

## Corrosion Inhibition of Carbon Steel in Saline Solution Using Anti-Bacterial Drug

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**Abstract:** Corrosion behavior of carbon steel in a solution in acidic medium ( $\text{pH} = 1$ ) was investigated in the absence and presence of different concentrations of anti-bacterial drug (tetracycline) as environmentally friendly corrosion inhibitor over temperature range (293-308) K. The investigation involved electrochemical polarization method using potentiostatic technique and optical microscopy, the inhibition efficiency increased with an increase in inhibitor concentration but decreased with increase in temperature. Results showed that the inhibition occurs through adsorption of the inhibitor molecules on the metal surface and it was found to obey Langmuir adsorption isotherm. Some thermodynamic parameters ( $\Delta G_{\text{ads}}$ ) and activation Energy ( $E_a$ ) were calculated to elaborate the mechanism of corrosion inhibition. The polarization measurements indicated that tetracycline is of mixed type. The surface characteristic of the inhibited and uninhibited metal samples were investigated by optical microscopy.

**Key words:** Corrosion inhibition, carbon steel, tetracycline, adsorption mechanism thermodynamic parameters, metal, microscopy

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### INTRODUCTION

Corrosion which is unavoidable issue faced by all industries can be considered one of the most important problems facing industries in the world as a whole. In addition to its direct economical cost, corrosion is definitely contributes to the exhaustion of our natural sources.

The importance of corrosion studies come from the urgent need to preserve the world natural resources (Stansbury and Buchanan, 2000). Prevention would be more practical and attainable than complete removal. The best choice of combating metals against deterioration against environmental effect is using inhibitors (Karthikeyan and Jeeva, 2015; Kumar and Bashir, 2015; Charitha and Rao, 2015; Rani and Basu, 2011; Matad *et al.*, 2014; Akpan and Offiong, 2014; Megalai *et al.*, 2013; Vaszilcsin *et al.*, 2012; Bhat and Alva, 2011). The toxicity of the using inhibitors to the environment encourage the search for eco-friendly corrosion inhibitors. Lately because eco-environmental nature of drugs they have been used as corrosion inhibitors (Samide *et al.*, 2011; Yousif *et al.*, 2013; Hebbbar *et al.*, 2014; Kumar *et al.*, 2013; Ahamad and Quraishi, 2010; Karthikeyan *et al.*, 2015a, b; Eddy and Odoemelam, 2008). Drugs are non-toxic, cheap and

negligible negative effects on environment and most of them can be easily synthesized from natural products, so, it suggested replacing the traditional toxic corrosion inhibitors (Mahdi, 2014). Several researches have proven that antibiotics can be used as corrosion inhibitors for various metals in acidic and alkaline media (Kumar and Karthikeyan, 2012; Naqvi *et al.*, 2011; Singh *et al.*, 2011; Gece, 2011; Fouda *et al.*, 2014; Kushwah and Pathak, 2014).

Generally, the adsorption of the inhibitor molecules on the metal surface is assumed to be the first step in the inhibition process, this process affected by kind and surface charge of metal, chemical structure of inhibitors, the distribution of charge in the molecule, the type of aggressive electrolyte and the type of interaction between organic molecules and the metallic surface. The main types of interaction between organic inhibitor molecules and metal surface are physical adsorption and chemisorption. In previous study, Abdallah (2004) dicloxacillin was used as corrosion inhibitor for corrosion of mild steel in acid medium it acted as cathodic inhibitor (Karthikeyan *et al.*, 2015a, b).

The aim of present research is to study the inhibiting effect of tetracycline on carbon steel corrosion in 0.1 M hydrochloric acid solution (Fig. 1).

Table 1: The chemical composition of carbon steel

C (%)	Si (%)	Mn (%)	P (%)	S (%)	Cr (%)	Mo (%)	Ni (%)	AL (%)	Cu (%)	Fe (%)
0.190	0.529	11.88	0.0472	0.0146	8.40	0.0181	0.844	0.0032	0.447	Bal.

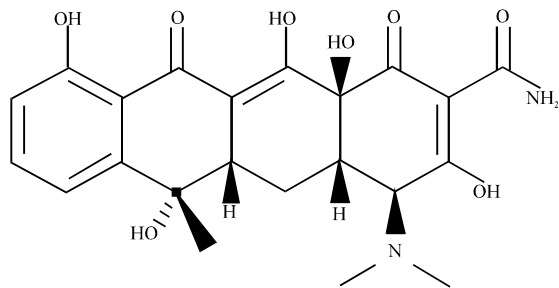


Fig. 1: Chemical structure of tetracycline

## MATERIALS AND METHODS

**Materials and chemicals:** The experiments were performed with a cylindrical rod of carbon steel (Table 1) show the composition of alloy) which was mechanically cut into circular form 2 cm diameter and 2 mm thickness with exposed area of 1 cm<sup>2</sup> was employed. Carbon steel specimens were abraded with different fineemery paper (100, 200, 400, 800, 1200 and 2000) to a mirror and degreased with acetone. The chemicals used in this research are include:

- Sodium chloride was used for preparation of the aggressive solution of 0.6 mol dm<sup>-3</sup>
- Hydrochloric acid
- Tetracycline (>99% purity)

**Solution:** The aggressive solution used was made of AR Sodium chloride to prepare 0.6 mol.dm<sup>-3</sup> concentration in pH = 1. Doubled distilled water was used for the preparation. For each experiment a freshly solution was made. Three concentrations of tetracycline were used (0.5×10<sup>-4</sup>, 1×10<sup>-4</sup> and 2×10<sup>-4</sup>) mol.dm<sup>-3</sup>.

**Potentiostatic polarization measurements:** The potentiostatic polarization studys were performed using a three electrodes cell with carbon steel specimen of 1 cm<sup>2</sup> exposed area, a platinum electrode and silver-silver chloride in saturated KCl solution were used as working, auxiliary (counter) and reference electrodes, respectively.

The measurements were carried out using MLab potentioestat/Galvanostat 200 Germany obtained from Bank Electronic Intelligent Controls Gnb H. It was connected to personal computer desktop. It provides electrochemical calculations like Tafel line evaluation, re-scaling the potential and integrating. The experiments

were preformed in 0.6 mol.dm<sup>-3</sup> NaCl solution of pH = 1 in absence and presence of three different concentrations of the inhibitor (tetracycline) over the temperature range (298-313) K. The experiments were carried out at a scan rate of 2 mV/sec.

From the polarization curve corrosion current density ( $i_{corr}$ ) and corrosion potential ( $E_{corr}$ ) were determined in addition other informations were obtained such as Tafel slopes ( $b_c$  and  $b_a$ ) weight loss and penetration values. In order to test the reproducibility of the results, the experiments were done in triplicate.

## RESULTS AND DISCUSSION

**Tafel polarization measurements:** Tafel polarization technique has been used to study the effect of 0.6 mol.dm<sup>-3</sup> NaCl solution in acidic medium onthe corrosion of carbonsteel. Figure 2 represent the potentiostatic polarization curves of carbon steel at four temperature in range of (298-313) K. Corrosion parameters such as corrosion potential ( $E_{corr}$ ), corrosion current density ( $i_{corr}$ ), anodic slope ( $b_a$ ) and cathodic slope ( $b_c$ ) are obtained from the Tafel polarization curves. Results are tabulated in Table 2. The results indicate the increase in the corrosion current density ( $i_{corr}$ ) with an increase in temperature.

The negative shift in the corrosion potential ( $E_{corr}$ ) with the increase in temperature indicates that the anodic process is much more affected than the cathodic process, this observation is in accordance with other published results (Ziaifer *et al.*, 2011; Raja *et al.*, 2014) which proposed the dependence of ( $i_{corr}$ ) and ( $E_{corr}$ ) on solution parameters.

**Effect of temperature:** The effect of increasing temperature on the corrosion rate of carbon steel (expressed by  $i_{corr}$ ) was studied at a temperature range 25-40°C in the absence and presence of tetracycline. Itis clear that raising the temperature led to increased of corrosion rate. Some activation thermodynamic parameters such as the activation energy  $E_a^*$ , the and the entropy of activation  $\Delta S^*$  for the corrosion of carbon steel in 0.1M HCl+0.6 M NaCl solutions were calculated as shown in Table 3. The dependence of the corrosion current density ( $i_{corr}$ ) on temperature followed Arrhenius Eq. 1:

$$\text{rate}(r) = i_{corr} = A \exp(-E_a/RT) \quad (1)$$

which can be expressed in logarithmic form Eq. 2:

Table 2: Data of polarization curve for corrosion of carbon steel in 0.6 mol.dm<sup>-3</sup> NaCl solution at pH(1) over the temperature range (298-313) K

T/K	i <sub>corr</sub> /μA.cm <sup>-2</sup>	-E <sub>corr</sub> /mV	-bc/mV decade <sup>-1</sup>	-bc/mV decade <sup>-1</sup>	Weight loss/g.m <sup>-2</sup> .day <sup>-1</sup>	Penetration loss/mm.year <sup>-1</sup>
298	265.12	472.0	64.3	72.7	6.63	3.08
303	373.24	466.9	64.1	81.0	9.33	4.33
308	410.16	461.0	61.7	82.9	1.03	4.76
313	783.72	480.0	64.4	61.6	1.96	7.94

Table 3: Activation Energy (E<sub>a</sub>), pre-exponential (A) and entropy of activation (ΔS\*) for carbon steel corrosion in 0.6 mol.dm<sup>-3</sup> NaCl solution in pH(1)

E <sub>a</sub> /kJ.mol <sup>-1</sup>	-ΔS*/J.k <sup>-1</sup> .mol <sup>-1</sup>	A/molecule m <sup>-2</sup> .S <sup>-1</sup>
25.31	108.1	12.35 10 <sup>32</sup>

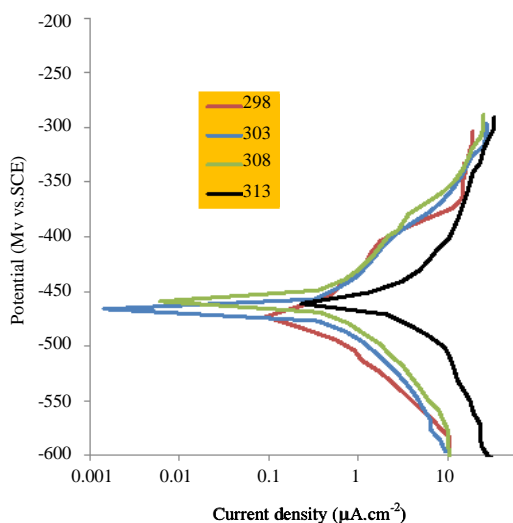


Fig. 2: Polarization curves of carbon steel corrosion in pH = 1 with NaCl solution at four temperatures in the range of (298-313) K

$$\ln i_{\text{corr}} = \ln A - \frac{E_a}{RT} \quad (2)$$

where, A and E<sub>a</sub> are respectively the pre-exponential factor and the activation energy of corrosion reaction.

Table 3 represents the values of E<sub>a</sub>, ΔS\* and the pre-exponential factor (A) for carbon steel sample in (pH = 1). From value of A using the relationship (3) Entropy of activation (ΔS\*) was calculated Eq. 3:

$$A = \frac{kT}{h} \exp \frac{\Delta S^*}{R} \quad (3)$$

Where:

k = Boltzman constant

h = Plank constant

R = The universal gas constant

T = The asbsolute temperature of the solution

Figure 3 represents the relation between ln icorr and 1/T for carbonsteel in 0.1 M HCl solution in the absence

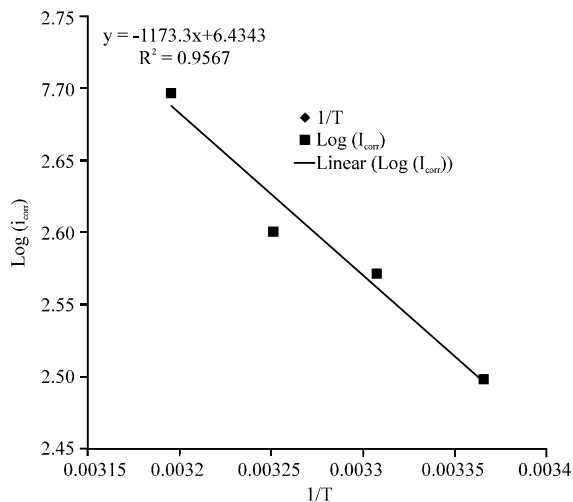


Fig. 3: Arrhenius plot relating Log (i<sub>corr</sub>) to 1/T for the corrosion of carbon steel in the pH = 1 in presence of NaCl over the temperature range (298-313) K

and presence of the studied antibiotic. A straight lines were obtained with slope equal to -E<sub>a</sub>\*/R. The calculated values of E<sub>a</sub> obtained from the slope of the straight line. The negative value of entropy of activation indicates implies that activated complex in the (rds) rate determining step explaine association than dissociation, suggests that a decreasein disorder takes place, ingoing from reactants to the activated complex.

#### Corrosion inhibition of carbonsteel by tetracycline:

Figure 4 shows the typical polarization curves of carbonsteel in 0.6 mol.dm<sup>-3</sup> NaCl (pH = 1) solution containing three different concentrations of antibacterial drug (tetracycline) as a green inhibitor over the temperature range (298-313) K. Table 4 presents the polarization data (E<sub>corr</sub> and i<sub>corr</sub> and IE%) showing that there is a decrease in corrosion current densities with presence of tetracycline, the inhibition effect of tetracycline increases as the concentration of the inhibitor increases in the range (0.5×10<sup>-4</sup> and -2×10<sup>-4</sup>) mol.dm<sup>-3</sup> at all temperatures of study.

Table 4 shows the values of Inhibition Efficiencies (IE%) which were calculated from Eq. 4:

$$IE\% = \frac{i_{\text{corr}}^0 - i_{\text{corr}}}{i_{\text{corr}}^0} \times 100\% \quad (4)$$

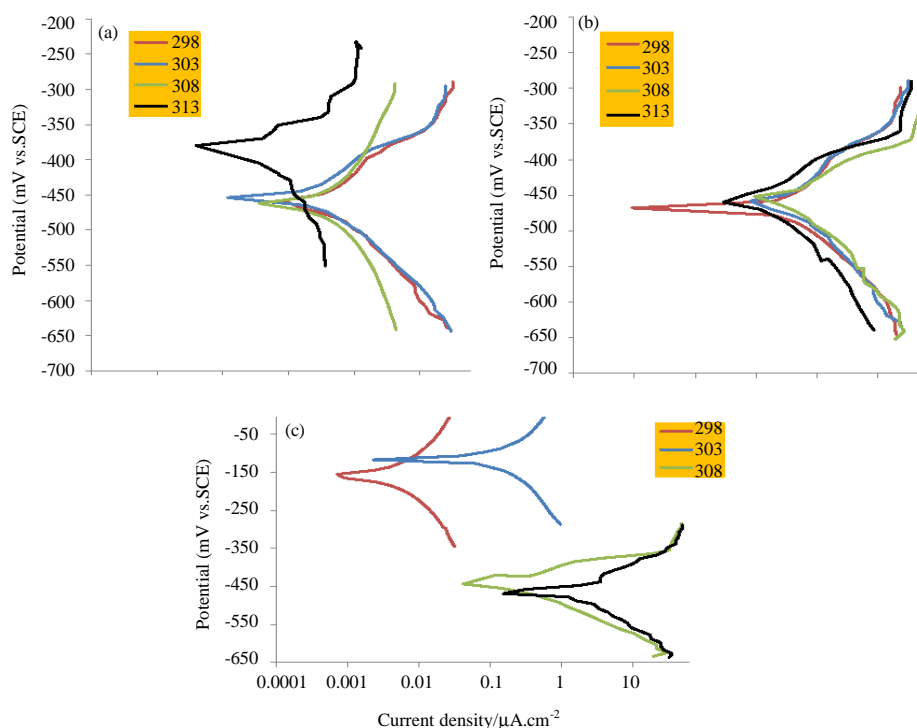


Fig. 4: Polarization curves of carbon steel corrosion in pH = 1 with NaCl solution in presence of different concentration of tetracycline at four temperatures in the range of 298-313 K: a)  $1 \times 10^{-4}$  mol.dm<sup>-3</sup>; b)  $0.5 \times 10^{-4}$  mol.dm<sup>-3</sup> and c)  $2 \times 10^{-4}$  mol.dm<sup>-3</sup>

Table 4: Values of  $E_{\text{corr}}$ ,  $i_{\text{corr}}$  with different concentrations of tetracycline at temperature range (298-313) K in pH = 1

T/K	Conc.inh.mol.dm <sup>-3</sup>	$-E_{\text{corr}}/\text{mv}$	$i_{\text{corr}}/\mu\text{A.cm}^{-2}$	$\theta$	IE (%)
308	0	472	265.12	-	-
	$1 \times 10^{-3}$	482	72	0.728	72.84
	$5 \times 10^{-3}$	461.8	69.42	0.738	73.81
	$1 \times 10^{-2}$	448.8	5	0.981	98.11
	0	466.9	373.24	-	-
	$1 \times 10^{-3}$	472.1	128.08	0.656	65.68
	$5 \times 10^{-3}$	453	113.53	0.696	69.58
	$1 \times 10^{-2}$	111.1	34.54	0.905	90.75
	0	461	410.16	-	-
	$1 \times 10^{-3}$	452.8	159.63	0.611	61.08
	$5 \times 10^{-3}$	448	140.38	0.658	65.8
	$1 \times 10^{-2}$	444.9	53	0.871	87.1
313	0	480	783.72	-	-
	$1 \times 10^{-3}$	438	314.8	0.598	59.83
	$5 \times 10^{-3}$	452.9	270	0.655	65.5
	$1 \times 10^{-2}$	424	189	0.758	75.88

where,  $i_{\text{corr}}^0$  and  $i_{\text{corr}}$  are corrosion current densities in absence and presence of the inhibitor, respectively. Regarding tetracycline effect of corrosion inhibition it is mainly attributed to the presence of electron donor groups (N, O) in its structure. The presence of heteroatoms, for nitrogen and oxygen in the structure of inhibitor molecules makes them good corrosion inhibitors (Mihit *et al.*, 2008). Most organic substance used as corrosion inhibitors can be adsorbed on the metal surface through their heteroatoms. Its inhibition efficiency

Table 5: Activation Energy ( $E_a$ ), pre-exponential (A) and entropy of activation ( $\Delta S^*$ ) for carbon steel corrosion in 0.6 mol.dm<sup>-3</sup> NaCl solution in pH (1) in presence of tetracycline

Conc. of tetracycline (mol.dm <sup>-3</sup> )	$E_a/\text{kJ.mol}^{-1}$	$-\Delta S^*/\text{J.K}^{-1}\text{.mol}^{-1}$	A/molecule m <sup>2</sup> .S <sup>-1</sup>
$0.5 \times 10^{-4}$	071.32	025.84	001.379
$1 \times 10^{-4}$	078.79	048.83	022.37
$2 \times 10^{-4}$	109.44	137.75	107.09

should follow the sequence O < N < S (Moretti and Guidi, 2002). Electron pairs on the heteroatoms can be shared with metal orbitals, forming an insoluble complex that protect the surface from the aggressive ions by blocking of its corrosion sites and hence, decreasing the corrosion rate (Khaled *et al.*, 2009). The presence of pairs of free electrons on nitrogen and oxygen atoms leads to form insoluble complex by ligand reaction with  $\text{Fe}^{2+}$  ion.

Some kinetic parameters for corrosion process  $E_a$  and  $\Delta S^*$  were calculated and summarized in Table 5 in the existence of inhibitor, activation Energy ( $E_a$ ) increased in the presence of tetracycline that means the addition of it has increased the energy barrier of the corrosion process of carbon steel in chloride acid solution (Fakrudeen *et al.*, 2012).

It was found that there is a direct relation between the values of  $E_a$  and A, i.e., simultaneous increase or decrease in  $E_a$  and  $\ln A$  for a particular system which can be

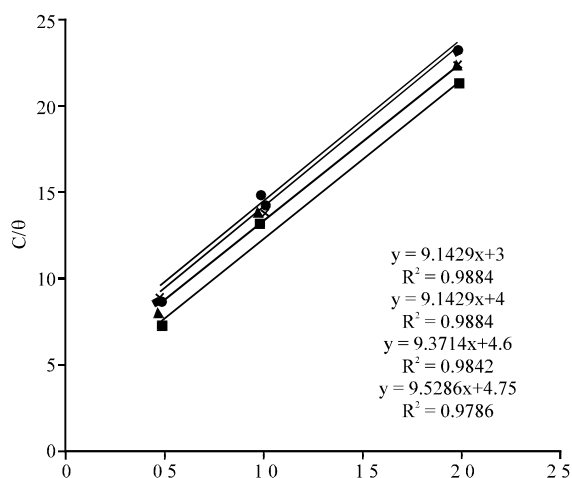


Fig. 5: Langmiure adsorption plots of tetracycline on carbonsteel in NaCl solution pH = 1 at various temperature

ascribed to the compensation effect which describe the kinetics of catalytic and tarnishing reaction on themetal.

The negative values of  $\Delta S^*_a$  in the existence of tetracycline meaning that the (rds) for the activated complexis association ratherthan dissociation step this means the activated molecules arein higher order than the initialstate.

#### Thermodynamic parameters ofthe adsorption isotherm:

The adsorption process wurface and the isas studied for abetter understanding of the electrochemical process occurring on the metal surface and the adsorption isotherm of langmiur was plotted Fig. 5 which is given bythe general Eq. 6 (Li *et al.*, 2011):

$$\frac{C_{inh}}{\theta} = \frac{1}{k_{ad}} + C_{glu} \quad (6)$$

Where:

$k_{ads}$  = The equilibrium constant of the adsorption/desorption process

$C_{inh}$  = The concentration of inhibitor(tetracycline)

From the intercepts of the straight lines on the  $C_{inh}/\theta$  axis Fig. 5 leads to the equilibrium constant for the adsorption/desorption of tetracycline process Table 6.

However,  $k_{ads}$  decreased with an increase of temperature indicating that adsorption of tetracycline on to the metal surface was favorable at lower temperatures. From Eq. 7 and based on  $K_{ads}$  values,  $\Delta G^0_{ads}$  was calculated and given in Table 6 (Eq. 7):

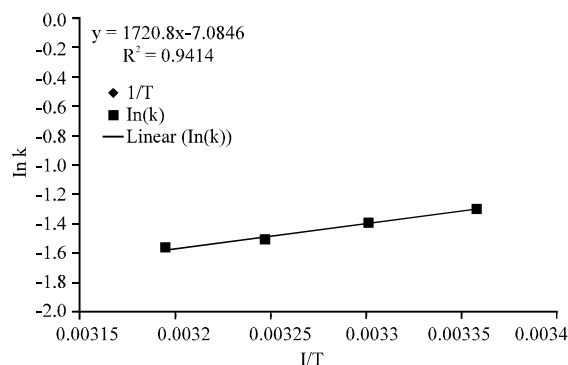


Fig. 6: Van't Hoff plot plot for of carbon steel in 0.6 mol.dm NaCl solution containing tetracycline at pH value (1)

Table 6: Equilibrium constant adsorption/desorption, standard free energy, enthalpy and entropy of adsorption onto carbon steel surface in 0.6 mol<sup>3</sup> NaCl solution in pH = 1 in the presence of tetracycline at various concentrations

T/K	$k_{ads}$ mol <sup>-1</sup>	$-\Delta G^0_{ads}$ kJ.mol <sup>-1</sup>	$-\Delta H^0_{ads}$ kJ.mol <sup>-1</sup>	$-\Delta S^0_{ads}$ kJ.mol <sup>-1</sup> .K <sup>-1</sup>
298	0.33	7.204		-0.0238
303	0.25	6.626		-0.0253
308	0.217	6.372		-0.0257
313	0.211	6.403		-0.0252

$$\Delta G^0_{ads} = -RT \ln(55.5k_{ads}) \quad (7)$$

Where:

R = The universal gas constant

T = The absolute temperature

55.5 = Concentration of water in solution

Generally,  $\Delta G_{ads}$  values of -20 kJ.mol<sup>-1</sup> or above are related with an electro static interaction between charged molecules of inhibitor and charged metal surface (physisorption) while those of -40 kJ.mol<sup>-1</sup> or below involves charge transferring from the inhibitor molecules to metal surface forming a coordinate covalent bond, (chemisorption). The valuesof  $\Delta G_{ads}$  are l isted in Table 6 ranged from (-6.3- to 7.2) kJ.mol<sup>-1</sup> from which it canbe concluded that adsorption of tetracycline on to carbonsteel surface occurs through physical adsorption (Ashassi-Sorkhabi *et al.*, 2004) (Fig. 6).

From Table 4 it was clearly seen that the presence of tetracycline reduces the corrosion rate of carbon steel in hydrochloric acid solution.  $E_{corr}$  shifts to little more negative values in the presence of tetracycline.

This result indicates that the inhibitors have been adsorbed to both cathodic and anodic areas. That means tetracycline is mixed type inhibitor. The same results have been reported by other researchers (Nnamna *et al.*, 2011).

As its well known that different substituents on the organic molecules polarize the functional group in a

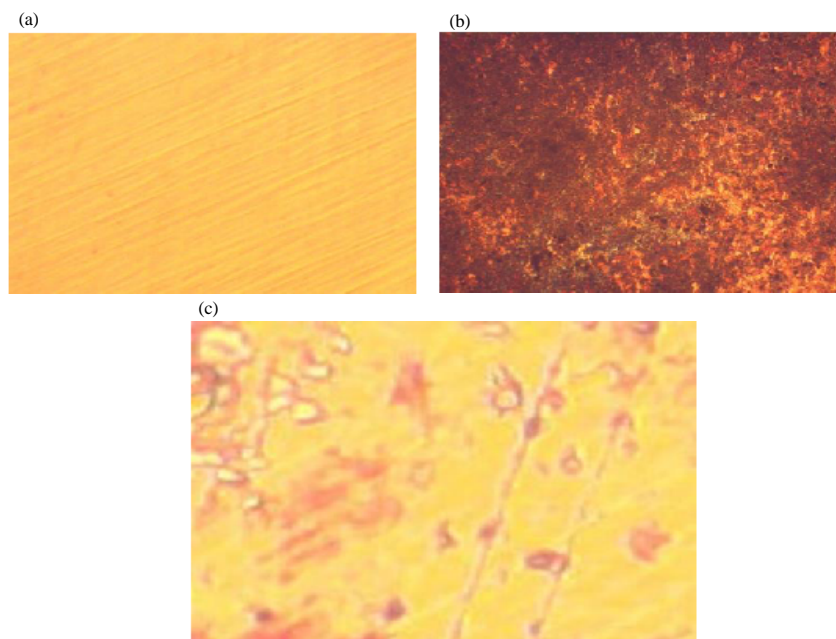
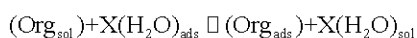


Fig. 7: Typical microstructure of carbon steel in the corroded medium, magnification power 400: a) Polished carbon steel; b) Immersed in (HCl+NaCl) and c) Immersed in (HCl+NaCl+tetracycline)

different manner. The adsorption of organic adsorbate on the surface of carbon steel electrode is regarded as substitutional adsorption process between the organic compound in the aqueous phase ( $\text{Org}_{\text{aq}}$ ) and the water molecules adsorbed on the carbon steel surface ( $\text{H}_2\text{O}_{\text{ads}}$ ):



where, X is the number of water molecules replaced by one organic molecule. From the values of thermodynamic parameters for corrosion process, mechanism of inhibition can be concluded.

Thermodynamically,  $\Delta S_{\text{ads}}^0$  were related to the  $\Delta H_{\text{ads}}^0$  and  $\Delta G_{\text{ads}}^0$  according to the following Eq. 8 (Megalai *et al.*, 2013):

$$\Delta G_{\text{ads}}^0 = \Delta H_{\text{ads}}^0 - T\Delta S_{\text{ads}}^0 \quad (8)$$

and  $\Delta H_{\text{ads}}^0$  can be calculated on basis of the Van't Hoff formula Eq. 9:

$$\ln K_{\text{ads}} = -\frac{\Delta H_{\text{ads}}^0}{RT} + \text{const} \quad (9)$$

A plot of  $\ln k_{\text{ads}}$  vs.  $1/T$  gives a straight line as show in Fig. 6. The slope of straight line is  $-\Delta H_{\text{ads}}^0/R$ . Generally, endothermic adsorption process is suggests chemisorption while exothermic process attributed to either physisorption or chemisorption. Negative sign of  $\Delta H_{\text{ads}}^0$  shows that adsorption of tetracycline molecules is

exothermic process (Fakrudeen *et al.*, 2012).  $\Delta H_{\text{ads}}^0$  of inhibitor can be compute from Eq. 8 according to Megalai *et al.* (2013) (Eq. 10):

$$\Delta S_{\text{ads}}^0 = \frac{\Delta H_{\text{ads}}^0 - \Delta G_{\text{ads}}^0}{T} \quad (10)$$

The values of  $\Delta H_{\text{ads}}^0$  in the existance of tetracycline molecules are negative that is accompanied with exothermic adsorption process, so, it must be accompanied by adecrease of entropy change.

**Optical microscopy observation:** To investegate the effect of tetracycline on carbon steel morphology, surface examination using optical microscopy was carried out Fig. 7a-c polished carbon steel surface, carbon steel surface immersed in (Hcl+NaCl) the specimen surface was strongly damaged, inpresence of tetracycline  $2 \times 10^{-2} \text{ mol.L}^{-1}$  shows that there was much less damage on the surface.

## CONCLUSION

Results gained from potentiodynamic polarization technique show that tetracycline acts as effective inhibitors for carbon steel dissolution in 3.5% NaCl solution at pH 1. Inhibition efficiency increases with increasing the concentration of inhibitors as well as with

decreasing temperature. Adsorption of tetracycline molecules on carbon steel surface occurs by interaction of  $\text{Fe}^{+2}$  with (N, O) atoms, blocking corrosion process. Values of  $\Delta G_{\text{ads}}$  indicate that the adsorption process of inhibitors on the carbon steel surface was spontaneous.

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