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## ***Rauvolfia vomitoria* Effect on the Degradation of Aluminium Alloy in 2.5 M Hydrochloric Acid Solution**

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### **Abstract**

*The deterioration of aluminium alloy in 2.5 M hydrochloric acid in the presence Rauvolfia vomitoria extract was examined using both gasometric and gravimetric techniques. Aluminium coupons measuring 4 cm by 1 cm were introduced into test solutions of uninhibited acid and also those containing extract quantities of 10, 20, 30 and 40 cm<sup>3</sup> at ambient temperature of 25°C for a total cumulative period of 117 minutes. The volumes of hydrogen gas evolved and weight loss readings were recorded and analyzed to obtain weight loss and corrosion rate for each coupon at specific time intervals. The results however, showed that while extract quantity was increasing; weight loss measurements increased but inhibitor efficiency decreased. Also, the metal surface-phytoconstituent interaction mechanism was consistent with the Temkin adsorption isotherm and the 3 minutes curve showed the best exposure time for phytochemicals to adsorb to alloy surface. Furthermore, microstructural studies showed an indirect relationship between metallic degradation and extract quantity, while uniformity of the irregular intermetallic phases increased with increasing extract quantity.*

*Key words-Environmentally induced failure, green inhibitors, hydrogen evolution, Aluminium alloy, adsorption isotherm.*

### **1. Introduction**

A material usually a metal is said to undergo chemical degradation otherwise called corrosion when it experiences a change in its properties as a result of an interaction with its environment. Corrosion is a natural process. For instance steel or iron has a natural tendency to combine with other chemical elements to return to their lowest energy states. Iron or steel normally combine with oxygen and water to return to lower energy states. This combination leads to the formation of hydrated iron oxides otherwise called rust and similar in chemical composition to the original iron ore. Corrosion processes leads to losses and affects society on a regular basis causing degradation and damage to household appliances, automobiles, airplanes, highway bridges, energy production and distribution systems. Corrosion is the major factor influencing the durability and dependability of pipelines that transport crucial energy through the country.

Presently, most of the aging infrastructure in the form of bridges, buildings, roads and tanks are reaching the end of their design lifetime in

Nigeria. Structural deficiency resulting from corroded steel and steel reinforcement has been fingered as the major factors responsible for these damages (Omotosho, 2011). The cost of controlling this naturally occurring phenomenon and cost associated with the damage it causes is substantial with figures rising yearly in developed countries like the U.S. The current per capita direct cost of corrosion for U.S residents based on 2001 figures is approximately \$970 per person per year (Koch et al, 2001). This amount does not include indirect/user cost which will essentially double that amount and is put at 6% of the Gross Domestic product (GDP) (Koch et al, 2001). However, these costs could be saved if optimum corrosion management techniques are employed. Thus, corrosion protection and control is a potent and vital tool for the techno-industrial and socio-economic development for any country that is determined to protect public safety, prevent damage to property and the environment and conserve funds.

Acids are utilized for rust removal and pickling; degreasing/desludging of petrochemical equipment; oil well acidizing and descaling; and the removal of calcareous deposit from steam



boilers and cooling tower units. These acids are known to have damaging effects on their substrates. Several established methods for preventing and controlling the damages depend on specific material to be protected, environmental concerns, type of product to be transported. The most generally used methods include organic and metallic protective coatings, corrosion resistant alloys, plastics and polymers, cathodic protection and corrosion inhibitors. However, bulk of the corrosion inhibitors available are synthetic chemicals, expensive and very hazardous to the environment. Therefore, it is desirable to source for environmentally safe and cheap green inhibitors ( Ajayi et al., 2011a, Ajayi et al., 2011b, Ajayi et al., 2011c, Omotosho et al. 2012a, Omotosho et al. 2012b, Ajayi et al., 2012, Omotosho and Ajayi, 2012, Omotosho et al. 2011).

Various assessment studies have been carried out to ascertain the potential of various inhibitors on different metals in the presence of variety of acids, most of them using either gasometric or gravimetric method (Ebenso et al, 1998; Ebenso and Ekpe 1996; Ekpe et al, 1994; Martinez and Tagljar, 2002; Ebenso et al, 2004; Oguzie, 2006; Njoku et al, 2006; Abiola et al, 2007; James and Akaranta, 2009; Saratha et al, 2009; Okafor et

Table 1. Chemical composition of Aluminium alloy sample utilized for the investigation

Elements	% Chemical Composition	Elements	% Chemical Composition
Silicon	0.157	Cadmium	0.0005
Iron	0.282	Calcium	0.0011
Copper	0.0025	Cobalt	<0.001
Manganese	0.51	Lithium	<0.0002
Magnesium	0.51	Sodium	0.0005
Zinc	<0.001	Phosphorus	<0.001
Nickel	<0.001	Lead	<0.0005
Chromium	0.023	Tin	<0.001
Titanium	0.0049	Strontium	0.0001
Silver	0.0004	Vanadium	0.0063
Boron	0.0007	Zirconium	0.002
Beryllium	0.0001	Aluminium	99
Bismuth	0.001		

The acidic extracts of RV were prepared from fresh leaves which were air dried and ground into powder, of which 20g was put into a 1000ml volumetric flask containing 1000 ml of ethanol. The resulting solution was left for 48hrs after which it underwent an evaporation process in the rotary evaporator to remove most of the ethanol. The remaining solution was then left for another 48 hours to allow the further evaporation of the remaining ethanol so that what is left afterwards, is the actual extract obtained from

al, 2010; Ajayi et al., 2011a; Ajayi et al., 2011b; Ajayi et al., 2011c; Omotosho et al. 2012a; Omotosho et al. 2012b; Omotosho and Ajayi, 2012; Ajayi et al., 2012 and Omotosho et al. 2011). Amongst the studies considered, none has yet worked on the effect of *Rauvolfia vomitoria* (RV) on Aluminium alloy in the presence of 2.5 M Hydrochloric acid (HCl). Thus, the aim of this study is to investigate the effect of RV on the deterioration of aluminium alloy in the presence of 2.5 M HCl at ambient temperature of 30 degrees celsius. The technique adopted in this study is the gasometric and gravimetric methods. Moreover, attempts were made to determine the mechanism of interaction between the aluminium sample and extract phytochemical.

## 2. Materials and Methods

Aluminium alloy specimens with dimension 4 x 1 cm were employed for the determination of corrosion inhibition of RV extract in 2.5 M Hydrochloric acid (HCl). Specimens were prepared by degreasing in ethanol and cleaning in acetone. It was then allowed to dry. The composition of the aluminium alloy samples was analyzed using Optical Emission Spectrometer (OES) and the result is presented in Table 1.

the leaves; which is considered as the phytochemical. In order to obtain the inhibited test solution, 1000ml of 2.5 M HCL is then mixed with 10, 20, 30 and 40 ml of the extract. The experimental set up is similar to the technique adopted elsewhere (Okafor et al. 2010; Ajayi, et al. 2011a; Ajayi, et al. 2011b; Ajayi et al. 2011c; Omotosho et al. 2011; Omotosho and Ajayi, 2012; Omotosho et al. 2012a and Omotosho et al. 2012b). The time was recorded and H<sub>2</sub> evolved was collected in a calibrated tube by downward displacement of water per time for

each coupon used. A plot of  $H_2$  evolved per unit area ( $ml\ cm^{-2}$ ) against time (minute) in the presence of different concentrations of RV was obtained as shown in Figure 2. The inhibition efficiency was then determined using Equation 1 below obtained from literature (Okafor et al. 2010; Ajayi, et al. 2011a; Ajayi, et al. 2011b; Ajayi et al. 2011c; Omotosho et al. 2011; Omotosho and Ajayi, 2012; Omotosho et al. 2012a; Omotosho et al. 2012b; Omotosho et al. 2012c ) [1]

$$I.E(\%) = \frac{(V_H - V_{HI})}{V_H} \times 100$$

(1)

Where,  $V_H$  is the volume of  $H_2$  evolved without inhibitor (i.e control experiment) and  $V_{HI}$  is the volume of  $H_2$  evolved with inhibitor.

Weight loss experiments were conducted by first weighing the sample before immersion and

recording the weight. Thereafter, the weight was obtained after completion of the hydrogen evolution experiment but before this was done the sample was separated from the test solution, washed with deionised water and acetone; and dried finally with a stream of air. From the weight loss results, the corrosion rate ( $R$ ), inhibition efficiency ( $\%I$ ) of the inhibitor and degree of surface coverage ( $\theta$ ), were calculated using equations 2, 3 and 4, respectively.

$$R\ (g\ h^{-1}\ cm^{-2}) = W/At$$

(2)

$$\%I = (1 - W_1/W_2) \times 100$$

(3)

$$\theta = 1 - W_1/W_2$$

(4)

Where  $W_1$  and  $W_2$  are the weight losses ( $g/dm^3$ ) for the sample in the absence and presence of the inhibitor in HCl solution, respectively, and  $\theta$  is the degree of surface coverage of the inhibition.

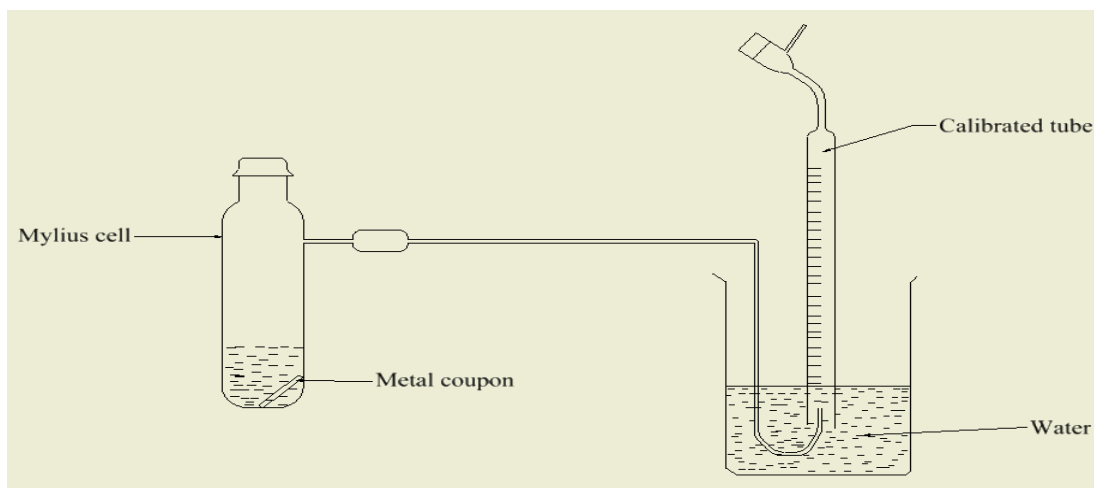


Figure 1: Schematic diagram of the experimental set up



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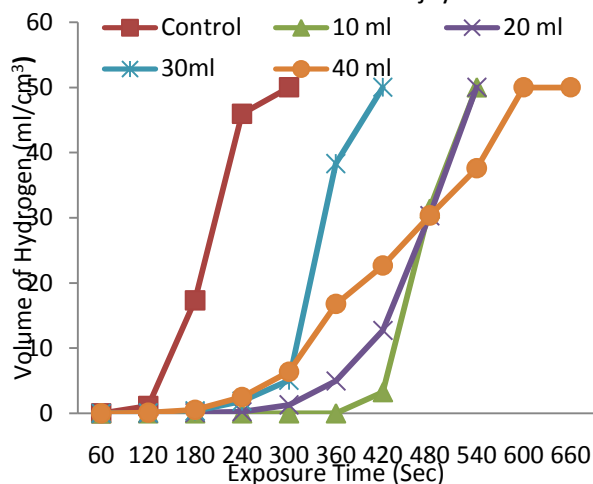


Figure 2: Variation of volume of hydrogen ( $\text{cm}^3$ ) evolved with time (sec) of Al coupons for varying quantities of RV extract in 2.5 M HCl at room temperature of 27°C using gasometric method

It is known that  $\text{H}_2$  gas is one of the products of reaction when Al-alloy reacts with HCl, it is thus, suitable to predict the rate of reaction by determining the rate of  $\text{H}_2$  gas evolution from the system. By inference, the rate of reaction corresponds to the rate of corrosion damage of the Al-alloy in HCl in the presence of RV extract. This therefore means that, modeling the rate of corrosion from the direction of rate of hydrogen gas evolution is indirectly a way to model the rate of material loss when the relationship between the weight loss and hydrogen gas evolution is known. The foundation for this is as represented in Equation (5) (Ajayi, et al. 2011a; Ajayi, et al. 2011b; Ajayi et al. 2011c; Omotosho et al. 2011; Omotosho and Ajayi, 2012; Omotosho et al. 2012a; Omotosho et al. 2012b):

$$\begin{aligned} \Delta V &\propto \Delta W \\ \frac{d\Delta V}{dt} &\propto \frac{d\Delta W}{dt} \\ \text{but } R &\propto \frac{d\Delta W}{dt} \propto \frac{dV}{dt} \end{aligned} \quad (5)$$

Where  $\propto$  = proportionality sign.

$\Delta V$  = volume of hydrogen gas evolved

$\Delta W$  = metal weight loss due to corrosion

$R$  = Rate of corrosion

$t$  = time (s)

Thus to determine Equation (5), it is necessary to develop a relationship between volume evolved and the time of evolution. This was

arrived at by a polynomial regression analysis of the volume of hydrogen gas against time leading to Equation (6) (Ajayi, et al. 2011a; Ajayi, et al. 2011b; Ajayi et al. 2011c; Omotosho et al. 2011; Omotosho and Ajayi, 2012; Omotosho et al. 2012a; Omotosho et al. 2012b):

$$V = c + bt + at^2 \quad (6)$$

Thus,

$$R = \frac{dV}{dt} = b + 2at \quad (7)$$

A particular case of this study will involve adapting Equations (6) and (7) to the volume-time measurement for individual quantity. For instance, for measurements relating to 5ml extract quantity, the corrosion rate model is presented as Equation (9) (Ajayi, et al. 2011a; Ajayi, et al. 2011b; Ajayi et al. 2011c; Omotosho et al. 2011; Omotosho and Ajayi, 2012; Omotosho et al. 2012a; Omotosho et al. 2012b):

$$V = 0.450t^2 + 0.352t - 3.104$$

(8)

$$dV/dt = 0.9t + 0.352$$

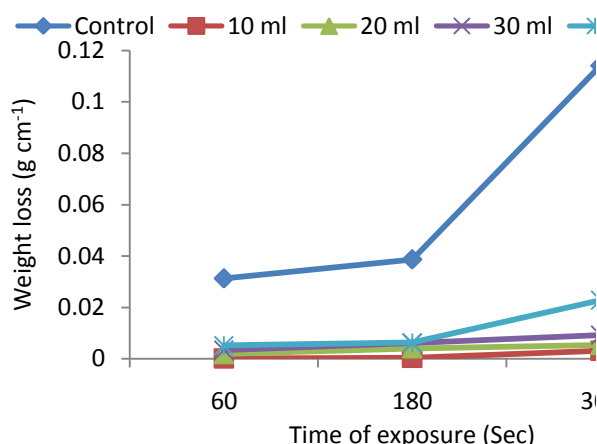
(9)

### 3. Results and discussion

An analytical look at Figure 2 revealed that the corrosion rate of Aluminium alloy as indicated by the amount of  $\text{H}_2$  evolved reduced in the presence of RV when compared to the control. The amount of  $\text{H}_2$  evolved in the presence of increasing volume of the extract did not follow a particular trend. However, based on the observations of the graph the trend of corrosion rate reduction starting from the least is as follows;  $30 < 40 < 20 < 10 < \text{control}$ . This indicates that the RV extract in the solution has an inhibitive effect on the corrosion of Aluminium in HCl and that the extent of inhibition depends on the amount of RV extract present.

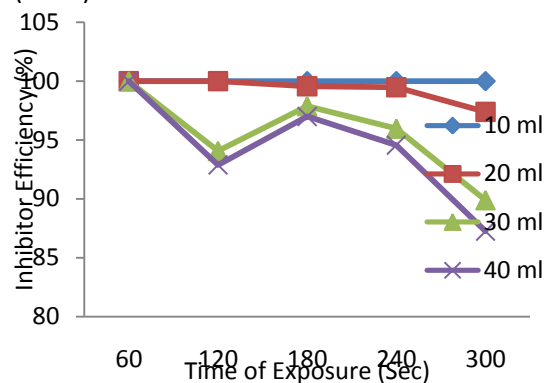


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**Figure 3:** Curves of weight loss of varying quantities of RV extract with time (minutes) on aluminium coupon in 2.5M HCl solution at room temperature

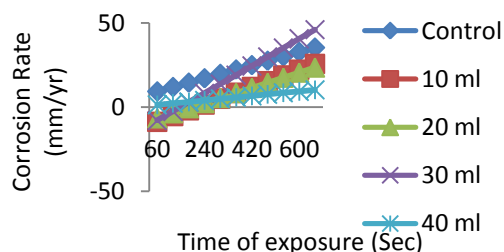
Figure 3 shows a better picture of the weight loss measurements as the curves do not cross over each other as in Figure 2. From Figure 3 the trend of weight loss measurement starting from the least is as follows;  $10 < 20 < 30 < 40 \text{ ml} < \text{control}$ . This same behavior was also observed in Figure 4 which shows the percentage inhibition efficiency (% I.E). The sample immersed in the 10 ml test solution showed best performance for the entire 5 minutes of the experiment as the inhibitor efficiency attained a hundred percent throughout. The sample immersed in the 20 ml test solution showed the same performance as that in the 10 ml test solution for the first two minutes of the experiment as 100 % inhibitor efficiency was attained; however after the second minute, the inhibitor efficiency began to decrease from that time till the end of the experiment. The sample immersed in the 30 and 40 ml test solution showed best performance for the first minute of the experiment as the inhibitor efficiency attained 100%; however after the first minute; the inhibitor efficiency began to decrease from that time till the end of the experiment. The order of inhibitor efficiency starting with the least is thus:  $40 < 30 < 20 < 10 \text{ ml}$ .



**Figure 4:** Percentage inhibition efficiency of varying concentrations of RV extract with time (minutes) on aluminium coupon in 2.5M HCl solution at room temperature of 27°C using gasometric method

In Figure 5, corrosion rate values for all the varying inhibitor additions were very low at the beginning of the experiment, with the 10 and 20 ml extract additions having sub-zero values for the first 4 minutes, while the 30 and 40 ml extract additions had above-zero values after the 3<sup>rd</sup> and 1<sup>st</sup> minute of the experiment respectively. However, the control experiment maintained above-zero values throughout the experiment with value increasing as the experiment progressed.

The order of performance is as follows:  $40 < 30 < 20 < 10 \text{ ml}$ . At the beginning of the experiment, the samples which were immersed in the 10, 20 and 30 ml test solutions had just adsorbed to the surface of the metal and since the bond was still strong at this timing of the experiment, corrosion rate was greatly reduced with no noticeable corrosion occurring. This explains the negative corrosion rate on the graph (Figure 5).



**Figure 5:** Corrosion rate of varying amounts of RV extract with time (minutes) on aluminium coupon in 2.5M HCl solution at room temperature of 27°C using gasometric method.





This same behavior was also observed in Figure 6 which shows the percentage inhibition efficiency (% I.E). The inhibitor performance followed the order: 40 < 30 < 20 < 10 ml.

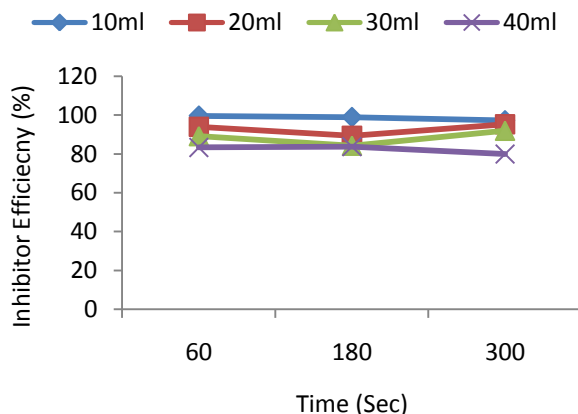


Figure 6: Inhibitor efficiency of varying amounts of RV extract with time (minutes) on aluminium alloy coupon in 2.5 M HCl solution at room temperature using weight loss method.

According to the observation in Figure 7, which is the graph of corrosion rate against time for the experiment based on weight loss measurements the same behavior that was shown by the graph for corrosion rate based on gasometric technique. The order of performance is thus as follows: 40 < 30 < 20 < 10 ml. This is exactly the same as that of Figure 5. The only difference is the level of responsiveness.

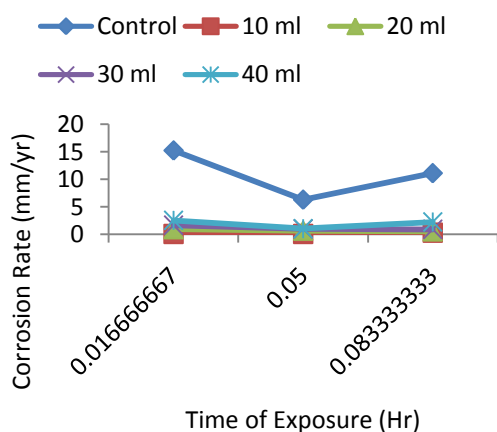


Figure 7: Corrosion rate of varying concentrations of RV extract with time on aluminium coupon in 2.5 M HCl solution at room temperature of 27 °C using weight loss method.

### Adsorption studies

The interaction between the phytochemicals in the RV extract and the metal surface suggests the existence of a relationship. This was thoroughly investigated by deploying various adsorption isotherms amongst which are Langmuir, Freundlich, Frumkin and Temkin. Surface coverage values,  $\theta$ , for the changing extract volumes were appraised on the measurement of the volume of  $H_2$  gas evolved while fitting the  $\theta$  values with the different adsorption isotherms were conducted, however, the Temkin isotherm was discovered to be best fitted to the  $\theta$  values at different time intervals. The Temkin isotherm has been reported (Febrianto et al, 2009) to be superior in the prediction of gas phase equilibria. The plot of  $\theta$  as a function of logarithm of concentration of RV is shown in Figure 8. This result justifies the notion that the corrosion inhibition of the RV extract is credited to the adsorption of phytochemical molecules on the metal surface by the chemisorption process. In this process which is a known tribological consideration in metal surface preparation a covalent bond is formed between phytochemicals and aluminium alloy.

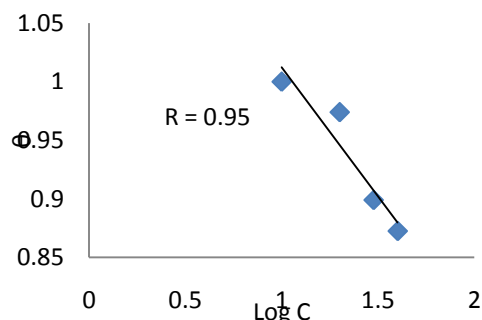


Figure 8: Plot of surface coverage ( $\theta$ ) with logarithm of concentration of acid extract of RV at a temperature of 300K in accordance with Temkin isotherm

Furthermore, the surface coverage strength study was conducted for different extract volume utilized at different time intervals of 1, 2, 3, 4 and 5 minutes as shown in Figure 9 to investigate if there are any influence of exposure times to the connection between  $\theta$  and C. The 2, 3, 4 and 5 minutes time curve showed the highest  $\theta$  value at extract volume of 10  $cm^3$ , while the 1 minute curve showed the least value at extract volume of 10  $cm^3$ . However, all the time curves showed a downtrend from the beginning to the end of the experiment as extract quantity increased. An



observation of Figure 9 shows that the surface coverage trend at different times can be depicted in the following order, 1 min < 5 min < 2 min < 4 min < 3 min.

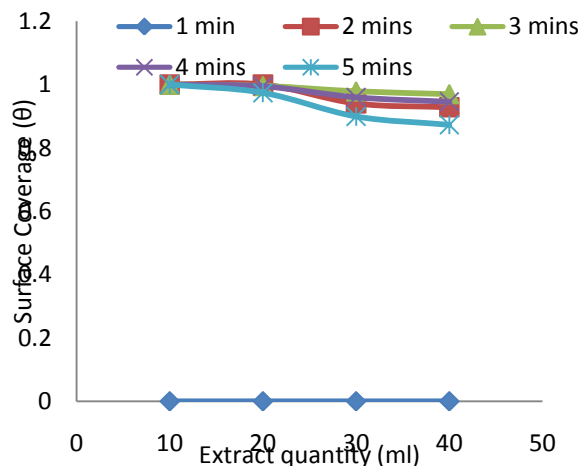


Figure 9: Variation of surface coverage ( $\theta$ ) with extract volume ( $\text{cm}^3$ ) at a temperature of 300K for different time intervals.

In addition, the values of  $\theta$  were also plotted against  $\ln C$  in Figure 10. Investigations on the adsorption phenomenon involved linking  $\theta$  with  $\ln C$  (natural logarithm of extract volume) using Equation 10 (Pandey et al, 2010):

$$\theta = \xi + \lambda \ln C \quad (10)$$

$$\text{where: } \xi(\text{ml/ml}) = \frac{RT}{b} \ln a \quad (11)$$

$$\lambda = \frac{RT}{b}$$

$a$  = Temkin constant related to the maximum adsorption capacity.

$b$  = Temkin constant related to the energy of adsorption.

$\theta$  = Surface coverage

$C$  = Extract quantity

$R$  and  $T$  are the gas constant ( $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$ ) and temperature (300 K) respectively. By comparing equation 10 to the straight line equation of the graph, it was possible to estimate the values of the energy of adsorption,  $b$ , and the maximum adsorption capacity,  $a$ , in terms of volume of hydrogen evolved to be  $26,117.3 \text{ J mol}^{-1}$  and  $2.494 \times 10^{-6}$  respectively.

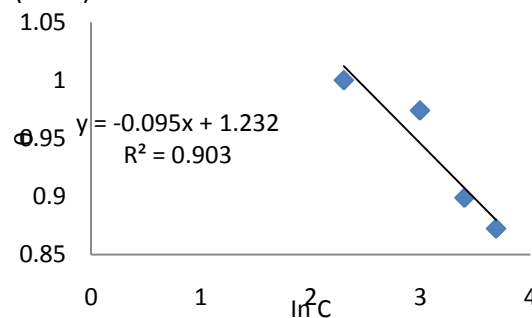


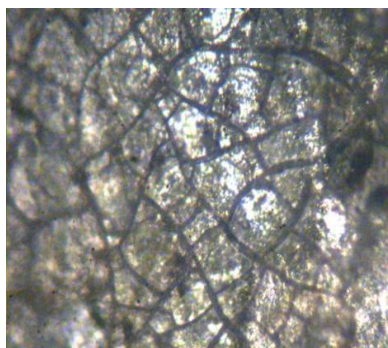
Figure 10: Plot of surface coverage ( $\theta$ ) with natural logarithm of extract quantity (ml) at a temperature of 300K.

The superficial effects of HCl action on the metal in the absence and presence of RV extract were investigated using optical microscope. Moreover, the studies were conducted on these samples with the aim of assessing the condition of the Al-alloy surface and grain structure. Nevertheless, the examinations were carried out on five metal samples which include that of the control experiment (having no inhibitor present) and samples from the 10, 20, 30 and 40 ml extract quantities. Superficial analysis was carried out and the micrograph of the alloy before the experiment as observed in Figure 11a, indicate the presence of three phases, which were the  $\alpha$ -Al (white) phase, the  $\text{Mg}_5\text{Al}_8$  (grey) phase and the thick spotted coarse  $\text{Mg}_2\text{Si}$  (black) phase. The first two mentioned phases were observed to be finely dispersed. In Figure 11b, which is the control sample, the micrograph indicate the presence of irregular flakes of the intermetallic phase along the grain boundaries which eventually resulted in deterioration. The effect of 10, 20, 30 and 40 ml acid extract of RV for over 60 minutes on the aluminium sample as shown in Figs. 11c, 11d, 11e and 11f respectively indicate that intergranular and pitting corrosion may not have occurred rather there were indication of noticeable grain roughening and/or reformation. The presence of surface flakes was ostensibly suppressed with increasing quantity of inhibitors than without. It was also shown that the irregular intermetallic phases were more regular after exposure to increasing quantity of inhibitors due to the adsorption efficiency of the RV extract which controls the acidic and cathodic processes that may affect the grain structure. Therefore, by comparing Figs. 11c, d, e and f, it is observed that the addition of RV extract has obviously slowed down the metal deterioration rate. It is therefore clear that the higher the RV extract

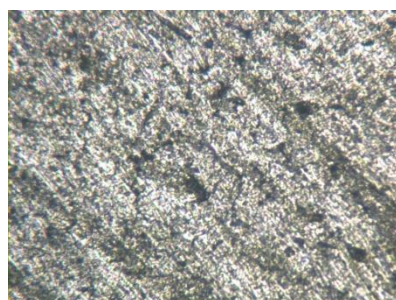
quantity the slower the degradation rate and the longer the life of the alloy.



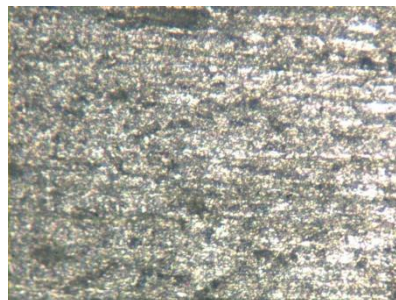
(a) Al metal before the experiment



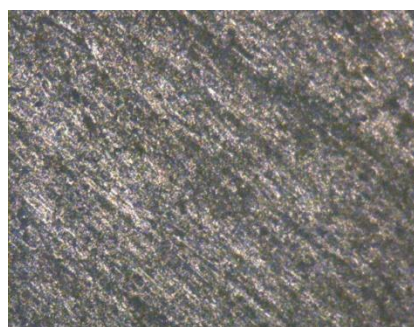
(b) Control



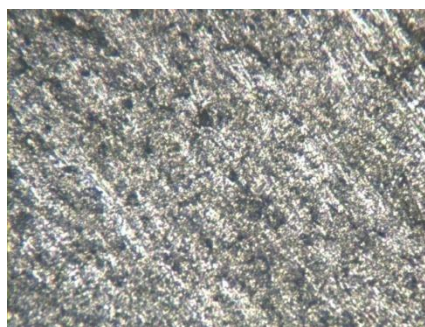
(c) Al metal in 10ml inhibitor



(d) Al metal in 20ml inhibitor



(e) Al metal in 30ml inhibitor



(f) Al metal in 40ml inhibitor

**Figure 11:** Micrographs for mild steel surface (a) before immersion in 2.5 M HCl solutions (b) after immersion in 2.5 M HCl for 60 min (c) after immersion in 10cm of RV extract (d) after immersion in 20 ml of RV extract.

## 4. Conclusions

The study investigated the environmentally induced damaged of Al-alloy by HCl acid in the presence of RV extract using gasometric and gravimetric techniques. Attempts were made to ascertain if any relationship existed: between  $H_2$  evolved and time; inhibitor efficiency and concentration; corrosion rate and time; hydrogen evolved and weight loss; and adsorption

mechanism with concentration. Several indices that portrayed the performance of the metal in the medium at different inhibitor concentrations were pinpointed. The results showed that as extract concentration was increasing,  $H_2$  evolution and corrosion rate increased, while only inhibitor efficiency reduced both for gasometric and gravimetric methods. The phytoconstituent-metal surface interaction mechanism was best described by the Temkin isotherm. The 3 minutes curve showed itself as





the best time for extract phytochemicals to adsorb to the alloy surface.

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