

# Corrosion Inhibition And Characterization Of Moringa Oleifera Leaves Extract (MOLE)

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## Abstract:

**Background:** The inhibitory effect of MOLE leaf extracts on mild steel at varied concentration of the extract and temperature of the acidic medium during corrosion in 2M HCl were studied using both gravimetric and electrochemical (potentiodynamic polarization measurement) techniques.

**Materials and Methods:** The plant materials investigated in this study is Moringa oleifera leaves and it was obtained from the vicinity of Federal Polytechnic, Ado Ekiti, Ekiti State, Nigeria, and it was authenticated at the Department of Biology, Bamidele Olumilua University of Education Science and Technology, Ikere, Ekiti State, Nigeria. The procurement and chemical composition of the mild steel used in this study were done at Universal steel company, Ogba Lagos.

**Results:** The result revealed that as the MOLE concentration increases the corrosion rate of the mild steel decreases but increases with increase in temperature. Electrochemical measurement of the corrosion capability revealed that the extract is a mixed type. Thermodynamic parameters revealed that the mechanism of the adsorption process is physisorption while the kinetics of the reaction follows a first order reaction. The E-core ranges from 420.02 to 456.64 suggesting that the extract is as a mixed inhibitor.

**Conclusion:** The current study's findings showed that MOLE can be applied to mild steel in 2 M HCl as a green corrosion inhibitor.

**Keywords:** Moringa oleifera leaves, Corrosion, Gravimetric method. Electrochemical measurement

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## I. Introduction

The industrial use of acids for chemical cleaning, pickling steel, de-scaling, and acidizing oil wells has encouraged more research into strategies to lessen the financial effects of mild steel corrosion over time. Due to steel's poor resistance to corrosion, particularly in acidic conditions, numerous studies on corrosion protection strategies have been conducted. The discovery of synthetic chemicals as efficient corrosion inhibitors was the result of unrelenting study. These artificial inhibitors effectively shielded metals in various media, but it was quickly shown that they posed a short- and long-term environmental harm. This revelation, together with their high cost and the growing interest in "going green" and safeguarding the environment, prompted more research into corrosion inhibition techniques that are safe for the environment. This resulted in the identification of natural origin inhibitors, or "green inhibitors" [1] [2] [3] [4] [5] [6] [7]. Numerous plant extracts and essential oils have been studied in a variety of environments as corrosion inhibitors [8] [9] [10] [11] [12] [13] [14] [15]. Through adsorption of the molecules on the metal surface, the presence of hetero-atoms (S, N, and O) with free electron pairs, -electrons, high molecular weight alkyl chains, and substituent groups enhances their inhibitory efficiency and function, forming a barrier between the metal and aggressive corrosion agents. [16] [11][17].

*Moringa oleifera* is a multipurpose tree with most of its parts being useful for a number of applications. It is generally used in a number of developing countries as a vegetable, medical plant and a source of vegetable oil. It has an impressive range of medicinal uses with high nutritional value. [18]. On the other hand, Moringa oleifera plants have been found to be a natural coagulant, flocculants, softener, disinfectant, and sludge conditioner. [19] [20] [21].

## II. Materials And Methods

### Mild Steel

The elemental composition of the mild steel samples used was as follows: C-0.215, Si-0.258, Mn-0.467, S-0.009, Cr-0.021, Ni-0.001, Cu-0.017, Ti-0.019 and balance Fe. Mild steel coupons cut into 19×16×5 mm were used for gravimetric measurements whereas coupons of size 3×1×0.1 cm with 1cm<sup>2</sup> exposed surface areas were used as working electrode for the polarization measurement. Prior to the experiments, the specimens were abraded

with a series of emery paper and then washed with distilled water and ethanol before immersion in the corrosive medium.

**Test Solution**

The test solution (2 M HCl ) was prepared from analytical grade HCl.

**Extract Preparation**

*Moringa oleifera* leaves were obtained at the vicinity of Federal Polytechnic Ado Ekiti, Ekiti State, Nigeria, cleaned with tap water to wash off dirt, dried for ten days in open air and pulverized. 100 g of the pulverized sample was soaked in 1 L of 98% ethanol for 72 hours after which the mixture was filtered. The filtrate was evaporated to dryness over a thermostat water bath at 90 °C. The dried extracts (MOLE) obtained were used to prepare different extract concentrations ranging from 0.2 to 1 g per 100 mL of 2 M HCl.

**Weight loss Measurement**

For four hours at room temperature, pre-weighed mild steel samples were submerged in 100 mL of 2 M HCl with and without varying MOLE concentrations. After being removed, the specimens were cleaned with distilled water, dried, and weighed. Weight loss was determined as the difference in weight between the pre- and post-immersion values. For excellent reproducibility, the experiment was run in triplicate.

Equation 1 was used to compute the corrosion rate

$$CR = \frac{\Delta W}{AT} \dots\dots\dots 1$$

Where t is the immersion period, A is the specimen's entire area, and Δw is the average weight loss.

Equation 2 was utilized to calculate inhibition efficiency

$$IE = 1 - \frac{CR_2}{CR_1} \times 100 \dots\dots\dots 2$$

Where CR<sub>1</sub> and CR<sub>2</sub> are the corrosion rates of the mild steel strip coupons in absence and presence of MOLE.

**Temperature Variation**

A thermostat-controlled water bath was used to maintain the temperature of the acidic solution for 303, 313, 323, and 333 K for three hours while pre-weighed coupons were submerged in 100 mL of 2 M HCl with and without different extract concentrations ranging from 0.2 to 1.0g/100 mL. The coupons were taken out, cleaned, dried, and weighed after being rinsed in distilled water. From the acquired data, which were also fitted into several isotherms, thermodynamic parameters were computed.

**Kinetic Study**

Pre-weighed coupons were submerged in 100 mL of the acidic solution with and without different extract concentration for 168 hours in order to examine mass loss and reaction sequence. The coupons were taken out, cleaned, degreased, dried, and weighed gradually every 24 hours. Equations 3 and 4, were respectively used to determine the order of reaction and half-life based on the observed results.

$$\ln(W_i - W_L) = -k_1 t + \ln W_L \dots\dots\dots 3$$

$$t_{1/2} = \frac{0.693}{k} \dots\dots\dots 4$$

where t is the immersion duration, k is the rate constant, and W<sub>1</sub> and W<sub>2</sub> are the starting and final weights of the coupons.

**III. Result And Discursion**

**Gravimetric measurements**

**Effect of the extract concentrations on corrosion rate and inhibition efficiency**

The effects of various MOLE concentrations on mild steel in a 2 M HCl solution are displayed in Fig. 1. The findings demonstrated that, at normal temperature, the rate at which mild steel corrodes in an acidic medium decreases as the concentration of the extract increases. The reason for this inhibitive behavior is that when the inhibitor's concentration rises, so does its adsorption coverage on the steel surface [22] [23]. [24] noted similar outcomes. According to the authors, a higher concentration of extract causes more extracts to adsorb on mild steel, preventing further corrosion and providing a barrier for mass transfer [25]. Therefore, the formation of a protective layer on the mild steel surface, which thickens with increasing extract concentration, may be the cause of the decrease in the mild steel's corrosion rate with increasing inhibitor concentration [25] [26].

Fig. 2 illustrates the impact of different extract concentration on inhibitory efficiency. The findings indicated that when the extract concentration increases, so does the inhibition efficiency. This can be explained by the fact that when a larger dosage of the extract was available in the medium, this will increased the proportion of mild steel's surface covered by the extract's adsorbed molecules [27] [28]. This further suggests that the process of adsorption between the inhibitor/adsorbate and the mild steel surface was effectively completed, resulting in

the development of a robust metal-inhibitor relationship and a reduction in the ability of the chloride ion (Cl<sup>-</sup>) to adsorb on the mild steel surface [29] [30] [31].

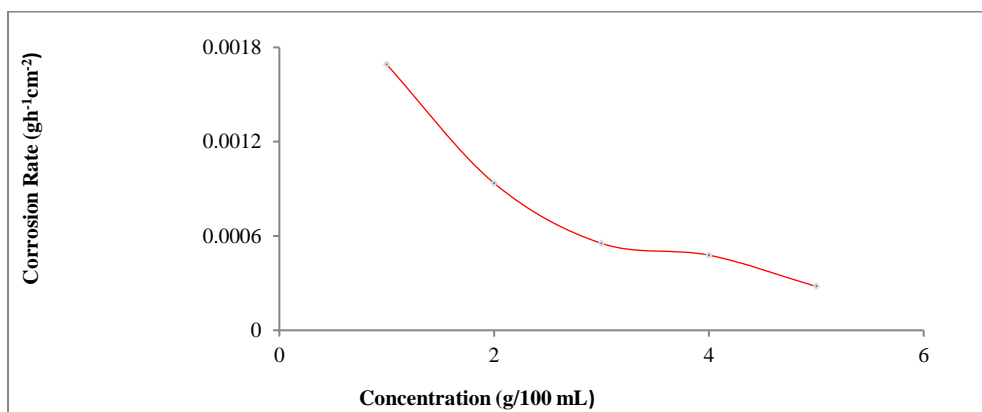


Figure 1: Effect of Concentration of MOLE on the Corrosion Rate of Mild Steel in 2 M HCl

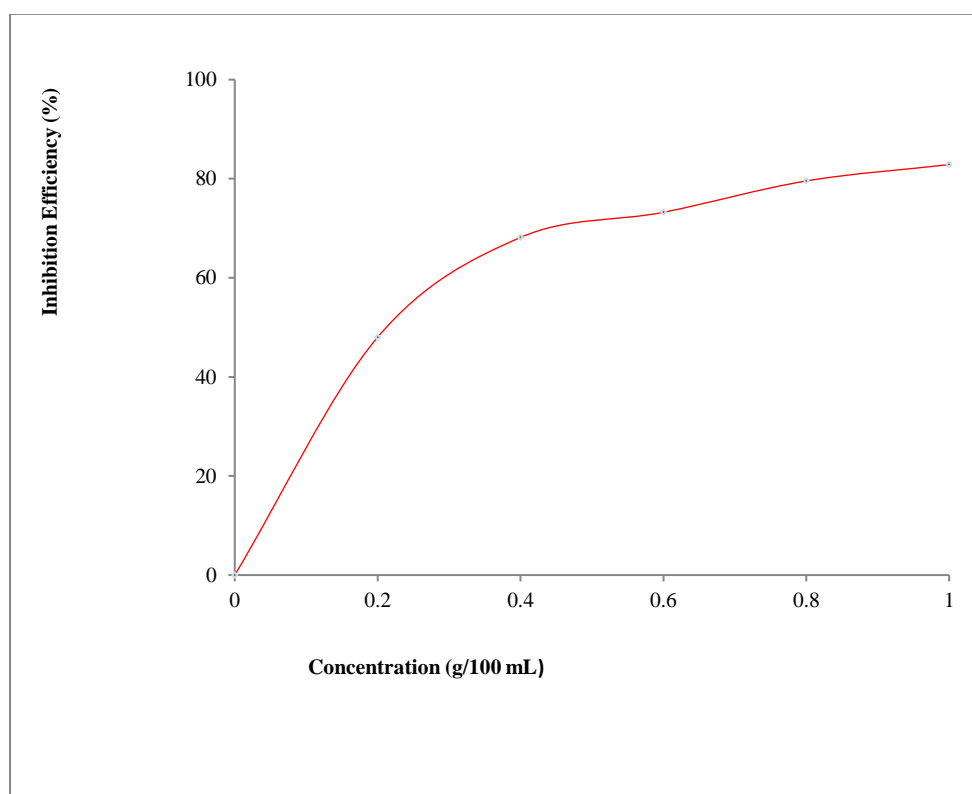


Fig. 2. Effect of Concentration of MOLE on the Inhibition Efficiency of Mild Steel in 2 M HCl

Moreover, it was observed that the inhibitory efficiency increases sharply from 0.4 g/100 mL to 1 g/100 mL, while it is relatively low at 48% at 0.2 g/100 mL. At 82.85%, maximum protection was attained. This demonstrates that the concentration of the extract determines its ability to adsorb on mild steel in an acidic media [32] [33]. This suggests that mild steel can be effectively inhibited by MOLE in an acidic medium at room temperature [34] [35].

#### Effect of temperature on corrosion rate and inhibition efficiency

For three hours, the temperature range of 303 to 333 K was used to study the impact of temperature on the corrosion rate and inhibition efficiency of mild steel in the inhibited and uninhibited solution. The results are displayed in Fig. 3. The findings demonstrated that while corrosion rate decreased with increasing extract concentration, it increased with rising temperature. According to Alinnor and Ejimeke [26] (2012), a rise in temperature causes the average kinetic energy of the interacting molecules to increase, which in turn accelerates the rate of mild steel corrosion [26]. Nonetheless, when compared to the blank solutions, the corrosion rate of

inhibited solutions is noticeably lower. The reduction in mild steel corrosion in acidic media can be attributed to the moderating effect of MOLE [27].

In the temperature range of 303 K to 333 K, the impact of temperature on the inhibition efficiencies of the extract in the inhibited and uninhibited 2 M HCl solution was also investigated. The outcome is displayed in Fig. 4. It is evident that the inhibitory efficacy increases with an increase in extract concentration but decreases with an increase in temperature, from 303 K to 333 K. The physical adsorption of the inhibitor molecule on the mild steel surface provides an explanation for the decrease in inhibition efficacy observed with temperature rise. As temperature rises, physical adsorption experiences a decrease in adsorption [28] [36]. Previous studies have demonstrated that an inhibitor's inhibition efficacy rises with temperature rise for a chemical adsorption mechanism (chemisorption) whereas it decreases for a physical adsorption mechanism (physisorption) [32] [37].

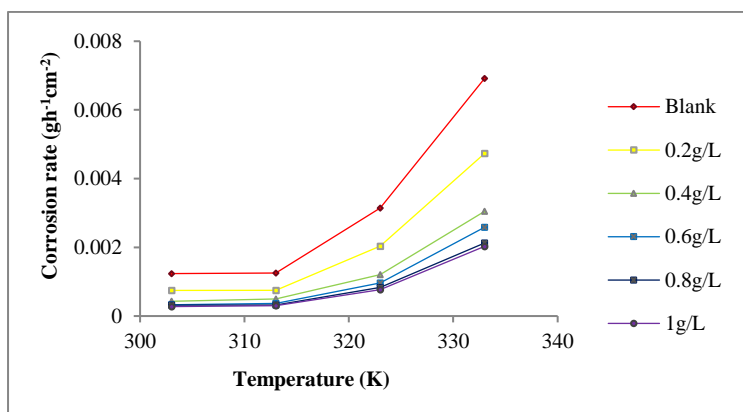


Fig. 3. Variation of Corrosion Rate with Temperature for Mild Steel in 2 M HCl in the Presence and Absence of MOLE at 303 K – 333 K

**Thermodynamic Studies**

Table 1 contains the values of Ea that were determined using the Arrhenius plot's slope. The inhibited solution had higher Ea values than the uninhibited one. This implies that the extract physically blocks the charge and mass transfer electrons, acting as an efficient inhibitor [25] [38]. Additionally, Ea increases with concentration when the extract is present. This illustrates how the inhibitive property is concentration dependant. Lower activation energy values in solutions containing inhibitors, according to earlier researchers, point to a particular kind of adsorption [31] [39]. Ea values that are higher when MOLE concentration is present (54.24 - 59.44 kJ/mol) than when it is absent (50.47 kJ/mol) provide additional support for the idea that physisorption is the mode of the extract adsorption [29].

The following formula was used to determine the values of additional thermodynamic parameters of the corrosion process, such as enthalpy (ΔH) and entropy (ΔS):

$$CR = \frac{RT}{Nh} \exp\left\{\frac{\Delta S}{R}\right\} \exp\left\{\frac{-\Delta H}{RT}\right\} \dots\dots\dots 5$$

where T is the absolute temperature, R is the universal gas constant, h is the Planck's constant, N is Avogadro's number, ΔS is the entropy of activation, and ΔH is the enthalpy of activation. The determined ΔH and ΔS values in the presence and absence of the MOLE are presented in Table 1

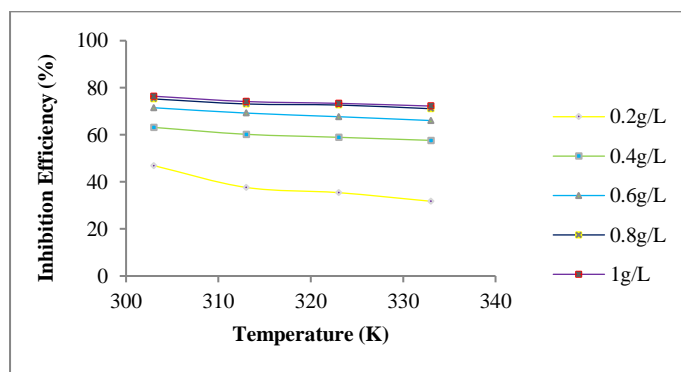


Fig. 4. Variation of Inhibition Efficiency with Temperature for Mild Steel in 2 M HCl in the presence of MOLE at 303 K – 333 K

According previous researchers, there should be a similar variation in the values of  $E_a$  and  $\Delta H$  [36]. This is noticed in the calculated values of the extracts. Furthermore, the endothermic nature of the reaction process is indicated by positive values of  $\Delta H$  in both the extracts' presence and absence [24]. This indicates that mild steel dissolves slowly in the presence of the extract [40].

But in the presence and absent of the extract, the values of  $\Delta S$  are negative. This indicates that the extract molecules were adsorbed onto the mild steel surface in a systematic manner [32].

**Table 1: Calculated Values of Activation Parameters for Mild Steel Corrosion in 2 M HCl in the Absence and Presence of Different Concentrations of MOLE**

C (g/100mL)	$E_a$ (kJ/mol)	$\Delta H$ (kJ/mol)	$\Delta S$ (kJ/mol/K)
Blank	50.4738	46.3418	-2.6666
0.2	54.2439	48.2872	-2.8156
0.4	56.1662	53.5258	-3.4187
0.6	59.4480	56.8096	-3.8529
0.8	55.1610	51.1937	-2.8946
1.0	57.4414	54.8010	-3.4319

**Adsorption Isotherms**

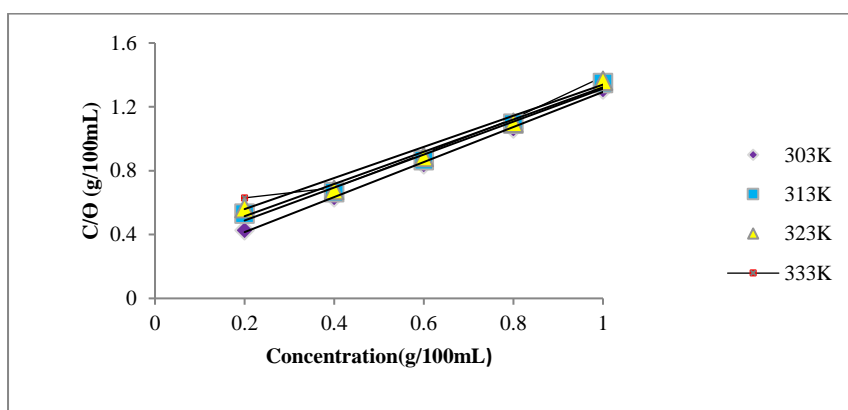
Adsorption isotherms offer valuable insights into how adsorbed molecules interact with the electrode surface [33] [41]. Studies on corrosion have made use of a number of adsorption isotherms. For these investigations, the Langmuir, Temkin, and Freundlich adsorption isotherms were employed. The Langmuir adsorption isotherm can be represented by the following equation [25] [42] [43] [44] [45].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \dots\dots\dots 6$$

where  $\theta$  is the inhibitor's degree of surface coverage, C is the inhibitor concentration, and  $K_{ads}$  is the adsorption equilibrium constant.

Where C is the inhibitor concentration,  $K_{ads}$  is the adsorption equilibrium constant and  $\theta$  is the degree of surface coverage of the inhibitor.

Fig. 5 showed the plot of  $\frac{C}{\theta}$  against C for extract. Fitted straight lines were obtained from the plots and its parameters are listed in Table 2. The correlation coefficient ( $R^2$ ) value is close to unity. This confirms that adsorption of the molecule of MOLE on mild steel in the acidic medium obeys the Langmuir adsorption isotherm [46].



**Fig. 5. Langmuir Adsorption Isotherm Plot for MOLE at 303 - 333 K**

The free energy of adsorption  $\Delta G_{ads}$  were calculated from the equation:

$$\Delta G_{ads} = -RT \ln(K_{ads} \times 55.5) \dots\dots\dots 7$$

where T is the temperature, R is the universal gas constant, and the concentration of water is 55.5. Table 2 demonstrates that the adsorption process is spontaneous and the adsorbed layer on the electrode surface is stable when  $\Delta G_{ads}$  is negative [29] [47].

More negative values than -40 kJ/mol are linked to the sharing or transfer of electrons from the inhibitor molecules to the metal surface to form a coordinate bond (chemisorption), whereas values up to -20 kJ/mol are generally consistent with the electrostatic interaction between the charged molecules and the unlike charged metal (physical adsorption) [28]. The determined values of  $\Delta G_{ads}$ , both in the presence and absence of MOLE, are less than -20 kJ/mol, indicating that physisorption—a physical form of adsorption—is the mode of adsorption [48].

**Table 2. Calculated Parameters of Langmuir Adsorption Isotherms of MOLE**

T (K)	Slope	R <sup>2</sup>	K <sub>ads</sub>	ΔG (kJ/mol)
303	1.0974	0.9985	5.1099	-14.2296
313	1.0324	0.9876	3.5524	-13.7530
323	1.0095	0.9831	3.1949	-13.9074
333	0.9749	0.9667	2.7442	-13.9170

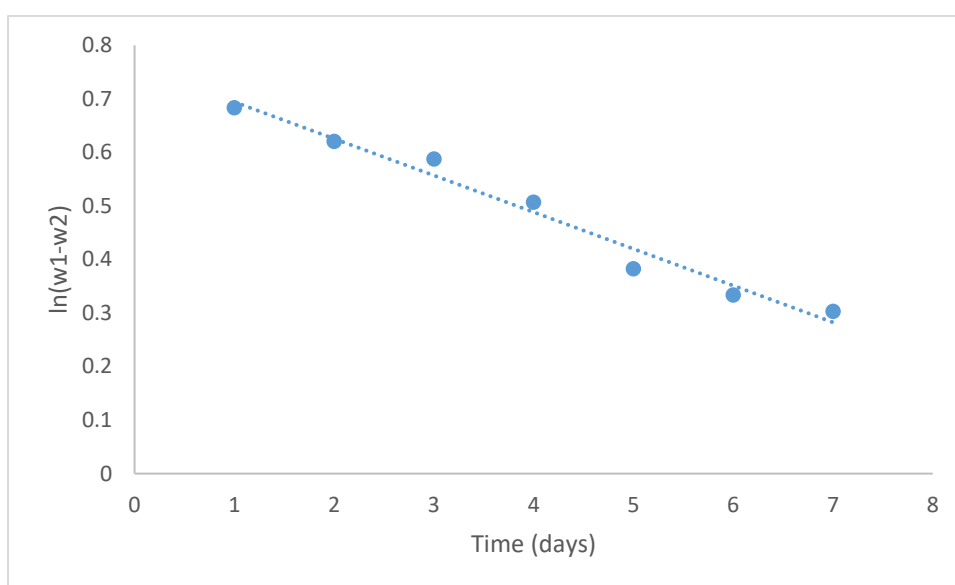
Additionally, the fact that the value of K<sub>ads</sub> falls as temperature rises indicates that adsorption also decreases with temperature, supporting earlier findings about the impact of temperature on the extract's ability to inhibit [25] [31].

**Kinetic Analysis**

Equation below was used to derive rate constants:

$$\ln(W_i - W_L) = -kt + \ln W_i \dots\dots\dots 8$$

Where t is the immersion period, k is the rate constant, W<sub>i</sub> is the metal's starting weight, and W<sub>L</sub> is the weight loss or change at time t [6]. Fig. 6 displays plots of ln(W<sub>i</sub>-W<sub>L</sub>) against periods (days) at room temperature for MOLE.



**Fig. 6 Plot of ln(w1-w2) of MOLE against days at room Temperature**

The plots were subjected to a linear variation, and Table 3 lists the computed parameters for each plot. The plot's slope is smaller than one, confirming a first-order reaction kinetics explanation for the extracts' inhibitory effect [49]. The graph's slope was used to determine the first order rate constant. Equation ix was used to calculate the reaction process' half- life:

$$t_{\frac{1}{2}} = \frac{0.693}{K} \dots\dots\dots 9$$

**Table 3. The Values of Rate Constant (k) and Half-life (t<sub>1/2</sub>) for Mild Steel in 2 M HCl in the Absence and Presence of Different Concentrations of Ethanol Extracts of MOLE**

g/100mL	K(day <sup>-1</sup> )	t <sub>1/2</sub> (days)
Blank	0.0297	23.33
0.2	0.0244	28.39
0.4	0.0226	30.71
0.6	0.0219	31.67
0.8	0.0219	31.67
1	0.0193	35.82

From the result it was observed yhat there is an increase in the half-lives (t<sub>1/2</sub>) of the metals immersed in the 2.0M HCl containing the extract with increase in its concentration. The increase in half-life of the mild steel with increase in concentration of the extract can be ascribed to the decrease in the dissolution rate of the metal in the solution with increase in concentration of the inhibitor

### Study of atomic absorption spectroscopy

Analyzing the concentration of a certain element (the analyte) in a sample is done using atomic absorption spectroscopy. The plot of  $\text{Fe}^{2+}$  dissolution versus MOLE concentration is presented in Figure 7. As the concentration of the extracts increases relative to the uncontrolled solution, there is a discernible drop in the amount of dissolved  $\text{Fe}^{2+}$  in the solution. The extracts' adsorption on the iron filling's surface in the acidic media caused the iron ion to become resistant to the fast oxidation of Fe to  $\text{Fe}^{2+}$ , which is what caused the drop in the concentration of  $\text{Fe}^{2+}$ . This provides more evidence that the adsorption mechanism is concentration-dependent. Decrease in dissolved iron also indicates that *Moringa oleifera* is a powerful inhibitor

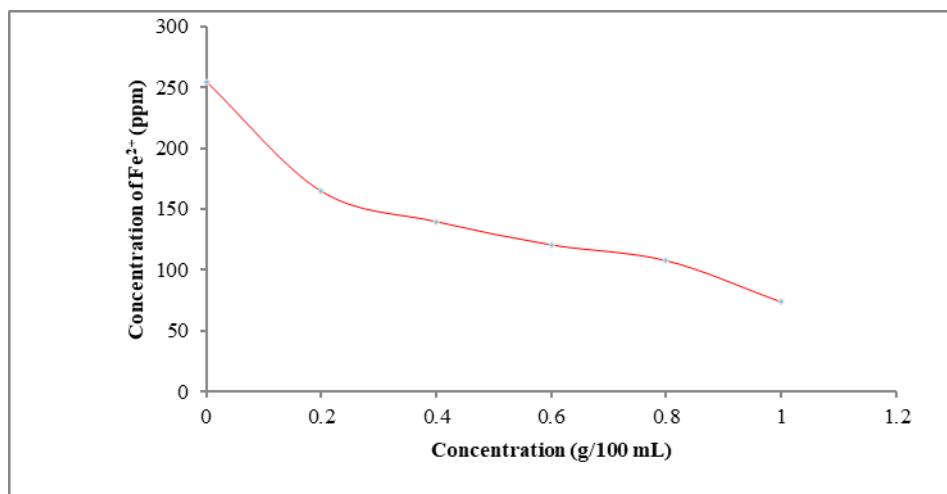


Fig. 7. Plot of Variation of Concentration of  $\text{Fe}^{2+}$  in 2 M HCl in Presence and Absence of Ethanol Extract of MOLE

### Analysis of scanning electron micrographs

Using SEM-EDX, the elemental makeup of the mild steels' surfaces was examined. Plate 1 displays a micrograph of mild steel prior to its immersion in an acidic solution. Plate 1 provides a clear image of the mild steel, and the EDX indicates that the weight percentage of Fe is 89.5 percent. The micrograph and EDX result of mild steel that was immersed in 2 M HCl for 4 hours at room temperature are displayed in Plate 2. When comparing the mild steel that was submerged in the acidic solution to the mild steel that was not, a significant difference was seen. This is because the corrosive solution is attacking the mild steel, causing tiny pits and cracks [50].

According to the edx, the weight percentage of Fe decreased to 62.5%. This demonstrates that the mild steel surface has experienced corrosion, leading to the decrease. The micrograph of mild steel submerged in an acidic solution with 1% (w/v) MOLE is displayed on Plate 3.

There is a discernible improvement in the mild steel's smoothness as compared to Plate 2. According to the EDX result, the weight percentage of Fe has increased to 85.5%. This demonstrates that the mild steel surface has experienced corrosion inhibition [51]. The mild steel that was submerged was also seen to be smoother than the steel that was left in an unrestricted solution. This demonstrates that the formation of a protective layer on the mild steel surface in the presence of MOLE prevents mild steel from corroding in corrosive media. This is the main cause of the mild steel's smooth surface in inhibited solution, which is equivalent to the surface of clean mild steel.

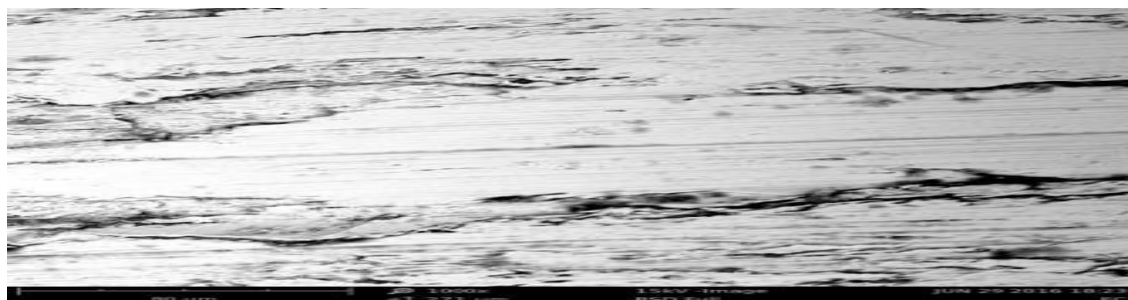


Plate 1. SEM Micrograph of Clean Mild Steel Strip (not immersed in acid)

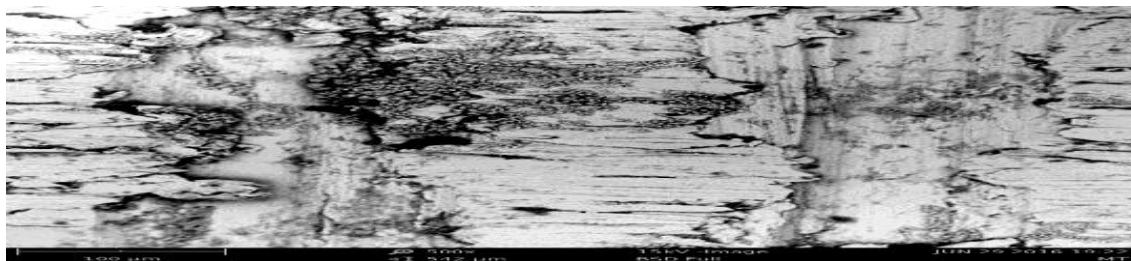


Plate 2. SEM Micrograph of Mild Steel Immersed in 2 M HCl containing 1g/100 mL of Ethanol Extract of *Moringa oleifera* leaves

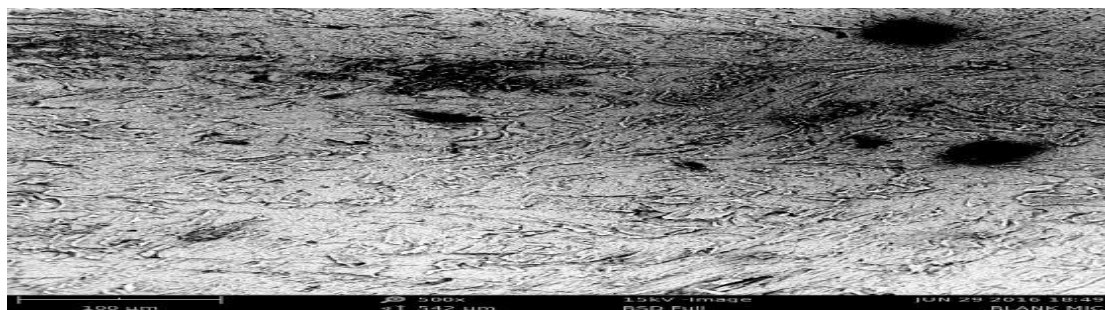


Plate 3. SEM Micrograph of Mild Steel immersed in 2 M HCl (Blank)

Moreso, Fe which is the major component of mild steel sample was found to decrease after immersion in the acidic solution in the absence and presence of the extract (Table 4). From the result, it was observed that the fresh mild steel have the highest composition of Fe, followed by mild steel treated in 1.0g/L of the extract and lastly the mild steel immersed in the acidic solution without the plant extract. The EDX result also corroborate our findings that corrosion rate of mild steel decreases in the presence of the extract.

Table 4: The composition of the mild steel samples regarding the EDX analysis

Sample	Elements					
	Fe	O	C	Si	Cl	Mn
Plate one mild steel composition	95.37	-	4.63	-	-	-
Plate two mild steel Composition	81.20	14.02	4.36	-	0.42	-
Plate three mild steel Composition	65.18	29.62	-	0.30	4.28	0.62

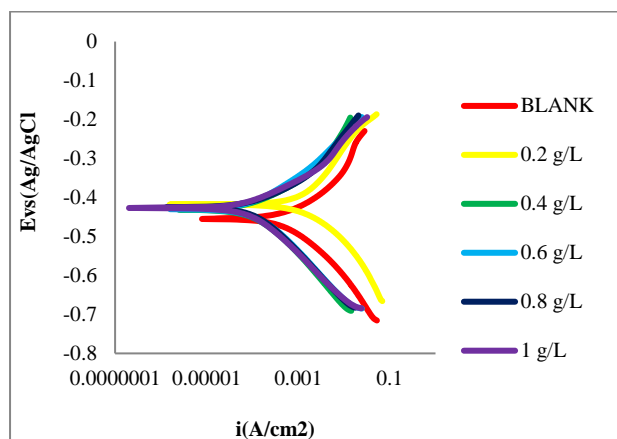
#### Measurements involving electrochemistry

.When comparing the values of the corrosion current densities of the inhibited solutions to the untreated solution (blank), there is a noticeable decrease in the values of corrosion current densities of inhibited solutions to the uninhibited solution (Table 5) This demonstrates that the plant extract slows down mild steel's rate of corrosion in an acidic environment (Hussin, H.M. and Jain Kassim, M. 2011). The results also showed that the value of  $E_{corr}$  changed somewhat depending on whether MOLE was present or not. El-Etre (2007) states that an inhibitor is neither anodic nor cathodic if the biggest displacement in the  $E_{corr}$  value obtained is less than 85 mV (El-Etre, A.Y, 2007). 37 mV is the biggest displacement in the  $E_{corr}$  value, It follows that MOLE is an inhibitor of mixed types. This outcome is consistent with the previously discussed gravimetric analysis data.

Table 5. Potentiodynamic Polarization Parameters for Mild Steel in 2 M HCl in Absence and Presence of Ethanol MOLE

C(v/v)	$-E_{corr}$ (mV)	$I_{corr}$ ( $\mu$ A/cm)	$\beta_a$ (mV/dec)	$\beta_c$ (mV/dec)	$C_R$ (mm/yr)	IE (%)
Blank	456.64	626.27	94.76	119.68	7.27	-
0.2	450.93	316.49	116.05	186.03	3.67	49.46
0.4	429.75	216.21	118.93	105.10	2.51	65.48
0.6	427.86	213.01	110.72	98.99	2.47	65.99
0.8	420.46	191.64	69.53	100.69	2.22	69.39
1.0	420.02	190.58	130.11	116.27	2.21	69.57





**Fig. 9. Potentiodynamic Polarization Curves for Mild Steel in 2 M HCl in the Presence and absence of MOLE**

#### IV. Conclusion

Using both gravimetric and electrochemical techniques, the effectiveness of MOLE as a corrosion inhibitor on mild steel in 2 M HCl was successfully investigated. The findings demonstrated that as extract concentration rises, so does the MOE's ability to suppress corrosion. The anodic and cathodic polarization curves demonstrated that MOE is an inhibitor of mixed types. The inhibitor is physically adsorbed to the mild steel surface, according to activation criteria. The Langmuir adsorption isotherm is followed by the adsorption of MOLE on mild steel surfaces, suggesting monolayer adsorption. The Langmuir adsorption isotherm is followed by the adsorption of MOE on mild steel surfaces. The adsorption process is endothermic, and physisorption is the manner of adsorption, according to thermodynamic characteristics. The current study's findings showed that MOLE can be applied to mild steel in 2 M HCl as a green corrosion inhibitor.

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