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Inhibition Efficiency of *Moringa Oleifera* Leaf Extract on the Corrosion of Reinforced Steel Bar in HCl Solution

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Abstract: *The inhibiting effect of Moringa Oleifera leaf extract on the corrosion of a reinforced steel bar in 2M solution of hydrogen chloride (HCl) was studied using gravimetric, gasometric and potentiodynamic polarization techniques. The study revealed that as the concentration of the extract increases, the inhibition efficiency increases in all three investigation scenarios. 1.0 g/l attained the efficiency of 92.31 % after 120 hours exposure during gravimetric measurement. The volume of the hydrogen gas evolved reduces with an increase in the exposure time during the gasometric test. It was noted that the extract slowed down the corrosion rate and the rate at which the hydrogen gas evolved. The formation of an adsorption layer on the surface of the metal reduces the rate at which hydrogen gas is evolved, which is a function of the concentration of the extract. Potentiodynamic polarization results revealed that the Moringa Oleifera leaf extract modifies the mechanism of anodic dissolution and cathodic hydrogen evolution. It was also observed that the corrosion current density decreases with the increase in the concentration of the extract. This decrease in corrosion is due to increased blocking of the metal surface by adsorption of the leaf extract. From the results, it can be concluded that Moringa Oleifera leaf extract can be used as a green inhibitor to slow corrosion of metals in aggressive media.*

Keywords: *Moringa Oleifera leaves, corrosion rate, weight loss, hydrogen evolution, potentiodynamic polarization*

1. Introduction

Corrosion processes are responsible for losses and failures in the industrial sector. It can be viewed as a universal phenomenon. Corrosion occurs in air, water, soil and in every environment and often affects most materials. Other than material loss, corrosion interferes with human safety, disrupts industrial operations and poses danger to the environment. Nations of the world loose a considerable amount of their gross domestic product (GDP) as a result of corrosion, for example the United States and the United Kingdom loose about $70 billion annually, accounting for about 4% of their national GDP (Gerhardus et al., 2002). A developing nation like Nigeria will most likely be spending a huge fortune on corrosion control annually.

Steel is one of the most versatile; least expensive and widely used engineering materials. It has a wide range of mechanical and physical properties that make it suitable for many applications in the industrial sector. The use of metal involves a major setback—corrosion attack (Raja and Sethuraman, 2009). Problems caused by corrosion can be reduced or minimised by many ways, such as conditioning the metal by coating the metal with another, or conditioning of the corrosive environment by the removal of oxygen, or electrochemical treatment of the metal by cathodic protection or the use of corrosion inhibitors (Bradford, 1993).

The use of a corrosion inhibitor had gained wide acceptance as a means of preventing corrosion in industries. Corrosion inhibitors can be defined as a chemical substance that retards corrosion when added to an environment in small concentration without significantly changing the concentration of any other corrosive agent (Bradford, 1993). It is widely accepted that the corrosion inhibition process takes place through physical or chemical adsorption of the inhibitor's molecules on the metal surface (Kairi and Kassim, 2013). In recent times, some industrial corrosion inhibitors are no longer used because they contain highly toxic compounds such as chromate, phosphate and arsenic. Thus, there is a need for a suitable substitute, which will not only be environmentally friendly but is also safe for the operator to use (Odusote and Ajayi, 2013). Green inhibitors contain alkaloids, tannins and flavonoids and other active agents (Chowdhary *et al.*, 2004; El-Etre *et al.*, 2005) which contribute to their inhibitory properties. The major source of green inhibitors is plants, and they are now being widely studied as a viable replacement for the toxic and harmful synthetic inhibitors being used in most applications.

Several studies have been conducted to examine the use of plant extracts as corrosion inhibitors in various media. Some of these plants include *Tithonia diversifolia* (Alaneme and Olusegun, 2012); *Isertia coccinea* (Lebrini et al., 2011); Rice husk (Alaneme *et al.*, 2015); *Telfaria occidentialis* (Oguzie, 2005); *Nicotiana tabacum* (Olasehinde *et al.*, 2013); *Justicia gendarussa* (Satapathy *et al.*, 2009); *Jatropha Curcas* (Odusote and Ajayi, 2013; Ajayi *et al.*, 2014; Olusegun *et al.*, 2013); *Water hyacinth* (Oloruntoba *et al.*, 2012); *Curcuma Longa* (Kairi and Kassim, 2013); *Phyllanthus amarus* (Okafor *et al.*, 2008); *Carica papaya* (Ebenso and Ekpe, 1996); *Azadirachta indica* (Ekpe *et al.*, 1994), and *Moringa oleifera* (Singh *et al.*, 2011).

Moringa Oleifera (MO) is a perennial plant that grows very fast and flowers within 12 months of planting. It is one of the most widely spread plant species that grows quickly at low altitude in the whole tropical belt, including arid zones. It can grow on sandy or loamy soil having relatively low humidity (Ndabigengesere *et al.*, 1995). All the parts of *Moringa Oleifera* (MO) plant are useful. Oil can be extracted from its seeds to be used as coolants; cosmetics, cooking and soap. The press cakes (i.e. what is left after the oil extraction) are used as soil fertilizer. Pods and leaves are consumed both by humans and animals, as they contain many vitamins. Significantly, the extract from the leaves of the tree is used in engineering as a corrosion inhibitor (Nnanna and Owate, 2014).

Singh *et al.* (2011) studied the effect of the fruit extract of shahjan (*Moringa oleifera*) on the inhibition of the corrosion of mild steel in hydrochloric acid solution using weight loss, electrochemical impedance spectroscopy (EIS), linear polarization, and potentiodynamic polarization techniques (Tafel). The inhibition was found to increase with increasing concentration of the fruit extract in the acid solution. In this study, the inhibiting effect of *Moringa Oleifera* leaf extract on the corrosion of reinforced steel bar in hydrochloric acid solution was evaluated using gravimetric, gasometric and potentiodynamic polarization techniques. This is in continuation of the developmental work on green corrosion inhibitors.

2. Experiment Details

2.1 Preparation of the steel test samples

The reinforced steel bar was obtained from Prism Steel Mill Limited, South West, Nigeria. Its chemical composition was determined using an Optical Emission Spectrometer and the results are presented in Table 1. The steel sample was cut into pieces measuring 12 mm × 6 mm, polished using emery paper, degreased in ethanol, dried in acetone and then stored in desiccators.

Table1. The chemical composition of the reinforced steel bar

Element	Percentage (wt%)	Element	Percentage (wt%)
C	0.2910	**Al**	0.0210
Si	0.1930	**Ti**	0.0080
Mn	0.5790	**Nb**	0.0001
S	0.0420	**B**	0.0020
P	0.0540	**W**	0.0001
Cr	0.2710	**Mo**	0.0001
Ni	0.1050	**V**	0.0001
Cu	0.3080	**Fe**	98.1260

2.2 Preparation of the *MO* leaf extract

The procedure for the preparation of the leaves extract is similar to that reported by Odusote and Ajayi (2013). Fresh green *Moringa Oleifera* leaves were obtained from the surroundings of The University of Ilorin, and then washed, cut into pieces, and air dried before pulverization. 200 grams of the pulverized leaves were soaked in 250 $cm^3$of methanol in a conical flask. The resulting solution was left for 48 hours and then filtered. The filtrate was subjected to evaporation at room temperature to obtain the extract. The inhibition test solutions were prepared from the extract to obtain 0.2 g, 0.4 g, 0.6g, 0.8 g and 1.0 g in 1 litre of 2M hydrogen chloride (HCl).

2.3 Weight loss measurement

The pre-weighed test specimens were immersed in the 2.0 M HCl solution with and without varying concentrations of *Moringa Oleifera* extract. After 24 hours, the test specimens were retrieved, washed with distilled water, rinsed with ethanol, dried with acetone and reweighed using an electronic weighing balance (HX 302T with 0.01 g accuracy). The difference in weight was taken as weight loss. The same procedure was repeated after 48, 72, 96 and 120 hours exposure. From the weight loss data, corrosion rate, inhibition efficiency (IE%) and surface coverage (θ) of the plant extract were calculated using Equations 1, 2 and 3, respectively.

$$CR = \frac{\Delta W}{AT} \quad (1)$$

where *CR* is corrosion rate, *W* is the weight loss (g), *A* is the area of the coupon in cm^2, and T is exposure time (hours).

Inhibition efficiency from weight loss (IE_{wt}) and surface coverage (Θ) were calculated using Equations 2 and 3, respectively.

$$IE_{wt}\% = \left(1 - \frac{CR_{inh}}{CR_{blank}}\right) x\ 100 \quad (2)$$

where CR_{inh} and CR_{blank} correspond to the corrosion rates in the presence and absence of the extract, respectively.

$$Surface\ coverage, \theta = \left(1 - \frac{CR_{inh}}{CR_{blank}}\right) \quad (3)$$

2.4 Hydrogen evolution measurement

The hydrogen evolution measurement was carried out using gasometric assembly. The prepared corrodent (2M HCl) was poured into 100 ml two-necked flask and the initial volume of the air in the 50 ml burette was recorded. Thereafter, the already weighed test specimen was dropped into the HCl solution. The flask was quickly closed to ensure that it was air-tight, preventing the escape of gases. The volume of the hydrogen gas evolved from the corrosion reaction was monitored by the downward displacement of water. The absorption time was noted. The displacement was monitored by the volume change in the level of water in the burette. The displacement was recorded at 20-minute intervals and progressively for 3 hours and 20 minutes. This procedure was repeated for a fresh inhibition test solution of different concentration of *Moringa Oleifera* leaf extract (0.2 g/l, 0.4 g/l, 0.6 g/l, 0.8 g/l and 1.0 g/l).

The inhibition efficiency and the degree of surface coverage from hydrogen evolution measurement were determined using Equations 4 and 5, respectively.

$$IE_{H_2}\% = \frac{V_{HO} - V_{H1}}{V_{HO}} X\, 100 \quad (4)$$

$$Surface\ coverage, \theta = \frac{V_{HO} - V_{H1}}{V_{HO}} \quad (5)$$

where V_{HO} is the volume of hydrogen gas evolved in the blank solution and V_{HI} is the volume of hydrogen gas evolved in the presence of *Moringa Oleifera* leaf extract.

2.5 Potentiodynamic polarization measurement

This involves changing the potential of the working electrode and monitoring the current that is produced as a function of time and potential. This test was carried out using a three-electrode cell assembly at room temperature with the AUTOLAB PGSTAT 204N instrument. The metal samples of area of 1 cm^2 were embedded in resins. The embedded metal sample was used as the working electrode; the platinum electrode was used as the counter electrode and saturated silver/silver chloride as the reference electrode. The electrolyte used for this study was 2M HCl solution with varying concentrations of the extract and without extract.

The working electrode was polished with different grits of emery paper until the surface became smooth. The open circuit corrosion potential was carried out for 30 minutes until a stable potential was attained. The potentiodynamic polarization study was carried out from cathodic potential of -250 mV to an anodic potential of +250 mV with a scan rate of 1.0 mVs^{-1} to determine the current density, corrosion rate and inhibition efficiency (IE%).

The surface coverage (Θ) and inhibitor efficiency (*IE%*) were determined using Equations 6 and 7, respectively.

$$Surface\ coverage, \theta = \frac{I^o_{corr} - I_{corr}}{I^o_{corr}} \quad (6)$$

$$IE\,\% = \frac{I^o_{corr} - I_{corr}}{I^o_{corr}} \times 100 \quad (7)$$

where I_{corr} and I^o_{corr} are corrosion current density in the presence and absence of the extract, respectively.

3. Results and Discussion

3.1 Phytochemical Screening of the *MO* Leaves

The phytochemical screening of the *Moringa Oleifera* leaves was carried out and the results are presented in Table 2. The results show that the extract contains tannins, saponins, alkaloids, cardiac glycosides, anthraquinones, flavonoids, phenol, oxalate, steroid and terpenoid. This result is similar to the experimental results reported by Kasolo *et al*. (2010). Previous results have attributed the inhibitory properties of most green plants to the presence of active agents such as flavonoids, tannins, saponins and alkaloids (Ekpe *et al.*, 1994; Ebenso and Ekpe, 1996; Lebrini *et al.*, 2011; Odusote and Ajayi, 2013; Ajayi *et al.*, 2014 and Alaneme *et al.*, 2015).

Table 2. Phytochemical screening result of the *Moringa Oleifera* leaves

S/N	Constituent	*Moringa Oleifera* leaves
1	Alkaloids	+
2	Cardiac glycosides	++
3	Tannins	+++
4	Saponin	+
5	Anthraquinones	-
6	Flavonoids	+++
7	Terpenoids	+
8	Anthraquinone	++
9	Oxalate	+
10	Phenols	+
11	Trypsin	++

Key: - = absent; + =present in minute amount; ++ =present in moderate amount; +++=present in an appreciable amount.

3.2 Weight loss measurement

Figure 1 shows weight loss with respect to exposure time to *Moringa Oleifera* leaf extract. It can be deduced that there is a significant reduction in the weight loss of test specimens immersed in varying concentrations of the extract in comparison to the blank solution. Additionally, as the concentration of the extract increases, the weight loss reduces. The reduction in the weight loss could be attributed to the adsorption of tannin, alkaloids, and flavonoids, among others. The adsorption of these compounds on the metal surface creates a barrier to the dissolution of the metal in corrosive medium (Oguzie, 2007). The 0.2 g/l recorded a weight loss of 0.19 g after 120 hours of exposure while at the same time for 0.4 g/l recorded 0.07 g, 0.6 g/l recorded 0.07 g, 0.8 g/l recorded 0.06 g and 1.0 g/l recorded 0.04 g as shown as in Figure 1. This indicates that the weight loss is highly dependent on the concentration of the inhibitor.

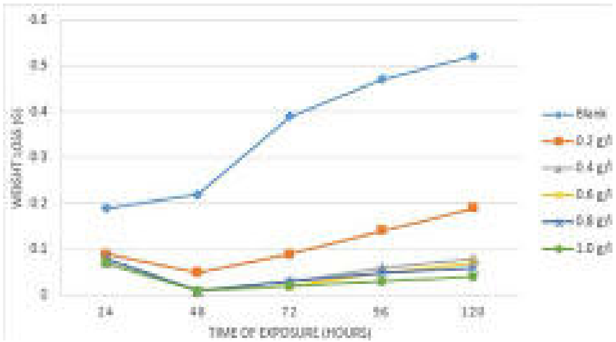

Figure 1. Weight Loss in the steel test sample against time of exposure in acidic medium with and without *Moringa Oleifera* leaf extract

Figure 2 presents the variation of corrosion rate against the exposure time. It is very obvious from Figure 2 that the corrosion rate of the reinforced steel bar in the absence of the plant extract decreases with the increase in concentration of the extract. The result after 24 hours revealed that the values of corrosion rate of the steel bar in 2M HCl decreased with increase in the concentration of *Moringa Oleifera* leaf extract. This suggests that as the concentration of the extract increases, there is an increase in adsorption of the extract constituents on the surface of the steel bar which resulted in the reduction of the corrosion rate (Nnanna and Owate, 2014; Okoronkwo *et al.*, 2015).

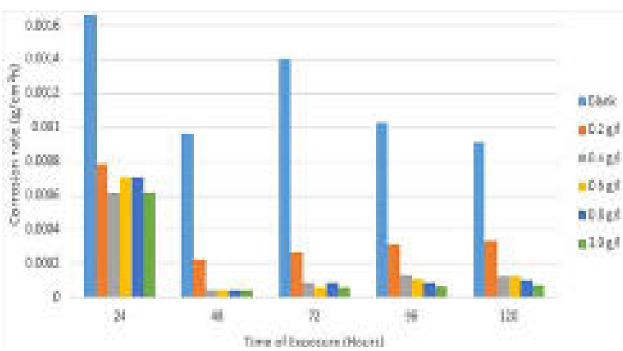

Figure 2. Variation in the corrosion rate of the reinforced steel bar against exposure time with and without *Moringa Oleifera* leaf extract

The stability of inhibitive behavior of the extract was assessed by the trend of the inhibition efficiency as a function of time. The values of inhibition efficiency (IE%) for each concentration of *Moringa Oleifera* leaf extract are recorded in Table 3. It was observed that the inhibition efficiency increases with an increase in inhibitor concentration but this was not consistent with the time of exposure. This is due to the formation of a protective film which is as a result of transition of the metal/solution interface from an active dissolution state to a passive state.

Table 3. Inhibition efficiency of reinforced steel bar for various concentration of *Moringa Oleifera* leaf extract at different time of exposure

Time of exposure (Hours)	Inhibition efficiency (%)					
	Blank	0.2 g/l	0.4 g/l	0.6 g/l	0.8 g/l	1.0 g/l
24.00	-	52.63	63.15	57.89	57.89	63.15
48.00	-	77.27	95.45	95.45	95.45	95.45
72.00	-	76.92	92.31	94.87	92.31	94.87
96.00	-	70.21	87.23	89.36	91.49	93.62
120.00	-	63.46	86.53	86.53	88.46	92.31

Therefore, the extract at this concentration protected the reactive surface of the metal from the aggressive environment of the acidic medium as reported by Ritchie and Bailey (1999). This observation agrees with the results obtained by Singh *et al.*, (2011), which revealed that the inhibition of corrosion of mild steel in hydrochloric acid solution by the fruit extract of shahjan (*Moringa oleifera*) increased with increasing concentration of the extract. They also concluded that the fruit extract of *Moringa oleifera* could serve as a viable inhibitor of the corrosion of mild steel in hydrochloric acid solution. However, there is a little decline in the inhibition efficiency after 72 hours exposure. The reason could be attributed to the adsorptive film of inhibitor that settles during the immersion time. After the formation of adherent and compacted adsorptive film, the effect of the exposure time become less significant because of the presence of barrier between the metal surface and the inhibitor (Faustin *et al*., 2015, Li *et al*., 2012).

3.3 Hydrogen evolution measurement

The volumes of hydrogen gas evolved in the presence and absence of *Moringa Oleifera* leaf extract are presented in Table 4. The results showed that the volume of hydrogen gas evolved increases with time but decreases as the concentration of extract increases. This indicates that the rate of the dissolution of the metal was retarded, leading to reduction of H^+. Readings of the lowest and highest volume of hydrogen evolved were noted in the 1.0 g/l and blank solution, respectively. These results conform to the conclusion drawn by Odusote and Ajayi (2013), that the rate of evolution of hydrogen gas decreases as the concentration increases and the least value of hydrogen gas was recorded at the highest concentration. The higher volume of hydrogen gas evolved in the blank solution suggests that there is no adsorption layer to inhibit the reaction of the acid on the surface of the metal according to Nnanna and Owate, (2014).

It can be deduced from Figure 3 that as the concentration of the extract increases, the inhibition efficiency also increases. This follows the same trend with the weight loss measurement. It can also be observed that there was a re-ordering of the inhibition efficiencies after 80 minutes. The corrosion inhibition of the plant extract on the surface of the steel may be due to the phytochemical constituents present in the leaf extract. It can be observed that the inhibition efficiency becomes more stable as the exposure time increases. This stability in the inhibition efficiency indicates that the extract must have been fully absorbed on the surface of the metal. It can also be observed from the plot that 1.0 g/l possessed the highest value of inhibition efficiency. This also suggests that the surface of the metal was protected against acidic attack at higher concentration as reported by Uwah *et al.* (2013).

Table 4. Volume of Hydrogen gas evolved with time of exposure in the presence and absence of *Moringa Oleifera* leaf extract

Time of Exposure (Minutes)	Volume of Hydrogen gas evolved in the presence and absence of the *Moringa Oleifera leaf* extract (ml)					
	Blank	0.2 g/l	0.4 g/l	0.6 g/l	0.8 g/l	1.0 g/l
0.00	0.03	0.03	0.20	0.20	0.20	0.10
40.00	0.80	0.60	0.60	0.40	0.40	0.50
80.00	2.80	1.10	1.30	0.80	0.70	0.80
120.00	4.00	1.60	1.90	1.30	1.10	1.20
160.00	5.20	2.20	2.20	1.90	1.50	1.30
200.00	6.80	2.90	2.60	2.40	2.00	1.50

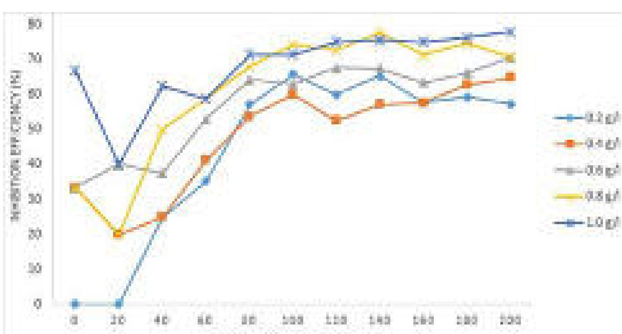

Figure 3. Variation in the inhibition efficiency with time of exposure in the presence and absence of the *Moringa Oleifera leaf* extract

3.4 Tafel polarization

Figure 4 shows the potentiodynamic polarization curves of corrosion inhibition of the mild steel in 2M HCl in the absence and presence of different concentrations of *Moringa Oleifera* leaf extract. It was observed that the polarization resistance (Rp) and inhibition efficiency (IE%) increase as the concentration of the extract increases. The highest inhibition efficiency of 81% was obtained at 1.0 g/l concentration. This also indicates that more inhibitor particles were absorbed on the surface of the metal thus providing a wider surface coverage and the extract acts as an adsorption inhibitor as reported by Mokhtari *et al.*, 2013. It can be observed from Table 5 that the corrosion current density (I_{corr}) decreases with increases in the concentration of the extract which is due to the increase in the blocked fraction of the metal surface by adsorption as reported by Fouda *et al.* (2014).

The shift in the Tafel slopes of both the cathodic reaction (βc) and anodic reaction (βa) as shown in Figure 4 in the presence and absence of the extract suggest that the inhibitor affects both the cathodic and anodic reactions. This also suggests that *Moringa Oleifera* leaf extract is a mixed type of inhibitor. It can also be observed from Figure 4 that as the concentration of the extract increases, there appears to be a shift towards more negative potential indicating that the *Moringa Oleifera* acts as a good corrosion inhibitor.

It can be observed that as the corrosion current density (I_{corr}) decreases, the inhibition efficiency (IE)% increases as the concentration of the extract is increased. This suggests that the extract retards the reactions at both the cathodic and the anodic sites as a result of coverage of these sites by the molecules of the extract as reported by El-Shafei *et al*. (2004).

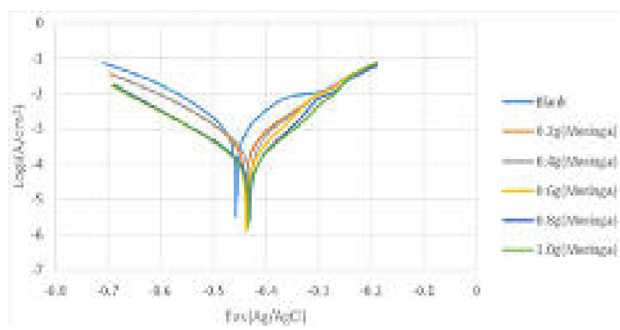

Figure 4. Polarization curves of reinforced steel bar in the presence and absence of different concentration of *Moringa Oleifera* leaf extract

Table 5. Corrosion parameters of the test sample in 2 M HCl in the presence and absence a *Moringa Oleifera* leaf extract

Concentration (g/l)	$-E_{corr}$ (mVvs SCE)	I_{corr} ($mAcm^{-2}$)	Tafel Slope		Rp (Ω)	Θ	IE%
			βa ($mVdec^{-1}$)	βc ($mVdec^{-1}$)			
Blank	458.06	617.53	70.375	72.329	25.085	0.000	---
0.200	438.9	442.52	123.47	99.994	54.223	0.283	28.30
0.400	430.28	413.72	137.63	91.053	57.524	0.330	33.00
0.600	436.60	147.81	121.34	74.798	135.96	0.761	76.06
0.800	431.71	134.17	121.65	87.757	165.02	0.783	78.27
1.000	434.94	113.73	104.44	89.484	184.03	0.816	81.58

The shift in βc and βa values as shown in Table 5 indicates that adsorption of *Moringa Oleifera* leaf extract modifies the mechanism of anodic dissolution and the cathodic hydrogen evolution, this reveals that the inhibition mechanism occurred by simply blocking the available cathodic and anodic sites of the metal surface according to Abdel-Gaber *et al*. (2009).

4. Conclusions

Based on this investigation into the inhibition of the corrosion of reinforced steel bar in hydrochloric acid solution by the leaf extract of *Moringa oleifera* using gravimetric, gasometric and potentiodynamic polarization techniques, the following conclusions can be drawn:

1) The corrosion rate of the steel bar in HCL solution was found to decrease with increasing concentration of the leaf extract of *Moringa Oleifera*.
2) The inhibition efficiency of the leaf extract of *Moringa Oleifera* is dependent on the concentration of the extract, and it increased with increasing concentration of the extract in the acidic medium.
3) As the concentration of the extract increased, the cathodic evolution of hydrogen gas decreased and the least evolution was recorded at the highest concentration.
4) Tafel polarization results showed that the extract acted as a mixed type inhibitor via a simple adsorption of the phytochemicals present in the extract on the mild steel surface in HCl solution.
5) The leaf extract of *Moringa Oleifera* could serve as an effective inhibitor of corrosion of mild steel in hydrochloric acid solution.

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