The Pharmaceutical and Chemical Journal, **2019, 6(3):53-62**

Available online www.tpcj.org

Research Article

ISSN: 2349-7092
CODEN(USA): PCJHBA

Optimization and Impedance Study on the Inhibition Efficiency of Moringa Leaf Extract as Corrosion Inhibitor of Aluminum in Alkaline Medium

Okafor, C. Sandra*[1], Onukwuli, O. Dominic[1], Anadebe, V. Chikaodili[2]

[1]Department of Chemical Engineering, Nnamdi Azikiwe University Awka Nigeria
[2]Department of Chemical Engineering, Federal University Ndufu Alike Ikwo, Nigeria
*Corresponding author: nwikechizoba@yahoo.com, anadebechika@gmail.com

Abstract This work is on optimization and impedance study of inhibition efficiency of moringa leaf extract as corrosion inhibitor of aluminum in alkaline solution. Gravimetric method, polarization study and impedance measurement was employed in the corrosion inhibition study. Response surface methodology (RSM) was applied in the optimization process. Central composite design tool of design expert software version 9 was used to evaluate the effects and interactions of four variables of acid concentration (AC), inhibitor concentration (IC), temperature (T) and time (t) on the inhibition efficiency of the extract. Analysis of variance (ANOVA) was used to study the data generated. Moringa leaf extract exhibited optimum inhibition efficiency of 96.65 % at optimum inhibitor concentration of 1.5 g/L^{-1}. The electrochemical impedance spectra revealed that moringa leaf extract acted as a mixed-type inhibitor. The extract is highly efficient in surface treatment of aluminum in alkaline environment.

Keywords Optimization, Corrosion, Aluminum, KOH, Moringa leaf.

1. Introduction

Metals and alloys are still the most widely used group of materials particularly in engineering industries. However, the usefulness of these metallic materials is constrained by one common problem called corrosion.

Previously, the use of chemical inhibitors in protection of metals against corrosion was successful, but due to their harmful effect to the environment and high cost of preventing corrosion in industries, there is need to focus research on suitable alternatives from renewable, eco-friendly and biodegradable sources. Agricultural by-products are been discarded as waste but they can be converted to wealth (put into economic use) as corrosion inhibitor. Many metals and alloys used in industries are prone to different mechanisms of corrosion due to their exposure to different aggressive environments. Acid solutions are often used in industry for cleaning, descaling and pickling of steel structure these processes are normally accompanied by considerable dissolution of metal quality [1,2]. The most acceptable method of protecting metals and alloys against corrosion is the application of inhibitor in contact with the surface in order to inhibit the corrosion reaction and reduce the corrosion rate.

It has become necessary to look towards nature for ‘‘clean’’ solutions to the world’s energy demands. Green inhibitors are biodegradable, non-toxic and contain no heavy metals. Plants products are inexpensive, renewable and readily available. Some of the plant organic compounds have been reported to function as effective inhibitors of metal in different aggressive environments [3–8]. The choice of an appropriate inhibitor for a particular application is restricted by a number of factors [9]. Attempt has been made in using response surface methodology (RSM) in optimization of inhibition efficiency of plant extract [10]. The aim of this work is to examine the corrosion inhibitive property of moringa leaf extract and optimize its application as corrosion inhibitor of aluminum. *Moringa* is a perennial plant and one of the valuable plant species that grows fast at minimal altitude. Moringa leaf extract is of interest because of its tremendous pharmaceutical and industrial applications [11].

2. Materials and Methods

Fresh leaves of moringa leaf (ML) were collected from *Moringa* orchard at Awka in Anambra State of Nigeria. The leaves were sun-dried for four days and then ground into powder form to increase its surface area. During the extraction process, 30 g of ML powder were measured and soaked in 1000 mL of ethanol for 48hrs. The mixture was filtered. The filtrate obtained is a mixture of the plant extract and the ethanol. Distillation process was applied to separate the solvent from the extract. The stock solution of the extract was weighed and stored for the corrosion inhibition study. The chemicals, ethanol and KOH, used for the study were of analytical grade.

2.1. Metals preparation

Corrosion tests were performed on aluminum sheet with the following compositions Pb (0.064%), Mn (1.22%), Si (0.3%), Cu (0.077%), Ti (0.026%), V (0.09), Fe (0.55%) and Al (97%). Prior to corrosion tests, the aluminum was mechanically cut into (5cm x 4cm x 0.1cm). The surface of each coupon was polished using different emery papers to expose shining polished surface. To remove organic impurities, the coupons were degreased with acetone, washed with distilled water and allowed to dry in air [12].

2.2 Weight Loss (Gravimetric) Method

2.2.1 Weight loss method using one factor at a time

Adapting one factor at a time, weight loss methods was carried out at temperatures of 303 K, 318 K and 333 K respectively. In this method, as previously expressed [13], weighed aluminum coupon was immersed in 250 mL beaker containing 200 mL of 3 M KOH. Also, other aluminum coupons were separately immersed in 250ml beakers containing 3 M KOH with various concentrations of ML extract. The difference in the weight loss was studied periodically at different temperatures in the uninhibited and inhibited medium with various concentrations of the ML. After the corrosion study the coupons were remove, immersed in acetone, scrubbed with brush, dried and reweighed. The weight loss (Δw), corrosion rate (CR), inhibition efficiency (IE) and degree of surface coverage were determined using the equations (1), (2) (3) and (4) respectively [14].

$$\Delta w = w_i - w_f \quad (1)$$

$$CR = \frac{W_{bl} - W_{inh}}{Area\ (m2)\ x\ t\ (time)day} \quad (2)$$

$$IE\% = \frac{W_{bl} - W_{inh}}{W_{bl}} \text{ x } 100 \quad (3)$$

$$\theta = \frac{W_{bl} - W_{inh}}{W_{bl}} \quad (4)$$

Where w_i and w_f are initial and final weight of aluminum (Al) samples respectively, w_i and w_o are the weight loss values of Al samples in presence and absence of the extract.

2.3 Electrochemical Technique

Thorough electrochemical analysis was performed with the aid of a potentiostat/galvanostat 263 electrochemical system workstation. Within the context of this study a graphite bar and a calomel electrode (CE) were employed as a supplementary material. All specimen of 1cm^2 dimension was used as working electrode. The experiment was performed in aerated and unstirred solution at the end of 30 mins of immersion; this makes the solution to stabilize. Experiment was performed considering ±250 mV VS E_{corr} and further examine sequentially at speed of 0.333 mV/s. This was evaluated using eq. (5).

$$IE\ \% = \frac{i_{corr\ (bl)} - i_{corr\ (inh)}}{i_{corr\ (bl)}} \text{ X } 100 \quad (5)$$

Where (*bl*) and (*inh*) are the current density values in the absence and presence of inhibitor.

2.4 Optimization of the Inhibition Efficiency

Using response surface methodology (RSM), the inhibition efficiency of the extract was optimized by central composite design (CCD) tool of Design Expert Software Version 9. The independent variables selected for this study were acid concentration (1 M - 3 M), inhibitor concentration (0.3g/L^{-1} – 1.5 g/L^{-1}), temperature (303 K – 333 K) and time (3hr – 6 hr).

3. Results and Discussion

The experimental data of weight loss and corrosion rate using one factor at a time are presented in Table 1. The inhibition efficiency increased with increase in concentration of the inhibitor (extract) reason being that the spontaneous accumulation of ML active species result to a near perfect coverage which impeded the invading of the corrodent. On the contrary, the protection efficiency decreased with increase in temperature because the rise in temperature breaks the heterocyclic bond of the ML active compounds thereby reducing the rate of surface adsorption on the degrading surface. This observation is in agreement with other research reports as contained in literature [15]. Maximum inhibition efficiency of 96.70 % and 92.82 % was obtained at inhibitor concentration of 1.5 g/L^{-1}. The high values of the inhibition efficiency showed that the extract can be used for corrosion control of aluminum in alkaline environment.

Table 1: Gravimetric results of Aluminum specimen in KOH with ML extract

M (g/L^{-1})	**1M**			**3M**		
	303 K	**318 K**	**333 K**	**303 K**	**318 K**	**333 K**
0.3 g/L^{-1}	55.46	55.38	37.54	51.63	42.55	40.38
0.6 g/L^{-1}	66.67	73.08	48.75	62.84	60.25	59.32
1.2 g/L^{-1}	81.47	78.02	63.55	77.64	65.19	64.17
1.5 g/L^{-1}	96.70	87.12	78.73	92.82	74.29	69.01

3.1 Adsorption Study

Elucidation of the phenomenon of adsorption from the experimental data requires estimation of the adsorption modes of the inhibiting species (whether molecular or ionic). The adsorption isotherm was tested for fitness with the experimental data in KOH at the temperature range of 303K – 313 K for ML extract. Due to adsorption, inhibitor molecules block the reaction sites and obstruct the rate of corrosion. The inhibitor molecules inhibit the corrosion of aluminum by adsorption on the aluminum surface; the adsorption provides vital information about the interaction around the adsorbed molecules themselves as well as their interaction with the aluminum surface respectively. Linearity accounts for physical adsorption. Langmuir adsorption was proposed to scrutinize the kinetics of adsorption of ML on Al surface. Langmuir adsorption isotherm is presented in Equation (6)

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (6)$$

C represent concentration of inhibitor, $\mathbf{K}_{ads}$ denotes adsorption equilibrium. The adsorption parameters are listed in Table 2. As seen in Fig 1 the plot of C/θ versus C yields a linear graph where the slope is near unity due to the surface adsorption of each ML molecule on individual active site on the aluminum surface. This mechanism of adsorption strongly conform to the Langmuir isotherm with a high correlation coefficient R^2 (0.9849) and slope of about unity (1.2982) [16]. Also Table 2 indicates the thermodynamics and activation energy of aluminum in KOH. The values of Ea recorded in the presence of different concentration of ML extract are larger than the values of Ea for the blank (in absence of inhibitors). This proves that the extracts retarded the corrosion of aluminum in the KOH corrodent media. Further observation from the result reveals that activation energy increases as the concentration of the inhibitor increases.

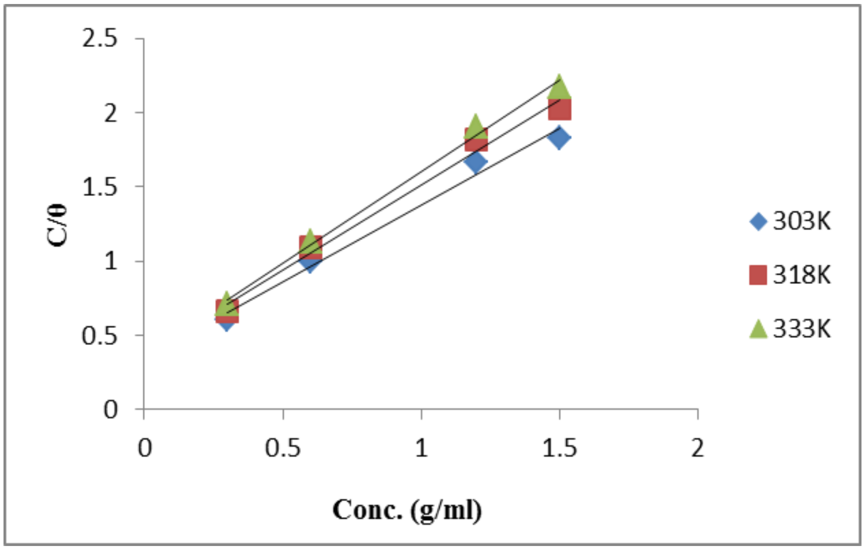

Figure 1: Langmuir isotherm for adsorption of ML on aluminum in KOH

Table 2: Langmuir Adsorption Parameter for Aluminum in 3M KOH with ML extract

Langmuir adsorption parameters					
Temp (K)	**Log K**	**K**	**Slope**	**ΔG (KJ/mol)**	**R^2**
303	0.1551	6.4475	1.3287	-14.8154	0.9843
318	0.2277	4.3917	1.2950	-14.5335	0.9847
333	0.3289	3.0404	1.2710	-14.2008	0.9857
Mean	0.2372	4.2153	1.2982	-14.5166	0.9849

Table 3: Activation and Heat of Adsorption studies

Conc. (g/L^{-1})	**Activation Energy (Ea)**	**Heat of Adsorption (Q_{ads}) KJ/mol**
Blank	36.59	
0.3	41.79	-14.34
0.6	37.61	-4.49
1.2	47.05	-16.95
1.5	63.51	-36.00

3.2 Polarization Study

Polarization test were carried out to distinguish the inhibitive effect of ML on the half reactions. Typical polarization curves for aluminum in 3 M KOH containing 0.3 g/L^{-1} and 1.5 g/L^{-1} inhibitor concentrations at 30°C are shown in Fig 2. The considered electrochemical parameters includes corrosion potential (E_{corr}), corrosion current densities (I_{corr}), cathodic Tafel slopes (b_c) and anodic Tafel slopes (b_a) obtained from polarization curves are presented in Table 4. The aluminum specimen is seen to exhibit active dissolution with no distinctive transition to passivation within the investigated range. Close observation of the polarization curve for aluminum in 3 M KOH reveals that the presence of ML Pushes the cathodic and anodic axis towards minimum current densities in the environment investigated. The introduction of ML into the KOH solution has no significant effect on the corrosion potential (E_{corr}). Also, the deviation in E_{corr} is not greater than 85 mV as shown in Table 4. Thus, ML is categorized as a mixed type inhibitor in KOH environment [17-18]. Furthermore, the results in Table 4, show that the corrosion current density (I_{corr}) reduced consistently in the presence of the inhibitor compared to that obtained in the unprotected solution and also decreased with an increase in the concentration of the extract.

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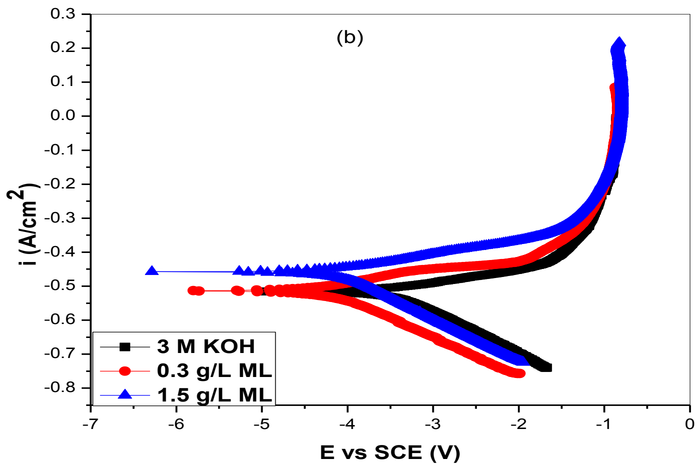


Figure 2: Polarization curves of Aluminum in the presence of ML in 3M KOH

3.3 Electromchemical Impedance Spectroscopy

The attachment of a known film layer on the aluminum surface causes a pronounced increment in the impedance of the corrosion process, thereby causing a promotion in the radius of the imperfect semi circle R_{ct}. Protection ability of an inhibitor can be evaluated by the test of the corrosion system impedance. The extent of the corrosion protection can be predicted by comparing of the impedance obtained in the unprotected and protected environment. Similar to the effect seen on the corrosion potential, the impedance of the aluminum dissolution system was affected severely in the protected medium. The Nyquist plot for the corrosion phenomenon of aluminum coupon in 3 M KOH in the absence and presence of ML are shown in Fig 3. The considered parameters in this study includes: charge transfer resistance (R_{ct}) and the double layer capacitance (C_{dl}) are presented in Table 5. When the aluminum coupon was immersed in KOH solution the Rct values was found to be at the range of 300.5 to 2400 Ω cm^2 in 3 M KOH at maximum concentration respectively. Rct values increased considerably with an increase in ML [19]. The increased Rct values and decreased double layer capacitance values obtained from impedance studies proves the unique action of ML as green corrosion inhibitor for aluminum in KOH solution.

The inhibition efficiencies (IE %) for inhibitor concentrations were calculated from Nyquist plots using the equation:

$$IE\% = \left(\frac{R_{ct\,(inh)} - R_{ct}}{R_{ct\,(inh)}}\right) \times 100 \qquad (7)$$

Where R_{ct} and $R_{ct\,(inh)}$ denotes charge transfer resistance in the absence and presence of the inhibitor. The result obtained using EIS technique follows the same trend compared to potentiodynamic polarization technique.

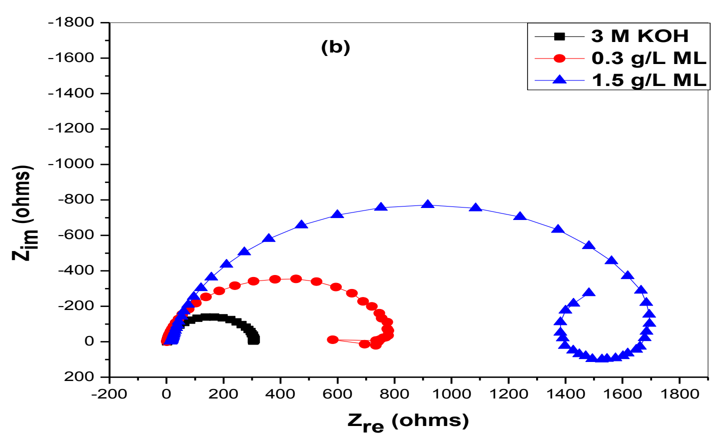


Figure 3: Impedance spectra of Aluminum in the presence of ML in 3M KOH

Table 4: Parameter from Tafel Polarization Measurements

System	E_{Corr} (mV vs. SCE)	I_{Corr} (μA cm^{-2})	βc (mV dec^{-1})	βa (mV dec^{-1})	Θ	IE (%)
1 M KOH	-536.4	107.1	106.4	92.6	-	-
1M KOH + 0.3g/L^{-1} ML	-540.9	57.3	100.1	77.6	0.464	46.4
1M KOH + 1.5g/L^{-1} ML	-438.9	10.3	98.4	72.2	0.903	90.3

Table 5: Impedance Data for Aluminum in 3M KOH

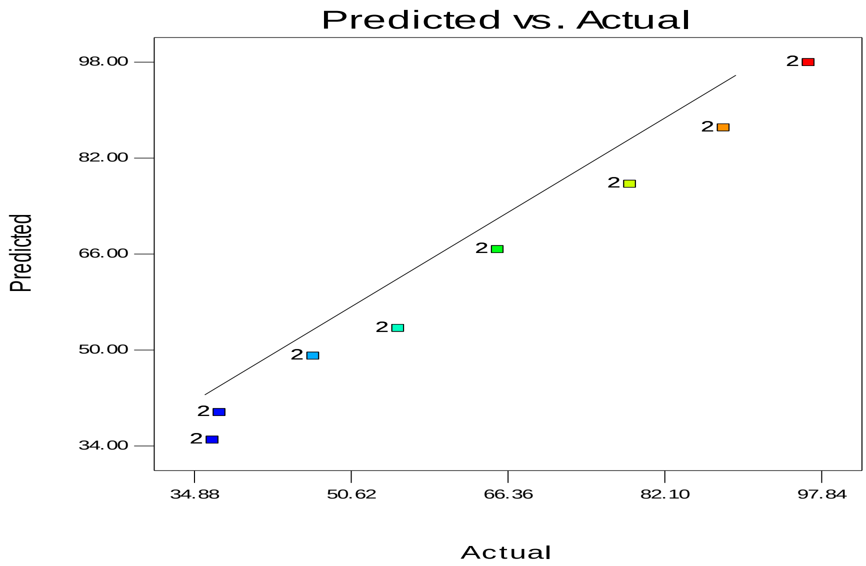
System	R_{ct} (Ω cm^2)	N	C_{dl} (F cm^2)	IE (%)
1 M KOH	300.5	0.89	6.907E-5	
1M KOH + 0.3g/L^{-1} ML	745	0.89	7.114E-5	59.7
1M KOH + 1.5g/L^{-1} ML	2400	0.89	7.061E-5	87.5

3.2 Results of the weight loss method using RSM

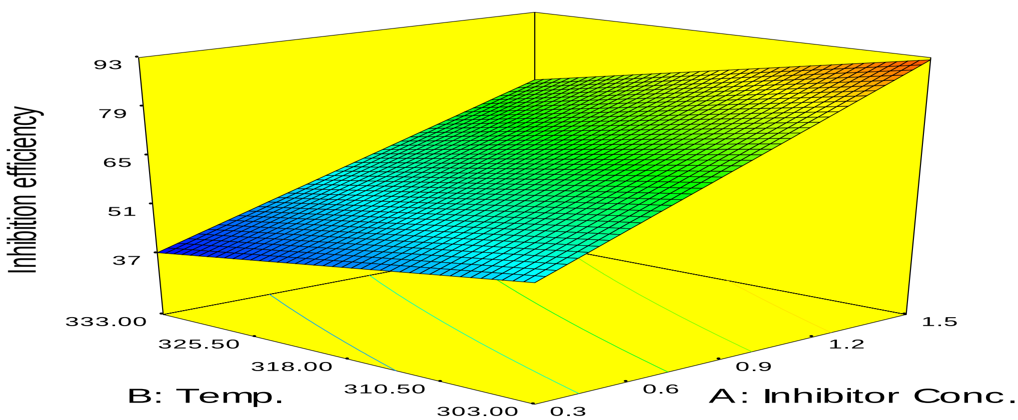
Using response surface methodology (RSM), the inhibition efficiency of ML extract was scrutinized and optimized by central composite design (CCD) tool of Design Expert Software (version 10). A total of 16 runs of experiments were conducted. The 3-D surface plot of efficacy of ML on aluminum surface is presented in Fig 4.The results of inhibition efficiency as functions of acid concentration, inhibitor concentration, temperature and time are presented in Table 6. The corrosion rate increased with increase in acid concentration, temperature and time, while increased inhibitor concentration decreases the corrosion rate. The inhibition efficiency of moringa leaf extract was dependent on acid concentration, inhibitor concentration, temperature and time. The analysis of inhibition efficiency of moringa leaf extract (inhibitor) on aluminum in KOH medium is presented in Figure 6. The predicted versus actual inhibition efficiency plot showed a linear graph (Fig 6 A), indicating that the regression model is able to predict the inhibition efficiency of the extract. The 3-D surface plots (Figure 6, B-D) showed the relationship between the factors affecting the inhibition process and inhibition efficiency of the extract. The nature of the three dimensional surfaces suggest that there is interactions among the considered factors of the corrosion inhibition process.

Table 6: Experimental range and levels of CCD for ML on Aluminum (AA3003) in KOH

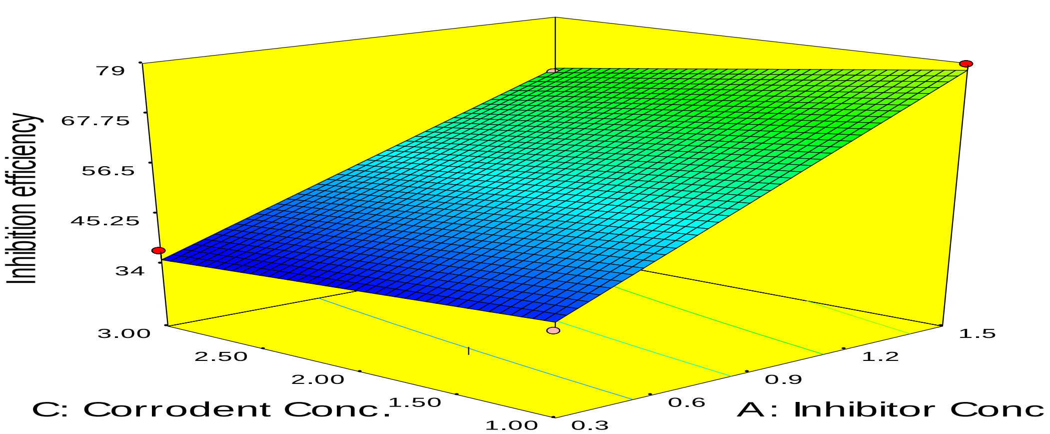
Run	F_1 (IC) g/L^{-1}	F_2 (T) K	F_3 (AC)	F_4 (hr)	Response $_1$ IE (%)
1	1.5	333	3	3	65.47
2	1.5	333	1	6	78.43
3	0.3	303	3	3	46.94
4	1.5	303	1	6	96.65
5	1.5	303	3	3	88.13
6	1.5	303	3	6	78.73
7	0.3	333	3	3	36.84
8	0.3	333	3	6	65.47
9	0.3	303	1	3	55.46
10	1.5	303	1	6	96.55
11	0.3	303	3	3	46.94
12	0.3	333	1	6	37.54
13	0.3	303	1	3	55.46
14	0.3	333	1	6	37.54
15	1.5	303	3	3	88.13
16	0.3	333	3	3	36.84


Predicted vs. Actual
Predicted
98.00
82.00
66.00
50.00
34.00
34.88
50.62
66.36
82.10
97.84
Actual

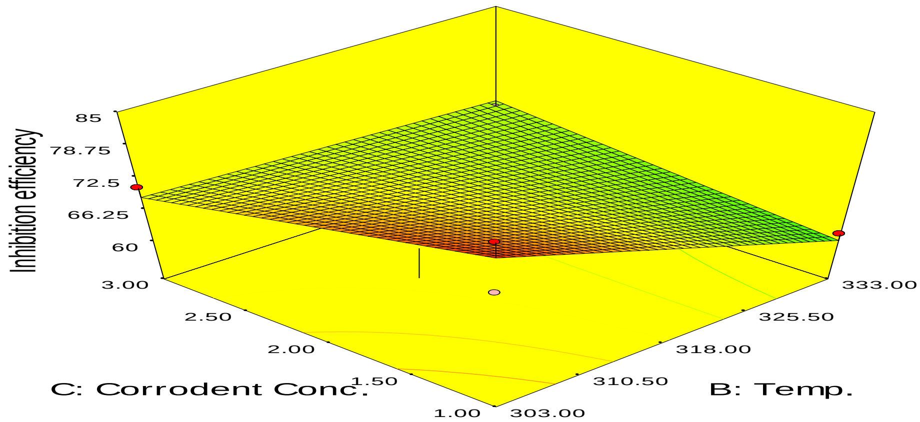
(A)


Inhibition efficiency
93
79
65
51
37
333.00
325.50
318.00
310.50
303.00
B: Temp.
0.3
0.6
0.9
1.2
1.5
A: Inhibitor Conc.

(B)


Inhibition efficiency
79
67.75
56.5
45.25
34
3.00
2.50
2.00
1.50
1.00
C: Corrodent Conc.
0.3
0.6
0.9
1.2
1.5
A: Inhibitor Conc.

(C)



(D)

Figure 6: Response Surface Optimization Plots for Inhibition Efficiency using Design Expert version 10 (a) Predict versus actual plot for ML on Aluminum in KOH (b) Temp. versus Inhibitor conc. (c) Acid conc. versus Inhibitor conc. (d) Acid conc. versus Temp.

Statistical Analysis of the Corrosion Inhibition Study

The factorial model equations obtained for the inhibition efficiency of ML molecules on aluminum in KOH is expressed in equation 8

$(IE\ \%) = +169.58650 + 92.41500X_1 - 0.41467X_2 -1.52000X_3 0.17444X_1X_2 - 2.61667X_1X_3$ -------------- (8)

The mathematical model of inhibition efficiency (IE %) of *moringa leaf* extract as a function of the considered factors is expressed by Equation (5). A linear model described the relationship between the inhibition efficiency and the considered independent variables. The model in terms of coded factors predicted the response for given levels of each factor. The coded model showed the relative impact of the factors. From the analysis of variance (ANOVA), the model F-value of 348.56 implies the model is significant (Table 7). In this model, the value of R^2 (0.9942) is in conformity with Adj R^2 (0.9914) indicating accurate statistical model [21].Values of "Prob> F" less than 0.0500 indicate model terms are significant. The "Pred R-Squared" of 0.9853 is in reasonable agreement with the "Adj R-Squared and the difference is less than 0.2. Linear model is adequate for the description of the inhibition efficiency with the considered factors of the inhibition process. The final factorial model equations obtained for inhibition efficiency of ML on aluminum in KOH after eliminating the insignificant model terms becomes as expressed in equations (9).

$IE\% = +63.22 + 19.03X_1 - 8.58X_2 - 3.88X_3 - 1.57X_1X_2 - 1.57X_1X_3$ -------------------- (9)

Table 7: ANOVA analysis for inhibition efficiency of ML on corrosion of aluminium in KOH

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F	
Model	7286.827	5	1457.365	348.5686	< 0.0001	Significant
X_1-Inhibitor Conc.	5791.21	1	5791.21	1385.126	< 0.0001	
X_2-Temp.	1176.49	1	1176.49	281.3896	< 0.0001	
X_3-Corrodent Conc.	240.25	1	240.25	57.4623	< 0.0001	
X_1X_2	39.4384	1	39.4384	9.4328	0.0118	
X_1X_3	39.4384	1	39.4384	9.4328	0.0118	
Residual	41.81	10	4.181			
Lack of Fit	41.81	2	20.905	5.16036	0.0763	Not sign.
Pure Error	0	8	0			
Cor Total	7328.637	15				

Std. Dev.	2.0447	R-Squared	0.994295
Mean	63.22	Adj R-Squared	0.991442
C.V. %	3.2343	Pred R-Squared	0.985395
PRESS	107.033	Adeq Precision	50.2736

4. Conclusion

Based on the analysis of the results, it can be inferred that:

1. Moringa leaf extract exhibited optimum inhibition efficiency of 96.65 % in 3M KOH environment
2. Moringa leaf extract is highly efficient in surface treatment of aluminum sheet in KOH solution.
3. A linear model adequately explains the relationship between inhibition efficiency and the considered factors of the inhibition process.
4. Electrochemical study signifies that moringa leaf extract acted as a mixed type inhibitor

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