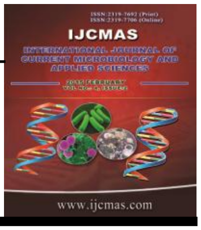

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Original Research Article

Triazoles used as a Corrosion inhibitor for mild steel in Hydrochloric Acid

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ABSTRACT

Keywords

Corrosion,
Mild steel,
Hydrochloric acid, Triazoles,
Langmuir adsorption,
Temkin's adsorption isotherm

Triazoles (Benzotriazole and Benzyl benzotriazole) were used as corrosion inhibitors for mild steel in hydrochloric acid solution. The inhibition efficiency depended on the concentration and type of the triazoles. The inhibition efficiency ranged between 80 and 98 % at the highest concentration (25 mM), and between 65 and 85% of the lowest concentration (5 mm) of inhibitor in 1 M HCl solution. Inhibition efficiency decreased with rise in temperature, this corresponded to surface coverage of the metal by the inhibitor. The calculated degrees of surface coverage, θ, were found to increase with the inhibitor concentration. The results also showed that, the inhibitors were adsorbed on the mild steel surface according to Langmuir and Temkin adsorption isotherm. Polarization study revealed that all the two triazole functioned as slightly anodic, but significantly cathodic inhibitors.

Introduction

Hydrochloric acid is generally used for the removal of undesirable scale and rust in several industrial processes. Thus, inhibitors are one of the most convenient methods for protection against corrosion, particularly in acid solutions to prevent unexpected metal dissolution and acid consumption (Sorkhabi et al., 2008). The use of acid solution during pickling and industrial cleaning leads to corrosive attack on mild steel. Therefore, corrosion of mild steel and its inhibition in acidic solutions have attracted the attention of a number of investigators (Abdennabi et al., 1996; Abboud et al., 2006; Al-Sarawya et al., 2008; Sayed et al., 2008; Desai, 2015; Desai, 2015). The protection of metal against corrosion can be achieved by adding a small concentration of organic compounds to the environment (Musa et al., 2009). A variety of organic compounds containing heteroatoms like nitrogen, sulfur and oxygen have been investigated as corrosion inhibitors (Quraishi & Sharma, 2005; Vrsalovic et al., 2005; Bouklah et al., 2006). A survey of literature reveals that the applicability of organic compounds as corrosion inhibitors for mild steel in acidic media has been recognized for a long time. Compounds including triazolederivatives (Bentiss et al., 1999), bipyrazolic derivatives (Touhami et al., 2000), surfactants (Algaber et al., 2004), aromatic hydrazides (Quraishi et al., 2001), organic dyes (Oguzie et al., 2004; Deasi & Vashi, 2010; Desai & Kapopara, 2014), drugs (Deasi & Vashi, 2011), poly (4-vinylpyridine) (Larbi et al., 2004) and thiosemi-carbazide (Ita & Ofiong 2001) type organic compounds were reported as inhibitors. These compounds can be adsorbed on the metal surface and block the active sites to reduce the corrosion rate.

In this work, the inhibition ability of Benzotriazole and Benzyl benzotriazole for mild steel corrosion in HCl solution was evaluated. The inhibitory mechanism of these organic compounds was discussed based on the analysis of the Langmuir adsorption isotherm, weight loss, thermodynamic and polarization studies.

Experimental

Materials preparation

Mild steel contains Fe = 99.746; Mn = 0.100; C = 0.058; Al = 0.033; Si = 0.010; Cr = 0.008; Cu = 0.004; Ni = 0.0029; Mo = 0.002% were used in this study. Each sheet, which was 0.12 cm in thickness, was mechanically press-cut into coupons of dimension 3.5 x 3 cm with small hole of about 5 mm diameter near the upper edge.

These coupons were used in the "as cut" condition, inhibition efficiency without further polishing, but were de-greased in absolute ethanol, dried in acetone, weighed and stored in a moisture-free desiccator prior to use. All chemicals and reagents used were of analytical grade and used as source without further purification. The aggressive media were, respectively, 1, 2 and 3 M HCl solution. Triazoles were used as inhibitors in the concentration range 5, 10, 15, 20 and 25 mM.

Weight loss method

The test specimens were immersed in 1 to 3 M HCl solution with without inhibitors. Only one specimen was suspended by a glass hook, in each beaker containing 230 ml of the test solution and was open to air at room temperature for 24 h. After the immersion, the surface of the specimen was cleaned with double distilled water, followed by rinsing with acetone and the sample was weighed again in order to calculate inhibition efficiency (η %). Triplicate experiments were performed in each case and the calculated mean values of the weight loss data are presented in figure 1.

To study the effect of temperature on corrosion rate, the specimens were immersed in 230 ml of 1.0 M HCl solution with various triazoles as inhibitor having concentration 5, 10, 15, 20 and 25 mM at solution temperatures of 313, 323 and 333 K for a period of 3 h. To study the effect of temperature, thermostat assembly with an accuracy of $\pm 0.5^0$C was used (ASTM, 1950; Wesley, 1956). From the weight loss data, calculate the Inhibition efficiency (η %), energy of activation (E_a), heat of adsorption (Q_{ads}), free energy of adsorption (ΔG^o_{ads}), enthalpy of adsorption (ΔH^o_{ads}) and entropy of adsorption (ΔS^o_{ads}) were calculated.

Polarization studies

For polarization study, metal specimens having an area of 0.2265 dm^2 were exposed to corrosive solutions. Mild steel was used as a working electrode, SCE was used as reference electrode and the auxiliary graphite electrode was placed in 230 ml of corrosive media through which external current was supplied automatically from the computerized polarization instrument. The change in potential was measured by potentiostate/galvanostate (Gamry-Make, USA) on the potentiostate mode with 5 MV/s scan rate. Polarization study was done with and without inhibitors in 1.0 M HCl.

Result and Discussion

The results are presented in Tables 1 to 2 and Figहाs 1 to 6. To assess the effect of corrosion of mild steel in HCl, derivatives of benzotriazole were used as corrosion inhibitors.

The corrosion rate of mild steel in HCl was increased with the acid concentration. The inhibition efficiency (η %) and degree of surface coverage (θ) at each concentration of benzotriazole derivatives were calculated by comparing the corrosion loss in the absence (W_u) and presence of inhibitors (W_i) using the relationships:

$$\eta\% = \left(\frac{Wu - Wi}{Wi}\right) X100 \qquad (1)$$

$$\theta = \left(\frac{Wu - Wi}{Wi}\right) \qquad (2)$$

As a constant inhibitor concentration, the inhibition efficiency decreased with the increase in acid concentration. At 25 mM inhibitor concentration, the inhibition efficiency of benzotriazole was 93.46, 70.16 and 50.65 with respect to 1.0, 2.0 and 3.0 M acid concentrations respectively. At a constant acid concentration, the inhibition efficiency of the benzotriazole derivatives increased with the inhibitor concentration, e.g. In 1.0 M HCl, the inhibition efficiency was found to be 71.90, 79.09, 86.27, 92.81 and 93.46 with respect to 5, 10, 15, 20 and 25 mM benzotriazole respectively, which was shown in Figure 1.

The temperature had significant influence on the metal corrosion rates. The effect of change in temperature on the corrosion rates of mild steel in 1.0 M HCL was examined. It was found that, the corrosion of mild steel increased with the increase in temperatures (Table 2). Corrosion rate was measured in 1.0 MHCl containing 5, 10, 15, 20 and 25mM inhibitor concentration at a solution temperature of 313, 323 and 333 K for an immersion period of 3 h. In 1.0 M HCl solution with 25 mM Benzyl benzotriazole concentration, the inhibition efficiency was found to be 92.31, 91.38 and 86.79 at 313, 323 and 333 K respectively (Figure 2a & 2b).

In the present study general type of corrosion occurs predominately and less pitting. Plotting of log θ/1-θ versus log C (%), straight lines were obtained, indicating that the adsorption of the added inhibitors followed the Langmuir adsorption isotherm (Fig.3-a,3-b). Therefore, adsorption of these compounds is assumed to occur uniformly over the metal surface.

Plot a graph θ versus log *C* gives straight line (Figure-4-a,4-b) showing that the adsorption of the compound on the mild steel surface from 1 M HCl obeys also Temkin’s adsorption isotherm. It also finds that the degree of adsorption of the inhibitors increases with their concentration.

The values of the free energy of adsorption (ΔG^0_{ads}) were calculated with the slope of the following equation (Abdel & Saied, 1981).

$$LogC = Log\left(\frac{\theta}{1-\theta}\right) - LogB \qquad (3)$$

Where $LogB = -1.74 - \left(\frac{\Delta G^0_{ads}}{2.303RT}\right)$ and C is the inhibitor concentration. The mean $\Delta G^0{}_a$

values are negative almost in all cases and lie in the range of -8.10 to -11.50 kJ. mol^{-1} shown in Table-1. The most efficient inhibitor shows more negative ΔG^0_a value. This suggests that they be strongly adsorbed on the metal surface. This statement was supported by the work of Talati and Darji (1988). The values of heat of adsorption (Q_{ads}) were calculated by the following equation.

$$Q_{ads} = 2.303R\left[Log\left(\frac{\theta_2}{1-\theta_2}\right) - Log\left(\frac{\theta_1}{1-\theta_1}\right)\right] X \left[\left(\frac{T_1T_2}{T_2-T_1}\right)\right] \qquad (4)$$

From Table -1, it is evident that in all cases, the (Q_{ads}) values are negative and ranging from -5.09 to -52.91 kJ. mol^{-1}. The negative values show that the adsorption, and hence the inhibition efficiency, decreases with a rise in temperature.

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Anodic and Cathodic polarization curves for mild steel in 1M HCl at 25 ppm inhibitor concentration of the presence and absence of inhibitors are shown in Figure 6 to 8. The value of the corrosion potential with inhibitors were found become more positive than the without inhibitors. Polarization study reveals that the inhibitors function as little anodic, but significant cathodic inhibitors, inhibitor functions as a mixed inhibitor. It is evident from the figure that cathodic tafel slopes (βc) remain almost unchanged with increasing inhibitor concentration. This indicates that hydrogen evolution is activation controlled and the addition of inhibitor did not change the mechanism of cathodic hydrogen evolution reaction (Ateya et al., 1976 ; Li et al., 2008). The values for the Tafel parameters obtained from this plot with and without inhibitors are given in Table-2.

The values of corrosion current densities in the presence and absence of inhibitor were obtained from the graph while percentage efficiency (η %) was calculated using the Equation (8). The inhibition efficiency from Tafel plots agrees well (within ±8%) with the values obtained from weight loss data.

$$\eta(\%) = \left[\frac{i_{corr}(u) - i_{corr}(i)}{i_{corr}(u)}\right] \times 100 \qquad (8)$$

The linear polarization study was carried out from cathodic potential versus OCP to an anodic potential of versus OCP with a sweep rate 0.01 V S^{-1} to determine the polarization resistance ($R_p^{'}$). From the measured polarization resistance value, the inhibition efficiency has been calculated using the relationship:

$$\eta\% = \frac{Rp' - Rp^0}{Rp'} X\ 100 \qquad (9)$$

Where R_p^0 and $R_p^{'}$ are the polarization resistance in the absence and in the presence of inhibitor, respectively.

Mild steel dissolves in acid solutions due to the hydrogen evolution type of attack. The reaction-taking place at the micro electrodes of the corrosion cell being represented as under,

$$Fe^{+2} \rightarrow Fe^{+3} + e^- \qquad (10)$$

(anode)

$$H^+ + e^- \rightarrow H \qquad (11)$$

(cathode)

Followed by the reaction

$$H + H \rightarrow H_2 \qquad (12)$$

The following secondary reaction can also take place in acid solutions (Godard, 1976).

$$2M + 2H^+ \rightarrow H_2 + 2M^+ \qquad (13)$$

(anode)

$$O_2 + 4H^+ + 4e^- \rightarrow 2H_2O \qquad (14)$$

(cathode)

Therefore, only if the hydrogen evolution type of attack is predominate and no other factors influence the corrosion process, corroded by the strong acid should be maximized. The inhibitory mechanism is a separation process involving (i) the inhibitor is adsorbed on the surface of the metal forming a compact protective thin layer and (ii) the inhibitor forms a precipitate on the surface of the metal, acting on the aggressive media to form protective precipitates or remove the aggressive agents (El-Sayed et al., 2010). Adsorption, on the other important notes can be described by two main types of interaction, which are physisorption and chemisorption, where (Noor, 2008): 1) *Physisorption*: involves electrostatic forces between ionic charges or dipoles on the adsorbed species and the electric charge at the metal/solution interface. The heat of adsorption is low, thus, this adsorption is only stable at relatively low temperature. 2) *Chemisorption*: involves the charge transfer or sharing from the inhibitor molecules to the metal surface to form a coordinate type bond. This type of adsorption is known to have much stronger adsorption energy compared to the other mode of adsorption. Thus, such bond is more stable at higher temperature.

Triazole ring can be considered to be a typical example of π excessive N heterocyclic compound. Owing to the presence of heteroatoms, having an unshared pair of electrons in the triazole ring, its 6π electrons are disposesd in its ring and therefore belongs to the aromatic group of compounds. These compounds inhibit corrosion by following mechanism.

The interaction between lone pair of electrons of the nitrogen atoms and the positively charged metal surface; and the interaction of electrons of the triazole rings with the positively charged metal surface and the interaction of thiazole cations formed in acidic solutions with negatively charged metalsurface. The de-localized π - electrons of inhibitors facilitate its strong adsorption on the mild steel surface leading to the outstanding corrosion inhibition (Desai, 2015).

In conclusion, as a constant inhibitor concentration, the inhibition efficiency of all inhibitors decreases as the concentration of acid increases. At all concentration of acid, as the inhibitor concentration increases inhibition efficiency increases and corrosion rate decreases. As the temperature increases corrosion rate increases in plain acid. Addition of inhibitors in corrosive media indicates that as the temperature increases corrosion rate increases while inhibition efficiency decreases. In all cases, the value of heat of adsorption (Q_{ads}) and the value of free energy of adsorption ($\Delta G^0{}_a$) is negative.The Value of change of enthalpy ($\Delta H^0{}_a$) and entropy of adsorption ($\Delta S^0{}_a$) is positive. A mean value of 'Ea' in inhibiting acid is higher than the value of 'Ea' in acid only. In almost all the cases, the inhibition efficiency from Tafel plots agrees well (within ± 6 %) with the values obtained from weight loss data.

Table.1 Energy of activation (E_a), heat of adsorption (Q_{ads}) and free energy of adsorption ($\Delta G^0{}_{ads}$) for mild steel in 1.0 M HCl containing 25 mM inhibitors

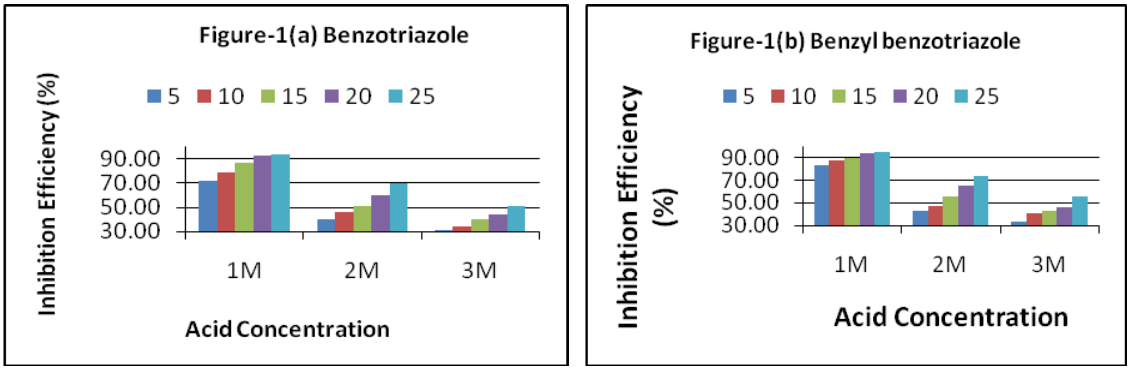
System	Mean Ea from Eq.(2) (kJ. mol^{-1})	Ea from Arrhenius plot (kJ. mol^{-1})	Q_{ads} (kJ. mol^{-1}) 313-323 (K)	Q_{ads} (kJ. mol^{-1}) 323-333 (K)	Mean value (kJ. mol^{-1}) $\Delta G^o{}_{ads}$	Mean value (kJ. mol^{-1}) $\Delta H^o{}_{ads}$	Mean value (kJ. mol^{-1}) $\Delta S^o{}_{ads}$
Blank	72.79	70.39	-	-	-	-	-
Benzotriazole	111.67	107.44	-16.71	-52.90	-28.29	74.25	0.2967
Benzyl benzotriazole	96.65	92.87	-34.89	-42.76	-29.14	79.27	0.3997

Table.2 Polarization data and inhibition efficiency of inhibitors for mild Steel in 1 M HCl

Inhibitor concentration: 25 ppm — Effective specimen area: 0.2265 dm²

System	E_{corr} (mV)	I_{corr} (A/cm²)	*R*p (ohm)	Tafel slope (V/decade)			Inhibition efficiency (%)		
							Pol. Method		Wt. loss method
				-βc	+βa	β	From I_{corr}	From *R*p	
Blank	-557	4.866×10^{-3}	12	5.751	1.436	2.64	-	-	-
Benzotriazole	-543	1.767×10^{-5}	2405	5.191	5.040	5.12	99.64	99.50	94.77
Benzyl benzotriazole	-532	2.941×10^{-4}	76	6.220	12.18	9.20	93.96	84.21	92.16

Figure.1 Effect of inhibition efficiency of Triazole derivatives for mild steel at different acid and inhibitor concentrations for 24 h at 301 K



Figue.2 Effect of inhibition concentration of Triazoles for mild steel in 1M HCl at different temperatures for3h

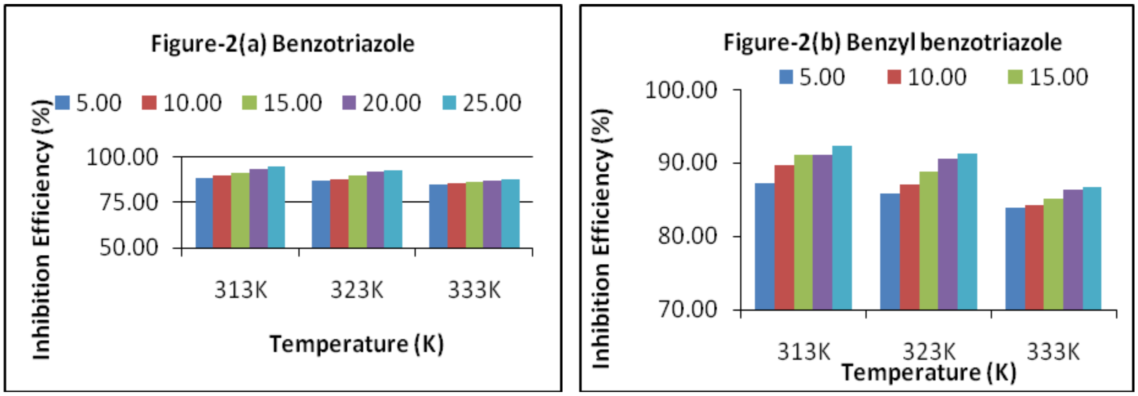


Figure.3 Plot of log (θ /1- θ) versus log C for Triazoles at different concentrations of HCl for 24 h at 301 K

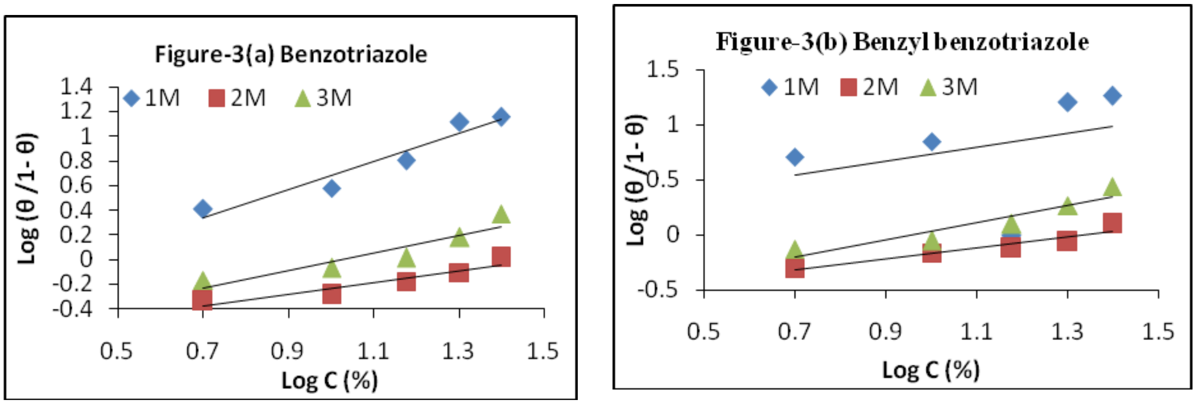


Figure.4 Plot of θ versus log C for Triazoles at different concentrations of HCl for 3h

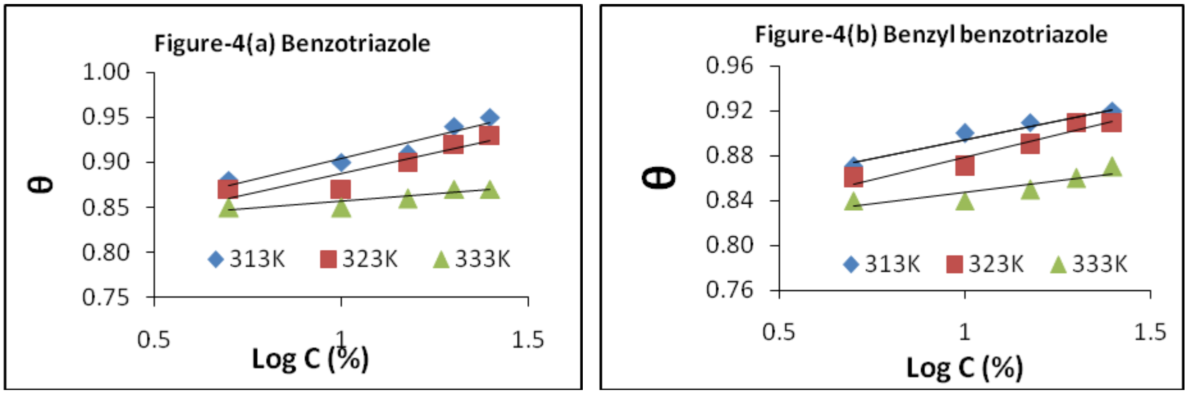


Figure.5 Arrhenius plots for corrosion of mild steel in 1 M HCl in absence and presence of Triazoles

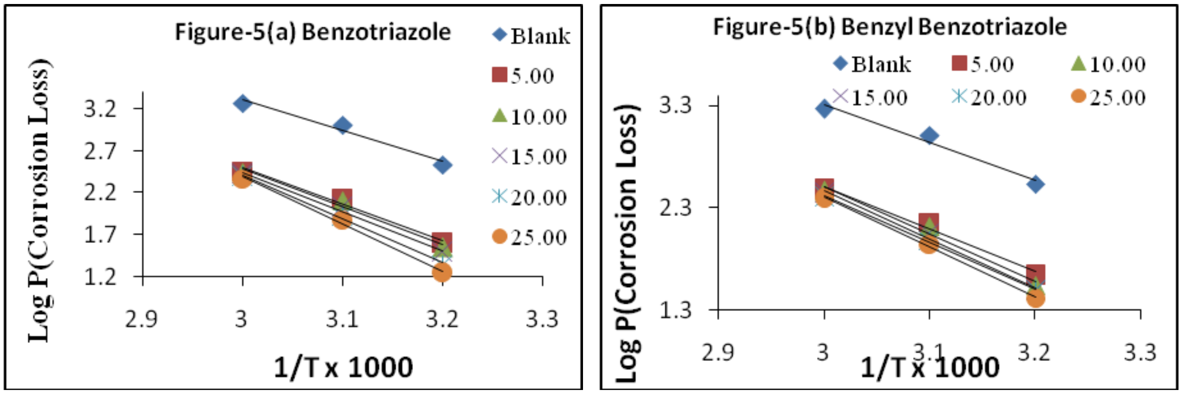


Figure.6 Polarisation curves for corrosion of mild steel in 1 M Hcl without inhibitors

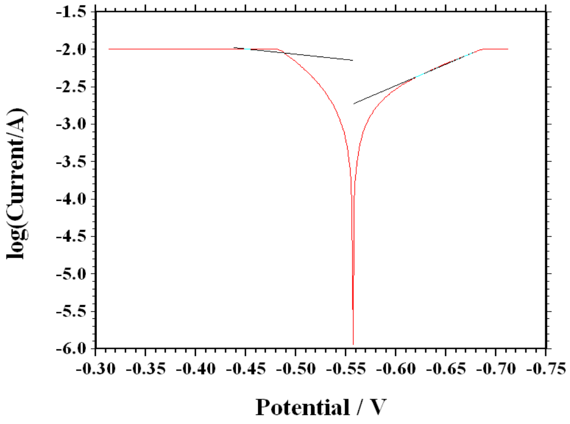


Figure.7 Polarisation curves for corrosion of mild steel in 1 M Hcl containing 25 mm benzotriazole inhibitors

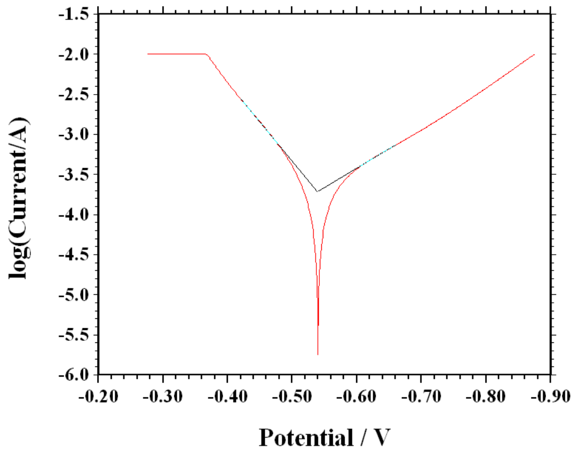
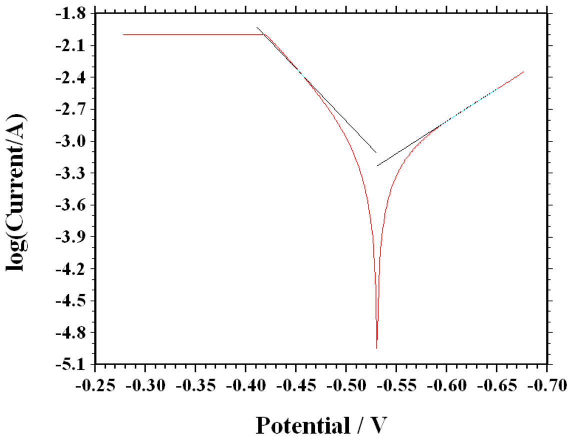


Figure.8 Polarisation curves for corrosion of mild steel in 1 M Hcl containing 25 mm benzoyl benzotriazole inhibitors



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