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Inhibitive Potential of Prosopis Africana on Corrosion of Low Carbon Steel in 1M Hydrochloric Acid Medium

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ABSTRACT

Recently, there is quest for the use of inexpensive, non-toxic, non-biodegradable, readily available and environmentally acceptable corrosion inhibitor. Studies have shown that these properties could be achieved through the use of plants as inhibitor. Inhibiting effect of *Prosopis Africana* (Iron Tree) seed extracts were assessed on a sample of low carbon steel in 1M HCl with varying proportion of the seed extract using gravimetric, Tafel polarization and gasometrical measurement techniques. The results show that weight loss/corrosion rate decreases with increase in the extracts' concentrations. Good inhibiting efficiency of *Prosopis Africana* extracts with optimum inhibiting efficiency of 97.7% at 1.0 g/l after 120 hours exposure in gravimetric measurement was attained. TAFEL polarization results revealed that the *Prosopis Africana* extract shows that the corrosion current density decreases with the increase in the concentration of the extract. The extract is found suitable as green inhibitor for corrosion of low carbon steel in the studied medium.

Keywords: Mild steel, *Prosopis Africana*, Weight loss, Inhibiting efficiency, Tafel polarization

1 INTRODUCTION

Low carbon steel is a popular metal that has a wide range of applications due to its availability and excellent mechanical properties [1,2]. Low carbon steel is much prone to corrosion when exposed to acidic or basic environment [3]. Corrosion occurs in air, water, soil and in every environment and often affects most materials. Other than materials loss, corrosion interferes with human safety, disrupts industrial operations and possesses danger to the environment [4]. Corrosion is a natural process that cannot be totally prevented, but it can be minimized or controlled by proper selection of materials, design, coating and application of inhibitors [5]. For closed systems or sometimes even under flowing conditions, it is effective and convenient to employ corrosion inhibitors. Corrosion inhibitors are used to protect metals by adsorption from corrosion attack. Some of these inhibitors are synthetic in nature and therefore not eco-friendly because of the toxic product released to the environment after usage [6]. It has been established that most of the efficient inhibitors are organic compounds that contain in their structures mostly nitrogen, sulphur or oxygen atoms that create barrier to corrosion attack on the metal surface [7].

However, the toxicity of organic compounds combined with their cost and non-availability, have made natural products an excellent alternative possible corrosion inhibitors [8 - 10]. Among the organic compounds, the majority of recent studies have been focused on plant extracts. The main constituents of the plant extracts have been reported to be a wide variety of organic compounds, including polyphenols, terpenes, carboxylic acids and alkaloids [11]. Some of these plants include *Jathropha Curcas*, Banana peel, Curcuma extract, Watermelon waste and Moringa Oleifera among others [1, 12-17].

Prosopis Africana is a perennial leguminous tree of the *Mimosoidae* subfamily and is mostly grown in the savannah regions of West Africa. In Nigeria, this tree can be found between lat. 7° N and 10° N [18]. Because this species is not cultivated, it is often referred to as wild, endangered but edible [19]; as a lost crop [20], or as a lesser crop. Fruit setting, maturation, and dropping takes place in *Prosopis Africana* in Nigeria between November and March. The trees produce many legume pods during the months of January and March. In Nigeria, the seeds are very important

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to the Idoma, Ighala, Ebira, and Tiv people (mostly of the Guinea savanna) who use them to prepare a spice known as locust beans that is rich in protein and fatty acids [21]. When dried, the roots are used by the Yorubas to make chewing sticks called "ayan", which prevent dental root and gum decay in adults. The hardwood from *Prosopis Africana* offers good material for furniture, hoes, bows, pestles, mortars, and charcoal. The tree contributes to nutrient recycling and prevention of soil erosion [22].

Prosopis Africana seed is abundantly available in Nigeria, most especially in the University of Ilorin, where it is properly utilized and regarded as waste materials. Utilization of the plant seed as corrosion inhibitor will curtail the possible environmental hazard that may arise as a result of its presence in the environment. The use of the plant as an inhibitor may be channelled to create wealth through job creation. However, the aim of this study is to investigate the corrosion potential of the *Prosopis Africana* seed extract (PASE) and establish the possible efficiency it could offer as low carbon steel corrosion inhibitor in hydrochloric acid (HCl) medium.

2 METHODOLOGY

2.1 Sample Preparation

The elemental composition of the low carbon steel as shown in Table 1 was determined at Midwal Engineering Service Limited, Lagos using Spectromaxx LMF06 Spectrometer Machine (Serial Number: 15007384).The sample was prepared in accordance with ASTM G1-03 & G4 guidelines [23, 24]. The metal sheet specimen was cut using guillotine machine into proper dimension of 2.2cm ×1.7cm× 0.15cm and drilled at the top to make hole (1mm) on the specimen for easy hanging, removal from tested solution and identification. Emery papers of different grades (220, 320, 400,800, 1200) was use to grind the specimens (low carbon steel) to attain smooth and uniform surfaces. The specimens were then degreased with acetone, washed with distilled water and hot dried, and then immediately kept in desiccators.

2.2 Preparation of the Plant Extract (Inhibitor)

Prosopis Africana seeds were removed from the pod and sun dried for 7 days, when it was fully dried. The dried seeds were crushed to remove the shell. The seed were then grinded into powder form and then soaked in ethanol for 48 hours, sieved using a sieve and filtered to remove the shaft. It was then left for more than 48 hours in a salt bath to allow ethanol to get evaporated. The samples of the *Prosopis Africana* (ponds, seeds and shell) are presented in Figure 1.

2.3 Preparation of Test Solution

The Hydrochloric Acid, HCl (sp.gr.1.18) was prepared to obtain 1.0 molarity of HCl using the equation 1. The preparation was carried out in the Corrosion Laboratory at the Department of Materials and Metallurgical Engineering, University of Ilorin, Ilorin, Nigeria.

$$\text{Molarity} = \frac{\text{Molecular Weight of HCl}}{\text{Specific gravity of HCl} \times \text{Percentage of Purity}} \times \text{M} \quad (1)$$

Table 1: Chemical composition of low carbon steel sample [1]

Elements	C	Si	Mn	Cr	Al	Cu	Ni	P	S	Co	Fe
Weight (%)	0.033	0.034	0.220	0.038	0.011	0.021	0.007	0.015	0.008	0.004	99.50

(a) (b) (c)

Figure 1: *Prosopis Africana*, (a) Pond (b) Shell after removing seeds from the pond (c) Seeds

2.4 Phytochemical Analysis of the Plant Extract

The phytochemical analysis of *Prosopis Africana* seed and pod were carried out at Department of Chemistry Laboratory, University of Ilorin, Ilorin, Nigeria to determine the presence of inhibitive functional groups.

2.5 Gravimetric Analysis

A 140 ml of 1M Hydrochloric Acid was taken into a container from the prepared acidic solution. The corrosion test using weight loss measurements was carried out in accordance with the guidelines in ASTM standards [25]. The pre-weighed specimens from the desiccators were completely immersed in the 140 ml of 1M HCl solution with or without varying concentrations of *Prosopis Africana* extract covered from the atmosphere (Figure 2). The concentrations of the inhibitor are presented in Table 2. The test specimens were exposed to the medium between 24 hours and 2160 hours. This method was carried out in line with ASTM G1 standards [23]. The experimental set-up is shown in Figure 2. Sequel to removal of the specimens from the medium of exposure, chemical method of cleaning was employed to remove the corrosion products formed on the surface and edges of the specimens using a prepared solution in accordance with the ASTM G1 guidelines [23]. The solution containing 500ml of HCl, 3.5g hexamethylene tetramine and reagent water which makes 1000 ml solution. The coupons after corrosion test are shown in Figure 3.

The specimen was hot dried and then reweighed using Electronic weighing balance (HX 302 with 0.01g accuracy) to determine the difference in weight (weight loss).The same procedure was repeated for the specimens exposed to the medium for the periods between 24 hours and 2160 hours. From the weight loss, the corrosion and the percentage inhibiting efficiency (IE %) of the plant extract were calculated using equations 2 and 3 respectively.

$$\text{Corrosion rate (mpy)} = \frac{\mathbf{kW}}{\boldsymbol{DAT}} \quad [23] \quad \text{or} \quad \frac{\boldsymbol{\Delta W}}{\boldsymbol{AT}} \ (gcm^{-2}h^{-1}) \qquad [1,4,26] \tag{2}$$

Where W is the mass loss (g), A is surface area of the specimen (steel coupon) in cm^2, K is constant = 3.45×10^6 mils per year (mpy) [23], D is density of the steel (g/cm^3) and T is the time of exposure (hours).

$$\text{I.E (\%)} = \frac{\mathbf{CR_{Blank} - CR_{Inh}}}{\mathbf{CR_{Blank}}} \times \mathbf{100} \ [26] \tag{3}$$

Where CR_{Blank} is corrosion rate in the absence of inhibitor, CR_{inh} is the corrosion rate in the presence of inhibitor.

Table 2: Varying concentration of *Prosopis Africana* seed extract

g/l	0.0	0.2	0.4	0.6	0.8	1.0

Figure 2: Gravimetric measurements set-up

Figure 3: Cleaned specimen after corrosion test

2.6 Tafel Polarization Techniques

The steel sample was cut into 1.0 cm × 1.0 cm dimension. Each of the specimens was held together with aluminium foil by connecting it with a flexible cable and placed on a cup mould. In another cup mould, hardener was added to a polyester resin and thoroughly mixed. To the mixture, an accelerator was added and mixed together to form a solution. The prepared solution was then poured in the initial mould where the specimens were placed, left for a period between 15-20 minutes to solidify. For surface exposure into the aggressive environments, the coupon was further polished. The electrochemical measurements were performed using a single compartment electrochemical cell design for varieties of flat samples for electrochemical test at room temperature. The cell consists of a three electrode system, saturated calomel electrode as the reference electrode, working electrode (low carbon steel) and graphite rod as the auxiliary/counter electrode (versaSTAT) as electrochemical work station. For the data analysis, Electrochem software was used. Electrochemical measurement setup analysis and the specimens connectivity is shown in Figure 4 and 5 respectively.

2.7 Gasometric Measurement

Gasometric measurement was carried out using the gasometric set-up described by Awe *et al.* [7]. The assembly was setup as in Figure 6 to measure the volume of evolved hydrogen gas from corroded mild steel in the test solution. This set-up consists of two necked conical flask (reaction flask) which contained test solutions and mild steel specimens, separating funnel, calibrated tube with retort stand, taps, and water bath. The measurement was carried out with different test solutions that contained in the reaction flask. And the reaction flask was connected to an inverted calibrated tube through a delivery tube. Initial volume of water in the calibrated tube was recorded and mild steel specimens were carefully dropped in the test solution and quickly closed. The volume of evolved hydrogen gas from in reaction flask was monitored by the downward displacement of water. This displacement was monitored at 10 minutes interval, progressively for 100 minutes and the difference in the level of water was recorded as the volume of hydrogen gas. From obtained values of evolution hydrogen gas, the inhibition efficiency (IE %) was calculated using equation 4 [7].

$$\text{IE \%} = 1 - \frac{\text{CRinh}}{\text{CRabs}} \tag{4}$$

Where CRabs is the hydrogen evolution rate in absence of inhibitors and CRinh is the hydrogen evolution rate in the presence of different concentration of inhibitors.

Figure 4: Electrochemical measurement analysis setup

Figure 5: Specimens held in foil and connected with cable for electrochemical measurement

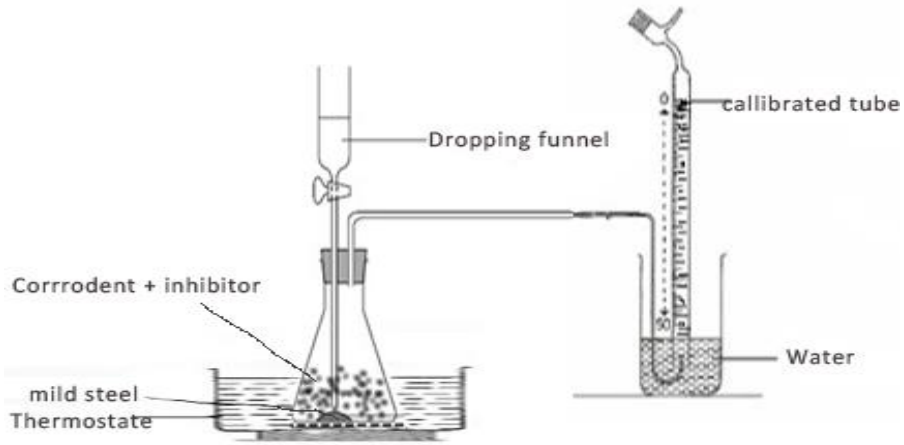


Figure 6: Schematic diagram of the set-up used for gasometric measurements [5]

Table 3: Qualitative and quantitative analyses of *Prosopis Africana* seed extract

Parameters (mg/100g)	*Prosopis Africana*
Flavonoids	0.80 (+)
Tannin	4.10 (+)
Saponin	8.50 (++)
Steroids	2.20 (+)
Phenol	3.05 (+)
Alkaloids	11.70 (++)

Table 4: Phytochemical analysis of *Prosopis Africana* pod extract

Parameter (mg/100g)	*Prosopis Africana*
Saponin	108.70
Alkanoids	101.60
Tannin	83.80
Phenol	9.90
Flavonoid	2.10
Cardiac cilycosides	1.06
Steroids	7.80

3 RESULTS AND DISCUSSION

3.1 Phytochemical Analysis

Phytochemical compounds and herbal-based extracts have garnered increasing interest in the field of sustainable material protecting products [11]. The role of protection is to form a barrier of one or several molecular layers against corrosive media attack and this depends widely on the part of the plant and its geographical location [7]. From the results obtained in Table 3 and 4, high presence of alkaloid (11.70 mg/l), alkaloid (10.60 mg/l) and saponin (108.70 mg/l) in *Prosopis Africana* seed extract is revealed. The presence of these compounds and their structures contribute to the inhibition efficiency due to the many functional groups that was found present in the compounds [7, 27]. The identified compounds as summarized in Tables 3 and 4 were based on standard methods in literature [7, 27]. The presence of organic compounds such as tannins, alkaloids, steroids, amino acids, flavonoids, among others in some natural plant products has been found to be responsible for their inhibitory action [1,4, 28-33]. It was previously reported that bitter leaves extract contained many phytochemical compounds like tanins, saponins, alkaloids, and flavanoids and this make it more effective for inhibition of corrosion [27]. Studies have also shown that formation of protective film on the metal surface due to adsorption of molecules of phytochemicals present in the plants on the surface of the metal is responsible for the inhibitive effect of some plants solution extract [33-35].

3.2 Gravimetric Measurement (Mass loss)

The variation of mass loss recorded in low carbon steel at different concentrations of *Prosopis Africana* seed extract in 1M HCl at different time of immersion are shown in Figure 7. Generally, the mass loss of low carbon steel in HCl increased with increase in time of exposure, but decreases as the concentration of inhibitor increases. This implies that

the rate of corrosion of the low carbon steel in HCl within the period of exposure would decrease as the concentration of the extract increased. Though, the mass loss of the specimens with the presence of *Prosopis Africana* seed extract in 1M HCl reduced drastically (Figure 8). In line with the views of Lai *et al.* and Shuaib-Babata *et al.* [1, 36], the reduction in mass loss might be as a result of adsorption of the inhibitor on the metal surface. For instance, the mass loss in the medium after 168 hours of exposure with and without 1.0g/l of inhibitor was 0.02 g and 0.54 g respectively. This is an indication of effective potential inhibiting of *Prosopis Africana* at all concentrations. It is also in agreement with Odusote *et al.* [4]s' view that the mass loss of steel decreased as the concentration of the inhibitor increased. With 0.2g/l of inhibitor in the medium, mass loss of 0.13g after 168 hours of exposure was recorded, while at the same time 0.4g/l recorded 0.1g, 0.6g/l recorded 0.06g, 0.8g/l recorded 0.04g and 1.0g/l recorded 0.02g as shown in Figure 8. These indicate that the weight loss increases with concentration of the inhibitor in line with the findings of Loto *et al.* [37]. The results revealed formation of significant inhibitory properties of passive film on the surfaces and edges of the specimens, which could be traced to synergistic concentrations of phytochemical constituents of *Prosopis Africana* [1]. It is interested to note the specimens could not survive reaction in the media beyond 168 hours without the presence of *Prosopis Africana* (inhibitor) due to the aggressiveness of the environment, while other specimens in the medium with *Prosopis Africana* survived beyond 2160 hours. This is an indication of good inhibiting efficiency of *Prosopis Africana*. The specimens in the medium without *Prosopis Africana* after 168 hours of exposure is shown in Figure 8a & b, while Figures 8c show the nature of the specimen without inhibitor in the medium after corrosion test.

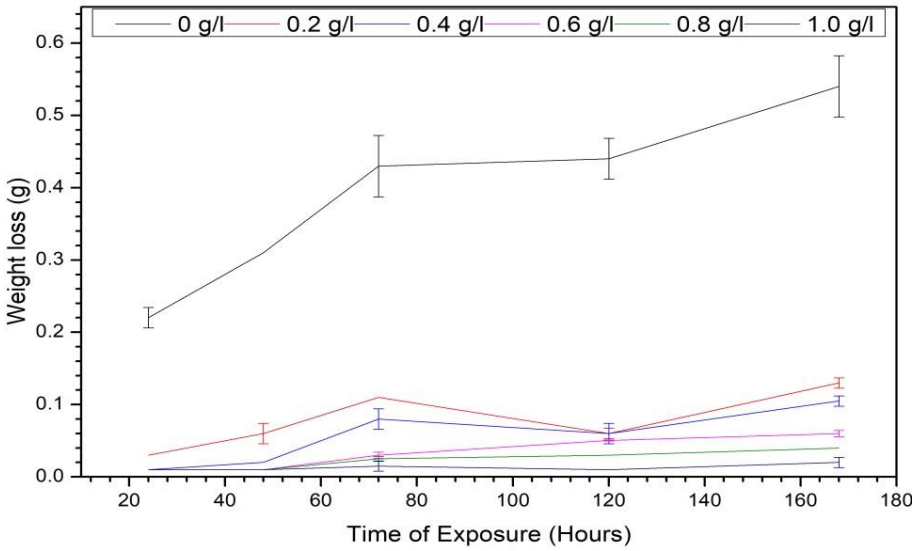
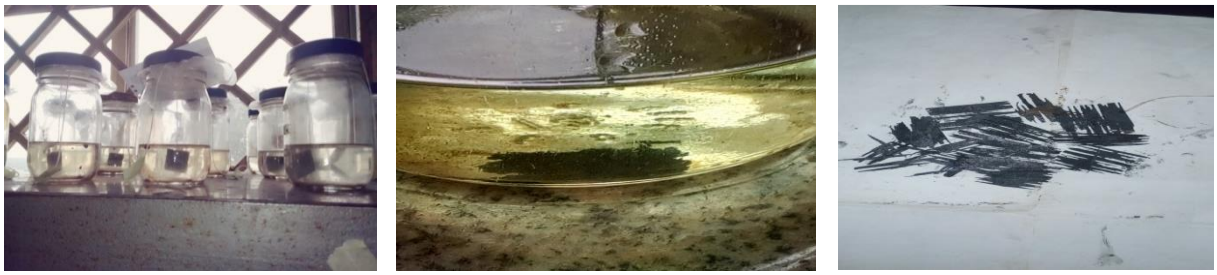


Figure 7: Variation of mass loss with time for the corrosion of low carbon steel in 1M HCl with and without inhibitor.



(a) (b) (c)

Figure 8: Effectiveness of *Prosopis Africana* as an inhibitor in HCl medium. (a) Nature of the low carbon steel immediately (b) The low carbon steel totally destructed after 168 hours after immersion without inhibitor of exposure in the medium without inhibitor due to aggressiveness of the evironment (c) Nature of the low carbon steel without inhibitor in HCl after corrosion tests.

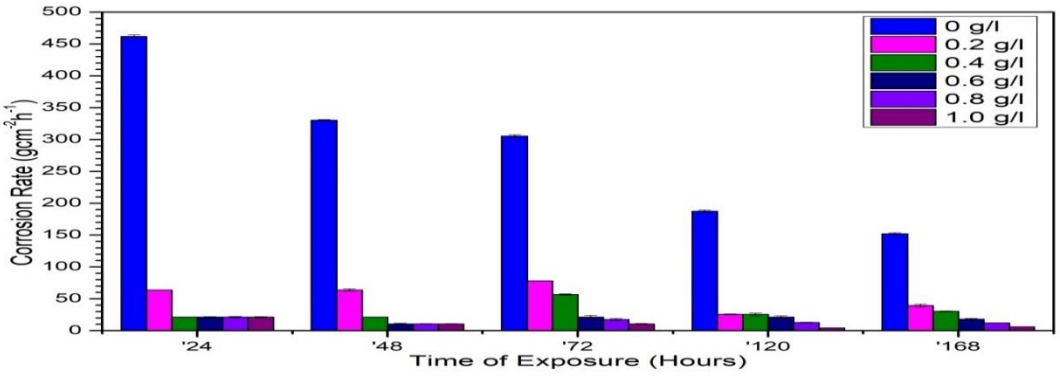


Figure 9: the variation of corrosion rate in *Prosopis Africana* seed extracts

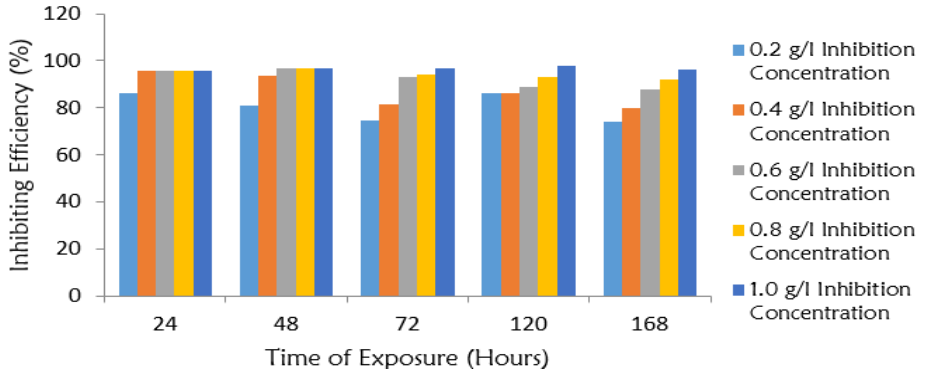


Figure 10: The percentage inhibition efficiency of *Prosopis Africana* seed extract at different concentrations

The variation of corrosion rates within the time of exposure are presented in Figure 9. It is shown in the results that the corrosion rate of low carbon steel in HCl with plant's extract in the medium decreases with increase in time of exposure, while that of specimens in medium without plant extract decreases with increase in time of exposure. After 24 hours of exposure, the specimens in the medium with 0.0 g/l recorded corrosion rate of 461.72 mpy, while 152.18 mpy was recorded after 168 hours at the same medium. More so in HCl with plants extract, the specimen's corrosion rate decreases with increase in concentration of the extract. Considering the corrosion rate results after 24 hours of exposure, the specimen exposed in 1M HCl with the 0.2 g/l concentration of *Prosopis Africana* seed extract recorded 63.92 mpy, while specimen in the medium with 1.0 g/l recorded 10.65 mpy for the same period. Again, in medium with 0.2 g/l and 1.0 g/l inhibitor after 72 hours of exposure, the recorded corrosion rate was 78.12 mpy and 10.65 mpy respectively. In medium with 0.2 g/l and 1.0 g/l inhibitor after 168 hours of exposure, corrosion rate was also 39.57 mpy and 6.09 mpy respectively. As a result of this, it is assumed that there is increase in adsorption of the constituents of the extract on the surface of the low carbon steel as the concentration of *Prosopis Africana* seed extract increases, which led to reduction in corrosion in the medium [1, 38, 39].

Figure 10 shows the variation of *Prosopis Africana* seed extract inhibiting efficiency within the period of immersion in 1M HCl solution. The results indicate that the *Prosopis Africana* seed extract inhibiting efficiency increases with its increasing concentration, but it was not consistent with the time of exposure. It is as a result of formation of a protective film which results to transition of metal interface from an active dissolution state to a passive state. The optimum inhibiting efficiency of 97.7% was recorded with concentration of 1.0 g/l after 120 hours of exposure. From the results (Figure 10), the steadiness inhibitive behaviour of *Prosopis Africana* seed extract was revealed through the calculated percentage inhibition efficiency. The IE% ranged between 74.0 and 97.7% within 24 to 168 hours of exposure. The percentage inhibition efficiency of *Prosopis Africana* seed extract in HCl medium beyond 168 hours of exposure could not be calculated since the specimens in HCl without *Prosopis Africana* seed extract could not withstand the aggressiveness of the medium as shown in Figure 8. Though, the specimens in HCl medium with *Prosopis Africana* seed extract were intact (Figures 3 & 4) even after 2160 hours of exposure. This proved the effectiveness of *Prosopis Africana* seed extract as an inhibitor of low carbon steel in HCl medium beyond 2160 hours. The effectiveness may be attributed to transition of metal interface from an active dissolution state to a passive state which resulted to the formation of protective film on the surfaces and edges of the specimens.

3.3 Tafel Polarization Technique

Corrosion rate is being measured with Tafel extrapolation. TAFEL behaviour (polarization curves) exhibits linear behaviour in the corrosion potential (Ecorr) against log corrosion current density (Icorr) plots for an electrochemical reaction under activation control. Starting from a cathodic potential of -200mV/s to an anodic potential of +250mV/s at a cam rate of 0.166, polarization measurements were carried out. The linear Tafel segment of the cathodic curves and the calculated anodic Tafel lines were extrapolated to corrosion potential to obtain the corrosion current densities Icorr [40]. Figure 11 shows that as the concentration of *Prosopis Africana* seed extract increased, the corrosion potential shifts toward a more noble direction. Furthermore, in the presence of *Prosopis Africana* seed extract, the corrosion current decreases markedly and the magnitude of such an effect increases with increasing *Prosopis Africana* seed extract concentration. The inhibitive action of the extract to corrosion of low carbon steel in the 1M HCl is reflected in the results (Figure 11). The inhibition efficiency increases with increasing extract concentration. The effect of the extract on both anodic and cathodic reactions is also reflected in the results (Figure 11). *Prosopis Africana* seed extract therefore acts as a mixed inhibitor.

3.4 Gasometric Analysis

Figure 12 shows the variation in volume of hydrogen gas evolved in 1M HCl at different concentration of the extract. It was revealed that the volume of hydrogen gas evolved increases with time until a level was reached when it remains constant. This constant level was reached quicker in the solutions containing inhibitors than that without inhibitor. This may be as a result of quicker formation of passive layer on the surface of the metal in the solution containing inhibitors. Thus, the higher the inhibitor concentration in a solution, the quicker the passivity level would be reached.

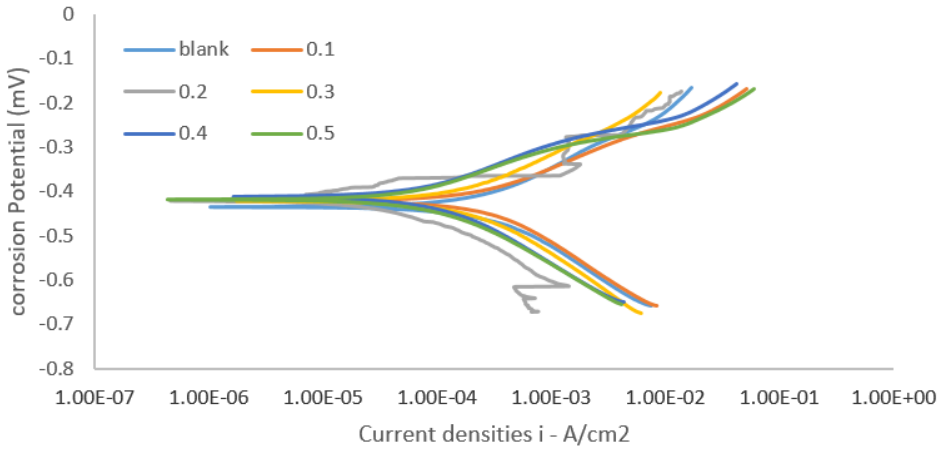


Figure 11: Corrosion potential against corrosion current density (*Prosopis Africana* seed extract)

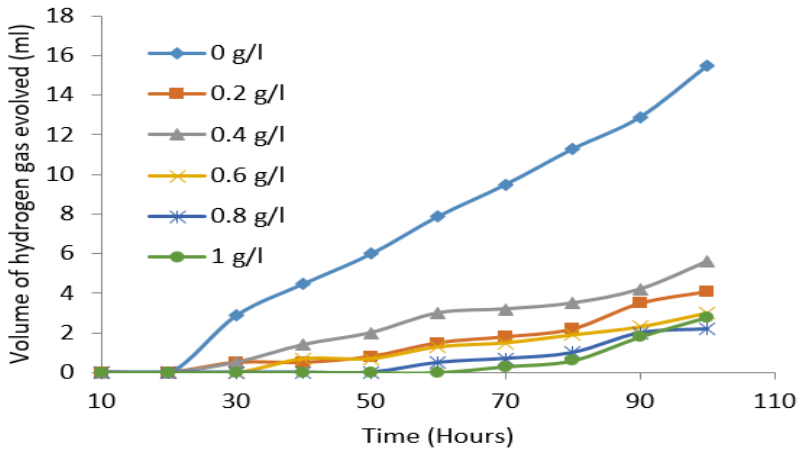


Figure 12: Rate of evolution of hydrogen gas in 1M HCl at different concentration of the extract.

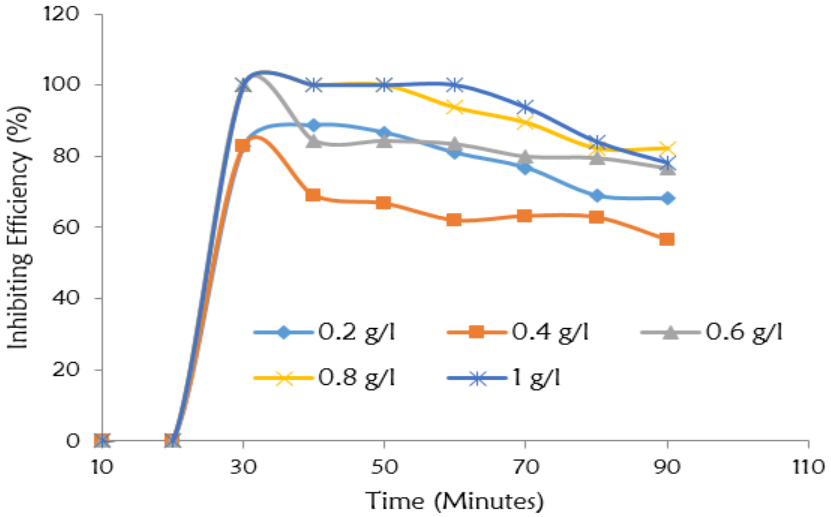


Figure 13: Variation of inhibition efficiency with time of exposure.

Figure 13 shows the results obtained by the variation of percentage inhibition efficiency with the time of exposure. The results show same initial 20 minutes of zero inhibition at all inhibitor concentration. This might be the period for formation of passive layer in the solution. The corrosion rate was high at the initial point with no inhibitive action from the extract irrespective of the concentration until adsorption of the inhibitor on the metal surface began. It is revealed in the results (Figure 13) that total inhibition of corrosion of the low carbon steel in 1M HCl solution was initially experienced (100% at 30 minutes) and later decreased to a level where it started increasing as the time of exposure increases. The initial behaviour might be as a result of slow adherence of the acid to the metal surface which in turn slowed down the attack of the metal by the acid. The point of initiation of the decrease in inhibition level may be as a result of the absorption of the extract on the steel surface before corrosion retardation occurred. The increase in the inhibition with the concentration of the extract is probably due to the retardation of the corrosion rate by the inhibitor as suggested by Odusote *et al.* [4] for the inhibition of mild steel corrosion by *Moringa Oleifera* leave extract.

CONCLUSIONS

According to the assessment of inhibition of corrosion of the low carbon steel in an acidic solution (HCl) by the use of *Prosopis Africana* seed extract using gravimetric, electrochemical measurement (Tafel polarization techniques) and gasometric analysis. The following conclusions are drawn from this study:

1. The inhibition of the corrosion of the low carbon steel in the acidic solution by *Prosopis Africana* seed extracts is due to the phytochemical constituents of the plant extract
2. The corrosion rate of the low carbon steel in the acidic solution (HCl) solution decreases with increase in the concentration of *Prosopis Africana* seed extract
3. The optimum percentage inhibition efficiency of *Prosopis Africana* (97.7%) was attained with 1.0 g/l concentration of the extract after 120 hours of exposure
4. In gravimetric technique, the *Prosopis Africana* seed extract's inhibition efficiency depends on its concentration. The efficiency decreases with increase in the extract's concentration in the medium.
5. Tafel polarization technique result showed that the extract acted as a missed type inhibitor via a simple absorption of the phytochemicals present in the extract on the low carbon steel surface in HCl.
6. Gasometric technique revealed quicker formation of passive layer on the surface of the metal in the solution containing *Prosopis Africana* seed extract (inhibitors). Thus, the higher the inhibitor concentration in a solution, the quicker the passivity level would be reached.
7. The *Prosopis Africana* seed extract was found to be an effective inhibitor of corrosion of low carbon steel in HCl solution.

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