

Degradation Study of Aluminum Alloy in 2 M Hydrochloric Acid in the Presence of *Chromolaena odorata*

¹Oluseyi O. Ajayi, ¹Olugbenga A. Omotosho,

²Kolawole O. Ajanaku and ¹Babatunde O. Olawore

¹Department of Mechanical Engineering,

²Department of Chemistry, Covenant University, P.M.B. 1023, Ota, Nigeria

Abstract: The deterioration behaviour of aluminum alloy in 2 M hydrochloric acid solution in the presence of *Chromolaena odorata* extract was investigated using gasometric technique. Aluminum alloy coupons of dimension 4 by 1 cm were immersed in test solutions of uninhibited acid and also those containing extract concentrations of 0.065, 0.098, 0.13 and 0.16 M at ambient temperature for 30 min. The volumes of hydrogen gas evolved as a result of the rate of reaction were documented and analyzed. The outcome revealed that maximum inhibitor efficiency which corresponds to the lowest corrosion rate was obtained at optimum inhibitor concentration of 0.16 M with reduction in the corrosion rate observed to follow in order of increasing extract concentration. The adsorption study further revealed that Freundlich isotherm best described the metal surface interaction with the extract phyto-chemicals with 20 min becoming the best exposure time for the phyto-chemicals to adsorb to the metal surface at all concentrations. Statistically modeling the corrosion rate yielded an important relationship suitable for estimating corrosion rate values, once concentration of the extract is known. Furthermore, the microstructural studies revealed that crack growth rate slowed down considerably at 0.16 M extract concentration and irregular intermetallic phases were more uniform as extract concentration increased.

Key words: Environmentally induced failure, green inhibitors, hydrogen evolution, aluminum alloy, observed, volumes

INTRODUCTION

The phenomenon of machine components failure in service has been identified as a major industrial problem because of the economic losses associated with such destruction. Failure of machine components is caused in most instances by a load applied either cyclically or singly, direction of the critical overload and the influence of outside or environmental forces such as residual stresses or corrosion. The relationship between loads and deformation is such that in a sound design, the load is not excessive because the stress does not exceed the yield point and the part deforms elastically. When a fluctuating or fatigue load is applied to a machine component such as a shaft or rotating part, it experiences cracking as soon as the fatigue strength is exceeded.

This fatigue crack can slowly work its way across a part until fracture occurs and corrosion has been pinpointed as a major factor that can affect the fatigue strength of a mechanical component. Industrial operations of oil well acidizing, cleaning, pickling and descaling expose mechanical components to aggressive

species like hydrochloric or sulphuric acid. These aggressive agents have been found to be effective in removing rust, mill and oxide scale but they do so by reacting with the substrate metal to form metal ions, Hydrogen gas (H₂) and acid vapour. Depending on period of exposure, the reaction also leads to material wastage in the form of reduction in diameter and in effect reduces strength resulting in catastrophic failure. Retarding agents or catalysts often referred to as inhibitors are used to reduce the effect of these agents on the substrate metal (Fontana, 1987). These inhibitors are synthetic in nature and therefore not environmentally friendly meaning that its use place additional burden of disposal on the users and solution to such issues are not forthcoming.

Studies by researchers (Omotosho *et al.*, 2010; Ralston, 2008; Faltermeier, 1992; Iannuzzi *et al.*, 2006; Ralston *et al.*, 2009) have established that synthetic inhibitors are beneficial in reducing corrosion rate on one hand and deleterious on the other hand because of the toxic products released to the environment after usage. However, inhibitors from plant sources often referred to as green inhibitors have been found to be effective in

retarding the effect of these aggressive agents and at the same time easily and cheaply disposed without any side effects. Various studies have been reported on the protective action of green inhibitors on metals in acidic and alkaline solutions (Saratha *et al.*, 2009; Ebenso and Ekpe, 1996; Ebenso *et al.*, 1998, 2004; Ekpe *et al.*, 1994; Oguzie, 2006; Martinez and Tagljar, 2003) with a few of them focusing on Al-alloy (Obot and Obi-Egbedi, 2009; Abiola *et al.*, 2007; James and Akaranta, 2009). Most of these previous studies deduced corrosion rate from weight loss measurements without any analyzing data in such a way as to investigate if there are any contribution of times of exposure to the relationship between surface coverage and extract concentration. However, the approach adopted in this study was to employ the rate of evolution of H₂ gas as a measure of the rate at which the alloy is corroding in the presence of *Chromolaena odorata* (CO). The technique adopted in this study was based on the hypothesis that the rate at which the metal degraded after exposure corresponds to the rate of evolution of H₂ gas, since it was discovered that increased metal loss resulted in rapid rate of evolution of hydrogen gas.

CO belongs to the family asteraceae and its common names include Awolowo, independence weed, siam weed, trifid weed, bitter bush or jack in the bush (Okon and Amalu, 2003). In traditional medicine, it is used as an antispasmodic, antiprotozoal, antitypanosomal, antibacterial, antifungal, antihypertensive, anti-inflammatory, astringent, diuretic and hepatotropic agent (Iwu, 1993; Akinmoladun *et al.*, 2007). It is reported to contain alkaloids which include cyanogenic glycosides, flavonoids (aurone, chalcone, flavone and flavonol), phytates saponins and tannins.

Phytic acid and tannins are well reported non-toxic corrosion inhibitors for metal in aggressive media (Notoyaa *et al.*, 1995). Hence, the anti-corrosion activity of CO extract can be attributed to phytic acid and tannins. It is well known that H₂ gas is frequently evolved when metals in service degrade in the presence of acids. The rate at which the H₂ gas is evolved is equivalent to the rate of metal dissolution in the environment shown by weight loss. Thus if there is a way to measure, the volume of H₂ gas evolved as a relationship with the metal mass loss, the rate of corrosion of the metal within the environment can directly be simulated without necessarily leading to plant downtime. This will enable the engineers to have a pre-knowledge of the situation and know when to carry out specific type of repair. The focus of this study, therefore is to establish the relationship between corrosion rate and the rate of evolution of H₂ gas as well as determine other essential parameters that compare metal surface phenomenon in a particular environment.

The present study, therefore seeks to investigate the inhibitive effect of this extract on the environmentally assisted cracking of Al-alloy in 2.0 M Hydrochloride (HCl) using the gasometric technique at ambient temperature. Furthermore, inhibitor efficiency was determined by the Tafel method (Okafor *et al.*, 2010) and the corrosion rate modelled statistically. The investigation further goes on to analyze the adsorption mechanism of the extract and also its effect on the microstructure of the alloy.

MATERIALS AND METHODS

Rectangular specimens of Al metal coupon of dimension 4×1 cm were employed for the determination of corrosion inhibition of BP extract. Specimens were prepared by degreasing in ethanol and acetone. It was then allowed to dry.

The composition of the Al samples was analyzed using optical emission spectrometer and the result as obtained were (wt. %), Silicon (0.157%), Cadmium (0.0005%), Iron (0.282%), Calcium (0.0011%), Copper (0.0025%), Cobalt (<0.0010%), Manganese (0.024%), Lithium (<0.0002%), Magnesium (0.51%), Sodium (0.0005%), Zinc (<0.0010%), Phosphorous (<0.0010%), Nickel (<0.0010%), Lead (<0.0005%), Chromium (0.023%), Tin (<0.0010%), Titanium (0.0049%), Strontium (<0.0001%), Silver (<0.0001%), Vanadium (0.0035%), Boron (0.0007%), Zirconium (0.0020%), Beryllium (<0.0001%), Al (99%) and Bismuth (<0.0010%).

The acidic extracts of CO were prepared from its fresh leaves after it has been air dried and ground into powder. About 10 g of the powder was then put into a flat bottom flask containing 200 cm³ of 2 M HCl solution. The resulting solution was refluxed for 2 h and left over night before it was filtered.

Extract concentration of 2-5 cm³ corresponding to 0.065, 0.098, 0.13 and 0.16 M, respectively were employed for the investigation. The experimental set up is as shown in Fig. 1. Each specimen was dropped into the mylius cell containing 50 cm³ of test solution system at room temperature. The volume of H₂ gas evolved per 120 sec interval was recorded for 30 min in a calibrated tube by downward displacement of water and the plot of volume against the interval was carried out and shown in Fig. 2. The inhibition efficiency was then determined using the Tafel method (Okafor *et al.*, 2010) as:

$$\text{I.E. (\%)} = \frac{(V_H - V_{HI})}{V_H} \times 100 \quad (1)$$

Where, V_H is the volume of H₂ evolved without inhibitor (i.e., control experiment) and V_{HI} is the volume of

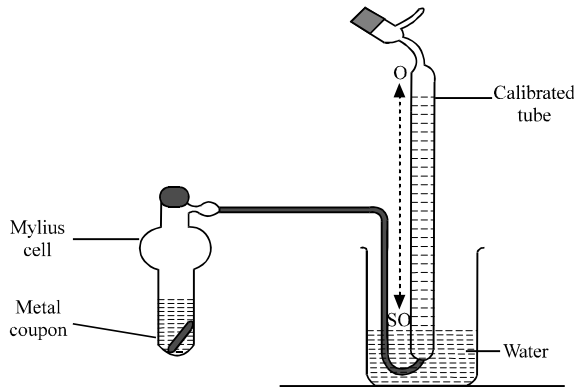


Fig. 1: Schematic diagram of the experimental set up

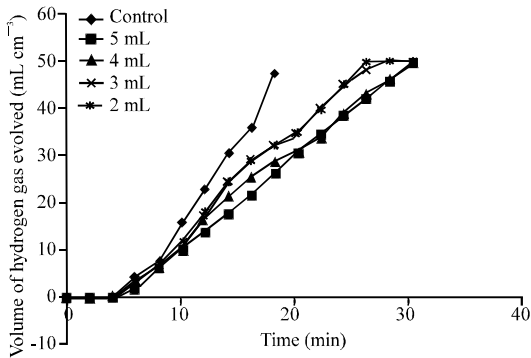


Fig. 2: Variation of volume of hydrogen (cm³) evolved with time (min) of Al coupons for different concentrations of CO extract in 2 M HCl at room temperature

H₂ evolved with inhibitor. Because H₂ gas is given off as one of the products of reaction when Al-alloy reacts with HCl, it is suitable to predict the rate of reaction by determining the rate of H₂ 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 CO extract.

Thus, the rate of corrosion corresponding to the rate of H₂ evolution was modeled based on the idea that the rate of H₂ gas evolution is directly proportional to the rate of corrosion which will also be directly proportional to the rate at which the weight of the metal is lost.

This therefore means that modeling the rate of corrosion from the direction of rate of H₂ gas evolution is indirectly a way to model the rate of material loss when the relationship between the weight loss and H₂ gas evolution is known. The foundation for this is as represented in Eq. 2:

$$\Delta V \propto \Delta W$$

$$\frac{d\Delta V}{dt} \propto \frac{d\Delta W}{dt}$$

but,

$$R \propto \frac{d\Delta W}{dt} \propto \frac{dV}{dt} \tag{2}$$

Where:

- α = Proportionality sign
- ΔV = Volume of H₂ gas evolved
- ΔW = Metal weight loss due to corrosion
- R = Rate of corrosion
- t = Time

Thus to determine Eq. 2, 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 H₂ gas against time leading to Eq. 3:

$$V = c + bt + at^2 \tag{3}$$

Thus:

$$R = \frac{dV}{dt} = b + 2at \tag{4}$$

A particular case of this study will involve adapting Eq. 3 and 4 to the volume-time measurement for individual concentration. For instance for measurements relating to 5 cm³ extract concentration, the corrosion rate model is shown as Eq. 6:

$$V = -1.935 + 0.587t + 0.0165t^2 \tag{5}$$

$$\frac{dV}{dt} = 0.587 + 0.033t \tag{6}$$

RESULTS AND DISCUSSION

A keen observation of Fig. 2 shows that the corrosion rate of Al-alloy as indicated by the amount of H₂ evolved decreased in the presence of CO when compared to the control. The amount of H₂ evolved also decreased with increasing concentration of the extract. This indicates that the CO extract in the solution has an inhibitive effect on the corrosion of Al in HCl and that the extent of inhibition depends on the amount of CO extract present.

This same trend was also observed in Fig. 3 which shows the percentage inhibition efficiency (I.E. %). However, the I.E.% readings for samples in the 2 and 3 mL extract and also the 4 and 5 mL extract were observed to be very closely related thus, pointing to the fact that there was only slight improvement in the performance of the extract when concentration was increased from 2-3 mL

and from 4-5 mL. This means that there might be a need to further increase extract concentration in order to increase I.E. (%) values since, the increases only produced a marginal improvement. Further observation of I.E. (%) values showed that samples in the 5 cm³ extract concentration had the best performance followed the by 4 cm³ extract and then closely followed by the 3 and 2 cm³ extract.

All extract concentration had peak I.E. (%) values between 12 and 16 min, thus indicating the period for optimal performance or best exposure time range. However, the inhibitive effects depreciated at about the same time (16th min) for all inhibitor concentration.

Furthermore from Fig. 4, it was observed that the rate of corrosion, generally decreases in the presence of CO extract. The 2 cm³ extract of CO shows the lowest effect of reducing the corrosion rate of Al followed very closely by the 3 cm³ extract. The 5 cm³ extract however, began by slowing down the rate but towards the latter part of the reaction, the rate tends to increase above every other concentration of the inhibitor though only slightly. The reduction in corrosion rate in the presence of CO extract

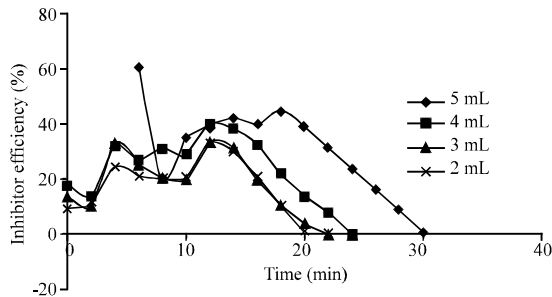


Fig. 3: Percentage inhibition efficiency of varying concentrations of CO extracts with time (min) on Al coupon in 2 M HCl solution at ambient temperature

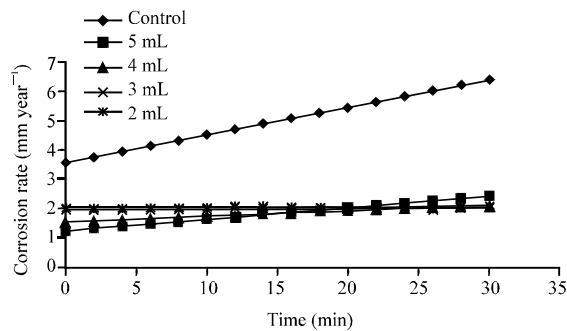


Fig. 4: Corrosion rate of varying concentrations of CO extract with time (min) on Al coupon in 2 M HCl solution at room temperature

at different concentrations was observed to follow the order; 2<3<4<5 cm³ extract. Further investigating, the relationship between corrosion rate, R and the changing concentration to obtain reaction constant and also the specific reaction constant for the HCl-corrosion of Al-alloy in the presence of CO extract involved carrying out a statistical regression analysis of the values of R against those of concentration. However, it has been proven (Ehteram and Al-Moubaraki, 2008; Mathur and Vasudevan, 1982) that corrosion rates can be correlated with acid concentration with the equation:

$$\text{LogR} = \text{Logk} + \text{BLogC}$$

Where:

R = Corrosion rate

K = Reaction constant

B = Specific reaction constant

C = Concentration

Therefore to obtain reaction constant, the corrosion rates obtained from H₂ evolution method must be converted to the same unit (mol min⁻¹) by assuming that H₂ evolution reaction occurred at 1.01325×10⁻⁵ Pa. Figure 5 shows the relationship between Log R and Log C for the Al-alloy coupon sample.

The straight line in the graph correlates all the points with correlation coefficient of 0.52. The estimated k and B values were 9.33×10⁻⁷ mol min⁻¹ and 0.072.

Adsorption studies: The mechanism of interaction between the phytochemicals in the CO extract and the metal surface can be explained using various adsorption isotherms namely Langmuir, Freundlich, Boris-Swinkels and Temkin. The degree of surface coverage, θ for the different inhibitor concentrations were evaluated based on volume of H₂ gas evolved measurements. Efforts were then made to fit the θ values with the different adsorption isotherms and the Freundlich isotherm was found to be best fitted to the θ values. This was expected as the Freundlich isotherm which is an empirical equation has the ability to fit nearly all experimental adsorption data

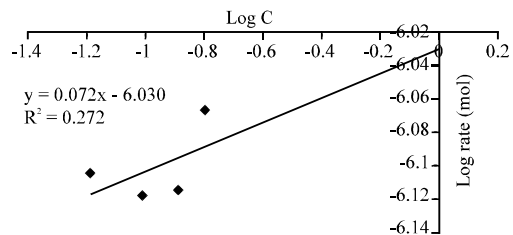


Fig. 5: Plot of log of corrosion rate against log of the acid extract concentration

(Febrianto *et al.*, 2009). It is also amongst the most widely used isotherms for the description of adsorption equilibrium and capable of describing the adsorption of organic compounds. The Freundlich equation is expressed as:

$$\theta = K_c C^{1/p}$$

And in linearized logarithm as:

$$\log\theta = \log K_c + \frac{1}{p} \log C$$

Where:

θ = Surface coverage

K_c = Characteristic constant related to the adsorption capacity

C = Concentration

p = Characteristic constant related to adsorption intensity or degree of favourability of adsorption

A graph of $\log\theta$ versus $\log C$ has a slope value of $1/p$ and an intercept magnitude of $\log K_c$ as shown in Fig. 6. The value of $\log K_c$ is equivalent to $\log \theta$ when C equals unity. From the plot and equation of the straight line obtained, the estimated value of p and K_c is 3.95 and 1.879 while a good correlation coefficient of 0.98 was also obtained. However, the value of p depicts a favourable adsorption which is defined by a Freundlich constant that has a value between 1 and 10 (Febrianto *et al.*, 2009).

This result therefore, confirms the assumption that the corrosion inhibition of the CO extract is attributed to the adsorption of molecules of phyto-chemicals on the metal surface by a physical adsorption process where a bond is formed between phytochemicals and Al-alloy. Typically, the adsorption process is known to take place easily when free energy change is a negative value. The adsorption free energy can be obtained or calculated by using the Gibbs equation (Yang *et al.*, 2009):

$$\Delta G = -RT \int_0^a P \frac{da}{a} \quad (7)$$

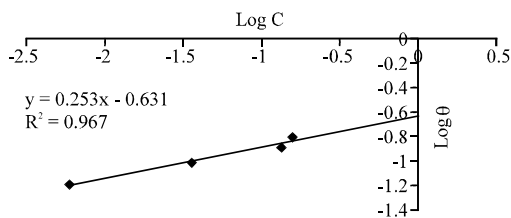


Fig. 6: Variation of logarithm of surface coverage (θ) with logarithm of concentration of acid extract showing conformity with Freundlich isotherm

From the Freundlich isotherm, the Gibbs equation (Eq. 7) can be expressed as:

$$\Delta G = -pRT \quad (8)$$

Where, ΔG , p , R and T are the free energy change, parameter of the Freundlich equation, gas constant and temperature, respectively. Therefore, it is clear that adsorption of CO extract on the Al-alloy is a spontaneous process because of the negative value of ΔG ($-9.786 \text{ KJ mol}^{-1}$). In addition, the degree of surface coverage, θ for the extract at different concentration was plotted for different time intervals of 8, 12, 16, 20 and 24 min as shown in Fig. 7 to investigate if there are any contribution of times of exposure to the relationship between θ and C .

For the 8 and 24 min curve, the surface coverage, θ was very low at extract concentration of 0.098 M but the rate of surface coverage during this time frame increased rapidly this concentration until it reached the optimum level of 0.2 and 0.232. This shows that during these time frames, the Al-alloy experienced the lowest surface coverage by the phytochemicals in the extract.

However for the other time frames of 12 and 16 min, changes in θ were very similar and the values were closely related. This means that during these time frames surface coverage of CO extract at different concentration ranged between 0.2038 and 0.388. The θ values displayed during the 20 min time frame was the highest across all the concentration showing that this time frame is the best for the phytochemicals to adsorb to the Al-alloy surface and inhibit corrosion effectively.

The surface effects of the HCl action on the metal in the presence of CO extract were investigated using optical microscope. Also, the photomicrograph studies were performed on these samples in order to evaluate the condition of the Al-alloy surface and grain structure. However, the investigation were carried out on three metal

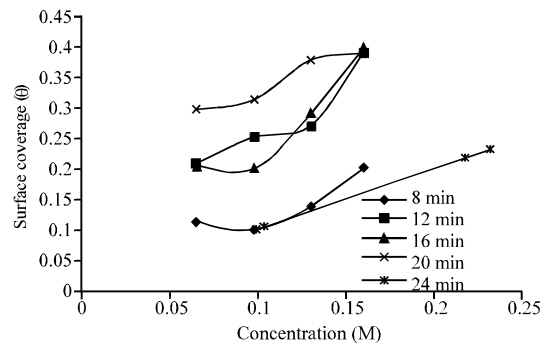


Fig. 7: Variation of surface coverage (θ) with extract concentration (M) at different time intervals

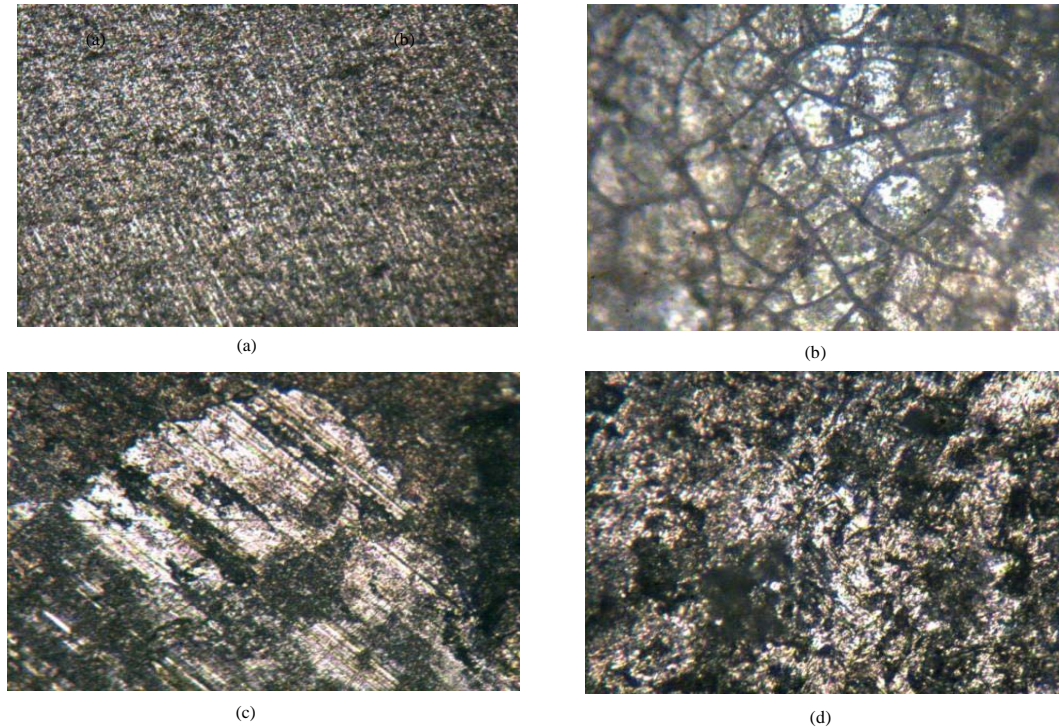


Fig. 8: Micrographs for Aluminum surface (a) before immersion in 2 M HCl solutions (b) after immersion in 2 M HCl for 30 min (c) after immersion in 0.13 M of CO extract for 30 min and (d) after immersion in 0.16 M of CO extract for 30 min

samples which include that of the control experiment (having no inhibitor present), sample from the second highest inhibitor concentration (sample from 0.013 M or 4 cm³ extract) and sample from the highest extract concentration. These were chosen to study the phenomenon on the case scenario of direct 2.0 M HCl attack, the second highest and highest inhibitive effect scenarios, knowing that all others will fall within these limits.

Superficial analysis was carried out and the micrograph of the alloy before immersion as observed in Fig. 8a, shows the presence of three phases which were the α -Al (white) phase, the Mg₂Al₃ (grey) phase and the thick spotted coarse Mg₂Si (black) phase. The first two mentioned phases were observed to be finely dispersed.

In Fig. 8b 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 intergranular cracks. The influence of 0.13 and 0.16 M acid extract of CO for 30 min on the aluminum specimen as shown in Fig. 8c and d, respectively shows that intergranular and pitting corrosion may not have occurred rather there were evidence of prominent grain coarsening and/or restructuring. The appearance of surface flakes was

apparently lowered with increased concentration of inhibitors than without. Figure 8c showed a slight introduction of crack along the α -Al phase. Corrosion attack was reduced in the 0.16 M extract than the 0.13 M extract, since the sample in the 0.16 M extract showed that the α -phase was present and coarse with no evidence of crack.

It was also discovered that the irregular intermetallic phases were more uniform after exposure to the 0.16 M extract due to the adsorption efficiency of the CO extract which controls the acidic and cathodic reaction that may affect the grain structure.

Therefore by comparing Fig. 8c and d, it is observed that higher extract concentration has apparently slowed down the crack growth rate thereby prolonging the life of the alloy.

CONCLUSION

The study investigated the environmentally induced damage of Al-alloy by HCl acid in the presence of CO extract using gasometric technique. The various indicators that characterized the behaviour of the alloy in the medium at different inhibitor concentration were highlighted and a new relationship that represents a vital contribution to knowledge was also obtained.

Investigations revealed that maximum I.E.% and lowest corrosion rate were obtained at optimum extract concentration of 0.16 M. The mechanism of interaction between the phytochemicals in the plant extract and Al-alloy surface was best described by the Freundlich isotherm which is an empirical equation in nature having the ability to fit nearly all experimental adsorption data.

The 20 min time frame was also revealed as the best exposure time for the phytochemicals at all concentrations utilized to suitably adsorb to alloy surface. Statistical modeling of corrosion rate yielded a vital relationship suitable for estimating corrosion rate once concentration of CO extract is known.

The microstructural studies revealed that crack growth rate slowed down considerably at 0.16 M extract concentration and irregular intermetallic phases were more uniform as extract concentration increased which is in agreement with the initial inference of lowest corrosion rate at optimum extract concentration.

Based on the outcome of the investigation, the researchers suggest the development and calibration of an apparatus that measures corrosion rate based on the theory that metal depletion after exposure is directly proportional to the volume of H₂ gas evolved. This may require that further tests or experiments be conducted to determine the exact relationship between the volume of H₂ gas evolved and the corresponding amount of metal depleted.

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