

## Tannin as Corrosion Inhibitor for Aluminium in Acidic Medium

Enas Hussien Ali and Takialdin A. Himdan

*College of Education for Pure Sciences Ibn-Al-Haitham, University of Baghdad, Baghdad, Iraq*

**Key words:** Corrosion inhibition, aluminium, tannin, adsorption mechanism, thermodynamic parameters

**Abstract:** Corrosion of aluminium in  $0.6 \text{ mol. dm}^{-3}$  NaCl solution in acidic medium ( $\text{pH} = 1$ ) was investigated in the absence and presence of different concentrations of tannin as environmentally friendly corrosion inhibitor over temperature range (298-313) K. The investigation involved electrochemical polarization method using potentiostatic technique and optical microscopy, the inhibition efficiency increased by increasing inhibitor concentration but decreased with temperature raising. 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 inhibition. The polarization measurements indicated that tannin is of mixed type. The surface characteristic of the inhibited and uninhibited metal samples were investigated by optical microscopy.

### Corresponding Author:

Enas Hussien Ali

*College of Education for Pure Sciences Ibn-Al-Haitham,  
University of Baghdad, Baghdad, Iraq*

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## INTRODUCTION

Corrosion is destruction of metal as a result of chemical reaction between metal and its environment that leads to a deterioration of metal physical and chemical properties<sup>[1]</sup>. It is a continues problem and often difficult to complete deletion, so, prevention is more practical<sup>[2]</sup>. Aluminium has excellent formability, low density, high thermal and electrical conductivity, attractive appearance and good resistance to corrosion when exposed to atmosphere or various aqueous environments because its ability to form a highly compact passive oxide layer<sup>[3-5]</sup>. These properties made aluminium one of the most attractive materials for industrial applications like automobiles, aviation, aerospace, food handling, containers, electronic devices, buildings, marine, etc.<sup>[6]</sup>. Although, the oxide film formed on aluminium relies for its corrosion immunity, this film is amphoteric, so, it

dissolves in case of exposes to high concentrations of acids or bases especially in present of chloride<sup>[7]</sup>. Among the most common protection method is inhibitors (organic and inorganic)<sup>[8-11]</sup> which when added to corrosive media in a small quantity reduce the corrosion are commonly used to reduce corrosion<sup>[12]</sup>. Most of the used inhibitors are toxic, non-biodegradable and expensive. Therefore, green corrosion inhibitors which are natural compounds have been widely used to protect aluminium because they are environmentally acceptable, readily available and renewable source for a wide range of inhibitors<sup>[13-19]</sup>.

The present work is another attempt to obtain environmentally and cheap inhibitor for aluminium in acidic medium containing (3.5% w/w) NaCl where tannin is tested.

Tannins comprise two different classes of polyphenolic compounds; hydrolysable and condensed tannins. The condensed tannins are found in substantial

concentration in the wood and bark of several trees, for instance, black wattle<sup>[20]</sup>. The tannin extracted from the bark of the black wattle tree contains flavonoid units such as (-)-robinetinidol, (+)-catechin and (+)-gallocatechin<sup>[21]</sup>. The flavonoid units are trimeric flavonols with fifteen carbons. The molecular weight of condensed tannins ranges from around 500 to over 20,000<sup>[20-22]</sup>. Due to the OH- groups in the ortho position on the aromatic rings, tannins are able to form chelates with iron and other metallic cations (e.g., copper)<sup>[23]</sup>. The application of tannins in corrosion studies has been investigated by several authors and their efficiency is controversial. The mechanism of the adsorption of mimosa tannin onto low-carbon steel in sulphuric acidic solutions has been investigated previously. At pH = 1 and 2, the value of the free energy of adsorption suggests a chemisorption mechanism. This mechanism occurs due to the formation of an adsorption bond between the oxygen lone-pair electrons of the tannin -OH group and the metal surface. At pH ≥ 3, ferric tannate is formed and the value of the free energy of adsorption suggests a physisorption mechanism of ferric tannate adsorption onto the steel surface<sup>[24]</sup>.

Electrochemical polarization measurements were used to evaluate the inhibition efficiency of tannin. Furthermore, this study aim to throw some light on the role of the functional groups of the inhibitors used in the adsorption process as well as the influence of the nature of the medium on the corrosion mechanism.

Optical microscopy was used to observe the morphology of aluminium surface corroded in test solution.

## MATERIALS AND METHODS

### Experimental part

**Materials:** The experiments were performed with aluminium sheets (99.8% purity) of 2 mm thickness which was mechanically press-cut into circular form 2 cm diameter. Chemicals used in this work include:

- Sodium chloride was used for preparation of the aggressive solution of 0.6 mol dm<sup>-3</sup>
- Hydrochloric acid
- Tannin tested as inhibitor (>99% purity). Its structure is shown in Fig. 1

**Solution:** The aggressive solution used was made of A.R. 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 the inhibitor (tannin) were used (3 × 10<sup>-5</sup>, 6 × 10<sup>-5</sup> and 9 × 10<sup>-5</sup>) 0.6 mol dm<sup>-3</sup>.

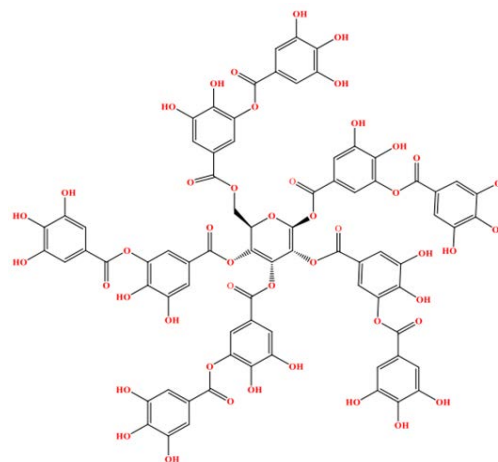


Fig. 1: Structure of tannin

**Potentiostatic polarization measurements:** The potentiostatic polarization measurements were carried out with aluminium sheets (explained before) having an exposed surface area of to corrosive medium (in absence and presence the inhibitor).

The open side of the working electrode (aluminium sheet) was grinded and polished mechanically using wheel machine. All samples were grinded via a series grades of emery papers (100,200,400,800,1200 and 2000) and polished with smooth cloth to a mirror finish. The specimen were degreased with acetone and thoroughly washed with distilled water then dried and kept in a desicator until use.

A three electrodes cell with aluminium specimen of 1 cm<sup>2</sup> exposed area, silver-silver chloride in auxiliary (counter) saturated KCL solution and a platinum electrode as working, reference and electrodes respectively were used to get polarization measurements.

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

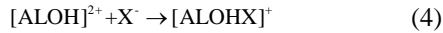
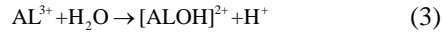
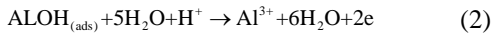
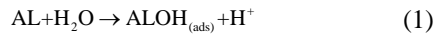
The experiments were performed in 0.6 mol dm<sup>-3</sup> NaCl solution of pH = 1 in absence and presence of three different concentrations of the inhibitor (tannin) over the temperature range (298-313)K. The experiments were carried out at a scan rate of 2 mV sec<sup>-1</sup>.

Corrosion current density ( $i_{\text{corr}}$ ) and corrosion potential ( $E_{\text{corr}}$ ) were determined from the polarization curve 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 was used to study the corrosion of aluminium in 0.6 mol dm<sup>-3</sup> NaCl solution in acidic medium at four different temperature. Figure 2 shows potentiostatic polarization curves. Corrosion parameters ( $E_{corr}$ ), ( $i_{corr}$ ), ( $b_a$ ) and ( $b_c$ ) are obtained from the Tafel polarization curves and tabulated in Table 1 and 2.

The results indicate the increase in the corrosion current density with temperature rising. From negative shift of with temperature rising can be concluded that anodic process is much more affected than cathodic, this observation is in accordance with other published results<sup>[1, 2]</sup> which proposed the dependence of ( $i_{corr}$ ) and ( $E_{corr}$ ) on solution parameters. The electrochemical cathodic and anodic reactions of aluminium in acidic solution can be described as follows<sup>[25]</sup>:



Thus, soluble complex ion formed leads to the dissolution of the metal.

**Effect of temperature:** The effect of temperature on the corrosion rate of aluminium (expressed by  $i_{corr}$ ) was studied by measuring the corrosion at the temperature range of (293-308)K which followed Arrhenius equation<sup>[26]</sup>:

$$\text{rate } (r) \equiv i_{corr} = A \exp(-E_a/RT) \quad (5)$$

which can be expressed in logarithmic form:

$$\ln i_{corr} \equiv \ln A - \frac{E_a}{RT} \quad (6)$$

where, A and  $E_a$  are respectively the pre-exponential factor and the activation energy of corrosion reaction. A typical linear plot relating values of  $\ln i_{corr}$  to the reciprocal of temperature ( $1/T$ ) as shown in Fig. 3 was obtained.

The values of  $E_a$  could be derived from the slope of the line and when the linear plot of Fig. 3 was extrapolated to  $\ln i_{corr}$  value at ( $1/T$ ) = 0, the value of A could be obtained. Table 3 represents the values of  $E_a$ ,  $\Delta S^*$  and the pre-exponential factor (A) for aluminium sample in pH = 1. ( $\Delta S^*$ ) was computed using Eq. 7:

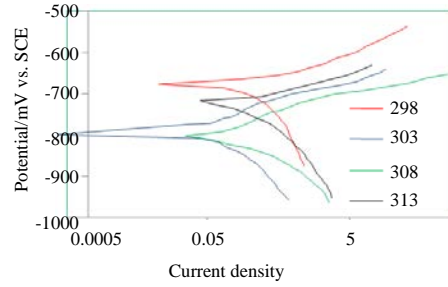


Fig. 2: Polarization curves of Al in (0.1 mol dm<sup>-3</sup>) HCl with (0.6 mol dm<sup>-3</sup>) NaCl solution

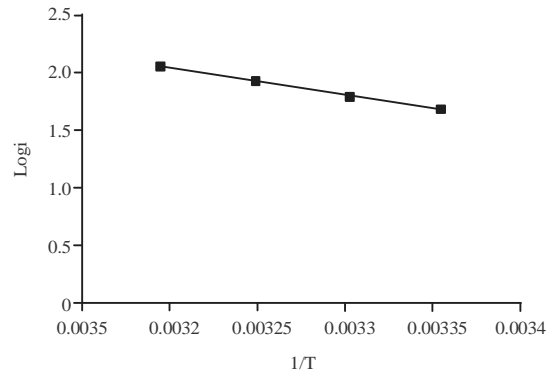


Fig. 3: Arrhenius plot relating  $\log(i_{corr})$  to  $1/T$  for Al in the pH = 1 with (0.6 mol dm<sup>-3</sup>) NaCl over the temperature range (298-313) K

$$A = \frac{kT}{h} \exp \frac{\Delta S^*}{R} \quad (7)$$

where (k) is boltzman constant, (h) is Plank constant, (R) is the universal gas constant and (T) the absolute temperature of the solution<sup>[27]</sup>.

The entropy of activation is negative, this implies that activated complex in the rate determining step represents association than dissociation indicating that a decrease in disorder takes place in going from reactants to the activated complex.

Figure 4 shows the typical polarization curves of aluminium in 0.6 mol dm<sup>-3</sup> NaCl solution containing three different concentrations of tannin as a green inhibitor over the temperature range (298-313)K. Table 4 and 5 presents the polarization data ( $E_{corr}$  and  $i_{corr}$ ) which shows that there is a decrease in corrosion current densities of aluminium due the addition of tannin.

The inhibition effect of tannin increases as the concentration of the inhibitor increases in the range (3×10<sup>-5</sup>-9×10<sup>-5</sup> mol dm<sup>-3</sup>) at all temperatures of study. The values of inhibition efficiencies (IE%) which were calculated from Eq. 4:

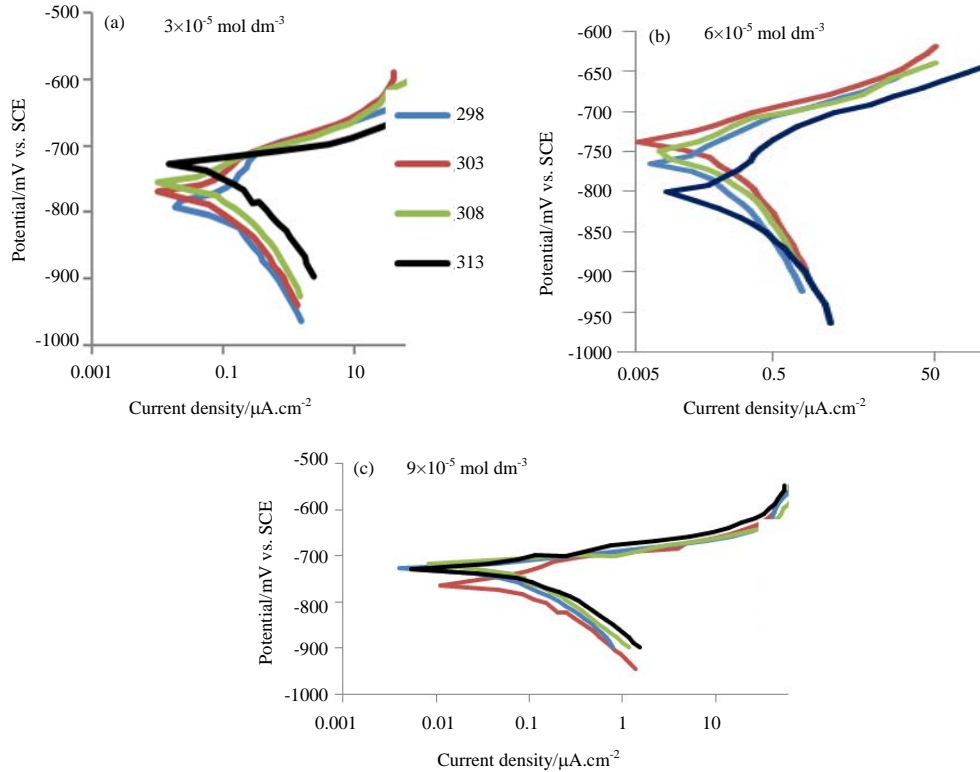


Fig. 4(a-c): The typical polarization curves of pure Al in with (0.6 mol dm<sup>-3</sup>) NaCl solution containing of Tannic acid as inhibitor (a = 3×10<sup>-5</sup>, b = 6×10<sup>-5</sup>, c = 9×10<sup>-5</sup> mol dm<sup>-3</sup>) over the temperature rang (298-313) K pH = 1

Table 1: The chemical composition of pure aluminium

Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ga	V
0.07	0.2	0.05	0.05	0.001	-	-	0.009	-	0.005

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

T/K	Corrosion		b		Weight loss/g <sup>-2</sup> .d <sup>-1</sup>	Penetration loss/mm year <sup>-1</sup>
	E <sub>corr</sub> /mV-	i <sub>corr</sub> /μA cm <sup>-2</sup>	-b/mV-decade <sup>-1</sup>	+b/mV-decade <sup>-1</sup>		
298	733.2	56.110	95.7	36.80	4.52	6.1×10 <sup>-1</sup>
303	793.8	58.490	125.8	131.9	4.71	6.36×10 <sup>-1</sup>
308	820	64.900	113.3	152.1	5.22	7.06×10 <sup>-1</sup>
313	714	166.61	155.7	24.70	1.34	1.81×10 <sup>-1</sup>

Table 3: Activation energy (E<sub>a</sub>), pre-exponential (A) and entropy of activation (ΔS<sup>‡</sup>) for pure aluminium corrosion in 0.6 mol.dm<sup>-3</sup> NaCl solution in pH = 1

E <sub>a</sub> /kJ.mol <sup>-1</sup>	ΔS <sup>‡</sup> J.K <sup>-1</sup> .mol <sup>-1</sup>	A/molecule m <sup>-2</sup> S <sup>-1</sup>
47.63	-56.77	619.07×1031

$$IE\% \equiv \frac{i_{corr}^0 - i_{corr}}{i_{corr}^0} \times 100\% \quad (8)$$

where i<sub>corr</sub><sup>0</sup> and i<sub>corr</sub> are corrosion current densities in absence and presence of the inhibitor, respectively.

**Corrosion Inhibition by tannin:** It is known that the presence of hetero atoms (O, N or S) in the structure of molecules makes them good corrosion inhibitors<sup>[12]</sup>.

The essential effect of corrosion inhibition is because of the presence of electron donor groups (O) in the molecular structure of the tannin. It is well know that the presence of heteroatoms, for (O, N or S) in the inhibitor structure results in better inhibition<sup>[12]</sup>. Also most organic substance employed as corrosion inhibitors can adsorb on the metal surface through heteroatoms, its inhibition efficiency should follow the sequence 0<N<S<sup>[28]</sup>.

Theses atoms can shared there electron pairs with metalorbitals forming insoluble complex which blocking corrosion sites and decreasing corrosion rate<sup>[29]</sup>. Due to the OH- groups in the ortho position on the aromatic rings, tannins are able to form chelates with iron and other metallic cations.

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

T/K	Inhibitor conc. mol. dm <sup>-3</sup>	-E <sub>corr</sub> /mv	i <sub>corr</sub> (μA. cm <sup>-2</sup> )
298	0	733.2	56.11
	3×10 <sup>-5</sup>	783	27.37
	6×10 <sup>-5</sup>	739.9	15.73
	9×10 <sup>-5</sup>	738	12.59
303	0	793.8	58.49
	3×10 <sup>-5</sup>	771	31.76
	6×10 <sup>-5</sup>	768.5	24.18
	9×10 <sup>-5</sup>	728.3	18.29
308	0	834	64.90
	3×10 <sup>-5</sup>	755	39.35
	6×10 <sup>-5</sup>	746.6	29.88
	9×10 <sup>-5</sup>	721	25.17
313	0	714	166.61
	3×10 <sup>-5</sup>	727	106.20
	6×10 <sup>-5</sup>	802	78.55
	9×10 <sup>-5</sup>	755	68.29

Table 5: Values of inhibitor efficiencies (IE%) calculated from  $i_{corr}$  for pure aluminium in pH = 1

Conc. of Tannic acid	T/K	IE% from $i_{corr}$
3×10 <sup>-5</sup>	298	51
	303	45.7
	308	39
	313	36
6×10 <sup>-5</sup>	298	71.9
	303	58.6
	308	54
	313	53
9×10 <sup>-5</sup>	298	77.5
	303	68
	308	61
	313	59

The activation parameters such as activation energy ( $E_a^*$ ) and the entropy of activation ( $\Delta S_a^*$ ) in the range of the studied temperatures (298-313)K for corrosion inhibition of aluminium in 0.6 mol dm<sup>-3</sup> NaCl solution in the presence of various concentrations of tannin were calculated using Arrhenius equation:

$$\text{rate} \equiv i_{corr} = A \exp\left(\frac{-E_a}{RT}\right) \quad (9)$$

Table 6 showed  $E_a^*$  and  $\Delta S_a^*$  values in presence of tannin an increase in  $E_a^*$  values were observed in presence of different concentrations of tannin the addition of tannin raise the energy barrier for the aluminium corrosion in chloride acid solution<sup>[30]</sup>.

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

The negative values of  $\Delta S_a^*$  in the existence of tannin meaning that the (r.d.s) for the activated complex is association rather than dissociation step this means the

Table 6: Activation energy ( $E_a$ ), pre-exponential factor (A) and entropy of activation ( $\Delta S^*$ ) for the corrosion of pure aluminium in the pH = 1 in 0.6 mol. dm<sup>-3</sup> NaCL solution+tannin

Conc. of Tannic acid mol. m <sup>-3</sup>	$E_a$ /kJ.mol <sup>-1</sup>	$\Delta S^*$ J.K <sup>-1</sup> .mol <sup>-1</sup>	A/molecule m <sup>-2</sup> .S <sup>-1</sup>
3×10 <sup>-5</sup>	81.7	49.5	2.4×10 <sup>39</sup>
6×10 <sup>-5</sup>	83.5	60	3.7×10 <sup>39</sup>
9×10 <sup>-5</sup>	86.9	62.5	0.12×10 <sup>39</sup>

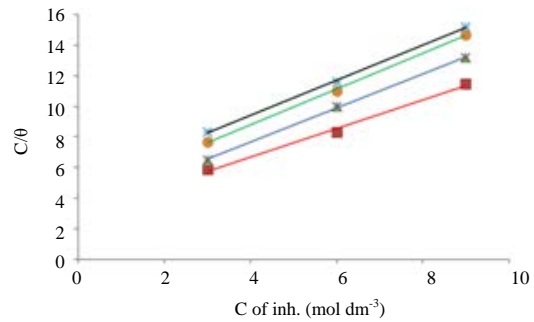


Fig. 5: Langmuir adsorption plots of Tannic acid on the pure Al in 0.6 mol dm<sup>-3</sup> NaCl solution at various temp

activated molecules are in higher order than the initial state in addition tannin molecules adsorption is accompanied by an increase in entropy which is the driving force for the adsorption process on to the aluminium surface.

**Thermodynamic parameters of the adsorption isotherm:** Since, the process of inhibition is mainly based on adsorption of inhibitor molecules on the metal surface it is necessary to study the adsorption type and adsorption isotherm. The adsorption isotherm fitted the experimental data for adsorption of tannin molecules on aluminium surface is due to langmuir as shown in Fig. 5 which is given by the following equation<sup>[31]</sup>:

$$\frac{C_{tan.}}{\theta} = \frac{1}{k_{ad}} + C_{tan.} \quad (10)$$

where,  $k_{ads}$  is the equilibrium constant for the adsorption and  $C_{tan.}$  tannin concentration.  $k_{ads}$  can be calculated from the intercepts of the straight lines on  $C_{tan.}/\theta$  the axis Fig. 5  $k_{ads}$  relate to the standard free energy of adsorption  $\Delta G_{ads}^0$  according to the equation:

$$\Delta G_{ads}^0 = -RT \ln(55.5 k_{ads}) \quad (11)$$

where, 55.5 is concentration of water in solution. The standard free energy of adsorption were calculated and are given in Table 7 and 8. Generally the standard free energy of adsorption values -20 kJ mol<sup>-1</sup> or less negative are associated with an electrostatic interaction between

Table 7: Corrosion parameter, surface coverage and corrosion inhibition efficiency for pure aluminium in 0.6 mol.dm<sup>-3</sup> NaCl solution in pH = 1 with different concentrations of Tannin at various temperature

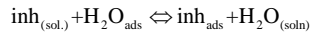
T/K	Conc. Tan., mol.dm <sup>-3</sup>	(-E <sub>corr</sub> )/mv	i <sub>corr</sub> /(μA.cm <sup>-2</sup> )	θ	IE%
298	0	733.2	56.11	-	0
	3×10 <sup>-5</sup>	783	27.37	0.51	51
	6×10 <sup>-5</sup>	739.9	15.73	0.719	71.9
	9×10 <sup>-5</sup>	738	12.59	0.775	77.5
303	0	793.8	58.49	-	0
	3×10 <sup>-5</sup>	771	31.76	0.457	45.7
	6×10 <sup>-5</sup>	768.5	24.18	0.586	58.6
	9×10 <sup>-5</sup>	728.3	18.29	0.68	68
308	0	834	64.90	-	0
	3×10 <sup>-5</sup>	755	39.35	0.39	39
	6×10 <sup>-5</sup>	746.6	29.88	0.54	54
	9×10 <sup>-5</sup>	721	25.17	0.61	61
313	0	714	166.61	-	0
	3×10 <sup>-5</sup>	727	106.20	0.36	36
	6×10 <sup>-5</sup>	802	78.55	0.53	53
	9×10 <sup>-5</sup>	755	68.29	0.59	59

Table 8: Thermodynamic parameters for adsorption of tannin on aluminium in 0.1 Hcl+NaCl

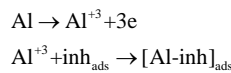
T/K	k <sub>ads</sub> mol <sup>-1</sup>	ΔG <sup>0</sup> <sub>ads</sub> kJ.mol <sup>-1</sup>	ΔH <sup>0</sup> <sub>ads</sub> kJ.mol <sup>-1</sup>	ΔS <sup>0</sup> <sub>ads</sub> J.mol <sup>-1</sup> .K <sup>-1</sup>
298	0.340	7.278	-28.144	0.0700
303	0.313	7.188		0.0691
308	0.243	6.659		0.0698
313	0.2066	6.347		0.0696

charged metal surface or more negative involves charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate covalent bond. So, it can be concluded that adsorption of tannin on to aluminium surface takes place through physical adsorption<sup>[31, 32]</sup>.

From Table 4 it was clearly seen that tannin decrease the corrosion rate of aluminium in hydrochloric acid solution E<sub>corr</sub> shifts to little more negative values in the presence of tannin tested-as shown in Table 4. This result indicates that the inhibitors have been adsorbed to both cathodic and anodic areas. That means tannin is mixed type inhibitor. The same results have been reported by other authors<sup>[20, 21]</sup>. It is known that the first step in the adsorption of tannin molecules is to replace one or more molecules of water that are adsorbed on the metal surface<sup>[22]</sup>.



The inhibitor tannin may then combine with freshly generated Al<sup>+3</sup> ions on the aluminium surface, forming metal inhibitor complex<sup>[23]</sup>:



From the values of thermodynamic parameters for the adsorption can be obtained with information on the inhibition mechanism ΔG<sup>0</sup><sub>ads</sub> were related to ΔH<sup>0</sup><sub>ads</sub> and ΔS<sup>0</sup><sub>ads</sub> according to Lundvall *et al.*<sup>[8]</sup>:

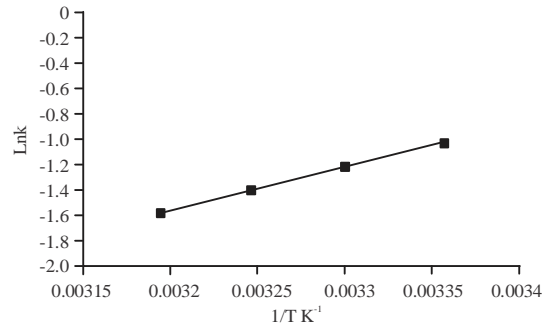


Fig. 6: Van't Hoff plot for Al in containing tanninat pH = 1

$$\Delta G_{\text{ads}}^0 = \Delta H_{\text{ads}}^0 - T\Delta S_{\text{ads}}^0 \quad (12)$$

On basis of Van't Hoff relation, the values of standard enthalpy of adsorption can be calculated:

$$\ln K_{\text{ads}} = -\frac{\Delta H_{\text{ads}}^0}{RT} + \text{constant} \quad (13)$$

The slope of straight line of the plot of lnk<sub>ads</sub> vs.1/T is -ΔH<sup>0</sup><sub>ads</sub> as show in Fig. 6. Generally, endothermic adsorption process is suggests chemisorption while exothermic process attributed to either physic-sorption or chemisorption. Negative sign of ΔH<sup>0</sup><sub>ads</sub> shows that adsorption of tannin molecules is exothermic process<sup>[16]</sup>. ΔS<sup>0</sup><sub>ads</sub> of inhibitor can be compute from Eq. 12 according to Lundvall *et al.*<sup>[8]</sup>:

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

The values of ΔS<sup>0</sup><sub>ads</sub> in the existance of tannin molecules are negative that is accompanied with exothermic adsorption process. This is compatible with the expected, that exothermic process must be accompanied by a decrease of entropy change.

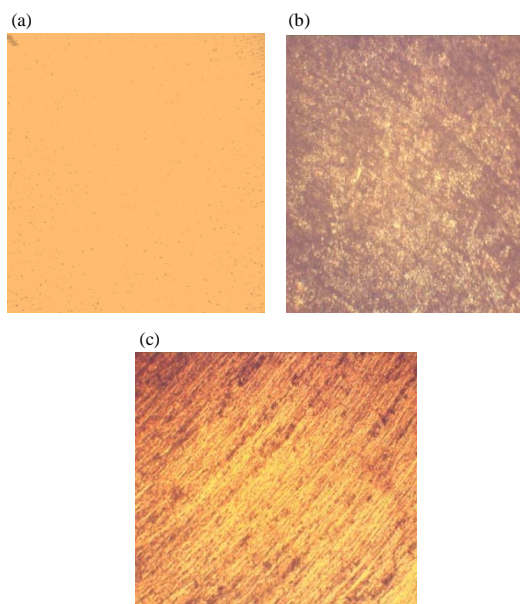


Fig. 7(a-c): Typical microstructure of pure Aluminium in the corroded medium at pH = 1, magnification power 400, (a) Pure Al, (b) Immersed in (HCl+NaCl)

**Optical microscopy observation:** To study the effect of tannin on the surface morphology of aluminium, surface out examined by optical microscope technique. Figure 7a polished aluminium surface, Fig. 7b aluminium surface immersed in (HCl+NaCl the specimen surface was strongly damaged) and Fig. 7c) in presence of tannin  $9 \times 10^{-5} \text{ mol dm}^{-3}$  shows that there was much less damage on the surface compared to the surface treated with uninhibited acid chloride solution.

### CONCLUSION

Results show that tannin is a good inhibitor for corrosion of aluminium 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 tannin on aluminium surface obeys Langmuir isotherm.

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