Glutamic Acid as Corrosion Inhibitor for Aluminium in Acidic Medium

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Abstract

Corrosion behavior of aluminium in 0.6 mol. dm^{-3} NaCL solution in acidic medium ($pH = 0.7$) was investigated in the absence and presence of different concentrations of amino acid, glutamic acid, 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 ($\Delta G_{ads.}$) and activation energy (E_a)were calculated to elaborate the mechanism of corrosion inhibition. The polarization measurements indicated that glutamic acid is of mixed type. The surface characteristic of the inhibited and uninhibited metal samples were investigated by optical microscopy.

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Introduction

Aluminium and its alloys have a remarkable , economic and attractive materials for engineering applications due to its low density, high thermal and electrical conductivity, attractive appearance and relatively good corrosion resistance. Alumininum is actually a very active metal, meaning that its nature to oxidize is very quickly, while a weakness for most metals; this quality is in fact the key to its ability to resist corrosion. The combination of these properties makes it a preferred choice for many industrial applications such as automobiles, aviation, aerospace, food handling, containers, electronic devices, buildings, etc [1].

Corrosion is the deterioration of metal by chemical attack or reaction with its environment which is often difficult to eliminate completely. Prevention would be more practical and achievable than complete elimination [2].

Corrosion of metal is caused by electrochemical reaction between a metal (or an alloy) and aqueous phase. It proceeds according to a complex electrochemical process that is related to the atomic structure of matter. The corrosion of metal is the result of two simultaneous reactions that are in electrical equilibrium, i.e. oxidation of metal to ions and reduction of hydrogen to hydrogen gas [3].

Hydrochloric acid and sulphuric acid solutions are used for pickling of aluminium or for its chemical or electrochemical etching. It is very important to add corrosion inhibitors to decrease the rate of metal dissolution using organic compounds. It is known that the organic compounds are effective corrosion inhibitors due to their ability to form an adsorbed protective film at the metal surface [3,4].

Unfortunately, most of the inhibitors available in the markets are not only expensive but also toxic to living beings. Again, the safety of environmental issues of corrosion inhibitors which may arise industry has always been a global concern [5].

To avoid toxicity of numerous compounds tested as inhibitors [5,6], natural compounds, regarded as environmentally friendly inhibitors. Amino acids have been tested as inhibitors of corrosion of a variety of metals, for example copper, aluminium and steel corrosion in acid media. They are relatively easy to produce with high purity at low cost, non-toxic and soluble in aqueous media [6,7].

In the present work, the inhibitive effectiveness of amino acid (glutamic acid), have been studied in retarding corrosion of aluminium in 0.2M H_2SO_4 by using of electrochemical polarization measurements. Furthermore, this study aims to examine the role of the functional groups of the inhibitors which are used in the adsorption process as well as the influence of the nature of the medium on the corrosion mechanism.

Optical microscopy was employed to observe the surface morphology of the aluminium corroded in test solution.
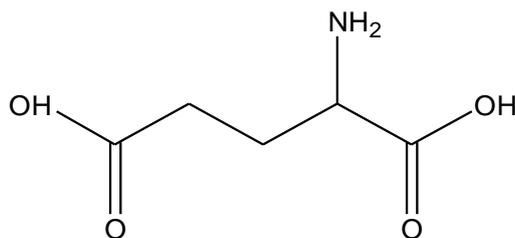
2. Experimental Part

2.1 Materials

The experiments were performed with aluminium sheets (99.8% purity) of 2mm thickness which was mechanically press-cut into circular form 2cm diameter.

All chemicals and reagents used are with analytical grade and use without further purification, they include:

a. Sodium chloride, was used for preparation of the aggressive solution of $0.6 mol\ dm^{-3}$.

b. Sulphuric acid.

c. The amino acid (L-Glutamic Acid) tested as inhibitor (>99% purity).Figure below shows the structure of amino acid which used.

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$$NH_2$$

OH ⟍ ⟋ OH

O   O

2.2. Solution

The aggressive solution used was made of A.R. Sodium chloride to prepare $0.6 mol\, dm^{-3}$ concentration in $pH = 0.7$. Doubled distilled water was used for the preparation. For each experiment a freshly solution was made. The concentrations of the inhibitor (glutamic acid) were used $(10^{-3}, 5 \times 10^{-3}\ and\ 10^{-2}) mol.\, dm^{-3}$.

2.3 Potentiostatic Polarization Measurements:

The polentiostatic polarization measurements were carried out with aluminium sheets (explained before) having an exposed surface area of $1 cm^2$ to corrosive medium [in absence and presence of 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.

Polarization measurements were performed in a three electrodes cell with aluminium specimen of $1 cm^2$ 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 M lab potentionstat / Galvanostat 200 Germany obtained from Bank Electronic Intelligent Controls Gnb H. It was connected to personal computer desktop the M lab software cares for controlling the potentiostat, recording and processing data. It provides electrochemical calculations like Tafel line evalution, re-scalling the potential and integrating.

The experiments were preformed in $0.6 mol\, dm^{-3}\ \ NaCL$ solution of $pH = 0.7$ in absence and presence of three different concentrations of the inhibitor (glutamic acid) over the temperature range (293-308) K. The experiments were carried out at a scan rate of $2mV/s$.

The extrapolating of straight line allows to determination of corrosion current density (i_{corr}) and corrosion potential (E_{corr}) , 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.
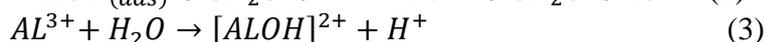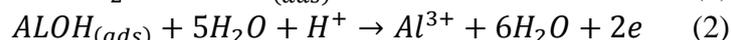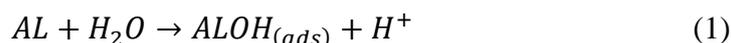
3. Results and Discussion

3.1 Tafel polarization measurements

The effect of 0.6 mol. dm^{-3} NaCl solution in acidic medium on the corrosion of aluminium sample was studied using Tafel polarization technique. Figure (1) represents the potentiostatic polarization curves of aluminium at four temperatures in range of (293-308)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 which listed in Table (2).
The results indicate the increase in the corrosion current density (i_{corr}) with an increase in temperature.

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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, these results are in agreement with many litretures[1,2] which proposed the dependence of (i_{corr}) and (E_{corr}) on solution parameters. The change in (b_a) and (b_c) reflects the mechanism of corrosion process.

The electrochemical cathodic and anodic reactions of aluminium in acidic solution can be described as reported in many references [8].

$$AL + H_2O \rightarrow ALOH_{(ads)} + H^+ \qquad\qquad (1)$$
$$ALOH_{(ads)} + 5H_2O + H^+ \rightarrow Al^{3+} + 6H_2O + 2e \quad (2)$$
$$AL^{3+} + H_2O \rightarrow [ALOH]^{2+} + H^+ \qquad\qquad (3)$$
$$[ALOH]^{2+} + X^- \rightarrow [ALOHX]^+ \qquad\qquad (4)$$

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

3.2 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. The dependence of the corrosion current density (i_{corr}) on temperature followed Arrhenius equation[9].

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

which can be expressed in logarithmic form:

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

Where A and E_a are repectively the pre-exponential factor and the activation energy of corrosion reaction.

Ploting values of $\ln i_{corr}$ to the reciprocal of temperature $\left(\frac{1}{T}\right)$ was shown in Figure (2).

The values of E_a could be derived from the slope of the line, and when the linear plot of Figure (2) was extrapolated to $\ln i_{corr}$ value at $\left(\frac{1}{T}\right) = 0$, 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 $(pH = 0.7)$.

Entropy of activation (ΔS^*) was calculated from the value of (A) using the relationship:

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

Where (k) is boltzman constant, (h) is Plank constant, (R) is the universal gas constant and (T) the asbsolute temperature respectively[10,11].

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 (3) shows the typical polarization curves of aluminium in $0.6 mol\ dm^{-3}\ NaCl$ solution containing three different concentrations of amino acid (glutamic acid) as a green inhibitor over the temperature range (293-308)K. Table (4) showes $(E_{cor}\ and\ i_{corr})$ which showes that

the addition of glutamic acid caused a decrease in corrosion current densities of aluminium, the inhibition effect of glutamic acid increases as the concentration of the inhibitor increases in the range $(10^{-2} - 10^{-3})mol\ dm^{-3}$ at all temperatures of study.

Table (5) shows the values of inhibition efficiencies ($IE\%$) which were calculated from equation(4):

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

Where i_{corr}^0 and i_{corr} are corrosion current densities in absence and presence of the inhibitor respectively.

3.3 Corrosion Inhibition of aluminium by Glutamic Acid:

The essential effect of corrosion inhibition is because of the presence of electron donor groups such (N,O) in the molecular structure of the glutamic acid. It is well known that the presence of heteroatoms, for nitrogen and oxygen in the inhibitor structure results in better inhibition[12]. Also the most organic substance employed as corrosion inhibitors can be adsorbed on the metal surface through heteroatoms, Its inhibition efficiency should follow the sequence $O < N < S$ [13] . 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 [14]. The presence of pairs of free electrons on nitrogen and oxygen atoms favors adsorption of glutamic acid and, thus , the best efficiency of this inhibitor.

The activation parameters such as activation energy (E_a^*) and the entropy of activation (ΔS_a^*) in the range (293-308)K for corrosion inhibition of aluminium in $0.6 mol\ dm^{-3}\ NaCl$ solution within various concentrations of glutamic acid were calculated using Arrhenius equation:

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

Table (6) shows values of E_a^* and ΔS_a^* which increased in the presence of the inhibitor, an increase in E_a^* values were observed, the variation of activation energy E_a^* in the presence of different concentrations of glutamic acid can be illustrated as follows[15]: higher values of (E_a^*) were found in presence of the inhibitor than those without inhibitor, i.e., the addition of glutamic acid raise the energy barrier for the corrosion process of aluminium in chloride acid solution[16].

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 the metal.

The entropy values of activation, ΔS_a^*, in the presence of glutamic acid are negative implying the rate determining step for the activated complex is the association rather than dissociation step, in addition the adsorption process is accompanied by an increase in entropy which is the driving force for the adsorption of glutamic acid on to the aluminium surface[17].

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3.4 Thermodynamic parameters of the adsorption isotherm

The efficiency of organic compounds as good corrosion inhibitors depends mainly on their adsorption ability on the metal surface. Basic information about the interaction between inhibitor and metal can be provided by the adsorption isotherm. The investigation of the relation between corrosion inhibition and adsorption of inhibitor is of great importance.

The surface coverage Table (7) and the concentration of glutamic acid solution (c_{meth}) were tested by fitting to various isotherms like: Langmuir, Tempkin and freundlich. However, the fit was obtaind with Langmuir isotherm as shown in Figure (4) which is given by the following equation[18].

$$\frac{C_{glu}}{\theta} = \frac{1}{K_{ad}} + C_{glu} \qquad \cdots (10)$$

Where K_{ads} is the equilibrium constant of the adsorption / desorption process, and it reflects the affinity of the inhibitor molecules towards surface adsorption sites.

From the intercepts of the straight lines on the C_{met}/θ axis Figure (4) leads to the equilibrium constant for the adsorption / desorption of glutamic acid process. Table (8). The high value of K_{ads} reveals that the glutamic acid molecule possesses strong adsorption ability on to the aluminium.

However, k_{ads} decreased with an increase of temperature indicating that adsorption of glutamic acid on to the metal surface was favorable at lower temperatures.

The equilibrium constant of the adsorption / desorption (k_{ads}) was related to the standard free energy of adsorption according to the equation.

$$\Delta G_{ads}^0 = -RTln(55.5 \, k_{ads}) \qquad \cdots (11)$$

Where R is the universal gas constant, T is the absolute temperature, and 55.5 is concentration of water in solution.

The standard free energy of adsorption were calculated and are given in table (8). Generally the standard free energy of adsorption values $-25 kJ.mol^{-1}$ or less negative is associated with an electrostatic interaction between 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 glutamic acid on to aluminium surface takes place through both physical and chemical adsorption[19].
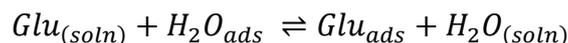
From Table (4) it was clearly seen that the presence of glutamic acid reduces the corrosion rate of aluminium in sulphuric acid solution. As its well known that different substituents on the organic molecules polarize the functional group in a different manner[11]. This indicates that the protective effect of glutamic acid is not solely due to their reactivity with sulphuric acid in acidic media, NH_2 of amino acid molecule is readily protonated to form NH_3^+. Also the addition of amino acids with aliphatic radical R leads to decrease corrosion current density but when the radical contains heteroatom as N,O or S a decrease of corrosion current densities in the presence of glutamic acid at $10^{-3}, 5 \times 10^{-3}$ and $10^{-2} mol.dm^{-3}$ was noted.

E_{corr} shifts to little more negative values in the presence of glutamic acid tested-as shown in Table (7).
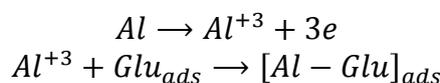
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This result indicates that the inhibitors have been adsorbed to both cathodic and anodic areas. That means glutamic acid is mixed type inhibitor. The same results have been reported by other authers[20,21].

Then the addition of amino acids molecules in sulphuric acid solution seems that no effect on anodic behavior is observed. This result shows that the inhibitory action of these compounds depends on the potential for higher E_{corr} potential inhibitory character disappears.

It is generally accepted that the first step in the adsorption of glutamic acid on the metal surface usually involves the replacement of one or more water molecules adsorbed at the metal surface[22].

$$Glu_{(soln)} + H_2O_{ads} \rightleftharpoons Glu_{ads} + H_2O_{(soln)}$$

The inhibitor glutamic acid may then combine with freshly generated Al^{+3} ions on the aluminium surface, forming metal inhibitor complex[23]

$$Al \longrightarrow Al^{+3} + 3e$$
$$Al^{+3} + Glu_{ads} \longrightarrow [Al - Glu]_{ads}$$

Valuable information about the mechanism of corrosion inhibition can be provided by the values of thermodynamic parameters for the adsorption of inhibitor. Thermodynamically, ΔG^0_{ads} were related to the standard enthalpy, ΔH^0_{ads} and standard entropy, ΔS^0_{ads} according to[8].

$$\Delta G^0_{ads} = \Delta H^0_{ads} - T\Delta S^0_{ads} \qquad \dots (8)$$

And the standard enthalpy of adsorption can be calculated on basis of the Van't Hoff formula:

$$\ln K_{ads} = -\frac{\Delta H^0_{ads}}{RT} + constant \qquad \dots (9)$$

A plot of $\ln k_{ads} \ vs. \ 1/T$ gives a straight line as shown in Figure (5). The slope of straight line is $-\frac{\Delta H^0_{ads}}{R}$.

The negative sign of ΔH^0_{ads} reveals that the adsorption of inhibitor molecules is an exothermic process. Generally, an exothermic adsorption process suggests either physisorption or chemisorption while endothermic process is attributed to chemisorption. The unshared electron pairs in investigated molecules may interact with P-orbitals of aluminium to provide a protective chemisorbed film[16].

The values of ΔS^0_{ads} in the presence of inhibitor are negative that is accompanied with exothermic adsorption process. ΔS^0_{ads} of inhibitor can be calculated from equation (8) according to[8].

$$\Delta S^0_{ads} = \frac{\Delta H^0_{ads} - \Delta G^0_{ads}}{T} \qquad \dots\dots\dots(10)$$

3.5 Optical Microscopy observation

The surface morphology of Aluminium specimens were characterized by i optical microscope technique. Figure (6a) polished , (6b) immersed in (H_2SO_4 + $NaCl$) and (6c) immersed in H_2SO_4 + NaCl in presence of inhibitor (glu) respectively. It can be observed that the specimen surface was pithely damaged in absence of glutamic acid owing to metal

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dissolution in the aggressive media (6b) , applying glutamic acid brings down corrosion wherein sample surface is covered with (glu) molecules showing less damage.

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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 $0.6 mol.dm^{-3}$ $NaCl$ solution at pH(0.7) over the temperature range (293-308)K.

T/K	$i_{corr}/\mu A.cm^{-2}$	$-E_{corr}/mv$	$-bc/mv$ decade^{-1}	ba/mv decade^{-1}	Weight loss/ $g.m^{-2}.day^{-1}$	Penetration loss/mm.year^{-1}
293	129	634	528.3	47.1	10.3	1.4
298	163	678	777.6	44.2	13.1	1.78
303	203	696	303.1	44.5	16.3	2.21
308	326	739	195.9	64.7	26.2	3.55

Table (3): Activation energy (E_a), pre-exponential (A) and entropy of activation (ΔS^*) for pure aluminium corrosion in $0.6 mol.dm^{-3}$ $NaCl$ solution in pH0.7.

$E_a/kJ.mol^{-1}$	$-\Delta S^* J.K^{-1}.mol^{-1}$	A/molecule $m^{-2}.S^{-1}$
45.6	49.4	1513×10^{31}

Table (4): Values of (E_{corr}, i_{corr}) with different concentrations of glutamic acid at temperature range (293-308) K in pH0.7

T/K	Inhibitor $conc.$ $mol.dm^{-3}$	$-E_{corr}/mv$	$i_{corr}/\mu A.cm^{-2}$
293	0	634	129
	1×10^{-3}	630	40
	5×10^{-3}	635	39
	1×10^{-2}	630	37
298	0	678	163
	1×10^{-3}	641	58
	5×10^{-3}	642	56
	1×10^{-2}	633	50
303	0	696	203
	1×10^{-3}	646	80
	5×10^{-3}	620	77
	1×10^{-2}	660	69
308	0	739	326
	1×10^{-3}	662	133
	5×10^{-3}	660	128
	1×10^{-2}	680	114

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Table (5): Values of inhibitor efficiencies (IE%) calculated from i_{corr}. For pure aluminium in pH0.7

Conc. of glutamic acid	T/K	IE% from i_{corr}.
1×10^{-3}	293	69
	298	64.5
	303	60.6
	308	59.5
5×10^{-3}	293	69.8
	298	65.7
	303	62.1
	308	60.8
1×10^{-2}	293	71.4
	298	69.4
	303	66.1
	308	65.1

Table (6): Activation energy (E_a), pre-exponential factor (A) and entropy of activation (ΔS^*) for the corrosion of pure aluminium in the pH value 0.7 in $0.6 mol.dm^{-3} NaCL$ solution and different concentrations of L-glutamic acid.

Conc.of L-glutamic acid $mol.dm^{-3}$	$E_a/kJ.mol^{-1}$	$-\Delta S^* J.K^{-1}.mol^{-1}$	A/molecule $m^{-2}.S^{-1}$
1×10^{-3}	29	95	675×10^{28}
5×10^{-3}	31.5	85.5	17370×10^{28}
1×10^{-2}	32.3	84.6	21370×10^{28}

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Table (7): Chosen corrosion parameter, degree of surface coverage and corrosion inhibition efficiency for pure aluminium in $0.6 mol.dm^{-3} NaCl$ solution in pH $= 0.7$ with different concentrations of glutamic acid at various temperature.

T/K	$conc.$ glu. $mol.dm^{-3}$	$-E_{corr}/mv$	$i_{corr}/\mu A.cm^{-2}$	θ	IE%
293	0	634	129	-	
	1×10^{-3}	630	40	0.69	69
	5×10^{-3}	635	39	0.698	69.8
	1×10^{-2}	630	37	0.714	71.4
298	0	678	163	-	
	1×10^{-3}	641	58	0.645	64.5
	5×10^{-3}	642	56	0.657	65.7
	1×10^{-2}	633	50	0.694	69.4
303	0	696	203	-	
	1×10^{-3}	646	80	0.606	60.6
	5×10^{-3}	620	77	0.621	62.1
	1×10^{-2}	660	69	0.661	66.1
308	0	739	326	-	-
	1×10^{-3}	662	133	0.595	59.5
	5×10^{-3}	660	128	0.608	60.8
	1×10^{-2}	680	114	0.651	65.1

Table (8): Equilibrium constant adsorption / desorption, standard free energy, enthalpy and entropy of adsorption onto pure aluminium surface in $0.6 mol.dm^{-3} NaCl$ solution in pH $= 0.7$ in the presence of glutamic acid at various concentrations.

T/K	$k_{ads} mol^{-1}$	$-\Delta G^0_{ads} kJ.mol^{-1}$	$\Delta H^0_{ads} kJ.mol^{-1}$	$\Delta S^0_{ads} J.mol^{-1}.K^{-1}$
293	5000	30.48		256
298	3333	30.00	44.47	250
303	2500	29.77		245.9
308	2000	29.69		241

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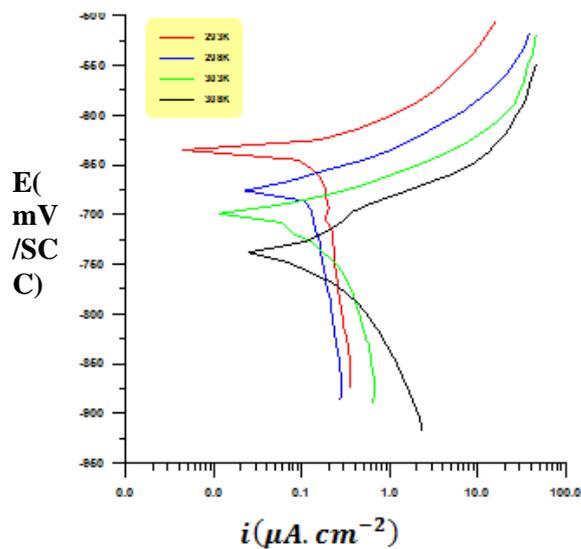
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Figure (1): Polarization curves of pure Al at temperatures range of (293-308)K.

Figure (2): Ploting Log (i_{corr}) vs 1/T for the corrosion of pure Al in the pH = 0.7 in presence of (0.6 mol.dm^{-3}) NaCl over the temperature range (293 – 308) K.
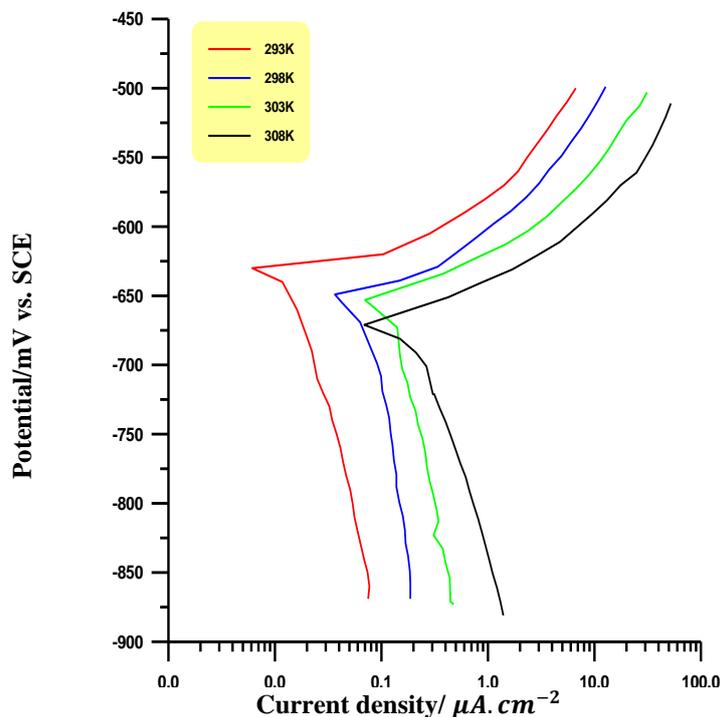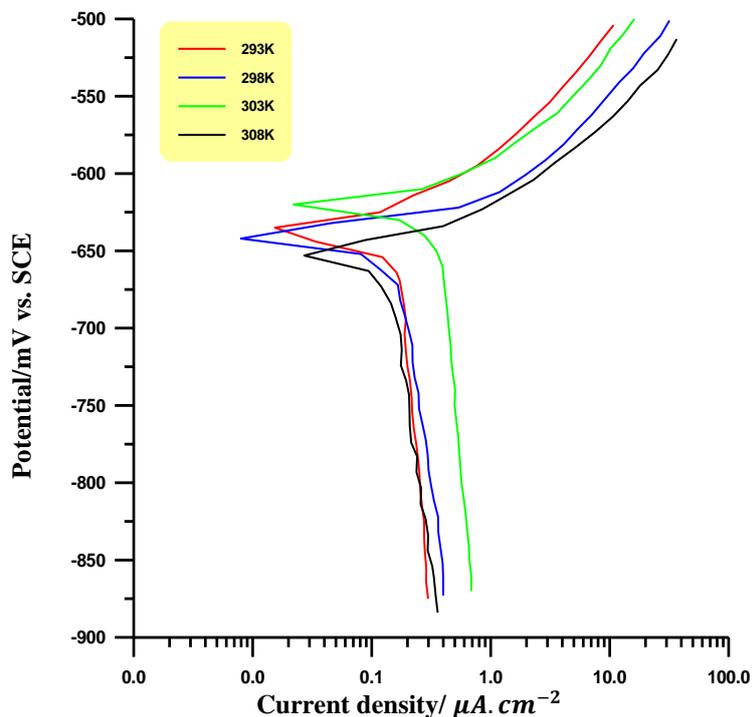
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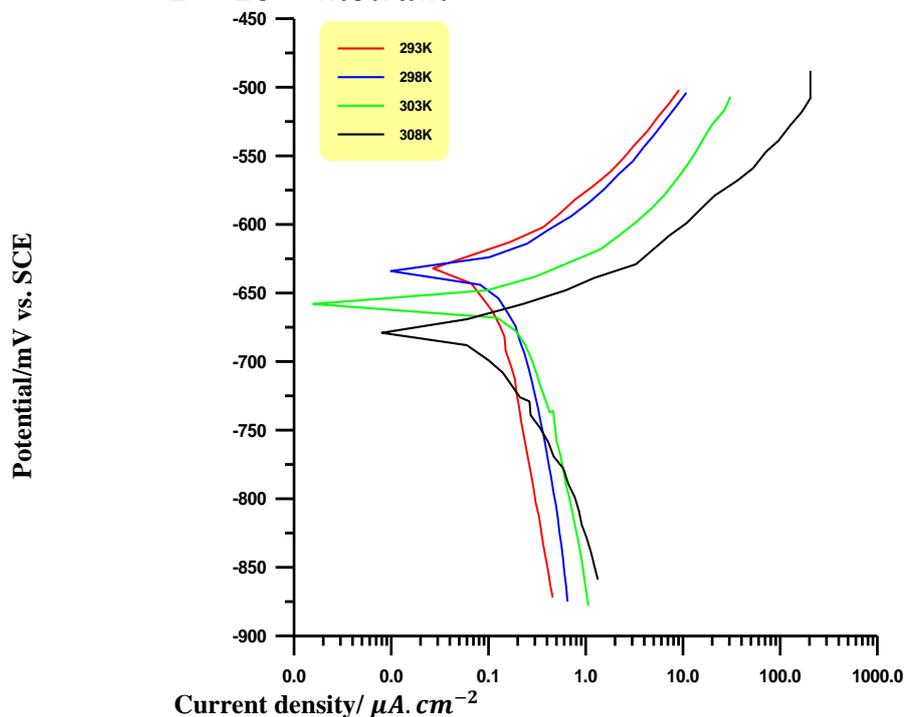
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Figure (3): The typical polarization curves of pure Al with (0.6 mol.dm^{-3}) NaCl solution containing of L-glutamic acid as inhibitor (a= 1×10^{-3}, b = 5×10^{-3}, c =1×10^{-2} mol.dm^{-3}) over the temperature range (293-308)K.

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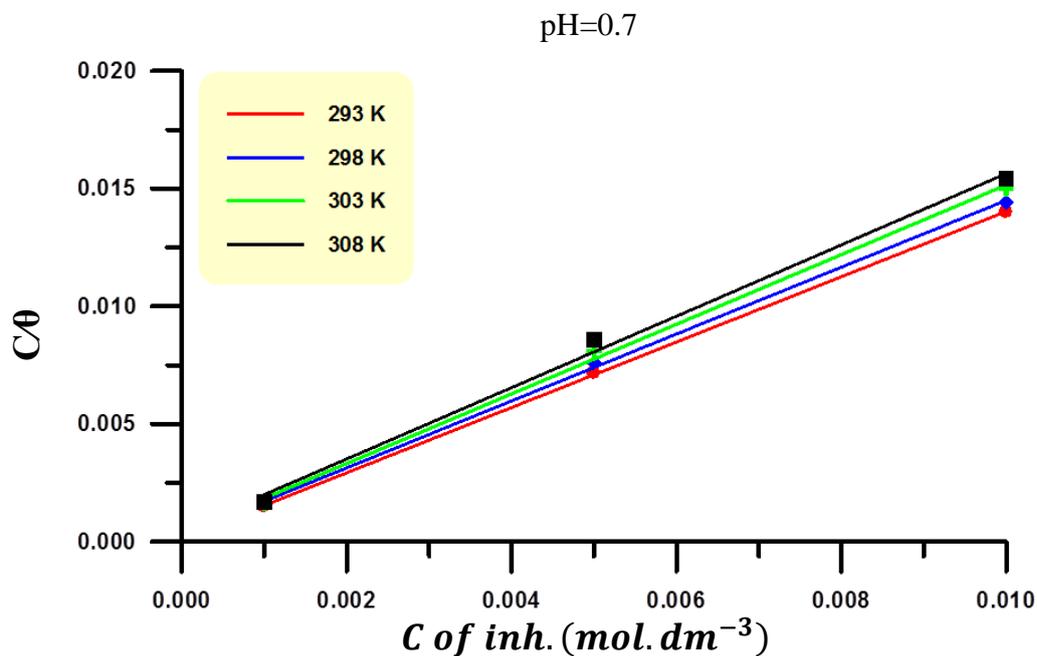
pH=0.7

Figure (4): Langmire adsorption plots of L-glutamic acid on the pure Al in 0.6 mol.dm⁻³ NaCl solution at various temperatures.

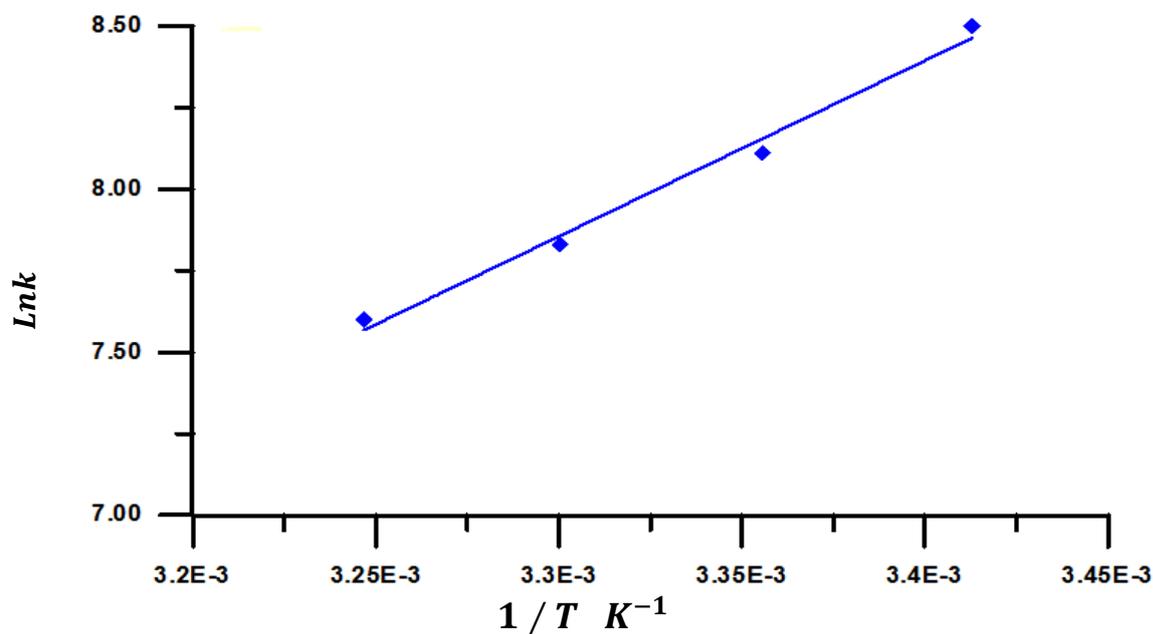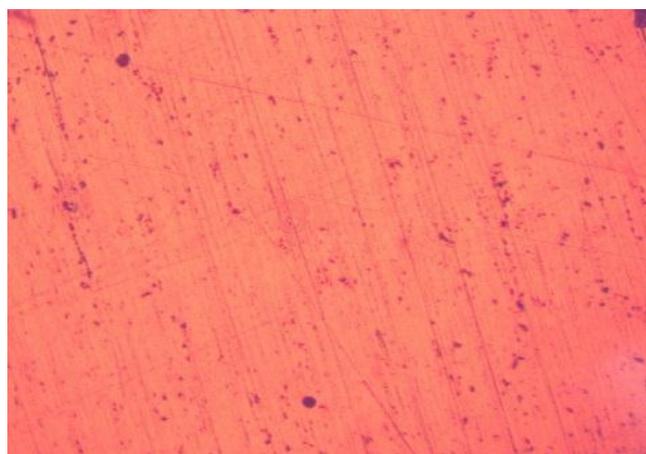**Figure (5): Van't Hoff plot for pure Al in $(0.6\ mol.dm^{-3})$ NaCl solution containing L-Glutamic acid at pH value (0.7)**

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a.polished pure Al

b.after immersion in corrosive medium

c.after immersion in the corrosive medium + L-Glutamic acid

Figure (6): Typical microstructure of pure aluminium in the corroded medium at $pH = 0.7$, magnification power 400×