

Influence of bis-(2-benzothiazolyl)-disulfide on corrosion inhibition of mild steel in hydrochloric acid media

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Abstract The inhibiting behavior of bis-(2-benzothiazolyl)-disulfide on mild steel corrosion was evaluated in 1 M HCl solution. Weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) techniques were used in this study. In EIS technique, by performing Kramers–Kronig transformations, the experimental results validated, before fitting any theoretical model on them. The obtained results showed that the inhibition efficiency (%*IE*) increases by increasing the concentration of bis-(2-benzothiazolyl)-disulfide up to 2.02×10^{-4} M (%*IE*~62–68). Between the results obtained from various used techniques, a good agreement was found. Polarization curves indicate that the inhibition of the inhibitor is a mixed anodic–cathodic nature and Langmuir isotherm is found as an accurate isotherm describing the adsorption behavior. It also found that its inhibition mechanism of bis-(2-benzothiazolyl)-disulfide is at the interval of physical adsorption and chemical binding. The inhibition efficiency of the inhibitor decreased by rising of temperature in the range of 25–55 °C and these results, verified adsorption behavior of the inhibitor.

Keywords Mild steel · Corrosion inhibition · Bis-(2-benzothiazolyl)-disulfide

Introduction

In chemical industry, to remove the scales from the metallic surfaces, different types of organic inhibitors are used in acid

solutions, and corrosion inhibition of steel in these media has been extensively studied [1–10].

Among all acceptable inhibitors, many substituted Aazole compounds have been still studied in considerable details as effective corrosion inhibitors for steel in acidic media [11–15].

The previous works show that most organic inhibitors containing hetero atoms such as N, O, P, and S, triple bonds or aromatic rings act by adsorption on the metal surface [16–20]. Investigating and exploring new corrosion inhibitors for steel corrosion in acid solutions is important for its practical application. A study of about action of corrosion inhibitors is useful and necessary, both from point of view of the search for new inhibitors and also for their effective use [17].

In this work, the corrosion inhibition of bis-(2-benzothiazolyl)-disulfide ($C_{14}H_8N_2S_4$) on mild steel in 1 M HCl have been studied by using weight loss method, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) techniques. The selected organic compound which still has not been studied contains nitrogen and sulfur atoms and also aromatic rings.

In EIS technique, the experimental results can be validated, before fitting any theoretical model on them, by performing Kramers–Kronig transformations (KKTs). Kramers and Kronig developed a number of integral transforms between the real and the imaginary components of a complex transfer function. These integral transforms were derived assuming four basic conditions, which are Linearity, Causality, Stability, and Finiteness [21–24]. Hence, in this study, we applied the KKTs to evaluate the validity of the impedance data. This study is helpful in efforts to develop new inhibitors with good efficiency and also to further explain the mechanisms of the inhibition process.

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Experimental details

Material

The employed specimens were prepared from a mild steel with the chemical composition of (wt.%): 0.11 C, 0.25 Si, 0.26 Mn, 0.03 P, 0.03 S, and the remainder Fe. The specimens were polished with a sequence of emery papers of different grades (400–1,200), washed with double-distilled water and degreased in absolute ethanol and then dried.

The molecular structure of bis-(2-benzothiazolyl)-disulfide is given in Fig. 1. The employed concentration range of bis-(2-benzothiazolyl)-disulfide were from 5×10^{-5} to 4×10^{-4} M. The acid solutions (1 M HCl) used were made by dilution of analytical grade 37% HCl with double distilled water. The volume of test solutions for each experiment was 200 ml and the temperature of the solutions was controlled by thermostat.

Electrochemical measurements

Electrochemical experiments were carried out using an Autolab (PGSTAT 30). The measurements were carried out in a three electrodes electrochemical cell with a platinum counter electrode and a saturated calomel electrode as reference. The working mild steel electrode soldered with Cu-wire for electrical connection embedded in Araldite to offer its cross-sectional area (1 cm^2) in contact with the solution. Before experiments, working electrode was first immersed into the test solution for 30 min to establish a steady state open circuit potential.

EIS measurements were carried out in a frequency range of 30 kHz to 0.1 Hz using an amplitude of 5 mV peak to peak A.C. signal at the open circuit potential and in this method, inhibition efficiency (%IE) was calculated by using Eq. 1:

$$\%IE = \frac{R_{ct(\text{inh})} - R_{ct(\text{Blank})}}{R_{ct(\text{inh})}} \times 100 \quad (1)$$

Where $R_{ct(\text{Blank})}$ and $R_{ct(\text{inh})}$ are the charge transfer resistances of mild steel in blank and inhibited solution. As mentioned to validation of impedance data, the use of

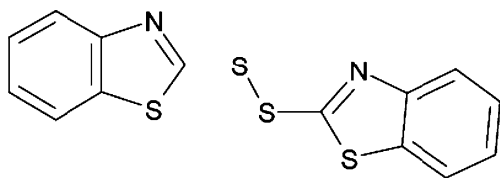


Fig. 1 Chemical structure of bis-(2-benzothiazolyl)-disulfide

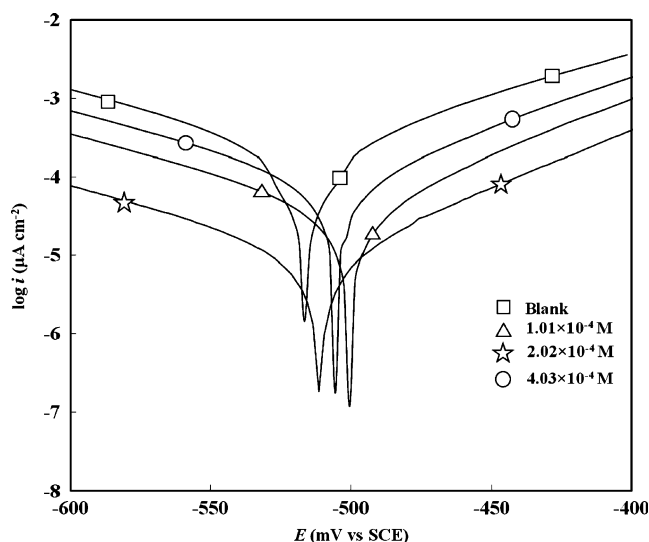


Fig. 2 Potentiodynamic polarization curves for the mild steel electrode in 1 M HCl solutions containing different concentrations of bis-(2-benzothiazolyl)-disulfide at 25 °C

KKTs is usual. KKTs in its most popular form, as applied to EIS data, are written as Eqs. 2 and 3 [22].

$$Z'(\omega) = Z'(\infty) + \left(\frac{2}{\pi}\right) \int_0^{\infty} \frac{xZ''(x) - \omega Z''(\omega)}{x^2 - \omega^2} dx \quad (2)$$

$$Z''(\omega) = -\left(\frac{2\omega}{\pi}\right) \int_0^{\infty} \frac{Z'(x) - Z'(\omega)}{x^2 - \omega^2} dx \quad (3)$$

Where ω is the frequency of transform, x is the frequency of integration, and Z' and Z'' are the real and imaginary components of the impedance, respectively. Equation 1 provide imaginary axis-to-real axis transformations, while Eq. 2 yields the real axis-to-imaginary axis transform and the average error (AE) was also calculated with Eq. 4 [22].

$$AE\%(Z) = \frac{100 \sum |Z_{ex}(\omega) - Z_{KKT}(\omega)|}{NZ_{ex,max}} \quad (4)$$

Where $Z_{ex}(\omega)$ and $Z_{KKT}(\omega)$ are the values for real or imaginary part observed experimentally and calculated by the appropriate KKT, respectively. $Z_{ex,max}(\omega)$ is the maximum value of the real or imaginary part in the experimental data set, and N is the total number of real/imaginary pairs.

The Tafel polarization scan was carried out by polarizing to ± 250 mV with respect to the OCP at a scan rate of 1 mV s^{-1} from the cathodic side. In polarization method, Stern–Geary equation [25] and Eq. 5 were used to calculate corrosion current (i_{Corr}) and the inhibition efficiency (%IE), respectively, where $I_{Corr(\text{Blank})}$ and $I_{Corr(\text{inh})}$ are the corrosion current density values of the

Table 1 Polarization parameters for mild steel in 1 M hydrochloric acid in the absence and presence of bis-(2-benzothiazolyl)-disulfide at various concentrations (at 25 °C)

Concentration (M)	$-b_c$ (mV dec ⁻¹)	b_a (mV dec ⁻¹)	R_p (Ω cm ²)	$E_{corr.}$ (mV)	$i_{corr.}$ (A cm ⁻²)	%IE
Blank	110	90	253	-584	8.51×10^{-5}	—
5.04×10^{-5}	106	88	452	-510	4.62×10^{-5}	45.7
1.01×10^{-4}	100	70	469	-501	3.80×10^{-5}	55.3
1.51×10^{-4}	99	75	616	-508	3.01×10^{-5}	64.7
2.02×10^{-4}	103	73	683	-511	2.71×10^{-5}	68.2
3.02×10^{-4}	96	75	416	-509	4.40×10^{-5}	48.3
4.03×10^{-4}	94	76	388	-505	4.71×10^{-5}	44.7

steel electrode in 1 M HCl solutions without and with the inhibitor, respectively.

$$\%IE = \frac{I_{Corr(Blank)} - I_{Corr(inh)}}{I_{Corr(Blank)}} \times 100 \quad (5)$$

The Polarization resistance (R_p) was calculated at over-voltage lower than ± 20 mV. Tafel slopes were calculated at over voltage more than ± 70 mV in the Tafel regions.

Weight loss measurements

The used mild steel specimens had rectangular form (length= 2 cm, width=1.5 cm, thickness=1 cm). The samples carefully weighted and immersed in non-stirring test solutions for 24 h and at the end, the specimens were washed in acetone, dried, and then weighed. Inhibition efficiency determined by using Eq. 6, where $W_{Corr(inh)}$ and $W_{Corr(Blank)}$ are the corrosion rates of mild steel with and without the inhibitor, respectively.

$$\%IE = \frac{W_{Corr(Blank)} - W_{Corr(inh)}}{W_{Corr(Blank)}} \times 100 \quad (6)$$

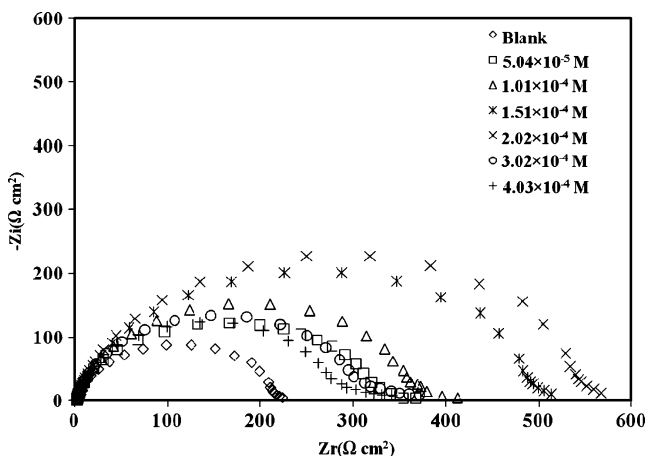


Fig. 3 Nyquist diagrams for the mild steel electrode in 1 M HCl solutions in the absence and presence of bis-(2-benzothiazolyl)-disulfide at various concentrations at 25 °C

Results and discussion

Polarization studies

Figure 2 shows the influence of bis-(2-benzothiazolyl)-disulfide concentration on the cathodic and anodic polarization curves of mild steel in 1 M HCl at 25 °C. In this figure, for better illustration, some of the polarization curves have been removed. The related corrosion parameters are illustrated in Table 1. Increasing the bis-(2-benzothiazolyl)-disulfide concentration leads to decreasing the current density values of the two branches of the polarization curves and consequently

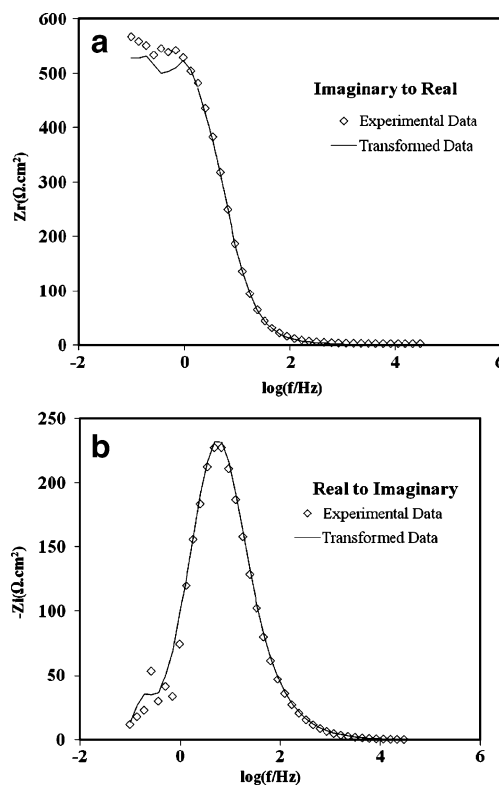


Fig. 4 Comparison of the experimental EIS data and KKT data of mild steel in the presence of 2.02×10^{-4} M of bis-(2-benzothiazolyl)-disulfide

Table 2 Values of %AE in transforming the experimental data by KKTs

Blank	5.04×10^{-5} (M)		1.01×10^{-4} (M)		1.51×10^{-4} (M)		2.02×10^{-4} (M)		3.02×10^{-4} (M)		4.03×10^{-4} (M)		
Z'	Z''	Z'	Z''	Z'	Z''	Z'	Z''	Z'	Z''	Z'	Z''	Z'	Z''
2.2	2.8	1.8	4.1	1.3	1.8	2.1	2.5	1.39	1.92	2.4	3.7	2.4	3.7

Table 3 Electrochemical impedance measurement of mild steel in 1 M Hydrochloric Acid in the absence and presence of bis-(2-benzothiazolyl)-disulfide at various concentrations (at 25 °C)

Concentration (M)	R_s (Ωcm^2)	CPE (F cm^{-2})	α	R_{CT} (Ωcm^2)	%IE
Blank	2.1	3.45×10^{-5}	0.87	224.8	–
5.04×10^{-5}	2.5	3.73×10^{-5}	0.89	367.8	38.9
1.01×10^{-4}	2.2	2.48×10^{-5}	0.89	412.2	45.4
1.51×10^{-4}	2.8	2.12×10^{-5}	0.92	513.2	56.2
2.02×10^{-4}	1.9	1.53×10^{-5}	0.94	584.2	61.5
3.02×10^{-4}	2.7	3.49×10^{-5}	0.89	371.6	39.5
4.03×10^{-4}	2.6	3.74×10^{-5}	0.89	341.8	34.3

the values of i_{corr} . Moreover, slight shifts were observed in E_{corr} values. This result reflects the diminution of both the cathodic hydrogen evolution reaction and anodic corrosion reaction with introducing the inhibitor in the aggressive solution. Decreasing the current density values of the two branches of the polarization curves leads to decreasing of the Tafel slopes. As a result bis-(2-benzothiazolyl)-disulfide is considered as a mixed-type inhibitor. The inhibition efficiency of bis-(2-benzothiazolyl)-disulfide increases with C_{inh} reaching its maximum value, 68.2%, at 2.02×10^{-4} M. Above this concentration, the %IE slightly decreases.

EIS studies

Nyquist plots for mild steel in 1 M HCl solution in the absence and presence of inhibitor at various concentrations of bis-(2-benzothiazolyl)-disulfide (at 25 °C) are shown in Fig. 3. Figure 4 shows the real and imaginary components of experimental EIS data of mild steel in the presence of 2.02×10^{-4} M of bis-(2-benzothiazolyl)-disulfide with corresponding

Table 4 Weight Loss results for mild steel in 1 M hydrochloric acid in the absence and presence of bis-(2-benzothiazolyl)-disulfide at various concentrations (at 25 °C)

Concentration (M)	Rate ($\text{gcm}^{-2}\text{h}^{-1}$)	%IE
Blank	3.42×10^{-3}	–
5.04×10^{-5}	1.94×10^{-3}	43.2
1.01×10^{-4}	1.71×10^{-3}	49.9
1.51×10^{-4}	1.32×10^{-3}	61.3
2.02×10^{-4}	1.16×10^{-3}	66.1
3.02×10^{-4}	1.87×10^{-3}	45.1
4.03×10^{-4}	2.05×10^{-3}	40.2

real to imaginary and imaginary to real KKTs data (calculated by Eqs. 2 and 3). Table 2 also shows the values of %AE in transforming the experimental data by KKTs for the all experienced concentration of bis-(2-benzothiazolyl)-disulfide (calculated by Eq. 4). As it can be seen, the value of %AE in all concentration is acceptable, and there are a relatively agreement between experimental EIS and KKTs data.

By considering Fig. 4 and Table 2, it can be resulted that the experimental data satisfy KKTs, and there is a relative stability in the system of experienced media. The semicircle Nyquist plots in Fig. 3 can be modeled by a simple “Randles” circuit including the “charge transfer resistance” (R_{ct}) parallel with constant phase element, CPE in series with solution resistance (R_s).

The appearance of the CPE element is often related to the electrode roughness or to the inhomogeneity in the

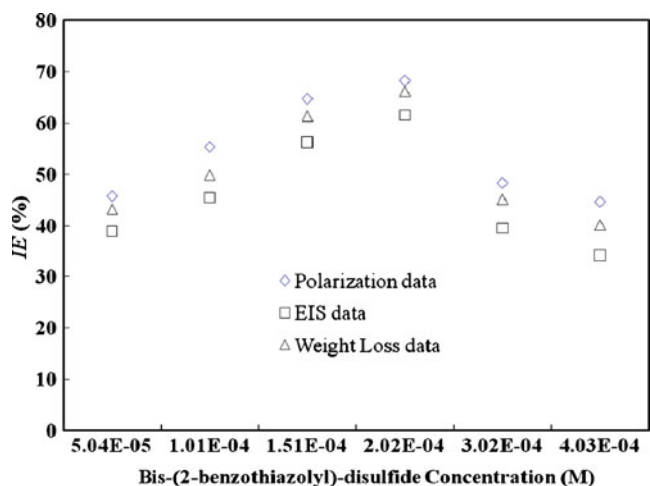
**Fig. 5** Comparison of weight loss, polarization, and EIS results of inhibitor efficiencies at various concentrations (at 25 °C)

Table 5 Comparison of weight loss, polarization, and EIS correlation coefficient (R^2) at various adsorption isotherms at 25 °C

Ads. Isotherm	Langmuir			Temkin			Flory-Huggins			Frumkin			
	Pol.	EIS	W.L	Pol.	EIS	W.L	Pol.	EIS	W.L	Pol.	EIS	W.L	
Method													
R^2	0.99	0.98	0.98	0.97	0.98	0.98	0.97	0.87	0.88	0.99	0.97	0.97	

WL weight loss, Pol. polarization

Table 6 The values of binding constant (K), free energy of adsorption (ΔG_{ads}) calculated using different methods for the bis-(2-benzothiazolyl)-disulfide at 25 °C

Method	Weight loss	Polarization	EIS
K	14,285	20,000	12,500
ΔG_{ads} (KJ mol ⁻¹)	-33.5	-34.4	-33.2

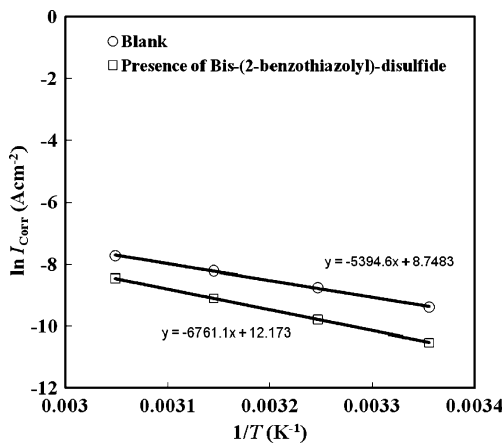


Fig. 6 Arrhenius slopes calculated from corrosion current density for mild steel in 1 M HCl in the absence and presence of the 2.02×10^{-4} M of bis-(2-benzothiazolyl)-disulfide at various temperatures

Table 7 Effect of temperature on the inhibition efficiency of bis-(2-benzothiazolyl)-disulfide (2.02×10^{-4} M) in 1 M hydrochloric acid without ($I_{Corr(Blank)}$) and with ($I_{Corr(inh)}$) the inhibitor

T (°C)	$I_{Corr(Blank)}$ (mAcm ⁻²)	$I_{Corr(inh)}$ (mAcm ⁻²)	%IE
25	8.51×10^{-5}	2.71×10^{-5}	68.2
35	1.59×10^{-4}	5.67×10^{-5}	64.3
45	2.74×10^{-4}	1.13×10^{-4}	58.7
55	4.46×10^{-4}	2.16×10^{-4}	51.6

conductance or dielectric constant [25, 26]. The CPE can be modeled as follows [3]:

$$Z_{CPE} = (j\omega C)^{-\alpha} \tag{7}$$

Where Z_{CPE} is the impedance, j the square root of -1 , ω the frequency, C the capacitance, and α is a measure of the non-ideality of the capacitor and has a value in the range of $0 < \alpha < 1$.

The quantitative results of equivalent circuits parameters (calculated by Zview program) for the bis-(2-benzothiazolyl)-disulfide, are given in Table 3. Inspection of the data in Fig. 3 and Table 3 for the bis-(2-benzothiazolyl)-disulfide reveals that the corrosion of mild steel was decreased in the presence of the inhibitor because the charge transfer resistance of mild steel was significantly increased. Increasing the concentration of bis-(2-benzothiazolyl)-disulfide increases R_{ct} and decreases the value of CPE and consequently enhances % IE until reaching their maximum values at 2.02×10^{-4} M ($R_{ct}=584.2$, %IE=61.5). With inhibitor concentration, increasing of charge transfer resistance may be result of more inhibitor molecules adsorption on the metal surface [4, 8].

Weight loss studies

Table 4 shows weight loss results for mild steel in 1 M HCl in the absence and presence of bis-(2-benzothiazolyl)-disulfide at various concentrations (at 25 °C), as shown the inhibition efficiencies increase with increasing inhibitor concentration and reaches to its maximum value at 2.02×10^{-4} M (%IE=66.1). The results of weight loss are agreement to EIS and Polarization results.

Adsorption isotherms and effect of temperature

Considering the inhibitor efficiencies at various concentrations (Fig. 5) revealed that the surface coverage values, θ ($\theta = \%IE/100$), increased with increasing concentration of the inhibitor. To describe bis-(2-benzothiazolyl)-disulfide adsorption behaviors, various adsorption isotherms [2, 3, 9] were used. Table 5 shows comparison of Weight Loss, Polarization, and EIS correlation coefficient (R^2) at various adsorption isotherms at 25 °C. Considering the results of Table 5 revealed the Langmuir kinetic-thermodynamic model fit the experimental data well for the inhibitors. This

isotherm assumes that the adsorbed molecules occupy only one site, and there are no interactions with other molecules adsorbed [2, 3]. The equilibrium constant of the process of adsorption (K) and free energy of adsorption (ΔG_{ads}) of inhibitor calculated and results are listed in Table 6. Equation 8 was used to calculate the ΔG_{ads} values.

$$\Delta G_{\text{ads}} = -RT \ln(55.5K) \quad (8)$$

The negative sign of ΔG_{ads} indicates that adsorption of the bis-(2-benzothiazolyl)-disulfide on the mild steel surface in 1 M HCl solution is proceeding spontaneously. The average value of ΔG_{ads} of adsorption process (Table 6) was calculated as $-33.7 \text{ kJ mol}^{-1}$ at the studied condition. Generally, the energy values of -20 kJ mol^{-1} or less negative are associated with an electrostatic interaction between charged molecules and charged metal surface, physisorption, those of -40 kJ mol^{-1} or more negative involve charge sharing or transferring from the inhibitor molecules to the metal surface to form a coordinate covalent bond, chemisorption [5, 7, 11]. The calculated $-33.7 \text{ kJ mol}^{-1}$ value is at the interval of physical adsorption and chemical binding and indicating the adsorption mechanism of the bis-(2-benzothiazolyl)-disulfide on mild steel in 1 M HCl was both electrostatic-adsorption and chemisorption. Iron is well known for its coordination affinity to hetero atoms (N, O, or S) bearing ligands [27]. By inspection of the chemical structure of bis-(2-benzothiazolyl)-disulfide (Fig. 1), the adsorption of the bis-(2-benzothiazolyl)-disulfide molecules on the mild steel surface is attributed to the donor acceptor interaction between π electrons of donor atoms N, S, and aromatic rings of inhibitors and the vacant d orbitals of iron surface atoms.

The corrosion inhibitor efficiency of bis-(2-benzothiazolyl)-disulfide was also studied as a function of temperature. Hence, polarization measurements were performed at temperatures range 25–55 °C in the absence and presence of the inhibitor ($2.02 \times 10^{-4} \text{ M}$, %IE=68.2) and the values of obtained polarization parameters (polarization curves are not show) are given in Table 7. Regarding to the Arrhenius equation, activation energy of metal dissolution can be calculated from the slope of Eq. 9 [1].

$$\ln I_{\text{Corr}} = \ln A - \frac{E_a}{RT} \quad (9)$$

The calculated activation energies in 1 M HCl with the absence and presence of bis-(2-benzothiazolyl)-disulfide were 44.8 kJ/mol and 56.2 kJ mol⁻¹, respectively (Fig. 6). Generally, the energy barrier of corrosion reaction increases with the presence of the inhibitors and could be interpreted by physical adsorption, which occurs in the first stage. Unchanged or lowered activation energy value of corrosion process in the

presence of inhibitor is linked to the existence of chemisorptions. However, it is accepted that mixed adsorption should be characterized by minor or no changes in activation energy [14, 25]. These results propose that physical adsorption occurring in the first stage explains the nature of organic molecule–metal interactions. Temperature can modify the interaction between the steel electrode and the acidic media without and with the inhibitors [1, 25, 28] and changes the adsorption inhibitor tendency toward to chemical adsorption mechanism.

Conclusions

Weight loss, Polarization and EIS were used to study the corrosion inhibition of bis-(2-benzothiazolyl)-disulfide on the mild steel in 1 M HCl solutions and the following conclusions can be deduced:

- The inhibition efficiency of bis-(2-benzothiazolyl)-disulfide increases with concentration to attain a maximum value of 62–68% at $2.02 \times 10^{-4} \text{ M}$.
- The inhibition efficiency of the inhibitor decreased by rising of temperature in the range of 25–55 °C.
- Bis-(2-benzothiazolyl)-disulfide is a cathodic and anodic (mixed type) inhibitor and obeys from Langmuir kinetic-thermodynamic model.
- Considering the values of free energy of adsorption, proposed the mixed physical adsorption and chemical binding adsorption for the inhibition mechanism of the inhibitor.
- The change in the activation energy value of corrosion process of mild steel in presence of bis-(2-benzothiazolyl)-disulfide was minor, and it seems adsorption mechanism is mixture of physical adsorption and chemical binding. This result is in good agreement with the result of free energy of adsorption.

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