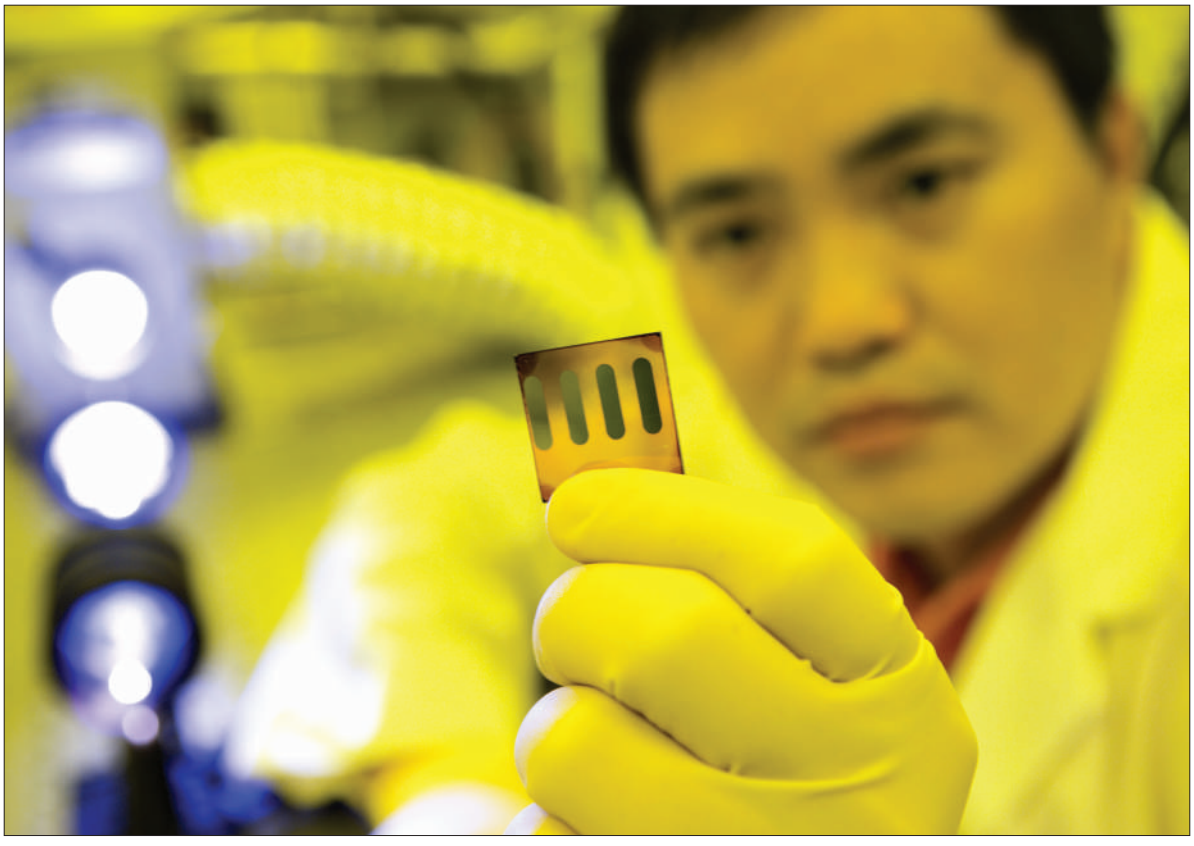

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Degradation Evaluation of Zinc in 2 M Hydrochloric Acid in the Presence of *Bambusa bambos*

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ABSTRACT

Failure evaluation of zinc sample in 2 M hydrochloric acid solution in the presence of *Bambusa bambos* extract was conducted using the gasometric method. Zinc coupons, each measuring 2.5 by 1.5 cm were completely immersed in uninhibited and inhibited test solutions containing extract quantities of 60, 100 140 and 180 mL at a temperature of 298 K for 20 minutes. The volumes of hydrogen gas data obtained during the experiment were documented and analyzed. Results showed that maximum inhibitor efficiency and lowest corrosion rate were obtained at extract quantities of 140 and 100 mL, respectively while corrosion rate reduction did not follow any particular trend. Frumkin isotherm best explained the extract-metal surface interaction adsorption mechanism. Once quantity of the extract is known, an expression for estimating corrosion rate values was also obtained. In addition, the surface analysis indicated that the rate of deterioration of the metal reduced as extract quantity increased to some extent and changed thereafter.

Key words: Gasometric, hydrogen evolution, corrosion, HCl, metal-phytochemical interaction mechanism

INTRODUCTION

An adequate and environmentally safe corrosion protection technique has been linked to improved and sustainable pollution free environment. The challenges associated with the use of harmful synthetic inhibitors in allied chemical industries have continuously been heightened by concerns for the environment. Although synthetic inhibitors like aniline and sodium nitrite (Okeniyi *et al*., 2012) represent a rich source of effective inhibitors, its damaging nature is a concern for the future. Thus, employing environmentally friendly sources for corrosion protection has gained wide acceptance across the globe. This is because these green sources have sustained potentials for abundant and cheap supply.

The use of plants as corrosion inhibitors requires establishing if they contain substances such as tannins, terpenes, alcohols, polyphenols, carboxylic acids and alkaloid. These substances have been proven to have corrosion inhibiting potentials (Loto *et al*., 2011; Saratha *et al*., 2009; Singh *et al*., 2010; Valek and Martinez, 2007; Oguzie, 2006, 2008; Okafor *et al*., 2008, 2010; De Souza and Spinelli, 2009; El-Etre, 2003; Ajayi *et al*., 2011a-c; Prabhu *et al*., 2009). In the case of Bambusa Bambos (BB), its leaves have been established to contain substances such as tannin and lignin which are capable of inhibiting corrosion. In addition, it is environmentally friendly and not harmful to humans.

Thus, several researchers have investigated the use of some plant extracts on the corrosion of zinc in acidic media (Abiola and James, 2010; El-Etre *et al*., 2005; Mahmoud, 2008; El-Gaber *et al*., 2008; Shylesha *et al*., 2011; Wang *et al*., 2003; Ghanbari *et al*., 2009; El-Aila *et al*., 2011) and all such studies suggest that, apart from the fact that green inhibitors can be cheaply sourced and applied without contaminating the environment, they are also organic compounds containing electron donor atoms particularly nitrogen, sulfur and oxygen in their functional groups with aromatic and heterocyclic rings with corrosion inhibiting capability. However, none of the investigations were done on zinc in Hydrochloric acid (HCl) in the presence of BB. For instance, Abiola and James (2010) reported the effects of Aloe vera extract on the corrosion of zinc in HCl solution using weight loss methods, while El-Etre *et al*. (2005) reported the outcome of the effect of henna (*lawsonia*) leaf extract on the corrosion inhibition of carbon steel, nickel and zinc in acidic, neutral and alkaline solutions employing polarization technique.

Thus, these reports are different from the focus of the present study whose object is to investigate the effect of acid extract of BB on the deterioration of zinc in 2 M HCl solution at a temperature of 298 K using gasometric method. Furthermore, this investigation also analyzed the various indices that portrayed the behavior of zinc metal in relation with extract quantity, metal-phytochemical extract adsorption interaction mechanism and micrograph examination. In addition, Inhibitor Efficiency (I.E) was determined by the method adopted by Ajayi *et al*. (2011a) and Okafor *et al*. (2010).

MATERIALS AND METHODS

Corrosion tests using gasometric measurements were performed using zinc metal coupons of dimensions 2.5×1.5 cm. The metal sheets were scraps purchased from a metal stockist and it was cut into dimensions suitable for the experiment. Surface preparation of the coupons involved degreasing in ethanol and drying in acetone. The coupons were then stored in a moisture free desiccator to avoid contamination before their use for the corrosion study. Weight percent compositions of the metal samples were analyzed using Optical Emission Spectrometer (OES) and the result obtained is shown in Table 1. Extracts of Bambusa Bambos (BB) were made from its fresh leaves by drying and then pulverizing into powder. The entire powdered mass was weighed, so that 20 g of it could be put into a 200 cm^3 flat bottom flask containing 100 mL of 2 M HCl solution. The extracts were prepared by refluxing the resulting solution for 3 h at 343 K (70°C) and it was then left to cool overnight. Filtration was done on the mixture and the filtrate obtained was taken as the stock inhibitor solution from which different quantities of the inhibitor solution was made. The quantities of the extract used for the study were 60,100 140 and 180 ml L^{-1} and they were prepared by serial dilution. Experimental set up was analogous to the technique adopted

Table 1: Composition of zinc sample employed for the investigation

Element	Content (%)
Al	0.0140
Cu	0.0049
Pb	0.60
Sn	<0.0010
Fe	0.01
Cd	0.02
Sb	<0.0001
Zn	99.40

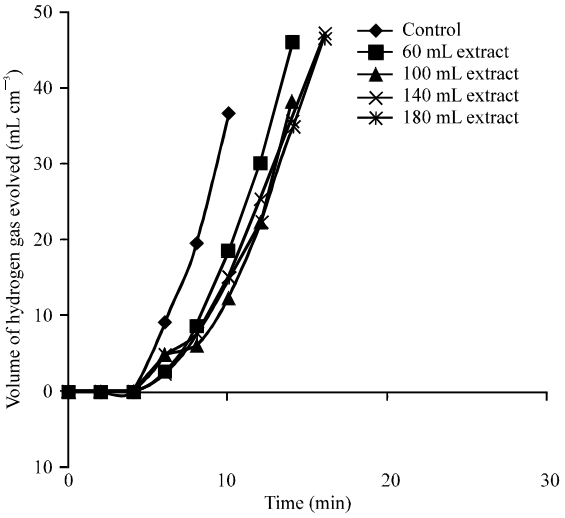

Fig. 1: Relationship of volume of H_2 (cm^3) evolved with time (min) of zinc coupons for different amounts of BB extract in 2 M HCl at a temperature of 298K

elsewhere (Ajayi *et al.*, 2011a-c; Omotosho *et al.*, 2012). Each zinc sample was put into the mylius cell containing 50 cm^3 of test solution and the experiments were conducted at ambient temperature of 298 K. The volume of Hydrogen (H_2) gas evolved and trapped in the inverted burette per 2 min interval was recorded for 20 min in a calibrated tube by downward displacement of water and the graph of volume of hydrogen evolved against time interval was plotted and is shown in Fig. 1.

The inhibition efficiency was computed by using Eq. 1 (Okafor *et al.*, 2010; Ajayi *et al.*, 2011a-c; Omotosho *et al.*, 2012):

$$\text{I.E}(\%) = \frac{(V - V_1)}{V} \times 100 \quad (1)$$

where, V and V_1, are the volumes of H_2 evolved from solutions without inhibitor (i.e., control experiment) and with inhibitor, respectively.

Since, it is a well known fact that hydrogen is given of when metals react with acids, it is therefore correct to associate reaction rate of corrosion deterioration of the zinc in HCl in the presence of BB extract with H_2 evolution. The method employed in literature (Ajayi *et al.*, 2011a-c; Omotosho *et al.*, 2012) was used in this study to also model the corrosion rate which explains the hydrogen evolution rate. From the foregoing, the hydrogen evolution rate has a close link with the rate at which metal weight is lost. Thus, in line with Ajayi *et al.* (2011c) corrosion rate analysis from the direction of H_2 gas evolution rate is completely a way of modeling weight loss rate when the link between the weight loss and H_2 gas discharged is proven.

Hence, Eq. 2 was generated (Ajayi *et al.*, 2011a-c; Omotosho *et al.*, 2012):

$$R \alpha \frac{dW}{dt} \alpha \frac{dV}{dt} \quad (2)$$

Where:

α = Proportionality sign
V = Volume of H_2 gas evolved
W = Metal weight loss due to corrosion reaction
R = Rate of corrosion
T = Time

Equation 2 was generated by the analysis of relating volume evolved with the time of evolution. Consequently, a polynomial regression analysis of the volume of H_2 gas evolved against time was done and it led to Eq. 3 (Ajayi *et al.*, 2011a-c; Omotosho *et al.*, 2012):

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

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

For measurements linked to 100 mL extract quantity, the corrosion rate model is represented as Eq 6. Via the alignment of Eq. 3 and 4 according to the technique used elsewhere (Ajayi *et al.*, 2011a-c; Omotosho *et al.*, 2012), Eq. 5 and 6 sufficed.

$$V = 1.3458-1.6485t+0.2951t^2 \tag{5}$$

$$\frac{dV}{dt} = -1.6485 + 0.5902t \tag{6}$$

RESULTS AND DISCUSSION

Figure 1 showed that the rate of corrosion of the zinc coupon in the control as indicated by the amount of H_2 evolved was the highest. Corrosion rate of zinc coupons immersed in the inhibited solutions declined significantly in contrast to the control. It was also shown to some extent that the amount of H_2 evolved also reduced as the quantity of the extract increased, considering that the 100, 140 and 180 mL extract quantities showed closely related values. This suggests that the BB extract in the solution slowed down the corrosion of zinc in HCl. As a result, the level of inhibition can be said to be controlled by the quantity of BB extract in solution. A slightly identical trend was also depicted in Fig. 2 in which the percentage inhibition efficiency (% I.E) was represented. The % I.E. readings for samples in the 60 and 140 mL extract quantities were closely related from the beginning of experiment to the 6th min of the experiment. The samples immersed in the 100 and 180 mL extract quantities also showed a similar behavior from the beginning to the 6th min of the experiment. However, in both cases % I.E. readings became slightly different as the experiment progressed to the end. From Fig. 2, it was also shown that peak % I.E. values of 0.714 and 0.736 was attained by samples in the 60 and 140 mL extract quantities at the 6th of the experiment. At the 10th min of the experiment % I.E. values attained by samples in all extract quantities were lower than at the 6th min. The % I.E. readings on the basis of average values in increasing order can be indicated as; 60 mL extract<180 mL extract<100 mL extract<140 mL extract.

Figure 3 revealed that in comparison to the control the corrosion rate at a temperature of 298 K, decreases in the presence of BB extract. The extract quantity of 60 mL showed the least effect of reducing the corrosion rate of zinc coupon. The corrosion rate values for the sample

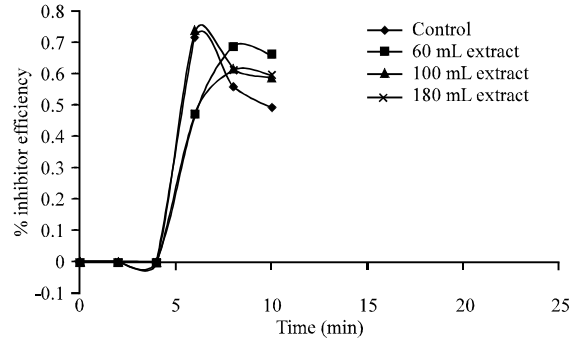

Fig. 2: Percentage inhibition efficiency of varying quantities of BB extracts with time (min) on zinc coupons in 2 M HCl solution at a temperature of 298 K

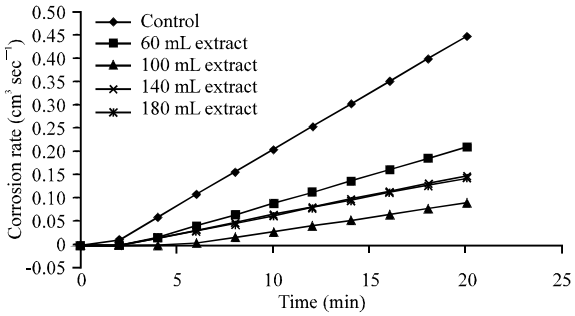

Fig. 3: Corrosion rate of varying quantities of BB extract with time (min) on zinc coupon in 2 M HCl solution at a temperature of 298 K

immersed in the 60 mL extract began at 0.018 cm^3 sec^{-1} and gradually increased until the end of the experiment at 0.21 sec^{-1}. Corrosion rate of sample immersed in the 140 and 180 mL extract solution which followed the 60 cm^3 extract in terms of reducing corrosion rate began at 0.017 cm^3 sec^{-1} and ended with values of 0.147 and 0.143 cm^3 sec^{-1}, respectively.

Sample immersed in the 100 mL extract produced the best result; its corrosion rate values began and ended at 0.006 and 0.0090 cm^3 sec^{-1}, respectively. Corrosion rate values on the average for the 60, 100, 140 and 180 mL extract quantities were 0.114, 0.048, 0.082 and 0.00722 cm^3 sec^{-1}, respectively. When these average values are weighed against that of the control (0.229 cm^3 sec^{-1}) it is obvious that the inhibitor was effective. The results also indicate that corrosion rate reduced as extract quantity increased to some extent since from the plot in Fig. 3 the 100 mL extract quantity is shown to be the best while the values for the 140 and 180 were closely related but better than the 60 mL extract. Corrosion rate reduction in terms of the average values from Fig. 3 was therefore observed to follow the order; control<60 cm^3 extract<140 cm^3 extract<180 extract<100 cm^3 extract.

Further corrosion study involved a regression analysis of the values of corrosion rate R against that of extract quantities. This was done to estimate the reaction constant and the specific reaction constant of the reaction by examining the relationship between corrosion rate and varying inhibitor quantity. This same procedure was adopted according to literature (Ajayi *et al.*, 2011a-c). It was revealed that corrosion rates can be linked with acid concentration with Eq. 7 (Mathur and Vasudevan, 1982; Noor and Al-Moubaraki, 2008; Ajayi *et al.*, 2011a-c; Omotosho *et al.*, 2012):

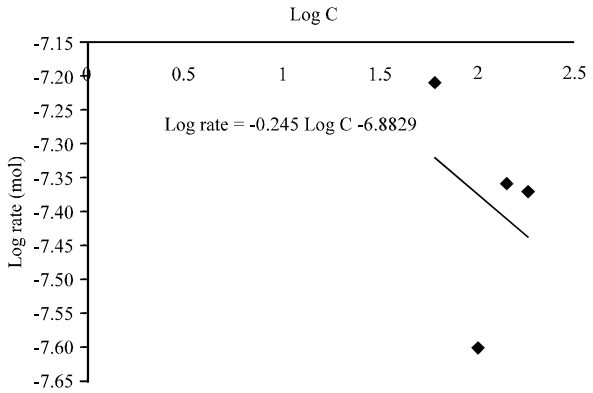

Fig. 4: Plot of Log of corrosion rate against log of the acid extract quantity

$$\text{Log}R = \text{Log}k + B\ \text{log}C \tag{7}$$

Where:
R = Corrosion rate
k = Reaction constant,
C = Specific reaction constant and
C = Concentration

Thus, in order to determine reaction constant it became essential to use same unit (mol min^{-1}). This was done by assuming that hydrogen evolution took place at 1.01325×10^{-5} Pa. The connection between Log R and Log C shown in Fig. 4 is for the zinc coupon sample and a correlation coefficient of 0.314 was obtained for the linear plot. From the linear expression obtained from the graph, the k and B values were calculated to be 1.309×10^{-7} mol min^{-1} and -0.245, respectively. The negative value of B obtained in this investigation suggests a decreasing slope which is at variance to other studies (Mathur and Vasudevan, 1982; Noor and Al-Moubaraki, 2008) where no inhibitors were used. This is worthy of note because it describes the inhibitive action of the BB extract on zinc coupon corrosion. Consequently, Eq. 7 was streamlined to obtain Eq. 8:

$$R = KC^{B} \tag{8}$$

And for the particular reaction Eq. 8 is written as:

$$R = 1.309\text{x}10^{-7}\ C^{-0.245} \tag{9}$$

Equation 9 completely explains the observations in Fig. 1 and 3 which shows an apparent variance between the uninhibited and inhibited solution.

Adsorption studies: The mechanism of interaction at the interface between phytoconstituents in the BB extract and the metal can be validated using various adsorption isotherms namely Langmuir (0.827), Freundlich (0.518), Temkin (0.495), Frumkin (0.866) and Boris-Swinkels (0.657). The degree of surface coverage, θ, for the different extract quantity was evaluated based on volume of H_2 gas evolved measurements. Also, the degree of corrosion inhibition depends on the surface conditions and mode of adsorption of inhibitors. The Frumkin isotherm was found to be best

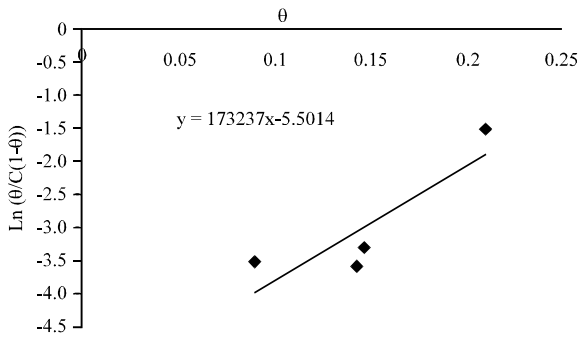

Fig. 5: Variation of Ln (θ/C(1-θ)) with surface coverage (θ) of acid extract showing conformity with Frumkin isotherm

fitted to the θ values as experimental data were made to fit with the different adsorption isotherms. The Frumkin isotherm expression (Ajayi *et al.*, 2011c) is represented as:

$$\mathrm{Ln}\left(\frac{\theta}{C(1-\theta)}\right)=\mathrm{Ln}\varphi+2\mu\theta \tag{10}$$

Where:

θ = Surface coverage
C = Quantity of inhibitor based on serial dilution
φ = Adsorption reaction binding constant
μ = Lateral interaction term describing the molecular interactions in the adsorption layer and the heterogeneity of the surface (it is a measure for the steepness of the adsorption isotherm)

The precise relationship between Ln (θ/C(1-θ)) and θ as shown in Fig. 5 was obtained by carrying out a linear regression analysis of Ln (θ/C(1-θ)) against θ which lead to Eq. 11:

$$y = 17.237x-5.5014\ (R^2 = 74.92\%) \tag{11}$$

In a bid to deduce values for φ and μ, a comparison of Eq. 11 with the Frumkin isotherm equation is necessary.

Therefore:

$$\mathrm{Ln}\varphi = -5.5014$$

$$\varphi = e^{-5.5014} = 0.00408$$

$$\mu=\frac{17.237}{2}=8.6185$$

The low value of φ shows that the investigated inhibitor is physically adsorbed on the zinc metal surface, while the positive value of μ infers that the interaction between the molecules boosts the adsorption energy with the increase of θ.

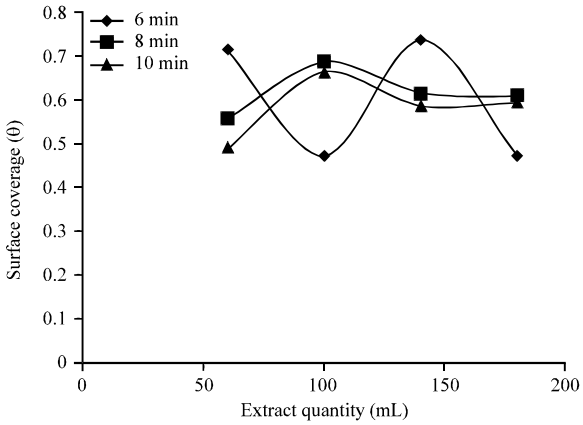

Fig. 6: Relationship of surface coverage (θ) with extract quantity (mL) at different time intervals

Additionally, the degree of surface coverage, θ, for the extract at different quantities was plotted for time intervals of 6, 8 and 10 min as shown in Fig. 6 to examine if there is any effect of times of exposure to the relationship between θ and C. The 6 min curve had the highest surface coverage value when the extract quantity was 60 mL, while the 8 and 10 min curve though closely related were second and third respectively. This implies that the 6 min time frame is the best for the phytochemical in the extract quantity of 60 mL to suitably adsorb to the metal surface. The 8 and 10 min curves showed closely related values that were higher than the 6 min curve at extract quantity of 100 mL, implying better adsorption at this extract quantity. Again the 6 min curve displayed better surface coverage when extract quantity was 140 mL, with the 8 and 10 min curve having closely related lower θ values. Finally at extract quantity of 180 mL the 8 and 10 min curves showed the best θ values with that of the 6 min curve being the lowest. Overall in terms of average θ values the best time frame across all extract quantities used is the 8 min curve. This was closely followed by the 6 and 10 min curve. Also the highest θ value of 0.736 was attained at extract quantity of 140 mL at the 6th minute of the experiment.

The surface effects of the HCl action on the zinc metal in the presence of BB extract were examined using optical microscope. The photomicrograph studies were performed on these samples in order to evaluate the condition of the zinc metal surface and grain structure. However, since increasing the extract quantities used did not translate to increased reduction in corrosion rate, the surface effects of all the samples used for the experiment was not done. Hence, the investigation were carried out on three metal samples which include that of the control experiment (having no inhibitor present), sample from the 60 and 100 mL extract quantity. These were chosen to study the phenomenon on the case scenario of direct 2 M HCl attack, the least and next to the least inhibitive effect scenarios, knowing that all others will fall within these limits. In addition, the values of the various indicators that characterized the behavior of samples in the 60 and 100 mL extract as well as 140 and 180 mL extract were closely related after immersion. The surface analysis was carried out and the micrograph of the metal before immersion as observed in Fig. 7a, indicate the presence of all phases with even distribution. In Fig. 7b which is the control sample, the micrograph indicate general corrosion with many localized pits at several sites. The black spots are evidences of the localized pits and severe corrosion.. In Fig. 7c, the chemical attack on the metal is such that the black spots are less in number showing that the localized pits is not as much as what was observed in the previous sample (control). However, in Fig 7d the uniform corrosion and

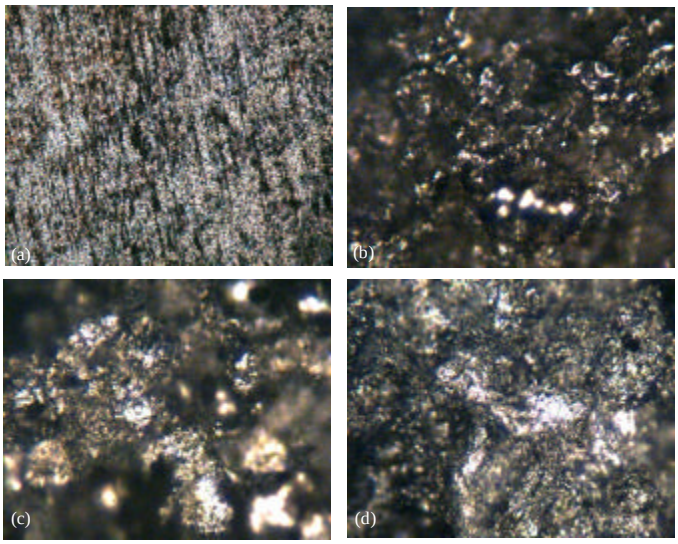

Fig. 7(a-d): Micrographs for zinc surface (a) before immersion in 2 M HCl solutions (b) after immersion in 2 M HCl for 20 min (c) after immersion in 60 mL of BB extract for 20 min (d) after immersion in 100 mL of BB extract for 20 min

localized pit formation is far less than the control and the severity of attack is also drastically reduced suggesting some level of corrosion inhibition. Therefore, exposure of the metal to BB extract increased the adsorption efficiency at the metal extract interface which slowed down the metallic deterioration.

CONCLUSION

The investigation examined and evaluated the damage of zinc by HCl acid in the presence of BB extract using the gasometric method. Several indices that described the behavior of the alloy in the medium at different inhibitor quantity were pinpointed and a relationship for corrosion rate measurement was also obtained. Results showed that maximum % I.E and lowest corrosion rate were obtained at extract quantities of 140 and 100 mL. The mechanism of interaction between the phytochemicals in the plant extract and zinc surface was best described by the Frumkin isotherm. The results also revealed that, the best time for phytochemicals to suitably adsorb to metal surface was 6 min at extract quantity of 140 mL. Statistical modeling of corrosion rate yielded a significant relationship suitable for estimating corrosion rate once quantity of BB extract is known. The superficial analysis revealed that rate of deterioration of the metal slowed down as extract quantity increased to some extent.

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