



## Effect of black tea as eco-friendly inhibitor on the corrosion behavior of mild steel in different media

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### ABSTRACT

Effect of Black tea extract (BTE) as green inhibitor on the corrosion behavior of mild steel in 1.0M HCl, 1.0M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl was studied by potentiodynamic polarization techniques open-circuit potential, linear polarization resistance and Tafel plots polarization, its clear BTE gives inhibition in both anodic and cathodic slopes in Tafel polarization, that indicate this inhibitor used as mixed-type inhibitor, the thermodynamic adsorption isotherm was studied and falls into Langmuir isotherm, and it is physisorption isotherm. Also the surface morphology of MS samples without and with the inhibitor was examined using scanning electron microscopy.

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### Introduction

Due to the aggressive nature of acid solution exposed to MS as an example of metals, a lot of studies were focused on the inhibition of metals corrosion especially the compounds have O, N, S and P as hetero cyclic atoms [1-5], some of these compounds cost in its preparation and have toxic effect for user. In the recent researches the choice of inhibitor directed to eco-friendly environment inhibitors (green inhibitor) because it's non toxic, safety for human uses, low cost and easy extraction from nature by simple methods. Two reviews include the using of green inhibitor to prevent, stop or retard the corrosion of metals exposed to aggressive media at recent years [6-7], recent studies on the corrosion inhibition by natural (green) inhibitors have been investigated [8-19]. In this present study Black tea used as corrosion inhibitor as it safety, easily extraction and cheap, also it follows Theaceae family and contains on some compounds such as (-)-Epicatechin (EC), Epicatechin 3-gallate (ECG), (-)-Epigallocatechin (EGC), Epigallocatechin 3-gallate (EGCG), Gallic acid and found of aromatic oils in its leaves, also, it contains on oxidized vitamins and amino acids specially theanine which exclusive to tea.

### Experimental Approach

#### Preparation of Black Tea Extract and Corrosives Media:

An aqueous extract solution was prepared by reflux 10 g of black tea (Al Arosa tea) in 100 ml of double distilled water for about 1 hr, then, filtering the refluxed solution to remove any contaminations, and evaporate 1 ml of filtered extract solution and weighted the residue [20] and preparation of 1.0M HCl, 1.0 M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl solutions as corrosives media.

#### Preparation of Specimens:

The mild steel specimens tested in this present paper, are in the sheet form, supplied by AL-EZZ Company in Alexandria. The designation and analysis of the material is listed in Table 1. Prior immersion the MS electrodes in the tested solutions, it was prepared by polishing with SiC paper up to 1200 grade, washing with distilled water and then degreased with alcohol about 5

minutes, washed again with distilled water then dried it by used filter papers.

#### Electrochemical Cell (Corrosion Cell):

The corrosion cell used in potentiodynamic polarization involving three electrodes, MS as working electrode, Saturated Calomel Electrode (SCE) as reference electrode and a platinum wire used as counter electrode. The exposed area of working electrode to solutions was 1Cm<sup>2</sup>.

#### Adopted techniques and other calculations:

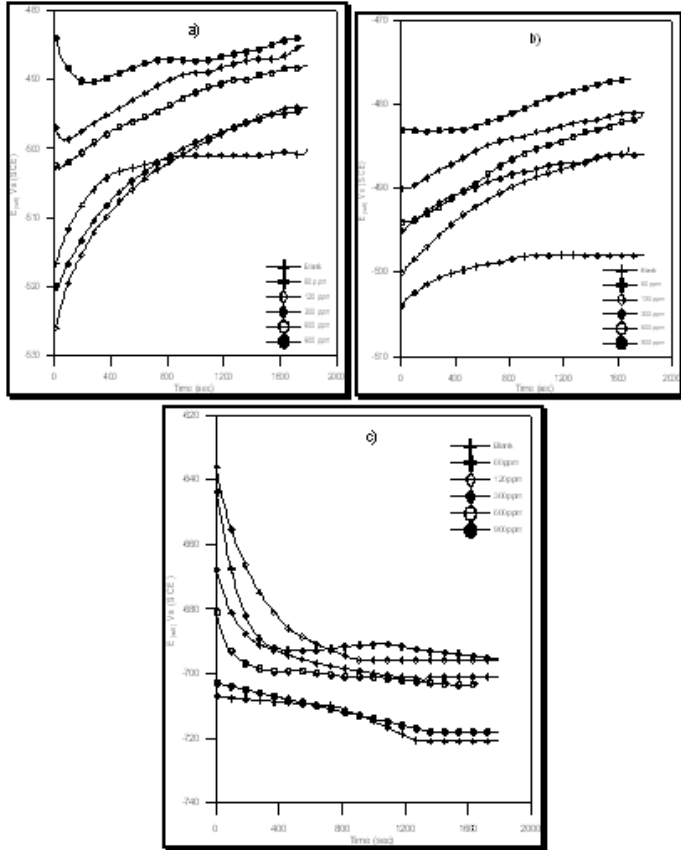
The major electrochemical techniques used in this study are open circuit potential (OCP) and Potentiodynamic polarizations (linear polarization resistance (LPR) and Tafel plots polarizations (TP)) which carried out with model 352/252 corrosion measurement system, which consists of EG & G potentiostat/galvanostat model 273A driven by software from IBM computer. Measurements of polarizations curves were started after immersing the working electrode in the used solutions for about 30 minutes, where the values of E<sub>corr</sub> became stable. The LPR was measured from +20 mV up to -20 mV vs E<sub>corr</sub> and the TP was measured from -250 mV up to +250 mV vs E<sub>corr</sub>. The scan rate for LPR and TP measurements were 0.166 mV/S and 0.3mV/S, respectively.

### Results and Discussion

#### Open -Circuit Potential (OCP) Measurements:

Fig. (1) shows the potential-time curves for MS exposed to 1.0 M HCl, 1.0M H<sub>2</sub>SO<sub>4</sub> and 35 g/l NaCl, respectively, at different concentration of BTE, from these curves the steady state potential (E<sub>s,s</sub>) for blank solutions shift to positive direction than immersion potential (E<sub>im</sub>) that indicate to forming of oxide films on the surface of tested metal. On the other hand at addition of BTE to blank solution at MS electrode, E<sub>s,s</sub> shift to more noble direction than blank solutions that indicates to adsorption on the active anodic and cathodic reactions on the surface of MS. Data listed in Table 2 illustrated the values of E<sub>s,s</sub> and E<sub>im</sub> for MS electrode in different corrosive media at addition the inhibitor and these data emphasize that E<sub>s,s</sub> shift to more positive direction than blank solutions, that attributed to blocking

of the active sites on the anodic and cathodic reactions on the surface of metal.



**Fig. 1. Potential-time curves for mild steel in a) 1.0M HCl, b) 1.0M H<sub>2</sub>SO<sub>4</sub> and c) 35g/l NaCl at different concentrations of BTE**

**Rate of Oxide Film Thickening:**

A theory for film thickening on the surface of metal and alloy based on OCP measurements has been developed by Abd El-Kader and Shams El-Din [21]. The essence of the theory is based on the idea that the potential is determined by simultaneous anodic (film formation) and cathodic (oxygen reduction) couple, in which the anodic reaction is the rate limiting. By presenting the data in form of potential–log (time) curves, straight lines were obtained satisfying the relation [21]

$$E = \text{constant} + 2.303 (\delta/\beta) \log t \dots\dots\dots (1)$$

Where (E) electrode potential with respect to a saturated calomel electrode, (t) is the time from the moment of immersion in solution,  $\delta$  is the rate of oxide film thickening per decade of time and  $\beta$  is given by equation (2):

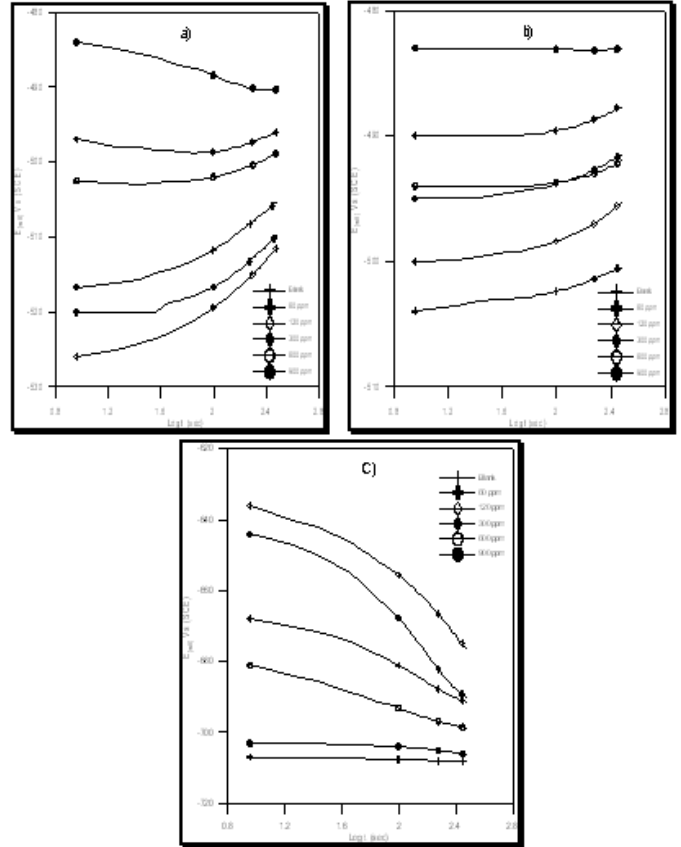
$$\beta = (nF / RT) \alpha \delta^{-1} \dots\dots\dots (2)$$

Where ( $\alpha$ ) is a transference coefficient similar to that found in electrochemical kinetic rate expressions ( $0.0 < \alpha < 1$ ), and ( $\delta^{-1}$ ) is the width of the activation energy barrier to be traversed by the ion during oxide formation. The theory was applied to the cases of oxide growth on nickel, titanium [22], Fe-Cr alloys [23], and molybdenum steels [24].

It is evident from the curves in Fig. 2 (a,b and c) that the relation between potential of the MS electrode versus the logarithm of the immersion time until  $E_{s,s}$  is confirmed (formation of oxide film on the surface of MS). This behavior can be readily understood when one takes into consideration the physical properties of iron oxides, covering the metal surface. Consideration of the curves in Fig. 2 (a,b and c) and the value of ( $\delta$ ) in Table (3) allows the following conclusion to be drawn:

(a) The rate of oxide film thickening ( $\delta$ ) for MS in 1.0M HCl, 1.0M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl solutions ( 0.222, 0.063 and 0.025 nm/log t), respectively.

(b) The rate of oxide film thickening for MS immersed in different concentrations of BTE as inhibitor at previous arranged blank media varied between (0.034 – 0.276 nm/log t), (0.004-0.091 nm/log t) and ( 0.062-0.928 nm/log t), respectively.



**Fig 2. Potential-Log t curves for mild steel in a) 1.0M HCl, b) 1.0M H<sub>2</sub>SO<sub>4</sub> and c) 35g/l NaCl at different concentrations of BTE**

**Potentiodynamic Polarization Measurements:**

The anodic and cathodic polarization curves (Tafel plot polarization) of mild steel in 1.0 M HCl, 1.0M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl solutions without and with (60, 120, 300, 600 and 900 ppm) of BTE show in Fig. 3. It was observed that both the cathodic and anodic curves showed lower current density in the presence of BTE concentrations than those observed in the solution without the inhibitor. This indicates that BTE inhibits the corrosion process. The results shown in Fig. 3 also suggested that the studied extract affect both anodic and cathodic corrosion process hence it reveals properties of a mixed-type inhibitor. The electrochemical parameters, i.e. corrosion current density ( $I_{corr}$ ), anodic ( $\beta_a$ ) and cathodic ( $\beta_c$ ) Tafel constants, polarization resistance ( $R_p$ ) and the inhibition efficiency (IE %) are shown in Tables (4, 5). It can be observed that the concentration of inhibitor has a little influence on values of  $\beta_a$  and more significant influence on the values of Tafel  $\beta_c$  indicating that inhibitor may change the mechanism of cathodic reaction and may not affect the process of anodic dissolution. The data clearly show that the  $I_{corr}$  values decreased and  $R_p$  values increased in the presence of BTE various concentrations as expected. Due to the inverse relationship between  $I_{corr}$  and  $R_p$ , with increasing concentration of the inhibitor, it can be assumed that the adsorption of the inhibitor molecules on metal surface makes a physical barrier for the mass and charge transfer, providing high degree of protection to the metal surface.

**Table 1. Chemical composition of mild steel by wt%**

C	Si	Mn	P	S	Cr	Mo	Ni
0.175	0.130	0.534	0.008	0.0094	0.0117	0.002	0.0188
Al	Co	Cu	Nb	Ti	V	W	Fe
0.005	0.0075	0.0047	0.003	0.002	0.005	0.172	99.07

**Table 2. Values of  $E_{im}$  and  $E_{s,s}$  (mV) for mild steel in different corrosive media at different concentrations of BTE**

Conc.	Media	$-E_{im}$ (mV)	$-E_{s,s}$ (mV)
1.0M blank	H <sub>2</sub> SO <sub>4</sub>	504	498
60	BTE	495	486
120		500	485
300		490	481
600		494	481
900		483	477
1.0M blank	HCl	495	488
60	BTE	520	495
120		526	494
300		499	485
600		502	488
900		484	490
35g/l blank	NaCl	707	721
60	BTE	644	695
120		636	696
300		668	701
600		681	703
900		717	713

**Table 3. Values of slopes of E (mV) vs. log t lines and the rate of oxide film thickening ( $\delta'$ ) (nm/log t) for corresponding the mild steel in different concentrations of BTE as natural product at different media.**

Test solution		Slope	Rate (nm/log t) $\delta'$
HCl 1.0 M (Blank)		8.72	0.222
BTE	60 ppm	8.12	0.207
	120	10.83	0.276
	300	1.33	0.034
	600	3.05	0.078
	900	4.81	0.122
H <sub>2</sub> SO <sub>4</sub> 1.0 M (Blank)		2.48	0.063
BTE	60 ppm	2.75	0.070
	120	3.56	0.091
	300	1.88	0.048
	600	1.54	0.039
	900	0.14	0.004
NaCl 35 g/l (Blank)		1.00	0.025
BTE	60 ppm	36.48	0.928
	120	29.87	0.760
	300	18.08	0.460
	600	12.42	0.316
	900	2.44	0.062

**Table 4. Polarization parameters from TP for mild steel in different media at different concentrations of BTE**

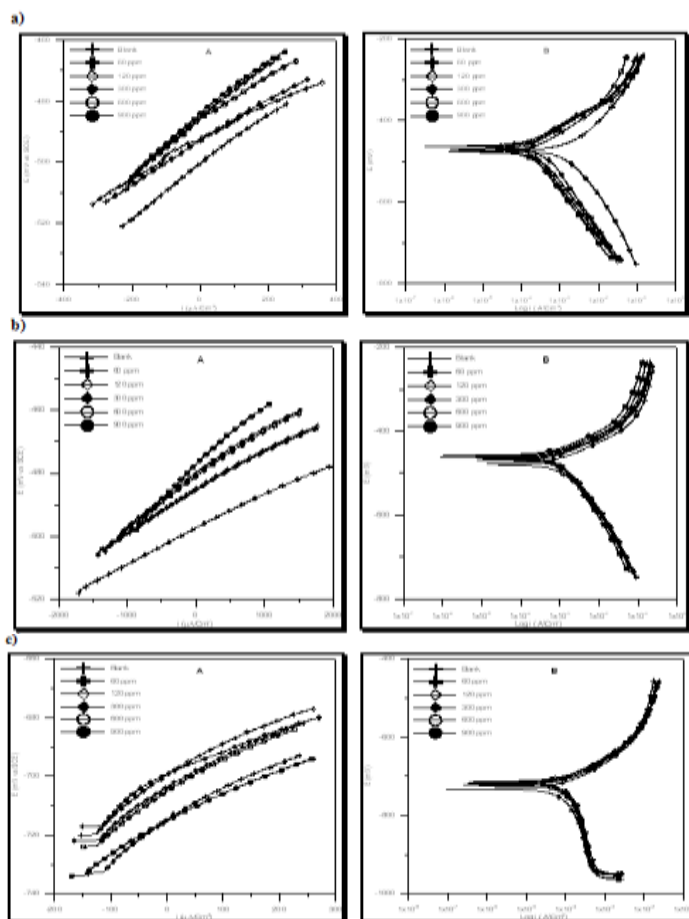
Media	C (ppm)	R <sub>p</sub> ( $\Omega$ )	Ba mV/decade	Bc mV/decade	-E <sub>corr</sub> (mV)	I <sub>corr</sub> ( $\mu$ A/Cm <sup>2</sup> )	C.R (mpy)	I.E (%)	Surface coverage ( $\theta$ )
1.0 M HCl	-	16.87	102.7	303.6	463.2	1126	1027	-	-
BTE	60	70.82	80.03	137.9	464.9	339.6	310	69.8	0.698
	120	71.11	76.81	131.4	477	298	272	73.5	0.735
	300	83.39	77.97	149.5	463.4	232.9	212.6	79.3	0.793
	600	89.40	68.2	129.6	474.2	180.9	165	83.9	0.839
	900	92.77	57.2	115.7	470.3	129.4	118	88.5	0.885
1.0 M H <sub>2</sub> SO <sub>4</sub>	-	4.9	115.1	185.7	476	4726	4313	-	-
BTE	60	12.63	81.16	152.8	469	1625	1484	65.6	0.656
	120	13.31	75.86	151.6	466	1484	1354	68.6	0.686
	300	15.29	53.78	135.3	459.8	1010	921.4	78.6	0.786
	600	17.05	56.32	128.4	463.5	922.6	842	80.5	0.805
	900	23.01	50.79	126.2	457.5	722	659.3	84.7	0.847
35 g/l NaCl	-	77.37	153	29.17	721	346.3	316.1	-	-
BTE	60	82.26	78.94	445.5	717.6	175	159.7	49.5	0.495
	120	97.5	83.89	502.9	713	162.2	148	53.2	0.532
	300	104.7	83.26	459.4	717.6	152.7	139.4	55.9	0.559
	600	130.6	72.07	398.4	715	142	129.6	59.0	0.590
	900	135	72.79	376.1	722.5	136.7	124.8	60.5	0.605

**Table 5. Polarization parameters from LPR for mild steel in different media at different concentrations of BTE**

Media	C (ppm)	R <sub>p</sub> (Ω)	-E <sub>corr</sub> (m V)	I <sub>corr</sub> (μA/Cm <sup>2</sup> )	I.E (%)	Surface coverage (θ)
1.0 M HCl	-	16.87	485.9	1288	-	-
BTE	60	70.82	493.2	306.7	76.2	0.762
	120	71.11	491.3	305.4	76.3	0.763
	300	83.39	484.8	260.4	79.8	0.798
	600	89.40	486.5	242.7	81.2	0.812
	900	92.77	484.7	234.1	81.8	0.818
1.0 M H <sub>2</sub> SO <sub>4</sub>	-	4.9	504.2	4395	-	-
BTE	60	12.63	485.9	1719	60.9	0.609
	120	13.31	485.7	1632	62.9	0.629
	300	15.29	480.8	1420	67.7	0.677
	600	17.05	480.8	1274	71.0	0.710
	900	23.01	477	943.7	78.5	0.785
35 g/l NaCl	-	77.37	711	280.7	-	-
BTE	60	82.26	700.3	264	5.95	0.059
	120	97.5	699.7	222.7	20.7	0.207
	300	104.7	704.6	207.4	26.1	0.261
	600	130.6	705.8	166.5	40.7	0.407
	900	135	713	160.8	42.7	0.427

**Table 6. Values of Langmuir adsorption parameters at exposed mild steel to BTE in different media**

Inhibitor	Type of polarization	Slope≈	Log K	R <sup>2</sup>	-ΔG <sub>ads</sub> , kJmol <sup>-1</sup>
BTE at 1.0 M HCl	TP	1.11	1.489048	0.999	18.45
	LPR	1.21	2.011955	0.999	21.43
BTE at 1.0 M H <sub>2</sub> SO <sub>4</sub>	TP	1.16	1.486126	0.999	18.43
	LPR	1.26	1.335656	0.995	17.57
BTE at 35g/l NaCl	TP	1.63	1.468124	0.999	18.33
	LPR	1.83	0.348629	0.970	11.94



**Fig 3. Linear (A) and Tafel plots (B) polarization curves of mild steel in a)1.0M HCl , b) 1.0M H<sub>2</sub>SO<sub>4</sub> and c) 35g/l NaCl at different concentrations of BTE**

The LPR and TP results were found to be similar Tables 4 and 5. IE% was computed for MS in 1.0 M HCl, 1.0M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl solutions containing different concentrations of BTE from the Tafel plots and polarization resistance measurements are shown in Tables 4 and 5. The IE% values were obtained from I<sub>corr</sub> and R<sub>p</sub> data using the equation 3 and 4 as follow:

$$IE\% = (I_{corr} - I_{corr\ inhib.} / I_{corr}) \times 100 \dots\dots\dots (3)$$

$$IE\% = (R_p (inhibited) - R_p (uninhibited) / R_p (inhibited) \times 100 \dots\dots\dots (4)$$

Where I<sub>corr</sub> (uninh.), R<sub>p</sub> (uninh.) and I<sub>corr</sub>(inh.), R<sub>p</sub> (inh.) are the corrosion current density and resistance without and with inhibitor presence, respectively.

The IE% values obtained from the TP experiments (I<sub>corr</sub>) were higher than those obtained through the LPR (R<sub>p</sub>). This behavior suggests that the inhibitor action is dependent on the potential applied and time of polarization, since in the R<sub>p</sub> experiments only ±25 mV around E<sub>corr</sub> was applied to the working electrode and the duration of the experiment was shorter. However, the data clearly showed that the CR of MS electrode decreased in the presence of BTE.

From results we can conclude the sequence of corrosive media used in this study arranged at H<sub>2</sub>SO<sub>4</sub> > HCl > NaCl because SO<sub>4</sub> ion is aggressive than Cl ion and the presence of Cl ion in HCl is more than present in NaCl where (35.5 g/l, 21 g/l) for HCl and NaCl, respectively.

**Adsorption Isotherm Calculations:**

Calculation of adsorption isotherm one of the important methods that represent the interaction between the molecules of inhibitor and the metal surface, Langmuir model agrees more with my experimental results obtained by applied the following equation (5)

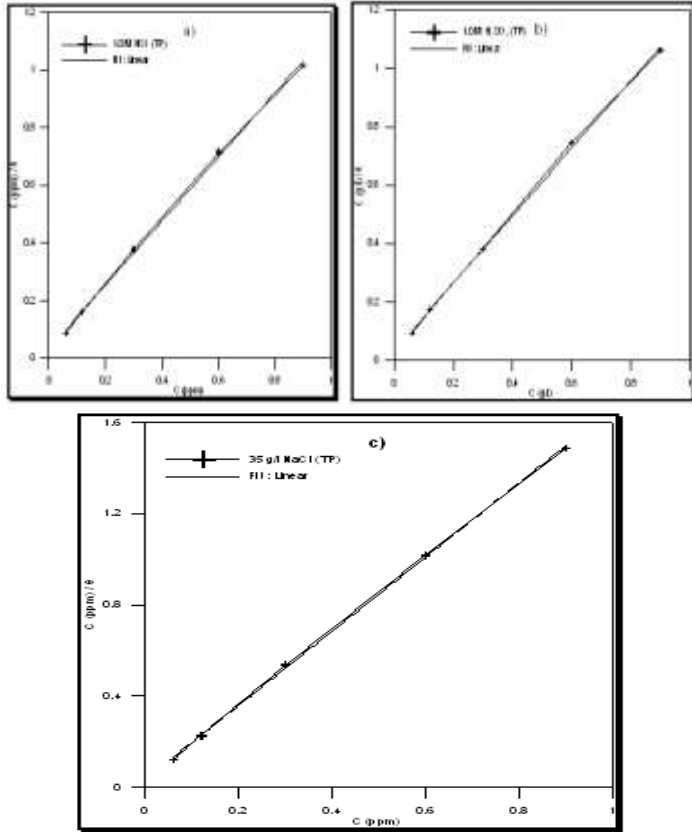
$$\theta / 1 - \theta = KC \dots\dots\dots (5)$$

The previous equation (5) can be modified into equation (6)

$$C/\theta = 1/K_{ads} + C \dots\dots\dots (6)$$

Where, ( $\theta$ ) surface coverage and obtained by  $\theta = IE\% / 100$ ,  
 (C) concentration of inhibitor and  $K_{ads}$  is equilibrium constant of adsorption. By using the parameters before in the following equation (6) for calculation the adsorption free energy [25]

$$\Delta G_{ads} = -2.303RT \log (55.5K) \dots\dots\dots (7)$$

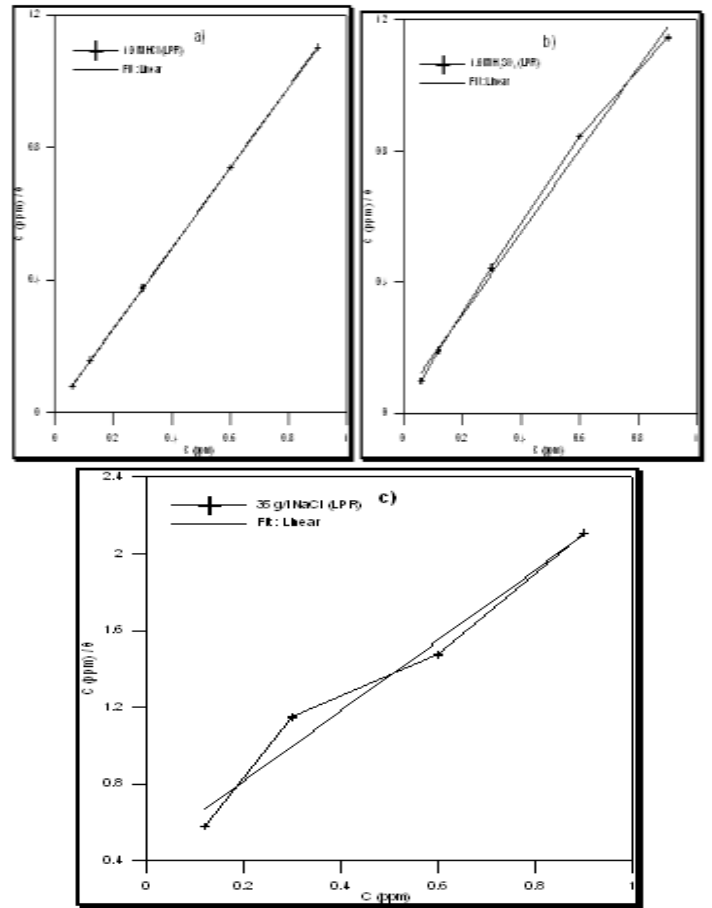


**Fig.(4). Curves fitting of different concentrations of BTE adsorption by Langmuir isotherm for mild steel at exposed to a) 1.0M HCl, b) 1.0M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl from ( Tafel polarization technique)**

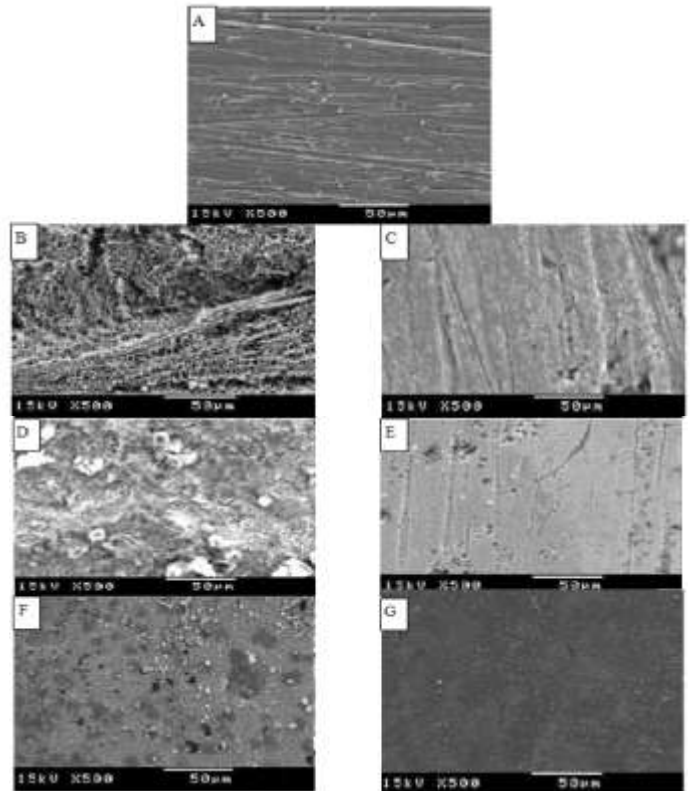
Fig.s 4 and 5 show that the linear line slope between C/ $\theta$  versus C which illustrated in equation 6, obtained from TP and LPR experiments, this linear line slope which equal to unity or near it and gives high correlation coefficient and illustrated the linear equation. The free energy of adsorption can calculated from equation 7, and Table 6 shows the values of  $\Delta G_{ads}$  which are negative in all cases, and it less than or over -20kj/mole but it lower than -40 kj/mole where this value of free energy adsorption is differ between physisorption and chemisorption, indicates to physisorption, where electrostatic forces between the inhibitor molecules and the surfaces of tested metals, strong physisorption at values over -20kj/mole not reach to -40 kj/mole.

**Scan Electron Microscope (SEM):**

SEM is one of the methods which explain the behaviour of metal surface against corrosive and inhibitor substances, fig. (6a) represented the morphology of MS surface before immersion in any solutions and it appears as smoothing surface except some scratching due to polishing before using the metal in experiments, in Fig. (6b, d,f) represented the surface exposed to corrosive media 1.0M HCl, 1.0M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl, respectively, and the surface damage as a result of deterioration of metal surface but at addition 5% of inhibitors at corrosive media shown in Fig (6c, e, g) the surface examine and layer of inhibitor formed or adsorbed on the surface of metal, that show hat the molecules of inhibitor act as defence for corrosive media to attack the surface of metal.



**Fig 5. Curves fitting of different concentrations of BTE adsorption by Langmuir isotherm for mild steel at exposed to a) 1.0M HCl, b) 1.0M H<sub>2</sub>SO<sub>4</sub> and 35g/l NaCl from (linear polarization resistance technique)**



**Fig 6. SEM of mild steel surface (A), MS+1.0M HCl (B), MS+1.0M HCl + 900 ppm of BTE (C), MS+1.0M H<sub>2</sub>SO<sub>4</sub> (D), MS+1.0M H<sub>2</sub>SO<sub>4</sub> + 900 ppm of BTE (E), MS+35g/l NaCl (F) and MS+35g/l NaCl + 900 ppm of BTE (G)**

**Conclusion:**

BTE is one of the natural products which gives best values of IE% for MS at acidic and neutral media, and the IE% increased as the concentration of BTE increased, physisorption occur in BTE at the surface of MS and the adsorption isotherm follow Langumir isotherm.

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