

Corrosion and Corrosion Inhibition of Mild Steel in Red Sea Water by EDTA

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Abstract

The corrosion of mild steel (MS) was studied in Red Sea water. The effect of disodium ethylenediaminetetraacetic acid salt (Na-EDTA) as an inhibitor in this medium was also studied. Weight loss, and Open Circuit Potential (OCP) were used as the tools of the study, where the Fe-surfaces before and after corrosion were examined using Scanning Electron Microscopy (SEM). We notice that, the corrosion reaction was reduced when Na-EDTA was added to the medium. As the concentration of Na-EDTA was increased, the corrosion rate decreases. It seems that, certain barrier of Na-EDTA adsorbed on Fe-surface which isolate the corroded medium from Fe-surface. The adsorption phenomenon was studied by different adsorption isotherms and found to fit well with Langmuir and Temkin. Thermodynamic parameters were determined from Arrhenius and Eyring equations. SEM shows the decrease of the roughness of MS after adding Na-EDTA.

Keywords: Corrosion; Scanning electron microscopy (SEM); Adsorption; Thermodynamic properties

Introduction

In our step by step life, incredible mechanical properties of iron and iron alloys are seen as a champion among the most fundamental materials. Regardless, these applications are confined when the iron is available in corrosive media as arrangements containing chloride or potentially acidic arrangements [1]. Inhibitors, generally, cause a change in the state of the metal surface through adsorption (physically or falsely) and moreover by forming an insoluble or hardly dissolvable compound with the metal cations. The adsorption process is enhanced by the existence of hetero atoms (as O, N, S, and P), aromatic rings, = or \equiv bonds in the structure of the inhibitor [2]. EDTA was used for complexation with the charged metal particles [3,4]. Complexation occurs between metal (vacant d-orbital) and EDTA (free or π)-electrons via the formation of donor-acceptor surface [3,5-7]. EDTA has been used as an inhibitor for metals in different environments [7-10]. The inhibition effect of EDTA is affected by different parameter including, temperature, medium pH, concentration, and type of the metal and additives. EDTA performed as good corrosion inhibitor for MS in hydrochloric acid media [11] and benzotriazole in HCl medium [3]. In physiological media, EDTA keeps the improvement of an inactive layer and extends the dissolvability of the steel electrode [7]. The addition of EDTA up to 300 ppm has resulted in improving the adherence of the oxide to carbon steel and decreasing the corrosion metal loss in pH from 7 to 10.5 [12]. EDTA is practical in decreasing the corrosion rate of the copper-nickel composite in seawater contaminated with sulfur [13]. In the present examination, the inhibition effect of disodium EDTA salt (Na-EDTA) for the corrosion of MS in Red Sea water will be studied using weight loss estimation and electrochemical methodologies (Tafel plot and OCP). The adsorption isotherms and thermodynamic parameters will be obtained by weight loss estimations are discussed in detail. The surface morphology of MS will be investigated in the closeness or nonattendance of Na-EDTA.

Experimental Section

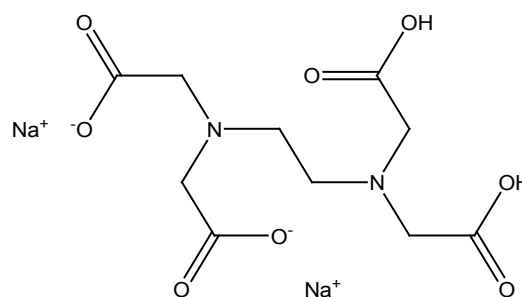
Chemical and reagents

The composition of MS coupons used in this study is shown in Table 1.

MS coupons (3.0 cm \times 4.0 cm \times 0.1 cm) dimensions for weight-loss tests and 4 mm diameter cylindrical electrode embedded in polyester for polarization curve tests, then polished using emery papers (grades 200 up to 1,200), degreased in acetone, dried at room temperature and stored in a desiccator. Na-EDTA (analytical grade) was obtained from Fluka AG, Switzerland. All solutions were prepared using freshly prepared bi-distilled water. Scheme 1 shows the chemical formula and

Element	Wt (%)
Mn	0.58
P	0.35
C	0.13
Si	0.04
Fe	Balance

Table 1: Chemical composition of MS samples (coupons and electrodes).



Scheme 1: Chemical structure of Na-EDTA disodium salt.

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the structure of EDTA disodium salt. Red Sea water is collected daily from four different places in Jazan beach, called B₁, B₂, B₃ and B₄.

The average parameters of the sea water samples are given in Table 2.

Methods

Weight loss measurements

The MS coupons were weighed using FA2104A analytical electronic digital weighing balance (sensitivity of 0.0001 gram) before immersion in an open 250 ml beaker containing 100 ml of Red Sea water at 298 K and desired temperatures. Each coupon was withdrawn from the Red Sea water after every 24 hours. The setups were fixed for a period up to 10 days. Each water sample (B₁-B₄) is examined separately and the average value is calculated. The weight loss after 6 days was taken by finding the difference in electrode weights as usual. Each experiment was performed three times in each case and the mean values are calculated. The inhibition efficiency (% IE), the degree of surface coverage (θ) and corrosion rate (η) were determined using eqns. (1)-(3) respectively.

$$IE\% = \left(1 - \frac{w_1}{w_2}\right) \times 100 \quad (1)$$

$$\theta = 1 - \frac{w_1}{w_2} \quad (2)$$

$$\eta(\text{mmy}^{-1}) = \frac{87.6xw}{A.t.d} \quad (3)$$

where w_1 and w_2 are the loss in weights (g cm^{-2}) for MS in the presence and absence of Na-EDTA, A is the mild steel coupon area (cm^2), t is the immersion time (h) and w is the average weight loss (mg) of MS after time t (min) and d is the MS density (g cm^{-3}).

Temperatures effects

The same steps adopted for weight loss studies, but with changing the temperature of each experiment. Each experiment was done twice at various temperatures with and without Na-EDTA in Red Sea for good reproducibility.

Electrochemical methods

A scanning potentiostat/galvanostat (EG&G 326A, USA) is used in all electrochemical experiments using 10 mV s^{-1} as scan rate. Each experiment was repeated to ensure reproducibility. A three-compartment cell has a reference electrode (SCE), auxiliary electrode (Pt-foil) and working electrode (MS).

Parameter	Average value
pH	7.98
Conductivity	53.62 mS cm^{-1}
Salinity	29.33 g kg^{-1}
Cl ⁻	24160 mg L^{-1}
SO ₄ ²⁻	3380 mg L^{-1}
Ca ⁺⁺	526 mg L^{-1}
Na ⁺	13500 mg L^{-1}
K ⁺	480 mg L^{-1}
Mg ⁺⁺	1620 mg L^{-1}
Br ⁻	80 mg L^{-1}
HCO ₃ ⁻	170 mg L^{-1}
Sr ⁺⁺	21 mg L^{-1}
TDS	43937 mg L^{-1}

Table 2: Physico-chemical parameter of used clean Red Sea water at 298 K with density of 1.235 g cm^{-3} .

Adsorption isotherm

Adsorption of inhibitors occurs at a metal/solution interface. The basic information of adsorption is obtained via the help of adsorption isotherm. In order to obtain the isotherm, the surface coverage values (θ) as a function of inhibitor concentration (c) must be obtained. The values of θ are determined from eqn. (2). We use different isotherms based on weight loss data, only the best fit Langmuir and Temkin are represented here.

The adsorption at a metal/solution interface is the major effect of adsorption of inhibitor. Adsorption isotherm provides basic information on the adsorption of the inhibitor on the metal surface. In order to obtain the isotherm, the fractional surface coverage values (θ) as a function of inhibitor concentration must be obtained. The values of θ are determined from eqn. (2). We use different isotherms based on weight loss data, only the best fit Langmuir and Temkin are represented here

Scanning electron microscopy (SEM)

To examine the surface morphology, the MS is observed in a Scanning Electron Microscope: JSM-T20 Electron Probe Micro-analyzer (JEOL, Tokyo, Japan). MS coupons after immersing in blank and in the inhibitor solution for a period of 6 days were rinsed with bi-distilled water and dried. We examine MS Sheets after polishing; MS sheet dipped in Red Seawater for 6 days at 303 K without inhibitor and MS sheet dipped 6 days in Red Sea water containing 0.13 M of Na-EDTA inhibitor at 303 K.

Results and Discussion

Open circuit potential (OCP) investigations

The behavior of MS in the absence and presence of Na-EDTA in Red Sea solutions was examined by OCP experiments. The OCP curves are shown in Figure 1. In the Red Sea, the potential of MS lies in a negative direction in the first time of immersion, this may be due to the dissolution of the MS surface under the effect of the aggressive chloride ions attack. The corrosion products from a layer that makes partial protection of MS from further attack and could lead to a slight shift in the potential towards the less negative values with time. In the presence of Na-EDTA, the potential rapidly shift to negative values in the first few minutes, as a result of the dissolution of the MS-Oxide film (that was formed in the air before immersion into the test solution) [14]. After that, a shifted towards the positive values occurs; by increasing Na-EDTA concentration the shift increase. Furthermore, at higher concentration of Na-EDTA, 0.11 and 0.13 M, a direct positive shift is recorded and increase with increasing time. It is concluded that both the absolute and the steady state potential are shifted to the more positive direction in the presence of Na-EDTA and by increasing Na-EDTA concentration.

Potentiodynamic polarization studies

The cathodic and anodic polarization curves of MS in Red Sea water in the absence and presence of different Na-EDTA concentration at 308 K is shown in Figure 2. The electrochemical kinetic parameters, namely, corrosion current density i_{CORR} , corrosion potential E_{CORR} and cathodic and anodic Tafel slopes, β_c and β_a , associated with the polarization curves have been simultaneously determined and are listed in Table 3.

Data infer that the addition of Na-EDTA to the Red Sea solutions increases both the anodic and cathodic overpotentials, decreases the

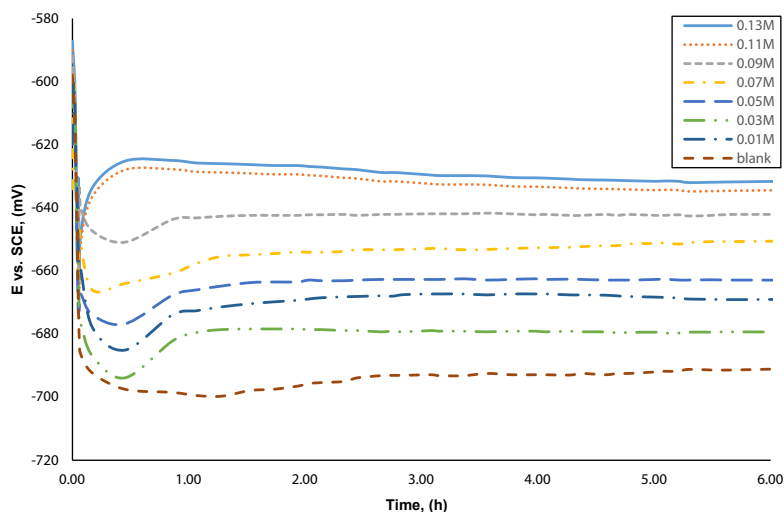


Figure 1: Potential time curves for MS electrodes immersed in Red Sea water in absence and presence of different concentrations of Na-EDTA at 308 K.

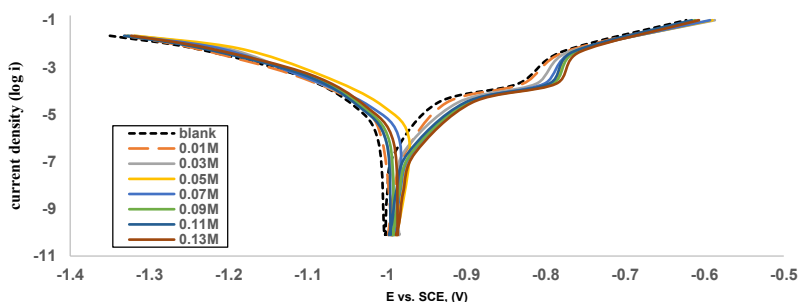


Figure 2: Potentiodynamic polarization curves for MS electrodes in Red Sea at 308 K with scan rate of 10 mVs⁻¹ with and without different concentrations of Na-EDTA.

Temperature (K)	c _{Na-EDTA} (mol L ⁻¹)	i _{corr} (mA cm ⁻²)	-E _{corr} (V)	-β _c (mV dec ⁻¹)	-β _a (mV dec ⁻¹)	IE (%)
318	Blank	0.038	1.011	122	0.69	--
	0.01	0.010	1.001	118	0.6	74
	0.03	0.009	1.006	116	0.53	76
	0.05	0.0059	0.972	118	0.52	84
	0.07	0.0054	0.984	112	0.51	86
	0.09	0.0049	0.997	115	0.5	87
	0.11	0.0047	1.001	114	0.5	88
308	Blank	0.03	1	110	0.65	--
	0.01	0.0085	0.98	109	0.62	57
	0.03	0.0082	0.97	111	0.63	57
	0.05	0.008	0.96	112	0.61	58
	0.07	0.0076	0.98	109	0.62	59
	0.09	0.0072	0.97	111	0.64	60
	0.11	0.007	0.96	107	0.63	61
298	Blank	0.028	0.96	106	0.61	--
	0.01	0.022	0.92	105	0.62	16
	0.03	0.02	0.93	104	0.63	21
	0.05	0.016	0.95	106	0.65	32
	0.07	0.012	0.96	104	0.62	42
	0.09	0.01	0.93	103	0.62	47
	0.11	0.009	0.92	106	0.63	50
	0.13	0.006	0.94	103	0.64	58

Table 3: The electrochemical kinetic parameters (i_{corr}, E_{corr}, β_c and β_a) and inhibition efficiency (IE%) obtained from polarization curves of MS electrodes immersed in Red sea water at different temperatures in absence and presence of Na-EDTA with scan rate of 10 mVs⁻¹.

corrosion current density, i_{CORR} , and shifts the E_{CORR} to more positive values. By regarding the polarization curves, only the anodic current densities diminish with the increase of the EDTA concentration. Conversely, the cathodic current densities increase with the EDTA concentration. This means that the EDTA may act as an anodic inhibitor. At different Na-EDTA concentrations and 308 K, IE% for MS electrode in Red Sea solution was calculated from the eqn. (4):

$$IE\% = \left(1 - \frac{i_{CORR}}{i_{CORR}^0}\right) \times 100 \quad (4)$$

Where, i_{CORR}^0 and i_{CORR} are corrosion current density for blank and inhibited solutions, respectively.

Mass loss

The mass losses of MS in Red Sea water samples were recorded for different time intervals (1-10) days of immersion at different temperatures. The samples are collected from 4 different places in Jazan area; we name it B₁, B₂, B₃ and B₄. The obtained data from each water sample is represented in Figure 3A and their average values represent in Figure 3B. From Figures 3A and 3B, no noticeable difference between samples is observed and weight loss, increase in all samples by increasing the time up to 6 days then began to decrease. This may

understand where corrosion films formed to isolate the MS surface from the corrosive sea water. At different temperatures in the presence or absence of Na-EDTA, the average mass-loss and corrosion rates are represented in Figures 3C and 3D, repetitively. The corrosion rates of MS were calculated using eqn. (3). The impacts on the corrosion rate of MS in Red Sea water at all studied temperatures were reduced. By increasing temperature and Na-EDTA concentration, the weight loss and corrosion rate decrease indicate that Na-EDTA may be used as an inhibitor to MS in Red Sea water. The high inhibition efficiency as the inhibitor concentration increases could be understood to be due to the reduction in corrosion rate. Thus, Na-EDTA could be considered as an inhibitor of Fe in Red Sea water given the high level of inhibition efficiency. IE% increased with the Na-EDTA concentration.

Figure 4 shows the inhibition efficiency in different concentration of the Na-EDTA and it is seen that the IE% increases linearly with the inhibitor concentration.

Adsorption studies

The increase in IE% of MS in Red Sea water with increasing additive concentrations of Na-EDTA can be explained on the basis of additive adsorption. The relation between the surface covered (θ) and

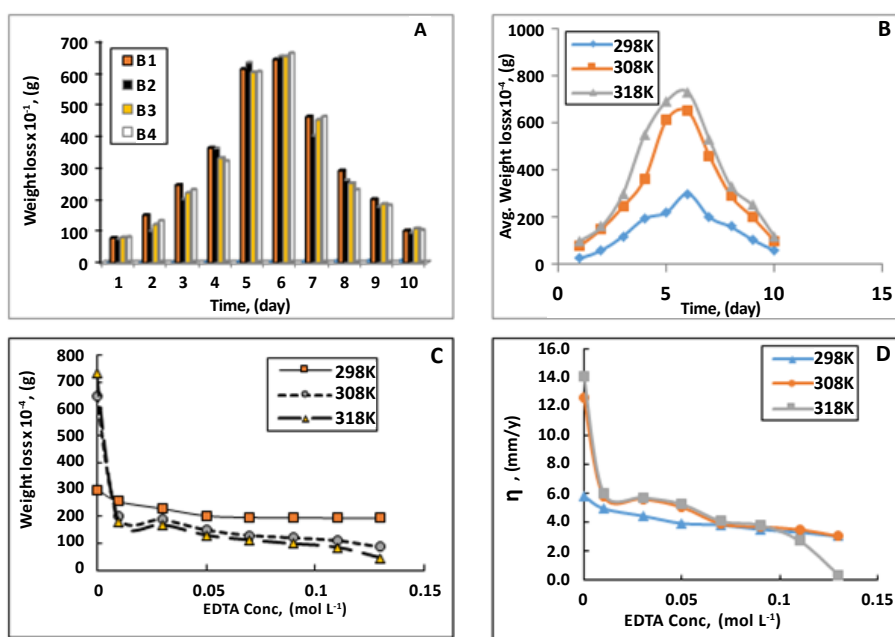


Figure 3: Mass loss of MS coupons immersed in 50 ml Red Sea water in the presence or absence of Na-EDTA at different temperatures and Na-EDTA concentrations.

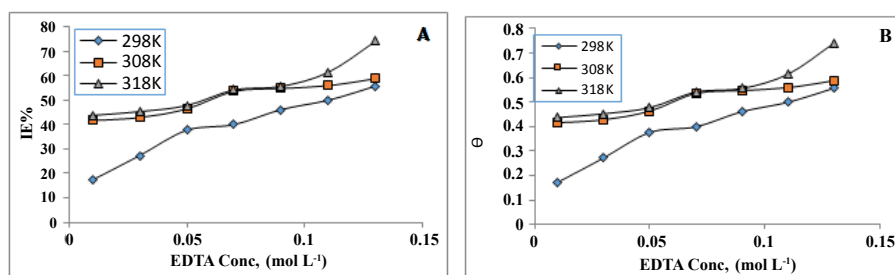


Figure 4: After 6 days in Red Sea water at different concentrations of Na-EDTA and at different temperatures. (A) %IE values and (B) θ values.

the concentration c of the inhibitor, was tested graphically by fitting it to Langmuir's isotherm, which assumes that, each adsorption sites of solid surface can hold one adsorbed species only, as in eqn. (5) [15-17].

$$c\theta^{-1} = K_{ads}^{-1} + c \tag{5}$$

Where, c and K_{ads} are inhibitor concentration and the equilibrium constant of adsorption, respectively. The higher value of K_{ads} indicates a strong adsorption of the inhibitor on metal surfaces [18]. Figure 5 shows a Langmuir-linear plots with an average correlation coefficient approach 1 ($R^2 > 0.99$), indicating a well fit with Langmuir's isotherm [15,19]. The intercept of the plot lines give the value of $\log K_{ads}$ which tabulated in Table 4. Values of K_{ads} are high, indicating a strong interaction between the inhibitor and the MS surface [18]. The relation between standard adsorption free energy (ΔG_{ads}^o) and K_{ads} is shown by eqn. (6) [15,16].

$$K_{ads} = \frac{1}{55.5} e^{-\left(\frac{\Delta G_{ads}^o}{RT}\right)} \tag{6}$$

Where 55.5 is the volume ratio of water content in solution in ml L^{-1} , R is the gas constant and T is the absolute temperature. From Table 4, the adsorption process is assumed to be spontaneous and stable where $\Delta G_{ads(average)}^o < -40 \text{ kJ mol}^{-1}$ [15-19]. The mechanism of adsorption is proposed to be physisorption, where $\Delta G_{ads(average)}^o < -40 \text{ kJ mol}^{-1}$ [18,19].

The nature of the interaction in MS/medium interface is elucidated using Temkin adsorption isotherm using eqns. (7a) and (7b) [20,21].

$$e^{(f\theta)} = K_{ads} \cdot c \tag{7a}$$

and it is rearranged

$$\theta = \frac{2.303}{f} \log c + \frac{2.303}{f} \log K_{ads} \tag{7b}$$

Where, K_{ads} , C , θ and f are equilibrium constant, inhibitor concentration, surface coverage, and interaction parameter, respectively. Attraction between adsorbed molecule meaning $f > 0$ and $f < 0$ indicate a repulsion. Figure 6 shows the change of concentration (log form) and the surface coverage (θ), the obtained line has correlation coefficient $R^2 \geq 0.94$ close to unity (Table 5). Average $K_{ads} \approx 0.86$ and $f > 0$ indicating a strong lateral attraction between the Na-EDTA and

Temperature (k)	R ²	K	-ΔG (kJ mol ⁻¹)
298	0.99	11.24	15.94
308	0.99	76.92	20.71
318	0.99	20.08	17.38

Table 4: Parameters of Langmuir isotherm for adsorption of Na-EDTA on MS coupon surface at Red Sea water.

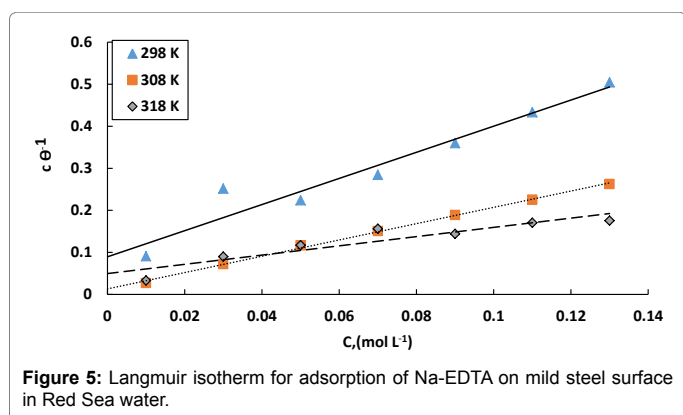


Figure 5: Langmuir isotherm for adsorption of Na-EDTA on mild steel surface in Red Sea water.

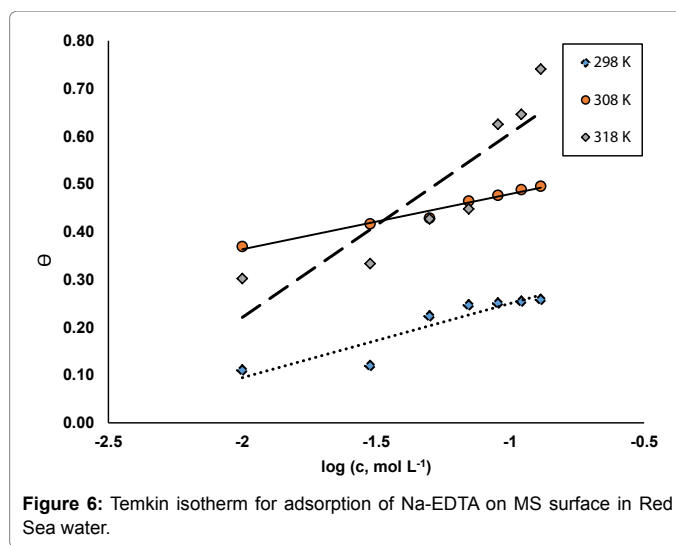


Figure 6: Temkin isotherm for adsorption of Na-EDTA on MS surface in Red Sea water.

Temperature (K)	1/f	k _{ads}	f	R ²	-ΔG (kJ mol ⁻¹)
298	0.229	0.79	4.37	0.94	8.38
308	0.1205	0.88	8.3	0.91	8.65
318	0.118	0.89	8.5	0.88	8.66

Table 5: Parameters of Temkin isotherm for adsorption of Na-EDTA on MS coupon surfaces at Red Sea water.

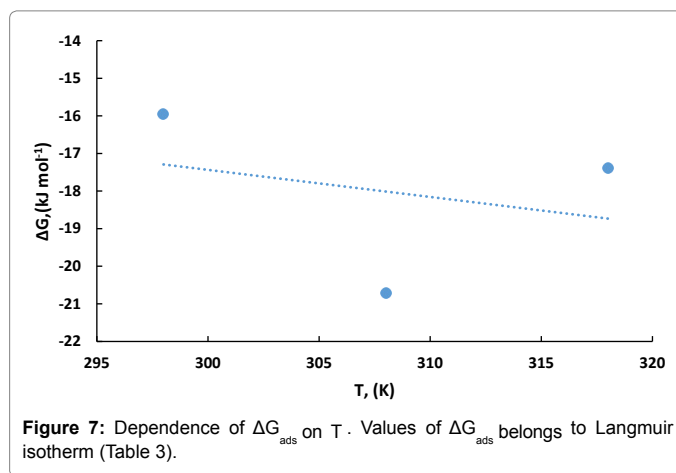


Figure 7: Dependence of ΔG_{ads} on T . Values of ΔG_{ads} belongs to Langmuir isotherm (Table 3).

MS surface [21]. $\Delta G < -40 \text{ kJ mol}^{-1}$ confirm the physisorption of the interaction between Na-EDTA and MS surface.

To calculate enthalpy ΔH_{ads} of the adsorption process, integrated version of Van-Hoff equation is used:

$$\ln K_{ads} = \left(-\frac{\Delta H_{ads}}{T}\right) + const \tag{8}$$

A plot between the variations of $\ln K_{ads}$ and T^{-1} gave a straight line, whose slope is equal to $-\Delta H_{ads} R^{-1}$ as shown in Figure 7.

The entropy of adsorption ΔS_{ads} was calculated using the following thermodynamic equation [22,23]:

$$\Delta S_{ads}^o = \frac{\Delta H_{ads}^o - \Delta G_{ads}^o}{T} \tag{9}$$

Table 6 listed the calculated values of ΔH_{ads} and ΔS_{ads} . The adsorption of Na-EDTA on MS surface in Red Sea water was found to occur endothermically (where, $\Delta H_{ads} > 0$). In the presence of Na-

EDTA, solvent entropy increase (where, $\Delta S_{ads} > 0$) as a result of water displacements from MS surface [24].

Effect of temperature

In order to get more information about the nature of adsorption and the activation processes, the influence of temperature on the inhibition process is important. By regarding the effect of temperature on inhibitors, we can classify inhibitors as:

1. Inhibitors whose IE% decrease with increasing temperature, and E_a will be greater than that in the blank solution;
2. Inhibitors whose IE% and E_a are not changing with temperature variation; and
3. Inhibitors whose IE% increases with raising temperature, but E_a less than E_a of blank solution.

Arrhenius equation eqn. (10) is helpful in studying the effect of temperature on the corrosion process [15,16,24]

$$\log \eta = \frac{-E_a}{2.303RT} + \log A \tag{10}$$

Where ‘ η ’, E_a , R, T, and A are the corrosion rate, the apparent activation energy, the molar gas constant, the absolute temperature, and the frequency factor, respectively. Figure 8 represents an Arrhenius

Temperature (K)	$-\Delta G_{ads}$ (kJ mole ⁻¹)	ΔH_{ads} (kJ mole ⁻¹)	ΔS_{ads} (J mole ⁻¹ ·K ⁻¹)
298	15.94	350.92	1231.07
308	20.71		1206.59
318	17.38		1158.18

Table 6: Adsorption thermodynamic parameters obtained using Langmuir isotherm.

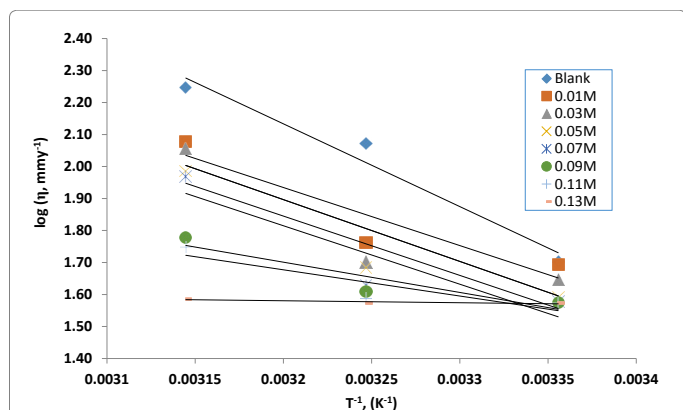


Figure 8: Arrhenius plot as $\log \eta$ vs T^{-1} for MS corrosion in Red Sea water in the absence and presence of various concentrations of Na-EDTA.

c (mol L ⁻¹)	E_a (kJ mol ⁻¹)	ΔH (kJ mol ⁻¹)	ΔS (J mol ⁻¹ K ⁻¹)
Blank	35.31	32.74	-119.60
0.01	-13.77	-16.34	-286.66
0.03	-11.77	-14.33	-280.76
0.05	-17.42	-19.98	-300.86
0.07	-22.15	-24.71	-317.12
0.09	-27.21	-29.77	-334.14
0.11	-32.63	-35.18	-352.37
0.13	-57.42	-59.97	-435.40

Table 7: Thermodynamic parameters, E_a , ΔH and ΔS for MS corrosion in Red Sea water in the absence and presence of various concentrations of Na-EDTA (data from weight loss technique).

plot as $\log \eta$ vs $1/T$ for MS in Red Sea and Red Sea with various concentration of Na-EDTA, E_a was calculated from the slope of the line obtained (Table 7).

From the table, it is seen that E_a increases in the presence of the inhibitors compared to the blank Sea water. The higher value of the E_a in the presence of Na-EDTA is attributed to its physisorption,

According to Eyring relationships eqn. (11), both of S and H could be obtained [15],

$$\ln \frac{\eta}{T} = \ln \frac{R}{Nh} + \frac{\Delta S}{R} + \frac{\Delta H}{RT} \tag{11}$$

Where h is the Planck’s constant, N is the Avogadro’s number, ΔH is the enthalpy of activation and ΔS is the entropy of activation. The kinetic results were found to fit the Arrhenius and Eyring equation, where plots of T^{-1} vs $\log (\eta T^{-1})$ resulted in good straight lines. The activation parameters ΔH and ΔS can be evaluated from the slopes and intercepts of the straight line. Figure 9 shows Eyring plot and all lines are straight from which ΔH and ΔS were evaluated and their values are put in Table 7. The positive values of ΔH reflect the endothermic dissolution of blank MS without any inhibitors, but in the presence of the inhibitor, the value becomes negative and the increase in ΔH with increasing of Na-EDTA concentration for MS corrosion reveals that the decrease in the MS corrosion rate is mainly controlled by kinetic parameters of activation. The negative values of ΔS may reflect the association mechanism of corrosion, i.e., the decrease in disorder takes place in going from reactants to the activated state.

SEM

The SEM image of the MS specimen before and after immersing in Red Sea water for 6 days in the absence and presence of inhibitor system are shown in Figures 10A-10C, respectively.

The SEM micrographs of MS surfaces are represented in Figure 10; no corrosion products appear at a polished MS surface (Figure 10A). The surface becomes rough after immersion in corrosive medium which considered as a corrosion indicator (Figure 10B). When, 0.13 mol L⁻¹ of Na-EDTA is added, MS surface lose the roughness; may be as a result of the formed Na-EDTA/inhibitor complex that reduce the dissolution of MS.

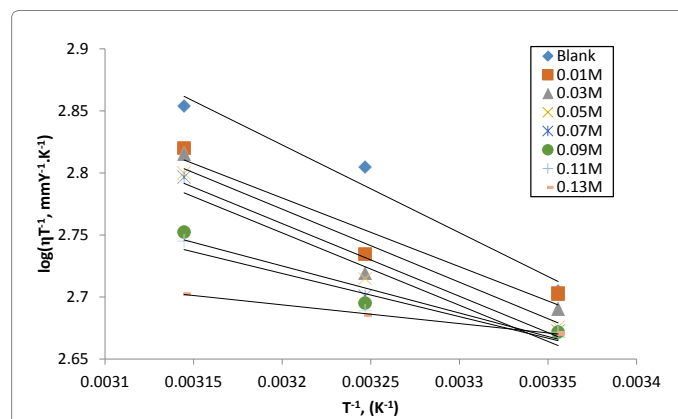


Figure 9: Eyring plot as $\log \eta T^{-1}$ vs T^{-1} for MS corrosion in Red Sea water in the absence and presence of various concentrations of Na-EDTA.

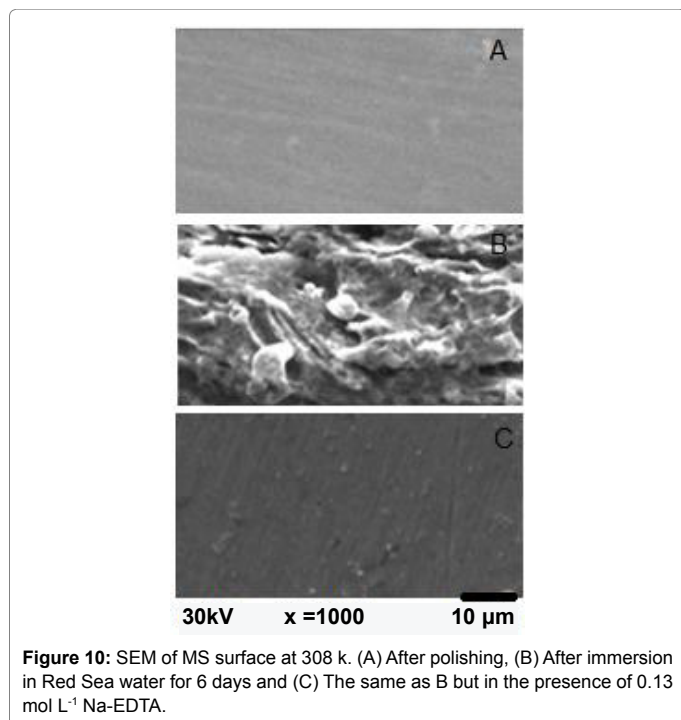


Figure 10: SEM of MS surface at 308 K. (A) After polishing, (B) After immersion in Red Sea water for 6 days and (C) The same as B but in the presence of 0.13 mol L⁻¹ Na-EDTA.

Conclusions

1. Na-EDTA is considered as a good inhibitor (efficiency~88%) for mild steel in the Red Sea water.
2. Increasing any of the reaction temperature and/or Na-EDTA concentrations enhance the inhibition efficiency.
3. The physisorption adsorption mechanism is proposed according to the negative sign of ΔG_{ads} .
4. Experimental data show a good fitting to Langmuir and Temkin isotherms.
5. Thermodynamic parameters revealed that the adsorption process is spontaneous.

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