Thermodynamic studies, Adsorption Behavior and Surface Investigation of Hexadecyltrimethylammonium Bromide on mild steel in Oilfield Brackish Water and Effluent Water

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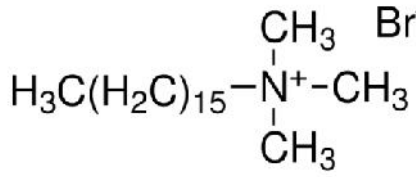
Abstract:- The effect of Hexadecyltrimethylammonium Bromide as corrosion inhibitor on mild steel in Brackish Water and Effluent Water was studied by weight loss technique at temperature range of 25°C, 30°C, 40°C and 50°C. The thermodynamics factors such as ΔH_a^* is the change in enthalpy of activation E_a^* apparent activation energy, ΔS_a^* is the change in entropy of activation were determined for inhibitive properties of Hexadecyltrimethylammonium Bromide and the adsorption parameters like ΔG_{ads}, ΔH_{ads} and ΔS_{ads} were determined and discussed. The corrosion rate (CR) of mild steel increased and inhibition efficiency (IE) decreased with increase in temperature and follows Arrhenius equation in all concentrations. Adsorption of HDTMABr on the mild steel surface obeyed the Langmuir adsorption isotherm. HDTMABr is a mixed type of inhibitor. The results were well supported by SEM studies.

Key Words:- Hexadecyltrimethylammonium Bromide (HDTMABr), Mild steel, Activation energy, Gibb's Free Energy, Scanning Electron spectroscopy (SEM).

Introduction:- Corrosion of mild steel in Brackish water and Effluent water is a major problem in oilfield industries and petrochemical industries. Mild steel is used in oil and gas industry due to cost-effective and it is easy to fabricate for various types of vessels, storage tanks, chemical reactors, heat exchangers, boiler systems, oil and gas transport pipelines, and many other equipment. **[1]** Corrosion of metals is inevitable and thus can never be stopped completely, but can be significantly controlled. Hexadecyltrimethylammonium bromide is used as corrosion inhibitor in this study. It is quaternary ammonium surfactant commercially available compound and it is an effective antiseptic agent against bacteria and fungi. The present work discuss about the thermodynamic factors and discuss the effect of temperature range of 25°C, 30°C, 40°C and 50°C on mild steel in the presence and absence of HDTMABr, activation energies and adsorption parameters. The molecular formula of HDTMABr bromide is $C_{19}H_{42}BrN$. The molecular structure of HDTMABr compound is given below in Fig.1



$$H_3C(H_2C)_{15}-\overset{\overset{\displaystyle CH_3}{|}}{\underset{\underset{\displaystyle CH_3}{|}}{N^+}}-CH_3 \quad Br^-$$

Fig 1: Molecular Structure of Hexadecyltrimethylammonium bromide

EXPERIMENTAL

Materials and methods

Surface Preparation of the Corrosion Coupon:

The specimens used for corrosion tests were Mild Steel (MS) coupons with a dimension of 2 x 2 cm area have been used. Chemical composition of Mild steel coupons (C=0.20%, Mn=1.00%, Si=0.05%, S=0.025%, P=0.25% and Fe=98%) have been used.**[2]** The surface of the specimens was polished using emery paper (Silicon carbide, grade 200 - 800), rinsed with distilled water, dried and immersed in acetone for 5 sec, and finally dried at room temperature and then weighed. These polished coupons were subjected to water test in agreement as per international standards i.e. NACE SP-0775 2013 and ASTM G1–03 to make sure the metal surfaces were free of scratches and other apparent defects like pits. All reagents that were used for the study were of analytical grades and double distilled water.**[3]**

Preparation of Hexadecyltrimethylammonium bromide (HDTMABr) Solution

Brackish and Effluent water were used for weight loss method. It was used to make different concentrations of 0.1, 0.2 and 0.3 Molarity prepared for Hexadecyltrimethylammonium bromide solutions. The inhibitor is soluble in hot water.

Weight Loss Corrosion Coupon Analysis

The mild steel corrosion coupons were immersed in BRW and EFW at different concentrations for 1 day weight loss. The experiments were carried out to study the corrosion inhibition effects of HDTMABr as inhibitor at different concentrations.

In the weight loss experiment, the mild steel coupons were accurately weighed and then immersed in the test solution with blank and remaining with inhibitor at different concentrations of 0.1M, 0.2M and 0.3M prepared for HDTMABr kept at constant temperature range 25°C, 30°C, 40°C and 50°C throughout the experiment. After the period of 1 day, the mild steel coupons were removed, scrubbed with 000 grade steel wool in soap solution, degreased with acetone, dried in warm air and re-weighed.

A calculation of average corrosion rate, expressed as uniform rate of thickness loss per unit time in mils per year (mpy), is shown in equation (1)

$$\mathrm{CR} = \frac{\mathrm{WX365X1000}}{\mathrm{ATDX}\,(2.54)^3} = \frac{2.227\mathrm{X}10^4\ \mathrm{XW}}{\mathrm{ATD}} \qquad (1)$$

Where CR = Average corrosion rate, mils per year (mpy)

W = Weight loss in grams (g)

A = Coupon surface area in square inches (in^2)

T = Exposed time in days (d)

D = Density of the metal coupon in grams per cubic centimeter (g/cm^3)

Guidelines for general and pitting corrosion rates are classified 'Low, Moderate, High and Severe' as per NACE as shown in **Table-1**

Table 1. Corrosion and Pitting Rates classification as per NACE SP0775-2018 (mpy = mils per year)

General Corrosion	Corrosion rate	Pitting Corrosion Rate	Pitting Rate
Low	<1 mpy	Low	<5 mpy
Moderate	1-4.9 mpy	Moderate	5-7.9
High	5-10 mpy	High	8-15

Severe	**>10 mpy**	**Severe**	**>15**

The inhibition efficiency of all the inhibitors was determined for three different concentrations was measured using the equation (2)

$$\text{Inhibition Efficiency (I\%)} = \frac{W_0\text{-}W}{W_0} \text{ X } 100 \qquad (2)$$

Where W is the loss in weight with inhibitor in the solution and W_0 is the loss in weight without inhibitor in solution.

During the weight loss study of mild steel in BRW and EFW, Hexadecyltrimethylammonium Bromide (HDTMABr) used as inhibitor.

RESULTS AND DISCUSSION:-

Analysis of Weight Loss Study

The corrosion rate is temperature dependent. The effect of temperature plays a significant role in electrochemical reaction on mild steel dissolution. To understand the effectiveness of corrosion inhibitors in BRW and EFW on mild steel at different temperatures with blank and with different concentrations from 0.1M to 0.3M of Hexadecyltrimethylammonium Bromide was measured in the temperature range of (25°C, 30°C, 40°C and 50°C) for 1 day of immersion time using weight loss measurements. A change in temperature directly influence not only on the inhibiting effect of corrosion inhibitor but also decompose the corrosion inhibitor which can increase the chances of damaging the protective film leading to further attack.Average rate of corrosion is calculated and explained as loss in thickness uniformly per time in unit as mils per year (mpy) is given in equation (1) and the efficiency of corrosion inhibition *IE* (%) was measured as per equation (2). The weight loss results of mild steel in brackish and effluent water with and without inhibitor of different inhibitors concentrations are presented in Tables 2 The plot of % IE versus Concentration of HDTMABr at different temperature shows the IE rises with rise in concentration and the plot of CR versus Concentration explains corrosion rate of mild steel decreases with rise in inhibitor concentration and increases with rise in temperature.**[5]** According to the Tables 2 and 3 results, HDTMABr had shown the maximum inhibition efficiency is found to be 86% in BRW and 85% in EFW at 0.3M concentration at 25°C and its corresponding corrosion rate is 0.18 mpy in BRW and 0.09 mpy in EFW this is the lowest corrosion rate among all the values of different concentration at different temperatures. The corrosion rate increases significantly with the rise in temperature reached from 1.26 to 3.26 mpy in BRW and from 0.6 to 2.31 mpy in EFW in the absence of inhibitor which is comparatively higher than the values obtained in the presence of inhibitor. From the Fig. 2 and 4 it is clear from the results that there is substantial increase in corrosion rate with rise in temperature. It should also be noted from the Fig. 3 and 5 that there is increased efficiency of inhibitor enhanced with addition of more amount in concentration. It is obvious that the values of IE in % increases with concentration of inhibitor. The corrosion rate decreased in presence of inhibitor as compared to the uninhibited solution. Therefore, we assumed that higher surface coverage will increase inhibition efficiency and inhibitor formed a protecting layer on the metal/solution interface.

Table 2. Different concentrations of Hexadecyltrimethylammonium Bromide (HDTMABr) for 1 day exposure at different temperatures with and without inhibitor by weight loss parameters of mild steel coupons in Brackish Water (BRW).

BRW								
Inhibitor	**Conc. (M)**	**Temp (ºC)**	**Initial Wt.**	**Final Wt.**	**Wt. Loss (mg)**	**CR (mpy)**	**IE (%)**	**Surface Coverage**
Blank	0	25	9.1316	9.1291	0.0025	1.26	0.00	0
HDTMABr	0.1		9.6668	9.6657	0.0011	0.55	56.00	0.560
	0.2		9.1394	9.1386	0.0008	0.40	68.00	0.680
	0.3		9.5679	9.5676	0.0003	0.18	86.00	0.860
Blank	0	30	9.1305	9.1268	0.00370	1.86	0.00	0
HDTMABr	0.1		9.6665	9.6646	0.00190	0.95	48.65	0.486
	0.2		9.1382	9.1369	0.00135	0.68	63.51	0.635
	0.3		9.5671	9.5663	0.00080	0.40	78.38	0.784
Blank	0	40	9.1291	9.1246	0.00450	2.26	0.00	0
HDTMABr	0.1		9.6657	9.6632	0.00250	1.26	44.44	0.444
	0.2		9.1373	9.1351	0.00220	1.10	51.11	0.511
	0.3		9.5660	9.5646	0.00140	0.70	68.89	0.689
Blank	0	50	9.1260	9.1195	0.00650	3.26	0.00	0
HDTMABr	0.1		9.6643	9.6600	0.00430	2.16	33.85	0.338
	0.2		9.1351	9.1317	0.00340	1.71	47.69	0.477
	0.3		9.5646	9.5613	0.00330	1.66	49.23	0.492

Table 3. Different concentrations of Hexadecyltrimethylammonium Bromide (HDTMABr) for 1 day exposure at different temperatures with and without inhibitor by weight loss parameters of mild steel coupons in Effluent Water (EFW).

EFW								
Inhibitor	**Conc. (M)**	**Temp (ºC)**	**Initial Wt.**	**Final Wt.**	**Wt. Loss**	**CR (mpy)**	**IE (%)**	**Surface Coverage □□□**
Blank	0.00	25	9.0560	9.0548	0.0012	0.60	0.00	0
HDTMABr	0.1		9.2285	9.2279	0.0006	0.29	52.50	0.525
	0.2		9.5831	9.5828	0.0003	0.15	75.00	0.750
	0.3		9.1619	9.1617	0.0002	0.09	85.00	0.850
Blank	0	30	9.0658	9.0643	0.0015	0.75	0.00	0
HDTMABr	0.1		9.2278	9.2270	0.0008	0.40	46.67	0.467
	0.2		9.5828	9.5821	0.0007	0.36	52.00	0.520
	0.3		9.1618	9.1613	0.0005	0.25	66.67	0.667
Blank	0.0	40	9.0643	9.0617	0.0026	1.31	0.00	0
HDTMABr	0.1		9.2267	9.2251	0.0016	0.80	38.46	0.385
	0.2		9.5819	9.5804	0.0015	0.75	42.31	0.423
	0.3		9.1601	9.1588	0.0013	0.65	50.00	0.500
Blank	0	50	9.0539	9.0511	0.0028	1.41	0.00	0
HDTMABr	0.1		9.2252	9.2231	0.0021	1.05	25.00	0.250
	0.2		9.5801	9.5783	0.0018	0.90	35.71	0.357
	0.3		9.1585	9.1569	0.0016	0.80	42.86	0.429

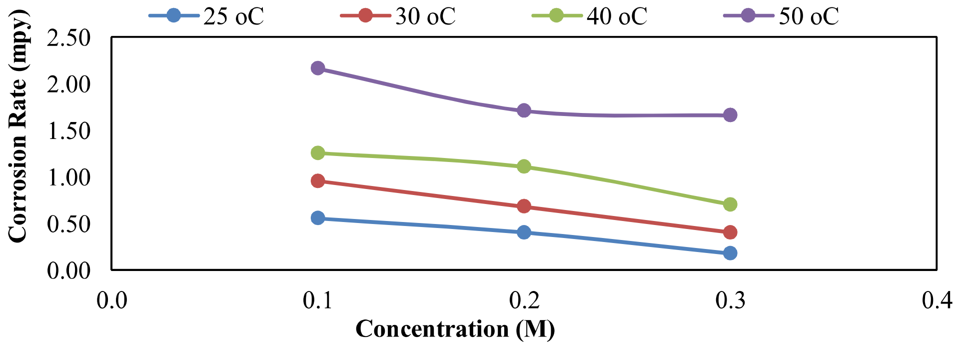


Fig 2. Corrosion rate versus Concentration at various temperature for HDTMABr for 1 day exposure in BRW

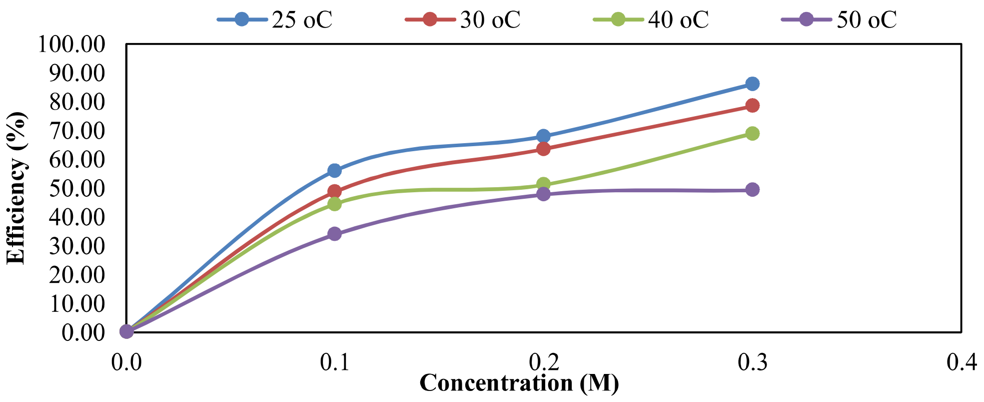


Fig 3. Efficiency versus Concentration at various temperature for HDTMABr for 1 day exposure in BRW

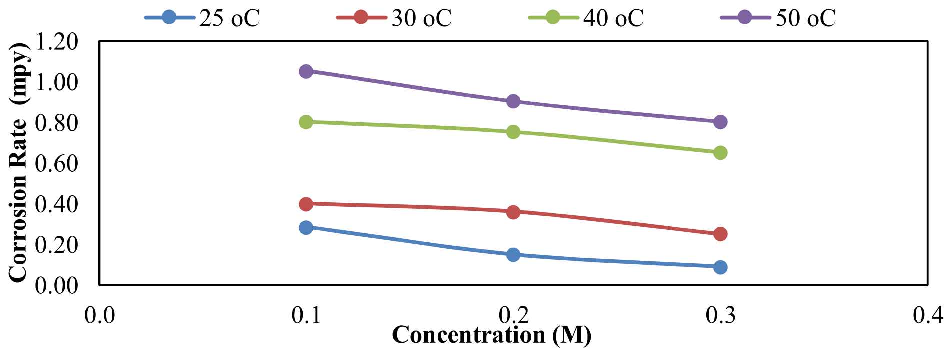


Fig 4. Corrosion rate versus Concentration at various temperature for HDTMABr for 1 day exposure in EFW

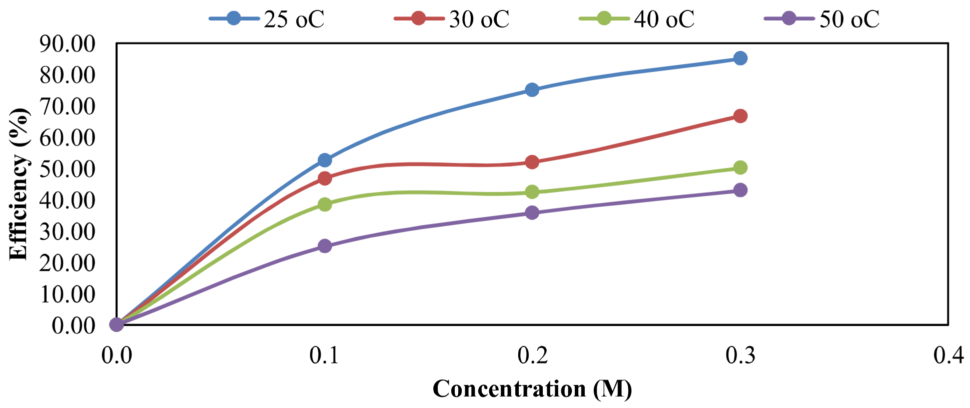


Fig 5. Efficiency versus Concentration at various temperature for HDTMABr for 1 day exposure in EFW

Thermodynamic studies for corrosion parameter

To check the temperature effect on mild steel and mechanism of corrosion reaction can be best represented by Arrhenius and transition state equations.**[8]**The activation energy of the metal dissolution with and without inhibitor can be evaluated by the following equation (3)

$$\mathrm{CR} = \mathrm{K}\exp\left(-\frac{\mathrm{E}_a^*}{\mathrm{RT}}\right) \qquad (3)$$

Arrhenius equation can also be written as (3)

$$\mathrm{CR} = \frac{\mathrm{RT}}{\mathrm{Nh}}\exp\frac{\Delta \mathrm{S}_a^*}{\mathrm{R}}\exp\left(-\frac{\Delta \mathrm{H}_a^*}{\mathrm{RT}}\right) \qquad (4)$$

Where, *N* is Avogadro's number, *h* is Planck's constant, *k* is pre-exponential constant, and *R* is the molar gas constant. Thermodynamic factors like the $\Delta \mathrm{H}_a^*$ is the activation in enthalpy change E_a^* apparent activation energy, $\Delta \mathrm{S}_a^*$ is the entropy of activation were calculated and values are tabulated in Tables 4. Using Eq. (3) and from a plot of the ln CR *versus* 1/*T* (Fig. 6. – 7.), the values of E_a^* and *k* at different concentrations of HDTMABr were calculated from slopes and intercepts. Further, using Eq. (4), plots of ln (C_R/T) *versus* 1/*T* gave straight lines (Fig. 8.-9.) with a slope of ($-\Delta \mathrm{H}_a^*/R$) and an intercept of [ln (R/Nh) + $\Delta \mathrm{S}_a^*/R$], from which the value of $\Delta \mathrm{H}_a^*$ and $\Delta \mathrm{S}_a^*$ were calculated. In the present study, it is observed that change in energy of activation E_a^* and in enthalpy change of activation $\Delta \mathrm{H}_a^*$ in the existence of inhibitor are more positive as compared to absence of inhibitor. Based on the data given in Tables 4 the values of activation energy are increases as inhibitor concentration increases. In BRW the value of E_a^* in blank solution is 27.76 kJ/mol and increases as the concentration of inhibitors increases. The value of E_a^* at the highest concentration of 0.3M is 66.43kJ/mol in case of HDTMABr. In EFW the value of E_a^* in blank solution is 28.41kJ/mol and increases as the concentration of inhibitors increases. The value of E_a^* at the highest concentration of 0.3M is 67.41kJ/mol in case of HDTMABr. The positive values of enthalpies ($\Delta \mathrm{H}_a^*$) indicates the endothermic structure of mild steel indicating that additional energy was required for the activated state and the inhibitor adhere firmly to metal surface at high temperature. Therefore, deterioration of metal is prevented in aqueous medium. The negative value of activation of entropy ($\Delta \mathrm{S}_a^*$) show that this reaction favors to product formation by an association of molecules than a dissociation.**[9]**

Table 4. Thermodynamic parameters for mild steel in BRW and EFW with and without inhibitor of different concentrations of HDTMABr,

Inhibitor	**Conc. (M)**	E_a^* **(kJ mol^{-1})**	**K (mg cm-2 h^{-1})**	$\Delta \mathrm{H}_a^*$ **(kJ mol^{-1})**	$\Delta \mathrm{S}_a^*$ **(J mol^{-1} K^{-1})**
BRW	0.0	27.76	$1*10^5$	28.00	-148.65
	0.1	39.62	$5*10^6$	39.98	-115.02
	0.2	44.00	$2*10^7$	44.42	-102.79
	0.3	66.43	$9*10^{10}$	67.11	-33.14

EFW	0.0	28.41	$6*10^{5}$	26.15	-160.94
	0.1	42.39	$8*10^{6}$	40.32	-119.55
	0.2	54.70	$7*10^{8}$	52.59	-81.85
	0.3	67.41	$8*10^{10}$	65.43	-42.65

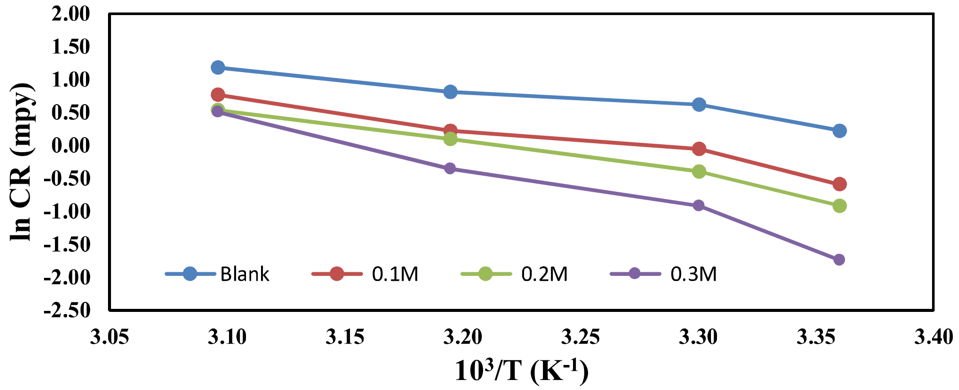


Fig. 6 Arrhenius plots for corrosion in BRW on mild steel in the presence and absence of inhibitor of various concentrations of Hexadecyltrimethylammonium Bromide

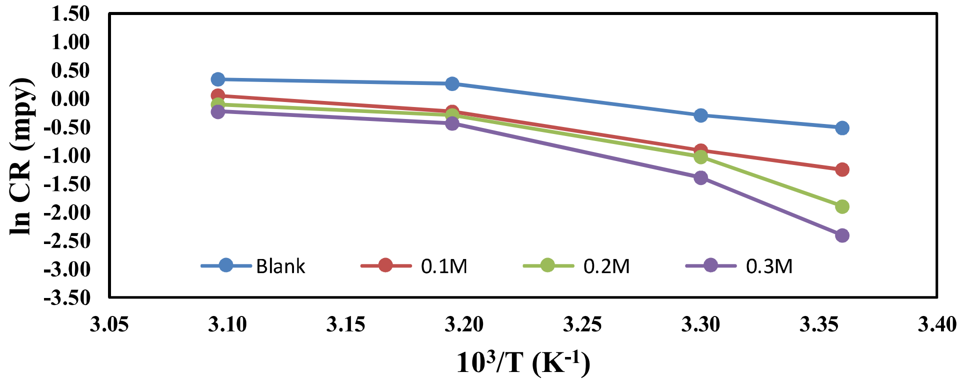


Fig. 7 Arrhenius plots for corrosion in EFW on mild steel in the presence and absence of inhibitor of various concentrations of Hexadecyltrimethylammonium Bromide

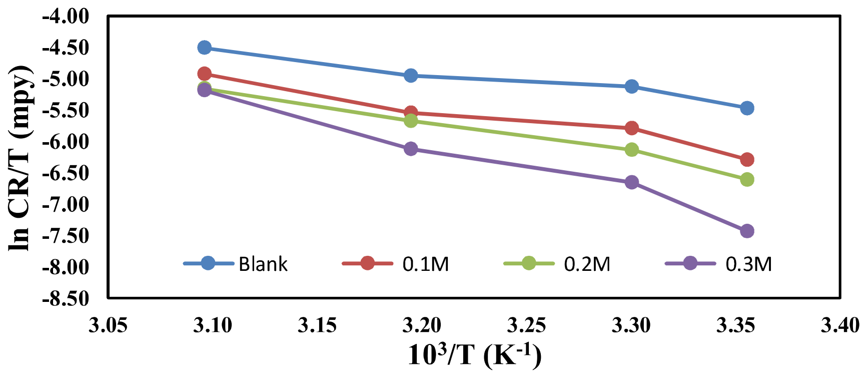


Fig. 8 Alternative Arrhenius plots for corrosion in BRW on mild steel in the presence and absence of inhibitor of various concentrations of Hexadecyltrimethylammonium Bromide

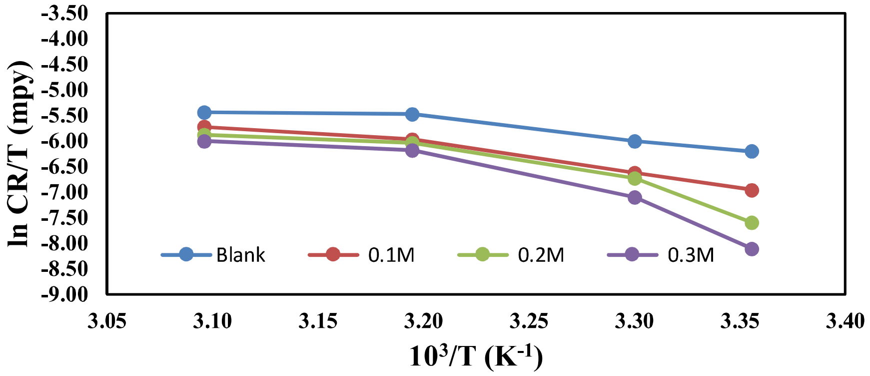


Fig. 9 Alternative Arrhenius plots for corrosion in EFW on mild steel in the presence and absence of inhibitor of various concentrations of Hexadecyltrimethylammonium Bromide.

Adsorption isotherm

The adsorption of inhibitor molecules describes the total surface coverage on mild steel.

Langmuir adsorption isotherms proved to be best to fit θ values which can be explained by the given equation.**[10]** (5)

$$\frac{\mathrm{C}}{\theta} = \frac{1}{\mathrm{K_{ads}}} + \mathrm{C} \qquad (5)$$

Where, K_{ads} is the equilibrium adsorption constant, C is expressed as inhibitor concentration in (g/L), θ is the surface coverage in degree, A plot of *C/θ* versus *C* gave a straight line (Fig. 10 – 11) with a slope almost equal to unity which confirm adsorption of HDTMABr on metal surface in BRW and EFW follows the adsorption by Langmuir isotherm with intercept equal ($1/K_{ads}$). The equilibrium adsorption constant, ($1/K_{ads}$) is associated to the adsorption of standard Gibb's free energy of (ΔG_{ads}) by the equation below (6)

$$\mathrm{K_{ads}} = 1/55.5\ \exp[-\Delta \mathrm{G_{ads}}/\mathrm{RT}] \qquad (6)$$

Where, T is the temperature in kelvin, 55.5 is the molar concentration of H_2O ((mol L^{1}), R is the gas constant. The measured ΔG_{ads} values of HDTMABr are listed in Tables 5. The change in enthalpy of adsorption and change in entropy of adsorption (ΔH_{ads} and ΔS_{ads}) can be measured by the following equation (7).

$$\ln \mathrm{K_{ads}} = \ln\frac{1}{55.5} - \frac{\Delta \mathrm{H_{ads}}}{\mathrm{RT}} + \frac{\Delta \mathrm{S_{ads}}}{\mathrm{R}} \qquad (7)$$

The Gibb's free energy can be measured by thermodynamic equation. (6)

$$\Delta \mathrm{G_{ads}} = \Delta \mathrm{H_{ads}} - \mathrm{T}\Delta \mathrm{S_{ads}} \qquad (8)$$

By (8) equation and from a plot of ΔG_{ads} *vs*. *T* (Fig. 10 and 11) the values of ΔS_{ads} and ΔH_{ads} were figured from slopes and intercepts, correspondingly and the results are tabulated in Table 5. The values of ΔS_{ads} and ΔH_{ads} provide related information of corrosion mechanism. The consistent negative values of ΔG_{ads} confirms that inhibitor molecule adsorbed the on mild steel.

From the thermodynamic point of view larger values gives good indication of strong adsorption. From the Table 5 the calculated values of free energy of ΔG_{ads} obtained ranges from -14.9 kJmol-1 to -16.3 kJmol-1 in BRW & from -15.3 kJmol-1 to -19.4 kJmol-1 in EFW.

The Thermodynamic parameter gives valuable information for the mechanism and the adsorption of inhibitor molecules. When $\Delta H_{ads} > 0$ signifies an endothermic reaction while $\Delta H_{ads} < 0$ is ascribed to an exothermic reaction either physical adsorption or chemical adsorption or both reaction. In BRW the measured values of ΔH_{ads} for the adsorption of inhibitor is negative in HDTMABr indicating the adsorption is exothermic process. While in EFW the calculated data of ΔH_{ads} for the adsorption of inhibitor shows positive values indicating the adsorption is endothermic process.

The ΔG_{ads}value for all inhibitors are negative which represent that the reaction favors to product by complex formation characterizes an association of molecules relatively than a dissociation signifying that jumbling occurs on moving from reactant to the product in the rate determining step.**[11]**

Table 5. Thermodynamic factors from Langmuir adsorption isotherm of Hexadecyltrimethylammonium Bromide on mild steel in BRW and EFW at different temperatures

Water	T (K)	K_{ads} **(L mol^{-1})**	**-ΔG_{ads} (kJ mol^{-1})**	**ΔH_{ads} (kJ mol^{-1})**	**-ΔS_{ads} (J mol-1K^{-1})**
BRW	298	9653	-15.6	-9.85	-19.5
	303	10593	-16.1		
	313	7138	-15.6		
	323	7843	-16.3		
EFW	298	9294	-15.5	27.22	-142.2
	303	8780	-15.6		
	313	12285	-17.0		
	323	20747	-18.9		

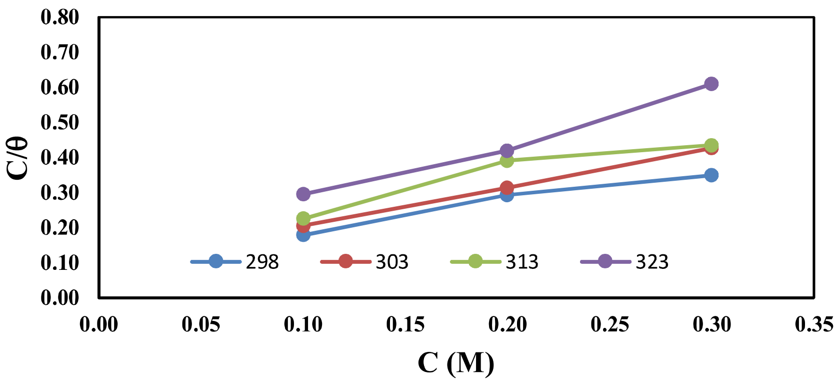


Fig. 10 Adsorption of Hexadecyltrimethylammonium Bromide by Langmuir isotherm in BRW at various temperatures in kelvin

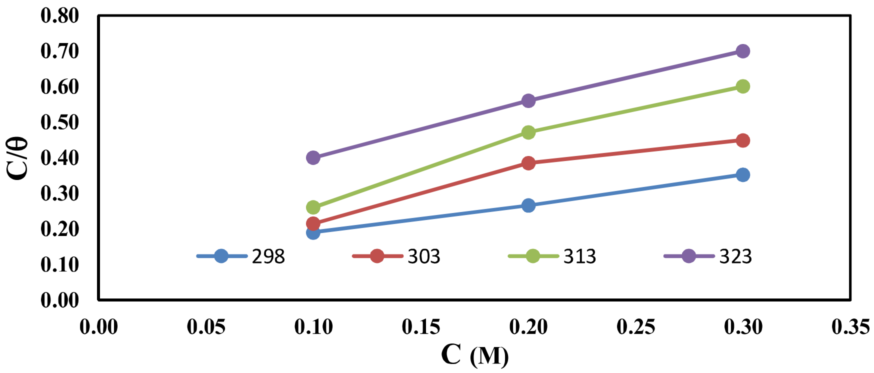


Fig. 11 Adsorption of Hexadecyltrimethylammonium Bromide by Langmuir isotherm in EFW at various temperatures in kelvin

Scanning Electron Microscopy (SEM) of Corrosion Coupons:-

The scanning electron microscope has distinctive abilities for studying the surfaces morphology of mild steel coupons and is accepted the most adaptable techniques used for the investigation of corrosion damage by identify the micro cells on metal.**[12]** Polished mild steel corrosion coupons used for scanning electron microscopic (SEM) studies to check the Morphological Surface analysis prepared in 0.5M solution with and without (HDTMABr) corrosion inhibitor in brackish and effluent water for 30 days at room temperature. After 30 days, the tested coupons were cleaned with demineralized water, dried in desiccator and then examine in scanning electron microscopy, then the results recorded gives the effectiveness of inhibitor adsorbed on mild steel surface. Zeiss sigma scanning electron microscope has been used in the present study. Fig. 12 show the clear images of new coupon to check the defects or any damages on the surface of mild steel and it was observed that new coupon was free from cracks or pits. Fig. 13 show the morphology of corroded surfaces, when mild steel coupons were exposed in brackish water without inhibitor. Uniform corrosion was observed, and pits can be seen clearly damaging the surface without inhibitor. Fig. 14 show the surface morphology of the corroded surfaces when mild steel coupons were exposed in Effluent water without inhibitor. Uniform corrosion with few pits and flakes are seen which show corrosion products like metal hydroxide and its oxides was observed in the micrographs. In Fig.15 the metal surface is completely covered with HDTMABr in BRW showing maximum corrosion protection by the formation of thin film and in Fig. 16 some white flakes can be seen with the thick layer of inhibitor in case of EFW. This observation reveals that HDTMABr was observed as effective corrosion inhibitor in both the field waters (BRW and EFW) which gives complete protection from corrosion. HDTMABr and HMTA works good in corrosion protection which is confirmed from the SEM investigations.

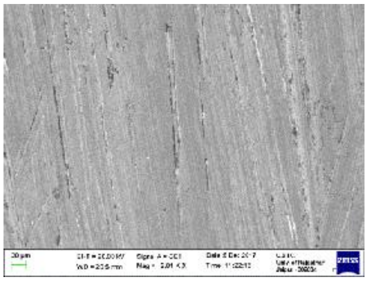
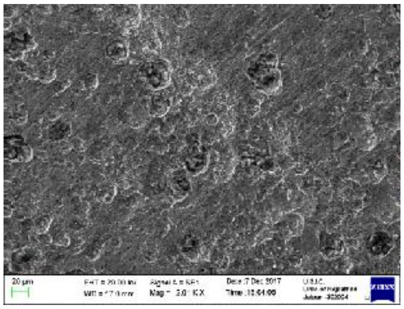
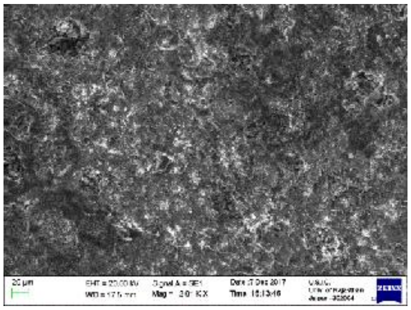


Fig. 12 SEM image at 2000 magnification on Plain Mild Steel coupon



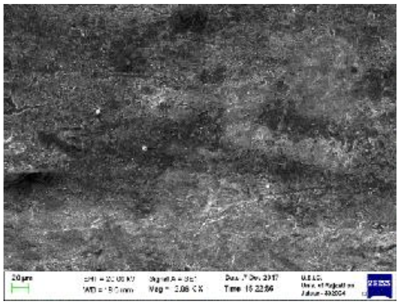
(Fig. 13)

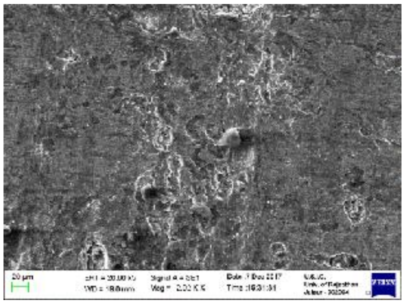


(Fig. 14)

Fig. 13 SEM image at 2000 magnification on Mild Steel coupon in BRW

Fig. 14 SEM image at 2000 magnification on Mild Steel coupon in EFW





CONCLUSIONS:- This research investigated the phenomenon of corrosion of mild steel metal in BRW and EFW. Weight loss method, SEM analysis were used in this study. From the result and discussions, the following conclusions were derived.

- Hexadecyltrimethylammonium bromide works as a good corrosion inhibitor mild steel in BRW and EFW.

- The inhibition efficiency increases with rise in inhibitor concentration and decreased with rise in temperature.
- In the present study, it is observed that activation energy E_a^* and enthalpy of activation ΔH_a^* in the existence of inhibitor are more positive as compared to absence of inhibitor.
- The adsorption of inhibitor molecules follows Langmuir adsorption isotherms.
- The ΔG_{ads} value are negative which represent that the reaction favors to product in the rate determining step.

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