



## Adsorption and Corrosion Inhibition of *Epiphyllum oxypetalum* Leaf Extract on Aluminum Alloy AA5052

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### Authors' contributions

This work was carried out in collaboration between all authors. Authors FON and OOI designed the study. Author FON performed the experiment under the supervision of authors OOI and EO did the statistical analysis, wrote the protocol, and the first draft of the manuscript. Author FON managed the analyses of the study. All authors read and approved the final manuscript.

### Article Information

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### ABSTRACT

**Aim:** The corrosion inhibition mechanism of aluminium Alloy AA5052 has been studied in acidic solution. The study used gravimetric and electrochemical impedance spectroscopy to verify the inhibition efficacy of *Epiphyllum Oxypetalum* Leaf Extract At Varied Concentrations.

**Study Design:** Laboratory research was conducted at a maximum exposure time of nine (9) hours.

**Place and Duration of Study:** The research was carried out at Material Science group laboratory, Abia State Polytechnics, Aba Abia State, Nigeria between May 2018 and July 2018.

**Methodology:** Gravimetric technique was employed at varied temperatures to note the general inhibitive behaviour of the leaf. Electrochemical Impedance Spectroscopy (EIS) technique was employed to determine the electrochemistry process. The leaf extraction was done with reflux set up under 65°C.

**Results:** An Optimal Inhibition Efficiency Value of 97% was obtained. The inhibition efficiency increased with increasing concentration of the inhibitor but decreases slightly with the increase in temperature. The electrochemical studies showed that the green leaf extract was able to alter the electrochemical mechanism of the corrosion process. The corrosion current densities  $i_{corr}$  and mass

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electron/charge transfer were observed to reduce due to the presence of the inhibitor in the test solution. The thermodynamic parameters were studied. Adsorption mechanism of the inhibitor molecules obeyed Langmuir and Temkin adsorption models. The calculated adsorption Gibbs free energy is negative showing that the adsorption process was spontaneous. OPTICAL micrograph was used to study the surface morphology of the metal surface. Vickers indentation studied on the metal showed how the inhibition process improved the mechanical hardness of the corroded metal. **Conclusion:** *Epiphyllum oxypetalum* extract has been developed as a corrosion inhibitor for aluminium alloy AA5052 in the hydrochloric acidic environment.

**Keywords:** Adsorption; *Epiphyllum oxypetalum*; corrosion inhibition.

## 1. INTRODUCTION

In many ways, man and nature fight over the state of materials, especially metals. Nature aims at returning material to their naturally occurring state called ore while man through scientific and technological research tends to preserve materials for the good purpose of applications. Metals in service often give a superficial impression of permanence. However, corrosion is one of the basic ways materials are affected in service. It's been defined by William and David [1] as a destructive and unintentional attack of a metal; it is electrochemical and ordinarily begins at the surface. Also, corrosion is an electrochemical phenomenon by which metallic structures are damaged slowly through anodic dissolution. In a statement offered by Avwiri and Osarolube [2] has it that corrosion of metals is determined by the type of environment the metal is in contact with. One of the major backdrops of corrosion is in its ability to disrupt the inherent properties of metals. Interestingly, the corrosion process causes a microstructural change of material. Such change in great length affects the mechanical and physical properties of metals and alloys.

Aluminium (Al) and its alloys are low cost and remarkable materials in industrial technology because of their lightweight, high thermal and electrical conductivity as well as high resistance to corrosion in a wide variety of corrosive environments [3]. Generally, the corrosion resistance of metals, such as Al and steel, in corrosive environments, may be attributed to the formation of a protective tightly adhered invisible oxide film on the metal surface. The film reduces or prevents the corrosion of such metals. This film is generally stable in the solutions of pH ranges of about 4.5 to 8.5 [4]. However, due to the solubility of the film in strong acidic or alkaline solutions, the metal shows a high rate of corrosion and dissolution in these conditions.

Therefore, inhibitors are used to control both metal corrosion and acid consumption [5].

The use of inhibitors for the control of corrosion of metals and alloys which are in contact with an aggressive environment is an accepted practice [6]. It is pertinent to note that one of the interesting ways to mitigate electrochemical corrosion of aluminum is to isolate the metal from corrosive agents and this can be achieved by using corrosion inhibitors which prevent the adsorption of the aggressive anions or by the formation of a more resistant oxide film on the metal surface. In a contribution by Fouda et al. [7] based on assumptions suggested that the first stage in the action mechanism of the inhibitors in aggressive acid media as hydrochloric acid is the adsorption of the inhibitors on the metal surface where HCl solutions are used for pickling [8] of aluminium for its chemical or electrochemical etching. However, the adsorption of an inhibitor on a metal surface which results on the inhibition mechanism depends on a few factors, such as the nature and surface charge of the metal, the adsorption mode, the inhibitor's chemical structure, and the type of the electrolyte solution [9].

In this work, we investigate the mechanism of inhibition of *Epiphyllum oxypetalum* (*E. oxypetalum*) leaf extract on the corrosion of aluminium alloy AA5042 in HCl solution using gravimetric and electrochemical impedance spectroscopy (EIS) technique.

## 2. EXPERIMENTAL

### 2.1 Materials Preparation

Aluminium alloy sheet of purity 98.8%, mechanically cut into 20 mm x 20 mm x 10 mm coupon size was used for this study. A hole of 2 mm was drilled to facilitate suspension of the coupon in the test solution and further prepared

for gravimetric and electrochemical impedance spectroscopy (EIS) according to earlier methods employed by Alinnor and Ejikeme [11], Ambrish and Quraishi [12], Nwosu et al. [13]. The test solution, 1.0 M HCl solution was prepared and used for reflux extraction of the green plant produce. Further dilution of the inhibitor to various concentrations was made.

The leaf of *E. oxypetalum* known as the queen of the night [10] was harvested within University of Port Harcourt (UPH) Choba Rivers State Nigeria environs and prepared according to Nwosu and Osarolube [14], Nnanna and Owate [15], Iroha and Hamilton-Amachree [16] to different test solution concentrations 0.0, 0.1, 0.2, 0.3, 0.4, and 0.5 g/L.

## 2.2 Gravimetric Technique

A gravimetric technique otherwise known as weight loss method was used to primarily note the mass of the Al alloy degraded in the test solution during the exposure period. The prepared coupons were first weighed using FA2104A analytical electronic digital weighing balance: sensitivity of 0.0001. Then, the coupons were fully immersed in a 500 mL beaker that contained 350 mL of the test solution. The water bath was used to keep the temperature variations of the system at constant 30°C and 60°C. The systems (both inhibited and inhibitor-free) were left unstirred for the corrosion inhibition process to take place for a maximum period of eight hours and each coupon retrieved at every one-hour exposure.

After each exposure time, the AA5052 coupons were removed, washed thoroughly to remove the corrosion product, rinsed with distilled water and dried in acetone as previously reported Umoren et al. [17], Noor and Al-Moubaraki [18]. The metal alloy was reweighed to determine the weight loss. This process was repeated in triplicate and the mean value taken for reproducibility. The weight loss measurements were used to calculate the corrosion rate,  $C_r$  (equation 1) of the Al alloy in the acidic medium.

$$C_r = \frac{87.6 \Delta W}{A \rho t} \quad (1)$$

Where  $\Delta W$  is the weight loss (in grams),  $A$  is the surface area of the immersed coupon (in  $\text{cm}^2$ ,  $\rho$  is the coupon density ( $\text{g/mm}^3$ ), and  $t$  is the exposure period (in hours) and  $C_r$  is the corrosion rate (in  $\text{mm.y}^{-1}$ ).

## 2.3 Electrochemical Measurements

The potentiodynamic polarisation test was carried out in a standard three-electrode glass cell of 500 mL capacity using Electrochemical System workstation (PAR 263). A graphite rod served as a counter electrode and, a saturated calomel electrode (SCE) was used as reference electrodes. The prepared coupons serve as the working electrode. Electrochemical measurements were carried out at  $30 \pm 1^\circ\text{C}$ , using standard procedures as outlined by Oguzie et al. [19], Shaju et al. [20], in aerated solutions at the end of 1800 seconds of immersion, which allowed the open circuit potential (OCP) values to attain a steady state. The potentiodynamic polarisation (PDP) experiments were then conducted at a scan rate of 0.333 mV/s. The potential range employed was -25 V to + 30 Volts versus corrosion potential. The power suit software was used in analysing the polarisation data. Tafel polarisation analysis was done by extrapolating anodic and cathodic curves to obtain corrosion current densities ( $I_{\text{corr}}$ ). The percentage of inhibition efficiency (%) was evaluated from the measured  $I_{\text{corr}}$  values using the following equation 4 [21].

## 2.4 Mechanical Hardness Test

The Hardness mechanical tests were performed on the coupons before and after the corrosion exercise. A load of 10 N was used. Vickers micro indentation tests were performed with a load of 10 N at different locations on the metal surface as reported earlier by Nwosu et al. [22]. Five different indentations were made on the coupons and the average value reported.

## 3. RESULTS AND ANALYSIS

### 3.1 Weight Loss, Corrosion Rate, and Inhibition Efficiency

The weight loss, corrosion rate, and inhibition efficiency (equation 2) [23] were calculated from the gravimetric measurements. Fig. 1 shows the weight loss variations to the exposure time. The plot (Fig. 1) suggests that at the early hours of the corrosion process, the inhibitor had neither little nor nothing to offer to the resistance of the metal. But, over time, the inhibitor was found to limit the weight loss of the Al alloy in the 1.0 M HCl solution. The weight loss of AA5052 in the inhibited system reduced with increasing inhibitor concentration. The inhibitor-free system was

found to degrade more than the inhibited system. Thus, the presence of the inhibitor quite decreases the material degradation.

*oxypetalum* greatly reduced compared to the inhibitor-free system. However, the Al alloy exhibits a sluggish sinusoidal curve which could be attributed to the *passivity* associated with the parent metal, aluminium. The rate of the corrosion attack was observed to reduce with increase in the inhibitor concentration.

The corrosion rate of AA5052 reported in Fig. 2 shows that the corrosion rates of the coupons immersed in different concentrations of *E.*

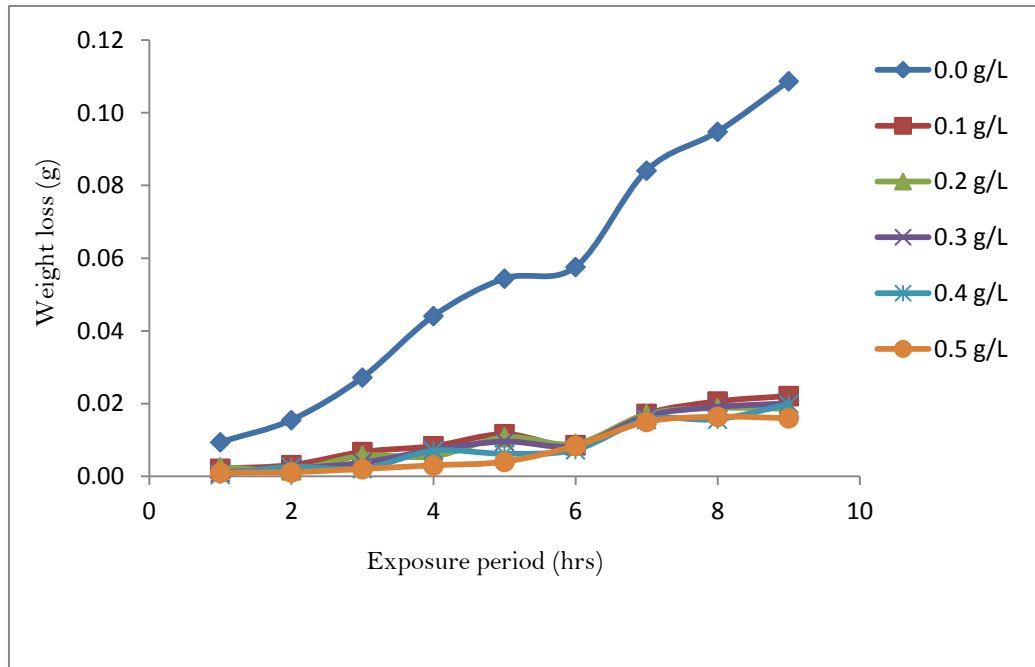


Fig. 1. Weight loss against exposure period of AA5052 immersed in 1.0 M HCl in the presence of *Epiphyllum oxypetalum* extract

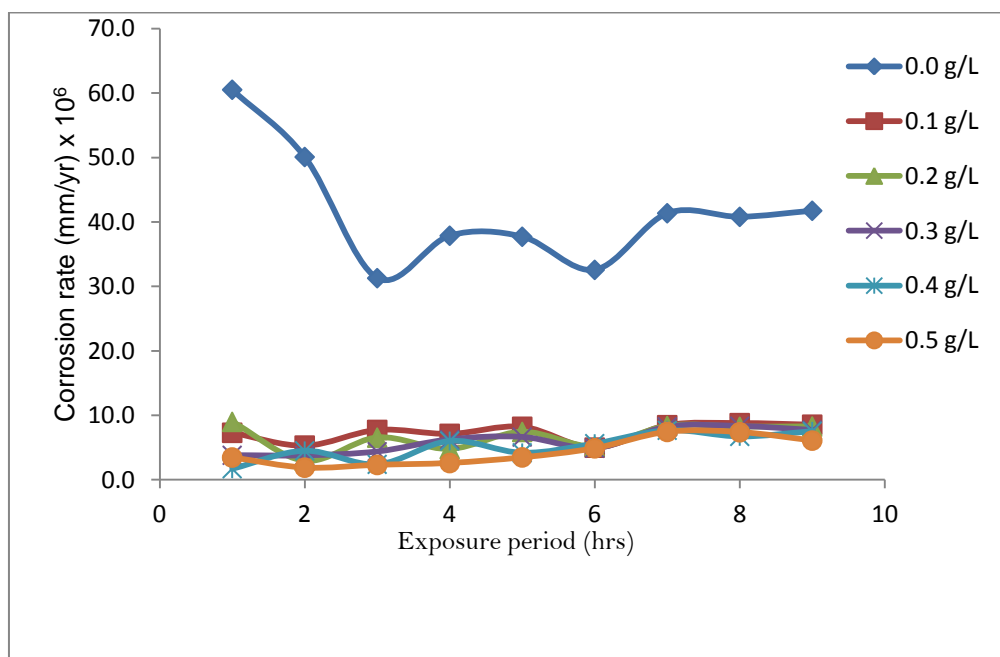


Fig. 2. Corrosion rate against exposure period of AA5052 in 1.0 M HCl in the presence of *Epiphyllum oxypetalum* extract

The efficacy of the leaf extract to inhibit the degradation of AA5052 is shown on Fig. 3.

$$\eta\% = \left(1 - \frac{W_i}{W_{i-f}}\right) \times 100 \quad (2)$$

Where  $\eta\%$  is the inhibition efficiency,  $W_i$  is the weight loss of the inhibited system,  $W_{i-f}$  is the weight loss of the inhibitor-free system. The inhibition efficiency of *E. oxypetalum* extract was found to increase with increasing concentration but exhibits an inverse relationship with temperature increase. This could be due to a sort of dissolution of the inhibitor molecule at high temperature which caused a loss of potency to protect the surface of the metal. However, even at a high temperature, the inhibitor proved to be useful as it recorded efficiency optimal value of 79%.

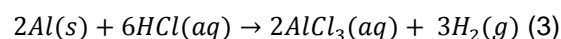
### 3.2 Potentiodynamic Polarisation (PDP) Study

The potentiodynamic polarisation curves of AA5052 in 1.0 M HCl solutions containing different concentrations of the green inhibitor at room temperature ( $30 \pm 1^\circ\text{C}$ ) are shown in Fig. 4. The corrosion kinetic parameters such as current density ( $i_{\text{corr}}$ ), corrosion potential ( $E_{\text{cor}}$ ), the anodic Tafel slope ( $b_a$ ), cathodic Tafel slope ( $b_c$ ), the degree of surface coverage ( $\Theta$ ) and the inhibition efficiency (%IE) for the green leaf extract are shown in Table 1. The corrosion

current densities were estimated by Tafel extrapolation of the cathodic and anodic curves to the open circuit corrosion potential.

Table 1 show that the values of the current density decreased with increasing inhibitor concentration. This indicates that there was barrier increase to the free-flow of currents between the electrodes through the environment. Conversely, the presence of *E. oxypetalum* in the solution altered the electrochemistry of the corrosion process. The cathodic Tafel slopes were decreased.

The result confirms the eye observation of a reduction of gas evolution in the inhibited system. Thus, green leaf extract influences the kinetic dissolution of the hydrogen evolution reaction (equation 3). Also, the anodic Tafel slopes were decreased. This result shows that the kinetics of the corrosion of the metal AA5052 was influenced by the actions of the inhibitor. The corrosion inhibitions for the advanced studies were calculated using equation 4 [24].



$$\% \eta = \left( \frac{i_{\text{corr}(bl)} - i_{\text{corr}(inh)}}{i_{\text{corr}(bl)}} \right) \times 100 \quad (4)$$

Where  $i_{\text{corr}(bl)}$  and  $i_{\text{corr}(inh)}$  represents the corrosion current density in the absence and presence of the inhibitor, respectively.

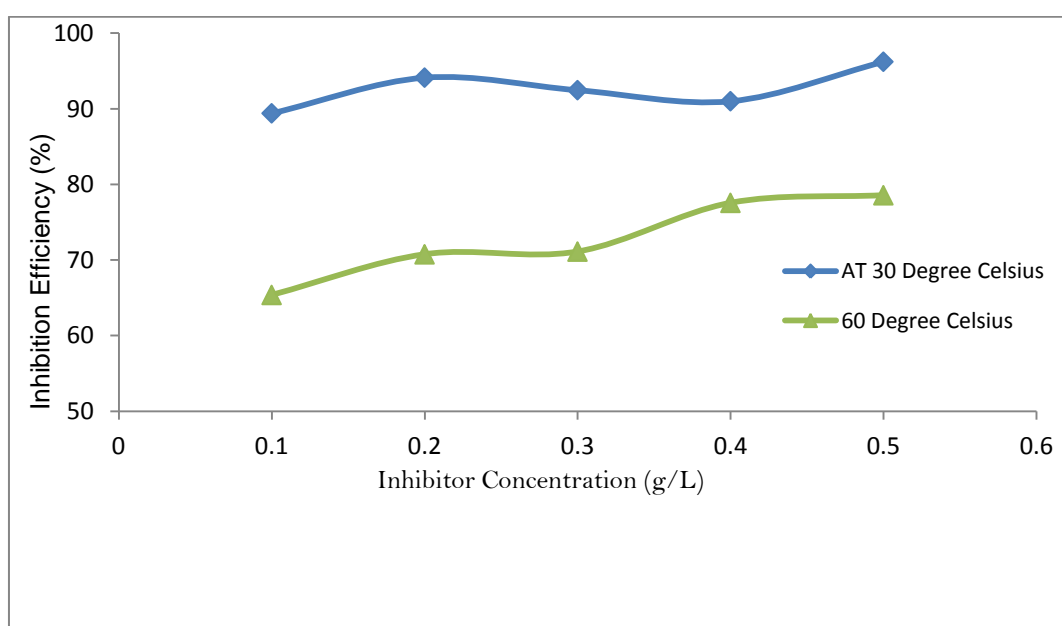


Fig. 3. Temperation of the Inhibition efficiency of *E. oxypetalum* extract in 1.0 M HCl

**Table 1. Polarization parameters for the AA5052 aluminium alloy in the absence and presence of *E. oxypetalum* in 1.0 M HCl solution**

System (g/L)	$I_{corr}$ ( $\mu\text{A}/\text{cm}^2$ )	$E_{corr}$ (mV)	$b_a$ (mV/dec)	$b_c$ (mV/dec)	Surface coverage, $\Theta$	$\eta$ (%)
Blank	192.5	-745	115.3	121.4	-	-
0.1	61.2	-656.4	97.6	109.8	0.682	68.2
0.2	43.6	-734.5	90.6	104.6	0.774	77.4
0.3	33.5	-796.5	89.5	100.3	0.826	82.6
0.4	30.9	-743.4	84.4	97.2	0.839	83.9
0.5	20.6	-764.5	80.7	96.6	0.893	89.3

Further, the value of the surface coverage and the inhibition efficiency were found to increase with increasing inhibitor concentrations. The *E. oxypetalum* extract is a mixed inhibitor through its ability to decrease both Tafel slopes.

The polarization curve (Fig. 4) shows that the cathodic reaction was inhibited by *E. oxypetalum*. This was observed in the shift of  $E_{corr}$  towards the more negative potential. The inhibition could be due to either or combination of the following: increase in cathodic and/or anodic overvoltage, adsorption of the inhibitor molecules or its ion on anodic and/or cathodic sites, and the formation of a protective barrier film [25]. The addition of the inhibitor further reduces the attack on the metal surface by not encouraging electron charge transfer in the redox process.

### 3.3 Adsorption Parameters and Thermodynamic Considerations

Corrosion inhibition is a surface phenomenon. The study of the adsorption models becomes pertinent in the present study. It is expected that the molecules of the inhibitor be attracted and adhered to the surface of the metal thereby limiting the access the corrosion active agents have to the metal surface. Adsorption models employed to study the effects of *E. oxypetalum* on AA5052 are Langmuir (equation 5) and Temkin (equation 6) among other isotherms tested. The models and the consequent thermodynamic derivations provide in-depth information into the mechanism of adsorption mode of attraction and adsorption strength [26-27]. Langmuir adsorption isotherm due to its comparatively higher correlation coefficient value.

$$\frac{c}{\theta} = \frac{1}{K_{ads}} + C \quad (5)$$

$$\theta = (1/f) \text{Log} C + (1/f) \text{Log} K_{ads} \quad (6)$$

Where  $c$  is the inhibitor concentration,  $\Theta$  is the surface coverage,  $K_{ads}$  is the thermodynamic equilibrium constant,  $f$  is the interaction parameter (which describes if the interaction between the lateral adsorbing molecules is repulsive or attractive).

The plot of  $C/\Theta$  against  $C$  (Fig. 5) of the green extract is linear with slope of 1.04 and correlation,  $R^2$  of 0.997. The  $R^2$  close to unity indicates strong adherence to Langmuir isotherm. Thus, *E. oxypetalum* leaf extract obeyed the Langmuir adsorption isotherm in 1.0 HCl on AA5052. The equilibrium constant for the adsorption process from Langmuir isotherm is related to the standard free energy of adsorption,  $\Delta G_{ads}$  by the expression 7 [28,29]. The Gibb's free energy was adopted to provide more insight into the thermodynamics of the corrosion process.  $\Delta G_{ads}$  values depend on metallurgical variables such as micro-pits, non-metallic impurities, elemental arrangements, microscopic fissures and crevices concerning grain orientation on the substrate [30].

$$\Delta G_{ads} = -2.303RT \text{Log}(55.5K_{ads}) \quad (7)$$

Where  $R$  is the universal gas constant (8.314 KJ/mol),  $T$  is the temperature of the system under study, and 55.5 is the concentration of water solution in (mol/L). The value of  $\Delta G_{ads}$  is -22.29 KJ/mol. The calculated value is below the threshold for chemical adsorption behaviour, thus the mechanism of the corrosion inhibition is through physical adsorption of the molecules on the metal surface. The negative sign expressed spontaneity adsorption of the inhibition process.

The Temkin adsorption isotherm relates the degree of surface coverage with inhibitor concentration. The plot of surface coverage,  $\Theta$  against  $\ln C$  from the weight loss method gives a straight line curve. The curve is found to conform to Temkin's model. The calculated  $f$  is 8.33 and the  $K_{ads}$  is 6.966 KJ/mol. From Temkin plot, it

was observed that the corrosion inhibition by inhibitor compounds is being a result of their adsorption on the metal surface.

### 3.4 Surface Screening

The scanning electron microscope image (SEM) of AA5052 immersed in 1.0 M solution with and without inhibitors are presented in Fig. 7.

The micrograph of the uninhibited coupon (Fig. 7a) was greatly attacked leading to a high density of porosity. The surface of the metal was exposed given the yellowish nature of the surface. Fig. 7b shows sort of film coverage hindering the appearance of the yellowish nature of the surface. This suggests that the molecules of the inhibitor adsorbed on the metal surface.

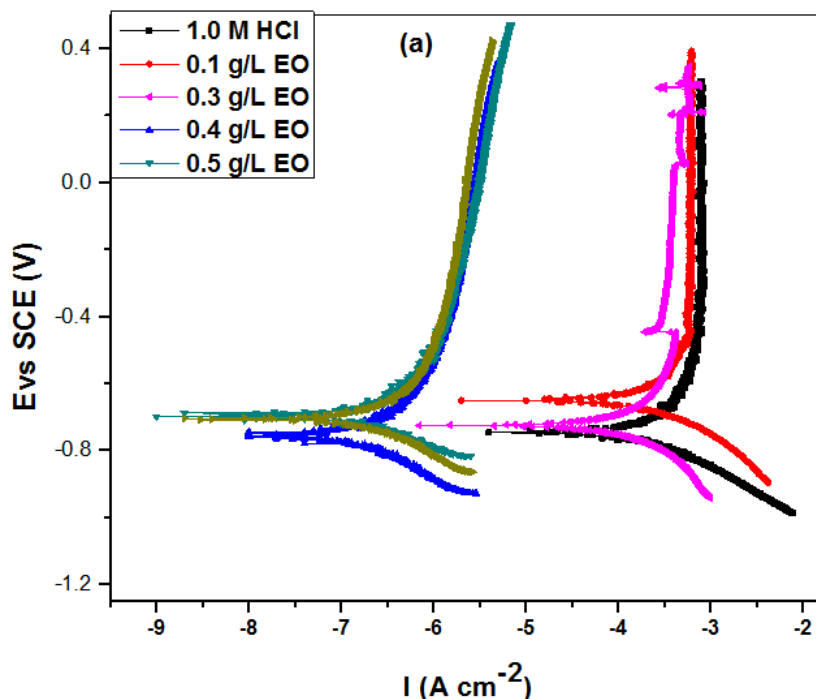


Fig. 4. Potentiodynamic polarisation curves of Aluminum in the absence and presence of *E. oxypetalum* in 1.0 M HCl solution

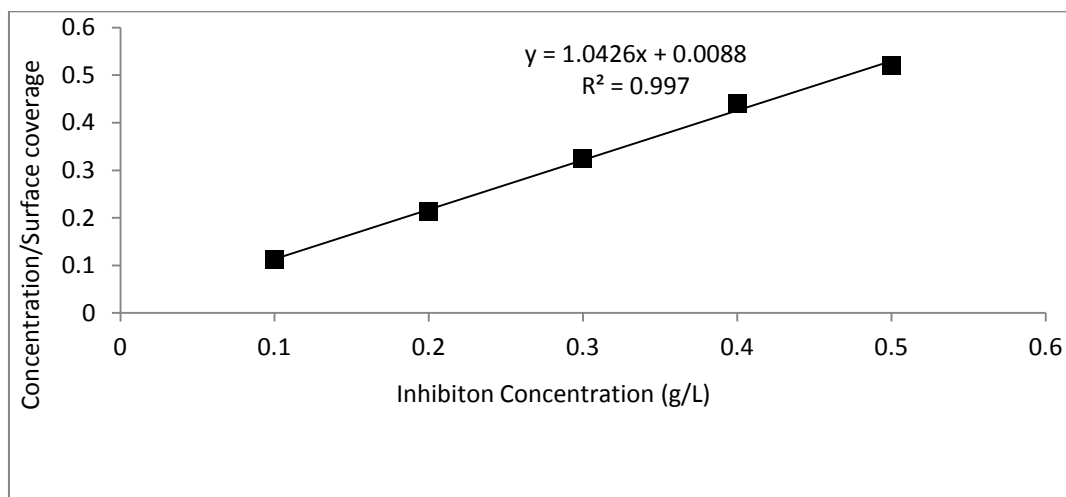
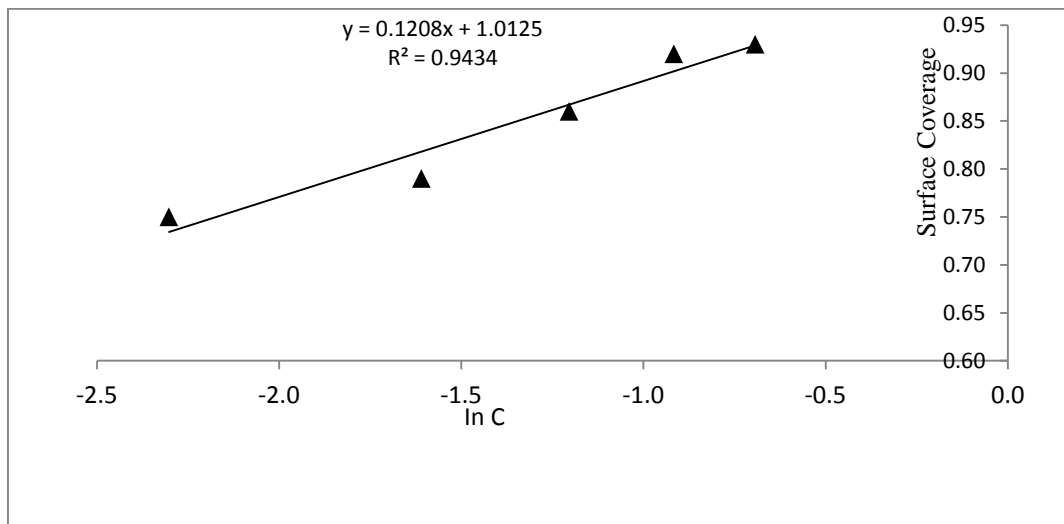
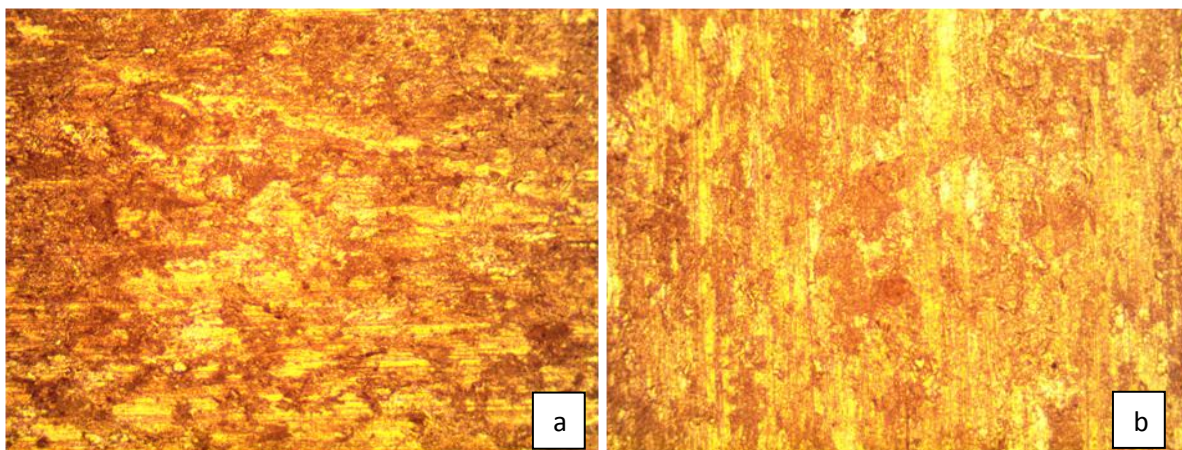


Fig. 5. Langmuir Adsorption Isotherm model of *E. oxypetalum* on AA5052 in 1.0 M HCl environment



**Fig. 6. Temkin adsorption isotherm model of *Epiphyllum oxypetalum* leaf extract on Al Alloy AA5052 in 1.0 M HCl environment**



**Fig. 7. Micrograph of AA5052 alloy immersed in 1.0 M HCl without inhibitor (a) and with *E. oxypetalum* extracts (b)**

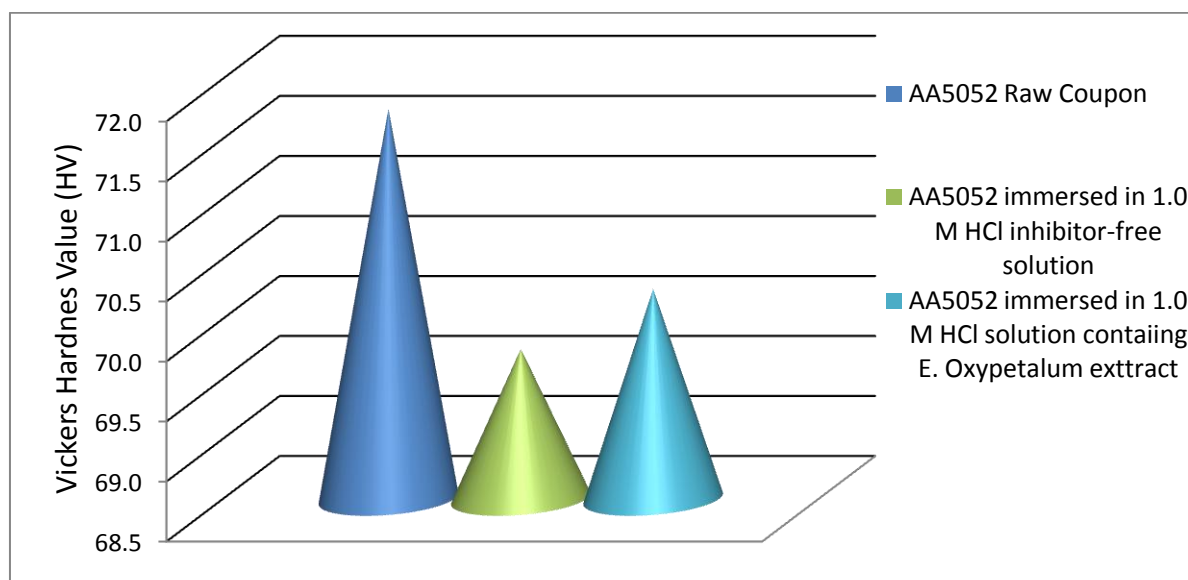
Furthermore, it could be possible that the parent metal must have undergone passivity. The formation of thin film oxide (aluminium oxide) on the metal surface could be another contribution to the barrier created between the metal surface and the active environment.

### 3.5 Mechanical Properties

The Vickers's hardness test performed on the metal helps to determine the influence of *E. oxypetalum* on the mechanical hardness behaviour of the metal. Fig. 8 shows how the metal's hardness was improved by the adsorption of the inhibitor molecules on the metal. The hardness indentation on the raw coupon showed that the microhardness was

greatest. The micro-indentation value of the coupon exposed to the inhibitor-free solution was very low compared with that of coupon dipped in the inhibited system. The low value recorded could be as a result of the high level of porosity created by the chemical attack. Obviously, material degradation is a surface phenomenon which extends to the reduction of the thickness of the material. The adsorbed inhibitor tends to discourage the thickness reduction thereby improved the surface coverage. The improvement of the micro indentation of the alloy could be attributed adsorption of the molecules of the inhibitor on the substrate which could cause an increase on the thickness when compared with the uninhibited system. Further, the presence of the inhibitor was found to reduce the





**Fig. 8. Mechanical Vickers micro indentation value of AA5052 exposed to an acidic environment**

level of porosity of the Al alloy are different between the inhibited and the uninhibited coupons. The decrease in the density of porosity is related to an increase in the hardness of materials [31]. The inhibition process is then responsible for the improvement of the metal's hardness value compared with the uninhibited.

#### 4. CONCLUSION

This paper investigated the adsorption inhibition mechanism of *E. oxypetalum* leaf extract on aluminium alloy, AA5052 in an acidic environment. The leaf extract effectively inhibited the corrosion of AA5052 in an acidic environment. The inhibition efficiency increases with increase in inhibitor concentration and decreases with increasing temperature. The electrochemistry of the corrosion process was greatly altered by the presence of the inhibitor. The charge-electron transfer between the electrodes was reduced due to the presence of the *E. oxypetalum* in the corrosive media.

Physical adsorption of the inhibitor molecule was observed according to Langmuir adsorption isotherm model. The Gibb's free energy of adsorption  $\Delta G$  at all temperature exposure indicates that the adsorption process was spontaneous. Also, the mechanical hardness properties of AA5052 were improved after the corrosion attack due to the presence of the green inhibitor.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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