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Corrosion of mild steel in NaCl solutions and effect of recycled plastic waste inhibitors

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ABSTRACT

Inhibition effect of recycled poly ethyleneterphthalate(PET) with both of Diethanolamine(DEA) and Triethanolamine (TEA)(ratios of 1:2 wt %) followed by estrification with stearic acid to give (D₂S and T₂S)on the corrosion of mild steel has been studied in 2 M NaCl solutions. The weight loss, potentiodynamic polarization techniques and open circuit potential measurements have been used. The results showed that the inhibition of the prepared recycled compounds (D₂ S& T₂S) occurs through adsorption of the inhibitor molecules on the metal surface and the inhibition efficiency was found to increase with increasing the inhibitor concentrations and temperature as well as these inhibitors act as mixed-type. The adsorption of these compounds on the metal surface is found to obey Langmuir adsorption isotherm. Thermodynamic functions for both dissolution and adsorption processes were calculated. The obtained results from weight loss and potention-dynamic polarization techniques are in a good agreement.

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KEYWORDS

Recycled Plastic Waste;
NaCl solutions;
Corrosion Inhibitors;
Mild Steel;
Adsorption isotherm.

INTRODUCTION

Steel is one of the most widely used materials of construction and frequently used in manufacture the pipelines due to the aggressiveness of the liquids which carried by them. These liquids may be petroleum containing sulphur or water containing chlorides and sulfate anions. The main problem in using mild steel in acidic solution is that of uniform corrosion. A possible solution to protect steel from acidic environment is the application of corrosion inhibitors or coatings.

Inhibition of mild steel corrosion in aqueous solutions by organic^[1-4] and inorganic^[5] compounds as well as synergetic inhibition^[6-8] was studied. The inhibition of steel corrosion in acidic solution was studied in considerable detail^[9]. In the previous works^[10, 11], Poly (ethyleneterphthalate) (PET) plastic waste was used as a cheap and safe corrosion inhibitors for C-steel in hydrochloric acid (1M HCL)and nitric acid corrosive medium.

PET is widely used in the manufacture of high-strength fibers, photographic films and soft drink

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bottles^[12]. The disposal of a large number of PET bottles causes serious environmental problems. Therefore, various methods used to recycle PET bottles^[13-15].

The addition of inhibitor compounds may reduce the partial anodic (anodic inhibitor), the partial cathodic (cathodic inhibitor) or the two partial reactions (mixed inhibitor). In many cases, the inhibition is related to adsorption of the inhibitor on the metal surface forming a barrier layer which separates the metal from the corrosive media. According to the type of inhibitor species and the nature of metal and alloy, adsorption may be chemical or physical adsorption^[16-20].

In the present work, Diethanolamine and Triethanolamine (DEA and TEA) were used to convert PET waste to water soluble oligomers followed by esterification of the products with stearic acid and evaluation of the obtained ester (D2S and T2S) as corrosion inhibitors for mild steel in NaCl solutions at different temperatures.

EXPERIMENTAL

Materials

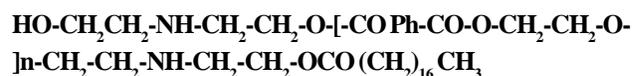
Poly(ethyleneterphthalate) (PET) waste is collected from beverage bottles. Diethanolamine (DEA), Triethanolamine (TEA) and manganese acetate and stearic acid were purchased from Aldrich Chemical Co. Ltd. (UK) England. sodium chloride (NaCl Shinyo Pure Chemicals, 99.99%).

Recycle process

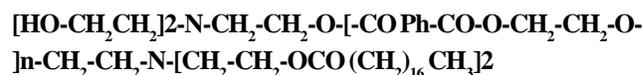
Poly(ethyleneterphthalate) waste is converted to glycolized oligomers products (D_2 and T_2). In this respect, PET bottle was depolymerized (1 wt% of PET: 2 wt% of DEA or TEA) using 0.5% of manganese acetate as a catalyst. The reaction mixtures were mixed into reaction flask fitted with mechanical stirrer, thermometer, condenser and nitrogen inlet. The reaction mixtures were heated under vigorous stirring in nitrogen atmosphere at temperatures from 170-190 °C for 4h and at 200-210 °C for 3h. The temperature of the reaction was lowered to 100 °C for 1h; the mixture was allowed to cool at room temperature^[21, 22]. (D2S and T2S) were prepared by esterification of 0.1 mol of D2 and/or T2 with stearic acid 0.1 mol in the presence of 1% (by weight) of p-toluene sulfonic acid (PTSA).

The reaction mixture was heated up to 140 °C to remove the theoretical condensed water using Dean-Stark separator. The reaction mixture was cooled to room temperature. The products were precipitated by using methanol as non-solvent to remove unreacted materials. The chemical structure of the used inhibitors is as follows:

D2S



T2S



Weight loss measurements

Mild steel alloy with the dimensions of 2x2x0.05 (cm) with chemical composition (wt %) of 0.17 C, 0.057 Mn, 0.011 P, 0.005 Sn, 0.043 Cu, 0.002 Mo, 0.011 Al and 0.027 Ni. The samples were polished successively with fine grade emery papers, cleaned with acetone, washed with double distilled water, dried, weighed and then introduced into test solution. Weight loss measurements were carried out in a double wall glass cell equipped with a thermostat-cooling condenser.

Potentiodynamic polarization measurements

The working electrode was made from mild steel rod that has the same composition as mentioned in point 2.2. The rod was axially embedded in araldite holder to offer an active flat disc shaped surface of an area 1 cm². Prior to each experiment, the working electrode was polished successively with fine emery paper, rinsed with acetone, washed with double distilled water and finally dried before dipping into the electrolytic cell. A platinum wire was used as a counter electrode and a saturated calomel electrode (SCE) as the reference electrode to which all potentials are referred.

The electrochemical experiments are performed using radiometer analytical, Volta master (PGZ301, DYNAMIC ELS VOLTAMMETRY). The experiments were carried out by changing the electrode potential automatically from the starting potential towards more positive values at the required scan rate till the end of the experiments.

Open circuit potential

The potential of steel electrode was measured

against saturated calomel electrode (SCE) in 2 M NaCl solution in absence and presence of 250 ppm of both D2S and T2S inhibitors at 25 °C. All measurements were carried out using Multi-tester until the steady-state potentials are reached.

RESULTS AND DISCUSSIONS

Evaluation of the prepared oligomers as corrosion inhibitors

The use of inhibitors is one of the most practical methods for protection against corrosion in acidic media^[18]. The choice of the inhibitor is based on two considerations:

First: it could be synthesized conveniently from relatively cheap raw materials,

Secondly: it contains the electron cloud on the aromatic ring or the electronegative atoms such as N₂ and O₂ in the relatively long chain compounds^[19]. In this respect, the present corrosion inhibitors were designed to prepare from recycled PET waste and introducing DEA and TEA followed by introducing stearate via esterification with stearic acid in their chemical structure to increase their abilities to soluble in water and to use as corrosion inhibitors for mild steel. The corrosion of mild steel in 2M NaCl Solution at different temperatures was studied by weight loss and by potentiodynamic polarization.

Techniques

Weight loss measurements

In the present work, two samples, D2S and T2S, were applied as corrosion inhibitors for mild steel. The 2M NaCl Solution was used as a blank solution for the weight loss measurements. For different concentrations from the prepared oligomers, 50, 100, 150, 200, and 250 ppm were prepared by dilution the required volume of each compound. Weight loss (mg/cm²) of the surface area was determined in an open system at various time intervals. Weight loss of the steel sample versus time of immersion was plotted in Figures (1, 2). It is evident from these Figures that in all cases. The weight loss increases with increasing the time of immersion. The curves obtained in the presence of additives fall significantly below that of free acid in all cases. In this

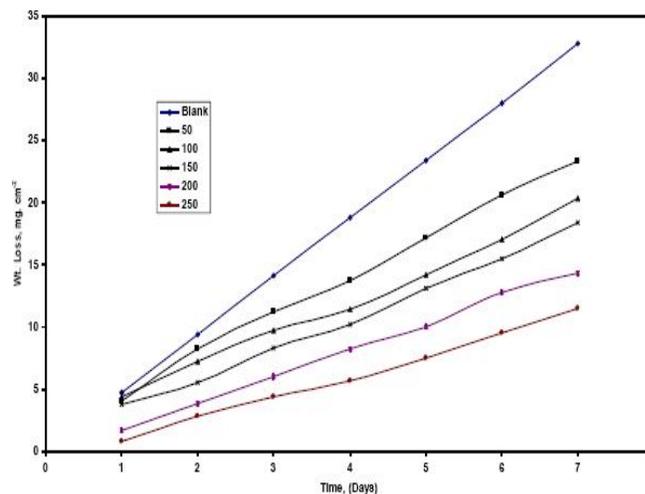


Figure 1 : Weight loss-time curves of mild steel alloy in 2M NaCl Solution in absence and presence of different concentration of inhibitor T2 S at 303 K.

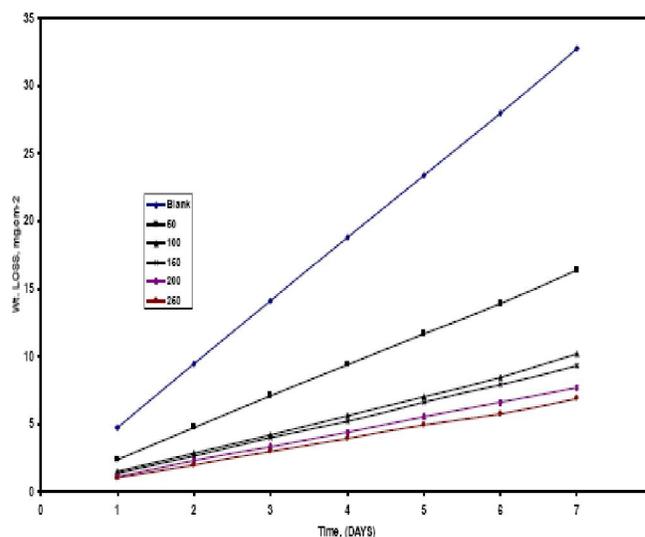


Figure 2 : Weight loss-time curves of mild steel alloy in 2M NaCl Solution in absence and presence of different concentration of inhibitor D2S at 303 K.

respect, the increase of the additive concentration was accompanied by a decrease of weight loss and an increase of the percentage of inhibition^[20].

The curves obtained in the presence of additives fall significantly below that of free acid in all cases. In this respect, the increase of the additive concentration was accompanied by a decrease of weight loss and an increase of the percentage of inhibition^[20]. The corrosion inhibition efficiency (%IE) was calculated by the following equation:

$$\%IE = ((\Delta w - \Delta w_i) / \Delta w) \times 100 \quad (1)$$

Where Δw and Δw_i are the weight loss per unit area in absence and presence of the inhibitor respec-

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tively. Δw can be calculated from the following equation:

$$\Delta w = w_b - w_a \quad (2)$$

Where w_b and w_a are the weight of the specimen before and after reaction, respectively. The obtained data of (%IE) and rate of corrosion (R_{corr}) of both D2S and T2S were summarized and listed in TABLE (1). The data show that R_{corr} value was decreased with increase of inhibitor concentrations and increase with time. On the other hand, the inhibition efficiency was increased with increasing of inhibitor concentrations. So that the inhibitor compounds may be chemically adsorbed on the steel surface and cover some sites of the electrode surface. The chemical adsorption can be explained on the basis of the effect of chemical structure of inhibitors on its inhibition efficiency. In this respect, the benzene ring and the carbonyl group (C=O) of D2S and T2S oligomers can form a big (π) bond accordingly, not only the (π) of benzene and carbonyl enter unoccupied orbital of iron, but also, the π^* orbital can accept the electron of d orbital of iron to form feedback bonds, which produce more than one center of chemical adsorption on the steel surfaces^[21]. On the other hand, the corrosion inhibition efficiency of the prepared oligomers of D2S is greater than T2S at lower surfactant concentration, (50- 250) ppm, as listed in TABLE (1).

This can be attributed to the presence of trisubstituted ethylene group attached to nitrogen group of PET which decrease the basicity of amine groups of T2S due to steric effect^[22]. If one supposes that the adsorption of inhibitor follows the Langmuir adsorption isotherm, the surface coverage could be given by the equation:

$$C_i/\theta = (1/K_{\text{ads}}) + C_i \quad (3)$$

Where, C_i is the inhibitor concentration and K_{ads} is the adsorption equilibrium constant. The degree of surface coverage (θ) for different concentration of the inhibitors in acidic media^[23], has been evaluated from weight loss measurements by using the following equation:

$$\theta = 1 - (\Delta w_i/\Delta w) \quad (4)$$

Careful inspection of these results showed also that, inhibition efficiency increases with increasment of both inhibitor concentration and surface coverage (θ).

In the present work, it was observed that, all isotherms have slope less than unity the deviation from

TABLE 1 : Degree of surface coverage (θ) and percentage inhibition efficiency (%IE) of the inhibitors T2S and D2S in 2 M NaCl at 303 K, obtained from weight loss measurements after 7 days.

Compound	conc. ppm	conc. M/Lx104	Wt. loss mg.cm	Corrosion rate (k) mg. cm-2 day-1	θ	%IE
BLANK	0	0	27.9	4	0	0
	50	0.74	8.7	1.3	0.58	58.3
	100	1.44	7.9	1.1	0.61	60.9
T2S	150	2.2	6.6	0.93	0.64	64.9
	200	2.97	5.8	0.77	0.67	67.3
	250	3.2	4.2	0.68	0.71	71.2
	50	0.32	7.3	0.8	0.58	67.8
	100	0.64	6.8	0.71	0.61	69.7
D2S	150	0.94	6.8	0.68	0.65	70.2
	200	1.3	6.7	0.58	0.67	72.3
	250	1.8	6.2	0.48	0.75	75.8

unity may be explained on the basis of interaction between the adsorption species on metal surface^[23]. The results of dissolution of carbon steel in 2M NaCl in presence of D2S and T2S might to interpret on the basis of interface inhibition mode. The inhibitors are acting effectively at the metal solution interface. Accordingly, a small concentration of inhibitors exhibits an effective corrosion inhibition. This may be due to the formation of adsorbed monolayer at the metal surface interface. The surface coverage values (θ) were tested graphically for fitting a suitable adsorption isotherm. The relation C_i/θ against C_i was illustrated in Figure [3].

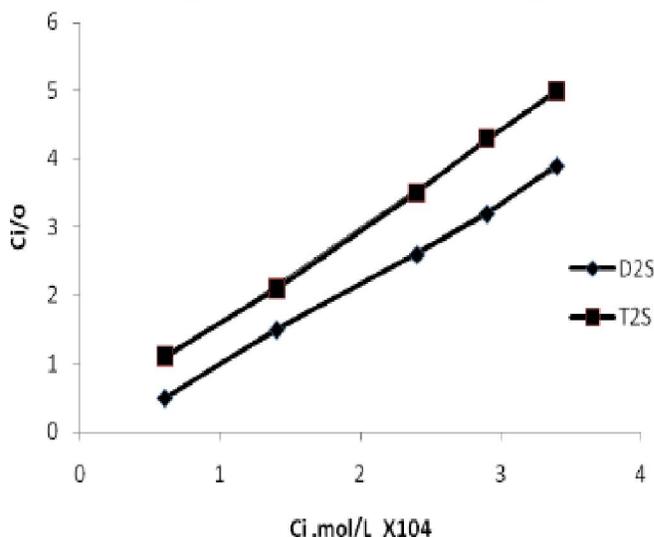


Figure 3 : Langmuir adsorption isotherm for T2S and D2S inhibitors

Effect of temperature

Influence of solution temperature on the corrosion behavior of carbon steel in 2M sodium chloride solution was studied by the weight loss method over temperature range (303 – 333K). The data display that the rate of corrosion decreases with an increase in temperature from 303 to 333K as represented in TABLE (2). Also, the Figures (4-5) indicate that the weight losses of mild steel in 2M NaCl increase with increasing temperature in case of blank. The results of mild

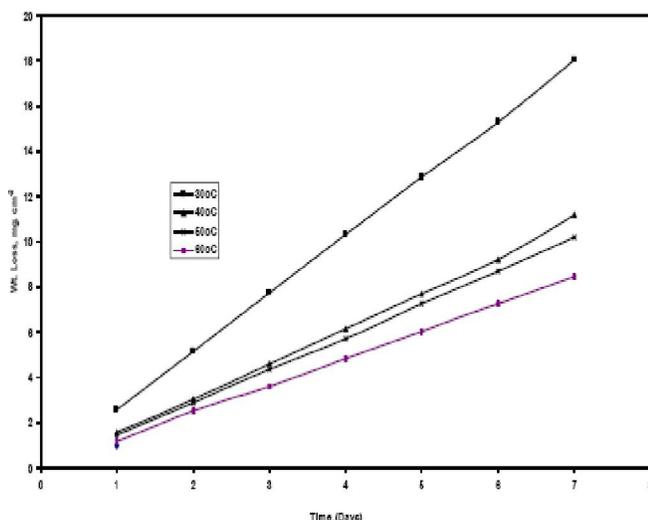


Figure 4 : Weight loss-time curves of mild steel alloy in 2 M NaCl in the presence of 250 ppm of inhibitors T2S at different temperatures

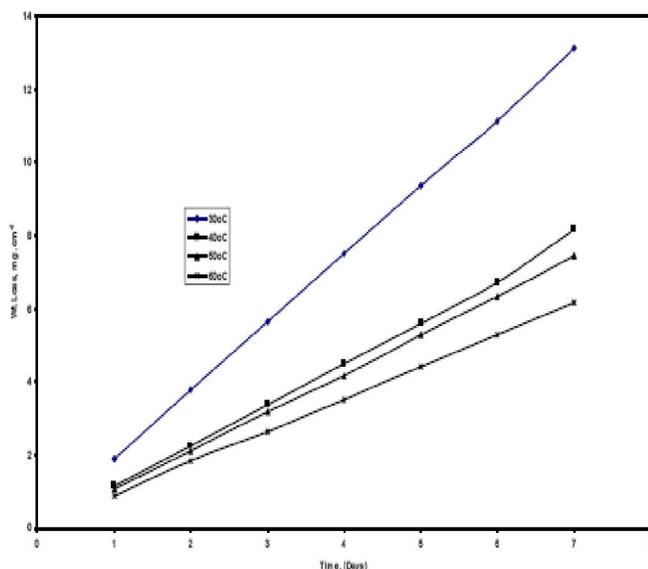


Figure 5 : Weight loss-time curves of mild steel alloy in 2 M NaCl in the presence of 250 ppm of inhibitors D2S at different temperatures.

steel inhibition efficiency of 250 ppm of the prepared oligomers at different temperatures show that the weight losses decrease (corrosion rate) with increasing temperature from 303 – 333K which indicate chemical adsorption^[24].

Activation energy of corrosion

Corrosion is an electrochemical phenomenon and follows the laws in chemical kinetics. The corrosion rate increases with temperature as a result of decreasing the apparent activation energy, E_a^* of the charge transfer reactions. Increasing of the temperature also enhances the rate of H^+ ion diffusion to the metal surface beside the ionic mobility, which increases the conductivity of the electrolyte. The effect of temperature on the inhibition efficiency of corrosion inhibitors is important in elucidation of the mechanism and kinetics of their action. Moreover, at lower temperature, absorbed hydrogen atoms (exothermic process) blocked the cathodic area.

The activation energy (E_a) can be calculated from Arrhenius equation^[24].

$$\log R_{corr} = \log A - E_a/2.303RT \tag{5}$$

A is pre-exponential factor related to concentration, steric effect and metal surface characteristics, etc. Plotting of $\log R_{corr}$ against $1/T$ for the free acid solution (blank) and 250ppm of the D2 and T2 inhibitor was represented in Figure (6). The activation energies were calculated and listed in TABLE (2). The results showed that, the values of activation energy (E_a^*) increase in the same order of increasing the inhibition efficiency of the inhibitors. It is also indicated that the whole process is controlled by surface reaction^[25].

TABLE 2 : Degree of surface coverage (θ) and percentage of inhibition efficiency (%IE) of the inhibitors T2S and D2S in 2M NaCl at different temperature

Compound	T, oK	(1/T) x10-3	R _{corr.} , mg.cm2.day-1	Log R _{corr.}	%IE	θ	E _{act} ive, k.J.mol-1
T2S	303	3.3	0.68	-0.1674911	71.2	0.712	122.9
	313	3.19	0.61	-0.2146702	73.8	0.798	
	323	3.095	0.56	-0.251812	79.8	0.798	
	333	3.003	0.49	-0.3098039	82.6	0.826	
D2S	303	3.3	0.48	-0.3187588	75.7	0.757	131.6
	313	3.19	0.41	-0.3872161	79.6	0.796	
	323	3.095	0.38	-0.4202164	82.4	0.824	
	333	3.003	0.31	-0.5086383	85.6	0.856	

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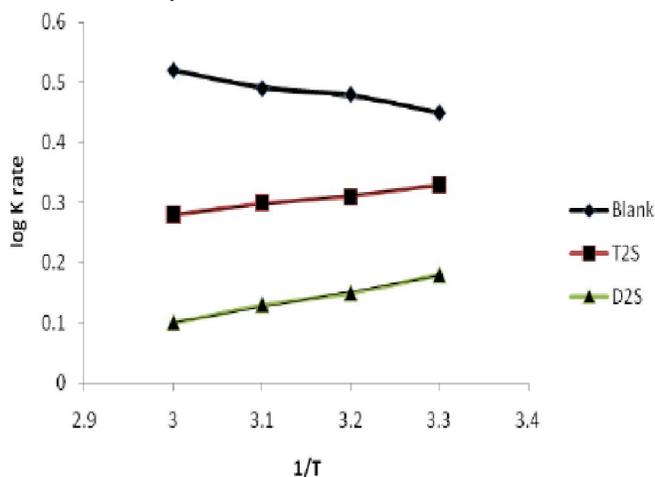


Figure 6 : relation between $\log R_{\text{corr.}}$ against $1/T$ at different temperature in presence and absence of 250 ppm of D2S and T2S.

Thermodynamic functions of activation

The free energy of adsorption (ΔG^*) at different temperature was calculated from the following equation:

$$\Delta G^* = RT (\log R_{\text{corr.}} - \log KT/h) \quad (6)$$

K, equilibrium constant, values is given by

$$K = \frac{\theta(7)}{C_i(1-\theta)}$$

The values of E_a and ΔG^* were listed in TABLE (3). The negative values of ΔG^* indicate that the spontaneous adsorption of inhibitors on the surface of mild steel. The negative values of ΔG^* also suggest the strong interaction of the inhibitor molecules onto the steel surface^[26]. The thermodynamic functions, entropy of activation, ΔS^* , enthalpy of activation, ΔH^* and free energy of activation, ΔG^* is calculated from this equation:

$$\Delta H^* = E_a + RT \quad (8)$$

Where, R is the universal gas constant. Entropy of inhibitor adsorption (ΔS^*) can be calculated using the following equation:

$$\Delta S^* = (\Delta H^* - \Delta G^*)/T \quad (9)$$

The calculated data were listed in TABLE (3). All obtained data show that ΔS values are positive and increased by increasing the temperature which indicate that the inhibitors more oriented and more disordered on the surface of the metal. TABLE (3) show that ΔH has negative values, which indicated that, the reaction is exothermic. On the other hand, the negative value of ΔH indicated that the adsorption of inhibitors is chemi-

cal adsorption and the surfactant forms stable layer at the surface of steel. This behavior protects steel from the environment (sodium chloride solution) and decrease the corrosion rate.

TABLE 3 : Thermodynamic activation parameters for mild steel in 2M NaCl in absence and presence of 250 ppm of the inhibitors at different temperature.

Compound	Temp., °K	E^*	ΔG^* , k.J. mol-1	ΔH^* , k.J. mol	ΔS , k.J. mol.k-1
Blank	303	70.23	-39.04	-21.1	0.187
	313		-39.07	-21.5	0.196
	323		-40.1	-22.1	0.194
	333		41.8	-22.8	0.192
	303		55.6	-23.4	0.13
T2S	313	128.9	58.7	-24.6	0.12
	323		61.6	-25.2	0.132
	333		63.9	-26.8	0.13
	303		64.2	22.8	0.14
D2S	313	131.6	72.3	24.2	0.15
	323		73.8	26.3	0.14
	333		75.2	27.6	0.141

Potentiostatic polarization measurements

Anodic and cathodic polarization curves for mild steel in 2 M NaCl with and without various concentrations of used inhibitors were represented in Figures (7, 8). The curves were swept from -750 to 0.3 V (SCE) with scan rate of 20 mVs-1. Figures (7,8) illustrate the effect of adding progressive addition (50 – 300ppm) of selected inhibitors on the cathodic and anodic polarization curves of steel alloy in 2 M NaCl at 30°C. The addition of the used inhibitors decreases the corrosion current densities (I_{corr}) and shifts the corrosion potential (E_{corr}) towards more positive values. Furthermore, it was also found that all inhibitors behave as mixed-type, i.e. both cathodic and anodic polarization curves are affected by the inhibitors^[27]. The variable values of the cathodic Tafel slopes suggest that the inhibition action of such compounds occurs by simple blocking of the electrode surface area^[27]. The cathodic current-potential curves give rise to parallel Tafel lines, which indicate that hydrogen evolution reaction is controlled^[28]. Addition of inhibitors under study increases both the cathodic and anodic over potential of mild steel alloy and causes displacement of the cathodic and anodic polarization curves. Therefore, these oligomers could be classified

as mixed type (anodic/cathodic) inhibitors. These results indicate that D2S and T2S products inhibit NaCl corrosion of mild steel via their adsorption on both anodic and cathodic active sites without modifying the mechanism of corrosion reaction. This means that the adsorbed inhibitor molecules block the surface active sites and decrease the area available for hydrogen evolution and metal dissolution reactions^[28].

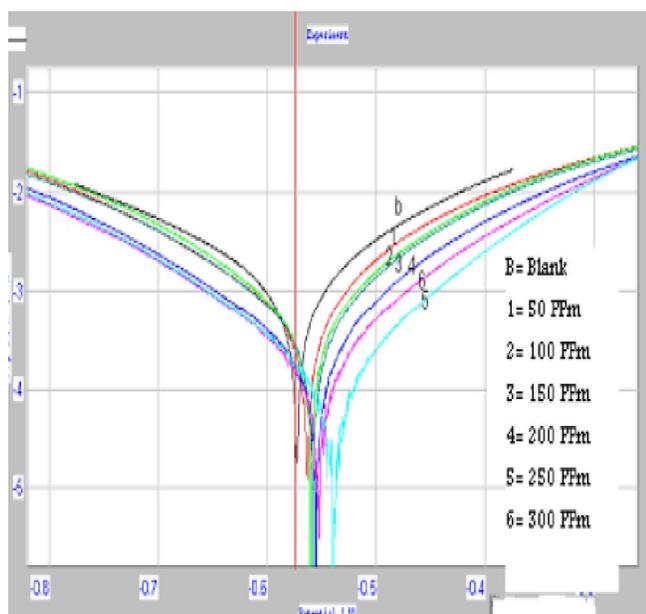


Figure 7 : Potentiostatic polarization curves of mild steel in 2 M NaCl in absence and presence of different concentration of inhibitor T2S at 303 K.

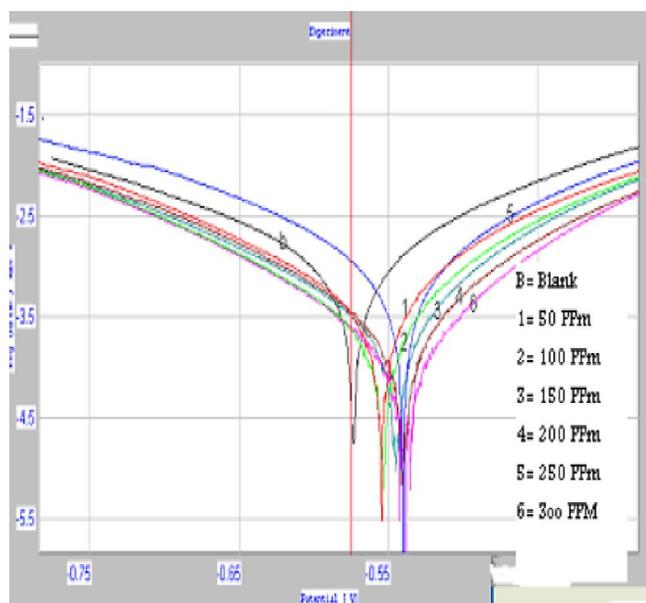


Figure 8 : Potentiostatic polarization curves of mild steel in 2 M NaCl in absence and presence of different concentration of inhibitor D2S at 303 K.

The corrosion potential (E_{corr}) and the corrosion current density (I_{corr}) were determined from extra-pollation of cathodic and anodic Tafel lines. The polarization resistance (R_p) and the slope plotting of potential vs. current density were also determined. The values of E_{corr} , I_{corr} , R_p , Tafel slopes (bc , ba), degree of surface coverage (θ) and inhibition efficiency (%IE) were calculated for each sample and listed in TABLE (4). It is clear that the values of corrosion potentials E_{corr} remain almost unchanged and indicate that the prepared surfactants acts mainly as mixed type inhibitors^[24]. Addition of all prepared inhibitors to sodium chloride solution decreases the values of I_{corr} and increases the values of R_p for carbon steel alloy.

The inhibition efficiency (%IE) of inhibitors was calculated from polarization measurements by using the following equation:

$$(\%IE) = 1 - (I_{\text{corr}}/I_{\text{corr}0}) \times 100 \quad (10)$$

Where $I_{\text{corr}0}$ and I_{corr} are the corrosion current densities in the absence and presence of inhibitor respectively.

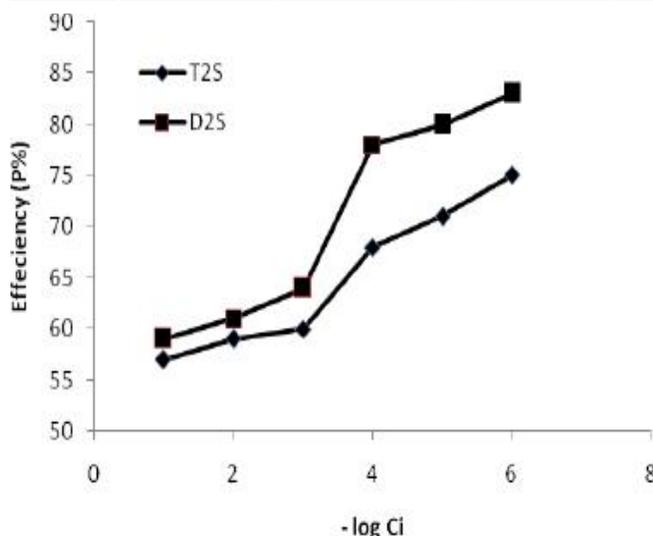
The inhibition efficiency calculated from the polarization measurements were listed in TABLE (4). It is obvious that the inhibition efficiency increases with increase of the concentration of inhibitors. These results are comparable with those obtained from weight loss measurements. However there is difference in the values obtained by the two methods. This difference was also mentioned by several investigators^[23].

The relation between the measured efficiency and concentrations of the selected surfactants was represented in Figure (9). S-shaped adsorption mode, indicated from Figure (9), which proved that the mechanism of inhibition involves the formation of monolayer at the metal-solution interface.

The shape of isotherm seems to reflect two modes of adsorption. At very low concentration, the polymer is adsorbed either at the vertical mode or the all active sites on the surface are not partially occupied. The plateau form may be explained by the formation of a monolayer of polymer. Further increase of inhibitor concentration leads to the formation of multilayer generally at horizontal mode. The presence of N, NH and ester groups reinforces the adsorption phenomenon by inducing the anodic action.

TABLE 4 : Polarization data of the prepared inhibitors.

Sample	Conc., ppm	I_{corr} , mA/cm ²	$-E_{corr}$, mV	Rb, ohm.cm ²	ba, mV	-bc, mV	%IE	Θ	Ci x10 ⁴ , mol/L
Blank	0	1.1565	51.732	24.561	158.31	185.67	0	0	0
	50	0.3951	482.8	78.4	132.1	161.2	59.6	0.596	0.7
	100	0.3402	475.83	76.41	124.74	154.89	63.45	0.6345	0.9
T2S	150	0.2961	470.61	54.77	121.14	163.53	66.87	0.6687	1.8
	200	0.30375	473.94	57.87	109.71	149.4	66.33	0.6633	2.79
	250	0.37602	463.95	49.5	119.61	165.51	60.66	0.6066	3.69
	300	0.36102	461.2	48.5	112.61	158.2	60.8	0.608	3.97
D2S	50	0.4213	462.6	38.6	133.6	216.2	57.4	0.574	0.68
	100	0.39564	456.66	44.856	128.43	208.8	59.13	0.5913	0.792
	150	0.36819	445.59	47.295	124.65	208.26	61.29	0.6129	1.53
	200	0.31905	442.89	60.678	117.63	194.4	64.89	0.6489	2.34
	250	0.15291	478.08	85.95	121.608	151.2	78.03	0.7803	3.15
	300	0.15024	438.2	68.3	119.211	149.3	80.1	0.801	3.92

Figure 9 : Relation between P% and $-\log C_i$ for the used inhibitors

Open circuit potential measurements

The potential of mild steel electrode immersed in 2 M NaCl solution was measured as a function of immersion time in the absence and presence of 250 ppm of both D2 S and T2 S as shown in Figure (10). It is clear that the potential of mild steel electrode immersed in 2 M NaCl solution (blank) tend towards more negative potential firstly, giving rise to short step. This behavior was reported by other investigators^[29], which represents the breakdown of the pre-immersion air formed oxide film presents on the surface according to the following equation:



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This is followed by the growth of a new oxide film inside the solution, so that the potential was shifted again to more noble direction until steady state potential is established. Addition of inhibitors molecules to the corrosive medium produces a negative shift in the open circuit potential due to the retardation of the cathodic reaction. The results of final steady state potential (E_s) without inhibitor is -590 mV (vs. SCE) while that using the best dose (250ppm) of the used inhibitor is -456 mV (vs. SCE) in case of D2S and equal to -462 in case of T2S.

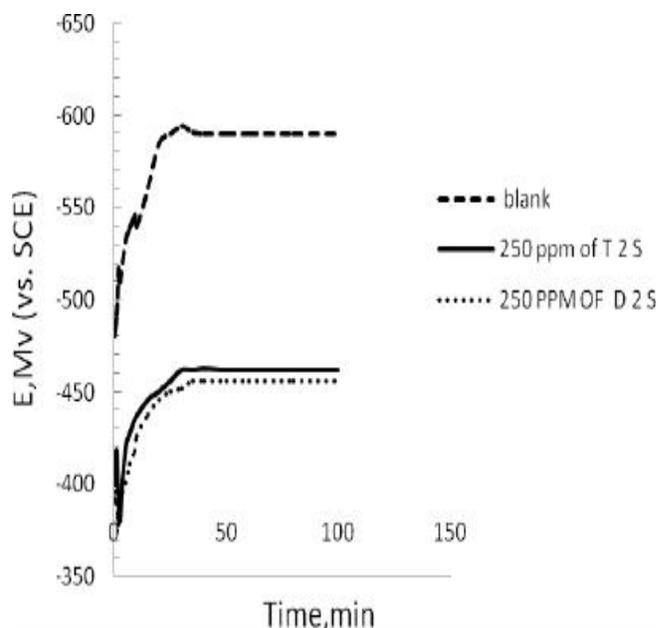


Figure 10 : Potential-Time curve for mild steel in 2 M NaCl solution in absence and presence of 250ppm of D2S and T2S.

CONCLUSIONS

Based on the previous results, the following conclusions are accomplished:-

1. All studied oligomers are good inhibitors and act as the mixed type inhibitors for mild steel corrosion in 2 M Sodium chloride solution.
2. Inhibition efficiencies increase by an increase in the inhibitor concentration and an increase in temperature up to 333 K.
3. All entropy parameters for adsorption of inhibitors molecules on steel are positive and increase by increasing the temperature which indicate that the inhibitors more oriented and more disordered on the surface of the metal.
4. The activation parameters of the dissolution (E^* , ΔH^* and ΔS^*) were calculated and showed that the used inhibitors decrease the rate of corrosion.
5. The adsorption of these inhibitors on steel surface obeys Langmuir's adsorption isotherm.
6. Addition of inhibitors molecules to the corrosive medium produces a negative shift in the open circuit potential due to the retardation of the cathodic reaction.

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