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Determination of the Effect of Newbouldia Leavis Leaf Extract on the Corrosion of Mild Steel in Sulphuric Acid

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ABSTRACT:
The inhibitive properties of Newbouldia leavis leaf extract for the corrosion of mild steel was investigated using gravimetric technique in 1 mole of sulphuric acid. The leaves were gotten and dried after which they were ground to micron size. The extract of the leaves was obtained using ethanol reflux method and the weight of the leaves extracted deduced. The quantities for different concentrations (0.1g/L, 0.2g/L, 0.3g/L, 0.4g/L and 0.5g/L) of the extract were also deduced and applied into the acidic medium, and the mild steel coupons dipped into them over four hours intervals (4 to 20hrs). The weight loss values for each metal coupon were obtained and the inhibition efficiency calculated. The gravimetric technique used confirmed that Newbouldia leavis leaf extract is a good inhibitor of mild steel corrosion with increasing inhibition efficiency as concentration of the inhibitor increases. Inhibition efficiency values of 72% to 90% were obtained. From the results obtained for weight loss and corrosion rate, it was revealed that Newbouldia leavis leaf extract act as an efficient inhibitor showing weight loss (from 0.173g to 0.017g) and corrosion rate (from 43mm/yr. to 4.2 mm/yr.) decreasing with increase in concentration (from 0.0g/L to 0.5g/L) of the plant extract. Plant extracts are excellent substitute as corrosion inhibitors because of it availability, biodegradability, not expensive, less harmful to humans and environment. This research work has shown that Newbouldia leavis leaf extracts if applied in the right proportions will prolong the life of mild steel in service.

Keywords: Corrosion, Gravimetric technique, Inhibition Passivation, Reflux.

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1. INTRODUCTION

Base on American Society of Testing and Materials (ASTM) International, corrosion is defined as the chemical or electrochemical reaction between a metal and the substances around it. This tends to deteriorate the properties of the metal (Brycki *et al.*, 2018). It is also defined by International Organization for Standardization (ISO) as the physiochemical reaction that occurs between a metal and the substances around it. As a result of this metal properties deterioration, this interaction may also lead to weakened metal that cannot perform optimally (ISO 8044, 2015). In nature, metal atoms are present in chemical compounds, and corrosion can take place due to differences in the electrolytic composition. Such variances may be present as resistivity of the soil, concentration of oxygen in the soil, humidity content and various ionic concentrations (Papavinasam, 2011).

Corrosion control of metals has been of great importance particularly in the production industry (Rani & Basu, 2012). Corrosion inhibitor is a major solution used in the oil and gas production and processing industries as a defense against corrosion. Corrosion can lead to failures in plant infrastructure and machines which are usually costly to repair, costly in terms of lost or contaminated products, corrosion can also be a major factor that can hamper safety. In order to make the best decision the condition causing the corrosion of any component or parts must be critically studied to know the rate of deterioration.

With the result of the critical study better assessment can be implemented on repair and management of such component (Roberge, 2008).

Corrosion can also hamper the cost of production, construction, design, the cost of corrosion-related maintenance, rehabilitation and restoration, and depreciation of the cost of unusable corroded materials due to be replaced (Koch, 2017).

Corrosion is associated to chemical and electrochemical processes. Chemical corrosion occurs in gases (in gas bearing containers) with low humidity and non-conductive liquids in which electrons cannot pass through. Chemical corrosion causes an oxide layer to form on the surface of the material. McCafferty (2010) resolved that electrochemical corrosion takes place in the interface between metallic materials and electrolytes. Redox reaction occurs due to the potentials difference in which a fraction of the metal reacts as an anode where metal oxidizes and becomes ion, while the other fraction reacts as a cathode, where depolarization occurs; it is basically a process of reduction of oxygen and hydrogen cation (Hou *et al.*, 2017; Stewart *et al.*, 2012).

When mild steel is exposed to a corrosive environment, for instance, sulphuric acid, an oxide film will form and corrosion defects begins from part of the steel with mechanical dent like scratches or some natural discontinuities in the film, i.e. grain boundaries, inclusions, dislocation networks at the surface of the steel. Thus, in other words, corrosion is more likely to occur on an uneven surface.

Some research groups have reported the successful use of naturally occurring substances to inhibit the corrosion of metals in acidic and alkaline environments. Delonix regia extracts inhibited the corrosion of aluminum in hydrochloric acid solution (Abiola *et al.*, 2007). Rosemary leaves were studied as corrosion inhibitor for the Al + 2.5Mg alloy in a 3% NaCl solution at 25°C (Kliskic *et al.,* 2000). El-Etre (2003) investigated natural honey as a corrosion inhibitor for copper. El-Etre (1998) investigated the effect of opuntia extract on corrosion inhibition of aluminum in hydrochloric acid solution.

Okra mucilage, a natural grade polysaccharide, was used to inhibit the corrosion of mild steel in 0.5 mole of H_2SO_4, presented as a cathodic type inhibitor for corrosion in mild steel (Amar *et al.*, 2017).

Gerengi *et al.* (2012) reported that Schinopsis lorentzii extract has a marginal Cathodic inhibition efficiency. Nevertheless, inhibitor can either be anodic, cathodic or mixed inhibitor. A report discovered some disparities depending on the environment, this indicated that a guanidine derivative behaves as cathodic in 1 mole of HCl and as mixed-type in 0.5 mole of H_2SO_4 (Khaled, 2008).

Amin *et al.* (2010) worked on evaluating the performance of three preferred amino acids, namely alanine, cysteine and S-methyl cysteine as safe corrosion inhibitors for iron in aerated stagnant 1 mole of HCl solution. The study used computational studies evaluating the HOMO (Highest Occupied Molecular Orbit)-LUMO (Lowest Unoccupied Molecular Orbit) orbitals and many hypotheses about modes of adsorption (physical, chemical and adsorption via H-bonding) of the three amino acids on the electrode surface were formulated.

Other works were able to make some speculations on the adsorption mechanism and therefore the inhibition mechanism is lacking in deeper explanations supported by theoretical or experimental data. The corrosion inhibition effect of a Henna extract was investigated, probable interpretation of the observed inhibition action was given without any strong data support, providing hypothesis relatively to complexes formation rather than adsorption mechanism (El-Etre *et al.*, 2005).

In another work on Henna extract, Ostovari *et al.* (2009) highlighted the magnitude of investigating the mechanism beyond the corrosion inhibition effect. Conductimetric titration was used as a regular technique to corroborate the speculation of formation of insoluble complex compounds mixed with the metallic cations and adsorbed lawsone molecules.
Additionally, they measured separately the inhibition efficiencies of the constituents of the extract and of the extract itself, lawsone displayed the highest inhibition efficiency among the other henna constituents such as gallic acid, glucose and tannic acid; it also had higher inhibition efficiency than the henna extract itself.

Shukla *et al.* (2009) claimed that inhibition occurs with the test of Streptomycin on a metal surface as the drug was absorbed by the metal surface without adjusting the mechanism of corrosion. However, only guess was given with no study of the corrosion mechanism.
Previous works explored the effect of corrosion inhibition and antibacterial drugs like ampicillin, cloxacillin, flucloxacillin and amoxicillin. The Process of inhibition process was credited to the development of insoluble complex film on the surface of the metal; as at this time this guess is not yet supported by experiments or theoretical calculations. The cost of using antibacterial drugs should have been taken into consideration since it is relatively expensive if used as an inhibitor (Abdullah, 2003).

There has been some research on the inhibitive action of Newbouldia leavis on metal corrosion, Nnanna *et al.* (2011) showed that the inhibition efficiency on an aluminum sample tested depends on plant extract concentration and the duration of exposure of the samples to H_2SO_4 solutions containing the extract. Nwosu *et al.* (2018) revealed the effect on hydrochloric acid corrosion on mild steel. The existence of the Newbouldia leavis leaf extract drastically decreased the weight loss and also lessened the rate of redox reaction on the aluminum alloy. The inhibition efficiency of the inhibitor is directly proportional to the concentration. However, an increase in temperature causes reduction in inhibition efficiency. The inclusion of the leaf extract changes the electrochemical behaviour of the metal in the acidic environment.
According to Charu *et al.* (2012) phytochemical analysis carried out on the Newbouldia Leavis Leaf extracts shows that it contains flavonoid and tannins which gives the leaves its inhibitive properties.
This research work was conducted to find a suitable replacement for inorganic corrosion inhibitors (for example: Chromates, Nitrites and Nitrates Salts) which are harmful to both humans and the environment.

2 MATERIALS AND METHODS

2.1. Newbouldia leavis Leaf Preparations

With reference to the work of Nnanna *et al.* (2012) the chemicals and reagents used for the study were of analytical grade.
The leaves of Newbouldia leavis plant were collected from Umuahia, in the Eastern region of Nigeria. The leaves were air dried away from direct daylight. The corrosive medium used was 1 mole of sulphuric acid (H_2SO_4). Stock solutions of the plant extract was prepared by boiling weighed amounts of the dried and ground leaves of Newbouldia leavis under reflux for 3 hours in H_2SO_4. The resulting solutions was allowed to cool to room temperature and filtered. The amount of plant material extracted into solution was quantified by comparing the weight of the dried residue with the initial weight of the

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powdered plant material before extraction. Inhibitor test solutions ranging from 0.1 to 0.5g/L was prepared from the stock solution by appropriately diluting with the acidic medium.

2.2. Metal Preparations

The experiment was carried out with cold rolled moderate metal sample of chemical composition (C = 0.05%, Mn = 1.00%, Si = 0.05%, S = 0,025%, P = 0.25%, and Fe = 78%). The steel sheets were cut into coupons of dimension (15 x 15 x 1mm) and they were abraded with diverse grades of emery paper (120, 600 and 1200) and washed with detergent, degreased in ethanol, air dried and weighed.

2.3. Gravimetric Method

The pre-weighed coupons were exposed to diverse test solutions. The study was conducted with the diverse concentrations (0.1g/L to 0.5g/L) of the inhibitor (Newbouldia Leavis Leaf extracts) at room temperature. After the exposure period of between 4hrs to 20hrs, the coupons were retrieved, rinsed with plenty of water, dried and thereafter weighed. The weight loss was estimated. The procedure was repeated three times and the average result was taken.

The corrosion rate was calculated using equation 2.

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

$$CR = \frac{K\Delta W}{At\rho} \qquad (2)$$

Where K = Rate constant equal $8.78x10^4$
W_1 = Weight before immersion in g
W_2 = Weight after immersion in g
ΔW = Weight loss in g
ρ = Density of material in g/cm^3
T = Exposure time in hours
A = Exposed area of coupon cm^2
The efficiency was calculated using the formula

$$\eta_{gravimetric} = (1-\frac{w_i}{w_o})x100\% \qquad (3)$$

Where w_i = Weight of test piece when dipped into corrosive environment with leaf extract.
w_o = Weight of test piece when dipped into corrosive media without leaf extract.

3. RESULTS AND DISCUSSION

3.1 Gravimetric Measurements

Below are the results of the gravimetric experiments.

Table 1: Weight loss measurements for mild steel in 1 mole of sulphuric acid in the absence and presence of *Newbouldia leavis* leaf extract.

S/N	Exposure Time (hrs.)	Weight loss at Different Concentration (g/L)					
		Control 0.0	0.1	0.2	0.3	0.4	0.5
1	4	0.025	0.007	0.005	0.005	0.005	0.005
2	8	0.069	0.016	0.010	0.009	0.008	0.009
3	12	0.106	0.019	0.016	0.012	0.012	0.011
4	16	0.132	0.023	0.018	0.015	0.013	0.013
5	20	0.173	0.044	0.024	0.018	0.018	0.017

Table 1: illustrate the weight loss measurements for mild steel as it degrades in 1 mole of suphuric acid in the absent of inhibitor and the mild steel resistance to corrosion in the presence of inhibitor (Newbouldia leavis leaf extracts).

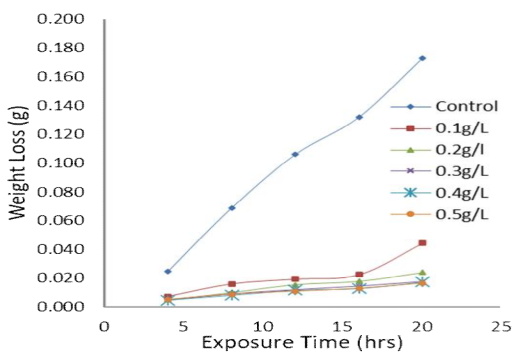

Figure 1: Plot of weight loss versus Exposure time

Figure 1: shows weight loss versus exposure time for the corrosion of mild steel in 1.0M H_2SO_4 in the absence and presence of different concentrations of Newbouldia Leavis leaf extract. From the plots, the rate of weight loss in the controlled environment (1 mole of H_2SO_4) was progressively increasing over time, this was observed for 20hrs at 4hrs intervals, but when different concentrations of leaf extract was introduced there was remarkable decline in the weight loss according to the various concentrations of the inhibitor which shows that Newbouldia Leavis leaf extract is a potential good inhibitor. The reduction in weight loss in the presence of the inhibitor was due to the adsorption of the constituents in the extract on the surface of mild steel sample.

Table 2: Corrosion rate values for mild steel in 1 mole of sulphuric acid in the absence and presence of *Newbouldia leavis* leaves extract.

Time (t) of exposure (hrs.)	Corrosion rates for control experiment and different concentration of Newbouldia leavis leaves extraction in corrosive media.					
	Control (0.0g/L)	0.1 g/L	0.2 g/L	0.3 g/L	0.4 g/L	0.5 g/L
4	31	8.7	6.2	6.2	6.2	6.2
8	43	10	6.2	5.6	5.0	5.6
12	44	8	6.6	5.4	5.0	4.5
16	41	7	5.6	4.6	4.0	4.0
20	43	11	6.0	4.7	4.5	4.2

Table 2: shows the corrosion rate values when there was no inhibition. The corrosion rates were seen to be increasing as exposure time increases, but when leaf extract was added there was noteworthy drop of corrosion rate.

The control values in Table 2 above shows that there was passivation within the first four hours of exposure of the metals, before the metal resistance to passivation, the resistance to corrosion observed between the hours of 8 to 16 was as a consequence of films build up on the surface of the test piece, It is not to be believed that corrosion has stopped, it is only delayed. Due to the aggressive corrosive nature of the environment, the films soon gave in to further corrosion after 16 hours. The corrosion rate values of the control experiment do not show a distinct pattern, but reduces uniformly over time for inhibited samples. The results also depict that the mild steel metal degrades slowly over time.

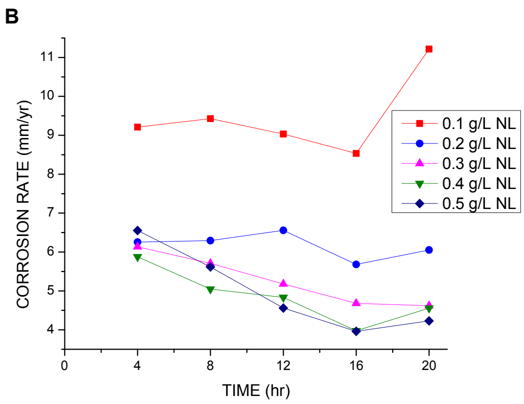

Figure 2: Plot of Corrosion rate (mm/yr.) (for various concentrations) versus Exposure time (t).

Figure 2: is an elaborate plot of the inhibited corrosion curves. At concentration of 0.1g/L, the corrosion rate showed a uniform decline from 0 to 16 hours, progressively reducing, evidencing that the leaf extract has formed a protective layer on the surface of the test piece thereby inhibiting the mass transfer of charges in the corrosive environment. But during the period between 16 to 20 hours there was an upsurge in the corrosion rate from about 7 to 11 mm/year, this observation was an indication that concentration 0.1g/L of the leaf extract has been completely consumed and the film initially formed on the surface of the test

piece succumb to the corrosive environment.

0.2g/L concentration exhibited a corrosion rate gradually increasing from 0 to 12 hours, before a sharp drop from 12 to 16 hours; this drip was as an upshot of increased in concentration from 0.1 to 0.2g/L, towards the aging of 20 hours, there was noticeable increase in corrosion signifying that the concentration of leaf extract was depleting in the corrosive environment.
From the plot, it can be seen that leaf extract concentrations from 0.3 to 0.5g/L demonstrated similar trends to the concentration of 0.2g/L.
The graph shows that Newbouldia leavis leaf extract has great influence against acidic corrosion of mild steel. The inhibition reaction continued until after the 16th hour of exposure where it can be seen to lose resistance against sulphuric acid. This characteristic is complimentary to the controlled experiment where the reduction in corrosion rate indicated that the metal rate of corrosion lost it resistance to the acid.

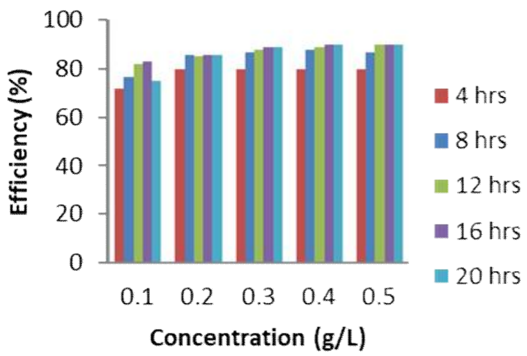

Figure 3: Percentage Efficiency versus Concentration of inhibitor (g/L)

Figure 3: shows the inhibition efficiency of the inhibitor plotted against the concentration of the plant extract. It can be seen that Newbouldia leavis leaf extract is a good inhibitor of mild steel corrosion in sulphuric acid. The inhibition efficiency increased from 72% to 90% with increase in inhibitor concentration.

4. CONCLUSION

In the light of the evidence gathered during the course of investigating the inhibitive properties of Newbouldia Leavis Leaf extract on the Corrosion of Mild Steel in Sulphuric Acid, the following conclusion can be drawn:

i. In the absence of Newbouldia leavis leaves extract in 1 mole of H2SO4 solution, gravimetric method reveals aggressive deterioration of the mild steel coupon immersed in the corrosive environment, the corrosion rate and weight loss increase drastically showing that the active site of the test piece was bare to acid attack.
ii. The addition of Newbouldia leavis leaf extract to 1 mole of H_2SO_4 solution reduces the corrosion rate of mild steel in the acid as revealed by the weight loss measurement. The inhibition efficiency increases with increase in concentration of the plant extracts.
iii. Mild steel was protected from corrosion when the plant extract was introduced by adsorption of compound of the extracts on the metal surface thereby causing a barrier for charge and mass transfer making the metal less susceptible to corrosion reaction.
iv. The findings show that Newbouldia leavis leaf extract is a very good corrosion inhibitor for mild steel in sulphuric acid (H_2SO_4).

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 researchers recommend that the need to grow Newbouldia leavis 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 Newbouldia leavis leaf extracts on mild steel in sodium chloride (NaCl) should be carried out.

iv. To investigate the effect of temperature on Newbouldia leavis leaf extracts as a corrosion inhibitor of mild steel in corrosive acid media.

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