THE EFFECTS OF INHIBITION OF FERN EXTRACTS ON THE ALKALINE AND ACID CORROSION OF ALUMINIUM ALLOY

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ABSTRACT

The deterioration behavior of aluminum alloy in 1M Hydrochloric acid and 1M Sodium hydroxide solution in fern extract was investigated using a weight loss technique. Aluminum alloy coupons of dimension 4 by 4 cm were immersed in test solutions of uninhibited acid and alkaline solutions, and also those containing the extract concentrations of 1, 2, 3, 4, and 5 g/l at ambient temperature for 168 hours. The test results show that the fern extract exhibited lower inhibitive properties in the alkaline than in the acidic medium. The observed adsorption characteristics were best described using Langmuir isotherm. The thermodynamic properties obtained were best described by physical adsorption mechanisms.

Keywords: Fern Extracts, Alkaline Medium, Langmuir Isotherms, Adsorption Characteristics, Molecular Interaction, Inhibition Efficiency.

INTRODUCTION

Metals are widely used in the industry today. These industries include the manufacturing industry, metallurgy industry, automobile, food packaging, construction, paints and dye making, electrical applications, and many other sectors. Some of these metals include copper, iron, nickel, cobalt, and aluminium¹-². Aluminium is the most common metal found in the earth’s crust; hence it is commonly used in industries because of its availability. Pure aluminium is very light and therefore too weak and soft to be of much use, that is the reason why most of the “metals” we use are not virtually metals but alloys. For the aluminum alloy to be formed, pure aluminium is combined with different metals to make them stronger, harder and better.²-³ Aluminium alloy is one of the most versatile metallic materials. It is widely used in industries for construction, transportation, food processing, food production, and food storage purposes. Aluminium also has economic importance due to its lightweight, electrical and thermal conductivity, corrosion resistance, diversity of the alloys and intermediates, recycling property and ease of use. Despite the usefulness and good properties of aluminium alloys, harsh conditions such as an acidic environment lead to deterioration of the aluminium alloy caused by corrosion and this is an issue of concern because of the resulting damages.⁴-⁵ Some consequences of these corrosion processes include plant shutdowns, loss of product, contamination of the environment, waste of valuable resources, loss of valuable products, etc.⁶

Corrosion occurs when the protective oxide film is broken as a result of electro-chemical action on the surface of the metal.⁷ The factors influencing metal corrosion is the nature of the environment and the nature of the metal, which includes the effect of pH, temperature, humidity, presence of impurities in the atmosphere, the conductance of the medium, presence of oxygen, oxidation potential, relative areas of the anode and cathode, nature of the corrosion product, etc. some forms of corrosion are atmospheric, pitting, crevice, intergranular, uniform, filiform, exfoliation, erosion, microbiological, etc.⁸ The hydrogen ion concentration of an acidic media is usually more corrosive than alkaline media. Aluminium alloy is mostly used in industries; this is why researchers are very concerned about how to inhibit corrosion on it. Corrosion is an unavoidable process, but it can be controlled. Metallic corrosion has a noticeable effect on the country’s development which can be in comparison with natural disasters like floods etc. For instance,
the direct cost of metals in the US was summed up to approximately $276 billion annually, which is far greater than the normal loss incurred as a result of natural disasters ($17 billion per annum). The control of corrosion of metals or their alloys is of economic, environmental, technical, and aesthetical importance. Literature has revealed that the presence of N, S, and O in organic compounds shows an excellent efficiency of inhibition. Unfortunately, they are toxic to the environment, human health, aquatic and animal life; also, they are expensive as well. Some of the consequences of the use of toxic inhibitors are temporary or permanent damage to the liver or kidney. The use of toxic materials has been constrained by agencies such as the Occupational Safety and Health Administration (OSHA), the U.S. Environmental protection Agency (EPA), and the U.S. Department of Transportation (DOT). Therefore, to replace the toxic corrosion inhibitors for mitigating corrosion of metals, green corrosion inhibitors which are non-toxic, biodegradable, environmentally benign and at low cost, have been studied. Also, research on natural products such as plant extracts, purified compounds, and essential oils to obtain benign inhibitors to corrosion has been on the increase. The first evidence of the use of inhibitors of natural origin to corrosion was in the mid-1930 when extracts of plants were utilized in H₂SO₄ pickling baths. Research to apply naturally occurring inhibitors to control corrosion is developing successfully as environmental awareness is encouraging it.

It has been observed from various researches that organic compounds act as inhibitors to corrosion via adsorption on the interface of the metal solution. The adsorption characteristics of the organic or green inhibitors depend on a few physicochemical properties of the constituent molecules, inclusive of the electron density of the donor atoms, the possible interactions of d-orbitals of the surface atoms and the p-orbitals of the inhibitors, their functional groups, and the possible steric effects. The result of the adsorption is a barrier film formation which obstructs the active surface sites and decreases the rate of corrosion by slowing down the cathodic or anodic reaction, diffusion of the aggressive species to the metal’s surface and reducing the electrical resistance of the surface of the metal. There are mainly two types of adsorption of corrosion inhibitors on the surface of a metal: physical adsorption (physisorption) and chemical adsorption (chemisorption). Physisorption is due to the electrostatic interaction between the charged metal surface and charged centers of the molecules which leads to dipole interaction of the molecules and metal surface while chemisorption is a process that involves sharing or transferring electrons from the inhibitor to the metallic surface which leads to the formation of the coordinate covalent bond and the strength of the bonding is much larger than that of physisorption. This article discusses the various forms of aluminium alloy corrosion in both acidic and alkaline environments and how to mitigate it using a plant extract called ferns.

**EXPERIMENTAL**

**Materials and Methods**

The plant extract was obtained from Fern. The leaves of the fern were obtained from Lagos, Western Nigeria. The reagents used were the analytical grade of 1M HCl, 1M NaOH, 96% Ethanol (Absolute Ensure, Germany) and 99% purity Acetone (Fischer Scientific). Rotary Evaporator used to separate the solvent from the extract was a product of Bibby Scientific Limited, UK. The water Bath - Cliffon SBS40 was used for heating the solute to further concentrate the solute (extract) to paste.

**Preparation of Coupon**

Aluminium alloy was utilized as a test material for this research. The aluminium alloy was mechanically cut to coupons of dimension, 4cm by 4cm with a thickness of 0.065cm and washed with distilled water. Silicon carbide (SiC) emery paper of grade No.600 was used to mechanically polish the square coupons before they were degreased using ethanol and acetone, respectively. The clean coupons were then exposed to air drying before they were immersed in both alkaline and acidic media. The blank corrodent was sodium hydroxide and hydrochloric acid. Analytical grade reagents were used throughout the experiment unless stated otherwise.

For use in this project, Aluminium was cut into rectangular coupons. The area of the specimen was obtained from the relation:

\[ A = 2(xy + yz + xz) \]  

Where \( A \) = area of specimen,
x = breadth of specimen = 4cm,
y = length of specimen = 4cm,
z = thickness of specimen = 0.065cm

Preparation of Fern Extract
The leaves of the fern were spread in a container and spread under the sun for drying. They pulverized to powder using a dry-blending machine. 300 g of the fern leaves powder was then weighed in a beaker to which 96% ethanol was added and allowed to soak for 72 hours. The mixture was filtered to obtain a particles-free filtrate. The filtrate was then taken to a rotary evaporator where it was concentrated to a paste-like slurry. The residue was soaked again in ethanol to obtain more of the plant extract which was filtered and concentrated as before. Varied weights (0.1 g – 0.5 g) of the inhibitor were measured and added to 1 M HCl solution in different beakers.

Phytochemical Analysis
The phytochemical analysis was carried out on the extract to be screened to determine the phytochemicals contained in the fern extract used for the inhibition process in this experiment. The Phyto-chemical analysis of the extracts was carried out in the Department of Biochemistry, Covenant University, Ota. Below is the tabulated procedure for the analysis.

Gravimetric Corrosion/Weight Loss Method
To undertake the corrosion study, five (5) separate beakers containing 100 ml each of 1 M hydrochloric acid (HCl) and 0.1, 0.2, 0.3, 0.4, and 0.5 g of fern extracts were clearly labeled. A sixth beaker without the fern extract was also set up and was marked as blank. Six metal coupons that were prepared were then weighed and placed in the six beakers. The beakers and their contents were maintained at room temperature. These metal coupons were retrieved progressively at 24 hours intervals for 7 days, washed with running water to remove any corroded product(s), dipped in a solvent (acetone), dried and then reweighed. The loss in weight (Wl) was recorded as the weight difference between the coupons (specimens) before and after they were immersed in the inhibitors and blank solution. A digital weighing balance of 4-decimal places was used to obtain weight loss.

The above experimental runs were repeated but this time 1 M sodium hydroxide instead of 1 M HCl solution was used.

0.1g inhibitor in 100 ml of the acid or alkaline solution gives a concentration of 1 g/l. The corresponding concentrations for the 0.2, 0.3, 0.4, and 0.5 g of fern or inhibitor in 100 ml of the acid or alkaline solution were calculated to give 2, 3, 4, and 5 g/l respectively.

From the corrosion study, the degree of surface coverage (θ), corrosion rate (CR), film attractive free energy (ΔGa), the efficiency of inhibition (IE), and adsorption equilibrium constant (K) were all determined.

Determination of the Rate of Corrosion, Degree of Surface Coverage, and Inhibition Efficiency

The Rate of Corrosion
The rate of corrosion (C.R.) was measured using eqn.-2 and it is expressed in millimeters penetration per year (mm/yr) for all the specimens under study.

\[
\text{C.R. (mm/yr)} = \frac{W}{At} \quad \text{(2)}
\]

Where

- \( W \) = weight loss (mg),
- \( t \) = exposure time (hr)
- \( A \) = total surface area of coupon respectively (cm²).

and

\[
W_L = W_I - W_f \quad \text{(3)}
\]

Where

- \( W_I \) is the initial weight of the coupons before immersion
- \( W_f \) is the final weight of the coupons after immersion

Inhibitor Efficiency
The inhibitor efficiency (% IE) was obtained using eqn.- 4.17
Inhibition efficiency (% IE) = \( 1 - \frac{CR_p}{CR_a} \times 100 \)  
\( (4) \)

Where  
\( CR_p \) = corrosion rates with inhibitor present in the test media  
\( CR_a \) = corrosion rates with the inhibitor absent (blank solution)

**Degree of Surface Coverage**

The extent or degree of surface coverage (\( \theta \)) of the aluminium coupons by the inhibitor’s molecules can be expressed using eqn.-5.

Degree of surface coverage  
\( \theta = 1 - \frac{CR_p}{CR_a} \)  
\( (5) \)

**Film Attractive Power**

From the degree of surface coverage (\( \theta \)) at any given temperature and inhibitor’s concentration, the attractive film power was computed using eqn.-6.\(^{16}\)

\[ K_{ads} = \frac{\theta}{C(1-\theta)} \]  
\( (6) \)

Where,  
\( K_{ads} = \) film attractive power at the adsorption equilibrium constant  
\( \theta \) gives the degree of surface coverage (\( \theta \))  
\( C \) gives inhibitor’s concentration.

**Free energy of Adsorption \( \Delta G_{ads} \)**

This measures the energy that is available to do work outside the system. It is the driving force behind a process and determines the spontaneity of a reaction. Equation-7 was used to obtain the free energy of adsorption (\( \Delta G_{ads} \)) at room temperature.\(^{19}\)

\[ \Delta G_{ads} = -RT\ln(55.5 \ K_{ads}) \]  
\( (7) \)

Where  
\( \Delta G_{ads} = \) Gibbs energy of adsorption (KJ/mole).  
\( R \) = the universal gas constant (8.314J/molK).  
\( T \) = the absolute temperature (K).  
\( K_{ads} = \) the adsorption equilibrium constant

**Determination of the Adsorption Isotherm**

**Langmuir Adsorption Isotherm**

The data generated from the weight loss study were fitted into the Langmuir adsorption isotherm by using eqn.-8\(^{20}\).

\[ \frac{C}{\theta} = \frac{1}{K_{ads}} + C \]  
\( (8) \)

Where  
\( K_{ads} = \) the adsorption equilibrium constant.  
\( \theta = \) Surface coverage  
\( C = \) the concentration of the inhibitor

**Temkin Adsorption Isotherm**

For this type of adsorption isotherm, eqn.-9 was used to relate the degree of surface coverage (\( \theta \)) to the concentration (\( C \)) of the inhibitor used.\(^{21}\)

\[ \exp(-2a\theta) = KC \]  
\( (9) \)

Where  
\( a = \) the attractive parameter  
\( K = \) the adsorption equilibrium constant.

and taking the natural logarithm of both sides of eqn.-9 and rearranging results in eqn.-10.

\[ \theta = \frac{-2.30310\log K}{2a} - \frac{2.30}{2a} \]  
\( (10) \)
The Frumkin Isotherm
Equation-11 was used to describe the Frumkin adsorption isotherm:\textsuperscript{20}
\[
\log \left( C \times \frac{\theta}{1-\theta} \right) = 2.303 \log K + 2\alpha \theta
\] (11)
Where
\( \theta \) = Surface coverage
C = the concentration of the inhibitor
K = the adsorption equilibrium constant
\( \alpha \) = the lateral interaction term describing the interaction in adsorbed layer.

RESULTS AND DISCUSSION
The total geometric surface area of the specimen (the coupon) was found to be 32.52cm\(^2\). Table-1 represents the chemical compounds that are present and absent in each of the plant extracts after the phytochemical analysis was successfully carried out on the plant extract. The results show that fern extract contained the following chemical constituents; Alkaloids, Tannins, Saponins, Phenols, Terpenoids, Quinones, Cardiac Glycosides except for Flavonoids, Carbohydrates and Glycosides. The presence of chemical compounds such as Tannins, Alkaloids, Cardiac Glycosides, Saponins in the leaf of the fern resulted in a low corrosion rate in the acid medium when compared with the blank medium. The presence of functional groups such as Nitrogen (N), Sulphur, and Oxygen (O) in molecules of the fern extracts serves as the active centers that will facilitate inhibitor’s adsorption onto the surface of the metal as a result of their higher electron density and basicity.

| Chemical Constituents | Fern Leaves Extracts |
|-----------------------|----------------------|
| Tannin                | +                    |
| Alkaloids             | +                    |
| Flavonoids            | -                    |
| Saponins              | +                    |
| Cardiac glycosides    | +                    |
| Glycosides            | -                    |
| Terpenoids            | +                    |
| Phenol                | +                    |
| Quinones              | +                    |

Key
+ represents Presence
- represents Absence

The availability of lone pair and p-electrons in the functional groups contained in the inhibitors helps to facilitate electron transfer from the inhibitor to the surface of the metal. The above findings are in agreement with the reported results.\textsuperscript{19,22}

Figures-1 and 2 are the graphical representations of the relationship between corrosion and the time of immersion of coupons in both media, which are the solutions without and with the inhibitor or plant extract of various concentrations at an average ambient temperature of 27\(^\circ\)C.

Weight Loss
Figures-1 to 3 shows the corrosion rate (mm/yr) for aluminium specimen immersed in 1M HCl and 1M NaOH for 7 days at 1-day intervals for each of the six samples as a function of exposure time at the ambient temperature of 27\(^\circ\)C used. The results show that the rate of corrosion of aluminium alloy in 1M NaOH was higher than in 1M HCl, this signifies that at the same concentration of alkaline and acidic media, aluminium alloy corrodes more in alkaline medium (Fig.-3) and it shows that the corrosion inhibition performance in an acidic environment is better than in the alkaline environment. The results show that the rate of corrosion decreased as the days increased from 1 day to 7 days for the different concentrations of extracts ranging from 1.5 g/l. This behavior may be attributed to the higher adsorption level of the active inhibitor molecules on the metal surface by forming a thin film on the metal surface to prevent it from further attack. It is this
action of the inhibitor that results in the reduction in the weight loss of the specimen, which agrees with the earlier findings.19

Fig.-1: Corrosion Rate-time Curve of Aluminium in 1M Hydrochloric Acid (HCl) in the absence and presence of varied Concentrations of Fern Extract at Room Temperature (27°C)

Fig.-2: Corrosion Rate-time Curve of Aluminium Alloy in 1M Sodium Hydroxide (NaOH) in the absence and presence of varying Concentrations of Fern Extract

Fig.-3: Corrosion Rates of Various Concentrations of Fern Extract in 1M NaOH and 1M HCl on Aluminium Alloy

**Effect of Inhibitor Concentration**

Figures-4 and 5 represent the graphs of the percentage inhibition efficiency versus the concentration of the inhibitors for the corrosion of the aluminium alloy in both the 1M NaOH and 1M HCl solutions at room temperature (27°C). An increase in the concentration of the inhibitors in the acidic and alkaline media
resulted in adsorption of more active ions from the fern extract on the surface of the specimen with the acidic and alkaline environment which led to an increase in percentage inhibition efficiency and the reduction in a weight loss of the specimen. This implies that the higher the concentration of the fern extract in HCl and NaOH solution, the better the inhibition efficiency of aluminium in both media. This is in agreement with the result obtained.\textsuperscript{19,22}

**Fig.-2:** Percentage Inhibition Efficiency (% IE) with varied Concentrations of Fern Extract or Inhibitor in 1 M HCl

**Fig.-3:** Percentage Inhibition Efficiency (% IE) with varied Concentrations of Fern Extract or Inhibitor in 1M NaOH

**Thermodynamic Parameters and Adsorption Isotherm**

Figures 6-11 represent the plots of the various isotherms fitted with experimental data while the thermodynamic parameters – adsorption equilibrium constants, the slopes and the correlation coefficient derived from the isotherms, are tabulated in Tables-2 to 4. From the regression coefficients or $R^2$ values shown in Tables-2 to 4, it can be inferred that the adsorption of the corrosion inhibitor or extract fitted the Langmuir, Temkin, and Frumkin adsorption isotherms. However, the Langmuir isotherm produced the overall best fit of the experimental data with an $R^2$ value of 0.9999 in both acid and alkaline media. The linearization of the nonlinear isotherm equations has, however, been argued to alter their error structure violate the error variance, and the normality assumptions of standard least squares.\textsuperscript{25}

The adsorption equilibrium constants ($K_{ads}$) represent the strength of adsorption between the adsorbent and adsorbate.\textsuperscript{24} The Larger the values of the $K_{ads}$ the more favorable is the adsorption and this implies better inhibition efficiency of the leaf extract. The various values of $K_{ads}$ for the extract are shown in Tables-2 to 4. Figures-6 and 7 represent the linearized plots of $C/\theta$ versus the inhibitor’s concentrations ($C$) for both the HCl and NaOH media. From the Langmuir isotherm, the reciprocal of the intercepts of the plotted lines give the adsorption equilibrium constant ($K_{ads}$) and it signifies the extent or degree of adsorption. Figures 8
and 9 show the linear plots of $\theta$ against log $C$ (concentration) for both HCl and NaOH media with $K_{ads}$ obtained from the intercept of Temkin plotted lines using eqn.-10. Figures-10 and 11 show the linear plots of log ($C^* (\theta/(1-\theta))$) versus 0 for both the HCl and NaOH media with $K_{ads}$ obtained from the intercept of Frumkin plotted lines using eqn.-11. From all adsorption isotherms, the value of $K_{ads}$ was higher for Langmuir isotherm with its slope and linear regression coefficient $R^2$ nearly equal to unity, which signifies that every molecule of the extract (inhibitor) occupies an active site on the metal surface, resulting in strong adsorption of the inhibitors. This is a strong indication of the adherence of the adsorption assumption data for the Langmuir isotherm. The values of ‘a’, known as the molecular interaction parameter, obtained from the Temkin adsorption isotherm, were observed to be negative and they indicate a repulsive effect at the molecular level within the adsorbed layer of the adsorbate. The adsorption process has the basic features of physical adsorption; hence it cannot be fitted or modeled using the Temkin isotherm because it is usually used for modeling or describing chemisorption or chemical adsorption. Also, the lateral interaction parameter ($\alpha$) from the Frumkin isotherm gave positive values, which indicate attractive interactions in the adsorbate layer, and therefore, it cannot be applied or modeled using the Frumkin isotherm. Equation-7 was used to calculate $\Delta G_{ads}$ - the Gibbs free energy of adsorption and the values are tabulated in tables 2-4. Except for the Frumkin isotherm, the values of the $\Delta G_{ads}$ for Langmuir and Temkin isotherms were all negative. The negative values infer that the adsorption of the corrosion inhibitor onto the metal or aluminium alloy’s surface occurs spontaneously and it gives an indication of strong interactions between the molecules of the extract and the surface of the aluminium alloy. This observation is in agreement with the work of other researchers. In general, when the values of the Gibbs free energy of adsorption ($\Delta G_{ads}$) is less or equal to $-20\text{kJ/mol}$, the electrostatic interactions exist between the charged molecules of the extract (inhibitor) and those of the charged metal surface, in which case, physisorption or physical adsorption is said to take place. But when $\Delta G_{ads}$ are more negative, greater than $-40\text{kJ/mol}$, then charge transfer or sharing between the extract or molecules of the inhibitor and that of the surface of the metal takes place to form a coordinate bonding known as chemical adsorption or chemisorption. From Tables 2-4, the values of $\Delta G_{ads}$ obtained from the isotherms show that they are less than $-20\text{kJ/mol}$, which validates the experimental data that the inhibition mechanism of aluminium alloy in the two media of 1M NaOH and 1M HCl solution is physisorption at room temperature (27°C).

![Image](image.png)

**Fig.-4: Data Fit for Langmuir Isotherm for the Corrosion Study of Aluminium Alloy using varied Concentrations of Fern Extract in 1M HCl**

| Medium  | Linear Regression Coefficient ($R^2$) | $K_{ads}$ | $\Delta G_{ads}$ (kJ/mol) |
|---------|-------------------------------------|-----------|--------------------------|
| 1M HCL  | 0.9999                              | 3.2744    | -12.9761                 |
| 1M NaOH | 0.9999                              | 3.0497    | -12.7988                 |

Table-2: Values of Thermodynamics Parameters from the Langmuir Adsorption Isotherms for the Adsorption of Fern Extract on Aluminium Alloy in Both Acidic and Alkali Media
Fig.-5: Data Fit for Langmuir Isotherm for the Corrosion Study of Aluminium Alloy using varied Concentrations of Fern Extract in 1M NaOH

Fig.-6: Data Fit for Temkin Isotherm for the Corrosion Study of Aluminium Alloy using varied Concentrations of Fern Extract in 1M HCl

Fig.-7: Data Fit for Temkin Isotherm for the Corrosion Study of Aluminium Alloy using varied Concentrations of Fern Extract in 1M NaOH

Fig.-8: Data Fit for Frumkin Isotherm for the Corrosion Study of Aluminium Alloy using varied Concentrations of Fern Extract in 1M HCl
CONCLUSION

The plant extract was successfully formulated from the leaf of ferns as a corrosion inhibitor. The ethanolic extract of fern leaves has proved to be a good corrosion inhibitor for aluminium alloy in NaOH and HCl media. The inhibitive efficiency of the fern extract at room temperature is higher in the hydrochloric acid (HCl) medium than in the sodium hydroxide (NaOH) medium. The inhibition efficiency of the fern extract (inhibitor) increases with its increasing concentration. The negative values of the Gibbs free energy of adsorption portray the spontaneous interaction with the high adsorptive ability of the extract on the aluminium at ambient temperature, which is consistent with the Langmuir isotherm. At the same concentration, aluminium corrodes at a faster rate in an alkaline medium than in an acidic medium. The adsorption mechanism of inhibition of aluminium in both 1M HCl and 1M NaOH solutions is Physisorption.

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