

Synthesis and Characterization of Oligomer Bis(*trans*-2,3-dibromo-4-hydroxy-2-butenyl)terephthalate as a Green Corrosion Inhibitor on Mild Steel in 1 M H₃PO₄ Solution

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Abstract: Poly(ethylene terephthalate) (PET) waste was depolymerized by *trans*-2,3-dibromo-2-butene-1,4-diol in the presence of manganese acetate as a catalyst using microwave irradiation as opposed to the conventional heating process in order to reduce the time required for PET depolymerization. The depolymerization product bis(*trans*-2,3-dibromo-4-hydroxy-2-butenyl)terephthalate (BDBHBT) was isolated, characterized, and evaluated as a green inhibitor for mild corrosion steel in corrosive 1 M H₃PO₄ medium. This product was characterized using FTIR and ¹H-NMR. The effects of immersion time, inhibitor concentration, and reaction temperature were studied. The chemical technique utilized in this study was weight loss, while the electrochemical technique employed an open circuit potential. With 0.6 g/L of BDBHBT inhibitor, the highest corrosion inhibition efficiency of 83.3% was observed. The kinetic and thermodynamic functions were calculated, and the results indicated that the investigated inhibitor was physically adsorbed on the surface and confirmed to the Langmuir adsorption isotherm. This study aims to lessen pollution of the environment by transforming PET waste to a beneficial oligomer BDBHBT and estimating the inhibitory effect of this product on the corrosion of mild steel in 1 M H₃PO₄.

Keywords: poly(ethylene terephthalate) waste; green corrosion inhibitor; phosphoric acid; weight loss; open circuit potential

■ INTRODUCTION

Poly(ethylene terephthalate) (PET) is an important polymer in the world of plastics and packaging due to its most significant applications [1-2]. Due to its non-biodegradability in the environment, it is widely used in thermoplastics, which generate tons of waste that would destabilize the ecosystem equilibrium; therefore, it is an excellent material for recycling [3-4]. Recycling PET is one of the most successful and widespread examples of polymer recycling, which has become the most important process from an ecological standpoint and provided a business opportunity due to the widespread use of PET bottles, fibers, and packaging [5-6]. PET waste can be recycled through various processes, including chemical and physical recycling. Chemical recycling can be defined as the reaction of PET with different chemicals to produce chemical industry-relevant products [7]. Various chemical

recycling methods for PET, including glycolysis, methanolysis, hydrolysis, ammonolysis, aminolysis, and hydrogenation, have been used to investigate the chain scission-induced degradation of PET's main chain [8-9]. Nathaniel Wyeth patented the PET container in 1973, and it became widely used in the 1980s to make throwaway soft drink bottles. In 1987, over 700 million pounds of PET were utilized to make them [10].

Besides, corrosion is the deterioration of metals and alloys in corrosive environments. Constantly occurring in nature, the corrosion reaction process of metals is one of the primary concerns of the gas and oil industry. Globally, the annual economic loss caused by corrosion is enormous; thus, controlling and reducing the cost of this economic issue costs billions of dollars [11-12]. Corrosion protection of metal constructions is therefore considered a very important subject. Acidic solutions are

utilized extensively to clean, descale, pick, and acidify oil wells. Inhibitors of corrosion are necessary to prevent corrosion of metal alloys in these acid solutions [13].

Phosphoric acid has numerous industrial uses and it is compatible with a great deal of chemical and petroleum apparatus [14-15]. The majority of this apparatus is composed of mild steel that is susceptible to corrosion when exposed to acid. For this reason, corrosion inhibitors are used to shield mild steel from different forms and corrosive conditions, and the presence of these inhibitors decreases the corrosion rate of steel, thereby increasing its lifetime [16-17]. The majority of efficient acid inhibitors are organic compounds with oxygen, nitrogen, and/or sulfur, and these compounds bind on the surface of the steel to inhibit corrosion [18-19].

This is one of a series of works aimed at mitigating environmental contamination by transforming waste PET into beneficial materials and evaluating the inhibitory effect of these useful products on the corrosion of certain metals and alloys in various aqueous media. In this study, *trans*-2,3-dibromo-2-butene-1,4-diol was used to convert PET waste into the oligomer bis(*trans*-2,3-dibromo-4-hydroxy-2-butenyl)terephthalate (BDBHBT). The product (oligomer) was investigated as a green inhibitor for the corrosion of mild steel in 1 M H₃PO₄.

■ EXPERIMENTAL SECTION

Materials

After removing covers and labels, used PET bottles were collected, washed, and dried before being reused. These bottles have been split into tiny chips (6 × 6 mm²). The materials *trans*-2,3-dibromo-2-butene-1,4-diol and manganese acetate were purchased from Sigma Aldrich Chemical Company in England. The corrosive solution (1 M H₃PO₄) was made by diluting H₃PO₄ of analytical grade with double-distilled water. Specimens of mild steel were used in all experiments; these specimens contained metals listed in Table 1.

Instrumentation

Using Fourier transform infrared spectral data recorded over the 400–4000 cm⁻¹ wavenumber range, a Bruker FTIR-spectrometer was utilized to determine the chemical's structure. The prepared substance was thawed by DMSO-*d*₆ and investigated with a ¹H-NMR spectrometer model Bruker spectrophotometer (500 MHz) as an additional spectroscopic method for determining the chemical structure.

Procedure

Microwave depolymerization of PET: The reaction of PET with *trans*-2,3-dibromo-2-butene-1,4-diol

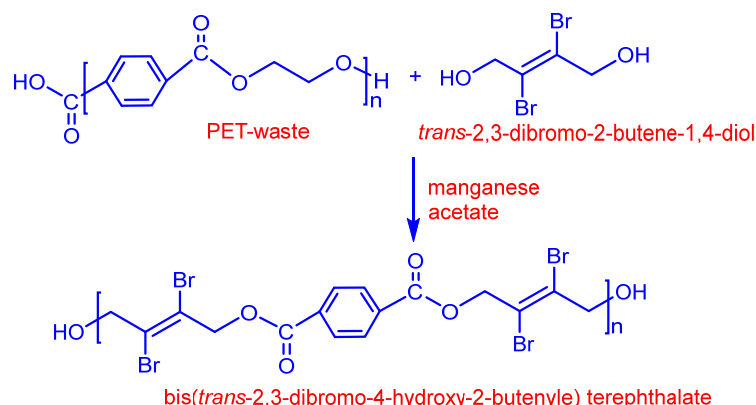
PET waste (1 g) was depolymerized with *trans*-2,3-dibromo-2-butene-1,4-diol (7.37 g) using 0.5% manganese acetate (0.005 g). PET was depolymerized using an electromagnetic source for microwave generation oven (LG, 2.45 GHz, maximal power: 900 W). The mixture was introduced into a reaction vial with a loose stopper, which was then placed in a microwave reactor. The reaction was permitted to continue with 450 W for a prolonged duration of time (30 min). This mixture was permitted to cool at room temperature. At the end of the reaction, an excess amount of distilled water was added to the mixture while vigorously stirring. The mixture was then separated. The collected filtrate was stored in a refrigerator for 24 h. Black crystalline powder was obtained in the filtrate, indicating that an oligomer BDBHBT had precipitated from the product, and it was separated and desiccated [20-21], yielding 4 g. The mechanism was illustrated in Scheme 1.

Weight loss measurements

Mild steel coupons of 4 × 8 × 0.1 cm dimensions were used as test specimens. Emery paper was used manually to clean them, degreased with acetone, rinsed with distillate water, and dried. The described treatment was performed promptly prior to each measurement.

Table 1. Contents of mild steel specimens

Element	C	Mn	P	Mo	Al	Sn	V	Nb	Ni	Cu	Fe
Weight (%)	0.170	0.057	0.011	0.022	0.011	0.005	0.004	0.007	0.027	0.043	Balance



Scheme 1. Preparation of bis(*trans*-2,3-dibromo-4-hydroxy-2-butenyl)terephthalate (BDBHBT)

Weight loss studies were conducted on specimens of mild steel submerged in 1 M H_3PO_4 medium in various concentrations of inhibitor (0, 0.1, 0.2, 0.4, and 0.6 g/L) at different temperatures (303, 323, and 333 K) for different immersion time (2, 4, and 8 h). The corrosion rate and efficiency of the inhibitor were determined based on weight loss for every coupon of every test configuration in every group. Corrosion rate (CR) is calculated from the change in weight of specimens in mils per year (mpy) using the relationship [22-23], Eq. (1):

$$\text{Corrosion rate (mpy)} = \frac{534 \cdot W}{A \cdot d \cdot t} \quad (1)$$

W is weight loss due to corrosion (mg), d is specimen's density (mg/cm^3), A is exposed surface area (in^2), and t is time of the immersion (h).

Regarding inhibitor efficiency (%IE) and surface coverage (θ), Eq. (2) and (3) are utilized [24-25]:

$$\%IE = \frac{W_0 - W}{W_0} \times 100 \quad (2)$$

$$\theta = \frac{W_0 - W}{W_0} \quad (3)$$

where W_0 , W indicate loss of weight the metal without and with inhibitor, respectively.

Adsorption mechanism for inhibitor on metal sample

In order to understand the relationship between an inhibitor (adsorbate) and an adsorbent surface, adsorption isotherms were utilized. Using the Langmuir, Freundlich, and Temkin adsorption isotherms, the mechanism of adsorption for inhibitor molecules on mild steel was predicted. Optimal adsorption isotherm was determined by using the equations of linear for their

models, plotting their individual graphs with the appropriate parameters, and comparing the R^2 values calculated from the line of best-fit plots for the three isotherms.

Open circuit potential measurements

The potential of a mild steel electrode was measured against a saturated calomel electrode (SCE) in a 1 M H_3PO_4 solution as a function of time of immersion without and with various concentrations of BDBHBT derived from PET waste until open circuit potential was reached. The open circuit potential test lasts a total of 30 min, allowing the electrochemical test system to reach equilibrium.

RESULTS AND DISCUSSION

Characterization for BDBHBT Inhibitor

From FTIR spectra, the structure of the obtained compound, BDBHBT was confirmed. The presence of a strong band at 3428 cm^{-1} in the spectra of BDBHBT in Fig. S1 indicates that the product terminates with hydroxyl groups. An absorption band at 3031 cm^{-1} corresponds to aromatic $=\text{C}-\text{H}$ stretching, whereas the peaks at 893 and 723 cm^{-1} correspond to aromatic and olefinic $=\text{C}-\text{H}$ out-of-plane bending, respectively. The other peak at 2972 cm^{-1} is determined to be stretching of vibration aliphatic $\text{C}-\text{H}$. The presence of peaks at 1722 and 1267 cm^{-1} are ascribed to stretching vibration of $\text{C}=\text{O}$ ester and stretching vibration of $\text{C}-\text{O}$ ester, while the peaks at 1507 and 1408 cm^{-1} indicate stretching of $\text{C}=\text{C}$, and the peak at 607 cm^{-1} correspond to $\text{C}-\text{Br}$ bending. Fig. S2 shows the $^1\text{H-NMR}$ of BDBHBT, the

peaks at 8.03, 8.05, 8.08, 8.10, and 8.13 ppm are imputed to protons of the aromatic ring, while the peak at 4.85 ppm is singlet attributed to the terminal O-H group. At the same time, 4.59 and 3.85 ppm are attributed to H-atoms of methylene groups nearer to carbonyl ester and O-H group, respectively. Whereas a peak at 2.50 ppm is assigned to the solvent DMSO- d_6 .

Weight Loss Measurements

Effect of immersion time and inhibitor concentration on the corrosion process

Table 2 shows the effect of increasing the immersion duration in 1 M H_3PO_4 containing the BDBHBT green inhibitor on the weight loss rate (W) and rate of corrosion (CR) of mild steel at various concentrations and temperatures. In general, as immersion time increased at a particular temperature, the weight loss of steel increased, and consequently, the corrosion rate decreased. As shown in Fig. 1-3, the relationship between weight loss and time in uninhibited and inhibited 1.0 M H_3PO_4 is linear. This demonstrates that during corrosion, there are no insoluble surface films. In this instance, an inhibitor is initially adsorbed on the surface of a metal,

and then it inhibits corrosion either by blocking the reaction sites (anodic and cathodic) or by changing the mechanism of anodic and cathodic partial processes [26-27]. In addition, at temperatures 303, 323, and 333 K, an increase in BDBHBT inhibitor concentration led to a decrease in the weight loss and corrosion rate. Thus, a BDBHBT inhibitor concentration of 0.6 g/L resulted in

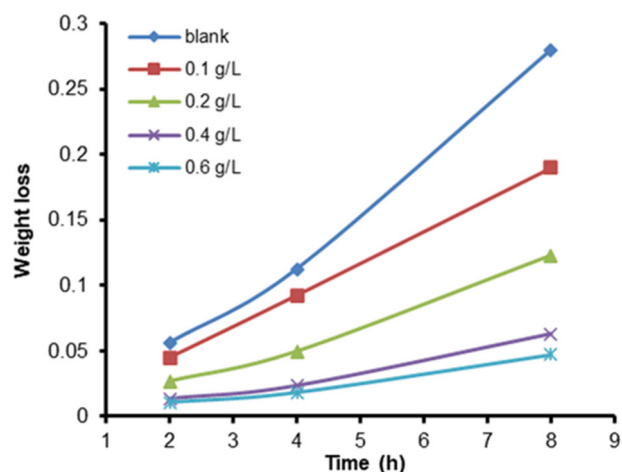


Fig 1. Curves of weight loss-time of mild steel in 1 M H_3PO_4 without and with various concentrations of PET waste-derived BDBHBT inhibitor in 303 K

Table 2. Weight loss and corrosion rate of steel in 1.0 M H_3PO_4 in the presence and absence of various concentrations of BDBHBT in various temperatures

Conc. g/L	T (K)	Time (h)					
		2		4		8	
		W (g)	CR (mpy)	W (g)	CR (mpy)	W (g)	CR (mpy)
blank	303	0.0562	401.6000	0.1125	330.0900	0.2799	333.2300
0.1		0.0449	320.3400	0.0924	254.0800	0.1898	224.3000
0.2		0.0269	195.9800	0.0498	151.0900	0.1231	144.8000
0.4		0.0134	97.1080	0.0234	69.4360	0.0632	62.0430
0.6		0.0109	89.0050	0.0185	66.0950	0.0475	55.0090
blank	323	0.0633	452.0600	0.1345	365.1100	0.3861	459.7600
0.1		0.0450	393.0000	0.0970	309.0000	0.2776	304.8000
0.2		0.0335	243.7900	0.0621	178.0000	0.1914	177.6000
0.4		0.0183	132.7100	0.0389	95.0090	0.0989	93.4000
0.6		0.0112	128.2300	0.0256	91.1900	0.0849	89.9000
blank	333	0.0694	495.0000	0.1566	462.1100	0.4335	531.9000
0.1		0.0552	465.6000	0.1339	406.8700	0.3147	395.8000
0.2		0.0395	287.2000	0.0745	230.4500	0.2167	227.2000
0.4		0.0201	182.0900	0.0413	150.2200	0.1611	148.6000
0.6		0.0172	197.8200	0.0368	167.0500	0.1287	163.0000

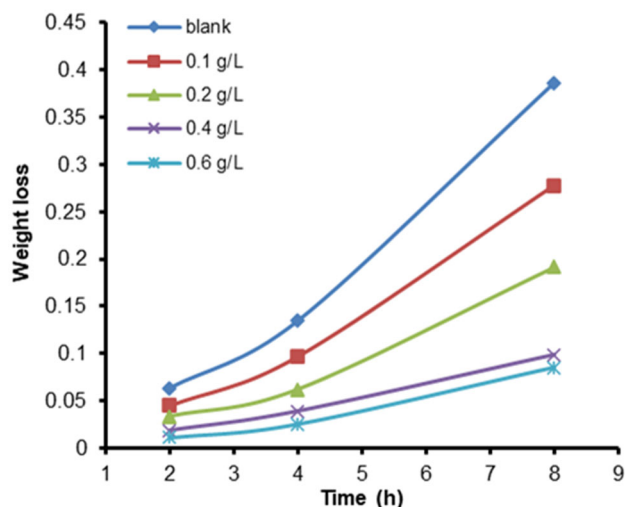


Fig 2. Curves of weight loss-time of mild steel in 1 M H_3PO_4 without and with various concentrations of PET waste-derived BDBHBT inhibitor in 323 K

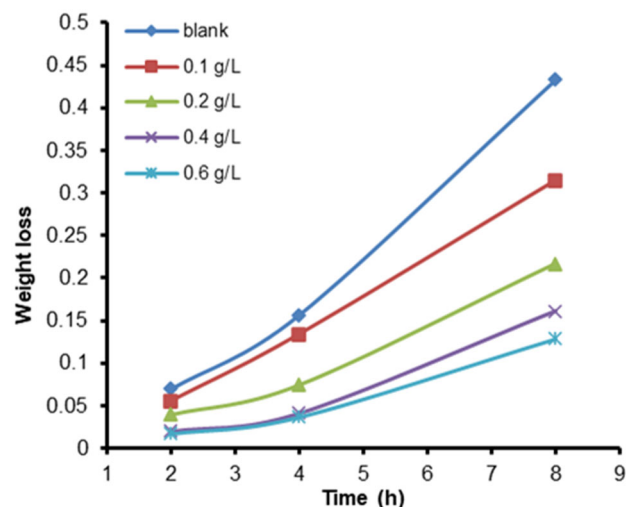


Fig 3. Curves of weight loss-time of mild steel in 1 M H_3PO_4 without and with various concentrations of PET waste-derived BDBHBT inhibitor in 333 K

Table 3. Effect of temperature on the rate of corrosion and effectiveness of preventing corrosion for mild steel in 1 M H_3PO_4 with various concentrations of BDBHBT inhibitor produced from PET waste for 8 h

Conc. (g/L)	T (K)	CR (mpy)	%IE	θ
blank		333.2300	-	-
0.1		224.3000	32.20	0.3220
0.2	303	144.8000	56.02	0.5602
0.4		62.0430	77.42	0.7742
0.6		55.0090	83.30	0.8330
blank		459.7600	0.00	0.0000
0.1		304.8000	28.10	0.2810
0.2	323	177.6000	50.42	0.5042
0.4		93.4000	74.38	0.7438
0.6		89.9000	78.01	0.7801
blank		531.9000	0.00	0.0000
0.1		395.8000	27.40	0.2740
0.2	333	227.2000	50.00	0.5000
0.4		148.6000	62.83	0.6283
0.6		163.0000	70.31	0.7031

the least amount of weight loss at each temperature considered. As the weight loss decreased as the concentration of the inhibitor increased, this indicated that the inhibitor was effective at inhibiting corrosion reactions.

Effect temperature on inhibition efficiency

Table 3 shows the results of CR and %IE of the inhibitor BDBHBT on mild-steel specimens when these

specimens were exposed to an acidic solution of 1 M H_3PO_4 for 8 h at various concentrations of inhibitor BDBHBT and temperatures. It is clear that as the temperature went up, the values of inhibition efficiency went down. That is, when the temperature went up, the rate of corrosion went up, which made the inhibitors less effective and sped up the corrosion process. This was because the molecules of the reactants had more kinetic

energy at higher temperatures, which made them move and crash into each other more quickly. This increased the rate of oxidation, or corrosion [28]. Fig. 4 shows how temperature affects the effectiveness of an inhibitor BDBHBT in the presence of different concentrations of the inhibitor. Fig. 5 shows how temperature affects the rate of corrosion in steel without and with different concentrations of the inhibitor.

Kinetic Study of the Corrosion of Steel in 1 M H₃PO₄

Thermodynamic parameters are essential for comprehending the inhibitive mechanism, activation energy (E_a^*) and thermodynamic functions of activations like activation enthalpy (ΔH_a^*) and activation entropy (ΔS_a^*) are studied. Thus, activation energy is calculated according to Arrhenius, Eq. (4):

$$CR = A \exp \frac{-E_a^*}{RT} \quad (4)$$

A represents the pre-exponential factor. Arrhenius plots for the corrosion of mild steel in 1 M H₃PO₄ (Fig. 6), slopes of these linear plots allow us to calculate E_a^* values, which are stated in Table 4. The results showed that the E_a^* value of the inhibited solutions was greater than that of the blank solution; this indicates that the corrosion process became more difficult in the presence of BDBHBT by increasing the energy barrier for the corrosion process [29-30]. Thermodynamic functions of

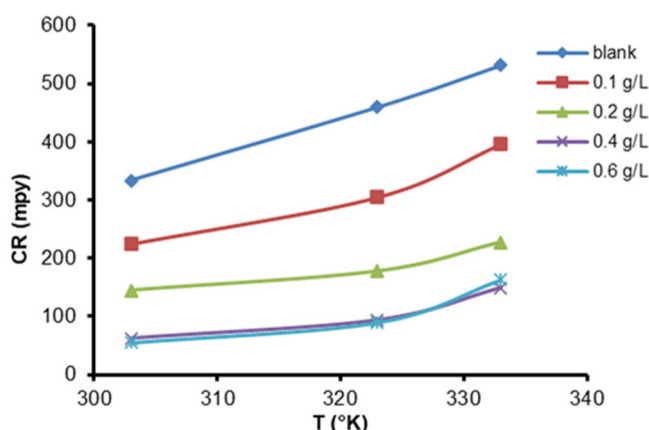


Fig 5. Relation between rate of corrosion and temperature for steel in 1 M H₃PO₄ with various concentrations of BDBHBT obtained from PET during 8 h

activation, such as ΔH_a^* and ΔS_a^* , can be determined using Eq. (5) of the transition state:

$$CR = \left(\frac{RT}{Nh} \right) \exp \left(\frac{\Delta S_a^*}{R} \right) \exp \left(\frac{-\Delta H_a^*}{RT} \right) \quad (5)$$

The plot of $\log (CR/T)$ versus $1/T$ of mild steel in 1 M H₃PO₄ solution with various concentrations of BDBHBT yields straight lines, as depicted in Fig. 7. The positive indications ΔH_a^* indicate the endothermic character and mean that mild steel corrosion is difficult in the presence of a BDBHBT inhibitor [31-32]. Whereas

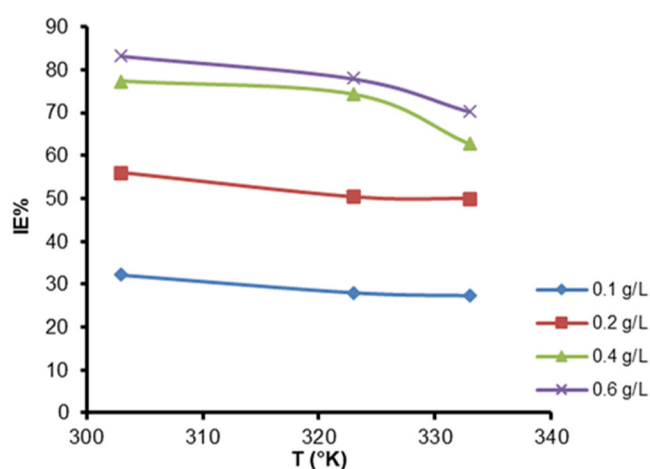


Fig 4. Relationship between percentage inhibition efficiency and temperature for steel in 1 M H₃PO₄ with various concentrations of BDBHBT obtained from PET during 8 h

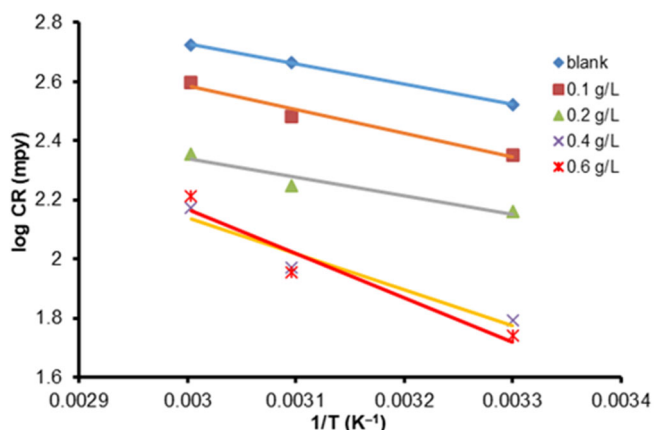


Fig 6. Arrhenius plots for mild steel after 8 h in corrosive solution in the absence and presence of different concentrations of BDBHBT

Table 4. Parameters of thermodynamic activation of adsorption of steel in 1 M H₃PO₄ without and with different concentrations of BDBHBT inhibitor

Conc. (g/L)	E _a [*] (kJ/mol)	ΔH _a [*] (kJ/mol)	ΔS _a [*] (kJ/mol K)
Blank	13.08	10.45	-0.16
0.1	15.33	12.69	-0.16
0.2	11.90	9.26	-0.17
0.4	23.15	20.52	-0.14
0.6	28.68	26.04	-0.13

ΔS_a^{*} value is higher and negative, this indicates that the activated complex favors an association step over a dissociation step, resulting in a reduction of the disorder [33-34].

Results of Adsorption Isotherm of Corrosion Process

A mechanism for corrosion inhibition in the phosphoric acid solution can be explained by using an adsorption isotherm. The adsorption process findings demonstrated that the plot of the Langmuir isotherm model was the best appropriate isotherm for describing the adsorbed BDBHBT on a mild steel surface and gave R² values closer to 1, as shown in Fig. 8. The linear equation of this model is, Eq. (6) [35]:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \tag{6}$$

where C is the amount of inhibitor and K_{ads} is the adsorption equilibrium constant, and θ is the amount of

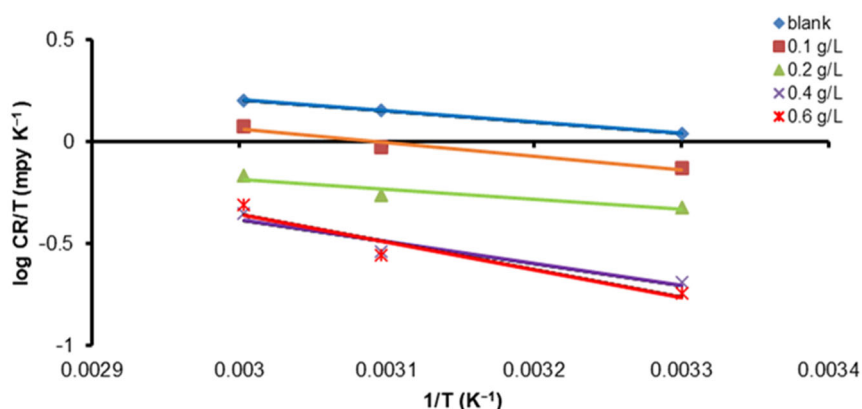


Fig 7. Transition state for mild steel after 8 h in corrosive solution in the absence and presence of different concentrations of BDBHBT

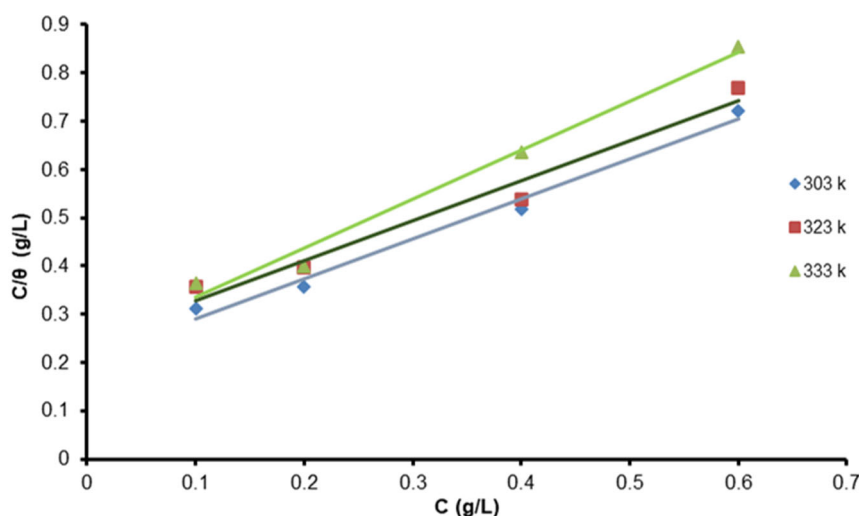


Fig 8. Langmuir isotherm of BDBHBT on steel in 1 M H₃PO₄ in various temperatures

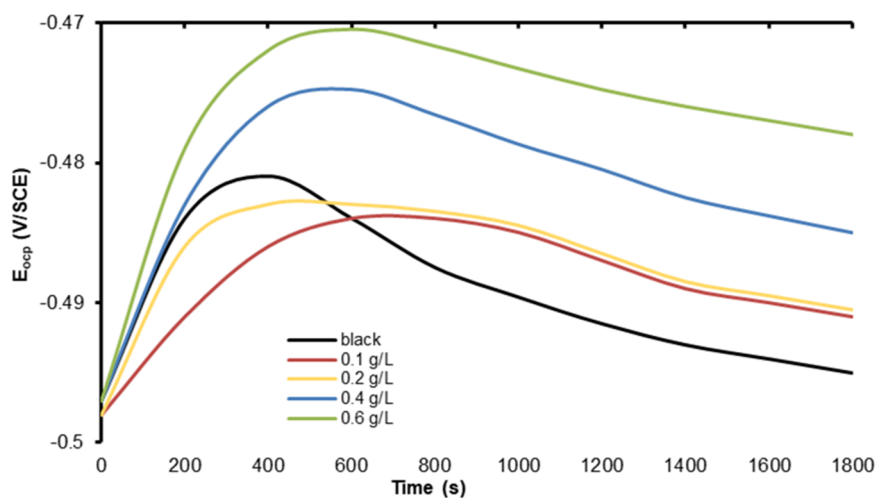


Fig 9. Open circuit potential of mild steel in 1 M H_3PO_4 without inhibitor and with different concentrations of BDBHBT

Table 5. Values of ΔG°_{ads} and K_{ads} registered in various temperatures using the Langmuir isotherm model

T (K)	K_{ads} (L/g)	ΔG°_{ads} (kJ/mol)
303	4.808	-21.357
323	4.072	-22.321
333	4.299	-23.163

surface coverage for various concentrations of inhibitor in acidic conditions. Table 5 illustrates values of the Gibbs free energy ΔG°_{ads} for the adsorption of BDBHBT inhibitor on steel surface in 1 M H_3PO_4 in various temperatures. The data of ΔG°_{ads} in Table 5 are negative, which shows that the adsorption of BDBHBT on mild steel in 1 M H_3PO_4 happens spontaneously and that the data are also approximately -20 kJ/mol, which shows that the process is physical [36-37].

Open Circuit Potential Measurements

Corrosion potential can be used to characterize the tendency of metals to corrode in a particular acidic environment. As depicted in Fig. 9, open circuit potential curves of mild steel in 1 M H_3PO_4 in the absence of inhibitor and the presence of various concentrations of BDBHBT inhibitor were plotted to provide a better understanding of this phenomenon. It is evident that the mild steel electrode's potential when submerged in 1 M H_3PO_4 solution (blank curve) first increased and then

tended gently; the constant state of each sample persists until the conclusion of the test. This shows the initial dissolution of the air-formed oxide film on the surface of mild steel [38]. The addition of BDBHBT inhibitor molecules in concentrations 0.1 and 0.2 g/L to the aggressive medium exhibited no difference in open circuit potential, whereas the corrosion potential of mild steel in concentrations 0.4 and 0.6 g/L moved to a more favorable state.

CONCLUSION

PET waste could be depolymerized by microwave irradiation with *trans*-2,3-dibromo-2-butene-1,4-diol with the presence of manganese acetate (0.5%, w/w). When compared to the traditional thermal glycolytic process, which needs at least 8 to 9 h, the use of a microwave could significantly shorten the reaction time needed for recycling (30 min). The weight loss measurements showed that oligomer BDBHBT obtained from PET waste was an excellent green inhibitor of corrosion of mild steel in H_3PO_4 solution. The corrosion inhibition efficiencies of BDBHBT rise with the rising of inhibitor concentration and with decreasing of temperatures. Adsorption of inhibitor BDBHBT on steel is slowed down corrosion and obeys Langmuir isotherm. Negative free energy ($-\Delta G^{\circ}_{ads}$) values imply spontaneous and strong adsorption of inhibitor BDBHBT on the mild steel surface.

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