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**Assessing the deterioration behaviour of mild steel in 2 M sulphuric acid
using *Bambusa glauscescens***

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ABSTRACT

The study investigated the deterioration of mild steel in 2 M sulphuric acid solution in the presence of *Bambusa glauscescens* extract using gasometric technique. Steel coupons measuring 4 cm by 1.5 cm were immersed in test solutions of uninhibited acid and also those containing extract concentrations of 60, 100 and 140 cm³ at a temperature of 333K for 60 minutes. The volumes of hydrogen gas evolved from the reaction were documented and analyzed. The analysis showed that while extract concentration was increasing, H₂ evolution and % I.E increased. There was also a corresponding reduction in corrosion rate as extract concentration increased. The decrease in corrosion rate was observed to follow in order of increasing extract concentration. Phytochemical-metal interaction mechanism was best described by Freundlich isotherm, with the 4 and 12 minutes curve becoming the best and worst contact time for the extract phytochemicals to suitably adsorb to metal surface for all concentrations used. An expression suitable for calculating corrosion rate values once concentration is known was also deduced. Furthermore, the microstructural studies revealed that increasing the extract concentration resulted in significant reduction in the dominant coarsening of the oxide of iron phase, while the pearlite and ferrite phases became finely dispersed.

Keywords: Mild steel deterioration, green inhibitors, hydrogen evolution, mild steel, regression analysis, adsorption isotherm, corrosion rate.

1. Introduction

Damage of mechanical components through corrosion is still a major concern in most industry today. These damages result in direct and indirect costs which translate to productivity losses, interruptions, breakdowns, environmental pollution and in some cases legal action. Developed countries of the world have conducted several studies on corrosion related cost issues and the outcome suggests that improved steps need to be taken to eliminate or reduce these costs. Corrosion mitigation techniques in the form of organic and metallic coatings, cathodic protection, plastics and polymers, corrosion resistant alloys and corrosion inhibitors have frequently been used to achieve these reductions (Ajayi et al, 2011a and Ajayi et al, 2011b). However, the use of inorganic inhibitors in industrial operations of acid pickling, descaling, oil well acidizing and cleaning of steel and ferrous alloys which reduce effective size of mechanical components has become an issue of concern because quite a number of synthetic inhibitors are harmful and toxic to the environment. The toxic effects not only affect living organisms but also poison planet earth. Most harmful effects are reversible and do not cause permanent damage but complete recovery may require time (Yin, 2004 and

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Omotosho et al, 2010). On the other hand some can cause irreparable permanent damage. For these reasons efforts have been made by several researchers to replace these toxic inhibitors with environmental friendly inhibitors.

Green inhibitors are known to be cheaply available, easily applicable and disposable without contaminating the environment. Thus, research efforts and studies (Ajayi et al, 2011a; Ajayi et al, 2011b; Oguzie, 2008; Okafor et al, 2005; Valek and Martinez, 2007; Singh et al, 2010; Raja and Sethuraman, 2008; Singh et al, 2010, Obot and Egbedi, 2009; Buchweishajia and Mhinzi, 2008; El-Etre, 2003; Saratha et al, 2009 ; Oguzie, 2009 and De-Souza and Spinelli, 2009) geared towards green inhibitors have become increasingly relevant because of the need to preserve the environment. Of the several studies considered none seemed to have specifically investigated the effect of 2M sulphuric acid on mild steel in the presence of extracts of *Bambusa glauscescens* (BG) at a temperature of 333K. The focus of this work is therefore to ascertain the degradation behaviour of mild steel at a temperature of 333K and correlate it with extract concentration, metal-phytochemical extract adsorption interaction mechanism and photomicrograph examination of the mild steel coupon. Also, inhibitor efficiency was determined by the method adopted elsewhere (Ajayi et al, 2011a, Okafor et al, 2010).

2. Materials and Method

Specimens of mild steel measuring 4 x 1.5 cm were employed for the determination of corrosion inhibition of BG extract in 2 M sulphuric acid (H₂SO₄). Samples were prepared by degreasing and cleaning in ethanol and acetone respectively. They were then allowed to dry. Chemical composition of the samples was analyzed using Optical Emission Spectrometer (OES) and the result is shown in Table 1. Extracts of BG were made from its fresh leaves by drying and grinding into powder. Powder was then weighed, from which 10g of the powder was then put into a 200 cm³ flat bottom flask containing 100ml of 2 M H₂SO₄ solution. The solution obtained was refluxed for 2 hours and left over night. The refluxed solution was then filtered. The concentrations of the extract used for the study were 60,100 and 140 cm³/l and they were prepared by serial dilution. The set up of the experiment was similar to the technique adopted elsewhere (Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c). Each sample was put into the mylius cell containing 50 cm³ of test solution and the experiments were conducted at a temperature of 333K. The volume of Hydrogen (H₂) gas evolved per 4 minutes interval was recorded for 60 minutes in a calibrated tube by downward displacement of water and the graph of volume of hydrogen evolved against time interval was plotted and is represented in Figure 1. Determination of the inhibition efficiency was carried out using Equation 1 (Okafor et al, 2010; Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c):

$$IE(\%) = \frac{(V-V_1)}{V} \times 100 \quad (1)$$

Where V and V₁, are the volumes of H₂ evolved from solutions without inhibitor (i.e. control experiment) and with inhibitor respectively.

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Because hydrogen is released when metals react with acids, it is correct to correlate reaction rate of corrosion deterioration of the mild steel in H_2SO_4 using BG extract with H_2 evolution.

The technique adopted elsewhere (Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c) was used in this investigation to also model the corrosion rate which translates to the hydrogen evolution rate. The hydrogen evolution rate has a direct relationship with the rate at which the weight of the metal is lost. Therefore, according to Ajayi et al (2011c) corrosion rate modeling from the path of H_2 gas evolution rate is implicitly a means of modeling weight loss rate when the connection between the weight loss and H_2 gas evolved is established. Thus, Equation 2 was obtained (Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c):

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

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

Table 1: Composition of mild steel sample employed for the investigation

Element	%Content	Element	%Content	Element	%Content
Bi	0.0024	Cu	0.035	Sn	< 0.001
Ca	0.017	Co	0.0057	Zn	0.022
Ce	< 0.0040	Ti	0.001	As	0.0059
Zr	0.0016	Nb	0.0046	C	0.131
La	0.0018	V	0.0075	Si	0.028
Fe	99.4	W	< 0.010	Mn	0.397
Ni	0.025	Pb	< 0.0020	P	0.0066
Mo	< 0.0020	Mg	0.002	S	0.018
Al	0.125	B	0.0009	Cr	0.0075

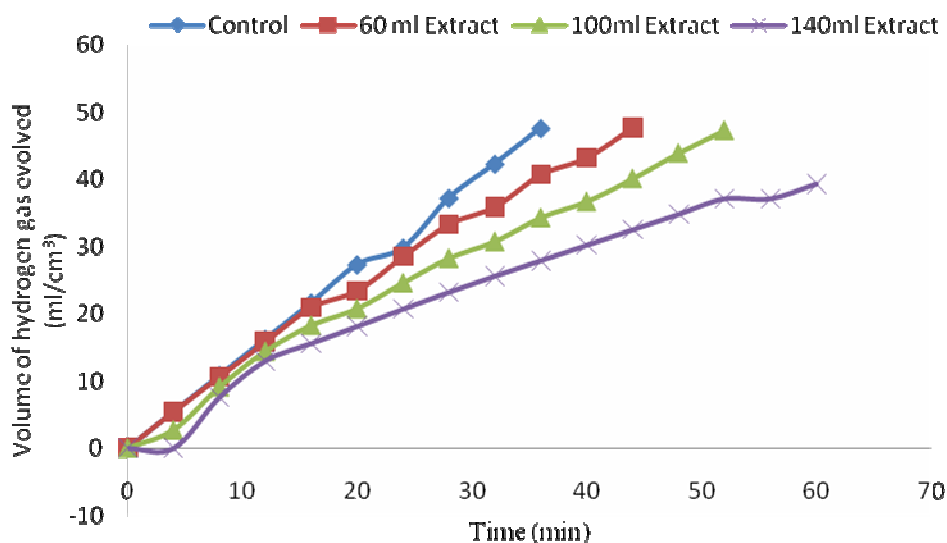


Figure 1: Relationship of volume of H₂ (cm³) evolved with time (minutes) of mild steel coupons for different concentrations of BG extract in 2 M H₂SO₄ at a temperature of 333K.

Equation 2 was obtained by a correlation analysis of volume evolved and the time of evolution. Thus, a polynomial regression analysis of the volume of H₂ gas evolved against time was carried out. This led to Equation 3 (Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c):

$$V = c + bt + at^2 \quad (3)$$

$$R = \frac{dV}{dt} = b + 2at \quad (4)$$

For measurements relating to 100 cm³ extract concentration, the corrosion rate model is represented as Equation 6. By adapting Equation 3 and 4 according to the approach used in literature (Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c), it was possible to obtain Equation 5 and 6.

$$V = 0.0352 + 1.1313t - 0.0047t^2 \quad (5)$$

$$\frac{dV}{dt} = 1.1313 - 0.0094t \quad (6)$$

3. Results and discussion

It was revealed from Figure 1 that the corrosion rate of mild steel in the control as indicated by the amount of H₂ evolved was the highest. Corrosion rate of samples immersed in the inhibited solutions reduced drastically when compared to the control. It was also noted that the amount of H₂ evolved also reduced as the concentration of the extract increased. This infers that the BG extract in the solution had a retarding effect on the corrosion of mild steel in H₂SO₄. Thus, the degree of inhibition can be said to be governed by the amount of BG

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extract present. A similar situation was also noted in Figure 2 in which the percentage inhibition efficiency (% I.E) was presented. However, the % I.E. readings for samples in the 60, 100 and 140cm³ extract were distant at the beginning of experiment but became very close as the experiment progressed. Precisely at 8 minutes the % I.E. readings for all the extract concentration became closely related while the readings for 60 and 100 cm³ extract were observed to be even closer at 20 minutes. Therefore, from Figure 2 it was discovered that % I.E. increased as extract concentration increased.

From Figure 2 it was observed that peak % I.E. values was obtained for 100 and 140 cm³ extract concentration at the beginning of the experiment. The retarding effects then declined afterwards for the 100 and 140 cm³ extract concentrations. The 60 cm³ extract had its lowest % I.E. at the beginning of the experiment, but afterwards % I.E. readings began to increase gradually until the experiment ended. However, the % I.E. readings in increasing order can be indicated as; 60 cm³ extract < 100 cm³ extract < 140 cm³ extract. An observation of Figure 3 revealed that the corrosion rate at a temperature of 333K, decreases in the presence of BG extract in contrast to the control. The extract concentration of 60 cm³ showed the least effect of diminishing the corrosion rate of mild steel sample. The corrosion rate values for the sample immersed in the 60 cm³ extract began at 0.02207cm³/s and gradually reduced until the end of the experiment at 0.0103 cm³/s. Sample immersed in the 100 cm³ extract which followed the 60 cm³ extract in terms of reducing corrosion rate began at 0.0189cm³/s and ended with 0.0095cm³/s.

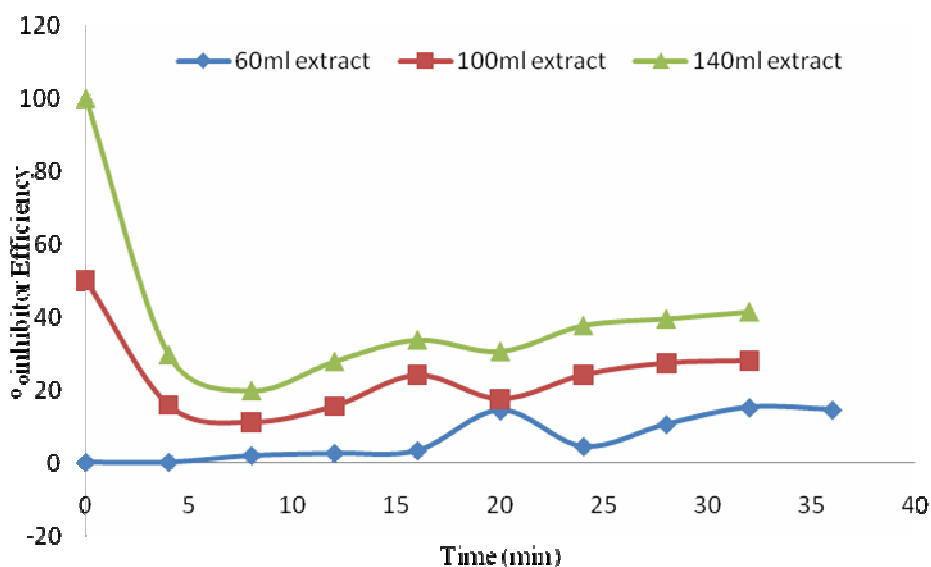


Figure 2: Percentage inhibition efficiency of varying concentrations of BG extracts with time (minutes) on mild steel coupons in 2 M H₂SO₄ solution at a temperature of 333k.

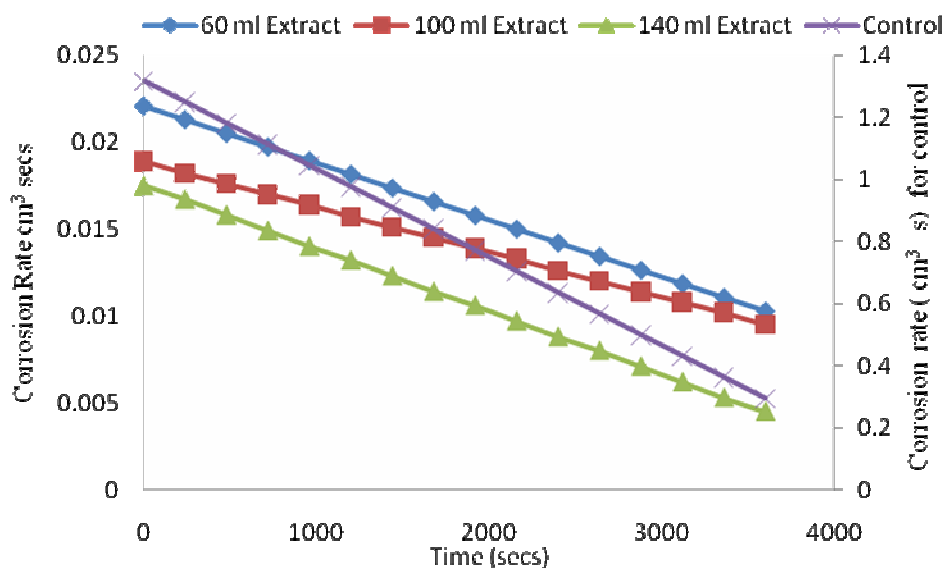


Figure 3: Corrosion rate of varying concentrations of BG extract with time (minutes) on mild steel coupon in 2M H₂SO₄ solution at a temperature of 333k.

The 140 cm³ extract produced the best result, as corrosion rate values started at 0.0175cm³/s and ended at 0.0045 cm³/s. Average corrosion rate values for the 60,100 and 140 cm³ extract were 0.00162, 0.0142 and 0.0011cm³/s respectively. Comparing these values to that of the control (0.08048cm³/s) shows that the inhibitor was effective. The results also indicate that corrosion rate reduced as extract concentration increased. The reduction in corrosion rate from Figure 3 was therefore observed to follow the order; 60 cm³ extract < 100 cm³ extract < 140 cm³ extract. A regression analysis of the values of corrosion rate R against that of corrosion was conducted in order to obtain the reaction constant and the specific reaction constant of the reaction through the investigation of the relationship between corrosion rate and varying inhibitor concentration. This technique was in accordance with the approach adopted elsewhere (Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c). By adopting this method discovered that corrosion rates can be related with acid concentration with Equation 7 (Mathur and Vasudevan,1982; Noor and Al- Moubaraki, 2008; Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c):

$$\text{LogR} = \text{Logk} + \text{BLogC} \quad (7)$$

where R = corrosion rate,

k = reaction constant,

B = specific reaction constant and

C = concentration.

However, a switch to same unit (mol min⁻¹) is necessary to determine reaction constant.

Thus, this was conducted by assuming that hydrogen evolution took place at 1.01325 x 10⁻⁵ Pa.

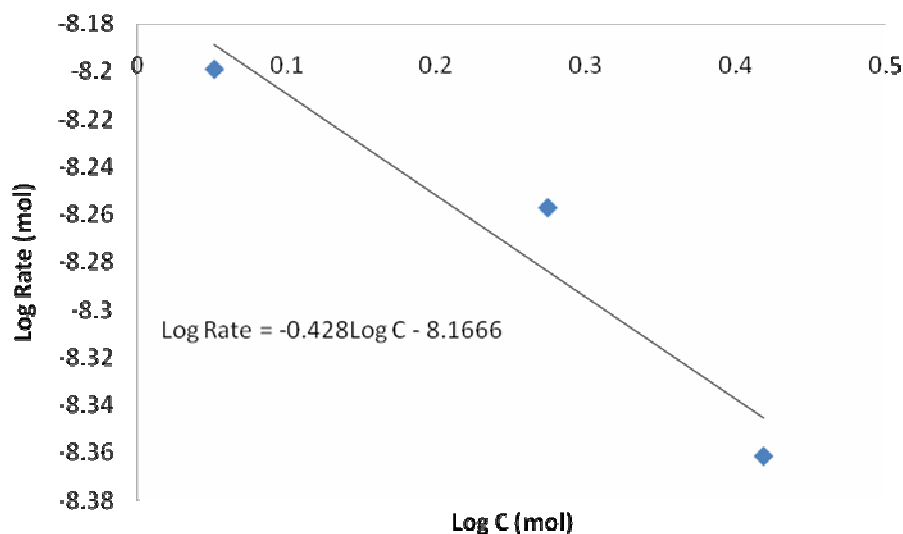


Figure 4: Plot of Log of corrosion rate against log of the acid extract concentration

The relationship between Log R and Log C shown in Figure 4 is for the mild steel coupon sample. A good correlation coefficient of 0.96 was obtained for the straight line in the graph. From the linear expression obtained from the graph, the k and B values were calculated to be $6.814 \times 10^{-9} \text{ mol min}^{-1}$ and -0.428 respectively. The negative value of B obtained in this study infers a decreasing slope and is different from other studies (Mathur and Vasudevan, 1982 and Noor and Al- Moubaraki, 2008) where no inhibitors were used. This is significant because it described the inhibitive action of the BG extract on mild steel corrosion. Thus, by further simplifying Equation 7 we obtain Equation 8:

$$R = KC^B \quad (8)$$

And for this specific reaction Equation 8 is written as:

$$R = 6.814 \times 10^{-9} C^{-0.428} \quad (9)$$

Equation 9 completely explains the observations in Figure 1 and 3, which shows a distinct difference between the uninhibited and inhibited solution.

3.1 Adsorption studies

The mechanism of interaction at the interface between the metal and the extract can best be resolved by using several adsorption isotherms. Such isotherms include Temkin, Freundlich, Langmuir, and Boris-Swinkels. This was done by analyzing the degree of surface coverage, θ , for all the extract concentrations utilized based on volume of H_2 gas evolved measurements. The surface coverage values were then fitted to the various adsorption isotherms and the Freundlich isotherm was discovered to have the best fit to the θ values. Studies (Febrianto et al, 2009; Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c) have shown the Freundlich isotherm as a practical calculation with the capability of fitting all data obtained experimentally and the most extensively utilized mechanism for depicting adsorption of

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organic compounds. The equation is thus expressed as (Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c):

$$\theta = D_c C^{1/H} \quad (10)$$

and in linearized logarithmic form as:

$$\log\theta = \log D_c + \frac{1}{H} \log C \quad (11)$$

Where

θ = surface coverage

D_c = characteristic constant related to the adsorption capacity

C = concentration

H = characteristic constant related to adsorption intensity or degree of favourability of adsorption.

After plotting a graph of $\log\theta$ against $\log C$ it was discovered that the graph has gradient and intercept values of $1/H$ and $\log A_c$ as depicted in Figure 5. The magnitude of $\log A_c$ is equivalent to $\log \theta$ when C is 1. The equation of the straight line obtained was evaluated to estimate the value of H and D_c as 1.526 and 4.99 respectively. In addition a good correlation coefficient of 0.9987 was also obtained. The value of H obtained therefore shows a favourable adsorption, clearly shown by a Freundlich constant that has a magnitude between 1 and 10 (Febrianto et al, 2009; Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c). The corrosion inhibition of the BG extract is thus credited to the adsorption of molecules of the phyto-constituents on the metal surface by a physical adsorption where a union is formed between the molecules and mild steel surface. Adsorption process is ordinarily known to occur simply when free energy change value is negative. Therefore by employing the Gibbs equation the adsorption free energy can be calculated (Yang et al, 2009; Ajayi et al, 2011a; Ajayi et al, 2011b and Ajayi et al, 2011c):

$$\Delta G = -RT \int_0^a H \frac{da}{a} \quad (12)$$

Equation 12 can be expressed as:

$$\Delta G = -HRT \quad (13)$$

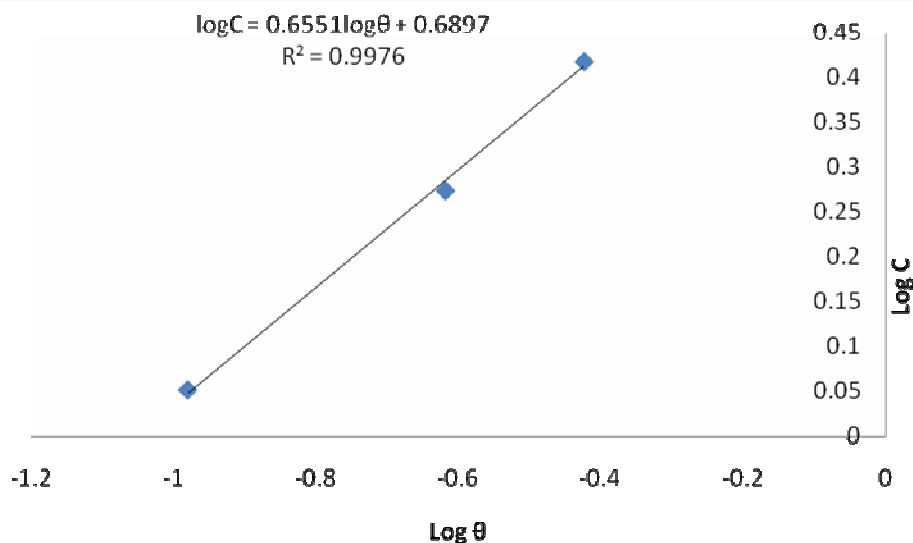


Figure 5: Plot of logarithm of surface coverage with logarithm of concentration of acid extract showing agreement with Freundlich isotherm.

The free energy change, parameter of the Freundlich equation, gas constant and temperature are represented as ΔG , H , R and T . The negative value of ΔG ($-4.225 \text{ KJ mol}^{-1}$) depicts the adsorption of BG extract on the mild steel surface as a spontaneous process.

The graph of the degree of surface coverage, θ , against the extract concentrations was plotted for different time intervals. The presentation in Figure 6 was done to examine if there were any influence of times of exposure to the relationship between θ and C . At extract concentration of 60 cm^3 , the 4 and 32 minutes curve had the lowest and highest surface coverage values while at 100 cm^3 the 4 and 12 minutes curve had the highest and lowest θ value respectively. At a concentration of 140 cm^3 the highest overall θ value of 1 was attained by the 4 minutes curve with the lowest θ value of 0.1975.

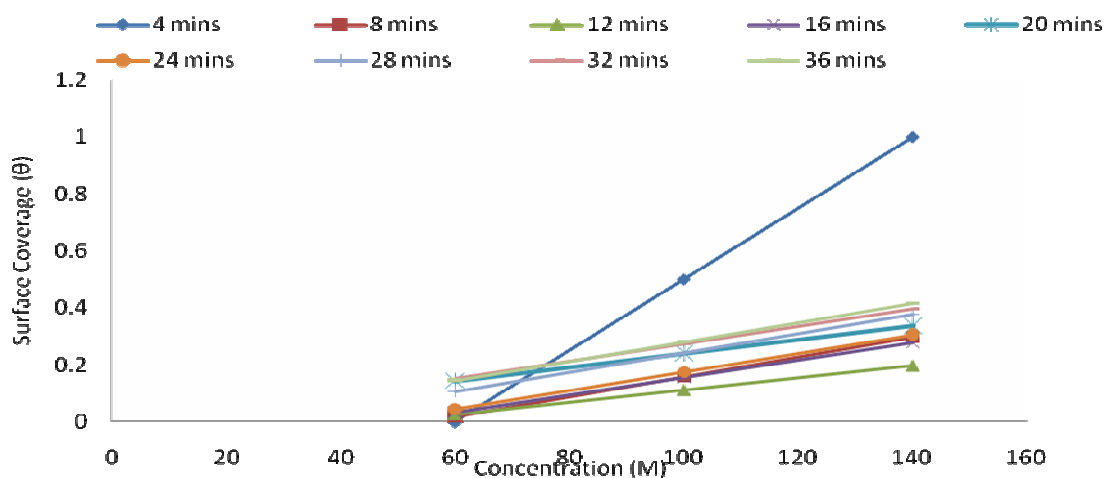
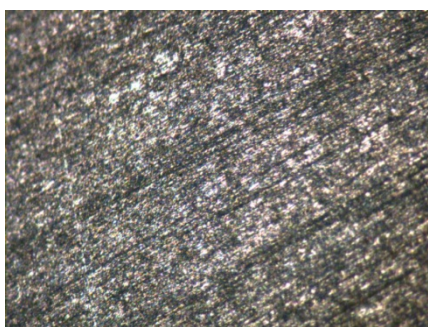


Figure 6: Relationship of surface coverage (θ) with extract concentration (M) at different time intervals.

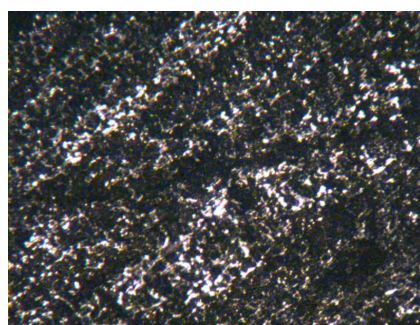
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The 4 and 12 minutes curve displayed the overall best and worst surface coverage values respectively across all concentrations utilized. This infers that the 4 minutes time frame was the best exposure time for the phytochemicals to adsorb to the steel surface and inhibit corrosion effectively. However, the other time frames showed closely related θ values throughout the experiment.

The effects of the H_2SO_4 action on the metal in the presence of BG extract were examined using the optical microscope. The studies were performed on these samples in order to investigate the state of the steel surface and its grain structure. The investigations were however, carried out on three metal samples which consist of the control sample, sample immersed in least inhibitor concentration and that of the highest extract concentration. The three samples were chosen because whatever occurs on the surfaces will not be far from what is expected to have happened to others. Figure 7a shows the micrograph of the metal before immersion. Observation of the micrograph showed that the microstructure of mild steel had three phases present, these included; the first, second and third phases respectively. These are the ferrite or white phase, the pearlite or grey phase and the black or oxide of iron phase. In Figure 7a it is observed that the ferrite and pearlite phases are evenly dispersed whereas the pearlite phase is observed to be more dominant. Figure 7b shows the mild steel in the H_2SO_4 without any inhibitor (control). The observation of the microstructure showed a dominant coarsening of the third phase, which is a vivid sign of corrosion. Figure 7c shows a gradual decrease in the third phase with a corresponding increase in the ferrite and pearlite phase while Figure 7d shows a fine dispersion in the ferrite and pearlite phase with a substantial decrease in the third phase evident around the edges of the mild steel. This suggests that the third phase is more predominant in the sample immersed in the $60cm^3$ extract than that in the $140cm^3$ extract. Therefore, a link existed between corrosion retardation and BG extracts concentration which clearly suggests that increases in extract concentration were completely relational to reduction in corrosion rate. Increasing the extract concentration in practical terms translated to a reduction in metal deterioration and an extension in the useful life of the metal.



(a)



(b)

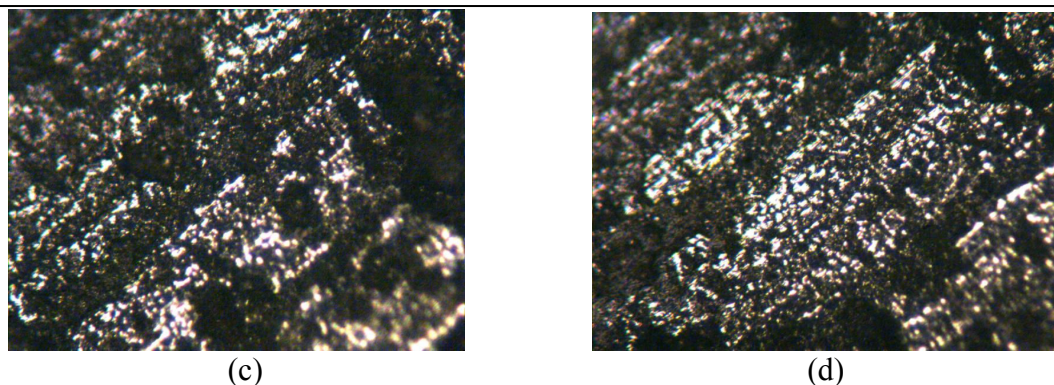


Figure 7: Micrographs for mild steel sample surface (a) before the experiment (b) after immersion in 2M H₂SO₄ for 60 min (c) after immersion in 60 cm³ of BG extract for 60 min (d) after immersion in 140 cm³ of BG extract for 60 min. All the immersions were done at a temperature of 333K.

4. Conclusion

The investigation examined the various indices that characterized the behavior of the mild steel sample when it was immersed in the sulphuric acid solution using the gasometric technique at a temperature of 333K. Investigations revealed that while H₂ evolution and corrosion rate reduced extract concentration and % I.E increased. The extract phytochemical-metal surface interaction mechanism was best explained by the Freundlich isotherm. The 4 and 12 minutes curve was also revealed as the best and worst interaction time for the extract phyto-constituents across all concentrations employed to adequately adsorb to steel surface. Photomicrograph studies indicated that increasing the extract concentration resulted in significant reduction in the coarsening of the oxide of iron phase, with the pearlite and ferrite phases becoming finely dispersed. The retardant was more effective as concentration increased; this is of course an indication of a decrease in the rate of damage of the steel.

5. References

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