

Investigation on Corrosion Inhibition of *Hura Crepitans* for 2101 Duplex Stainless Steel in an Acidic Environment

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ABSTRACT

The inhibition effect of *Hura Crepitans* seed extract (HCS) on the corrosion of Duplex Stainless Steel 2101 (DSS2101) in 0.5M H₂SO₄ solution has been studied for the first time by weight loss test method and potentiodynamic polarization method. The extracts elemental compositions were characterized using atomic absorption spectroscopy (AAS). The phenolic and aromatic functional groups in the extracts were revealed with Gas Chromatography Mass Spectroscopy (GC-MS). From the result, phytochemicals such as saponin, tannins, steroid and flavonoids were observed. The presence of the organic group accounted for the inhibitive efficiency recorded in the environment. Potentiodynamic polarization test was also conducted to confirm the effectiveness of inhibition. The polarization studies revealed that generally the inhibitor produced from seed extract of *Hura crepitans*, improved the corrosion resistance of DSS2101 in 0.5M H₂SO₄. The optimum corrosion inhibitor efficiency was observed at 0.2% inhibitors in 0.5M H₂SO₄ for 2101DSS. Polarization curves reveal that HCS extract acts as a mixed-type inhibitor in 0.5M H₂SO₄.

Keywords: corrosion, steel, *Hura crepitans*, inhibitor, sulphuric acid.

INTRODUCTION

Corrosion as a natural universal phenomenon has turned into a global threat, as its trigger materials interaction with their immediate environment [1]. The versatility of steels in structural and load bearing applications makes its corrosion worthy of investigation [2, 3]. Corrosion causes material loss which affects domestic and industrial operations severely [4, 5]. The key to the decrease of corrosion failure depend on the adaptation of timely and appropriate control measure and awareness to corrosion [6, 7]. Like other natural hazards, the catastrophic nature of corrosion in structural and industrial environments comes with devastating consequences [8, 9]. Losses sustained by corrosion amounts to many billions of

dollars annually [10, 11]. The economic factor is the main objective, for much of the current research in corrosion; others include safety of machineries and radioactive waste vessels [12].

Corrosion inhibitors can either be synthetic or natural, they decrease the effect of corrosion on metals. Several synthetic compounds are known to be applicable as good inhibitors of corrosion for metals, some of them are expensive and toxic in nature [13, 14, 15]. Organic inhibitors are now the choice for many researchers since they are eco-friendlier and economical [16] Good eco-friendly organic inhibitors like plant extracts are antioxidants. The application of plant extracts as corrosion inhibitor has become important because of their availability,

renewability and environmental acceptability for a wide range of green inhibitor [17, 18].

Corrosion inhibitors can serve as temporary or permanent protection depending on the corrosion aggressiveness of the environment. An inhibitor efficiency depends on the effectiveness, economical and compatibility of the environment. Steel as one of the major construction materials is extensively used in chemical and allied industries for handling acid, alkali and salt solutions [19, 20]. The standard life span of a pipe line is less than half a century. Research findings shows that corrosion contributes a greater percentage to failure in material integrity [21, 22, 23].

An overview of the researches done in corrosion inhibition provides insight on the level and nature of studies undertaken in this field. Corrosion control and prevention has been tailored towards organic inhibitors among other corrosion control methods because of its inherent health and safety considerations. It is noteworthy that researches have been geared towards environmentally acceptable and renewable substances [24, 25, 26]. The objective of this work was to study the corrosion inhibition of Duplex Stainless Steel 2101 (DSS2101) in a 0.5M H₂SO₄ solution in the presence of Hura Crepitans seed extract (HCS) as an inhibitor respectful of the environment by potentiodynamic polarization curves and weight loss measurements.

MATERIALS AND METHODS

The metal used for this research was 2101 Duplex Stainless Steel. It was obtained from Advanced Materials and Electrochemical Research Group (AMERG) at Federal University of Technology, Akure, (FUTA). It was cut into corrosion coupons in accordance to ASTM G59-97 (2014) for polarization analysis. The selected steels were cold mounted using polyester resin, after which the samples were polished with series of emery paper from 60 – 1200 µm grade to remove any mill scale. The 2101DSS coupons used in this study were prepared from plate having the composition (in wt.%) is C (0.03), Si (0.45), Mo (0.5), P (0.023), Ni (1.5), Cu (0.001), N (0.22), Cr (21.5), Mn (4.80), Fe (balance). The weight loss test was carried out with steel metal plates of sizes 0.1×1×1 cm. The working metal steel samples surface was grounded with emery paper of different grits and then rinsed with ultrapure water (Milli-Q, Millipore) water, degreased with acetone, dried and stored in desiccators prior to use.

Preparation of plant extract

Ripe and Dried *Hura crepitans* seeds (HCS) were collected from trees grown within the premises of the campus of Afe Babalola University, Nigeria, the matured and healthy fruits were randomly picked. These were then kept in a dark polyethylene bag and transported immediately to the laboratory for further processing. It was identified and authenticated at Botany Department of Ekiti State University, Ado-Ekiti, Nigeria. The pods were cut open and the seeds were removed from the pods while the creamy white cotyledons inside the endocarp was gently removed. It was further sun dried for a week and the seeds were latter grinded into fine powder using electronic blender. It was then stored in a polythene bag for extraction and additional study.

The dried seeds were blended to fine powder using electronic blender. Ethanol was used as a medium of extraction by weighing 300 g of the sample to a 1000 ml beaker and 500 ml of ethanol was added. The extract obtained was concentrated in water bath heated to 80 °C until all ethanol was evaporated to a semi-solid mass and the extract thus obtained was used for the research.

Polarization measurement

The Autolab Potentiostat with Nova 2.0 software and with a potential of -0.2 mv to 1.2 mv at a scan rate of 0.01 mv was used to determine the polarization of the test sample in the solution using platinum counter electrode. The Autolab potentiostat after carrying a Linear Voltammetry sweep test on the steel sample in different varying environments analyzed and recorded the parameters of the corrosion such as the corrosion current density (I_{corr}) in area per centimeter square, corrosion potential (E_{corr}), in voltage and the corrosion rate in millimeter per year among other parameters.

For polarization measurements, a potentiostat Voltalab301 PGZ monitored by a PC computer and Voltmaster 4.0 software were used to run the tests, collect and evaluate the experimental data. During each experiment, the test solution was mixed with a magnetic stirrer.

The inhibition efficiency (IE %) depends on the degree of coverage of the mild steel surface by inhibitor molecules and can be calculated with the following equations:

$$IE = \frac{C_{\text{blank}} - C_{\text{inhibitor}}}{C_{\text{blank}}} \times 100\% \quad (1)$$

where: IE is the inhibition efficiency;
 C_{blank} and $C_{\text{inhibitor}}$ are corrosion rate without inhibitor and with inhibitor respectively [27].

Weight loss measurements

DSS2101 specimens with the same composition used in the electrochemical measurements with dimension of 0.1×1×1 cm were immersed in 100 mL of electrolyte with and without optimal concentrations of HCS extract. The weights of the specimens before and after immersion were detected by analytical balance (sensitivity ±0.0001 g). After one week and everyday exposure, the specimens were rinsed thoroughly with deionized water, dried for one hour in electrical oven at 70°C then cooled in desiccator and weighed again as quickly as possible. Three parallel experiments were done for each test. Weight loss calculation of the IE (%) of extracts [27]:

$$CR = \frac{\text{weight loss (g)}}{\text{area (m}^2\text{)} \times \text{time (day)}} \quad (2)$$

Corrosion rate (gravimetric method)

The test for corrosion was done by immersing the DSS2101 in of 0.5 M Hydrogen tetraoxosulphate (VI) acid (H_2SO_4) corrosive medium with and without extract. While the value of corrosion rate was calculated for exposure time 48, 96, 192, 384 and 768 hours. The varying concentration of the extract ranging from blank, 0.1%, 0.2%, 0.3%, 0.4% and 0.5% was immersed into the H_2SO_4 acid

medium. The sample was removed from the corrosion medium after the completion of corrosion process at a given time. This was then followed with washing with double distilled water with the aid of soft brush, rinsed with acetone and dried at room temperature.

The corrosion rate (CR) was calculated using the expression in equation:

$$CR = \frac{87.6 \times (W_{\text{blank}} - W_{\text{inhibitor}})}{\rho St} \quad (3)$$

where: ρ – steel density ($g\ cm^{-3}$);
 S – area of the metal exposed (cm^2);
 t – time of immersion (h);
 W_{blank} and $W_{\text{inhibitor}}$ – the weight loss (g) for DSS2101 with and without the inhibitor in H_2SO_4 solution respectively [27].

RESULTS AND DISCUSSION

1. Phytochemical screening

Phytochemical screening of the HCS extract was performed to identify the chemical constituents present and the result is shown in Table 1.

The phytochemical analysis of the HSC extracts, show that the plant extract contains phytochemicals that are corrosion resistant organic agents (e.g. saponin, tannins, steroid and flavonoids). Tannin compound as organic agent forms complexes with Fe (III) on metal surface [28]. The presences of saponin and flavonoid on metal form complex affinity that is effective for corrosion inhibitor performance. This shows that the presence of these phenolic compounds on the DSS2101 metal surface provided strong adsorption molecules of the HCS extract film. Hence,

Table 1. Phytochemical analysis of plant extracts

S/No	Phytochemical	Tests	Inference leaf	Bark	Root
1	Carbohydrates	Molisch	+	+	+
2	Anthraquinones	Bontrager's	-	-	-
3	Cardiac glycosides	Kella killani	+	+	+
4	Glycosides	Fehling	+	+	+
5	Saponins	Froth	+	+	+
6	Steroids/ terpenes	Liebermann	+	+	+
7	Tannins	Ferric chloride	+	+	+
8	Flavonoids	Shinoda	+	+	+
9	Alkaloids	Dragendorff	+	+	+
10	Phenols	Pyridine -FeCl ₃	+	+	+

Note: (+) means the presence of the chemical constituent and (++) means the presence of more chemical constituent.

Table 2. AAS result for *Hura crepitans* seed showing the concentration of some metals

Samples	Ca (ppm)	Fe (ppm)	Mg (ppm)	Cu (ppm)	Mn (ppm)	K (ppm)	Ni (ppm)	Zn (ppm)
Seed	91.800	0.647	10.915	0.302	0.245	64.500	40.200	1.245

Table 3. Density and pH result of samples

Parameter	Seed
Loss volume	50
Compact volume	44
Mass	47.84
Density	1.087273
pH	5.08

it added to the inhibition efficiency of the inhibitors which might be as a result of the cyclic compounds [29].

2. Elemental constituents of the HCS extract

Analyses were performed by AAS depending on the level of concentration of metals present and the result is as shown in Table 2. The Atomic Absorption Spectroscopy result showed that heavy metals present are in minute concentration in the plant extracts, hence making the plant extract suitable for use as inhibitor and also responsible for the inhibition efficiency of the extract on the metal samples used.

3. pH and density (Table 3)

4. Proximate analysis (Table 4)

5. Gas chromatography/mass spectroscopy (GC/MS) of *Hura crepitans* seed extract

The extracts that obtained by soxhlet from HCS are a golden yellow colour at room temperature. The *Hura crepitans* seeds extractions with different solvents have shown that the best efficiency is with water and then with ethanol. The extract composition was determined by GC/MS. The structures of the major compounds are shown in the chromatogram Fig. 1.

Earlier researchers have confirmed that GC/MS is a powerful instrument that can be used to determine the major organic inhibitors adsorbed on the surface of metals or alloys [30]. A GC/MS spectrum was further used to characterize the chemical constituents that contribute to the corrosion inhibition of the extracts and were found to have specific functional compounds. Fig.1 show the GC/MS spectrum of the HCS extract.

As described in Table 5 GC/MS is used to determine the different composition of HCS extract, while the chromatogram in figure I showed the

Table 4. Proximate analysis result of samples

Parameters (%)	Ref	Ref	Ref	Avg
Moisture content	3.243	3.876	4.795	3.971
Fat content	52.703	54.211	52.899	53.271
Crude fibre	17.093	17.413	17.507	17.337
Ash content	3.243	3.101	3.425	3.256
Protein content	10.501	10.824	10.115	10.480
Carbohydrate	13.217	10.576	11.260	11.684

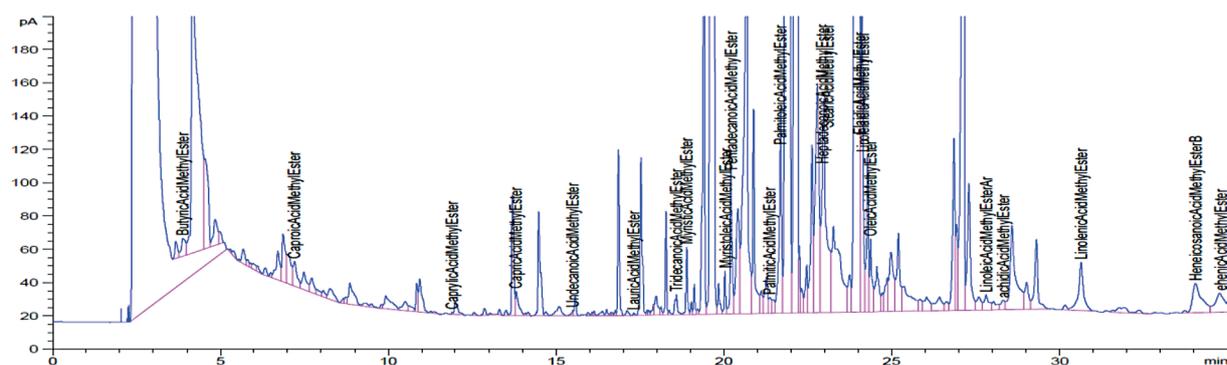


Fig. 1. The GC/MS spectrum of the *Hura crepitans* seed extract

Table 5. The chemical profile of the identified compounds in the chromatogram

Compound Names	Non-C/H Atom count	Metal atom Count	Electro negativity atom count	Rotatable bond count	Ring closure count	Aromatic ring count	Aromatic atom count	SP ³ atom count	Amine Count	Alky-Amine Count	Aromatic Nitrogen atom count
Stearic acid	2	0	2	16	0	0	0	18	0	0	0
Myristic acid	2	0	2	16	0	0	0	14	0	0	0
Palmitic acid	2	0	2	14	0	0	0	16	0	0	0
Lauric acid	2	0	2	10	0	0	0	12	0	0	0
Linoleic acid	2	0	2	14	0	0	0	14	0	0	0
Myristoleic acid	2	0	2	11	0	0	0	12	0	0	0
Palmitoleic acid	2	0	2	13	0	0	0	14	0	0	0
Linolelaidic Acid	2	0	2	14	0	0	0	14	0	0	0
Arachidic acid	2	0	2	18	0	0	0	20	0	0	0
Linolenic acid	2	0	2	13	0	0	0	12	0	0	0
Behenic acid	2	0	2	20	0	0	0	22	0	0	0

major compounds. The study of the HCS extracts as anticorrosion gave the same results as Ajmal et al., Arockia et al. and Desai [31, 32, 33].

Generally, the extract characterization of the showed that it can used as corrosion inhibitors.

Hence, in this study the extract of HCS was used as corrosion inhibitor of 2101DSS in 0.5M H₂SO₄ acid media.

6. Potentiodynamic polarization analysis

The use of polarization resistance technique for corrosion testing is intended to observe the changes in sample resistance to oxidation at the present of external potential. The superimposed potentiodynamic polarization curves of 2101duplex stainless in 0.5 M H₂SO₄ with and without HCS extract at 298 K was presented in Figure 2.

The electrochemical parameters values for different concentrations of ethanol extracts of HCS are given in the Table 6.

Table 6 shows that the corrosion I_{corr} decreases for HCS extract by increasing their concentration and thereby increasing the inhibition efficiency. The presence of different HCS extract produced a shift in the E_{corr} toward positive values between -0.55627V and -0.639433V.

The superimposed potentiodynamic polarization curves of the corrosion of DSS2101 in 0.5M H₂SO₄ solution are as shown in Figure 2. Table 6 shows the corrosion parameters such as E_{corr} and I_{corr} of anodic and cathodic slopes. The polarization curves indicated that both anodic and cathodic reactions are inhibited on addition of HCS extract. The values obtained show that the corrosion rates (CR) decrease with addition of inhibitors. The inhibition by the organic inhibitor is as shown in Table 6.

This result showed the maximum IE at 58.77% which means that there are moderate corrosion inhibitors for DSS2101 metal. This

Table 6. Electrochemical polarization parameters for 2101 duplex stainless steel immersed in 0.5M H₂SO₄ solution at different concentrations of *Hura crepitans* seed extract

Seed				
Samples concentration (ppm)	E _{corr} (V)	I _{corr} (A/cm ²)	Corrosion rate (mm/yr)	Inhibitor efficiency (%)
0%	-0.55627	1.7895E-10	2.0794E-06	-
0.1%	-0.54543	1.0068E-10	1.1699E-06	43.73
0.2%	-0.63943	7.3764E-11	8.5713E-07	58.77
0.3%	-0.61680	8.1131E-11	9.4274E-07	54.66
0.4%	-0.62999	8.2273E-11	9.5601E-07	54.02
0.5%	-0.62424	1.4193E-10	1.6492E-06	20.69

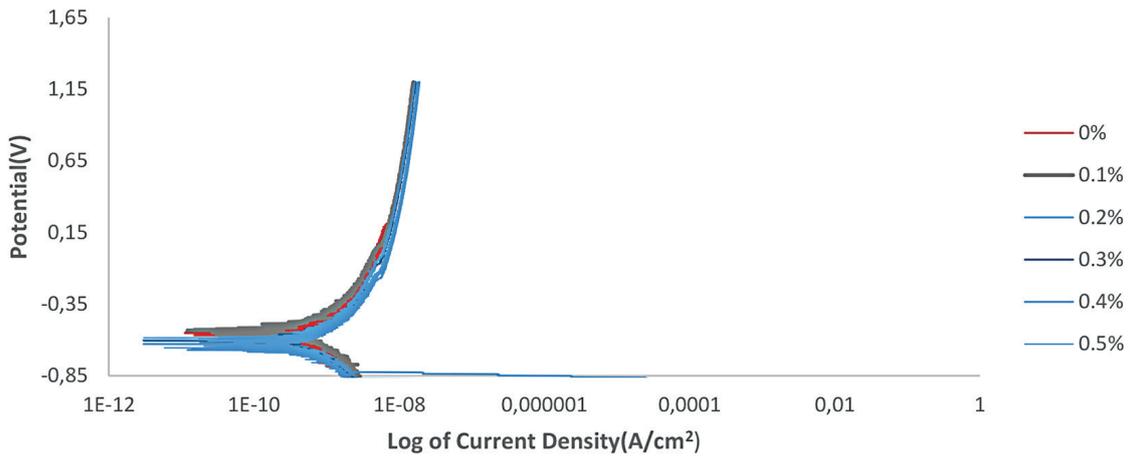


Fig. 2. Polarization curves for DSS2101 immersed in 0.5M H₂SO₄ in the presence and absence of *Hura crepitans* seed extract as inhibitor at 298 K

also agreed with earlier works on inhibition by *Cyamopsis tetragonoloba* seed [34] and *Calotropis* plant [35].

7. Weight loss measurement

The effectiveness of corrosion inhibitor HCS extract solution on DSS2101 metal in 0.5M H₂SO₄ can be proven with increase in the HCS extract concentration. The result is shown in Figure 3.

Figure 3 clearly shows a loss in the weight of the DSS2101 metal coupons on addition of HCS extract compared to the absence of the extracts. It can also be observed that corrosion is a time dependent, because the CR changes directly with time, and also there is an increase at a specified condition which agreed with the findings of Ebenso et al., [36]. The gravimetric result agrees with the potentiodynamic results in this study.

CONCLUSION

From the research work carried out, the following conclusion can be made. Results of the electrochemical performance of *Hura crepitans* seed extract organic compound on the corrosion inhibition of high carbon steel in dilute H₂SO₄ confirms the organic derivative to be highly effective. The presence of saponin, tannins, steroid and flavonoids, and other organic compound group observed from the GC/MS spectrum accounted for the inhibitive efficiency recorded in the subjected environment. The polarization curves indicated that both anodic and cathodic reactions are inhibited on addition of *Hura crepitans* seed extract. The values obtained shows corrosion resistance with addition of inhibitors for all the environment. The optimum resistance was observed

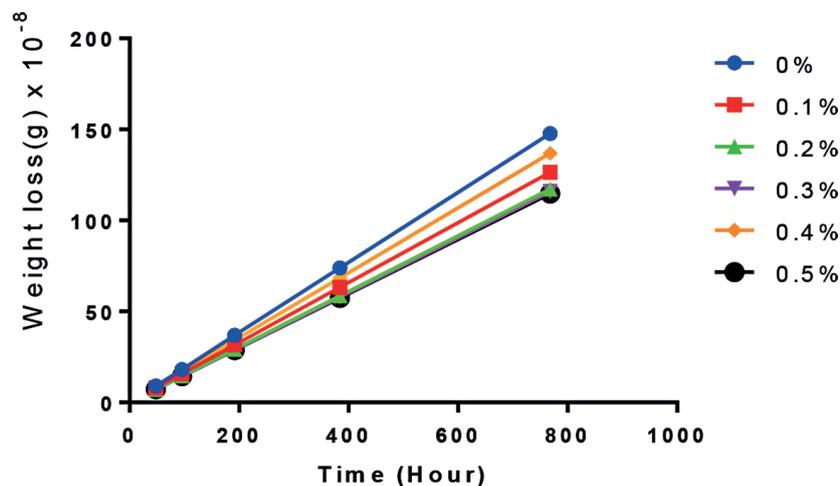


Fig. 3. Weight loss variation with exposure time for 2101 duplex stainless sample immersed in 0.5M H₂SO₄ with various *Hura crepitans* seed extract concentrations

at 0.2% for both potentiodynamic and gravimetric test. Statistical analysis show that *Hura crepitans* seed extract concentration and exposure time had influence on its inhibition efficiency values.

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