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Anti-corrosion Process of *Epiphyllum oxypetalum* Extract on Aluminum Alloy in Acidic Environment

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Author's contribution

The sole author designed, analysed, interpreted and prepared the manuscript.

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ABSTRACT

Major problems encountered in industries, science and engineering is corrosion failure. The gravimetric, the electrochemical impedance spectroscopy (EIS) technique and Vickers hardness test were employed in investigating the anti-corrosion process of Aluminum alloy in acidic environment (0.5 M HCl solution) using *Epiphyllum oxypetalum* leaf extract. The leaf extraction was prepared using reflux set-up. Addition of the leaf extract in the corrosive media significantly reduced the mass loss and the corrosion (damage) rate. The inhibition efficiency increased linearly with the inhibitor concentration with a peak value of 89% for gravimetric technique and 87% for EIS technique, but slightly reduced with temperature rise. The charge-transfer resistance, R_{ct} increased linearly with the increase in concentration of the inhibitor in the corrosive environment. The Vickers's microindentation hardness test investigated revealed a preservation of the indentation value of the AA5052 exposed to inhibitive environment compared with those exposed to uninhibited environment. Surface screening was conducted and examined the surface morphology of the metals before, and after exposure to the various inhibitive solution. The experimental data obeyed Langmuir adsorption isotherm. Thermodynamic parameters (heat of adsorption, ΔH^*, activation energy, E_a, entropy, ΔS^* and Gibb's free energy, ΔG_{ads}) were evaluated through the Eying and Arrhenius plots. Upon the addition of the inhibitor, ΔH and E_a increased while the ΔS^* decreased.

Keywords: Corrosion; adsorption; AA5052; gravimetric; electrochemical; Epiphyllum oxypetalum.

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1. INTRODUCTION

A report by Karthik and Sundaravadivelu [1] has it that the environmental consequence of corrosion is enormous and its inhibition has been deeply investigated by researchers. Organic compounds recently have captured researcher's attention especially in the area of corrosion inhibition. The inhibition ability of an organic molecules depends on their efficiencies to adsorb on the surface of the metals. Inhibitive impacts of organic molecules are usually based on the substitution of H_2O molecules from the metal surface and formation of barrier film of the inhibitor compound on the surface of the meta [2,3]. The use of corrosion inhibitors is one of the most practical and economical methods for diminishing corrosion rates and the protection of metal surfaces against corrosion [4].

Adsorption of inhibitors on ingot surface is a function of some inhibitor group physicochemical properties not limited to the following: density of the electron at the donor atom, the nature and surface charge of the metal, the type of aggressive electrolyte, orbital character and electronic structure of the molecule (the chemical structure of the inhibitors) [5,6]. The process of inhibitor's action is critically on the surface of the adsorbent. Further, efficiency of a given inhibitor depends on the characteristics of the environment in which it acts, the nature of the metal surface and electrochemical potential at the interface [7].

The position of aluminum and its alloy in the industries, and homes are very enormous to mention. Aluminum is known as the third abundant element on the earth crust [8]. The corrosion resistance ability and the mechanical properties of Al-alloy are in constant attack as a result of service condition, environmental treat, and other factors. Due to their wide usage, there is need to seek for a way of maintaining and protecting the metal against environmental attack especially as acid is mostly applied in the cleaning and pickling process. Acid have been found as a cleaning agent of aluminum alloys. Thus, the use of *Epiphyllum oxypetalum* leaf extract drew attention on the need to the combat to corrosion attacks of Aluminum alloy, AA5052 in acidic environment.

2. MATERIALS AND METHODS

2.1 Materials Preparation

The metal, AA5052 alloy were mechanically cut into coupons of sizes, 20 mm x 20 mm x 1.5 mm for the gravimetric studies, while a size of 1 mm x 1 mm x 1 mm was sectioned for electrochemical studies. The metal was prepared according to ASTM G1 (2003) [9] early employed by [9,10]. The coupons were weighed using electronic weighing balance of 0.0001 sensitivity.

The leaf extraction was conducted using Reflux setup. The leaf harvested within Abia State Polytechnic, Aba (5.1338°N, 7.3554°E) were ground to powder called stock. The stock extracted into the corrosive environment in the ratio 1:30. The solute-solvent mixture was subjected to heating at a temperature (60°C). And the various concentrations of the inhibitor were prepared from the filtrate using ($C_1V_1 = C_2V_2$). The solvent used for the extract is 0.5 M HCl.

2.2 Gravimetric Technique

The gravimetric technique involves taking note of the initial weight of the metal immersed in the corrosive environment and the final weight after the corrosion inhibition process. The final weight of the coupons was measured after the corrosion and the inhibition process. The procedure outlined on ASTM G31-72(2004) [10] STANDARDS were followed for reliability. The system of immersion practice was planned interval test. The corrosion inhibition process involved full immersion of the coupon in 0.5 M HCl solution containing the various concentrations of the inhibitor without disturbance was. The setup was allowed for a maximum period of 9-hours under ambient temperature and each coupon retrieved every 1-hour corrosion process quenched. The temperature of the environments was raised to 45°C and 60°C using water-bath for 2-hours. The process was carried out three times for reproducibility and the average value reported.

2.3 Electrochemical Impedance Spectroscopy (EIS) Technique

Electrochemical methods are used in many different ways to assess the effectiveness of corrosion inhibitors and coatings for metals and alloys [11]. Electrochemical impedance spectroscopy (EIS) has been an advanced technique used in corrosion investigations. The EIS studies the system response to the application of a periodic small amplitude ac signal [12]. Electrochemical Workstation Potentiostat/Galvanostat electrochemical analyzer with a tripod stand: The working

electrode being the coupon (AA5052), the reference electrode and the counter electrode were used. Before measurement, the working electrode was immersed in test solution (0.5 M HCl containing different concentrations of the leaf inhibitor) for approximately 30-minutes until a steady open-circuit potential (OCP) was reached. The EIS was performed at -0.25 to +0.40 volts versus door circuit potential, at a scan rate of ± 0.333 volts per second, step height of 1.0 mm and a step time of 3 seconds. The data obtained from the electrochemical tests were analyzed using Echem analyst 5.0 software. To confirm the reliability and reproducibility of the electrochemical issues, duplicate experiments organized for each case, and under the same conditions. And the average value was reported. The following electrochemical parameters were determined: solution resistance, R_s, charge transfer resistance, R_{ct}, corrosion rate and the inhibition efficiency (from the equation 3).

2.4 Micro indentation Hardness Test

Microindentation hardness test (MHT) was used due to the size of the metal coupon. Three sets of aluminum alloy ingot were used in carrying out the mechanical hardness test. The AA5052 alloy was exposed to 0.5 M HCl corrosive solution containing *Epiphyllum oxypetalum* leaf extract at the various concentrations. The test was conducted according to ASTM Designation E 384 – 99 [13]. The Vickers indenter was notched at a face angle of 136°±30″ with a test force (HV 10) of 10 kgf for 10 s randomly (four different locations) across the surface of the metal. The mean hardness value (HV) was reported.

3. RESULTS AND DISCUSSION

3.1 Wight Loss and Corrosion Rate

Weight loss being a measurable change in corrosion process was employed in the study. Fig. 1 depicts the mass loss of Aluminum Alloy, AA5052 in 0.5 M HCl solution. The mass loss of the AA5052 alloy immersed in corrosive environment (blank) increases with exposure time except at the third hour of exposure where the non-ferric metal exhibited passivation phenomenon. Significantly, the presence of the *Epiphyllum oxypetalum* extract in 0.5 M HCl solution hinders the material's mass loss. This hinderance increases with increase in concentrations of the inhibitor.

The time dependent of the material deterioration was calculated using equation 1 [14]. Fig. 2 reported the corrosion rate against the exposure period. The plot exposed that the corrosion rate greatly reduced when the green leaf extract (in different concentrations) were added in the corrosion environment. The trend of the curves suggests that the attack on the metal in the corrosive environment followed the same mechanism of corrosion response. This corrosion rates decreases with an increase in the exposure period in the inhibition systems; and with the inhibitor concentration.

$$CR = \frac{K\Delta W}{At\rho} \quad (1)$$

Where CR is the corrosion rate, K is corrosion constant (which dependent on the parametric measurement of the Area), t is the exposed period (hours), ρ is the density of the metal (gram/dm^3), and ΔW is the weight loss (gram).

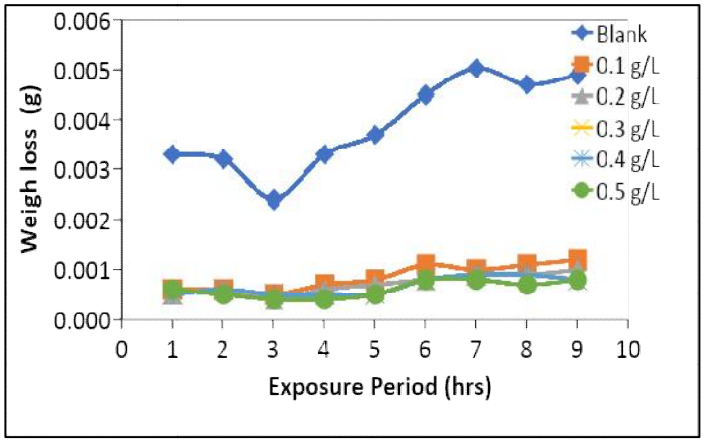

Fig. 1. Weight loss against exposure period of AA5052 in 0.5 M HCl in the presence of *Epiphyllum oxypetalum* extract

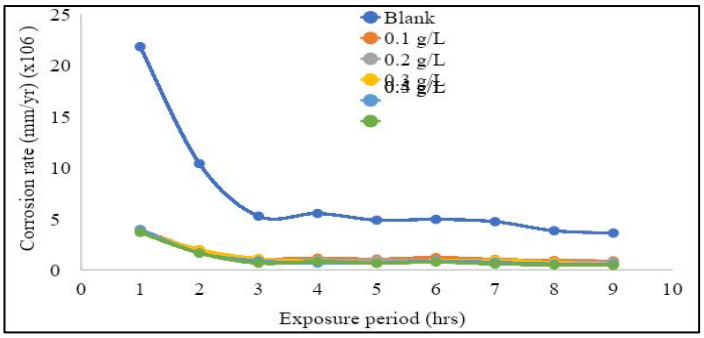

Fig. 2. Corrosion rate against exposure period of AA5052 in 0.5 M HCl in the presence of *Epiphyllum oxypetalum* extract

3.2 Electrochemical Impedance Spectroscopy (EIS) Results

The EIS is an advanced technique greatly enables the investigation of electrochemical processes occurring at the metal/solution interface. The impedance response of the AA5052 alloy in 0.5 M HCl solution containing the various concentration of *E. oxyptalum* extract are presented in the Nyquist plot (Fig. 3). The Nyquist Encirclement diameter increases with increase in the green extract concentration in then corrosive solution inferring that the corrosion process is controlled by charge transfer reaction [15]. The EIS data were fitted adequately by a circuit model shown in Fig. 4. The electrochemical parameters reported on table 1 showed that the value of charge transfer resistance and the solution resistance generally increases linearly with increasing inhibitor concentration. The higher the R_{ct} values, the slower the corrosion process. The formed inhibitive film hinders the diffusion of aggressive ions to the aluminum alloy surface; thus, caused an increase in R_{ct} values in presence of inhibitor [16]. The Nyquist plot from the circuit model developed a capacitive loop following the inductive loop. The complex Nyquist plot has two half-planes: the half-plane capacitive loop and half-plane inductive loop. The increase in the capacitive and inductive loops with the increased concentration of *E. oxypetalum* suggests that the impedance of the inhibited substrate increases.

3.3 Corrosion Inhibition Efficiency

The green leaf inhibition's abilities were considered using the two explored techniques: Gravimetric and Electrochemical Impedance Spectroscopy. The gravimetric focused on the material weight loss (equation 2) [17] while the EIS considers the charge-transfer resistance (equation 3) [16]. The inhibition efficiency was deployed to determine the impact of the green leaf extract on AA5052 corrosion attack.

$$\%IE = \left(\frac{W_a - W_b}{W_a}\right) x100 \quad (2)$$

$$\eta_{EIS}\% = \left(\frac{R_{ct} - R_{ct}^{'}}{R_{ct}}\right) x\ 100 \quad (3)$$

Where W_a and W_b are the weight loss of the uninhibited coupon and the inhibited coupon, respectively. R_{ct} and $R_{ct}^{'}$ are the charge-transfer resistance of the corrosion-free environment and the inhibited environment, respectively. The charge transfer resistance was calculated based on the difference between impedance values at the lower and higher frequencies. %IE and η_{EIS} are the inhibition efficiency for the gravimetric technique and the EIS, respectively.

Considering the gravimetric studies, the inhibition efficiency of *Epiphyllum oxypetalum* extract on aluminum alloy AA5052 (shown in Fig. 5) generally increases with increasing inhibitor concentration with an optimal value of 88% for fourth-hour exposed period. Similar trend was observed for the EIS technique (Table 2). Both techniques showed that the inhibition efficiency of *E. oxypetalum* leaf extract functions as a good corrosion inhibitor for AA5052. The somewhat sinusoidal wave form of the inhibition efficiency curve experienced (in Fig. 5) with the gravimetric technique is possibly attributed to the inherent passivity phenomenon associated with parent metal of the alloy [18].

3.4 Temperature Effect

Variation in temperature of the environments was conducted to determine the influence it would have on the inhibition ability of *E. oxypetalum*

leaf extract. Table 2 shows that the inhibition efficiency of *E. oxypetalum* extract on AA5052 alloy and; it decreased slightly with increase in temperature for every inhibitor concentration. The inverse relationship suggested that the adsorbed molecules of the inhibitor dissolve with increasing temperature. The temperature increase tends to increase the average kinetic energy of the adsorbed molecules. This causes an increase in the solubility. However, the inhibitor, *E. oxypetalum* showed high inhibition efficiency suggesting its availability even at high temperature range in the fight against corrosion of metals.

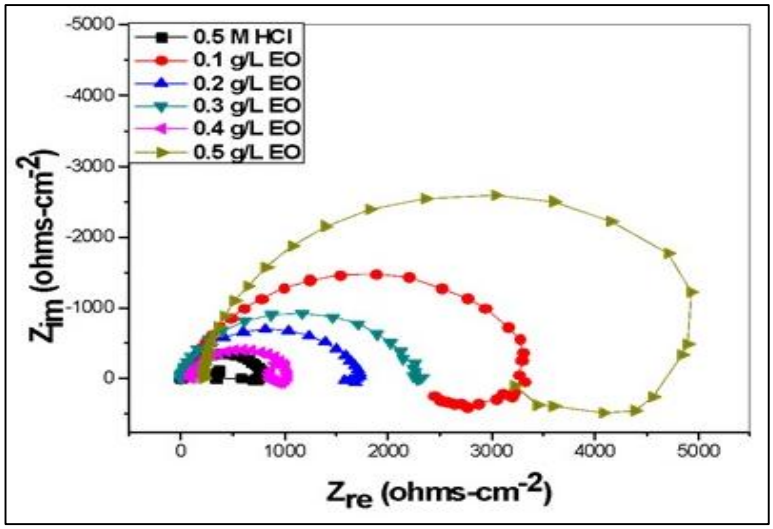

Fig. 3. Electrochemical impedance spectroscopy plots of AA5052 in 0.5 M HCl solution in the absence and presence of different concentrations of *E. oxypetalum*

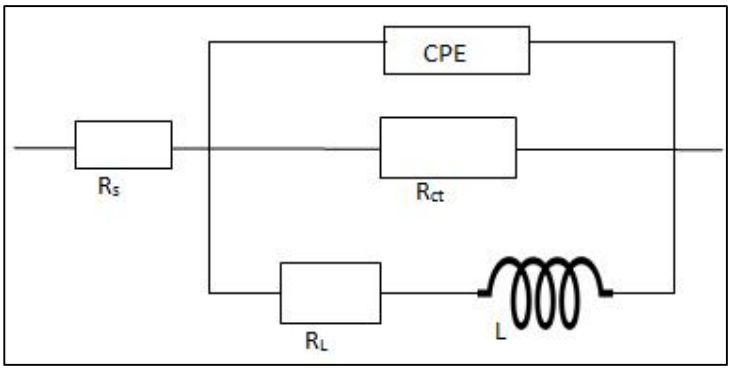

Fig. 4. Equivalent circuit used in fitting the electrochemical impedance spectra

Table 1. Electrochemical parameters of the aluminum alloy, AA5052 in the active environment of 0.5 M HCl solution containing of *E. oxypetalum*

Environment *E. oxypetalum* (g/L)	R_s (Ωcm^2)	R_{L1} (Ωcm^2)	R_{ct} (Ωcm^2)	L (H)
0.0	3.03	7.8	607	5.6
0.1	3.55	642	1128	651
0.2	3.93	3202	1857	1607
0.3	3.99	2007	2489	2112
0.4	4.13	2543	3345	2789
0.5	4.26	2983	4685	3365

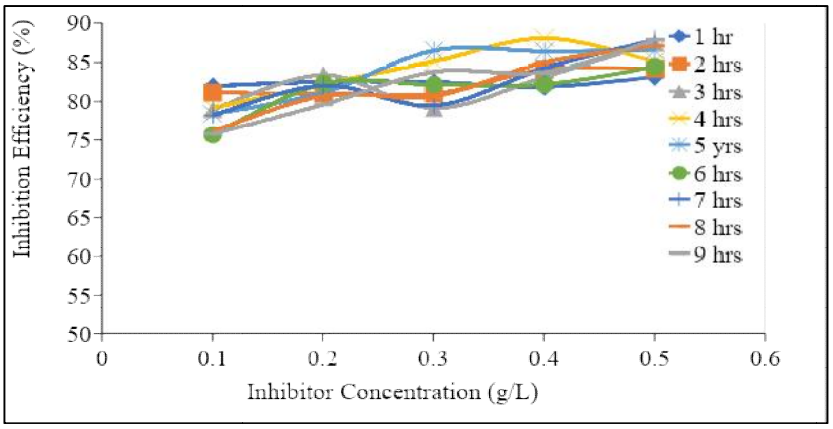

Fig. 5. Inhibition efficiency of *E. oxypetalum* on AA5052 alloy immersed in 0.5 M HCl solution

Table 2. Inhibition efficiency of *E. oxypetalum* leaf extract on AA5052 immersed in 0.5 M HCl solution

CONC. (g/L)	EIS	Gravimetric (at varied temperature)		
	303 K	303 K	318 K	333 K
0.1	46.2	81.18	75.75	74.34
0.2	67.3	81.06	76.02	75.12
0.3	75.6	81.03	76.92	74.78
0.4	81.8	84.14	79.08	76.15
0.5	87.1	84.20	80.57	78.87

3.5 Adsorption Models and Thermo-dynamic Parameters

Adsorption exists due to the presence of the inhibitor molecules and the metal surface. The interaction which arises due to this phenomenon forms the basis for investigating the difference adsorption modes [19]. Langmuir adsorption and the Temkin Adsorption models were exposed to the experimental data and were fitted by the Langmuir adsorption isotherm. Langmuir isotherm model expresses the relationship between the surface coverage and the inhibitor concentration. The Langmuir expressed in equation 4 [20,21] showed that the experimental data were in agreement with the isotherm model plotted in Fig. 6. The plot shows that the Langmuir parameter for the varied temperature conditions all exhibited linear relationship with a slope and correlation coefficient, R^2 approximated to unity (shown in Table 3). The recorded parameter shows that adsorption of the leaf extract follows the Langmuir adsorption isotherm.

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (4)$$

Where θ is the surface coverage, C_{inh} is the inhibitor concentration, and $K_{ads}$is the adsorption equilibrium constant. The standard free energy of adsorption, ΔG°_{ads} can be calculated from the adsorption equilibrium constant, K_{ads} shown in equation 5.

$$\Delta G_{ads} = -2.303RTLog(55.5K_{ads}) \quad (5)$$

Where R is the gas constant (8.314 J mol^{-1} K^{-1}), and T is the absolute temperature (K). The negative values the standard Gibb's free energy of adsorption with the range below -40KJ mol^{-1} estimated that the molecules of the *E. oxypetalum* were physically adsorbed on the metal surface at all temperature exposures. This is in line with the general concept highlighted by [22,23], that the values of ΔG°_{ads} around -20 KJ kol^{-1} are consistent with the electrostatic interaction between the charged metal (physical adsorption); whereas those more than -40 KJ mol^{-1} involves charge sharing or transfer from the inhibitor molecules to the metal surface leading to the formation of a donor-acceptor bond (chemical adsorption). Also, the negative value of ΔG^{0}_{ads} indicates spontaneity in the inhibitor adsorption on aluminum alloy, AA5052 surface.

The heat of adsorption, the adsorption energy and activation energy has been reported by [24] as the most important inter-related and frequently calculated parameters relevant to the thermodynamics of any sorption system. Thermodynamically, the activation energy of the corrosion process was calculated from the Arrhenius equation (equation 6) [21,25], which was then rearranged to equation 7 for clarity. The Eyring transition state equation (equation 8) [26] was employed to determine the enthalpy of adsorption (being the heat released or adsorbed during the adsorption process), and the entropy of adsorption using the gravimetric measurements. The equation 8 was linearized to equation 9. The Arrhenius temperature-dependent plot (Fig. 7) and the Eyring transition state plot (Fig. 8) gave straight lines with coefficient regression close to unity.

$$CR = Ae^{-\frac{E_a}{RT}} \quad (6)$$

$$LogCR = \frac{-E_a}{2.303RT} + LogA \quad (7)$$

$$CR = \left(\frac{RT}{N_a h}\right) exp\left(\frac{\Delta S^*}{R}\right) exp\left(\frac{-\Delta H^*}{RT}\right) \quad (8)$$

$$Log\left(\frac{CR}{T}\right) = \frac{-\Delta H^*}{2.303R}\left(\frac{1}{T}\right) + \left[Log\frac{R}{N_a h} + \frac{\Delta S}{2.303R}\right] \quad (9)$$

Where A is the pre-exponential factor, R is the universal gas constant 8.3145 J/mol, T is the absolute temperature (K), N_a is the Avogadro's number $6.022x10^{23}$ mol^{-1}, *h* is the Plank's constant $6.626x10^{-34}$ J.s, E_a is the activation energy, ΔS^* is the entropy of adsorption and ΔH^* is the enthalpy of adsorption.

Table 4 presents the calculated thermodynamic parameter from the Arrhenius and Eyring plots. The E_a of the inhibited systems were greater than that of the inhibited-free system. This establishes that the presence of the inhibitor increased the threshold energy for the corrosion process to take place; conventionally it increased the energy barrier for the corrosion process. Also, the ΔH^* increase on the addition of the inhibitor supports the concept of physical adsorption of the inhibitor substance on the metal surface [27]. The E_a values shows strong inhibitive. The less negative entropy of adsorption in the inhibited system compared with the inhibitor-free environment elaborates on the ordered arrangement of the *E. oxypetalum* molecules on the AA5052 surface. This confirms the surface coverage close to unity and it further bare forth the understanding that complex inhibitor molecules associate rather than dissociates on the metal surface [28-30].

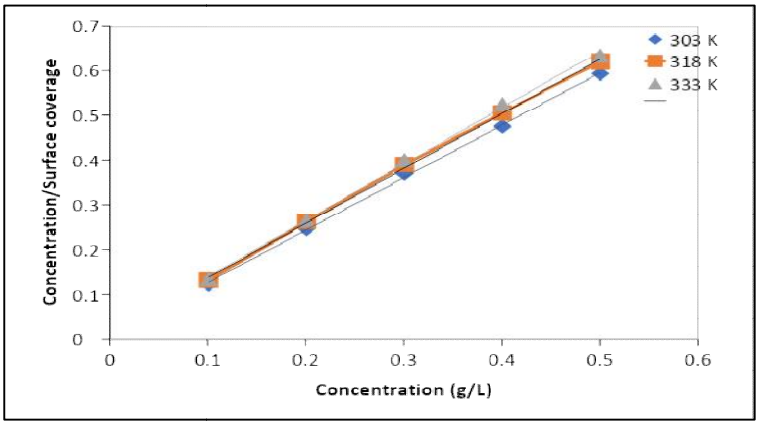

Fig. 6. Langmuir isotherm plot of corrosion of AA5052 in 0.5 M HCl containing *Epiphyllum oxypetalum* leaves extract

Table 3. Langmuir adsorption parameters of *E. oxypetalum* extract in 0.5 M HCl solution

Temp. (K)	Langmuir Isotherm			
	Slope	R^2	K_{ads}	ΔG_{ads} (KJ/mol)
303	1.1697	0.9999	91.7431	-21.5057
318	1.2200	0.9990	61.3497	-20.4918
333	1.2580	0.9980	67.1141	-20.7181

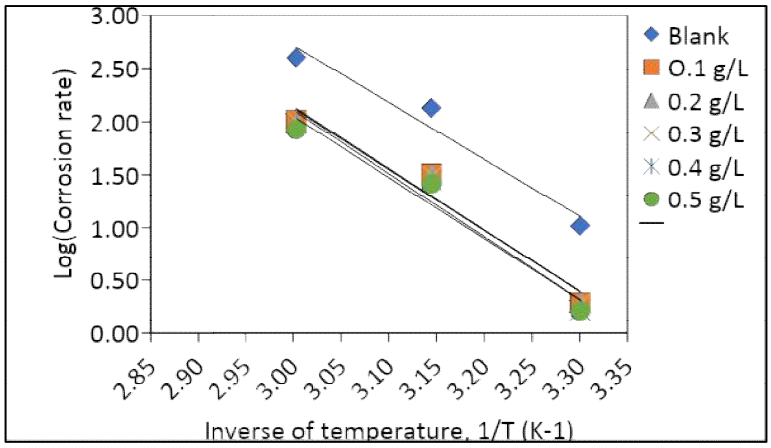

Fig. 7. Arrhenius plots of corrosion of AA5052 alloy in the presence and absence of *E. oxypetalum* extract in 0.5 M HCl

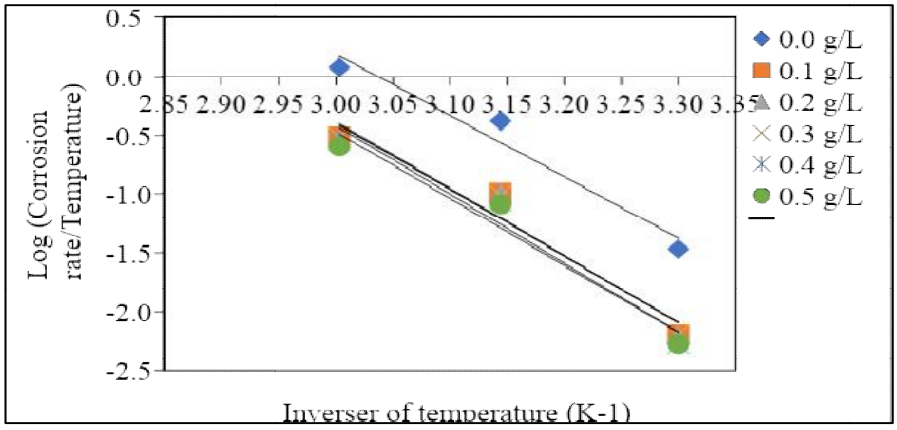

Fig. 8. Eyring plots of corrosion of AA5052 the absence and presence of *E. oxypetalum* extract in 0.5 M HCl solution

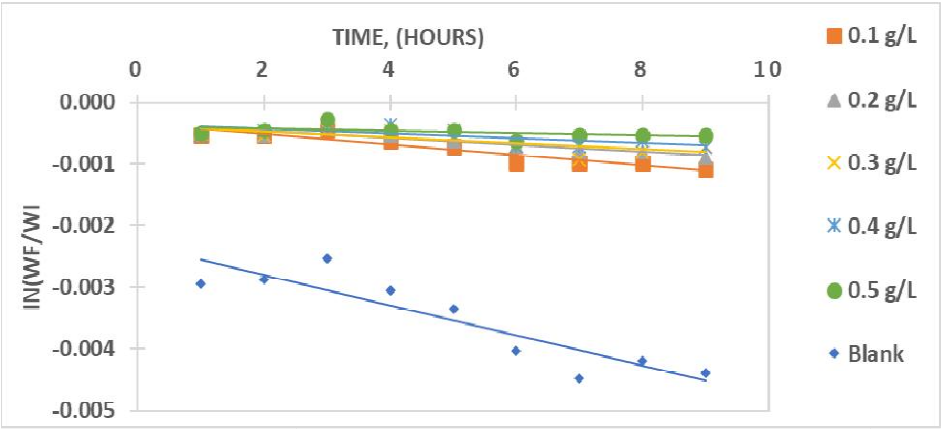

Fig. 9. Plot of lnWf/Wi against time for AA5052 alloy immersed in 0.5 M HCl solution containing different concentration of *E. oxypetalum* extract

Table 4. Thermodynamics parameters for AA5052 alloy corrosion in 0.5 M HCl solution containing *Epiphyllum oxypetalum* leaves extract

System *E. oxypetalum* (g/L)	Activation energy, E_{ads} (KJ/mol)	A x10^{19} (L/mol-s)	Enthalpy, ΔH^* ($KJmol^{-1}K^{-1}$)	Entropy, ΔS^* ($Jmol^{-1}K^{-1}$)	Rate constant k	Half-life
0.00	102.793	0.67	43.49	-65.46	$-2.57x10^{-4}$	2828.43
0.10	111.546	4.2	47.29	-58.85	$-8.27x10^{-5}$	8379.67
0.20	110.499	2.8	46.83	-60.32	$-5.60x10^{-5}$	12386.94
0.30	110.810	3.1	46.97	-59.92	$-4.71x10^{-5}$	14718.54
0.40	114.268	10.0	48.47	-55.60	$-3.92x10^{-5}$	17682.26
0.50	111.014	2.79	47.06	-60.29	$-1.87x10^{-5}$	37096.74

First order reaction kinetics with respect to AA5052 in 0.5 M HCl solution were observed when $In(W_f/W_i)$ was plotted against time according to the equation 10. The plot (Fig. 9) was used to obtain the rate constant and subsequently the half-life of the ingot in the environment using equation 11.

$$In\frac{W_f}{W_i} = -kt \quad (10)$$

$$t_{½} = \frac{0.693}{k} \quad (11)$$

Where W_f is the final weight after immersion, W_i is the weight before immersion, K is the rate constant and t is time. $t_{½}$ is the half-life of the aluminum alloy.

The obtained rate constant has inverse relationship with time; but a direct relationship with the inhibitor concentration. However, the calculated half-life shows a very significant increase when the inhibitor concentrations were added. Conversely, the presence of the inhibitor greatly influenced the half-life of the AA5052 in 0.5 M HCl environment.

3.6 Surface Morphology Examination

Surface screening examination was executed on three categories of the aluminum alloy, AA5052 after 9 hours of exposure shown in Fig. 10. sample (a) is the raw coupon (as bought), showing how the nature of the surface is before any corrosion and corrosion inhibition process. Sample (b) is coupon exposed to corrosive media without inhibitor. The surface is characterized by roughness linked to the attack of the aggressive media resulting to metal dissolution. However, the addition of *E. Oxypetalum* extract renders the metal surface of sample (c) with evidence of deposition. The deposit is attributed to surface protection [31]. The surface deposition of *E. oxypetalum* supports the adsorption model and the increases ininhibition efficiencies recorded at the gravimetric and the EIS techniques.

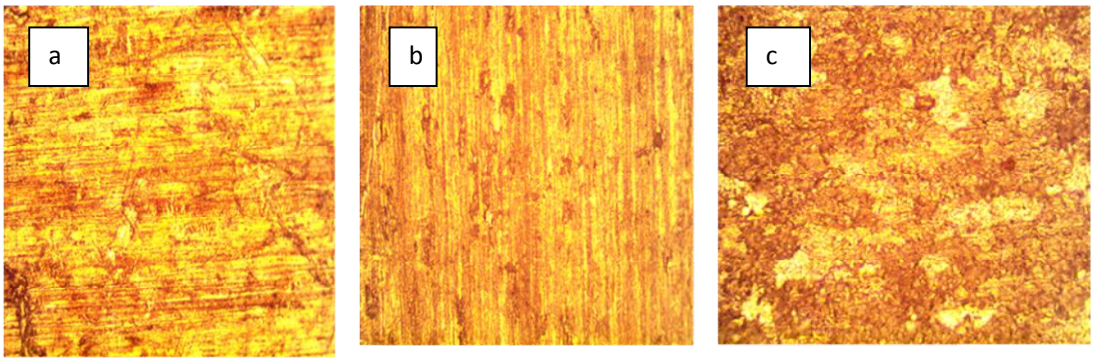

Fig. 10. Optical Micrograph of AA5052 alloy (a) Raw Material, (b) exposed to inhibitor-free (c) exposed to inhibited environment

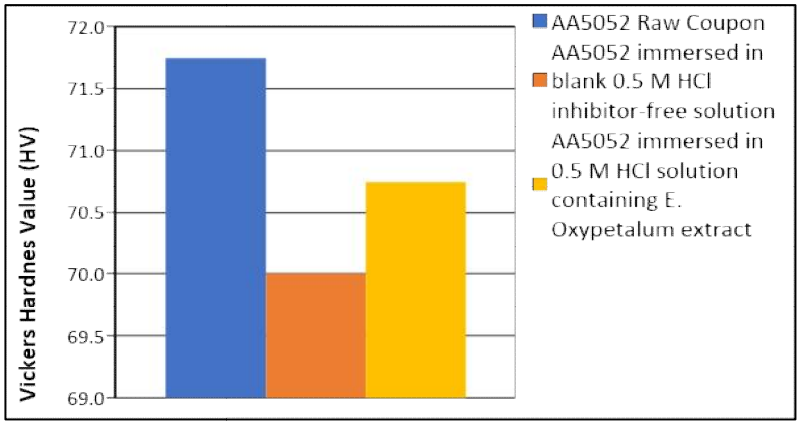

Fig. 11. Mechanical Vickers micro indentation value of AA5052 exposed to 0.5 M HCl solution

3.7 Vickers Micro-indentation Test

Hardness broadly defined as the resistance to permanent surface indentation or penetration by [32] paves the need for microindentation hardness testing (MHT) conducted on AA5052. The mechanical hardness test of AA5052 was investigated using Vickers's indention technique. A load of 10 kgf (N) was notched on four different random locations on the coupons. The influence of the inhibitor developed was accessed based on the Vickers Pyramid number (HV). The Vickers's number value of the coupon dipped in the inhibitor-free solution is lower compared with that exposed to inhibitor environment; while the value of the raw material is the highest. This implies that the adsorption of *E. oxypetalum* extract rescued the AA5052 alloy from corrosion since corrosion process is involved in material loss and mechanical test is relative to the thickness of the material. It was revealed (Fig. 11) that the presence of *E. oxypetalum* extract. These scale change in the HV confirms another potency of *E. oxyptalum.*

4. CONCLUSION

The influence of eco-friendly leaf extract of *E. oxypetalum* on the corrosion process of aluminum alloy AA5052 has been investigated in 0.5 M HCl environment using gravimetric and electrochemical impedance spectroscopy. The following conclusion have been made based on the introduction of different concentration of *E. oxypetalum* leaf extract in 0.5 M HCl solution:

1. The introduction of *E. oxypetalum* in the corrosive media reduced the weight loss and the corrosion rate of AA5052 alloy.
2. EIS method reveals that the charge-transfer resistance increases with increase in inhibitor concentration.
3. The inhibition efficiency of the green inhibitor increases with increasing inhibitor concentration, but reduced with temperature rise.
4. The E_a increase with inhibition introduction confirms adsorption of the inhibitor molecules on the metal surface as it created double layer.
5. Decrease of the entropy on the addition of the inhibitor extract explains the association interaction of the inhibitor molecules. While the heat of adsorption increase supports the evidence of adsorption on the metal/solution interface.
6. Adsorption isotherm of *E. oxypetalum* on the AA5052 surface follows Langmuir's adsorption isotherm. Adsorption was through physical adsorption.
7. Inhibition process followed first order reaction kinetic. The process was spontaneous according to the Gibb's free energy.
8. The mechanical hardness strength of the ingot was preserved due to adsorption of the inhibitor molecules of the metal surface. And surface rescued the metal surface from attack as revealed by the surface morphology.

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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