Nigerian Research Journal of Engineering and Environmental Sciences 8(2) 2023 pp. 510-516
p ISSN: 2635-3342; e ISSN: 2635-3350

Nigerian Research Journal of Engineering and Environmental Sciences
Journal homepage: www.rjees.com

Original Research Article

The Inhibiting Effect of *Entada Africana* Leaves Extract (EALE) on Corrosion of Mild Steel in 1 M HCL and 0.5 M H_2SO_4 Acidic Media

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http://doi.org/10.5281/zenodo.10442751

ARTICLE INFORMATION

Article history:
Received 26 Oct. 2023
Revised 07 Nov. 2023
Accepted 09 Nov. 2023
Available online 30 Dec. 2023

Keywords:
Corrosion
Inhibitors
Entada africana
Phytochemical
Acidic media

ABSTRACT

The use of inhibitors for the control of corrosion of metals and alloys which are in contact with aggressive environment is among the acceptable practices used to reduce and/or prevent corrosion. The inhibiting effect of Entada africana leaves extract (EALE) on corrosion of mild steel in 1 M HCL and 0.5 M H_2SO_4 acidic media was investigated in this study. The corrosion rate of mild steel and the inhibition efficiencies of the extract were calculated. The results obtained show that the extract could serve as an effective inhibitor for the corrosion of mild steel in acidic media. The inhibition efficiency increased with increase in concentration of the inhibitor and decreased with temperature. The inhibition efficiency was also found to be significantly increased with time of exposure for the first 3 hours of the experiment for both 1 M HCL and 0.5 M H_2SO_4 before it declined. Therefore, the Maximum Inhibition efficiency of EALE at 0.8%v/v concentration was found to be 96.2% in 1M HCl and 92.1% in 0.5 M H_2SO_4 at 3hr immersion. The phytochemical analysis of the EALE was performed; the result showed that organic constituents were present which made the Entada africana leaves extract a good inhibitor.

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1. INTRODUCTION

Corrosion is the deterioration or degradation of materials by chemical interaction with their environment. Corrosion can cause disastrous damage to metal and alloy structures causing economic consequences in terms of repair, replacement, product losses, safety, and environmental pollution (Umoren, 2008). Due to these harmful effects, corrosion is an undesirable phenomenon that ought to be prevented. There are several ways of preventing corrosion and the rates at which it can propagate with a view of improving the lifetime of metallic and alloy materials. The use of inhibitors is one of the most practical methods for protection against corrosion in corrosive environments (Hammajam, et al 2022). Inhibitors are substances that directly or indirectly coat a film on a metal surface to protect it from its environment. Most inhibitors are adsorbed by the metal surface from a solution or dispersed, but some are applied directly as coatings. Generally, the dissolution of can be suppressed by the action of adsorptive inhibitors which may prevent the adsorption of the aggressive ions, by the formation of a more resistant film on the metallic (El-Maghraby, 2009).

A corrosion inhibitor is a substance which, when added in small concentration to an environment, effectively reduces the corrosion rate of a metal exposed to that environment. Corrosion inhibitors can be divided into two broad categories, namely, those that enhance the formation of a protective oxide film through an oxidizing effect and those that inhibit corrosion by selectively adsorbing on the metal surface and creating a barrier that prevents access of corrosive agents to the metal surface (Leelavathi, and Rajalakshmi 2013). Almost all organic molecules containing heteroatoms such as nitrogen, sulphur, phosphorous, and oxygen show significant inhibition efficiency which have been reported to function as effective corrosion inhibitors of metals and alloys in different aggressive environments (Quarashi et al., 2009). studied corrosion inhibitive properties and adsorption behavior of ethanol extract of *Piper guinensis* as a green corrosion inhibitor for mild steel in H_2SO_4 solution. The study found that the plant extract retarded the acid induced corrosion of mild steel. Makanjoula et al. (2011) investigated corrosion of mild steel in hydrochloric (HCl) acid by tannins from *Rhizophora raceumosa*. Recently, there has been growing interest on some environmentally friendly substances; from natural resources that can be used to control corrosion problems, aside the conventional synthesized organic and inorganic chemicals which have adverse impacts on the environment (Adegoke, 2015). Despite these promising findings about possible corrosion Inhibitors, most of these substances is not only expensive but also toxic non-biodegradable thus causing pollution problems (Lekan *et al*., 2013).

Hence, these deficiencies have prompted the search for their replacement. The phytochemical screening of *Entada africana* leaves demonstrated the presence of flavonoids, saponins, terpenes, acanand alkaloids, tannins. However, the leaves have never been exploited as a corrosion inhibitor in an acid medium. The aim of this study is to investigate the potential of *Entada africana* leaves extract to act as corrosion inhibitor of mild steel in acidic media. The study will immensely contribute to the development of new corrosion inhibitors which are eco-friendly, non-toxic, and cost effective which will substitute the present inorganic inhibitors.

2. MATERIALS AND METHODS

2.1. Materials and Equipment

The materials used for this study were include mild steel specimens, *Entada africana leaves* extract *(Tafasa)*, commercial grade of hydrochloric and sulphuric acid, distilled water, ethanol, molish, mayers and acetone respectively. While the equipment used were Soxhlet extractor, desiccator, bench vice, hack saw, metal scraper, analytical mass balance, conical flask, separating funnel, filter paper, masking tapes, cotton wool, burette, dropper, hand gloves, thread, plastic container and abrasive papers respectively.

2.2. Methods

2.2.1. Preparation of the leaves sample

The fresh leaves of *Entada africana* were collected from Agricultural Research farm of the Ramat Polytechnic Maiduguri. Fresh leaves were cut in to small pieces and shade dried. *Entada africana* leaves were authenticated by botanical survey of the department. The extract was prepared by refluxing 25 g of powdered *Entada africana* leaves in 1 M HCl for 3 h and kept overnight for cooling. The cooled extract was filtered and made up to 500 ml with 1 M HCl acid to get 5%v/v extract of the inhibitor. Similar procedure was adopted in 0.5 M H_2SO_4 and 5%v/v extract in 0.5 M H_2SO_4 was also prepared. Concentration of EALE is expressed in %v/v. Phytochemical screening of the *Entada africana* Leaves extract was carried out using standard procedure.

2.2.2. Preparation of mild steel specimen

The mild steel samples were obtained from a locally available industrial Fe-C steel with very low concentration of carbon. A large sheet of cold rolled mild steel coupons with a chemical composition of carbon 0.13%, manganese 0.23%, silicon 0.03%, phosphorus 0.03%, sulphur 0.016%, chromium 0.022%, nickel 0.012% and iron 99.95% was utilized for the present study. The mild steel samples, with an active surface of 1 cm x 5 cm were used for mass loss measurements and the mild steel samples were mechanically polished, degreased, washed in double distilled water and dried in warm air. Mass loss experiments measurements were done according to ASTM G1-03

2.2.3. Experimental Procedure

The mass loss measurements were carried out using a Denver balance. The specimens were immersed in beaker containing 100 ml acid solution without and with different concentrations of EALE using glass hooks and rods for a predetermined time period at room temperature. At higher temperatures, a constant immersion period of ½ hr was selected and studies were conducted for various concentrations of inhibitors. In order to get good reproducibility, experiments were carried out in triplicate. At the end of exposure period, specimens were cleaned according to ASTM G-81 and the weight recorded. The average mass loss of three parallel mild steel specimens was obtained. The test specimens were removed and then washed with de-ionised water, dried and reweighed. The experiments were performed for various parameters such as:

a) Concentration variation (0.1%v/v ,0.2%v/v, 0.3%v/v, 0.4%v/v, 0.5%v/v, 0.6%v/v, 0.7%v/v and 0.8%v/v)

b) Different time intervals (1/2 h, 1 h, 3 h, 6 h, 12 h and 24 h)

c) Temperature variation (308 K, 318 K, 328 K, 338K, 348 K and 358 K)

2.2.4. Corrosion rate

The corrosion rate was calculated from the weight loss of the coupons at room temperature for various concentrations and immersion times as shown in Equation (1) (Sheeba *et al.,* 2014).

$$\text{Corrosion rate (CR)} = \frac{87.6w}{pAT}(mmpy) \quad (1)$$

Where W = Weight loss in mg, ρ = Metal density in g/cm^3, A = Exposed area of the test coupon in mm^2, and T = Exposure time in hrs.

2.2.5. Inhibition efficiency

The inhibition efficiency of the green inhibitor was calculated from weight loss measured for different inhibitor concentrations using Equation (2) modified from Vasudha and Shanmuga, (2013).

$$\text{IE (\%)} = CR_{blank} - CR_{inhibited} / CR_{blank} \times 100 \quad (2)$$

Where; CR_{blank} and $CR_{inhibited}$ are corrosion rate of mild steel in blank and inhibited solutions

3. RESULTS AND DISCUSSION

3.1. Phytochemical Analysis

The results on phytochemical analysis of the leave extract were shown in Table 1 and 2. The results show the presence (+) of carbohydrates, glycosides, Phlabotannins, saponins, steroids, tannins, flavonoids and alkaloids; anthraquinones were, however, absent (-) in the extracts. This indicates the potential of these plants to be used as green corrosion inhibitors. These substances are known to contain oxygen (O), nitrogen (N) and sulphur (S) atoms in their molecules which are regarded as centers of adsorption of the extracts on the metal surface thereby aiding the formation of a protective film on the metal surface which acts as a barrier separating the metal from the corrosive environment as reported by previous researchers. Generally, the dissolution of metal can be suppressed by the action of adsorption inhibitors which may prevent the adsorption of the aggressive ions, by the formation of a more resistant film on the metallic surface which is in line with (El-Maghraby, 2009).

Table 1: Phytochemical constituents of *Entada africana* leaves extract using qualitative screening

Constituents	Tests	Leaves
Carbohydrates	Molish test	+
Anthroquinons	Bontrager test	+
Glycosides	Lieberman Burchard‟s test	+
Phlabotannins	Frothin test	3.5%
Tannins	Ferric chloride test	6.2%
Flavanoids	Shinods test	8.9%
Alkaloids	Dragedoff‟s test	1.2%
Monosaccharide	Barfoed‟s test	+

Table 2: Quantitative phytochemical screening

Phytochemical constituents	Composition of leaves
Alkaloids	42mg/g
Flavonoids	7mg/g
Saponins	41mg/g
Tannins	10mg/g

3.2. Mass Loss Measurements

The mass loss method of monitoring corrosion rate is useful because of its simple application and reliability. Inhibition efficiencies of mild steel with different concentrations of *entada africana* leaves extract in 1 M HCl and 0.5 M H_2SO_4 solutions at room temperature at different periods of immersion are presented in table 3.3. From the table, corrosion rate decreases noticeably with an increase in EALE concentration, i.e. the corrosion inhibition efficiency enhances with the inhibitor concentration. This behavior is due to the fact that the adsorption and coverage of the inhibitor on the mild steel surface increase with the inhibitor concentration. Maximum Inhibition efficiency of EALE at 0.8%v/v concentration was found to be 96.2% in 1 M HCl and 92.1% in 0. 5 M H_2SO_4 at 3h immersion. At any given inhibitor concentration, the corrosion rate in 0.5 M H_2SO_4 is comparatively higher than that in 1M HCl solution hence inhibition efficiency is higher for the investigated inhibitor in 1M HCl. The high inhibitive performance of EALE suggests a higher bonding ability of inhibitor on to mild steel surface. Similar observations were reported by (Ahmed *et al*., 2021)

3.3. Effect of Immersion Time

In order to assess the stability of adsorbed inhibitor film at mild steel/ acid solution interface with time, mass loss measurements were performed in both acid media in absence and presence of inhibitors at various concentrations for ½ hr to 24 hr immersion time at 35°C. From Figure 1 and 2 it was noticed that a maximum inhibition efficiency of 96.2% and 92.05% were observed for 3hr of immersion period for 1 M HCl and 0.5 M H_2SO_4 respectively. Immersion studies revealed that as the time of immersion increased from 1/2 hr to 3 h the inhibition efficiency also increased from 84.9% to 96.2%. After 3 h there is a slight decline in the inhibition efficiency at 6 h yielding 92.6% and then stabilized at 24 hr (81.5%). This may be explained due to increase of adsorbed of inhibitor molecules on mild steel surface with time. Prolonged immersion may result in desorption of the extract from mild steel surface. Shriver *et al*., (1994) explained that decrease in inhibition for long period of immersion can be attributed to the depletion of available inhibitor molecules in the solution due to chelate formation between iron and the inhibitor ligands. The stabilization of the inhibition efficiency at 24 hr proves that EALE is a promising inhibitor for MS in 1M HCl and 0.5 M H_2SO_4 at various time of immersion.

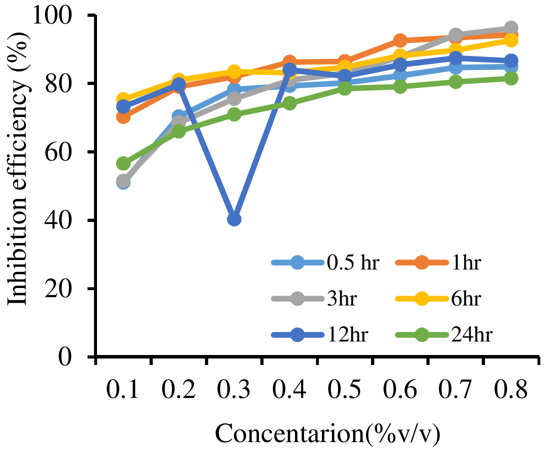

Figure 1: Effect of immersion time on corrosion inhibition efficiency in 1 M HCL solution on mild steel

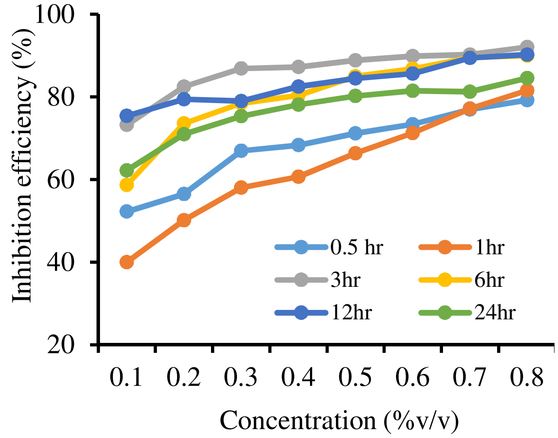

Figure 2: Effect of immersion time on corrosion inhibition efficiency in 0.5 M H_2SO_4 solution on mild steel

3.3. Effect of Temperature

To evaluate the adsorption of EALE in both acid media and activation parameters of the corrosion process of mild steel in acidic media, mass loss measurements were carried out in the range of temperature 308–358 K, and the results are depicted in Figure 3. As the temperature rises from 308 K to 328 K, inhibition efficiency was found to increase from 85.9% to 94.3% in 0.5M H_2SO_4 and 79.1% to 88.3% in 1M HCl at maximum concentration of 0.8%v/v. Further rise in temperature, decreased the IE but at higher concentration affording 81.6% in 1M HCl and 84.5% in 0.5M H_2SO_4. These observations established the effectiveness of EALE in reducing corrosion of mild steel in the temperature range of 308 K to 338 K in 1M HCl and in 0.5 M H_2SO_4.

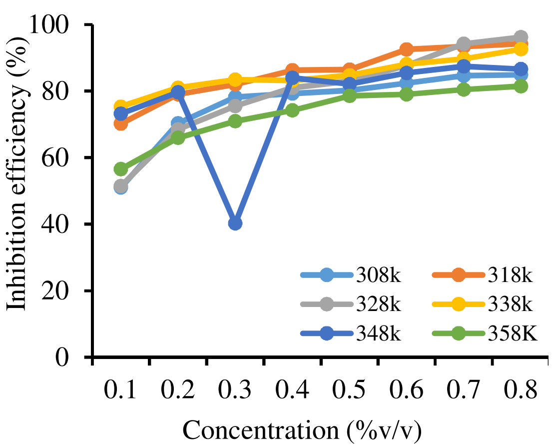

Figure 3: Effect of temperature on corrosion inhibition efficiency in 1 M HCL solution on mild steel

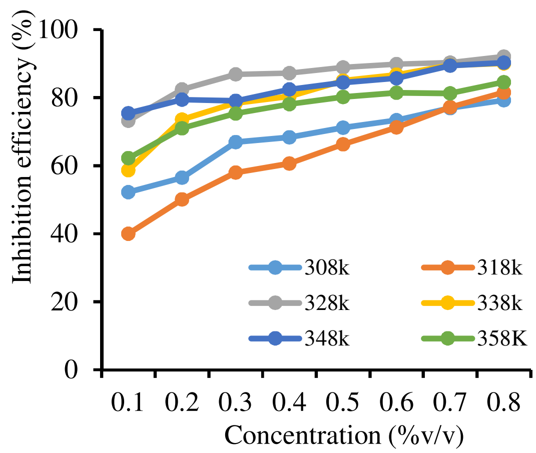

Figure 4: Effect of temperature on corrosion inhibition efficiency in 0.5 M H_2SO_4 solution on mild steel

As shown in Table 3, the adsorption and desorption of inhibitor molecules continuously occur at the metal surface and the equilibrium exists between these two processes at a particular temperature. With the increase of temperature, the equilibrium between adsorption and desorption process is shifted leading to a higher desorption rate than adsorption until equilibrium is again established at a different value of equilibrium constant. Values of inhibition efficiency with temperature indicate that the adsorption of EALE on mild steel surface is physical at low temperature and at higher temperature; chemical adsorption may also take place. This is evidenced from inhibition efficiency values of EALE in 1 M HCl (81.5 % at 358 K) and in 0.5 M H_2SO_4 (84.6 % at 358 K).

Table 3: Inhibition efficiency as a function of immersion time and concentration of EALE in 1M HCl and 0.5 M H_2SO_4

C (%v/v)	Inhibition efficiency for various immersion time in HCl						Inhibition efficiency for various immersion time in H_2SO_4					
	0.5	1	3	6	12	24	0.5	1	3	6	12	24
0.1	51.00	70.23	51.45	75.35	73.25	56.56	52.23	40.00	73.22	58.66	75.40	62.20
0.2	70.33	79.00	68.55	80.99	79.66	65.96	56.45	50.11	82.45	73.55	79.44	70.96
0.3	78.22	81.99	75.53	83.45	40.22	70.97	66.89	57.99	86.88	78.44	79.00	75.33
0.4	79.33	86.23	80.98	83.22	84.00	74.22	68.33	60.66	87.22	80.33	82.45	78.11
0.5	80.23	86.45	83.02	84.75	82.12	78.55	71.15	66.33	88.85	84.98	84.45	80.20
0.6	82.33	92.55	87.75	88.12	85.45	79.05	73.35	71.23	89.85	86.75	85.65	81.44
0.7	84.65	93.45	94.25	89.75	87.42	80.45	76.95	77.15	90.25	89.45	89.40	81.26
0.8	84.90	94.25	96.20	92.61	86.65	81.50	79.22	81.55	92.05	90.02	90.25	84.55

4. CONCLUSION

From the results of qualitative and quantitative analysis, it was concluded that these plant materials contain a wide range of natural compounds with different bioactive components. In the current study, the result has shown that the leaves extract from *entada africana* contain alkaloids, flavonoids, tannins, saponins and phenols. These chemicals contain atoms of oxygen (O), nitrogen (N) and sulphur (S) in their molecules which usually act as centers of adsorption on the metallic surface thereby producing a protective film that acts as a barrier separating the metal from the corrosive environment thereby protecting it from corrosion attack.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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