

Corrosion Inhibition for Carbon Steel of Benzimidazole Derivatives Synthesized in Sulfuric Acid Solution

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Abstract: Synthesis three organic inhibitors for carbon steel corrosion: 2-(propylthio)-1H-benzo[d]imidazole (PTBI), 2-(allylthio)-1H-benzo[d]imidazole (ATBI) and 2-(prop-2-ynylthio)-1H-benzo[d]imidazole (YTBI) were prepared from reaction of 2-mercapto benzimidazole with different alkyl halide. The melting point and TLC were used to confirm the purity of the inhibitors as well as using the [FTIR, $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$] for the identify structures. The synthesized inhibitors were examined by potentiostatic polarization measurement as corrosion inhibitors of carbon steel in acidic media [1M H_2SO_4]. The polarization measurement results showed that the mixed type inhibitors. In addition, the efficiency of inhibitors (YTBI) were studied at different concentration and temperature levels (308-338) K. the result, showed that the high conc. records high inhibition efficiency which ranged from (60-79%).

Keywords: heterocyclic inhibitor, carbon steel, corrosion inhibition, sulfuric acid

1. Introduction

Steel is the most important engineering and construction material in the world [1]. The benefits of corrosion inhibitors is protects the metals surfaces which are existed exposure to the acidic media such as HCl and H_2SO_4 [2, 3]. In the literature there are many methods are used to protect the carbon steel from the corrosion. However, the most useful one is organic inhibitor under different conditions of acid, concentration and temperature lead to mitigating metallic corrosion [4, 5]. Heterocyclic inhibitor containing π bonds and heteroatom [P, S, N, O] used for this purpose [6, 7]. In many research used organic corrosion inhibitors due to its usefulness in various industries [8]. benzimidazole and its derivatives investigated as corrosion inhibition in various acid solutions [9-14]. The corrosion rate is reduce by adsorption organic inhibitors on the surface of the metal through formation blocking the active centers on the surface of the metal [15]. The inhibition performance of organic inhibitors is due to physisorption and/or chemisorption on the surface of the metals [16, 17].

2. Experimental

2.1. Instruments

A Gallen Kamp melting point apparatus used to measure melting points. Shimadzu FTIR-8300 spectrophotometer as KBr disc, result are given cm^{-1} and Bruker spectro spin ultra-shield magnets 300 MHz instrument, using DMSO-d_6 as solvent and TMS as internal reference used to identification the organic inhibitor. Polarization measurements were conducted using advanced potentiostatic winking MLab-200(2007) [Bank Elektronik-Intelligent controls GmbH] with all accessories.

2.2. Chemical

All starting chemical compounds were obtained from Fluka and Aldrich or BDH.

2.3. Synthesis of 2-(alkylthio)-1H-benzo [d] imidazole derivatives

2-mercaptobenzimidazole was synthesized according to reported procedure [18]. 2-mercaptobenzimidazole (1.502 g, 0.01 mole) was dissolved in dry $\text{C}_2\text{H}_5\text{OH}$ (25 ml) and added to a solution of KOH (0.5611 g, 0.01 mole) in dry $\text{C}_2\text{H}_5\text{OH}$ (15ml). After 30 min of stirring at room temperature, different alkyl halides (0.01mole) was added in (2-3) portions and the resultant suspension was reflux with stirring for (4-5) hrs. The reaction mixture was cooled and poured into ice bath with stirring a solid was precipitated and left for overnight. The solid was filtered off, washed with cold water and air dried. The crude products were purified by recrystallization from ethanol. Table (1) show the physical properties of the dry products.

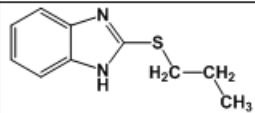
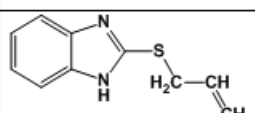
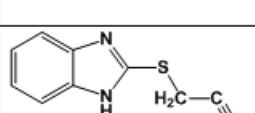
2.4. Polarization measurements

Carbon steel specimens with composition 0.086% C, 0.252% Mn, 0.003% P, 0.016% S and the remainder being Fe were used. Specimens of size 1.5cm diameter were used for polarization method. These samples were polished successively with belt grinding polishing machine then polished with emery papers of different grit (80, 150, 220, 240, 320, 400, 1000, 1200 and 2000) in sequence. After polishing, the specimens were washed with distilled water then acetone finally dried and kept in desiccator. The electrochemical measurements were performed in a typical three-compartment glass cell consisted of the carbon steel specimen as working electrode (WE), platinum counter electrode (CE), and a saturated calomel electrode (SCE) as the reference electrode. The reference electrode was connected to a Luggin capillary to minimize IR drop: Solutions were pre-

pared by diluted H_2SO_4 with distilled water; the carbon steel electrode was abraded with different grit emery papers, cleaned with acetone, washed with distilled water and finally

dried. The electrode potential was allowed to stabilize 15 min before starting the measurements.

Table 1: Physical properties and FTIR spectral of synthesized inhibitors

Comp: code.	Comp: structure	Melting point °C	Yield %	Color	Major FTIR Absorptions cm^{-1}				
					vN-H	vC-H arom.	vC-H Aliph.	vC=N	Other bands
PTBI		167-169	78	White	3220	3060	2964 2869	1625	----
ATBI		143-145	80	Off-white	3180	3047	2964 2860	1620	v=C-H: 3080 vC=C: 1655
YTBI		164-166	85	White	3200	3046	2975 2881	1622	v≡C-H: 3300 vC≡C: 2135

Tafel polarization curves were obtained by changing the electrode potential automatically from -200 mV versus open circuit potential (OCP) to +200mV versus OCP with scan rate of 2.0 mV s^{-1} . All experiments were conducted at $308 \pm 1 \text{ K}$ for different synthesized inhibitors at 100 ppm. Different concentrations (50,100,150 ppm) at 308-338 K were examined. Tafel lines of potential versus log (I) were plotted and corrosion current density (I_{corr}) and corrosion potential (E_{corr}) were determined in the absence and presence of inhibitor.

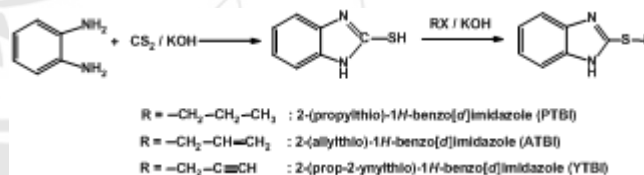
3. Results and discussion

3.1. Structure of Corrosion Inhibitors

Organic compounds containing (N, S atoms and functional group) prepared for used as corrosion inhibitors. The synthesis some of 2-(alkylthio) benzimidazole derivatives show in scheme (1). The route include condensation of the 2-mercaptobenzimidazole with different aliphatic alkyl halide and KOH in dry C_2H_5OH under reflux condition, and the end point of the reaction was examined by TLC. The chemical structures of (PTBI, ATBI and YTBI) were confirmed using FTIR spectroscopy listed in Table (1) for (PTBI) inhibitor as representative sample of the different inhibitors and showed the following spectra: $3220\text{-}3180 \text{ cm}^{-1}$ to vN-H, $3060\text{-}3046 \text{ cm}^{-1}$ to vC-H arom., $2975\text{-}2860 \text{ cm}^{-1}$ to vC-H aliph. and $1625\text{-}1620 \text{ cm}^{-1}$ to vC=N, (ATBI and YTBI) showed additional absorption band: 1655 cm^{-1} to vC=C alkenyl and 2135 cm^{-1} to vC≡C alkynyl. While the $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra data of inhibitors [19] δ ppm in $DMSO-d_6$ solvent are listed in Table (2). Other chemical test used to characterize the inhibitors (ATBI and YTBI) such as Br_2/CCl_4 and $AgNO_3$ alcoholic for terminal alkene and alkyne respectively [20].

Potentiodynamic evaluation

Polarization curves for carbon steel in H_2SO_4 solution (1M) in the absence and presence of 100 ppm of the (PTBI, ATBI and YTBI) at 308 K were shown in (Fig. 1).



Scheme (1): Synthetic route of 2-(alkylthio) benzimidazole inhibitors

The cathodic and anodic currents, corrosion current densities and corrosion potential were obtained at each inhibitor dose by the extrapolation of the linear parts of the anodic and cathodic current-potential curves. The electrochemical parameters including: corrosion potential, corrosion current, cathodic and anodic Tafel slopes of the corrosion process obtained from the polarization curves were listed in Table (3). As reflected from the graphs, the inhibitors exhibit a significant effect on the current-potential relations. The Tafel lines are shifted to more negative and more positive potentials for the cathodic and anodic processes relative to the blank curve. In presence of 100 ppm (wt. %) of the inhibitor, the anodic and cathodic Tafel slopes are shifted to more anodic and cathodic directions (Fig.1). This means that these additives influence both the cathodic and anodic processes. The corrosion potential (E_{corr}) remains almost constant at different inhibitors which indicate that the metal obtained an equilibrium potential at constant potential value. The value of the percentage inhibition efficiency (%P) and the extent of adsorption of inhibitor molecules onto metal surface in term of surface coverage (θ) were calculated using Eqs. 1 and 2 respectively [21].

$$\% P = [(I_{corr} - I_{corr(inh)}) / I_{corr}] \times 100 \dots\dots (1)$$

$$\theta = (I_{corr} - I_{corr(inh)}) / I_{corr} \dots\dots\dots (2)$$

Where I_{corr} and $I_{corr(inh)}$ are the corrosion current densities in the absence and in the presence of the inhibitors respectively.

Table 2: ¹H-NMR and ¹³C-NMR spectral data (ppm) for synthesized inhibitors

Comp: Code	Compound structure	¹ H-NMR signals (ppm) δ-H
		¹³ C-NMR signals (ppm) δ-H
PTBI		1.3(t,3H,-CH ₃); 1.6(m,2H,-CH ₂ -); 3.9(t,2H,-S-CH ₂ -); 7.1-7.3(m,4H,Ar-H); 12.6(b, 1H, NH).
		13.0(C ₁); 21.60 (C ₂); 38.6(C ₃); 118-128(C ₅ , C ₆ , C ₇); 140.20(C ₄).
ATBI		3.9 (d,2H,-S-CH ₂ -); 5.2(d,2H,=CH ₂); 5.9(m,1H,-CH=); 7.0-7.2(m,4H, Ar-H); 12.5(b, 1H, NH).
		33.86(C ₃); 46.61(C ₁); 49.73(C ₂); 110.29-121.61(C ₅ , C ₆ , C ₇); 135.0(C ₄).
YTBI		3.1(t, 1H, ≡C-H); 4.2(d, 2H,-CH ₂ -); 7.2-7.4(m, 4H, Ar-H); 12.4(b, 1H, NH).
		38.6(C ₃); 73.9(C ₁); 76.9(C ₂); 120-129(C ₅ , C ₆ , C ₇); 142.1(C ₄).

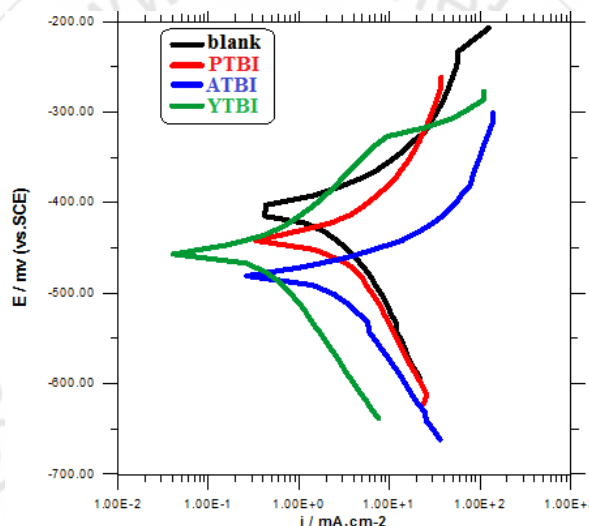


Figure 1: Polarization curves of carbon steel in 1M H₂SO₄ in the absence and presence of 100 ppm of the (PTBI, ATBI and YTBI) at 308 K

Table 3: Polarization parameters of carbon steel in the absence and presence 100 ppm for inhibitors at 308 K

Solution	E _{corr} Mv (SCE)	I _{corr} μA/cm ²	β _c mV/dec	β _a mV/dec	Weight loss g/m ² .d	Penetration loss mm/a	θ	% P
Blank	-410.4	1470	-107.6	72.2	368	17.9	-	-
PTBI	-443	1140	-57.9	61.8	286	13.3	0.2244	22.44
ATBI	-480.5	1030	-62.5	36.1	258	12	0.2993	29.93
YTBI	-456.1	335.84	-118.8	84.9	84	3.9	0.7715	77.15

Effect of inhibitor (YTBI) concentration

Effect of 2-(prop-2-ynylthio)-1H-benzimidazole (YTBI) was investigated in the concentration range (50–150) ppm. (Fig. 2) shows the Tafel polarization value of carbon steel immersed in 1M H₂SO₄ at 308 K with and without (YTBI) inhibitor. polarization parameters such as corrosion potential (E_{corr}), cathodic and anodic Tafel slopes (β_c and β_a) and corrosion current density (I_{corr}) were extracted by extrapolating the anodic and cathodic branches of Tafel curves at potentials beyond the region of E_{corr}±30 mV, using Softcorr III program and are listed in Table(4). From the results collected in Table (4), the corrosion rate is decreased by in-

creasing the inhibitor concentration and inhibition efficiency increased as shown in Fig. (3). Therefore, it can be concluded that they impede the corrosion by merely blocking the reaction sites on carbon steel surface without interfering the anodic and cathodic reactions. Moreover, this inhibitor cause no significant changes in the anodic and cathodic Tafel slopes, this suggest that this inhibitor behave as a mixed-type inhibitor. Indicating that the inhibitor can be classified as adsorptive-type [22].

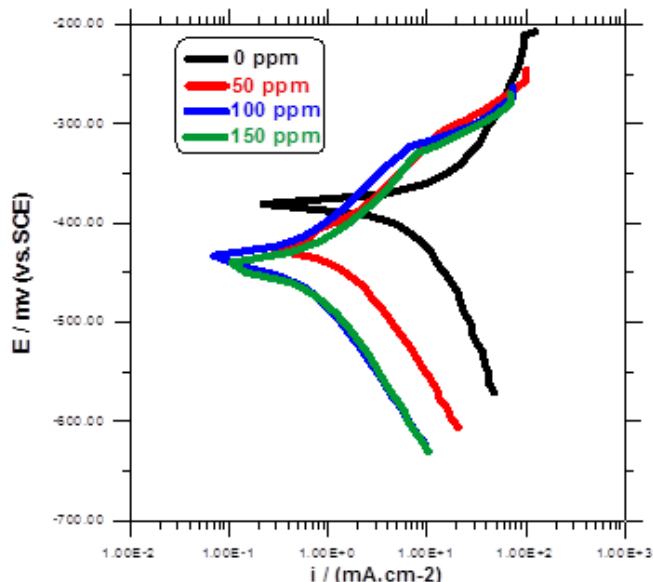


Figure 2: Polarization curves of carbon steel in the absence and presence of different concentrations of inhibitor (YTBI) at 308 K

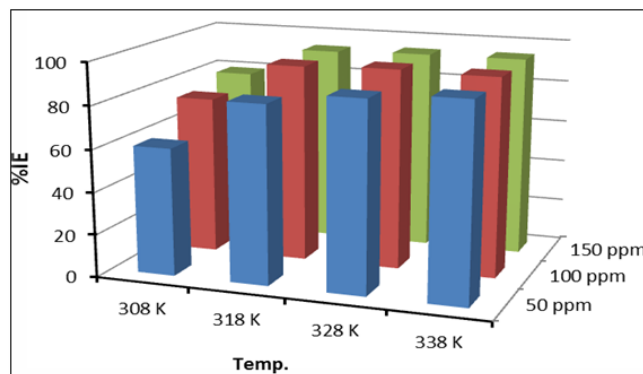


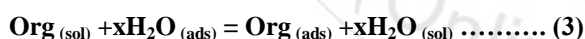
Figure 3: Relationship between inhibition efficiencies for inhibitor [YTBI] with various concentrations and different temperature for carbon steel in H₂SO₄ (1M)

Table 4: Polarization parameters of carbon steel in the absence and presence of different concentration for (YTBI)

Inhibitor conc. (ppm)	E _{corr} mV (SCE)	I _{corr} μA/cm ²	β _c mV/dec	β _a mV/dec	Weight loss g/m ² .d	Penetration loss mm/a	θ	% P
0	-410.4	1470	-107.6	72.2	368	17.9	-	-
50	-423.1	577.06	-119.8	69.4	144	67	0.6074	60.74
100	-456.6	367.40	-130.5	91.3	91.8	42.6	0.7500	75.00
150	-456.0	296.39	-112.0	89.0	74.1	3.44	0.7983	79.83

Adsorption isotherms

Basic information on the interaction between inhibitors and metal surface can be provided using the adsorption isotherms [23]. The adsorption of an organic adsorbate at metal-solution interface can occur as a result of substitution adsorption process between organic molecules presented in the aqueous solution (Org_(sol)), and the water molecules previously adsorbed on the metallic surface (H₂O_(ads)) [24]:



Where Org_(sol) and Org_(ads) are the organic species in the bulk solution and adsorbed one on the metallic surface, respectively, H₂O_(ads) is the water molecule adsorbed on the metallic surface and x is the size ratio representing the number of water molecules replaced by one organic adsorbate. In order to obtain the adsorption isotherm, the degree of surface coverage, θ, for different concentrations of inhibitor in 1M H₂SO₄ solution has been evaluated by equation (2). The θ values are presented in Table (5). According to the Langmuir's isotherm, the surface coverage (θ) is related to inhibitor concentration (C) by the following equation [25]:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \dots \dots \dots (4)$$

Where K_{ads} is the equilibrium constant of the inhibitor adsorption process. A straight line is obtained on plotting C/θ versus C as shown in Fig. (4). the linear correlation coefficient

(r²) is equal to (r² = 0.9974 - 0.9999) and the slope is very close to 0.99, indicating the adsorption of inhibitor (YTBI) on the carbon steel surface obeys the Langmuir adsorption isotherm. The high correlation factor (r²) of the Langmuir adsorption isotherm may confirm the validity of this approach. The equilibrium constant (K_{ads}) for the adsorption-desorption process of tested inhibitor can be calculated from reciprocal of the intercept. The adsorptive equilibrium constant (K_{ads}) values are listed in table (5). The most important thermodynamic adsorption parameters are the free energy of an adsorption (ΔG_{ads}) at different temperatures was calculated from the following equation [26]:

$$\Delta G_{ads} = -2.303RT \log(55.5K_{ads}) \dots \dots \dots (5)$$

Where R is the gas constant, T is the absolute temperature and the value 55.5 is the concentration of water in solution expressed in M, K_{ads} is the equilibrium constant of the inhibitor adsorption process. Values of ΔG_{ads} are listed in table (5). The values of ΔG_{ads} negative sign are usually characteristic of a strong interaction and a high efficient adsorption. Generally, values of ΔG_{ads} around -20 kJ mol⁻¹ or lower negative are consistent with the electrostatic interaction between the charged molecules and the charged metal (physisorption). Whereas, the more negative values than -40 kJ mol⁻¹ involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate type of bond (chemisorption) [27]. Calculated ΔG_{ads} values indicate that the adsorption mechanism of the synthesized inhibitor on carbon steel in 1M H₂SO₄ solution is physical adsorption

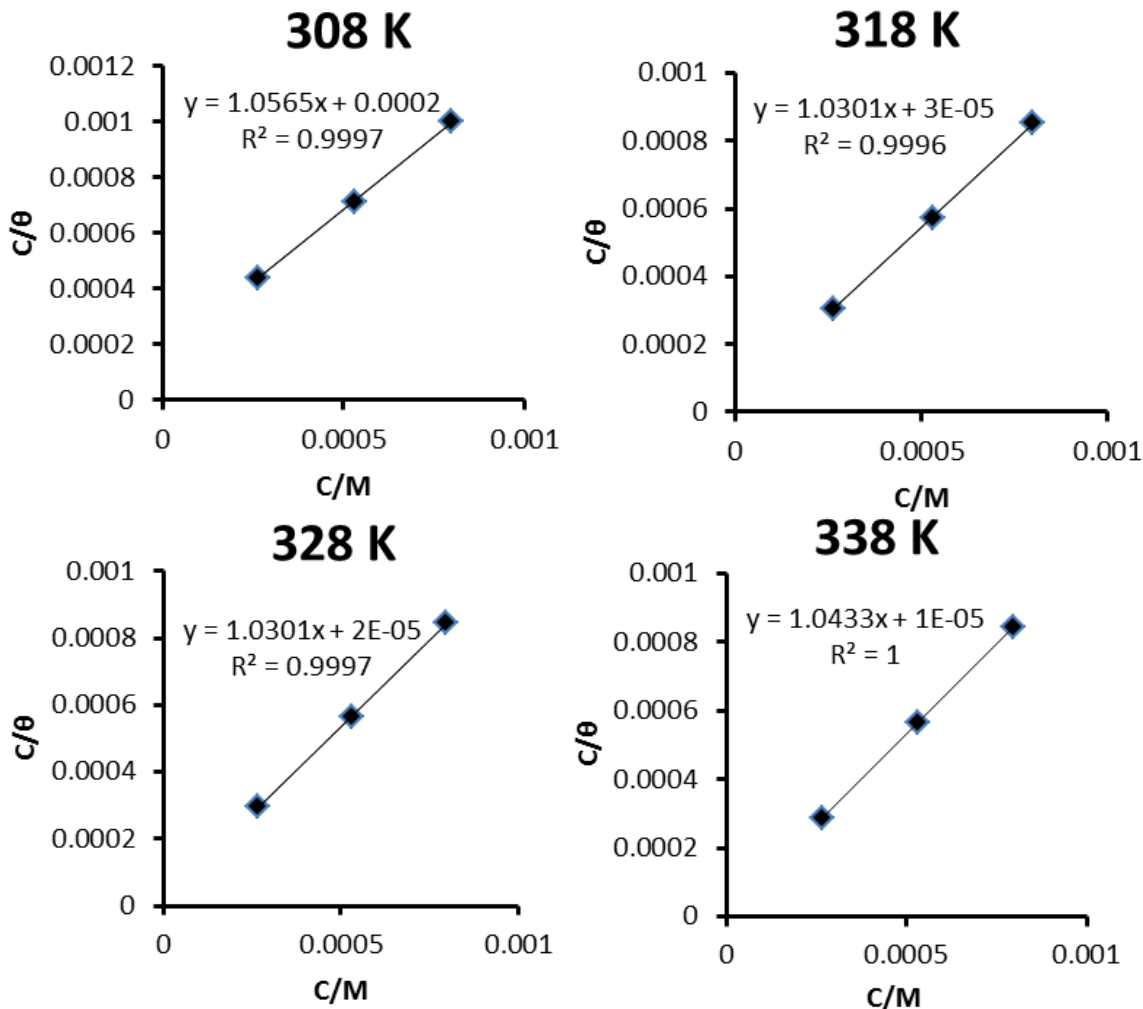


Figure 4: The Langmuir isotherm adsorption for carbon steel in the different concentrations of (YTBI) at (308-338) K

Table 5: Data of free energy of adsorption and the parameter of adsorption isotherm for carbon steel in different concentrations for (YTBI) at (308-338) K.

T/K	$(1/T) \cdot 10^{-3} \text{ K}^{-1}$	C/ppm	θ	K_{ads}	ΔG_{ads} KJ.mol ⁻¹ .k ⁻¹	r^2
308	3.2	50	0.6074	5000	-32.10	0.9997
		100	0.7500			
		150	0.7983			
318	3.1	50	0.8376	49.47	-20.93	0.9996
		100	0.9311			
		150	0.9330			
328	3.0	50	0.8913	74.21	-22.70	0.9997
		100	0.9426			
		150	0.9431			
338	2.9	50	0.9179	148.41	-25.34	1
		100	0.9361			
		150	0.9445			

Effect of temperature

In this part studied the change in the rate of the corrosion process with temperature in H₂SO₄ (1M) with and without various concentrations (50-150 ppm) for (YTBI) inhibitor t. The aim was to evaluate the apparent activation energy and the pre-exponential factor (A) of the corrosion process. The polarization exhibits Tafel behavior. The Polarization curves which are not presented here show that both the anodic and cathodic polarizations decrease upon heating while I_{corr} increases. Several electrochemical Parameters were calculated from these curves and the results are summarized in Table (6). I_{corr} increases upon heating both in uninhibited and inhibited solutions. The efficiency of YTBI increases with temperature in the studied temperature range. The fact that (%P) increases with temperature was considered by Ivanov [28] as due to change in the nature of the adsorption mode Fig. (3).The inhibitor is physically adsorbed at lower temperatures, while chemisorption is favored at higher temperatures.

Table 6: The effect of temperature on the corrosion parameters of carbon steel for different concentrations of (YTBI)

Conc. (ppm)	Temp: K	E_{corr} Mv (SCE)	I_{corr} $\mu A/cm^2$	β_c mV/dec	β_a mV/dec	Weight loss $g/m^2.d$	Penetration Loss mm/a	Θ	% P
0	308	-410.4	1470	-107.6	72.2	368	17.9	-	-
	318	-395.5	6160	-142.5	103.0	160	70.9	-	-
	328	-380.4	13350	-150.7	130.1	332	152	-	-
	338	-375.2	21450	-240.4	135.5	535	245	-	-
50	308	-423.1	577.06	-119.8	69.4	144	67	0.6074	60.74
	318	-420.5	1000	-125.1	98.5	251	11.7	0.8376	83.76
	328	-421.7	1450	-136.1	100.4	363	16.9	0.8913	89.13
	338	-423.2	1760	-100.7	100.3	441	20.5	0.9179	91.79
100	308	-456.6	367.40	-130.5	91.3	91.8	42.6	0.7500	75.00
	318	-434.8	424.42	-132.0	94.7	106	49.3	0.9311	93.11
	328	-437.4	765.73	-123.3	109.3	191	88.9	0.9426	94.26
	338	-425.6	1370	-134.6	123.9	341	15.9	0.9361	93.61
150	308	-456.0	296.39	-112.0	89.0	74.1	3.44	0.7983	79.83
	318	-443.5	412.22	-104.7	75.9	103	4.78	0.9330	93.30
	328	-436.7	758.45	-133.7	110.6	190	8.80	0.9431	94.31
	338	-435.9	1190	-128.8	116.4	298	13.9	0.9445	94.45

The kinetic model is another way to explain the mechanism of corrosion inhibition. The corrosion reaction can be regarded as an Arrhenius modified equation [23]:

$$\log i_{corr} = \log A - \frac{E_a^*}{2.303RT} \quad (6)$$

Where i_{corr} is the corrosion current density, E_a^* is the apparent activation energy of the corrosion reaction, R is the gas constant, T is the absolute temperature and A is the Arrhenius pre-exponential factor. Fig. (5) presents the Arrhenius plots of the natural logarithm of the current density $\log i_{corr}$ versus $1/T$, for 1M solution of H_2SO_4 , in presence and absence of different concentrations of (YTBI). Values of E_a^* and A for the corrosion reaction in the absence and presence of different concentrations of the (YTBI) inhibitor are calculated by a linear regression method and given in Table (7). It is found that the activation energy is lower in the presence of inhibitor than in its absence. Enthalpy and entropy of activation (ΔH^* and ΔS^*) are calculated from the transition state equation [29]. Values of ΔH^* and ΔS^* are calculated and listed in table (7).

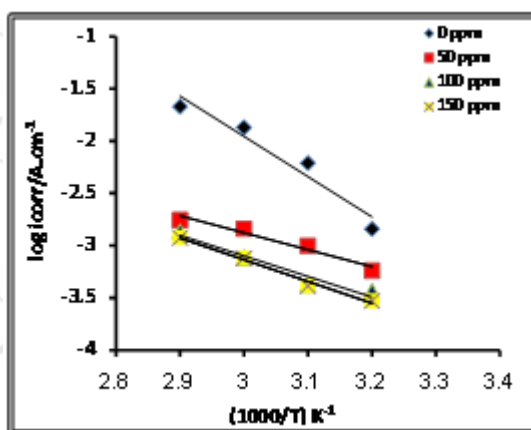


Figure 5: Arrhenius plots of $\log I_{corr}$ versus $1/T$ in presence and absence of different concentrations of (YTBI)

Table 7: The thermodynamic parameters of activation for (YTBI) at different concentrations

C ppm	A $A.cm^{-2}$	E_a^* $KJ.mol^{-1}$	ΔH^* $KJ.mol^{-1}$	ΔS^* $J.mol^{-1}.K^{-1}$
0	3.8×10^9	73.5	70.03	-955.75
50	9.35×10^1	30.9	28.72	-1095.38
100	6.58×10^2	37.8	35.04	-1080.66
150	1.25×10^3	39.8	37.53	-1073.98

E_a^* decreases with increasing (YTBI) concentration. It is obvious that concentration of the synthesized inhibitor is playing a role in decreasing the activation energy value, there by indicating a more efficient inhibiting effect. The decrease in E_a^* with the synthesized inhibitor concentration is typical of chemisorption process [30]. According to Eq. (6) low values of A and high values of E_a^* lead to lower corrosion rates. For the present study, E_a^* is lower in the presence of (YTBI). Therefore, the decrease in the steel corrosion rate is decided by the pre-exponential factor A, which reflects the effect of the variation of entropy ΔS^* during activation. It was found that A and consequently ΔS^* significantly decreased with inhibitor concentration reducing the corrosion rate of the steel. As a result, the corrosion rate of steel decreased with increasing inhibitor concentration. Clearly, the reduction of A is an important factor that determines the corrosion rate of steel in 1M H_2SO_4 in the presence of (YTBI). Generally one can say that the nature and the concentration of electrolyte affect greatly the activation energy for the corrosion process.

4. Conclusions

The inhibiting effect of 2-(prop-2-ynylthio)-1H-benzo[d]imidazole (YTBI) on carbon steel in H_2SO_4 (1M) was studied. The results are in good agreement and the main conclusions are:

- 1) The chemical structure of the synthesized inhibitors is identified by FTIR, 1H -NMR and ^{13}C -NMR.
- 2) The sequence (% P): YTBI > ATPI > PTBI.
- 3) The (% P) of YTBI increases with increase of inhibitor concentration.
- 4) YTBI was found to be the best inhibitor for carbon steel in H_2SO_4 (1M).
- 5) The results of the experiment lead to the suggestion of chemisorption of the inhibitor on the metal surface.

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