

CORROSION INHIBITION EFFECT OF *BALANITE AEGYPTIACA* LEAVES EXTRACT ON MILD STEEL IN 5M H₂SO₄ A GRAVIMETRIC AND THERMOMETRIC STUDY

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ABSTRACT

The corrosion inhibition effect of *Balanites aegyptiaca* leaves extract on mild steel in 5 M sulphuric acid solution has been investigated using gravimetric study and thermometric technique at 40 oC, 50 oC, and 60 oC. The main aimed of this research is to investigate the potential of *Balanite aegyptiaca* leaves powder for corrosion inhibition. The *Balanite aegyptiaca* fresh leaves was obtained from itas Gadau local government Bauchi and the leaves sample was washed and shade dried for two weeks under room temperature. The concentration of the inhibition 0.01, 0.02, 0.03 and 0.04 g/L was prepared and used at temperature of 313 K, 323 K, and 333 K respectively. The inhibition efficiency was found to be highest at 313 K and 0.04 g/l concentration of the extract with IE of 18.48 % and lowest at 333 K and 0.01 g/l concentration with IE of 16.42 %. Therefore the Inhibition efficiency increases with extract concentration but decreases with temperature. The thermodynamic parameters and results from the FTIR (shift from 1208 cm⁻¹ to 1205 cm⁻¹ and 1618 cm⁻¹ to 1611 cm⁻¹ and appearance of new 1016 cm⁻¹, 1676 cm⁻¹, 1834 cm⁻¹, 3905 cm⁻¹ bands," etc) revealed the adsorption of the inhibitor molecules on mild steel surface as comprehensive adsorption involving physical and chemical adsorption. Results show that *Balanites aegyptiaca* which is biodegradable, environmentally, and are obtained from a renewable resource with minimal health and safety concerns has the potential to a cost-effective alternative to synthetic corrosion inhibitors

Keywords: *Balanite aegyptiaca*, Corrosion rate, Fourier transform infrared spectrophotometer (FTIR), Inhibition Effect, Weight loss, Metal density

INTRODUCTION

Corrosion is a natural process that converts a refined metal to a more chemically stable form, such as its oxide, hydroxide or sulphide. Corrosion inhibitors are substances that when added in small concentrations to corrosive media decrease or prevent the reaction of the metal with the media (El- Etre *et al.*, 2015). The *Balanites aegyptiaca* plant also known as Adduwa in Hausa native is a species of tree, classified as a member of either the Zygophyllaceae or the Balanitaceae. This tree is native to much of Africa and parts of the Middle East. There are many common names for this plant. In English, the fruit has been called desert date, soap berry tree or bush, Thron tree, Egyptian myrobalan, Egyptian balsam or Zachum oil tree (Tula, 2020).

Balanites aegyptiaca is a multibranched, spiny shrub or tree up to 10 m high. Crown rounded, dense (but still seen through) with long stoutbranchlets. Trunk and bark grey, deeply fissured longitudinally (Orwa *et al* (2019).

The plant grows in tropical and desert areas it can be found in many kinds of habitats, tolerating a wide variety of soil types from sand to heavy clay and climatic moisture. (Salami *et al.*, 2024)

Balanite aegyptiaca is perennial plant used in food preparations mainly in africa and developing nations. It has variety of uses and most part of the plant is utilized including leaves thorns, back of root and fruit. The fruit is applied in the treatment liver aliments and as a purgative.(Kamaluddeen *et al.*, 2020).

Moringa Oleifera is similar to *Balanite aegyptiaca* is widely used in traditional human and veterinary medicine throughout Africa for its many properties. Through out the world the different part of this plant are used to treat many diseases such as bacterial and fungal infections, haemorrhagic menstruation, goiter, bilharzias, malaria, colic, yellow

fever,haematuria, hydrocele, and abdominal pain. (Hardman *et al.*, 2020)

Organic compounds use as inhibitors, occasionally, they act as cathodic inhibitor or anodic inhibitor or together, as cathodic and anodic inhibitors, neverthelass, as a general rule, act through a process of surface adsorption, designated as a film-forming. Naturally, the occurrence of molecules exhibiting a strong affinity for metal surface compounds shows good inhibition efficiency and low environmental risk (Yaro *et al.*, 2023).

Corrosion inhibitors are applicable in many ways such as in the oil, fertilizer, metallurgical and other industries where contact between metals and aggressive mediums is imperative (Eddy *et al.*, 2019). In order to solve the arising problem associated with corrosion, several measures including anodic and cathodic protection, lubrication, painting and electroplating have been adopted. Moreover one of the quality options available for the protection of metals against corrosion is the use of inhibitors (Abiola *et al.*, 2017).

Mild steel or Carbon steel is a steel with carbon content up to 2.1% by weight. Mild steel remains the most widely use material since it belongs to the family of plain low-carbon steels. The use of materials exposes them to different environments. It has adequate ductility and moderately high strength and it is relatively cheap and readily available. These qualities have made it stand out during the other materials for selection as the major constructional materials in different fields of engineering (Rejendra *et al.*, 2020) The use of organic compounds containing oxygen, sulphur and nitrogen to reduce corrosion attack on mild steel has been studied in the past decades (Khaled *et al.*, 2022). The existing data showed that most organic inhibitors get absorbed on the metal surface by displacing water molecules and forming a compact barrier film. The availability of lone pairs and pi electrons in

the inhibitor molecule facilitate electron transfer from the inhibitor to the metal forming a co-ordinate covalent bond (Heckerman *et al.*, 2020). The strength of the chemisorption bond depends on the electron density of the donor atom of the functional group and also the Polaris ability of the group.

A plant extract is a substance or an active with desirable properties that is removed from the tissue of the plant, usually by treating it with solvent to be used for a particular purpose. Therefore, plant extracts are viewed as an incredibly rich source of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in the environment. Entering into 21st century along with people's increasing awareness of protecting the environment, a large number of researches about plant extracts as effective corrosion inhibitors of iron or steel in acidic media have been reported. Through these studies it agreed that the inhibition performance of plant extracts is normally described as the presence in their composition of complex organic species such as tannins, alkaloids, nitrogen bases, carbohydrates, amino acids and proteins as well as hydrolysis products. These organic compounds contain polar functions with N, S O atoms as well as conjugated double bonds or aromatic rings in their molecular structures, which are the major adsorption centers (Abdel-Gabe *et al.*, 2018). Millions of dollars are lost each year because of corrosion including loss of prevention, maintainance and loss. Much of this loss is due to the corrosion of iron and steel, although many other metals may corrode as well. The condition of the environment in the factory is also main cause of corrosion where humidity, temperature and pH of air becomes the major port. Many of factories owners did not realize the disaster of corrosion and how to prevent it correctly causing many accidents and bad quality results in the product (Abiola *et al.*, 2017)

This problem therefore, necessitated the indispensable use of corrosion inhibitors mitigate the attacks on metallic materials by the corrosive media. Unfortunately, the inhibitors mostly used are inorganic and synthetic in nature which have been proven to be very hazardous to human health causing cancerous diseases, exorbitant-cycle cost and are not environmentally friendly. Given this development, the present research study investigated an economically viable and scalable source of green corrosion inhibitor *Balanites aegyptiaca* leaves extract which is biodegradable and environmentally friendly using gravimetric method. The source plant *Balanites aegyptiaca* is abundantly distributed across Africa, South Asia, Middle East, e t c and also possesses potential health benefits but neverthelass unkempt.

MATERIALS AND METHODS

Materials

- i. Flat bottom flask (1L)
- ii. Beakers (250 cm³, 100 cm³)
- iii. Glass funnel
- iv. Filter paper
- v. Analytical weighing balance
- vi. Water bath
- vii. Reagent bottle (60 cm³)
- viii. Measuring cylinder (1L).
- ix. Mild steel coupons (20x20 x4 mm).

Reagents

- i. Acetone (200 cm³)
- ii. Distilled water
- iii. Butanol (1L)
- iv. Sulphuric acid (5 M).

Methods

Preparation of H₂SO₄ Solution (5M)

The acid (5M) was prepared using the following formulas:

$$C_1V_1 = C_2V_2 \quad (1)$$

$$\text{Molarity}(CI) = \frac{10 \times \text{density of the acid} \times \% \text{purity}}{\text{Molar mass of the acid}} \quad (2)$$

Density, % purity and molar mass were obtained from the acid's bottle label and 10 = constant

The concentration of the acid needed was calculated using the formula:

Where,

C₁= the concentration of the acid in the bottle

V₁= the volume of the acid to be measured from the bottle

C₂= the concentration of the acid needed (5M)

V₂= the volume of the acid needed

Finally, the volume of H₂SO₄ measured from the bottle (V₁) was dissolved in the volume of the acid needed (V₂) using distilled water.

Plant Collection

Balanites aegyptiaca fresh leaves were obtained from Gadau, Itas-gadau LGA, Bauchi State, Nigeria. The leaves samples were washed and shade dried for 2 weeks, under room temperature so as to enrich the activity principles in them by reducing their moisture content. The dried leaves was then ground and sieved into fine powder.

Extract Preparation

The powdered leaf was weighed 50g and percolated with 1L butanol for two days with shaking and opening at room temperature yielded 14% ww extract. The solution was filtered in a Beaker using filter paper and a glass funnel. The filtrate was concentrated using a rotary evaporator in the University instrumental laboratory. This was then allowed to further dry by covering it with perforated aluminum foil paper to avoid dust and other contaminations. The concentration of the inhibition solution 0.01, 0.02, 0.03 and 0.04 g/L was prepared and used at temperatures of 313 K, 323 K and 333 K respectively and stored at 4 °C in amber bottles.

Extraction and Determination of total saponins in balanite aegyptiaca extract by spectrophotometer

Take 25 g depatted sample in 500 ml conical flask and add 250 ml absolute methanol 99.9% in ratio of dry wheight of the sample to methanol as 1:10 . Flask was tightly sealed and kept in a shaker at 25 C and 120 rpm for 24 hour following by centrifuging the contents at 3500 rpm for 20 min. After centrifugation, methanol extract was filtered using whatman filter paper No.1. The result methanolic extract where evaporated to dryness in vacuum condition using a rotaevaporator. After evaporation dried plant extract dissolve in minimum amount of distilled water (10 ml), transferred in to a separating funnel and extracted with equal volume of n-butanol (3 times).

Metal Preparations

The experiment was carried out with a medium carbon steel (C-1345) sample of chemical

Composition (C=0. 48%, Mn=2. 53%, Si=0. 64%, Cr=0. 46%, Ti=0. 15%, Co=0. 36%, Cu

=0. 07%, Mo=0. 68% and Fe=95. 7%). The metal sheets were cut into coupons of

Dimension (20x20x4mm) was abraded with diverse grades of emery paper (120, 600

And 1200) and washed with detergent, degreased in ethanol and air dried before weighing.

Gravimetric Method

The pre-weighed coupons were exposed to diverse test solutions. The study was conducted with the diverse concentrations (0.1 g/L to 0.5 g/L) of the inhibitor (*Balanites Aegyptiaca* Leave extracts) at room temperature. (*Balanites aegyptiaca* Leave extracts) At room temperature. After the exposure period of between 4, 8, 12, 16, 20 hrs respectively. The coupons were retrieved, rinsed with plenty of water, dried and thereafter weighed. The weight loss was decided. The procedure was repeated and the average result was taken.

Weight Loss

A known weight of mild steel was immersed in 100 ml of the test solution in an open beaker. The beaker was then transferred into a water bath maintained at 313 K, 323 K and 333 K. After a time interval, each sample was withdrawn from the test solution, washed with distilled water, rinsed in ethanol then acetone, dried in air and reweighed. The differences in weight of the coupons were taken as the weight loss which was used to compute the inhibition efficiency as:

$$IE(\%) = \frac{W_0 - W_1}{W_0} \times 100 \quad (1)$$

Where W_0 and W_1 are the weight losses of mild steel in the absence and presence of inhibitor in H_2SO_4 solution at the same temperature.

The corrosion rate is given as:

$$CR \left(\frac{g}{cm^2 hr} \right) = \frac{\Delta m}{At} \quad (2)$$

Where; Δm = mass loss (g), A = Area (cm^2), t = time (hr)

Corrosion rate can also be given as:

$$CR \left(\frac{mm}{yr} \right) = 87.6 \times \frac{w}{DA t} \quad (3)$$

Where,

W = Weight loss in g, D = Metal density in g/cm^3 , A = Area of sample in cm^2 T = Time of exposure of the metal sample in hours and 87.6= constant.

The Degree of surface coverage is given as:

$$(\phi) = \frac{W_0 - W_1}{W_0} \quad (4)$$

Where; w_0 = weight loss of the metal, w_1 = initial weight of the metal.

were retrieved, rinsed with plenty of water, dried and thereafter weighed. The weight procedure was repeated and the average result taken loss was decided.

RESULTS AND DISCUSSION

This study was carried out to identify a suitable substitute for some of the currently used inorganic and synthetic organic corrosion inhibitors, which are damaging to both humans and the environment. Potassium, magnesium, lead, chromates, and inhibitors containing a mixture of deadly anions (nitrates, phosphates, and fluorides) are no longer suitable or appropriate. As a result, a great number of inhibitors are currently utilized to protect metal surfaces from corrosion. Instead, eco-friendly inhibitors should be used. Unfortunately, there is a scarcity of knowledge about corrosion inhibitors that are safe for both humans and the environment. As a result of this research, corrosion inhibitor formulations made from *Balanites aegyptiaca* plant leaf extracts have been produced for use in sulphuric acid solutions to decrease low-carbon steel corrosion.

Table 1: Weight Loss at Different Concentrations

Exposure Time	Weight Loss At Different Conc						Mean SD±
	Control 0.0g/L	0.1g/L	0.2g/L	0.3g/L	0.4g/L	0.5g/L	
4	0.1395	0.821	0.767	0.659	0.413	0.739	0.644±0.02
8	0.2069	0.1076	0.91	0.746	0.536	0.578	0.69±0.01
12	0.3843	0.1345	0.1346	0.1305	0.816	0.852	0.48±0.03
16	0.6372	0.1976	0.2104	0.1782	0.1278	0.1113	0.15±0.01
20	0.9386	0.2722	0.2347	0.1871	0.1747	0.135	0.18±0.01

Table 1. Illustrate the weight loss measurements for medium carbon steel as it degrades in 1M sulphuric acid in the absence of an inhibitor and the medium carbon steel resisted corrosion in the presence of inhibitor (*Balanites aegyptiaca* leaves extracts). Weight loss versus exposure time for medium carbon steel corrosion in 1.0M H_2SO_4 in the absence and presence of various amounts of *Balanites aegyptiaca* leaf extract was shown in Table 1.

The rate of weight loss in the controlled environment (1.0M H_2SO_4) was gradually increasing over time, as shown in the plots, and this was observed for 20 hours, but when the inhibitor leaf extracts were introduced, the rate of weight loss decreased significantly according to the different inhibitor concentrations. The adsorption of elements in the extract on the surface of the medium carbon steel test piece caused the weight loss to be reduced in the presence of the inhibitor Which is similar to Kamaluddeen findings in 2022.

Table 2: Corrosion Rate Values for Medium Carbon Steel in 1 M sulphuric Acid in the Absence and Presence of *Balanites Aegyptiaca* Leaves Extract

Time (t) of exposure (Hrs)	Corrosion rates for control experiment and different concentration of <i>Balanites aegyptiaca</i> leaf extraction in corrosive media					
4	0.9700	0.5712	0.5336	0.4585	0.2873	0.5141
8	0.7200	0.3743	0.3165	0.2595	0.1864	0.2011
12	0.8900	0.3119	0.3121	0.3026	0.1892	0.1976
16	1.1100	0.3437	0.3659	0.3099	0.2223	0.2223
20	1.3100	0.3787	0.3266	0.2603	0.2431	0.1878

Table 2 shows the corrosion rate values when there was no inhibition to increasing as exposure time increased, but when leaf extract was added there was a noteworthy drop in corrosion rate. The corrosion rate was calculated using eq. (6).

$$\Delta W = W_1 - W_2 \quad (5)$$

$$CR = \frac{K \Delta W}{A t p} \quad (6)$$

Where K = Rate constant equal 8.78×10^4

W_1 = Weight before immersion in g

W2 = Weight after immersion in g
 ΔW = Weight loss in g
 ρ = Density of material in g/cm³

T = Exposure time in hours
 A = Exposed area of coupon cm²

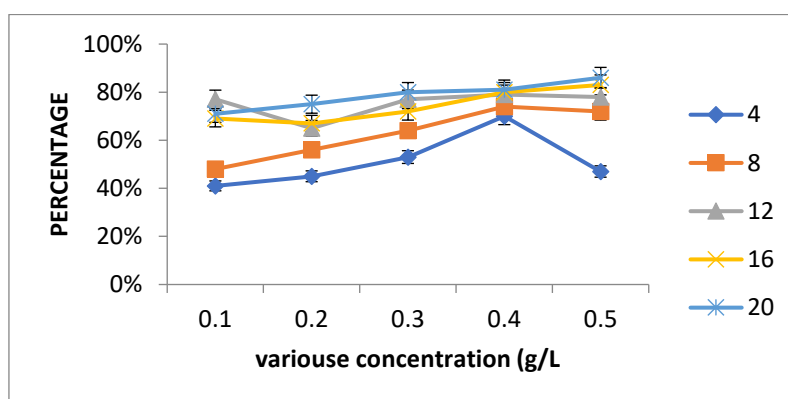


Figure 1: Showing efficiency increased with an increase in concentrations of inhibitor for gravimetric method of Analysis

Figure 1. Plot of Corrosion rate (mm/yr) (for control + various concentrations) versus exposure time (t). Figure 1 depicts the phenomenon where, in the absence of leaf extract (control experiment), a behavioural pattern of medium carbon steel corrosion rate with time was found. Passivation occurred within the first four hours of metal exposure, followed by metal resistance to passivation, as seen by the control curve. The creation of coatings on the test piece's surface caused the corrosion resistance observed between 4 and 8 hours; this does not mean that corrosion has halted; merely that it has been postponed. The films succumbed to further deterioration after 8 hours due to the environment's severe corrosive nature. The corrosion rate values in the control experiment continued to grow after 8 hours with no signs of slowing down. Medium

carbon steel degrades quickly, according to the findings which is similar to Lebrini's findings in 2015. The inhibited corrosion curves for all concentrations of 0.1 to 0.5 g/L showed a consistent pattern of the corrosion rate dropping steadily from 0 to 20 hrs, demonstrating that the leaf has formed a protective layer on the test piece's surface, blocking the active site and thus preventing corrosion. The corrosion rate continued to reduce at 0.1 g/L concentration until 16 hours, when a minor rise was noted, indicating that the extract concentration was depleting. From 8 to 20 hrs, the 0.5 g/L concentration of leaf extracts demonstrated the best corrosion rate values which is similar to Abdel- mecsoud's findings but deviate's from khadom's findings in 2019.

Table 3: Efficiencies (%) at Concentrations (g/L) for Gravimetric Method

Exposure time (Hrs)	Efficiencies at various concentrations (g/L)					Mean SD±
	0.1	0.2	0.3	0.4	0.5	
4	41%	45%	53%	70%	47%	56.66±0.03
8	48%	56%	64%	74%	72%	70±0.04
12	77%	65%	77%	79%	78%	78±0.04
16	69%	67%	72%	80%	83%	78.33±0.04
20	71%	75%	80%	81%	86%	82.33±0.04

Table 3 Elucidates how efficiency increased with an increase in concentrations of inhibitor. The efficiencies were calculated using the formula.

$$\eta_{\text{gravimetric}} = (1 - W_i/W_o) \times 100\% \quad (7)$$

Where W_i is the weight of the test piece when dipped into a corrosive environment with leaf extract and W_o is the weight of the test piece when dipped into corrosive media without leaf extract.

CONCLUSION

In the light of the evidence gathered during the course of investigating the inhibitive properties of *Balanites aegyptiaca* Leaf extract on the Corrosion of Medium Carbon Steel in Sulphuric Acid, the following conclusion have been drawn:

- In 1 M H_2SO_4 solutions, ethanolic extract of *Balanites aegyptiaca* Leaf is a suitable eco-friendly green inhibitor for medium carbon steel and can be used instead of hazardous chemicals.
- In the absence of *Balanites aegyptiaca* leaves extract in 1M of H_2SO_4 solution, gravimetric method reveals aggressive deterioration of the medium carbon steel

coupon immersed in the corrosive environment, the corrosion rate and weight loss increases drastically.

- showing that the active site of the test piece was bare to acid attack.
- The addition of *Balanites aegyptiaca* leaf extract to 1M H_2SO_4 solution reduces the corrosion rate of medium carbon steel in the acid as elucidated by the weight loss measurement. The inhibition efficiency increases with increase in concentration of the plant extracts, the maximum inhibition efficiency was 86%.
- Medium carbon steel was protected from corrosion when the plant extract was introduced by adsorption of compound of the extracts on the metal surface thereby creating a barrier for charge and mass transfer making the metal less susceptible to corrosion reaction.

RECOMMENDATIONS

In view of the experienced gained on this research, the following are recommended for further research.

- i. Further investigation is required to access the corrosion morphology so as to ascertain the active species in the adsorption layer.
- ii. The researcher recommends that the need to grow *Balanites aegyptiaca* trees at commercial be encouraged since they have very high efficiency as inhibitors. Being good inhibitors, they can reduce corrosion rate. Moreover, they are human friendly and ecologically acceptable.
- iii. Investigation of the inhibitive properties of *Balanites aegyptiaca* leaf extracts on medium carbon steel in sodium chloride (NaCl).
- iv. To investigate the effect of temperature on *Balanites aegyptiaca* leaf extracts as a corrosion inhibitor of medium carbon steel in corrosive acid.

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