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FULL LENGTH ARTICLE

Corrosion inhibition efficiency of water soluble ethoxylated trimethylol propane by gravimetric analysis

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KEYWORDS

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Abstract Polyoxyethylenated trimethylol propane monolaurate surfactants with varied ethylene oxide content were tested as corrosion inhibitors via the weight loss method. Weight loss measurements for carbon steel dissolution were performed in 1 N HCl containing different inhibitor concentrations at 30, 40 and 50 °C. These measurements were utilized for calculating corrosion rate, surface coverage area and percentage inhibition. The obtained data show that the adsorption of these inhibitors obeys Langmuir adsorption isotherm. Thermodynamic parameters (ΔG^*, ΔH^* and ΔS^*) of the corrosion process are evaluated and correlated to inhibitors structures.

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1. Introduction

Surfactants are widely used and find a very large number of applications because of their remarkable ability to influence the properties of surfaces and interfaces. They may be applied at all stages in petroleum recovery and processing industry, from oil well drilling, reservoir injection, oil well production and surface plant processes. They also find application as corrosion inhibitors in petroleum industry as it contains a wide variety of corrosive environments. Adsorption type inhibitors represent a large class of inhibitors that are adsorbed through a film onto metal surface to resist penetrations [1,2]. Such inhibitors should possess a hydrocarbon portion attached to a strongly polar group such as nitrogen, sulfur or oxygen. The hydrocarbon end will attract the molecules of the non-polar process stream to provide an additional barrier to a potentially corrosive medium.

An attempt is made by Gontmakher et al. [3] to develop and put into operation the easily available cheap inhibitors for protection of gas well equipment processed by hydrochloric acid in the presence of hydrogen sulfide. The efficiency of a series of inhibitors with surfactants was studied in 15% and 24% HCl at 60–98 °C. It was found that corrosion is slowed down by a factor of 10^2 and eliminating pitting. Growcock [4] proposed a germinal corrosion inhibition scheme for alpha-alkenylphenone benzoyl allyl alcohol (BAA). The corrosion kinetics of J55 steel in HCl solutions inhibited with a mixture

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of BAA and *n*-dodecyl-pyridinium bromide surfactant at temperatures of 10–95 °C was carried out. Al-Lohedan et al. [5] studied the influence of temperature on the adsorption of some cationic surfactants onto steel. The binging constant, the free energy of adsorption, the lateral interaction and the number of active sites are reported. Corrosion inhibition of *N*,*N*,*N*-dimethyl-4-methyl benzyl dodecyl ammonium chloride on mild steel in HCl was investigated by Abdel-Hamid et al. [6]. Also, the corrosion inhibitive effect of water soluble surfactants based on maleic anhydride-oleic acid adduct was investigated by Osman et al. [2]. Incorporation of nonionic surfactant with corrosion inhibitors [7–9] also imparts supplementary significant potency leading to improved inhibition efficiency. Synergistic effect of few surfactants i.e. Cetyl trimethyl Ammonium Chloride, Cetyl Trimethyl ammonium bromide and Cetyl pyridium Chloride toward the corrosion of Carbon Steel in 1.0 M hydrochloric acid solution has been investigated [10] using weight loss and metallurgical research microscopy techniques. Recently, weight loss measurements were used to investigate the corrosion behavior of carbon steel [11], ductile iron [12] and zinc [13] in different acidic environments.

The aim of the present study is to investigate corrosion inhibition efficiency of poly oxyethylentated trimethylol propane monolaurate with varied poly oxyethylene content. These surfactants were prepared in a previous article [14] and were selected due to their efficiency in reducing aqueous surface tension and their low free energy of adsorption. Gravimetric weight loss technique was used for evaluating their corrosion inhibition efficiency in 1 M HCl at different temperatures. These measurements were utilized for calculating corrosion rate, surface coverage area and percentage inhibition. The obtained data show that the adsorption of these inhibitors obeys Langmuir adsorption isotherm. Thermodynamic parmeters (ΔG^*, ΔH^* and ΔS^*) of the corrosion process were evaluated and correlated to inhibitors structures.

2. Experimental

Poly oxyethylentated trimethylol propane monolaurate surfactants with varied poly oxyethylene content namely 15, 30 and 49 ethylene oxide units were prepared (described in a previous work [14]). The chemical structures of the prepared surfactants are shown in Fig. 1. First, trimethylolpropane was ethoxylated with different oxyethylene contents using stainless steel autoclave (Parr Model 4530 USA). Trimethylolpropane (TMP) was mixed with appropriate amount of triethylamine as a catalyst (0.3 wt.% based on TMP) [15] and the temperature was raised to 80 °C. Ethylene oxide (eo) was then added until the desired molar ratio namely (15, 30 or 49 mol) was introduced [16] and the reaction mixture was kept at 80 °C. Second, 3.0 mol of polyoxyethylenated trimethylolpropane was monoesterified with 1.0 mol of lauric acid. The reaction was carried out at the reflux temperature of xylene until the theoretical amount of water of the reaction was collected azeotropically. The samples are denoted as 15TL, 30TL and 49TL, where 15, 30 and 49 denote the number of oxyethylene (eo) repeating units, T for trimethylol propane and L for lauric acid.

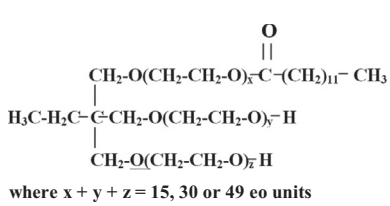

Figure 1 Chemical structure of surfactants.

Carbon steel test specimens were used in the form of sheets with dimensions $2 \times 2 \times 0.2$ cm^3 and 3.2 g average weight. Carbon steel used for the investigations was in the form of sheet having the following composition.

The test specimens were first polished by 310, 410 and 610 emery papers, degreased with acetone, washed with double distilled water and finally dried between two filter papers. Such treatment was carried out immediately before each measurement. Five concentrations namely 100, 250, 500, 1000 and 2000 ppm of each inhibitor were used to evaluate gravimetric loss of carbon steel samples in 1 N HCl at 30, 40 & 50 °C. Weight-loss measurements were carried out as described earlier [17].

3. Results and discussions

The effectiveness of polyoxyethylenated trimethylol propane monolaurate surfactants (15TL, 30TL and 49TL) as corrosion inhibitors for carbon steel in 1 N HCl solutions has been examined using the weight loss method. Weight loss (in mg/cm^3) of the surface area was determined in an open system at various time intervals in the absence and presence of different concentrations of the surfactants. Corrosion rate in mg.cm^{-1}.hr^{-1}, surface coverage area (Θ) and inhibition efficiency % (I%) were determined. Representative experimental results of weight loss in mg/cm^2 for 15TL at 30 °C, 30TL 40 °C and 49TL at 50 °C at different immersion times and different concentrations are represented in Table 1. It is evident from these data that in all cases the dissolution of carbon steel in HCl is characterized by initial slow rate. This may be due to the oxide film originally present on metal surface. S reflected from graphs, weight loss of carbon steel samples increases with increasing time of immersion. The curves obtained in the presence of surfactants fall significantly below that of free acid. The rate of corrosion (in mg.cm^{-1}.hr^{-1}) and surface coverage area Θ with different concentrations of 15TL, 30TL & 49TL surfactants in 1NHCl at different temperatures (30, 40 & 50 °C) are listed in Table 1. Fig. 2 presents variation of inhibition efficiency percentage (I%) of carbon steel upon using different inhibitors' concentrations at varied temperatures where solid lines present I% at 30 °C, dashed lines are for 40 °C and dotted ones are for 50 °C.

It is evident that in all cases the rate of corrosion decreases with increasing surfactant concentration while surface coverage area (Θ) and percentage of inhibition (I%) increase. These results result in a conclusion that, the investigated surfactants are fairly efficient as inhibitors for steel dissolution in acidic medium. Careful inspection of the percentage inhibition at the same temperature and inhibitor concentration, one can get that I% increases with increasing ethylene oxide content. This may be due to the formation of more intensive monolayer adsorption by the excessive electron density of ether linkages of ethylene oxide chains 202,292. In addition, this behavior may be related to minimum area occupied by surfactant molecule (A_{min}) (previously determined [14]) and Θ of inhibitor.

Table 1 Some corrosion parameters for carbon steel in 1 N HCl from weight loss measurements at different temperatures.

Temp. (°C)	Sample	Time (hr)	Weight loss (mg/cm^2)							Corrosion rate mg.cm^{-1}.hr^{-1}			Surface coverage area Θ		
			25	50	75	100	125	150	175	30 °C	40 °C	50 °C	30 °C	40 °C	50 °C
30 °C	1 N HCl	Concn	7.72	16.14	24.3	28.4	32.5	40.22	48.2	–	–	–	–	–	–
	15TL	100	3.18	6.59	9.09	12.72	15.68	21.13	22.45	0.14	0.28	0.31	0.54	0.41	0.33
		250	2.72	4.54	7.95	11.36	14.09	18.4	20.45	0.12	0.26	0.27	0.60	0.44	0.41
		500	2.72	4.09	6.59	8.18	11.8	13.63	16.5	0.10	0.24	0.26	0.66	0.49	0.46
		1000	2.27	3.18	6.14	7.72	10	10.9	13.56	0.09	0.22	0.25	0.71	0.52	0.49
		2000	2.27	3.18	5.6	6.59	7.95	10.22	11.9	0.08	0.21	0.23	0.74	0.55	0.52
40 °C	1 N HCl	Concn	13.6	21.5	34	45	55	60	65	–	–	–	–	–	–
	30TL	100	5.68	12.5	16.3	22.95	25.54	30	33	0.14	0.24	0.32	0.60	0.49	0.34
		250	5.68	10	15.45	22.27	24.77	27.72	32	0.12	0.22	0.28	0.66	0.52	0.42
		500	5.45	9.54	14.1	21.5	23.86	26.81	30.8	0.11	0.21	0.25	0.69	0.54	0.48
		1000	5.45	9.54	12.5	20.9	22.59	26.23	30.5	0.08	0.20	0.24	0.74	0.56	0.51
		2000	5	8.18	10.9	18.18	21.5	24.77	26.81	0.07	0.18	0.21	0.79	0.60	0.55
50 °C	1 N HCl	Concn	13.6	25.7	36	46	53	62	66	–	–	–	–	–	–
	49TL	100	9.52	17.6	21.4	24.76	29	34.5	34.5	0.11	0.20	0.29	0.62	0.57	0.40
		250	8.33	15	20	24	26.8	33.1	34.3	0.09	0.18	0.26	0.70	0.61	0.45
		500	7.85	14.2	17.6	22.4	25.7	30	32	0.07	0.16	0.25	0.76	0.65	0.49
		1000	7.38	13.1	16.4	20.7	24.76	26.2	28	0.06	0.15	0.23	0.78	0.67	0.53
		2000	6.42	11.4	15.2	19.5	21.9	23.57	24.76	0.04	0.13	0.21	0.85	0.70	0.58

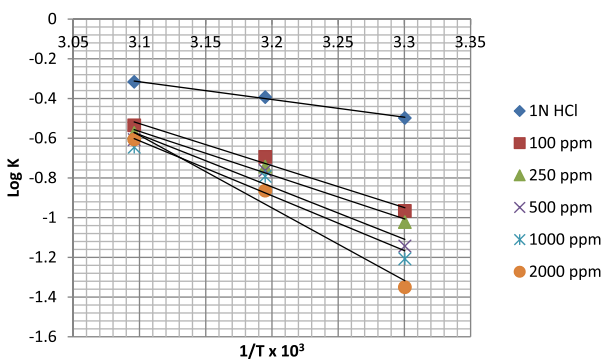

Figure 2 Log K−1/T curves for carbon steel dissolution in 1 N HCl in the absence and presence of different concentrations of 49TL inhibitor.

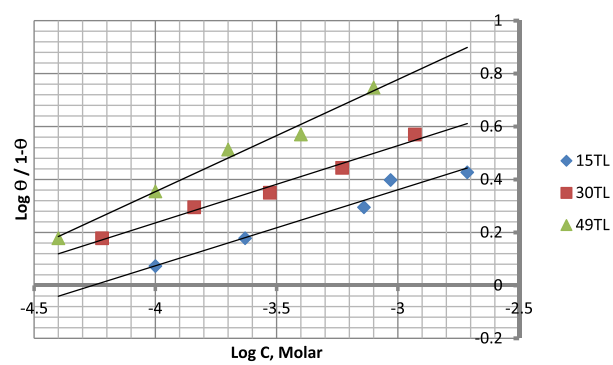

Figure 3 Dependence of log (Θ)/1-Θ on logarithm of concentration for inhibitors 15TL, 30TL and 49TL.

The study of the effect of temperature on inhibition efficiency of corrosion inhibitors is important in elucidation of the mechanism and kinetics of their action and ultimately the proper selection of these inhibitors for specific practical situations. Accordingly, the effect of temperature of the corrosive medium on the reaction proceeding in pure acids was reported by many authors [5,6,18–23]. By analyzing corrosion rate data for inhibitors at increased temperatures from 30 °C to 50 °C, it is clear that the corrosion rate increases with increasing temperature. This result is in accordance with the Arrhenius law (Eq. (1)) in the temperature range used. This behavior confirms that, inhibition of carbon steel dissolution occurs through physical adsorption of inhibitors on metal surface. In other words, the increase in temperature may enhance the desorption process [24]. Arrhenius equation shows that, the smaller the activation energy of reaction (lower energy barrier), the more rapid the reaction at a given temperature, and that for any reaction the rate of reaction will increase with increasing temperature.

Table 2 Energy of activation as determined by weight loss technique in 1 N HCl in the absence and presence of different concentrations of 15TL, 30TL and 49TL.

Concentration ppm	E_a^*, k J			
	Free acid	15TL	30TL	49TL
100	17.73	30.83	38.64	41.29
250		33.43	39.69	41.98
500		40.25	40.58	50.74
1000		42.24	45.78	52.50
2000		44.67	50.04	61.91

$$\text{Log K} = (\text{Ea}^*/2.303R)1/T + \text{const.} \quad (1)$$

Arrhenius plot of log K against reciprocal of absolute temperature (1/T) for 49TL (figures for 15TL and 30TL are not shown here for brevity) is shown in Fig. 3 and enables the activation energy to be calculated from the slope. Ea* values (Table 2) for the inhibitors are much high compared to that of free acid sample. Also it increases with increasing number of oxy-

ethylene repeating units from 15–49 through inhibitor structure. On the other hand, Ea^* values (see Table 2) increase with increasing inhibitor concentration. Ea^* values indicate that the whole process is controlled by surface reaction; since its values are over 20KJ [25,26].

To understand the mechanism of the inhibiting process, the adsorption behavior of the organic adsorbate on metal surface must be known [27]. Adsorption isotherms can provide valuable clues to the inhibition mechanism. Langmuir adsorption isotherm has been used to interpret the monolayer adsorption of the inhibitor molecules. The Langmuir isotherm for monolayer adsorption may be written as in Eq. (2):

$$\log(\Theta/1-\Theta) = \log C + \log A \quad (2)$$

where A is the temperature independent constant, and C is bulk concentration of inhibitor in mM.

Since inhibition efficiency has been taken to represent the surface coverage the results can be represented in the form of Langmuir plots, i.e. log (Θ/1-Θ) vs log C. Dependence of log Θ/1-Θ on logarithm of concentration for 15TL, 30TL and 49TL inhibitors is shown in Fig. 4. Langmuir adsorption isotherm indicates that the inhibitors control the metal dissolution by being absorbed on the metal surfaces and follows Langmuir adsorption mode (see Fig. 3). It is found that Langmuir adsorption gradient deviates from unity, a behavior that may be due to interaction between adsorbed species on metal surface [28–30] whereas ideal Langmuir isotherm is based on the assumption that the adsorbed species do not interact with each other.

Thermodynamic parameters of corrosion process play an important role in defining the spontaneity of metal conversion into corrosion products [5,31]. Thermodynamic parameters of activation for corrosion process (enthalpy (ΔH^*), free energy (ΔG^*) and entropy (ΔS^*)) were calculated for the three inhibitors (15TL, 30TL and 49TL) using Eqs. (3)–(5):

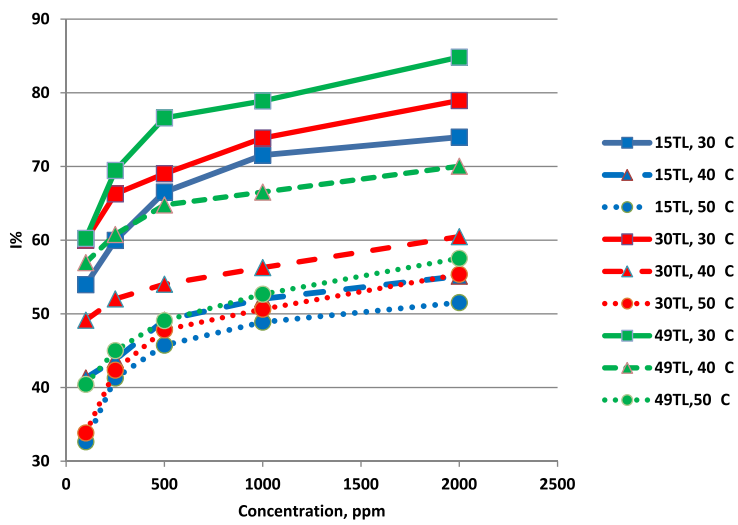

Figure 4 Dependence of corrosion inhibition efficiency (I%) on inhibitor concentration and temperature.

Table 3 Activation parameters of the dissolution reaction of carbon steel in 1 N HCl in the absence and presence of different concentrations of 15TL, 30TL and 49TL at different temperatures.

Concentration (ppm)	ΔH^* (kJ/mol)			ΔG^*(kJ/mol)			ΔS^*(kJ/mol/K)		
	30 °C	40 °C	50 °C	30 °C	40 °C	50 °C	30 °C	40 °C	50 °C
1 N HCl	20.249	20.332	20.415	97.818	100.144	103.287	−256	−255	−256
1 N HCl + 100 ppm of 15TL	33.355	33.428	33.530	99.709	101.548	104.438	−218	−217	−219
1 N HCl + 250 ppm of 15TL	35.953	36.032	36.114	100.180	101.715	104.775	−211	−209	−212
1 N HCl + 500 ppm of 15TL	42.771	42.854	42.947	100.726	101.935	104.906	−191	−188	−192
1 N HCl + 1000 ppm of 15TL	44.768	44.841	44.925	100.985	102.082	105.054	−185	−182	−186
1 N HCl + 2000 ppm of 15TL	47.109	47.279	47.357	101.332	102.244	105.267	−178	−175	−179
1 N HCl + 100 ppm of 30TL	41.167	41.250	41.333	100.167	101.780	104.439	−195	−193	−195
1 N HCl + 250 ppm of 30TL	42.214	42.298	42.381	100.577	102.009	104.782	−192	−190	−193
1 N HCl + 500 ppm of 30TL	43.099	43.182	43.265	100.838	102.104	105.005	−190	−188	−191
1 N HCl + 1000 ppm of 30TL	48.299	48.383	48.466	101.332	101.236	105.193	−175	−172	−175
1 N HCl + 2000 ppm of 30TL	51.884	51.967	52.050	101.854	102.484	105.472	−164	−158	−163
1 N HCl + 100 ppm of 49TL	43.815	43.891	43.981	100.542	102.311	104.631	−119	−119	−119
1 N HCl + 250 ppm of 49TL	44.503	44.586	44.669	100.795	102.543	104.855	−185	−185	−186
1 N HCl + 500 ppm of 49TL	53.260	53.344	53.428	101.530	102.673	105.068	−159	−157	−159
1 N HCl + 1000 ppm of 49TL	55.020	55.103	55.186	101.868	102.841	105.315	−159	−157	−159
1 N HCl + 2000 ppm of 49TL	64.427	64.512	64.596	102.696	103.242	105.580	−126	−127	−126

$$\Delta H^* = Ea^* + RT \quad (3)$$

$$\Delta G^* = RT((lnkT/h) - lnK) \quad (4)$$

$$\Delta S^* = (\Delta H^* - \Delta G^*)/T \quad (5)$$

where R is the universal gas constant, T is absolute temperature, h is Planck's constant, k is Boltzman constant and K is corrosion rate constant.

Table 3 summarizes ΔH^*, ΔG^* and ΔS^* values for 15TL, 30TL and 49TL at 30, 40 and 50 °C for different inhibitors concentrations. ΔH^* values reflect exothermic behavior of all inhibitors on steel surface for metal dissolution process. It is clear that ΔH^* values increase with increasing number of oxyethylene units at the same concentration. Values increase from 33.35 to 41.61 and 43.81 for 15TL, 30TL and 49TL, respectively at 100 ppm and 30 °C. Regarding varied inhibitor concentration, the data reveal that ΔH^* increases with increasing concentration at definite number of oxyethylene units. This increase may be due to inhibitor strength in adsorption on metal surface. Also, relatively low ΔH^* values verify physical adsorption of these inhibitors on steel surface.

Positive ΔG^* values for all inhibitors (see Table 3) reflect high-energy barrier for the corrosion process [30]. On the other hand, higher ΔG^* values for the tested inhibitors than that for 1 N HCl (blank) reveal that the process is activation controlled. The magnitude of ΔG^* increases with increasing number of oxyethylene units at definite temperature and concentration. In addition, the data show an increase in ΔG^* with increased inhibitor concentration.

Table 3 shows large negative values for ΔS^* which implies that the activated complex in rate determining step represents association rather than dissociation. In other words, a decrease in disordering takes place on going from reactants to activated complex [16,32,33].

4. Inhibition mechanism

The inhibition process could be explained as follows: Non-ionic surfactant molecules with long hydrocarbon chain are adsorbed on the metal surface through the hydrophilic parts, which contain oxygen, nitrogen or sulfur atoms. On the other hand, the hydrocarbon chains tend to be curled up in water to minimize the area of contact between themselves and water molecule [34].

The inhibition effect of oxyethylenated trimethylol propane, on the steel surface may be explained as follows: surface active agents have a characteristic structure consisting of a structural group (two dodecyl croups), that has very little attraction for the solvent, known as a hydrophobic group together with a group that has strong attraction for the water solvent (different numbers of oxyethylene repeating units) called the hydrophilic group. When the surfactant molecule is dissolved in water, the presence of the hydrophobic group in the interior of the solvent causes a distortion of the solvent liquid structure, increasing the free energy of the system [34]. In aqueous solution, the hydrogen bonding between water molecules is distorted by the presence of non-polar substances. Therefore, the water tries to expel the nonpolar substances completely as a separate phase. This, however, is not possible as the hydrophobic group of the surfactant is hydrated and expelling would require preventing the adsorption of the inhibitor molecules on the steel surface. Inspecting data presented in Fig. 2, show that reasonable inhibition efficiency toward the corrosion of steel in 1 N HCl was obtained. This can be explained as follows [35]: the binding force between the hydrophilic group (terminal OH group) and iron metal is stronger than the force between expelling the hydrophobic group and aqueous media. Under such conditions a diffusion barrier to chemical and/or electrochemical attack of the solution on metal surface is established. Also, the inhibition efficiency increases with increasing number of ethylene oxide units. This may be due to the interior oxygen atoms (of ethylene oxide units) which fix the molecule to adsorb on the surface of iron. This occurs by the hydrogen bonding between the oxygen atom of ethylene oxide unit and the hydrogen of water molecules.

5. Conclusions

The following conclusions can be drawn from the study:

(1) Surfactant inhibitors show high inhibitive efficiency for iron dissolution in 1 N HCl.
(2) Corrosion inhibition efficiency increases with increasing number of oxyethylene repeating units through inhibitor structure.
(3) The degree of surface coverage area-exerted by inhibitor molecules on carbon steel surface-increases with increasing inhibitor concentration.
(4) Inhibitor molecules are adsorbed on carbon steel surface blocking reaction sites.
(5) Energy of activation increases with increasing inhibitor concentration and number of oxyethylene repeating units through inhibitor structure.
(6) Relatively low ΔH^* values verify physical adsorption of these inhibitors on steel surface.

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