Study on the inhibition of Mild steel corrosion by N, N- dimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide in HCl Medium

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Abstarct: N, N- dimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide has been evaluated as a corrosion inhibitor for mild steel in 1MHCl solution using potentiodynamic polarization, electrochemical impedance spectroscopy and atomic force microscope and Scanning electron microscope measurements. According to the experimental results, inhibition efficiency increased with increasing inhibitor concentration. Potentiodynamic measurements showed that the presence of inhibitor molecules decreases cathodic and anodic currents and shifts corrosion potential to a more positive potential region. Adsorption of the inhibitor molecules to the mild steel surface obeyed the Langmuir adsorption isotherm. Adsorption free energy was calculated from the adsorption isotherms as -31.33 to -39.0kJmol-1. The negative value of ΔG_{ads} showed that the adsorption process is spontaneous for the studied conditions. In addition, measurements were carried out at different solution temperatures to determine temperature dependence of the adsorption process. At all studied temperatures, the inhibitor molecule protected mild steel with the same effect.

Keywords: AFM, Corrosion inhibitors, EIS, Mild steel, Polarization curves

I. Introduction

The use of hydrochloric acid in pickling of metals, acidization of oil wells in cleaning of scales is more economical, efficient and trouble free, compared to other mineral acids. Inhibitors are used to prevent metal dissolution as well as acid consumption. Most well known acid inhibitors are organic compounds containing nitrogen, sulfur and oxygen atoms. The surfactant inhibitor has many advantages such as high inhibition efficiency, low price, low toxicity and easy production [1-3]. Generally, it has been assumed that the first stage in the action mechanism of the inhibitor in the aggressive acid media is based on its adsorption on the metal surface. The process of adsorption of inhibitors are influenced by the nature and surface of the metal, the chemical structure of the organic inhibitor, the distribution of charge in the molecule, the type of aggressive electrolyte and the type of interaction between organic molecules and the metallic surface. It has been reported that quaternary ammonium compounds are important as inhibitor additives in hydrochloric acid. N, Ndimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide (DPDAB) is widely used in industrial and household commodities, including textile softeners, pharmaceuticals, disinfectants and human hair conditioners. To the best of our knowledge, no data are available in the literature regarding the behavior of N, N- dimethyl-N-(2phenoxyethyl) dodecan-1-aminiumbromide as inhibitor for metallic corrosion. In continuation to our goal for searching for safe and less toxic corrosion inhibitors, this work aims to investigate the inhibitive action of DPDAB towards the corrosion of mild steel; in 1M HCl using Tafel polarization and electrochemical impedance technique. The morphology of mild steel surface was investigated by using AFM and SEM techniques.

II. Experimental

DPDAB is dissolved in 1M HCl solution at various concentrations $(10^{-2}, 6X10^{-3}, 4X10^{-3}, 2x10^{-3} \text{ and } 10^{-3} \text{ M})$ and the solution in the absence of DPDAB was taken as blank for comparison.

Aggressive solution (1MHCl), was prepared by dilution of reagent grade 34% HCl with double distilld water. Composition of the mild steel was Fe, 98.7; C, 0.223; Mn, 0.505; Si, 0.164; S, 0.05. The cylindrical rod specimen was welded with copper wire for electrical connection and embedded in Teflon holder using epoxy resin with an exposed area 1 cm^2 . Before each experiment, the electrode was first mechanically polished with various grades of emery paper (0/0, 2/0, 3/0, & 4/0) and then cleaned with methanol and acetone followed by cleaning with double distilled water. MS samples (2cm x 1cm) were used for weight loss measurement studies at various temperatures (298K-328K) for 3h exposure time.

2.1 Electrochemical experiment

Electrochemical experiments were performed in a conventional three electrode cell, mild steel welded with copper wire and embedded in Teflon holder using epoxy resin with an exposed area 1 cm^2 as Working electrode, a platinum foil of 1 cm^2 was used as counter electrode & the reference electrode was a saturated

calomel electrode (SCE) with a Luggin capillary. All potentials are measured with respect to the SCE. Measurements were performed using CHI 660C model Electrochemical workstation.

Potentiodynamic polarization curves were obtained by scanning the potential range from -200mv + E_{corr} to +200 mv + E_{corr} at a scan rate of 10mv/s after 1h immersion time in

1M HCl and 1M HCl + inhibitor solutions.

Electrochemical impedance measurements were carried out at open – circuit potential over a frequency range of 0.01Hz – 100 KHz. The sinusoidal potential perturbation was 10mv in amplitude. Electrochemical data were obtained after 1h of immersion with the working electrode at the rest potential & all tests have been performed in non-deaerated solutions under stirred conditions. Potentiodynamic polarization curves were also obtained at different temperatures to calculate the activation energy of the inhibitor adsorption to mild steel surface.

2.2 Surface analysis by SEM and AFM

The morphology of the sample surface before and after being treated with optimal concentration of inhibitor was examined by SEM and AFM.

III. Results and discussion

3.1 Weight loss

The weight loss of mild steel strips in 100ml of 1M HCl solution in the temperature range $25^{\circ}C-55^{\circ}C$ without and with various concentrations (10^{-3} ,2X10⁻³, 4X10⁻³, $6X10^{-3}$ & $10^{-2}M$) of inhibitor was determined after 3h immersion period. The inhibition efficiency, %IE, was calculated as mentioned elsewhere [4]. The values of %IE and corrosion rate obtained from weight loss measurements for mild steel in 1M HCl and 1M HCl with different inhibitor concentration and at different temperature in the range $25^{\circ}C-55^{\circ}C$ are given in the Table 1. Fig.1 shows the variation of % inhibition efficiency with change in concentration. Corrosion inhibition efficiency increases with increasing in concentration as well as temperature as shown by the Fig. 1.

3.2 Potentiodynamic polarization:

Fig.2 shows potentiodynamic polarization curves of mild steel in 1M HCl in the absence and presence of inhibitor, it is very clear from the Fig.2 that both anodic and cathodic reactions of mild steel corrosion in the presence of 1M HCl were suppressed and the suppression effect increases with increase in the concentration of inhibitor. Corroison parameters such as corrosion potential (E_{corr}), corrosion current (i_{corr}) and %inhibition efficiency (%IE) are given in Table 2. The data show that the I_{corr} values decreased considerably in the presence of DPDAB and decreased with increasing inhibitor concentration. The decrease in I_{corr} with increasing inhibitor concentration was associated with the shift of corrosion potential, E_{corr} , to a less negative value. This indicates that the inhibitor reduces the corrosion rates predominantly by dissolution of metal. But the shift of E_{corr} is less than 85my reveals that the studied inhibitors act as mixed type inhibitor with predominant anodic effectiveness.

3.3. Electrochemical impedance spectroscopy:

The aim of EIS is to obtain more information concerning the dynamics and influence of working electrode in the presence of studied inhibitor. The impedance data in the form of Nyquist plots of mild steel at the open circuit potential in 1M HCl without and with studied inhibitor at 25^oC are shown in the Fig. 3a. Fig. 3b represents the equivalent circuit design used to fit the experimental data of EIS for 1M HCl in the absence and presence of studied inhibitor.

The circuit elements for the obtained data include a solution resistance (R_s) , a constant phase element (Q) and a charge transfer resistance (R_{ct}) . The value of R_{ct} was indicative of electron transfer across the interface. Taking in account the non-ideal frequency response of the display data, a constant phase element Q (CPE) was used instead of an ideal capacitance. CPE can represent all frequency dependent electrochemical phenomena, such as double layer capacitance, diffusion process and local frequency dispersion due to the microscopic roughness of the metal surface [5,6].

The impedance of a CPE is described by the equation 1

 $Z_{CPE=Y^{-1}(iw)^{-n}}$

Where y is proportional factor, *i* is $\sqrt{-1}$, *w* is $2\pi f$ and n has the meaning of phase shift, n is a factor satisfying the condition $0 \le n \le 1$. The fitted data follows almost the same pattern as the experimental results with R (QR) equivalent circuit using the Software Z_{simp} win. Inhibition efficiencies and other calculated impedance parameters are given in Table 3. When the inhibitor concentration increases, C_{dl} values tend to decrease. It can be attributed to the decrease in local dielectric constant or increase in thickness of surface film layer by the adsorption of the inhibitor molecules on the metal solution interface. The value of R_{ct} is a measure of the electron transfer across the surface and is inversely proportional to corrosion rate. R_{ct} increases with

increase in concentration. The inhibition efficiency increases with increase in concentration attain a maximum inhibition efficiency of 97.3% at 10^{-2} M. The semicircle shaped Nquist plots shown in figure 3a indicate the formation of a barrier on the surface and a charge transfer process mainly controlling the corrosion of MS. The values of n_{dl} related to (CPE)_{dl} are found in the 0.57-0.85 interval indicating the electrode surface are partially heterogeneous [7].

3.4 Effect of Temperature:

The activation energy of corrosion process can be obtained by investigating the influence of temperature on corrosion inhibition. Polarization curves of mild steel in 1M HCl at different temperature (298 – 328K) in the absence and presence of inhibitor are given in Figure. 2. Electrochemical parameters and inhibition efficiencies are presented in Table 2. The corrosion current density increases with the increase of temperature both in uninhibited and inhibited solutions, but inhibition efficiency of the compound remains nearly constant at higher temperatures in 1M HCl, thus studied inhibitor is temperature dependent.

The activation energy of the corrosion process can be calculated with the help of Arrhenius equation 2:

$$lni_{corr} = -\frac{E_a}{RT} + \ln A$$

Where A = pre –exponential factor and E_a is the activation energy of the corrosion process. Figure. 4 show the Arrhenius plots of lni_{corr} vs 1/T in the absence and presence of DPDAB. The calculated values of E_a are shown in Table 4. Inspection of the data shows that the activation energy is lower in the presence of inhibitor than in its absence. The decrease in E_a with inhibitor concentration is typical of chemisorptions. This was attributed by Hoar and Holliday to slow rate of inhibitor adsorption with a resultant closer approach to equilibrium at higher temperature. But Riggs and Hurd explained that the decrease in the activation energy of corrosion at higher levels of inhibition arises from the shift of the net corrosion reaction from the uncovered part of the metal surface to the covered part.

Following equation is the alternative formulation of Arrhenius equation 3:

$$i_{corr} = \frac{RT}{Nh} exp \frac{\Delta S^{\neq}}{R} exp \frac{\Delta H^{\neq}}{RT}$$
3

Figure 5 shows the plot of $\log[\frac{f(2\sigma r)}{T}]$ vs 1/T in the case of studied inhibitor in 1M HCl. A straight line is obtained with a slope of $(-\frac{\Delta H^{\neq}}{2.303R})$ and an intercept of $[\log R/Nh + \frac{\Delta S^{\neq}}{2.303R}]$ from which values of ΔS^{\neq} and ΔH^{\neq} are calculated and tabulated in the Table 4. The positive sign of the enthalpies reflect the endothermic nature of the steel dissolution process. Large and the negative values of ΔS^{\neq} imply that activated complex at the rate determining step represents association rather than dissociation step, meaning that increase in order takes place on going from reactant to the activated complex [8,9].

3.5 Adsorption isotherms and thermodynamic parameters:

Several adsorption isotherms were studied and Langmuir adsorption isotherm given as equation 4 was found to be closest to the description of the adsorption behavior of the studied inhibitor.

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{4}$$

Where C is inhibitor concentration and K is equilibrium constant of adsorption, which is related to standard free energy of adsorption ΔG_{ads} by equation 5:

$$\Delta G^{O} = -RT \ln(55.5K_{ads})$$
5

Figure.6 shows the plot of $\frac{C}{\theta}$ vs*C*. From the intercepts, the values of K_{ads} and ΔG_{ads} are calculated and are given in the Table 5.Table 5 shows that all the linear correlation co-efficients are almost equal to 1 and all the slopes are very close to 1, which indicates the adsorption of inhibitor onto steel surface accords with the Langmuir adsorption isotherm. The negative values of ΔG_{ads} along with high K_{ads} indicate a spontaneous adsorption process and a good chemical stability of inhibitor, magnitude of K_{ads} and ΔG_{ads} values confirm that inhibitor is adsorbed on the metal surface at all temperatures. Thermodynamic parameters are and given the Table 5. The positive value of ΔH_{ads} indicates the endothermic nature of steel dissolution and positive sign of ΔS_{ads} , means that the process is accompanied by an increase in entropy, which is the driving force for the adsorption of inhibitor onto the mild steel surface [10].

3.6 Atomic force microscope (AFM) surface examination:

Surface morphology of the polished mild steel and mild steel in 1M HCl in absence and presence of inhibitor were investigated through atomic force microscopy (AFM) technique. The results are shown in figure 7 (a-c). The average roughness of polished mild steel (Figure 7a) and mild steel in 1M HCl without inhibitor (Figure 7b) was calculated as 41nm and 785nm. It is clearly shown in figure 5b that mild steel sample is getting cracks due to the acid attack. However, in presence of optimum concentration of inhibitor (Figure 7c) the average roughness was reduced to180nm. Thus it can be concluded that corrosion was strongly inhibited when inhibitor was present in acid medium.

3.7 Scanning Electron Microscopy

The SEM micrographs of the corroded mild steel in 1M HCl in the absence and presence of inhibitor are shown in Fig.8 (a - c). In Figure 8a of polished mild steel and 8b, mild steel electrode surface in the presence of 1M HCl, defects and notches were observed, while a layer of closely packed film was obtained in Figure 8c, and the surface was free from pits and it was smooth. It can be concluded from Figure 8a-b that corrosion does not occur in presence of inhibitor and hence corrosion was inhibited strongly when the inhibitor was present in the solution, then scanning electron microscopy (SEM) observation, confirmed the existence of an absorbed protective film on the mild steel surface.

3.6 Mechanism of corrosion and inhibition in 1M HCl

In hydrochloric acid solution the following mechanism is proposed for the corrosion of mild steel.

The anodic dissolution mechanism of mild steel is:

The cathodic hydrogen evolution mechanism is:

$$Fe + H^+ \leftrightarrow (FeH^+)_{ads}$$

 $(\text{FeH}^+)_{ads} + e^- \rightarrow (\text{FeH})_{ads}$

$$(\text{FeH}^+)_{\text{ads}} + e^- + H^+ \rightarrow \text{Fe} + H_2 \text{ (rate determining step)}$$

Generally corrosion inhibition mechanism in acidic medium is due to the adsorption of the inhibitor onto the metal surface. The process of adsorption is influenced by the nature and charge of the metal, chemical structure of the inhibitor and the type of aggressive electrolyte. The charge of the metal surface can be determined from the potential of zero charge (PZC) on the correlative scale (\emptyset_c) by the equation

$$\mathcal{O}_{c} = E_{corr} - E_{q=0}$$

Where $E_{q=0}$ is the potential of the zero charge. However, a value obtained in HCl is -502.1 mv vs SCE.

Banerijee and Mallhotra reported the PZC of mild steel in HCl solution is -530mv VS SCE. Therefore the value of \emptyset_c is +27.9mv VS SCE, so the metal surface acquires slight positive charge. Studied quaternary ammonium compounds may chemisorb at steel / solution interface via chemical bond between positively charged nitrogen atoms and negatively charged mild steel surface as follows: Steel surface is positively charged in presence of HCl medium. While bromide ion is negatively charged surface of steel. By means of electrostatic attraction, Quaternary ammonium cation easily reaches mild steel surface, so bromide ion acts as an adsorption mediator for bonding metal surface and inhibitors. This gives rise to the formation of an adsorption composite film in which Br⁻¹ ion are sandwiched between metal and positively charged part of inhibitor. This film acts as a barrier facing corrosion process.

IV. Conclusion:

- The results proved that N,N- dimethyl-N-(2-phenoxyethyl) dodecan-1- aminiumbromide showed good corrosion inhibition for mild steel in 1M HCl solution.
- Studied inhibitor is a mixed type inhibitor because both anodic and cathodic curves decreased but corrosion potential remained constant.
- > The percentage inhibition increases with inhibitor concentration and increase in temperature.

- \triangleright The adsorption of the inhibitor molecules on mild steel surface obeys Langmuir adsorption isotherm.
- \triangleright Thermodynamic parameters indicated that the adsorption is spontaneous and endothermic process.
- Work is in progress of finding the inhibition efficiency in H₂SO₄ Medium.

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Table 1

Inhibition efficiency of various concentrations of the inhibitor for the corrosion of mild steel in 1M HCl by weight loss method at different temperatures (298K-328K).

		Temperature							
		298		308		318		328	
Inhib itor	Inhibitor concentrat ion (M)	%IE	Corrosion rate(mm/y)	%IE	Corrosion rate(mm/y)	%IE	Corrosion rate(mm/y)	%IE	Corrosion rate(mm/y)
D P D A B	Blank		36.44		66.4		153.6		278.0
	1 X 10 ⁻³	76.4	8.55	84.2	10.41	86.2	21.2	90.4	26.59
	2 X 10 ⁻³	79.8	7.25	89.6	6.88	90.1	15.25	92.2	21.76
	4 X 10 ⁻³	83.4	5.95	92.4	5.01	93.2	10.41	93.2	18.97
	6×10^{-3}	92.8	2.60	96.3	2.42	97.2	4.27	96.8	8.93
	1×10^{-2}	96.5	1.11	98.0	1.30	97.9	3.16	98.0	5.39

dimethyl-1	N-(2-phenoxyeth	yl) dodecan-1-amin	iumbromide at diff	ferent temperatures	
Concentration(M)	E_{corr} (mV) i_{corr} (μ A/cm ²)		b _a	b _c	IE(%)
			(mV/decade)	(mV/decade)	
298K					
HCl	-502.1	2640	151.7	130.1	
1 x 10 ⁻³	-465.6	455.0	143.7	263.8	82.8
2 x 10 ⁻³	-476.1	348.1	140.0	242.5	86.8
4 x 10 ⁻³	-472.9	277.6	136.1	219.5	89.5
6 x 10 ⁻³	-471.3	242.2	132.9	191.7	90.8
1 x 10 ⁻²	-502.8	209.9	172.1	132.1	92.5
308K					
HCl	-501.8	6354	178.8	158.7	
1 x 10 ⁻³	-486.1	560.0	146.8	230.0	91.2
2 x 10 ⁻³	-487.4	340.5	140.8	181.0	94.6
4 x 10 ⁻³	-480.3	281.2	137.2	156.8	95.6
6 x 10 ⁻³	-475.8	274.0	132.1	157.1	95.7
1 x 10 ⁻²	-481.6	244.7	131.2	149.0	96.1
318K					
HCl	-500.5	14130	200.8	189.5	
1 x 10 ⁻³	-495.7	830.0	154.0	177.1	94.1
2 x 10 ⁻³	-506.8	696.7	153.7	158.4	95.1
4 x 10 ⁻³	-506.9	607.1	154.2	154.0	95.7
6 x 10 ⁻³	-487.1	539.7	147.5	139.5	96.2
1 x 10 ⁻²	-487.1	573.2	146.2	137.9	95.9
328K					
HCl	-494.8	26620	201.7	206.3	
1 x 10 ⁻³	-507.7	1536	171.0	160.8	94.2
2 x 10 ⁻³	-510.0	1483	170.7	156.7	94.4
4 x 10 ⁻³	-500.8	1166	161.8	143.8	95.6
6 x 10 ⁻³	-496.5	1128	159.1	142.7	95.8
1 x 10 ⁻²	-492.0	1046	156.5	135.4	96.1

 Table 2.

 Electrochemical polarization parameters for MS in 1MHCl containing different concentration of N, N-dimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide at different temperatures.

Table. 3

Impedance parameters of mild steel in 1MHCl absence and presence of different concentration of N, Ndimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide at 25^oc

Concentration(M)	$\begin{array}{c} Y_{dl} \\ (\Omega^{-1} cm^{-2} S^{ndl} \\ 10^{-4}) \end{array}$	n _{dl}	$\mathbf{R}_{\mathrm{ct}}(\mathbf{\Omega})$	C _{dl} (µF/cm ²)	IE (%)
HCl	18.20	0.8561	9.979	927.8	
1 x10 ⁻³	26.21	0.5925	70.04	816.8	85.7
2 x 10 ⁻³	20.38	0.5849	90.2	612.6	88.9
4 x 10 ⁻³	19.19	0.5716	102.0	565.2	90.2
6 x 10 ⁻³	14.33	0.5778	142.0	449.8	92.9
1 x 10 ⁻²	13.48	0.5602	160.0	404.3	97.3

Table	4
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Corrosion kinetic parameters for mild steel in 1M HCl in the absence and presence of different concentrations of DPDAB

01 DFDAB						
Concentration(M)	E _a (kJ/mol)	$\Delta H^{\neq}(kJ/mol)$	$\Delta S^{\neq} (J/mol/K^{-1})$			
Blank	64.28	53.35	-115.89			
10 ⁻³	33.61	30.96	-207.59			
2 x 10 ⁻³	42.0	39.45	-182.89			
4 x 10 ⁻³	42.2	39.54	-184.26			
6 x 10 ⁻³	44.0	41.36	-179.2			
10 ⁻²	47.1	44.49	-169.9			

Table 5: Data obtained from the Langmuir adsorption isotherm for mild steel in 1M HCl at the temperature range of 298K -328K.

Temperature(K)	R	Slope	$\Delta G_{ads}(kJmol^{-1})$	$\Delta H_{ads}(kJmol^{-1})$	$\Delta S_{ads}(Jmol^{-1}K^{-1})$
298	0.9994	1.0656	-31.33	23.6	184.3
308	0.9999	1.0359	-35.40	23.6	191.5
318	0.9984	0.9980	-31.98	23.6	174.8
328	0.9999	1.0387	-39.00	23.6	190.8

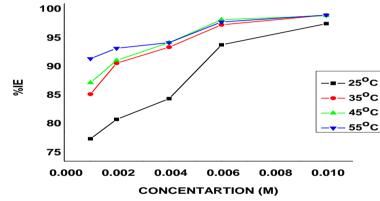
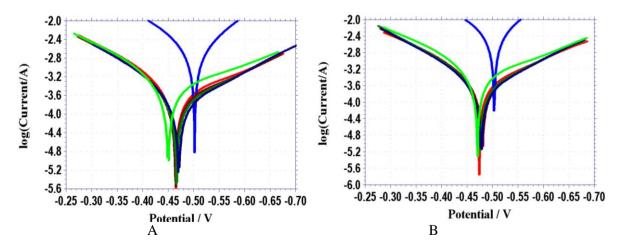


Fig. 1 Variation of %IE with change in concentration of the studied inhibitor in the different temperature range.



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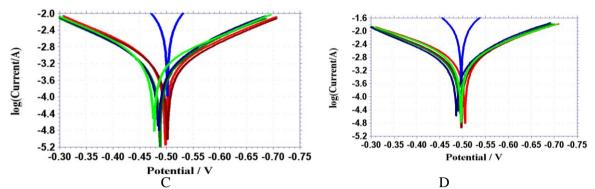


Figure 2: Typical Tafel plots of MS in 1MHCl in presence and absence of different concentration of N, N-dimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide at A). 25^oC B) 35^oC C) 45^oC D) 55^oC

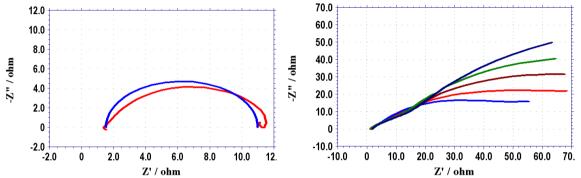


Figure 3a: Nyquist plots of MS in 1MHCl in the presence and absence of different concentration of N, Ndimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide at 25^oC

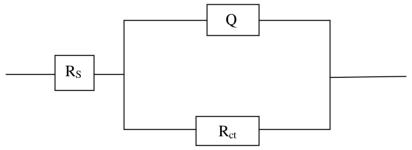


Figure 3b: Equivalent circuit used to Fit EIS Data

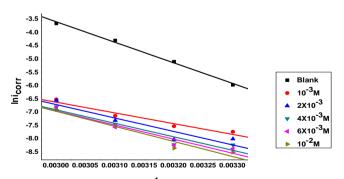


Figure 4: Arrhenius plots for mild steel in 1M¹HC1^{*} without and with various concentration of N, N- dimethyl-N-(2-phenoxyethyl) dodecan-1-aminiumbromide.

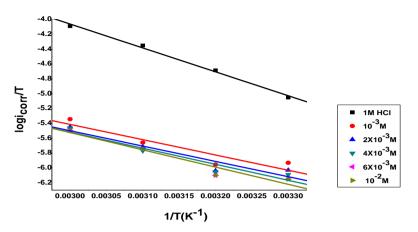


Figure 5: Transition state plots for MS in 1M HCl without and with various concentrations of N,N-dimethyl-N-(2-phenoxyethyl)dodecan-1-- aminiumbromide

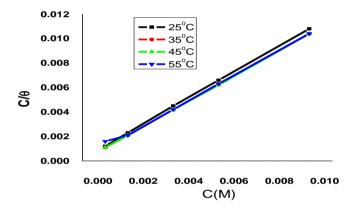
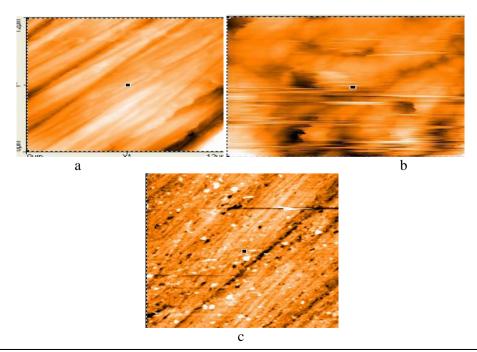


Figure6: Langmuir adsorption isotherms for MS in 1M HCl without and with various concentrationsof N,Ndimethyl-N-(2-phenoxyethyl)dodecan-1-aminiumbromide



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Figure 7 : Atomic force micrographs of mild steel surface (a) Polished mild steel (b) Mild steel in 1M HCl (c) Inhibited mild steel (1M HCl + 10^{-2} M)

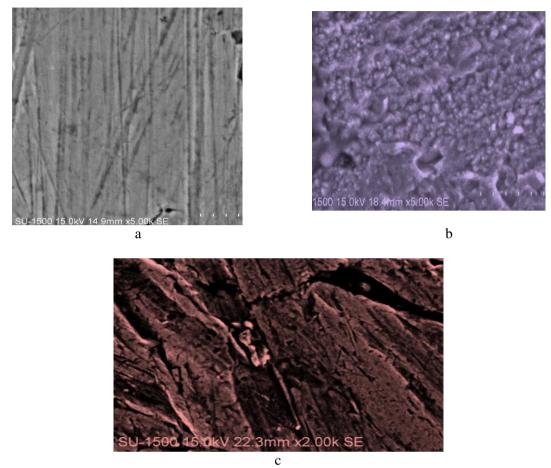


Figure 8: Surface Characterization by SEM for a) Polished mild steel b) Mild steel in 1M HCl in the absence of inhibitor c) In the presence of Inhibitor