

Study of Corrosion Inhibitive Behaviour of *Moringa Oleifera* Leaf Extract on Mild Steel

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ABSTRACT

The inhibiting effect of *Moringa oleifera* (MO) leaves extract on the corrosion of mild steel in 1.0 M Hydrochloric acid (HCl) has been investigated using gravimetric and electrochemical techniques. Gravimetric study for mild steel presented the best average inhibition at 0.3g/mol concentration, and decreased with increase in concentration. The application reduced weight loss of mild steel from 0.1850g to 0.0095g when inhibited at 0.3 g/mol concentration. Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization (PDP) measurements were also employed in this study. The Nyquist plots of mild steel was determined for different concentrations of *Moringa oleifera* leaf extract. It confirmed the inhibitive nature of *Moringa oleifera* leaves extract on the corrosion of mild steel as the corrosion current density decreased with increase in concentration of the extract. Furthermore, the potentiodynamic polarization measurement depicted *Moringa oleifera* to be a mixed-inhibitor for mild steel. Each technique proved that *Moringa oleifera* leaves extract is a good inhibitor of mild steel corrosion in acidic medium. Implementation of this research findings will reduce drastically the wastage of mild steel metal products in industries.

KEYWORDS: Mild steel, *Moringa oleifera*, Electrochemical analysis, Corrosion rate, Gravimetric analysis.

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1. INTRODUCTION

The corrosion scourge is globally recognized as one of the most serious problems in modern societies and has many serious economic, health, safety, technological, and cultural consequences.

Corrosion environments lead to compromise of structural integrity of materials resulting in damage, which leads to costly repairs and replacement as well as devastation of the environment. Corrosion is worth investigating, because corrosion problems represent a large portion of the total costs for oil and gas producing companies every year worldwide (Omotosho, 2016). Corrosion have been manifesting in the production sector and they have been either general or localized (Finšgar & Jackson, 2014). Corrosion involves the gradual destruction of materials by chemical reaction with the environment, this is more common in metal (Umoren *et al.*, 2011). Degradation is often fast in metals which starts with the destruction of the protective layer and then reactions that change the composition and performance of the metal as well as its direct environment. This activity thus leads to formation of oxides, metalcation, change in pH, and electrochemical potential.

In the manufacturing and production sectors, degradation of metallic materials has become a major factor affecting the reliability and availability of systems. The survival of industrial facilities will affect the effectiveness of the sector and the national economy in general. However, because a large majority of these installations which their components are made of carbon steel which are inevitably susceptible to corrosion or degradation (Omotosho, 2016). Often times, failures of these critical systems lead to wastages in terms of spillages which cause losses to the industry and overall risk to human and

environment. The system failure may lead to more losses in the area of compensation for damages. Thus, to curb these avoidable distractions in maximizing output, monitoring and inspection of these systems are very necessary. However, there can be prolonged or eliminated monitoring and inspection duration through the incorporation of good corrosion protection approaches. Moreover, these techniques will reduce corrosion rate and by extension prolong inspection or monitoring time (Omotosho, 2016).

Corrosion control can be achieved by many methods (Terence, 2018). However, the use of corrosion inhibitors is actually the most practical method used in industries and academic studies to secure metals and its alloys from aggressive environment (Beda *et al.*, 2017). It is worth noting that majority of the corrosion inhibitors in commercial scale that are utilized in industries are multi-component inhibitor consisting of nitrogen and sulfur functionalities. These inhibitors are stable and effective in corrosive environments, they can be expensive to formulate and can pose threat to public health and the environment because of their toxicity (Brycki *et al.*, 2018). Moreover, accompanied by other heavy metals, chromates, and phosphates, multi-component inhibitor systems are persistent and require expensive processes for their removal (Abbas *et al.*, 2014).

Increasing scientific research has been steered towards developing corrosion inhibitors from natural, green products because of the difficulties surrounding commercial corrosion inhibitors. These organic corrosion inhibitors were developed because of the positive characteristics exhibited. They include having wide temperature range, compatibility with protected materials, good water solubility ability, minimal costs and relatively low toxicity (Brycki *et al.*, 2018). Hence, these green products have the adsorptive capability on material

surface thereby forming protective film which displace water and protect the surface against degradation.

Moringa oleifera (MO) is one of the many leaf extracts that have gained interest in corrosion of engineering materials (Nnanna & Owate, 2014; Imohiosen *et al.*, 2014; Allaoui *et al.*, 2017; Aribo *et al.*, 2017). In each case, *Moringa oleifera* had shown positive response to the inhibition of metal corrosion. Hence, this research is aimed at investigating the effectiveness of moringa leaves extract as a natural occurring corrosion inhibitor on the corrosion of mild steel in 1M Hydrochloric acid (HCl). In addition, the assessment on the suitability of *Moringa oleifera* as green inhibitor for Mild steel corrosion in HCl was done using gravimetric and Electrochemical Measurements. The findings of this research are expected to assist in effective management of corrosion to ensure long term system integrity.

2. MATERIALS AND METHODS

The materials used in this work are: Mild steel series SAE1015, *Moringa oleifera* leaves, refluxing apparatus, volumetric flasks, beakers, measuring cylinder, silicon-emery paper (grade nos. 200, 400), digital weighing balance, thread, filter paper, Vernier caliper, micrometer screw gauge, desiccators, Hydrochloric acid, acetone, ethanol and distilled water.

2.1 Preparation of the Mild steel specimen and Corrosive Media

In order to conduct the weight loss experiment, the metals were cut into 2 cm by 2 cm sizes using the guillotine and snip. They were thereafter subjected to chemical treatments. The mild steel sample was degreased by immersion in acetone and then dried. After that it was immersed in a solution of HCl with acid to water ratio of 1: 4 for 30 seconds at room temperature to completely remove the rust and dried with clean cloth and stored in a

desiccator. The environment utilized in this study is HCl at concentration of 1M.

2.2 Preparation of the Plant Extract and Corrosion inhibition process

The procedure for the preparation of the leaf extracts is according to the process described by Nnanna and Owate (2014). To prepare the extract, *Moringa oleifera* leaves were obtained in their fresh forms and subjected to drying under aeration. This process was undertaken to ensure that the leaves retain their natural properties. This was followed by grinding using a grinding machine to fine particle grain size. The blank corrodant was 1.0M of HCL solution. Stock solution of the plant extract were prepared by boiling weighed amounts of dried and grounded leaves of Moringa under reflux for 3 hours in 1.0M HCL solutions. The solutions obtained after this process were cooled and filtered afterwards. The quantity of substance extracted into solution was gotten by subtracting the mass of the dried residue from the initial mass of the dried plant material before extraction. From the respective stock solutions inhibitor test solutions were prepared in the concentration range of 0.1 - 0.5 mg/l in the given corrodant.

The mild steel coupons were dipped in five (5) different concentrations of the inhibitor with a set of dipping (the blank) without any inhibitor added. They were dipped at two (2) hours intervals over 10 hours. They were also quenched with the same method as used in the cleaning process.



Figure 1 Coupons exposed to corrosion and inhibition at different concentrations

2.3 Electrochemical Analysis

Further study was conducted to investigate the inhibitive effect of the extract using Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization.

2.3.1 Electrochemical Impedance Spectroscopy (EIS)

Electrochemical Impedance spectroscopy (EIS) is a useful technique for characterizing a wide range of electrochemical systems. It also aids in the determination of the contribution of electrode or electrolytic processes in such systems. In electrochemical impedance spectroscopy, the equation

$$1 - \frac{R_{ct\text{uninhibited}}}{R_{ct\text{inhibited}}} \times 100\% \quad (1)$$

(Singh *et al.*, 2015)

was used to calculate inhibition efficiency for EIS.

To calculate I_{corr} and E_{corr} for the electrochemical analysis the following equations were used

$$CR = \frac{Z_{corr} \times Eq \times 10 \times 3.15 \times 10^7}{F \rho} \quad (2)$$

$$Fq = \frac{CR \times F \times q}{Z_{corr} \times 10 \times 3.15 \times 10^7}$$

(3)

(Singh *et al.*, 2015)

Where $CR = 3.7\text{mm/yr}$; $\rho = 7.85\text{g/cm}^3$

2.3.2 Potentiodynamic Polarization

The corrosion behavior of metals was evaluated using potentiodynamic polarization curves. Alternatively, the process can be called potential sweep, cyclic voltammetry or linear sweep voltammetry. The cyclic sweep test was conducted using appropriate range with adequate sweep rate. The sweep rate determines the linearity of the polarization curves. The cathodic portion and anodic portion of the Tafel plot was scanned and the cathodic and anodic mechanisms obtained. The extrapolation of the Tafel straight line determines the corrosion current density (I_{corr}) (Singh *et al.*, 2015). The inhibition efficiency was calculated using:

$$IE\% = \frac{I_{corr(Blank)} - I_{corr(Inhibited)}}{I_{corr(Blank)}} \quad (4)$$

(Singh *et al.*, 2015)

3. RESULTS AND DISCUSSION

3.1 Gravimetric Measurements

The corrosion rate versus time graph for the control experiment for mild steel is shown in Figure 2.

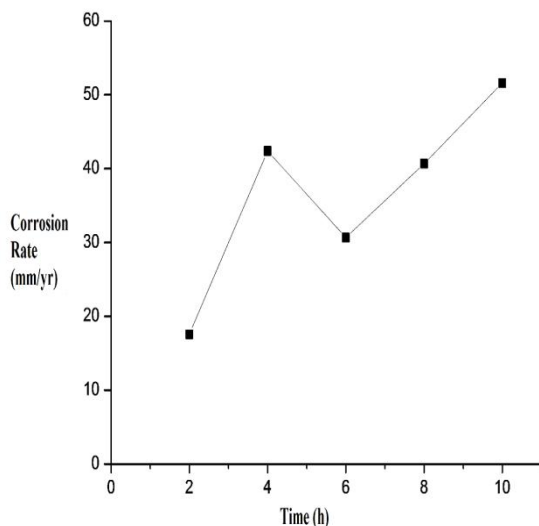


Figure 2 Corrosion rate plot of mild steel in 1M HCl in the absence of *Moringa oleifera*

It was observed that there was rapid corrosion within the first four hours of exposure of the metals. Between the fourth and the sixth hour there was passivation. After the sixth hour, it lost resistance to corrosion and corroded now at a uniform rate. The corrosion rate values for mild steel increased significantly uniform after the resistance. The result also depicts that the mild steel metal degrades slowly over time.

The corrosion rate vs time graph for the control experiments for mild steel was compared to the inhibited corrosion experiments in Figure 3. The graph showed that there was great corrosion inhibition reaction on the metals with introduction of *Moringa oleifera*.

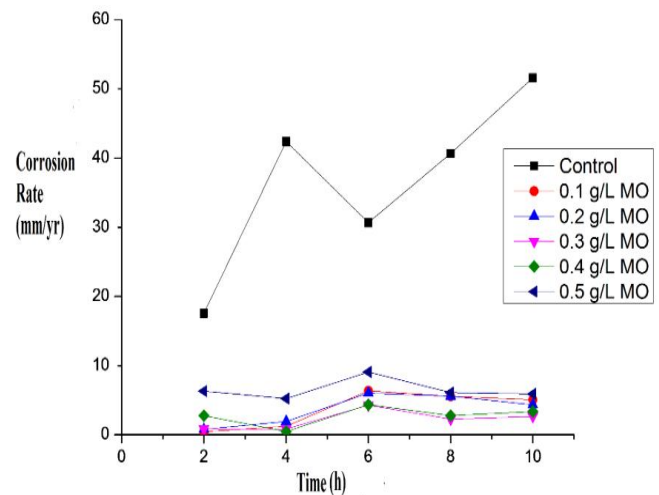


Figure 3 Corrosion rate plots of mild steel in 1M HCl in the absence and presence of *Moringa oleifera*

In mild steel corrosion, the pattern of degradation of the metal is generally uniform and low, with little passivation effect within the second hour unlike in the control experiment.

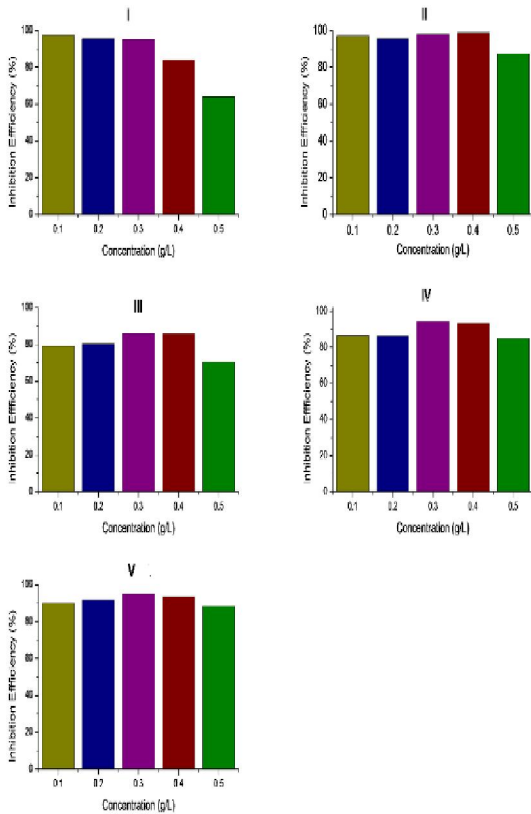


Figure 4 Inhibition efficiency plots of mild steel in 1M HCl in the presence of *Moringa oleifera* for five concentrations (0.1-0.5g/mol)

Inhibition efficiency plots for each concentration of *Moringa oleifera* for mild steel is shown in Fig 4. The plot depicted that the inhibitor had higher efficiency at 0.3g/mol concentration, and decreased with increase in concentration. The fifth concentration was least efficient with the third concentration having the best average inhibition efficiency.

When compared side by side Fig 3 with Fig 4, it is noticed that there is an indirect relationship between corrosion rate data and inhibition efficiency. It can be deduced that in a given corrosion-inhibitor medium, the higher the corrosion rate values the lower the efficiency (Omotosh, 2016).

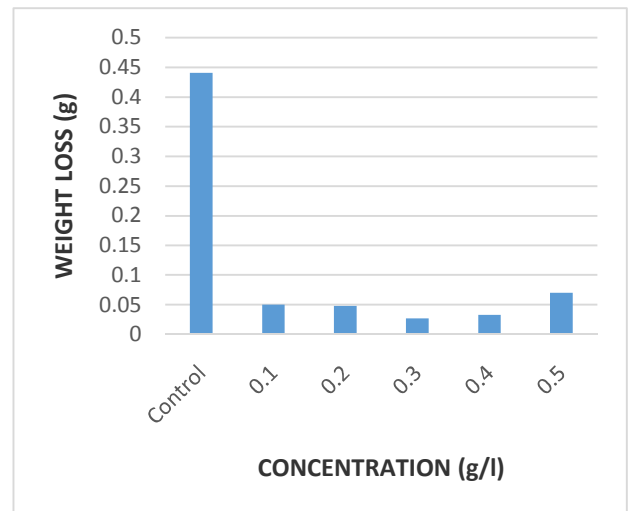


Figure 5 Mild Steel Weight Loss against Concentration

The graphical illustration of weight loss effect with concentration and time is as presented in Fig 5 for mild steel. From the graphs, it is observed that at 0.3g/mol concentration mild steel samples showed good resistance to corrosion over the investigation time. It can be inferred that *Moringa oleifera* is a good inhibitor for mild steel corrosion.

3.2 Electrochemical Impedance Spectrometry

The Nyquist plots of mild steel for different concentrations of *Moringa oleifera* leaf extract was obtained, as shown in Fig 6 by Electrochemical Impedance Spectroscopy (EIS) method. The Nyquist plots contain semi-circle and with inhibitor concentration, the size increases. It was observed that there is single semicircle which indicated that steel dissolution is made up of a single process of charge transfer which is not compromised after addition of inhibitors molecules. Again, the high frequencies inductive loop showed that there is relaxation of adsorbed species at metal-corrosive solution interface while the typical semicircle with a slight depression and a center located below the real axis presents inhomogenities of solid

electrodes during corrosion, like some degree of roughness and/or others.

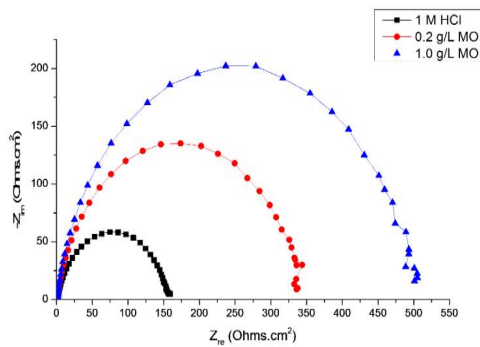


Figure 6 Electrochemical impedance curve for Mild steel in 1M HCl in the absence and presence of *Moringa oleifera* leaf extract

From the plot it can be deduced that inhibition increased at first as concentration increases but later lost resistance with further increase in concentration thus the inconsistency towards the end of the graph. In addition, the Nyquist curve showed system stability for the concentrations utilized where the curve counters the real part of the impedance axis.

3.3 Potentiodynamic Polarization (PDP) Measurements

The PDP plot was presented in Fig 7. It shows the anodic and cathodic polarization curve, for mild steel in 1M HCl. For mild steel, there is no much difference between the anodic and cathodic tafel slopes, therefore the inhibitor is of mixed-type-cathodic and anodic type inhibitor of mild steel. As corrosion current density decreased with increase in concentration of the extract which is due to the increase in blocked fractions of the metal surface by adsorption. The shift in the tafel slope of both the cathodic and anodic reactions as shown in the figure in the presence and absence of the extract suggest that the inhibition affects both the cathodic and anodic reactions. The inhibition efficiency increased with extract concentration

increase while the corrosion current density decreased. From this observation, it is submitted that the extract reduced the reactions at both the cathodic and anodic sites. This is due to the coverage of these sites by the molecules of these extracts. It can also be observed that as the concentration of the extract increased, there appears to be a shift towards more negative potentials indicating that *moringa oleifera* acts as a good corrosion inhibitor.

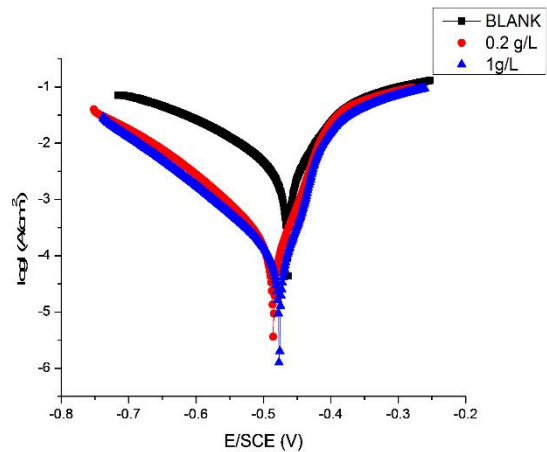


Figure 7 Potentiodynamic polarization curves of mild steel in 1 M HCl in the absence and presence of *Moringa oleifera*

Similar results as obtained in Fig 6 and Fig 7 on the inhibition effect and adsorption behavior of the inhibitor on Mild Steel were also in agreement with what was reported by Preethi *et al* (2014); Chuan *et al* (2017) and Chengduan *et al* (2018) in their works on corrosion inhibition of Mild Steel using various inhibiting materials.

4. CONCLUSION

The investigation on the inhibitive effect of *Moringa oleifera* leaf extract on the corrosion of mild steel was undertaken in this study. The gravimetric analysis showed that *Moringa oleifera* is a good corrosion inhibitor for mild steel corrosion. Weight loss analysis showed that mild steel had weight loss of 0.1850g for control against 0.0095g when inhibited at 0.3 g/mol concentration. The

electrochemical impedance measurements confirmed the inhibitive nature of *Moringa oleifera* leaves extract on the corrosion of mild steel. The potentiodynamic polarization measurement also depicted *Moringa oleifera* to be a mixed-inhibitor for mild steel. Thus, the improvement achieved in corrosion inhibition is therefore recommended for effective management of mild steel products.

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