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Corrosion Inhibition of Mild Steel in Hydrochloric Acid by Lawsonia inermis and Hibiscus sabdariffa Extracts: A Comparative Study

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Abstract
An assessment of comparative corrosion inhibition abilities of *Lawsonia inermis* (LI) and *Hibiscus sabdariffa* (HS) plant extracts for mild steel in hydrochloric acid media has been carried out. Weight-loss and potentiodynamic polarization methods were utilized to obtain the effectiveness of HS and LI to inhibit corrosion at 100mg/l to 3000 mg/l concentrations, for a period of 120 hours test on mild steel coupons Corrosion rates, inhibition efficiency, and surface coverage were determined, revealing significant inhibition by the plant extracts at optimal concentrations. Typical findings show *Lawsonia inermis* to have better inhibitor performance than *Hibiscus sabdariffa* as 95.7 mg/l and 95.0 mg/l for 0.1 M HCl and 97.8 mg/l and 96.5 mg/l for 1 M HCl respectively. Potentiodynamic polarization results revealed reduction of corrosion current density (I_{corr}) by 28.2 and 15.8 for HS and LI respectively in 0.1M HCl.

Keywords: AI-Assisted, Corrosion Inhibition, *Lawsonia Inermis*, *Hibiscus Sabdariffa*, Mild Steel

Introduction

The challenge of mild steel corrosion is witnessed across various industries like the petroleum, construction and chemical processing sectors (Schweitzer, 2010). The bid to look for sustainable and renewable plant alternatives to conventional synthetic corrosion retardants, is the latest trend in chemical research. The present work examined the potential of *Lawsonia inermis* (LI) and *Hibiscus sabdariffa* (HS) extracts to inhibit the corrosion of mild steel in hydrochloric acid medium. Weight-loss and potentiodynamic polarization (PDP) methods, were applied to assess inhibition efficiency, corrosion rate, and surface coverage over a 120-hour exposure period (Popoola et al., 2013). Awe et al. (2015) studied the ability of *Parinari polyandra* plant extract to prevent the deterioration of mild steel in sulphuric acid with gravimetric as well as polarization procedures. The result indicated a rise in inhibiton efficacy when the inhibitor concentration was raised. Chidiebere et al. (2016) investigated the capability of *Delonix regia* (DR) to retard the degradation of mild steel by applying gravimetric and electrochemical methods. From the results, DR leaf extract prevented the deterioration of the metal surrface and functioned as a multifaceted corrosion retardant. Awe et al. (2015) investigated the power of *Boscia senegalensis* to retard the degradation of mild steel corrosion by making use of gravimetric methods. From the result, it was shown that the increase in inhibition efficiency was concentration dependent, but was reduced when the temperature was enhanced. Akalezi et al. (2016) investigated the corrosion inhibition efficacy of *Pentaclethra macrophylla* (PM) plant in hydrochloric and tetraoxosulphate (VI) acidic media using gravimetric technique at 30-60^{0}C. The result indicated a proportionate increase of inhibition efficacy with PM concentration. Nwanonenyi et al. (2018) studied the suppression of carbon steel deterioration in 1M tetraoxosulphate (VI) acid with soy polymeric (SP) and polyvinylpyrrolidine (PVP) by making use of gasometric and electrochemical techiques.

Onyeachu et al. (2017) carried out an investigation on the deterioration of a Ni-Al combined film in 2M aqueous sodium chloride by electro-deposition procedure. The findings from the study showed that the electro-deposition of the aluminium on nickel substrate increased the porosity of the composite, decreased the corrosion potential and likewise increased the charge flow densities at the positive and negative electrode in the active region, when compared with the pure nickel coating. Nwanonenyi et al. (2016) investigated the inhibition power of hydroxy-propyl cellulose and potassium iodide ion on decomposition of mild steel in tetraoxosulphate (VI) acid medium using gravimetric technique. The inhibition efficiencies obtained were 76.43% and 89.73% for HPC and synergy with KI respectively. The work of Okore et al. (2024) provided excellent inhibition efficiencies for *Lawsonia inermis* (LI) extract and showed the power of LI to retard the deterioration of mild steel. Hajar et al. (2016) assessed the power of *Lawsonia inermis* to retard the degradation of mild steel in sea water. The findings from the research work showed that Lawsone, the active ingredient, reduced mild steel deterioration in brine within 60 days.

Tran et al. (2022) assessed the ability of *Sonneratia caseolaris* leaf extract (SCLE) to inhibit the degradation of steel in HCl solution using electrochemical procedures. The result presented SCLE as effective inhibitor of steel corrosion with inhibition efficiency reaching up to 98%. Seham et al. (2022) evaluated the inhibiting power of *Hibiscus sabdariffa* (roselle) on brass corrosion intrioxonitrate (V) acid, using potentiodynamic polarization technique, which presented roselle to have multi-faceted ability to retard degradation of brass by 94.89%.

Statement of Problem

Corrosion is a great challenge that has been posed across different construction, petroleum and chemical process companies. There is the need for green, sustainable retardants of metal deterioration, as opposed to toxic synthetic ones. There has been extensive research with regards to potential of green suppressants of metal degradation, but comparative analysis on the performance of natural dyes is scarce. Comparative analysis on corrosion inhibition activities of *Lawsonia inermis* versus *Hibiscus sabdariffa* has not been found in literature, which this work has attempted to address.

Specific Objectives

1. To extract the biochemically active constituents of *Lawsonia inermis (*LI) and *Hibiscus sabdariffa* (HS) as green retardants of mild steel deterioration in hydrochloric acid medium.
2. To evaluate the corrosion rate and inhibition efficacy of LI and HS extracts by employing gravimetric and potentiodynamic polarization methods.
3. To perform comparative analysis of the inhibition efficiency of LI and HS extracts and relate with the performance of plant-based corrosion inhibitors reported in the literature.

Materials and Methods

Reagents

High purity chemicals acquired from Sinopharm company and utilized concentrations for analysis. Different concentrations of HCl solutions were employed as corrodent.

Extraction process

The dye bearing plants *Lawsonia Inermis* (LI) and *Hibiscus Sabdariffa* (HS) were obtained from local market and validated at Agricultural science department of Alvan Ikoku Federal University of Education, Owerri, Imo State, Nigeria. Cold extraction process was used to extract the bioactive components of the plant materials at room temperature to avoid decomposition due to heating. The dried leaves of Lawsonia inermis were dissolved in ethanol in the ratio of 1:5 and mixed thoroughly to allow intimate interaction of sample and solvent. The container was sealed and allowed to stand at room temperature for seventy-two (72) hours. The solution was filtered with a clean cloth to obtain the filtrate, which was left open to evaporate the solvent, and dried to obtain a solid mass, which was stored in a clean container for further use. The same procedure was repeated for ground leaves of HS. The concentrations of the stock solutions were from 100 mg/l to 3000 mg/l. The corrodent used was 0.1M HCl solutions (Okore et al., 2024).

Metal specimen (mild steel)

The mild steel was cut into coupon of dimension 20 mm by 20 mm by 3 mm. The surface was polished with emery papers 120-grit to 2000-grit, to obtain a smooth surface that is free from oxides and rust. Thorough rinsing of the surface of the polished metal was carried out with distilled water so as to remove left-over polishing materials. Acetone was applied to remove water and organic matter. At this stage, drying of the specimen was done with dryer that blows hot air to it, which was then stored in moisture free desiccator, and kept for further use (Okore et al.,2024).

Method

Weight loss (gravimetric) method

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The metal specimen which had been prepared earlier was collected from the desiccator and used for the weight loss experiment. The initial weight of the metal coupon was measured with precision balance and recorded as W_1. The metal coupon was then immersed by hanging with threads, into 300 ml of the already prepared corrosive medium, in this case hydrochloric acid (0.1 M and 1M concentrations), at the temperature of 303 K. At intervals of twenty-four (24) hours of immersion in the corrosive medium, the metal coupon was removed, rinsed properly in distilled water, dried an oven for ten minutes, and re-weighed to obtain the final weight, which was recorded as W_2 (Okore et al., 2024). The determination of inhibition efficiency, I.E and corrosion rate, C.R., for mild steel in hydrochloric acid, *Lawsonia Inermis*, and *Hibiscus Sabdariffa* was carried out from equations below. (Murugavel & Gunavathy, 2012).

$$\Delta W = W_1 - W_2 \quad (1)$$

$$\%I = \left(1 - {}^{W_1}/_{W_2}\right)\ X100 \quad (2)$$

$$\theta = 1 - {}^{W_1}/_{W_2} \quad (3)$$

$$CR = {}^{\Delta W}/_{AT} \quad (4)$$

W_1= Initial weight of the metal coupon
W_2 =Final weight of the metal coupon.
θ = Degree of surface coverage
ΔW = Mass reduction
T = Exposure time in hours
A = Exposed area of metal coupon in cm^2

Electrochemical studies
The mild steel was cut into 1 cm^2 as the exposed area and polished with emery papers of size 120-2000 grit. The sample was cleansed with distilled water, acetone and air-dried with hot dryer. The coupon was placed in the electrode holder, exposing the 1 cm^2 to the electrolyte. The electrodes were connected to the potentiostat. The mild steel coupon was used as the working electrode. The reference electrode (the saturated calomel electrode), was then placed near the working electrode, while the counter electrode (in this case, graphite rod), was placed opposite the working electrode. The initial potential was set at $\pm$250 mV and scanned at the rate of 0.1-1 mV/s. Afterward, the Tafel graph, which is a plot of log I_{corr} against E_{corr}. Triplicate measurements were taken to ensure reproducibility (Okore et al.,2024)

Results

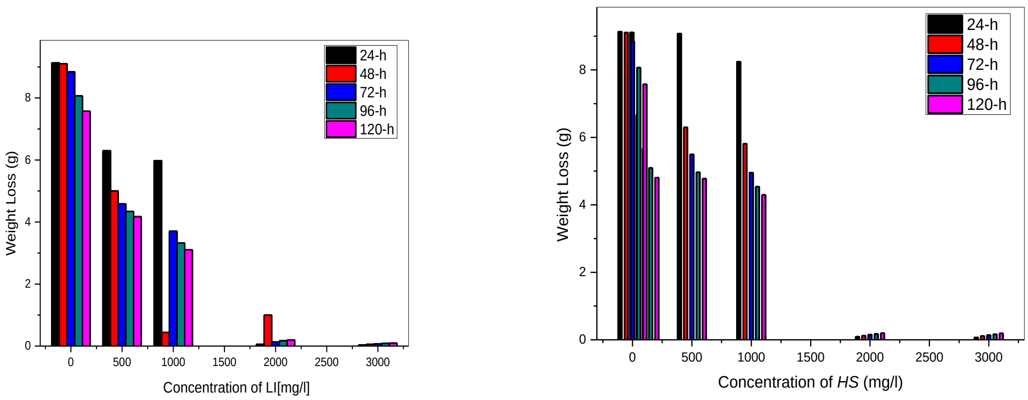

Figure 1: Mass loss vs concentration of (a) *Lawsonia inermis* and (b) *Hibiscus sabdariffa* for mild steel specimen in 0.1M HC at 303K

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Figure 2: Inhibition Efficiency versus concentration of (a) *Lawsonia inermis* and (b) *Hibiscus sabdariffa* for mild steel in hydrochloric acid at 303K

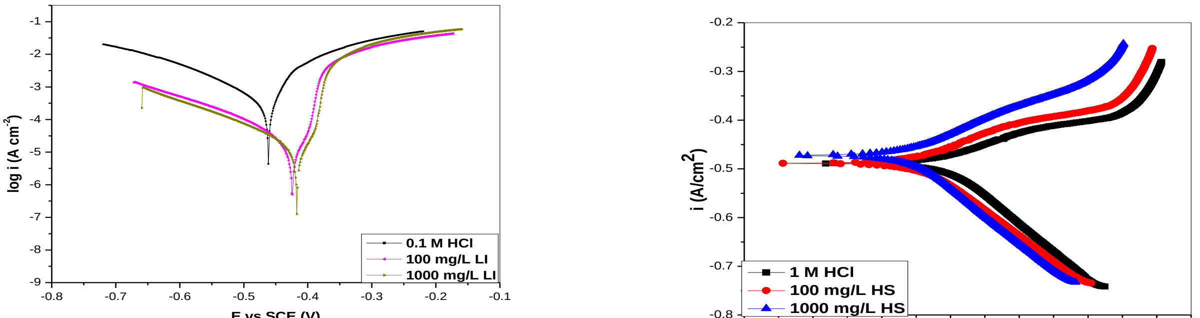

Figure. 3: Pdp graphs for mild steel corrosion with or without (a) *Lawsonia inermis* and (b) *Hibiscus sabdariffa* in 0.1M HCl

By making reference to Figure 1, the weight loss of *Lawsonia inermis* (LI) and *Hibiscus sabdariffa* (HS) with respect to concentration could be compared as follows. Figure 1(a) and (b) reflect reductions in weight loss with increase in concentrations, portraying good inhibition performance. However, Li produced greater reduction in weight loss than HS at same concentrations of the inhibitors, which presents LI as better inhibitor than HS within the experimental conditions of the study**.** Olusegun et al. (2011) in like manner noticed a closely-related pattern of behavior, in their study of the ability of Gossipium hisutum leaf extract to retard the dissolution of aluminium in HCl solution. The results showed a reduction in the rate of dissolution of aluminium with increase in concentration of the plant extract. Njoku et al. (2014) confirmed similar behavior with *Baphia nitidal*, which retarded the dissolution of mild steel in acidic environment. Figure 2 provides information on the change in inhibition efficiencies for LI and HS with respect to the increase in concentration, and reflect increment of the inhibition efficiencies of LI and HS with increase in concentration, which became more pronounced at higher concentrations. The inhibition efficiencies of LI and HS were recorded as 95.7 and 95.0 at 3000 mg/l, showing LI to have better inhibition performance at all the studied concentrations. The work of Ihebrodike et al. (2012) portrayed comparable pattern of action, because the increase in concentration of *Solanum melongena* produced proportional enlargement of the inhibitory efficacy. Figure 3 describes the potentiodynamic polarization behaviours of LI and HS for mild steel immersed in 0.1 M HCl acid. Graph 3 reflects the shifts in the cathodic and anodic arms, showing mixed-type inhibitory behaviour for both plants, which indicates that both plant extracts function by adsorbing on the cathodic and anodic sites. LI shifts the Ecorr more positively than HS, showing a more cathodic behaviour. LI produced lesser reduction in corrosion current density than HS as 15.8 and 28.2 respectively. Lower Icorr values signify better inhibition efficiency and better performance as corrosion inhibitor. The polarization studies of Nagiub et al. (2013) on ability of azo dyes to diminish deterioration of mild steel in acidic solution revealed a multi-faceted action of the azo dyes on the metal. *Lawsonia inermis* gave better inhibitor performance than *Hibiscus sabdariffa*.

Conclusion

Plant extracts of *Lawsonia inermis* and *Hibiscus sabdariffa* are powerful suppressors of deterioration of mild steel in hydrochloric acid. The inhibitor performance was enhanced at higher concentrations. The comparative study presented *Lawsonia inermis* as a better inhibitor than Hibiscus sabdariffa

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Recommendations

1. Characterization of the plant extracts using SEM, EDX and XPS should be carried out to provide better understanding of corrosion inhibition mechanism.
2. Stability studies should be carried out under different conditions of temperature and exposure to prolonged immersion time.
3. The potentials of the studied plants to suppress metal dissolution should be compared with those of synthetic inhibitors.

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