

Aloe Plant Extract as Environmentally Friendly Inhibitor on the Corrosion of Aluminum in Hydrochloric Acid in Absence and Presence of Iodide Ions

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Abstract

The study shows that the corrosion of Aluminum increases with increasing HCl concentration which indicates the dependence of Aluminum corrosion on the concentration of HCl. The inhibition efficiency of the corrosion by *Aloe* extract was found to increase with the increase of *Aloe* extract concentration. Its inhibitive action from chemical (hydrogen evolution and mass loss) and electrochemical (polarization and impedance) was in good agreement. Also, it was found that *Aloe* plant act as mixed type inhibitor. The corrosion of Aluminum is mainly controlled by a charge transfer process. The inhibition action occurred through the adsorption of the extract on Aluminum surface, the adsorption process follows Freundlich adsorption isotherm. The addition of iodide ions enhances the inhibition efficiency for *Aloe* plant extract.

Keywords: Corrosion, Corrosion inhibitor, Aluminum, Acid corrosion, *Aloe* plant inhibitor

1. Introduction:

Many important applications of Aluminum and its alloys have resulted in researches due to its electrochemical behavior and corrosion resistance in a wide variety of media, including investigation of the properties of the surface oxide film formed naturally or by iodization (G. Warnglen, 1985). Aluminum has high resistance to corrosion in many environments; this is attributed to a protective surface film formed rapidly in air or in neutral aqueous solutions, which is preserved even after long periods of immersion in acidic and alkaline media (M. Hurovic, R. Babic, Z. Grubac and S. Brinic, 1994), and in nearly neutral aqueous solutions (C. Brett, I. Gomes and J. Marhins, 1994).

Hydrochloric acid solutions are normally used for pickling of Aluminum and for its chemical and electrochemical etching to corrosion (E. E. Oquzic, G. N. Onuoha and E. N. Ejibe, 2007).

Efforts have been made to ameliorate the Aluminum surface film dissolution and hence protect the integrity of the metal in aggressive acid medium. One of the most important methods is the use of inhibitors to hinder the corrosion reaction and thus reduce the corrosion rate.

Investigations have been made recently into the corrosion inhibiting properties of natural products of plant origin, which was found to exhibit good inhibition efficiencies (F. Zucchi, I. H. Omar, 1985)(M. Kliskic, J. Radošević, S. Gudic and V. Katalinik, 2000)(A. Y. El-Eter, 2003)(M. Abdallah, 2004)(E. E. Oquzic, 2006). The use of natural products for metals protection is of much importance because in addition to being inexpensive, readily available and renewable sources of materials, plant products are environmentally friendly and ecologically acceptable.

Aloe plant extract is organic in nature and can be used in the production of green inhibitor and it is one of natural inhibitors which has an inhibitive action on the corrosion of metals. *Aloe Vera* gel is the colorless mucilaginous gel obtained from the parenchymatous cell in the fresh leaves of *Aloe Vera*. It contains various active

compounds such as: salicylates, magnesium lactate, acemannan, lupeol, campesterol, sterol, linolenic, aloctin and anthraquinones (T. Blitzke, A. Porzel, M. Masaoud and J. Schmidt, 2000).

In this study, chemical and electrochemical methods are applied to study the corrosion behavior of Aluminum in hydrochloric acid. The inhibition efficiency of *Aloe* extract as natural inhibitor for the corrosion of Aluminum in 0.5M HCl and the effect of iodide ions were studied. SEM was applied for surface morphology to confirm the obtained results.

2. Experimental

2.1 Specimen

The composition of the Aluminum sample used in these experiments is given in Table (1)

2.2 Surface Pre-treatment:

Before all measurements the specimen was polished first with a series of emery paper of type (231 Qwetordry Imperial Pape aesoc), starting with a coarse one and proceeding in steps to fine grade. Then the sample was thoroughly washed with deionised water then with acetone (A.R), dried by stream of air. The sample was then immediately immersed in the test solution.

2.3 Solutions:

Hydrochloric acid and NaI (Fluka) were used. All solutions were prepared with analytical grade reagents (A.R.). Deionised water was used throughout for the preparation of solutions.

Stock solution of *Aloe* plant leaves extract was prepared by extracting 0.5 kg *Aloe* leaves in water and boiled for some time then mixed in a plinder. The solution was cooled and filtered by air bump and then the filtered solution was kept at low temperatures.

From the respective stock solutions, the inhibitor test solutions were prepared in the concentration range (4-60) %v/v. The concentration range of NaI employed was $(1.0 \times 10^{-4} - 5.0 \times 10^{-2})$ M. For the inhibition study 0.5M HCl was used.

2.4 Techniques:

2.4.1 Chemical measurements:

The chemical measurements were carried out by two methods; hydrogen evolution method (HEM) and mass loss method (MLM) using a vessel which has the same form as that described by Mylius (F. Mylius and S. Niethem, 1957). The effect of hydrochloric acid concentration at (0.25, 0.5, 0.75, 1.0, 1.25 and 1.5 M) on the corrosion of Aluminium at 30°C was studied. Also, the effect of *Aloe* extract at different concentrations was studied in absence and presence of I^{-1} at 0.5M HCl solutions.

2.4.2 Electrochemical measurements:

Two kinds of measurements; potentiodynamic polarization (PDP) and impedance (EIS) measurements were carried out using an impedance spectrum analyzer (ACM instrument) connected to a Samsung computer (Bridgmax DVD ASUS 8X) (S. T. Arab and A. M. Al-Tukustani, 2002). Electrochemical impedance spectroscopy (EIS) measurements were conducted in deaerated HCl solutions of different concentrations at open-circuit (O.C) conditions.

2.4.3 Scanning Electron Microscopy (SEM) Analysis:

SEM analysis of the electrode surface was carried out using Scanning Electron Microscopy XL 20 PHILPS. The magnification varied from 10x up to 500x.

3. Result and discussion

3.1 Effect of the hydrochloric acid concentration at 30 °C:

a- Chemical study:

Figure (1) shows the plots of hydrogen volume with time, the increase in rate of hydrogen evolution depend on the acid concentration. As the concentration of the HCl solution increases, the slope of the straight lines also increases, indicating an increase in the dissolution of Aluminum, e.g. increases of corrosion rate. An induction period was observed especially at low concentrations of HCl due to the slow reaction between the acid and the air formed oxide film on Aluminum surface as reported before (K. Srivastava and P. Srivastava, 1981)(P. S. Khosla, J. B. Singh and R. K. Srivastava, 2000). The induction period decreases with increasing acid concentration. The presence of oxide film leads to blocking the surface of the Aluminum and prevent the solution reaching the metal surface, the oxide film causes the corrosion rate to decrease (A. A. Mazhar, S. T.

Arab and E. A. Noor, 2001). The variation in the induction period was explained to be either to the HCl concentration increase or time needed for penetration of Cl^- through the oxide film and make it destruction, then the dissolution of Aluminum will occur (E. Oquzic, 2007)(A. M. Al- Mayouf, 1996).

At high concentrations of HCl solution, the attack on oxide film by chloride ions was instantaneous and very quick, and Cl^- ions will accelerate the corrosion reaction by retarding film repair and forming intermediate soluble complex as follows (R. Ambat and E. S. Dwarakadasa, 1993):



The formed complexes facilitate the dissolution of the Aluminum ions (or atoms) from the lattice into the solution and leads to local thinning of the passive layer on the metal surface and to pitting (localized) corrosion. This illustrates that, as the immersion time increases, the hydrogen evolved by constant rate (especially at 1.0 M and 1.25 M of HCl), this may be explained as that, the formation of insoluble complex will adsorbed on Aluminum surface or due, to existence of thin oxide film formed before immersion and hindrance the solution diffusion to Aluminum surface.

The corrosion rate, R, from HEM in ($\text{ml.cm}^{-2}\text{min}^{-1}$) as well as, the corrosion rate obtained from MLM is calculated from the simple relationship $R' = dw/dt$ in ($\text{g. cm}^{-2} \text{min}^{-1}$) where dw is the mass loss and dt is the immersion time.

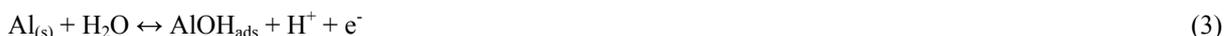
Table (2) illustrates the corrosion rate (R and R') values for Aluminum in HCl solutions from HEM and MLM, respectively. It was found that, the corrosion rate increases with the increase of HCl concentration; this indicates that Aluminum corrosion in HCl is concentration dependent. Corrosion of Aluminum in aqueous solution has been reported (A. M. Al- Mayouf, 1996)(T. H. Nguyen and R. T. Foley, 1982) to depend on the concentration of anions in solution.

Figure (2) shows the variation of $\log R$ and / or $\log R'$ with $\log C$ of HCl solutions for Aluminium at 30°C . It was obvious that, the slopes of the straight lines equal to 3.04 and 3.06, respectively, this means that the degree of the reactions between Aluminium and HCl is a third order reaction, by applying the relation:

$$\log R = \log A + n \log C \quad (2)$$

The first step in the pitting process is suggested to be, the adsorption of chloride ion (Cl^-) on the oxide covered Aluminium surface. When Cl^- interacts with an ionic surface, such as an oxide, the attractive forces consist of (E. Mc Cafferty, 2003): (i) columbic forces, (ii) induction of the adsorbent by the approaching ion, (iii) electrostatic polarization of the ion and (iv) non-polar van der Waals forces (J. H. de Boer, 1950). Of these attractive forces, the largest are the first two interactions, which are ionic.

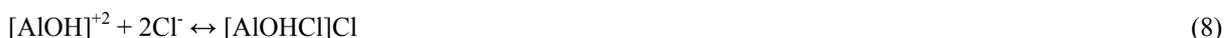
A general mechanism for the dissolution of Aluminium metal in aqueous solutions would be similar to that reported by Ford et al. (F. P. Ford, G. T. Burstein and T. P. Hoar, 1980) as well as Nguyen and Foley (T. H. Nguyen and R. T. Foley, 1982):



The controlling step in the metal dissolution is the complexation reaction between the hydrated cation and the anion present (eq. (6)). In the presence of chloride ions the reaction will correspond to:



Since the reaction obtained have third order, thus the soluble complex $[\text{AlOHCl}]\text{Cl}$ will formed as:



The soluble complex ion formed increases the metal dissolution at a rate which depends on the chloride concentration, this accounts for the observed increase in corrosion rate as the concentration of HCl is increased.

b- Electrochemical study:

Figure (3) shows the anodic and cathodic polarization curves of Aluminium in HCl solution at different concentrations. It was shown that, the increase of concentration of the acid leads to displacement the anodic and cathodic curves to high current densities and increase the corrosion rate will be found, Table (3). At higher concentrations of acid, the anodic polarization curves give small hump depending on the concentration of the acid, which is associated with the formation of an adsorbed thin layer on Aluminium surface or thin layer from

non-porous oxide film leads to minimise the passing current to Aluminium surface. Results of electrochemical parameters, E_{corr} , I_{corr} and Tafel constants (b_a & b_c) obtained from these curves are listed in Table (3).

It obvious from Fig. (3) and Table (3) that the increase in acid concentration lead to displacement in anodic and cathodic curves to high values of current density and increase in corrosion rate.

-At high HCl concentrations, e.g. (0.75, 1.0, 1.25 and 1.5 M) and high potential, a more or less passive region is detected during anodic polarization, which indicates onset of localized attack (pitting corrosion). The passive region may be attributed to existence of the air formed an oxide film on the metal surface (B. R. Baker and J. D. Balsler, 1976). These were confirmed by chemical measurements (HEM).

- The values of E_{corr} shift to more negative values with increase in HCl concentration.

Figure (4(a, b)) show the dependence of E_{corr} and I_{corr} on acid concentration. The linear relation in case of E_{corr} , Fig. (4a), can be represented by the equation (A. M. Beccaria and G. Poggi, 1986):

$$E_{\text{corr}} = E_{\text{corr}} + (2.303 \text{ RT} / n \text{ F}) \log C \quad (9)$$

Where E_{corr} is the corrosion potential when the acid concentration is equal to unity, R is the universal gas constant, T is the absolute temperature, n is the number of electrons, F is the Farad and C is the concentration, it is found that the value of $E_{\text{corr}} = -795.0 \text{ mV}$ and $n = 1.38 \times 10^{-3}$. The deviation of n from the expected value may be related to a multi-step process of oxide film protection. Figure (4b) also, illustrate the relation between I_{corr} and concentration follows the relation:

$$I_{\text{corr}} = a + b \log C \quad (10)$$

where a and b are characteristics dependent on the corroding surface.

From Fig.(4) it can be seen that, both E_{corr} and I_{corr} obey linear relation with HCl concentration with correlation constants equal to 92.5 and 98, respectively.

Figure (5) shows the Nyquist plots of the studied Aluminum sample in HCl solutions at 30 °C. It is seen from the figure that the impedance diagrams in most cases are not a perfect semi-circle, and this difference has been attributed to frequency dispersion. The semi-circle appearance indicates that the corrosion of Aluminium sample is mainly controlled by the charge transfer process, and it appears that there is a decrease in polarization resistance R_p values as judged from the decrease in the diameters of the semi-circles with increasing in HCl concentration to the dissolved of oxide film formed on Aluminium surface. The high frequency capacitive semi-circle is related to the dielectric properties and thickness of the oxide film (F. Zucchi, I. H. Omar, 1985), while the incomplete inductive loop may indicate to the presence of at least one adsorbed species (T. U. Chavanin, 1991), or the occurrence of localized corrosion (F. Zucchi, I. H. Omar, 1985). It is also seen that an inflection appears in the semi-circle especially at high HCl concentrations, which indicate the presence of an adsorbed film or localized attack, as previous observation.

At low concentrations of HCl (1.0 and 1.25)M, the passive oxide film locally ruptures and heals spontaneously as proved by A.C. impedance measurements (A. El-Etre, M. Abdullah and Z. El-Tantawy, 2005), while at higher concentrations (1.5M) prevent the healing process. This supports the motion that Cl^- ions responsible for the attack of the film and subsequent dissolution of the oxide layer, this appears from Fig.(1) in chemical measurements.

This in agreement with the study of Brett (C. N. A. Brett, 1990) by EIS technique. It was suggested that in the corrosion behavior of Aluminum in 0.1 M HCl the spectra indicates that the electrochemical processes in passive region are mainly controlled by a multi-step dissolution, ionic migration through the oxide layer and the influence of Cl^- ions. This behavior is similar to that obtained from polarization and HEM results.

Electrochemical parameters (R_{ct} and C_{dl}) established from Nyquist diagram are listed in Table (3). As can be seen from this Table, the charge transfer resistance (R_{ct}) for Aluminum sample in HCl solutions decreases with HCl concentration increase, and the C_{dl} values increased, this may be due to the formation of oxide-chloride complexes, or the adsorbed complex $[\text{AlOHCl}] \text{Cl}$ on the surface of Aluminum (E. Marboe and S. Bentur, 1961).

According to the mechanism proposed by Marbo and Bentur (E. Marboe and S. Bentur, 1961), O^{2-} ions are also formed in the solid phase by top chemical reaction. Consequently, $\text{Al}[\text{O}_x (\text{OH})_y (\text{H}_2\text{O})_z]$ is the idealized formula for the solid hydroxo complexes.

The presence of aggressive ions like Cl^- prevent the formation of the passive film and accelerates the process of anodic dissolution, the more the greater is the concentration of Cl^- ions. The pitting corrosion of Aluminum is due to the migration of chloride ions through the oxide film or due to the chemisorbed Cl^- onto the oxide surface

where they act as the reaction partners, aiding dissolution via the formation of different mixed oxo-hydro- and chloro- complexes.

Analysis of the polarization and impedance curves show that no passive active transition up to high current was observed. Anodic dissolution of the metal does occur at appreciable rate in solutions containing aggressive anions, particularly chloride.

3.2 Corrosion inhibition of Aluminium in 0.5M HCl at different concentrations of Aloe plant extract at 30°C.

The polarization curves (Fig. (6)) show the effect of aqueous extract of *Aloe* plant in the concentration range (4-60) %v/v on the corrosion of Aluminium in 0.5M HCl. It can be seen that, *Aloe* extract retarded the cathodic (hydrogen evolution) and the anodic (Aluminium dissolution) reactions, i.e., the increase of the *Aloe* extract concentration cause the displacement of the curves, this was more pronounced in the cathodic curves, thus the cathodic sites are blocked to a greater extent than the anodic sites by the extract components (A. M. Mohamed, A. Al- Nadjm and A. A, Fouda, 1998). This means that the *Aloe* extract act as mixed inhibitor mainly cathodic inhibitor. Also, the increase of *Aloe* extract concentrations cause gradual lowering in current density and corrosion rate values.

It is obvious from the relation between $I_{corr.}$ and $\log C_{inh.}$ (Fig.(7)) that the increases in *Aloe* extract concentration lead to lowering $I_{corr.}$ values which indicate the high ability of *Aloe* extract to inhibit the dissolution of Aluminium in HCl solution.

The percentage inhibition efficiency (Inh.%) was calculated using the equation:

$$\text{Inh. \%} = [I_{corr.add.} / I_{corr.free}] \times 100 \quad (15)$$

where $I_{corr.free}$ and $I_{corr.add.}$ are the corrosion current densities in the absence and presence the extract, respectively.

Table (4) shows the corrosion kinetics parameters, b_a , b_c , $E_{corr.}$, $R_{corr.}$ and Inh.%, the following observation could be drawn :

- 1) The anodic (b_a) and cathodic (b_c) Tafel slopes were changed in irregular form, this observation was previously recorded in many studied especially for Aluminium (M. Abdallah, 1981)(A. Fouda, H. El-Nadar and M. Moussa, 1987)(A. Mohamed, T. Rakha, M. Moussa, 1990)(A. Bouyanzer and B. Hammouti, 2004)(M. M. Saleh, 2006).
- 2) The values of $E_{corr.}$ change slowly to less negative values more positive (i.e., nearly remain constant) and the values of $I_{corr.}$ decrease. This indicates that *Aloe* plant primarily cause anodic polarization (M. Abdallah, Bull, 2000).
- 3) The values of Inh.% increases, indicating the inhibiting effect of *Aloe* plant at all concentrations except at the low concentration (4%v/v) it gives acceleration. This indicates that the inhibition efficiency depends upon the concentration of *Aloe* extract.

The inhibitive behavior could be attributed due to the adsorption of *Aloe* components on the Aluminium surface. The increase of the surface coverage (Θ) is found as the extract concentration increased. Competitive adsorption is assumed to occur on the surface of the metal between the aggressive Cl^- and the *Aloe* components at low concentrations, and the limited coverage leads to pitting corrosion which increase the rate of corrosion and acceleration will occur (V. Branzoi, F. Golgovici and F. Branzoi, 2002).

In order to get more information about the corrosion which increase inhibition phenomenon, solution resistance(R_s), polarization resistance(R_p), double layer capacitance(C_{dl}) and charge transfer resistance(R_{ct}) of Aluminium sample, impedance measurements have been carried for the Aluminium sample in 0.5 M HCl in the absence and presence of different concentrations of the aqueous extract of *Aloe* plant.

Figure(8) shows the effect of *Aloe* extract concentration on the impedance response of Aluminium sample in 0.5 M HCl deaerated solution at open circuit potential (OCP), after immersion of 15 minutes. The locus of the Nyquist plots was regarded as one part of a semicircle. The data reveal that the impedance diagrams in most of the cases is not a perfect sine-circle and this has been attributed to frequency dispersion and the half circle indicates that the corrosion of Aluminium is mainly controlled by the charge transfer process and the presence of *Aloe* extract in solution does not affect the mechanism of dissolution of Aluminium (F. Mansfeld, M. W. Kending and S. Tasi, 1980)(F. Mansfeld, M. W. Kending and S. Tasi, 1982). The charge transfer resistance R_{ct} has been calculated using Stern-Geary equation to get corrosion current from the difference in impedance at lower and higher frequencies as suggested by Tsuru et al. (T. Tsuru, S. Haruyama and B. Gijutsu, 1978)

For the calculation of double layer capacitance C_{dl} , the frequency at which the imaginary component of the impedance Z'' is maximum i.e., (Z''_{max}) is found and the double layer capacitance values are obtained from the relationship:

$$f(-Z''_{\max}) = 1/2\pi C_{dl} R_{ct} \quad (16)$$

Electrochemical chemical parameter R_{sol} , R_{ct} and C_{dl} established from the Nyquist diagram, obtained without and with the addition of different concentrations of *Aloe* extract in 0.5 M HCl solution, are listed in Table (4). It is seen from this table that the increase in the concentration of the *Aloe* extract increases the value of R_{ct} and decreases the corrosion of Aluminium in 0.5 M HCl solution and considerably reduced due to the adsorption of the *Aloe* component on Aluminium surface, or due to the increasing surface coverage by the *Aloe* molecules which leads to an increase in the inhibition efficiency with increasing *Aloe* plant concentration.

The inhibition efficiency of the corrosion of Aluminium is calculated as follows:

$$Inh_{R_{ct}} \% = R_{cto}^{-1} - R_{ct}^{-1} / R_{cto}^{-1} \times 100 \quad (17)$$

Where R_{cto} and R_{ct} are the values of charge transfer resistance without and with *Aloe* extract respectively. The values of inhibition efficiencies calculated are listed in Table (4).

It can be seen that the results of impedance studies are less in agreement with those obtained from polarization techniques, this attributed to that in the polarization process the clean of surface of Aluminium electrode will occurred as a result to beginning the cathodic polarization and the anodic polarization, this reduction cause the surface of Aluminium free from any adsorbed oxides.

-Adsorption Isotherm:

The decrease in the corrosion rate by the addition of *Aloe* extract is attributed to the adsorption of these extract molecules on the metal surface or to the formation of barrier film separating between the metal surface and the corrosion medium (B. K. Gary, R. A. V. Raff and R. V. Subramanian, 1977)(S. Rengamanni, S. Muralidharan, M. Anbu Kulandainathan and S. U. Iyer, 1994).

The process of adsorption can be described by two main types of interaction: physical adsorption and chemical adsorption. These are influenced by the nature and charge of the metal, extract composition and the type of electrolyte (F. Bentiss, M. Lagrenee, M. Traisnel, B. Mernani and H. Elattari, 1999).

The aqueous extract of *Aloe* plant investigated in this study is organic in nature and contain anthraquinones, coumarins and reducing substances. The constituents of *Aloe* extract are mostly composed of oxygen containing compounds. Adsorption of these constituents may occur through their oxygen active centres. Moreover, these compounds may also form complexes with metallic cations. The complexes can cause blocking of the micro anodes and / or the micro cathodes that are generated on the Aluminium surface when in contact with accordant (HCl), and hence can retard the dissolution of the Aluminium (M. Quraishi, I. Farooqi and P. Saini, 1999).

In hydrochloric acid, *Aloe* compounds forms its hydrochloride, or more specifically, it exists as a moiety of protonated *Aloe* or *Aloe* compounds ions and chloride ion (G. Banerjee and S. N. Malhotra, 1992). When Aluminum electrode is immersed in dilute solution of hydrochloric acid containing *Aloe*, three kinds of species can be adsorbed on its surface, which are described as (G. B. Hunt and A. K. Holiday, 1980):

-If the metal surface is positively charged (with respect to potential of zero charge (PZC)), the chloride ions will first be adsorbed on the metal surface, which in turn will attract the *Aloe* compounds ions and protonated water molecules. Therefore, a close-packed triple layer will form on the metal surface and inhibit Aluminium ions to enter the solution. Hence, with an increase in the positive charge on the metal surface, the adsorption of *Aloe* would increase and the *Aloe* content of the solution would decrease.

-If the metal surface is negatively charged with respect to PZC, the protonated water molecules and the *Aloe* compounds ions would be directly adsorbed on the metal surface. With increasing the negative charge on the metal surface, adsorption of *Aloe* increase and its concentration in solution would decrease.

-When the metal surface attains the potential at which the surface charge becomes zero, none of the ions (neither cations nor anions) adsorb on the surface through their centres. A few *Aloe* compounds molecules may however be physically adsorbed through their planar π orbitals on the metal surface (with vacant d orbitals). Because of this possibility, the concentration of *Aloe* in solution at PZC also decreases instead of remaining the same. However, the decrease is small due to the fact that, in the case of planar adsorption, the molecular density of *Aloe* on the Aluminium surface becomes smaller to a lesser degree than in the case of later-ionic adsorption. The amount of *Aloe* adsorbed is smaller in the former case than in the latter.

Figure (9) shows the variation of the protection efficiency from the polarization and impedance methods with the logarithm of the concentration of *Aloe* extract at 30°C. The plots show that the *Aloe* extract inhibits the corrosion of Aluminium and the inhibition efficiency increased with the increase in the extract concentration. These results suggest that the inhibitor retard the dissolution of Aluminium by adsorption of *Aloe* molecules at

Aluminium\ acid solution interface. One inflection point was observed, indicating perhaps that the *Aloe* extract form a monolayer on the surface of Aluminium.

The adsorption of *Aloe* molecules on the Aluminium surface was found to follow the Freundlich adsorption isotherm (Fig.(10)) which is given by (B. Berge, K. Grijothheim, C. Kronhn, R. Ncumann and K. Torkiep, 1976)(M. Kliskic, J. Radosevic, S. Gudic and V. Katalinik, 2000):

$$\theta = KC^n \quad (19)$$

where $0 < n < 1$

$$\log \theta = \log K + n \log C \quad (20)$$

θ is the degree of surface coverage for different concentrations of the extract in acidic media using the equations:

$$\theta = \frac{i_{\text{corr}(\theta=0)} - i_{\text{corr}\theta}}{i_{\text{corr}(\theta=0)} - i_{\text{corr}(\theta=1)}} \quad (21)$$

$$\theta = \frac{C_{\text{dl}(\theta=0)} - C_{\text{dl}\theta}}{C_{\text{dl}(\theta=0)} - C_{\text{dl}(\theta=1)}} \quad (22)$$

C is the *Aloe* extract concentration in the bulk of the solution and K is the binding constant (equilibrium constant) for the adsorption process in which is evaluated from the intercepts of the plots and related to the standard free energy of adsorption, $\Delta G_{\text{ads}}^\circ$, as in the relation (23).

$$K = 1/55.5 \exp.(-\Delta G_{\text{ads}}^\circ / RT) \quad (23)$$

The calculated values of K and $\Delta G_{\text{ads}}^\circ$ were found to be 1.06 and -10.26 k.J. mol.⁻¹ from polarization and the values 1.16 and -10.50 k.J. mol.⁻¹ from impedance measurements, respectively, this is in agreement with that obtained in previous studies (E. Oquzic, 2007)(E. El- Etre, 2003).

The value of K is relatively small indicating that low interaction between the adsorbed molecules and the metal surface is found and the extract molecules are physically adsorbed on the Aluminium surface through adsorption of oxygen atom with lone pair of electron on *Aloe* molecules. The values of $\Delta G_{\text{ads}}^\circ$ also are large and acquire negative sign suggesting that the adsorption occurs spontaneously.

The inhibition of Aluminium corrosion by *Aloe* extract can be explained on the basis that the inhibition process of *Aloe* molecules found through the simple blocked of active area on Aluminium surface, on another side the inhibitor decreases the surface area for anodic dissolution and hydrogen evolution without affect the corrosion mechanism. The reaction between HCl and Aluminium metal which covered by *Aloe* molecules probably occurred by diffusion of hydrogen ions and chloride ions through the pores passive layer (metal – oxide – hydroxide – inhibitor)_{ads}. Which was suggested previously (E. Khamis and M. Atea, 1994).

-Microstructure Study of Aluminium Surface by SEM at 30°C:

Scanning electron microscopy (SEM) was employed to study the surface morphology of Aluminium surface. The sample was studied after etching and \ or 90 minutes immersion in the test solution, in both cases after mechanical polishing. Figure (11(a)) reveals the microstructure of polished Aluminium before placing it in the test solution. The scan shows that a solid and homogeneous surface is found.

Figure (11 (b)) illustrates the effect of 0.5M HCl on Aluminium sample after 90 minutes immersion at 30°C, it appears that the presence of general corrosion and pitting corrosion (a large number of vacuoles with different sizes).

Figure (11 (c)) exhibit the effect of 4% v/v of *Aloe* extract in 0.5M HCl, it obvious that the presence of thin porous and protection layer on Aluminium surface contain of numerous pits less than that appears in case of HCl acid alone, the pits are deep and contain a local cell and pitting corrosion which increase the corrosion rate (acceleration), $\text{Inh}\% = -12.90\%$.

On comparing figure (11 (d)) which illustrates the effect of 48% v/v of *Aloe* extract in 0.5M HCl at 30°C on Aluminium sample, it appears that the disappearance of vacuoles (pits) and the formation of an adsorbed film on Aluminium surface due to adsorption of *Aloe* extract components lead to high corrosion inhibition at this concentration. This confirms with the previous results obtained from electrochemical studies.

3.3 Corrosion Inhibition of Aluminium in 0.5M HCl at Different Concentrations of the Aloe Plant and Constant Concentration of Iodide Ions.

The corrosion of Aluminium in 0.5M HCl solutions containing different concentrations of *Aloe* extract and 0.01 M of iodide ions was studied electrochemically using polarization and impedance methods.

Table (5) gives the values of corrosion potential (E_{corr}), corrosion current (I_{corr}), corrosion resistance (R_{corr}), charge transfer resistance (R_{ct}), double layer capacitance (C_{dl}) and inhibition efficiency (Inh.%) obtained from the polarization and impedance plots. It clears those values of E_{corr} remain constant with increase **Aloe** concentration in presence of 0.01M of NaI, where I_{corr} and R_{corr} were decrease. The inhibition efficiency is also was found to increase with increasing concentration of **Aloe** extract. The values of R_{ct} increase, while the values of C_{dl} decrease, this explained on the basis that addition of iodide ions enhance the adsorption of **Aloe** molecules on Aluminium surface, by synergistic effect between Γ ions and **Aloe** compounds (S. Syed Azim, S. Muralidharan and S. V. Iyer, 1995)(S. S. Abd El- Rahim, M. Sh. Shalaby and S. M. Abd El- Haleem, 1985)(S. Syed Azim, S. Muralidharan, S. V. Iyer, B. Muralidharan and T. Vasudevan, 1998).

The synergism parameter (S_{θ}) was calculated using the relationship given by Aramaki and Hackermann (K. Aramaki and N. Hackerman, 1969):

$$S_{\theta} = (1 - \theta_{1+2}) / (1 - \theta_1 - \theta_2) \tag{17}$$

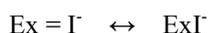
where: $\theta_{1+2} = (\theta_1 + \theta_2) - (\theta_1 \theta_2)$,

θ_1 = surface coverage by anion,

θ_2 = surface coverage by cation and,

θ_{1+2} = measured surface coverage by both anion and cation.

These values are given in Table (6), which calculated for various concentrations of **Aloe** extract in presence of 0.01M of Γ ions. Figure (12 (a& b)) shows the plot of synergism parameter (S_{θ}) against concentration of **Aloe** extract from polarization and impedance methods, respectively. The S_{θ} values given in Table (6) are more than unity and this indicate that the enhanced inhibition efficiency caused by the addition of iodide ions to **Aloe** extract is only to a synergistic effect between Γ ions and **Aloe** molecules (K. Aramaki, M. Hagiwara and H. Nishihawa, 1987)(G. K. Gomma, 1998)(M. A. Quraishi and J. Rawat, 2002), this can be attributed to cooperative adsorption between Γ ions and **Aloe** cations. This can explained as: the attraction between Γ ions and **Aloe** molecules lead to formation of ionic complex $Ex\Gamma$ adsorbed on Aluminium surface by columbic attraction which lead to decrease the rate of corrosion by stabilisation of adsorbed anions and increase the covered area and increase the inhibition efficiency (A. Mohamed, T. Rakha, M. Moussa, 1990)(G. K. Gomma, 1998)(N. S. Rawat and G. Udayabhanu, 1987). The following reaction can be suggested as:



The complex $Ex\Gamma$ adsorb on the Aluminium surface and cause the corrosion inhibition of Aluminium in HCl.

Figure(13) shows the variation of inhibition efficiency with the concentration of **Aloe** extract in 0.5M HCl containing 0.01M NaI obtained from polarization and impedance measurements at 30°C. The presence of Γ ions enhanced the protection efficiency of **Aloe** extract especially at low concentrations

3.3 Corrosion Inhibition of Aluminium in 0.5M HCl at Fixed Concentration of Aloe Extract and Different Concentrations of Iodide Ions.

The inhibition efficiency of 48% v/v of **Aloe** extract for Aluminium in 0.5M HCl was determined for various concentrations ($1.0 \times 10^{-4} - 5.0 \times 10^{-2}$)M of Γ ions (Figs.(14,15)). It can be seen from these figures and Table (7) that Inh.% was increased by adding Γ ions until reach to 2.5×10^{-2} M then a decrease in the inhibition efficiency with increasing concentration of Γ ions is occurred.

The increase of Inh.% with increasing the concentration of NaI may explained as to be due to a joint adsorption (co- adsorption) of Γ ions with inhibitor molecule. Adsorption of **Aloe** extracts molecules at Aluminium / solution interface occurred through physical adsorption via the oxygen atom with its positive charge and chemisorptions through the electron orbital of the benzene rings. It is known that Γ ions have strong interaction with Aluminium surface owing to chemisorptions, but it gives an acceleration in absence of **Aloe** extract as inhibitor.

When the anion and **Aloe** extract coexist in solution, in terms of electrostatic attraction of these species, the ionic complex $Ex\Gamma$ will formed between **Aloe** cations and Γ ions and adsorb on Aluminium surface which lead to increase the coverage and enhance the inhibition (Y. Feng, K. S. Siow, W. K. Teo and A. K. Hsieh, 1999).

Figure (16) and Table (8) show the relation between S_{θ} and C of Γ ions, which appears that $S_{\theta} > 1$ indicate that the presence of synergistic effect between Γ ions and **Aloe** molecules as a result of co- adsorption.

The addition of I^- leads to stabilisation of *Aloe* extract adsorption on Aluminium surface by the interaction between *Aloe* molecules and iodide ions. The formation of the ionic complex which adsorbed through iodide ions on the surface of Aluminium (Y. Feng, K. S. Siow, W. K. Teo and A. K. Hsieh, 1999).

From these results, iodide ions in NaI are suggested to have synergistic effect with *Aloe* extract only at low concentrations because it was prevent the acceleration and enhance the inhibition of Aluminium corrosion.

The decrease of Inh.% indicates that desorption takes place at Al / acid solution interface. Assuming that the adsorption of the inhibitor molecules and the anions is ionic in nature and of the overlap type, then the addition of more anions might give rise to a weak combination of anions and *Aloe* cations, and as a result make the cations desorbed. Desorption of the cations will then result in their replacement with water molecules.

From previous results, it is known that NaI could be considered as one of the effective anions for synergistic action within the *Aloe* plant inhibitor.

4. Conclusion:

The effect of acid concentration and the effect of addition the aqueous extract of *Aloe* plant on the corrosion of Aluminium has been studied. The following conclusions may be drawn:

- 1) The chemical results showed that the corrosion rate of Aluminium sample is increase with increasing acid concentrations (0.25-1.5) M.
- 2) The polarization measurements also showed that, the increase of HCl concentration leads to displacement of the anodic and cathodic curves to high current densities ($I_{corr.}$), also increase the corrosion rate will be found.
- 3) The electrochemical impedance measurements showed that the corrosion of Aluminium sample is mainly controlled by charge transfer process.
- 4) The aqueous extract of *Aloe* plant acts as good inhibitor for the corrosion of Aluminium metal in HCl solution.
- 5) Electrochemical polarization results indicates that the *Aloe* aqueous extract act as mixed type inhibitor and impedance results showed that the corrosion of Aluminium is mainly controlled by a charge transfer process and the presence of *Aloe* extract in acid solution does not alter the mechanism of Aluminium dissolution.
- 6) The adsorption of *Aloe* extract molecules on Aluminium metal surface follows Freundlich adsorption isotherm, the values of $K_{ads.}$ and $\Delta G_{ads.}$ indicate physical adsorption of *Aloe* molecules on Aluminium surface.
- 7) The addition of 0.01M NaI to Aluminium sample in HCl in presence of different concentrations (4-60)%v/v of *Aloe* extract enhances the inhibition efficiency of *Aloe* plant, and the addition of different concentrations ($1.0 \times 10^{-4} - 5.0 \times 10^{-2}$) M of NaI in the presence of 48%v/v of *Aloe* extract leads to increase the Inh.% by I^- ions reach to 2.5×10^{-2} M then a decrease in the Inh.% with increasing concentration will occurred.

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Table 1. Chemical composition of Aluminum specimen.

<i>Element</i>	<i>Mn</i>	<i>Ni</i>	<i>Fe</i>	<i>Pb</i>	<i>Si</i>	<i>Zn</i>	<i>Cr</i>
%	0.009	0.043	0.765	0.014	2.242	1.621	0.009

Table 2. Corrosion rates (R and R') for Aluminum sample at different concentrations of HCl at 30°C.

<i>C (M)</i>	0.25	0.5	0.75	1.0	1.25	1.5
<i>R</i> <i>ml. cm.⁻² min.⁻¹</i>	0.1623×10^{-2}	7.917×10^{-2}	13.21×10^{-2}	22.73×10^{-2}	29.25×10^{-2}	65.80×10^{-2}
<i>R'</i> <i>g. cm.⁻² min.⁻¹</i>	0.1300×10^{-5}	2.580×10^{-5}	9.011×10^{-5}	14.63×10^{-5}	15.20×10^{-5}	43.00×10^{-5}

Table 3. Electrochemical parameters for corrosion of Aluminium in different concentrations of HCl at 30°C.

<i>Conc.</i> <i>(M)</i>	<i>Polarization</i>					<i>Impedance</i>		
	$-E_{corr}$ <i>(mV)</i>	b_a <i>(mV dec⁻¹)</i>	b_{c^-} <i>(mV dec⁻¹)</i>	C_{dl} <i>(μF)</i>	R_{ct} <i>(Ωcm^2)</i>	R_{sol} <i>(Ωcm^2)</i>	R_{ct} <i>(Ωcm^2)</i>	C_{dl} <i>(μF)</i>
0.25	693.805	34.25	207.88	2.181	23.751	1.347	79.85	78.510
0.50	726.573	44.55	131.22	7.282	79.300	0.584	42.52	102.50
0.75	754.973	71.85	139.42	19.48	212.17	1.206	8.891	161.90
1.00	759.342	87.17	174.03	23.06	251.12	2.755	2.599	237.70
1.25	766.990	106.60	103.75	33.46	364.40	0.864	0.420	440.75
1.50	774.635	138.02	132.15	39.36	428.58	0.890	0.156	472.50

Table 4. Electrochemical parameters and inhibition percentages of Aluminium corrosion in 0.5M HCl in presence of different concentrations of *Aloe* extract at 30°C.

<i>Conc.</i> <i>V/V</i>	<i>Polarization</i>						<i>Impedance</i>			
	$-E_{corr}$ <i>(mV)</i>	b_a <i>(V dec⁻¹)</i>	b_{c^-} <i>(V dec⁻¹)</i>	I_{corr} <i>(mA. cm⁻²)</i>	R_{corr} <i>(mm day⁻¹)</i>	<i>Inh. %</i>	R_{sol} <i>(Ωcm^2)</i>	R_{ct} <i>(Ωcm^2)</i>	C_{dl} <i>(μF)</i>	<i>Inh. %</i>
0.0	762.57	44.55	131.22	7.28	79.30	---	0.584	42.52	102.5	---
4	737.36	85.82	15.05	7.36	80.11	-1.07	2.331	59.25	122.00	15.00
8	740.22	78.30	15.02	5.99	65.18	17.74	1.322	68.01	113.60	25.00
20	735.46	104.57	19.45	3.08	33.59	57.70	1.605	80.81	69.67	40.00
40	737.36	63.28	25.38	2.73	29.75	62.51	2.268	99.77	63.82	50.00
48	728.81	45.69	75.44	2.16	23.49	70.34	1.198	200.40	50.55	75.05
60	729.27	71.37	63.25	1.26	13.75	82.69	1.052	359.50	21.19	88.42

Table 5. Electrochemical parameters and inhibition percentages for the corrosion of Aluminium in presence of various concentrations of *Aloe* plant extract and in presence of 0.01M NaI at 30°C.

Conc V/V	Polarization						Impedance			
	$-E_{corr.}$ (mV)	b_a (mV dec ⁻¹)	b_c (mV dec ⁻¹)	$I_{corr.}$ (mA cm ⁻²)	$R_{corr.}$ (mm day ⁻¹)	Inh.%	$R_{sol.}$ (Ω cm ²)	$R_{ct.}$ (Ω cm ²)	C_{dl} (μF)	Inh. %
0.0	726.57	44.55	131.22	7.28	79.30	-----	0.584	42.52	102.5	-----
4	725.80	33.01	110.71	8.22	89.51	-12.9	0.696	47.78	99.50	12.5
8	737.03	97.22	14.60	6.01	65.47	17.47	1.514	50.90	96.80	18.33
20	731.49	33.98	119.81	3.27	35.62	55.10	2.665	56.71	84.50	26.67
40	726.07	72.32	24.75	3.17	34.56	56.50	0.737	62.52	59.20	33.38
48	732.05	71.89	15.14	2.68	29.16	63.20	0.525	87.23	57.80	52.23
60	731.19	79.64	15.17	1.45	15.79	80.09	3.592	181.1	37.94	77.0

Table 6. Synergism parameters for the corrosion of Aluminium in 0.5M HCl in presence of *Aloe* plant extract and 0.01M NaI at 30°C.

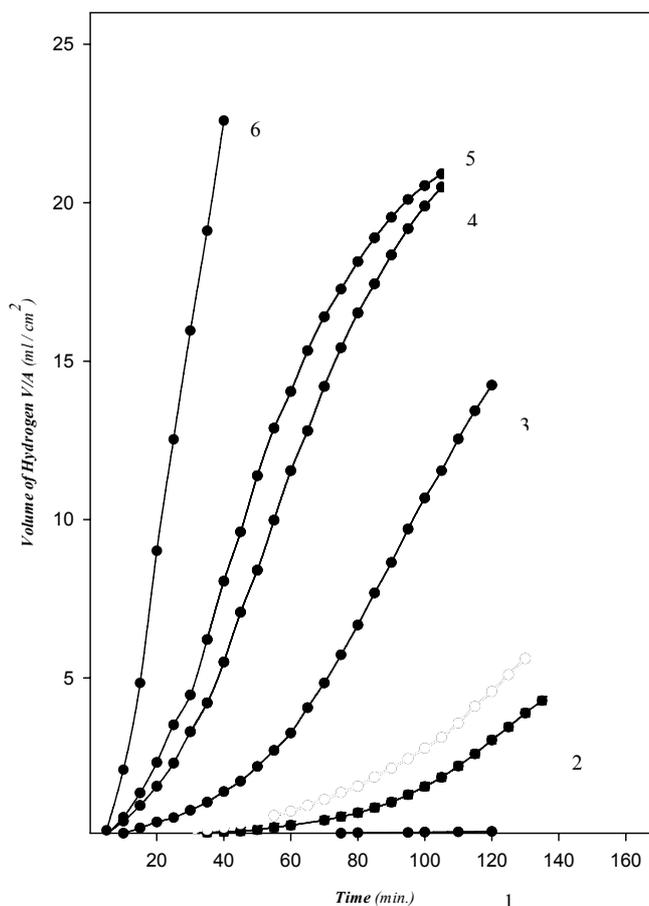
Conc. Of Iodide Ions	$S_{\theta}(P)$	$S_{\theta}(I)$
1.0×10^{-4}	1.81	4.25
2.5×10^{-3}	1.75	3.55
1.0×10^{-2}	2.31	3.47
2.5×10^{-2}	1.68	3.24
3.0×10^{-2}	1.45	2.73
5.0×10^{-2}	1.10	1.71

Table 7. Electrochemical parameters and inhibition percentages for the corrosion of Aluminium in 0.5M HCl in presence of 48%v/v *Aloe* extract and different concentrations of NaI at 30°C.

Conc. V/V	Polarization						Impedance			
	$-E_{corr.}$ (mV)	b_a (V dec ⁻¹)	b_c (V dec ⁻¹)	$I_{corr.}$ (mA cm ⁻²)	$R_{corr.}$ (mm day ⁻¹)	Inh.%	$R_{sol.}$ (Ω cm ²)	$R_{ct.}$ (Ω cm ²)	C_{dl} (μF)	Inh.%
48	732.05	71.89	15.14	2.68	29.16	63.20	0.525	87.23	57.80	52.23
1.0×10^{-4}	731.0	70.50	128.33	4.01	43.70	44.94	1.437	150.0	48.66	66.67
2.5×10^{-3}	726.08	67.17	141.87	3.16	34.40	56.61	0.822	164.1	58.39	69.53
1.0×10^{-2}	728.81	45.69	75.44	2.16	33.22	70.34	2.427	193.8	50.31	74.2
2.5×10^{-2}	747.22	111.04	30.97	1.96	30.93	73.08	3.792	304.0	30.59	83.55
3.0×10^{-2}	743.16	101.54	26.48	2.02	22.06	72.26	2.135	276.1	41.00	81.90
5.0×10^{-2}	731.77	92.19	32.05	2.11	22.96	71.02	1.467	198.9	48.14	74.90

Table 8. Synergism parameters for the corrosion of Aluminium in 0.5M HCl in presence of 48% of *Aloe* plant extract and different concentration of NaI at 30°C.

<i>Conc. Of Aloe V/V</i>	$S_{\theta}(P)$	$S_{\theta}(I)$
4	2.08	1.76
8	1.88	1.86
20	1.98	2.09
40	2.16	2.28
48	2.31	3.28
60	2.14	3.39



(1) 0.25 M (2) 0.50 M (3) 0.75 M (4) 1.00 M (5) 1.25 M (6) 1.5 M

Figure 1. Volume of hydrogen / time curves of Al in different concentrations of HCl at 30° C.

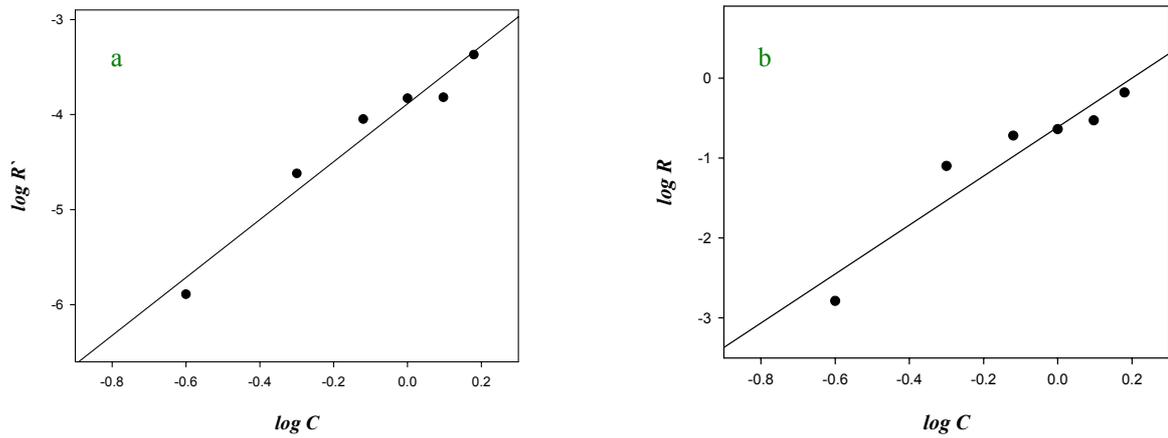


Figure 2. Variation of (a) $\log R'$ (b) $\log R$ of Al sample with the concentration of HCl at 30°C.

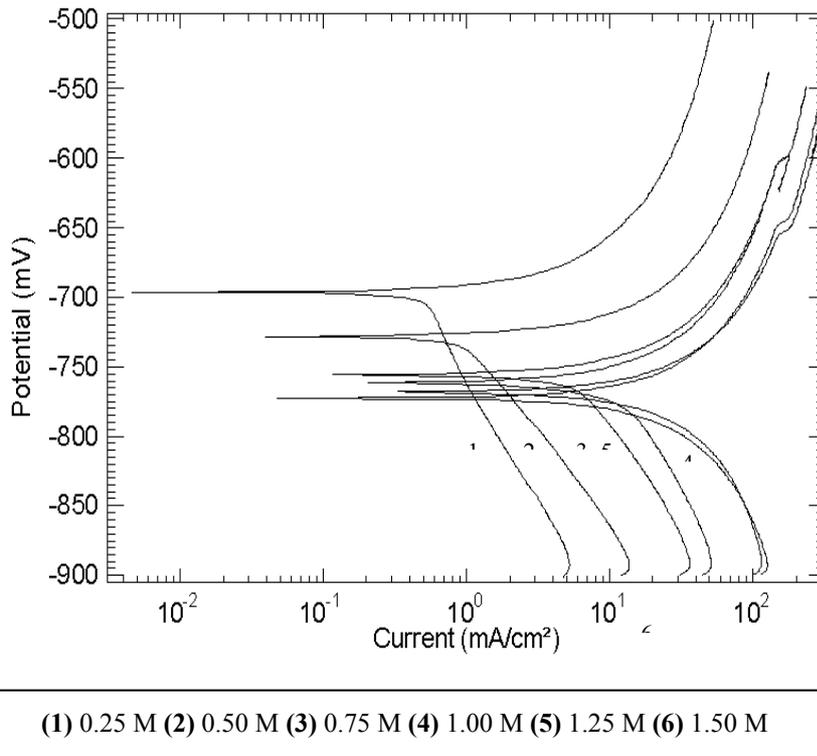


Figure 3. Polarization curves for Al sample corrosion in different concentrations of HCl solution at 30° C

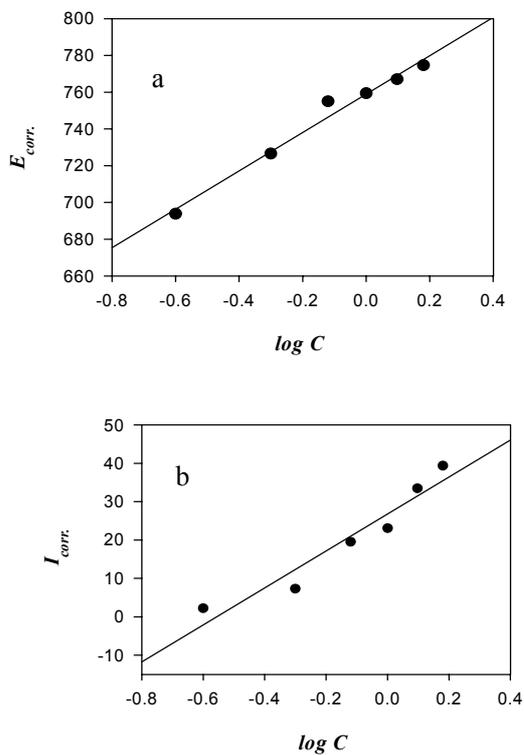
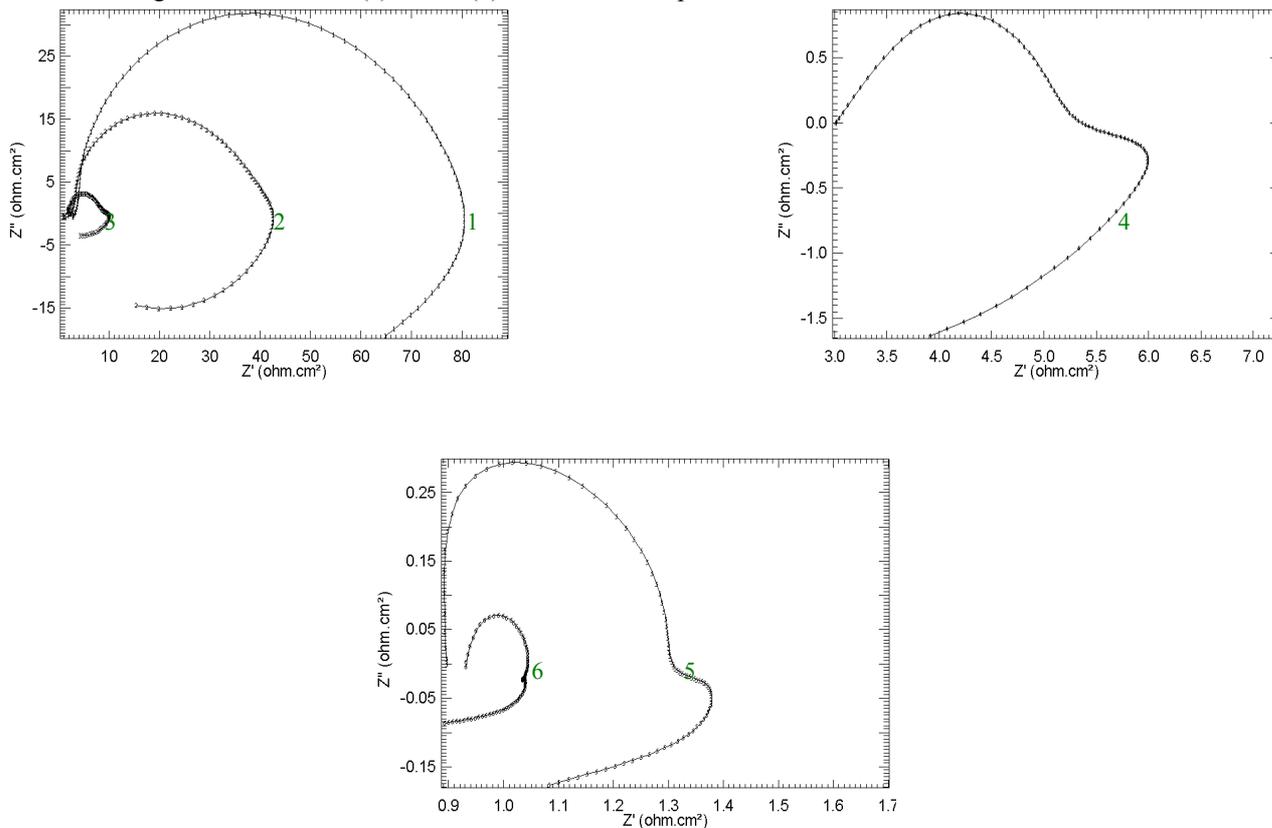
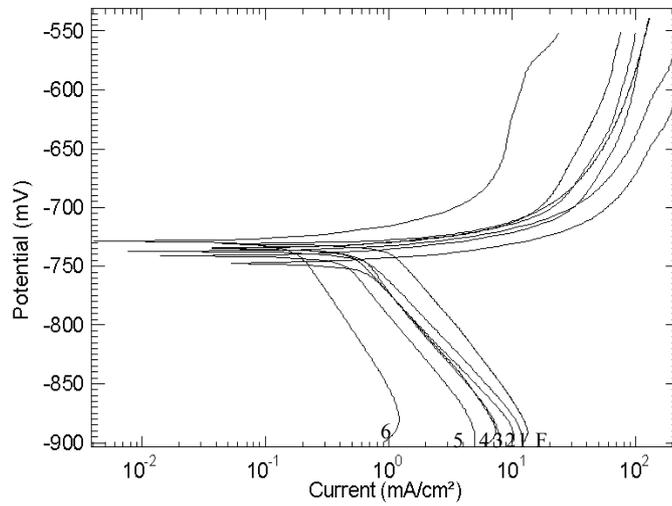


Figure 4. Variation of (a) E_{corr} . (b) I_{corr} . of Al sample with the concentration of HCl at 30°C.



(1) 0.25 M (2) 0.50 M (3) 0.75 M (4) 1.00 M (5) 1.25 M (6) 1.50 M

Figure 5. Nyquist plot for Al sample corrosion in different concentrations of HCl solution at 30°C.



[(F) 0.00 M (1) 4.0 (2) 8.0 (3) 20.0 (4) 40.0 (5) 48.0 (6) 60.0] V/V

Figure 6. Polarization curves for Al sample corrosion in different concentrations of *Aloe* extract at 30° C.

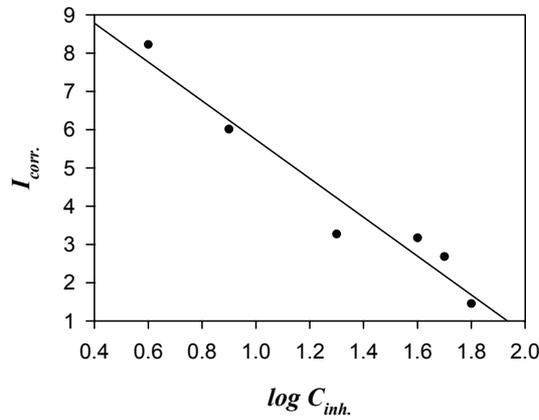


Figure 7. Variation of $I_{corr.}$ of Al sample with the concentrations of *Aloe* extract at 30°C .

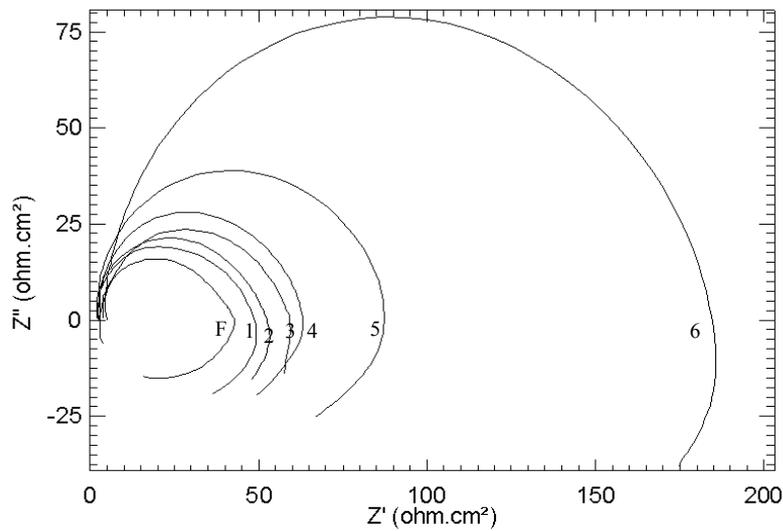


Figure 8. Nyquist plot for Al sample corrosion in different concentrations of *Aloe* extract at 30° C.

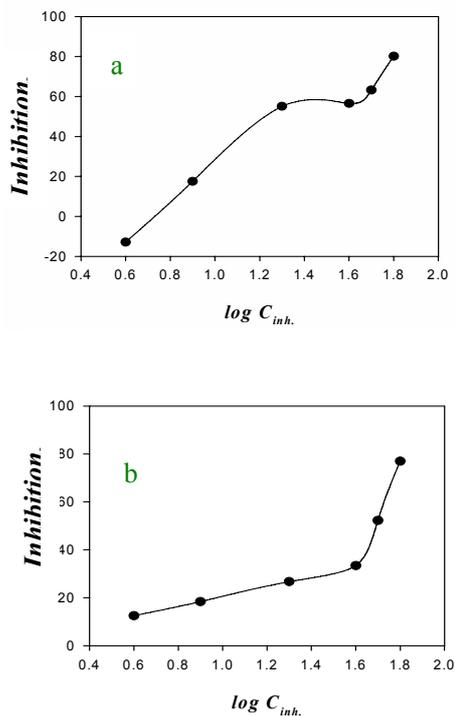


Figure 9. The relation between inhibition efficiency and the logarithm of concentration of *Aloe* extract for the corrosion of Al sample in 0.5 M HCl at 30° C from (a) polarization (b) impedance.

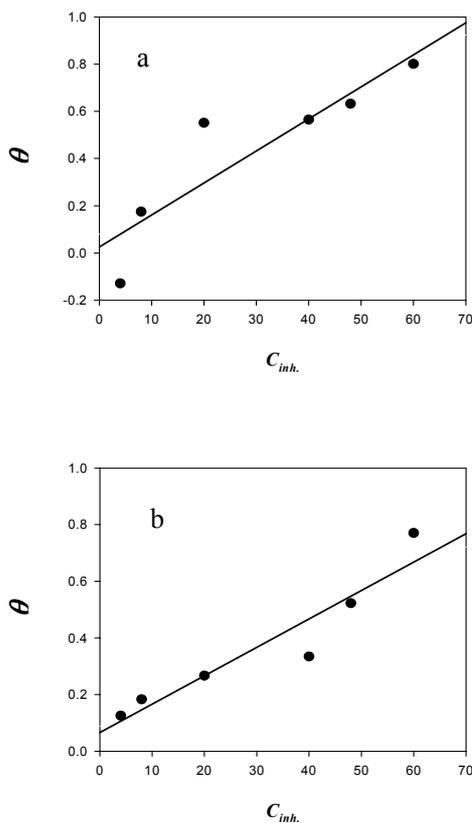


Figure 10. The relation between $C_{inh.}$ and for the corrosion of Al sample in 0.5 M HCl in presence of different concentrations of *Aloe* extract in at 30° C from (a) polarization, (b) impedance .

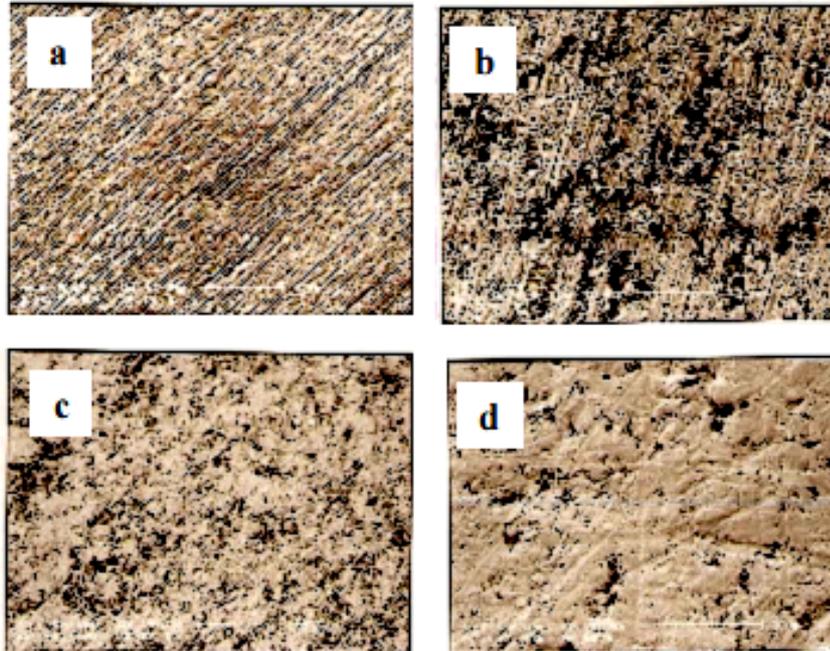


Figure 11. SEM photographs of aluminium sample, (a) before immersion, (b) after corrosion, (c) in presence of 4% v/v *Aloe* extract (low concentration) and (d) in presence of 48% v/v of *Aloe* extract (high concentration) in 0.5 M HCl solution at 30° C.

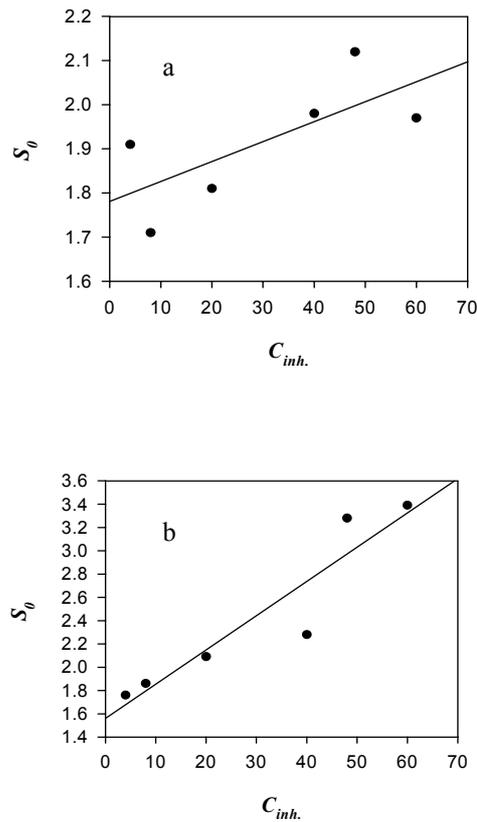


Figure 12. The relation between $C_{inh.}$ and S for the corrosion of Al sample in 0.5 M HCl in presence of different concentrations of *Aloe* extract+ 0.01 M NaI at 30° C from (a) polarization, (b) impedance .

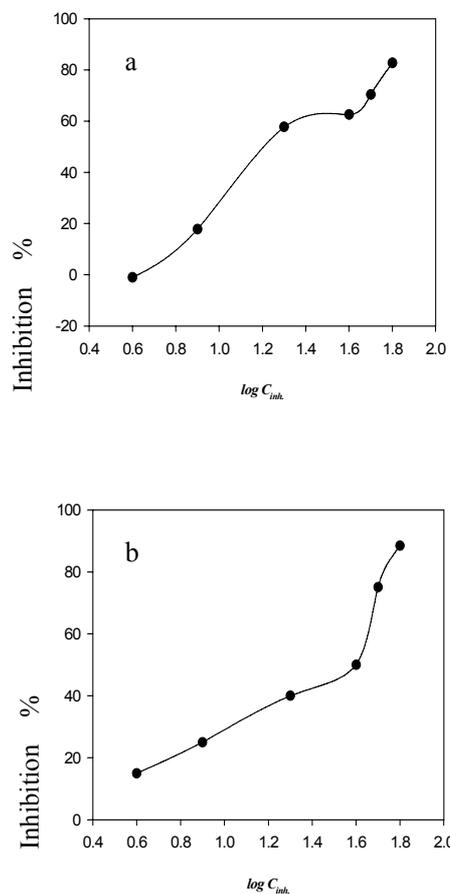
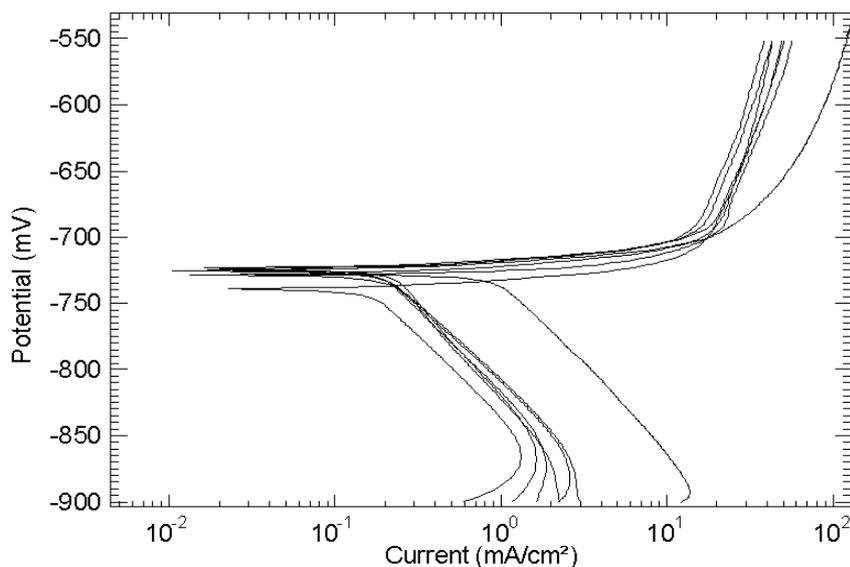
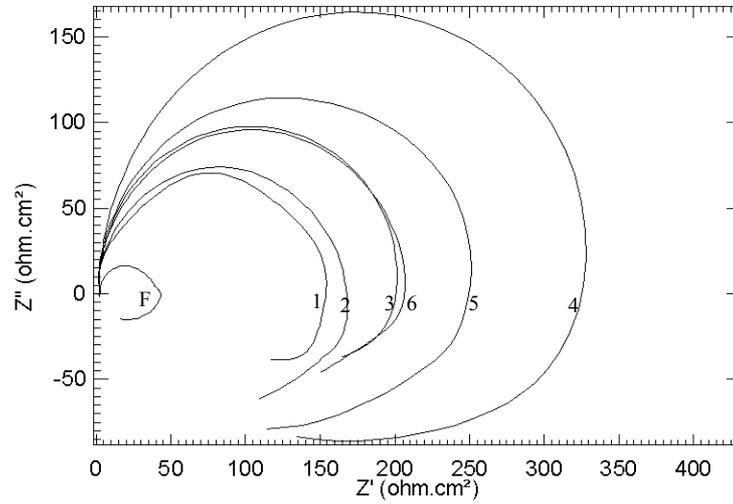


Figure 13. The relation between $\log C_{inh}$. and Inhibition efficiency for the corrosion of Al sample in 0.5 M HCl in presence of different concentrations of *Aloe* extract+ 0.01 M NaI at 30° C from (a) polarization, (b) impedance .



[(F) 0.00 (1) 1.0×10⁻⁴ (2) 2.5 ×10⁻³(3) 1.0 ×10⁻² (4) 2.5×10⁻² (5) 3.0×10⁻² (6) 5.0×10⁻²] M

Figure 14. Polarization plot for Al sample corrosion in 0.5 M HCl + different concentrations of NaI and presence of 48% v/v of *Aloe* extract at 30° C.



[(F) 0.00 (1) 1.0×10^{-4} (2) 2.5×10^{-3} (3) 1.0×10^{-2} (4) 2.5×10^{-2} (5) 3.0×10^{-2} (6) 5.0×10^{-2}] M

Figure 15. Nyquist plot for Al sample corrosion in 0.5 M HCl + different concentrations of NaI and presence of 48% v/v of *Aloe* extract at 30° C.

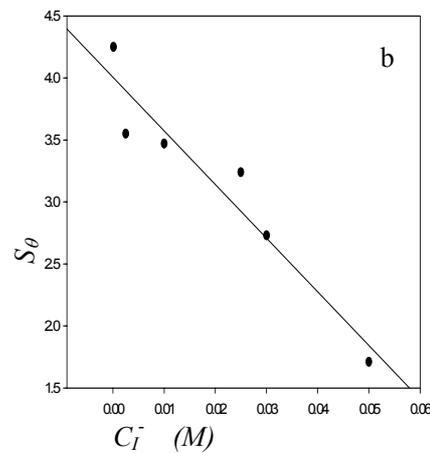
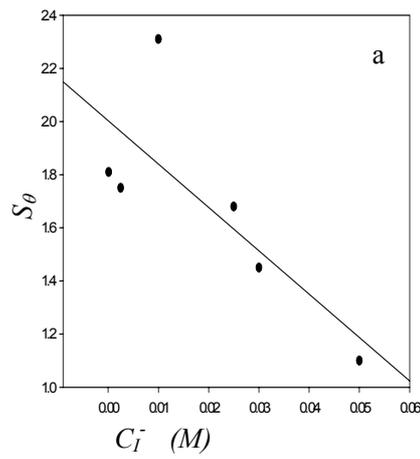


Figure 16. The relation between $C_{inh.}$ and S for the corrosion of Al sample in 0.5 M HCl in of presence 48% of *Aloe* extract and different concentration NaI at 30 C from (a) polarization, (b) impedance.