

Investigating the Inhibitive Powers of Orange Leaf Extort in Corrosion Studies of Mild Steel Metal in HCl

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Abstract: Continuous search for corrosion inhibitors, due to huge differences in the media encountered in industries remain a major point in corrosion eradication. The use of natural leaves extract (orange leaf extract) to inhibit corrosion has assumed great significance by its application in inhibiting corrosion in acidic environments. Natural leave extract (orange leaf extract) was investigated as corrosion inhibitor for mild steel using 0.1, 0.3 and 0.5 M HCl as corrodents. The inhibitive outcome of orange leaf extract on the corrosion of mild steel was scrutinized using weight loss technique at 30 to 60°C. In 0.1 M, 0.3M and 0.5M HCl as corrodents in 0.5M HCl. Results confirmed that inhibition efficiency improved with increase in concentration of orange leaf extract, decreased by increasing the acid concentration and increasing the temperature. Rate of corrosion increased by increasing the media concentration/ acid concentration, decreased in inhibitor concentration and decreased in acid concentration. Result got indicated that orange leaf extract is a good hinder for corrosion. Incorporation of 2g/lolo of inhibitor showed an effect in inhibiting corrosion. Response surface method in design expert software was used to design the experiment for the weight loss method. Temperature, time, inhibitor concentration as the measured factors while corrosion rate, inhibition efficiency and surface coverage as the expected response of the study. Disparity of reticence efficiency with concentration, temperature and time were analyzed using mathematical models determined by response surface methodology (RSM) of the design expert software. RSM is an effective way to achieve optimization by analyzing and modeling the effects of multiple variables and their responses and finally optimizing the process. Analysis of the data includes the 3-D graphical representations of the relationship between inhibition efficiency and factors of concentration, temperature and time. The mathematical models generated shown inhibition efficiency as a function of concentration, temperature and time. The interactions among the factors of concentration, temperature and time was identified.

Keywords: Corrosion inhibition, mild steel, orange leaves extract, weight loss, hydrochloric acid.

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I. Introduction

Corrosion reduction processes are inhibitory [1]. Metals are used in different situation in industries. They come in contact with aggressive environment which negatively affect their stability in service. This effect on metals is referred to as corrosion. A way of shielding metals against corrosion is to employ inhibitors. Corrosion steps are usually electrochemical, with features of a battery. Once metals atoms are bare to an environment which contains water molecules, electrons will be released, their ions will be positively charged ions with completed electrical circuit (Latanision and Searson, [2]. It is observed that most synthetic corrosion inhibitors are hazardous, the need to use a non-toxic, non-hazardous and biodegradable inhibitors are necessary. Using economical and green compounds as corrosion fighters for mild steel has been step up [3] and [4]. A natural inhibitors served this need. Plant extracts are inexpensive and can be got by simple extraction process. Extracts of naturally occurring products contain many compounds products contain many compounds which are biodegradable in nature. The development of technological uses for food residues may reduce the volume of waste discarded and increase the economic viability of appropriate waste management options. Reports have been made of using naturally occurring substances like ethyl ester of edible oil as corrosion inhibitors for metal in diverse media [5]. More over on the adsorption of inhibitors on the surface of the metal,

corrosion of metals were protected by inhibitors [6]. Some have reported the inhibition effect of non-toxic compounds on the metal corrosion and inhibition effect of amino acid on the steel [7]. The effect of some non-toxic organic compounds inhibition have been reported for steel [8] and [9].

This study was aimed at surveying the inhibitive effect of orange leaf extract on corrosion of mild steel in hydrochloric acid.

II. Experimental

2.1. MATERIAL PREPARATIONS

Mild steel used in this work were obtained in sheet form, from the Engineering workshop of the Federal University of Technology Owerri, Imo State. The sheets were pressed cut mechanically into 2cm x 2cm coupons respectively. tiny opening was pierced at one end of the coupons to allow their immersion into experimental solution. Coupons were not altered as cut but were degreased in ethanol, dehydrated in acetone, weighed and kept in free moisture desiccators before being used for corrosion examination. Inhibitor used was orange leaf extract. The leaves of orange were collected at Fegge-Onitsha, Anambra State, Nigeria. Leaves were dehydrated under the sun three days. After sun drying, they were crushed to elevate the surface area and kept in a closed container. 30 grams of the crushed orange leaves (fineness: particle size of 0.85 mm) measured and drenched inside 1000 ml of distilled water for 48 h. Towards the end of the of 48 h, the fusion was filtered. Filtrate got is a fusion of leaf extract and distilled water. Leaf extract got was weighed and stored for the corrosion study. Thereafter, five different concentrations of the leaves extract which include 2g/l, 4g/l, 6g/l, 8g/l and 10g/l were prepared.

2.2. Inhibitor preparation

Inhibitor utilized was Dangonyaro leaf extort. The leaves of orange plant was got at Fegge – Onitsha, Anambra State, Nigeria. The leaves were sun dried for three days. Dried leaves were ground to boost the surface area and kept in a blocked container. 30 grams of the ground dangonyaro leaves (fine : particule size of 0.85mm) were gauged and immersed in 100ml of distilled water for 48 h. The mix up was sieved at the end of the 48 h. The remains got was a blend of plant extract and distilled water. Extract got was weighed and stored for the corrosion studies. Five different concentrations of the leaves extract which include 2 g/l, 4 g/l, 6 g/l, 6 g/l, 8 g/l and 10 g/l were prepared.

2.3. Weight loss measurement

In weight loss method, 250 ml beakers containing blank solutions of 0.1 M, 0.3M and 0.5M of HCl were prepared. The test was performed in immersion in the test solution maintained at 30⁰C, 40⁰C, 50⁰C and 60⁰C respectively. Weighed metal coupons were perched in the glass with the help of wooden bars along with twines and immersed in a thermoseted water bath set at a given equilibrated study temperature stated above. Coupons were rescued by 24 hour gap.

From the results obtained, the following corrosion parameters were tabulated.

$$i. \text{ Corrosion rate (mm/yr)} = \frac{87,600 W}{\rho A t} \quad (1)$$

were W = weight loss in (g), ρ = density of the coupon (g/cm³), A = exposed surface area of the coupon (cm²) along with exposed time (h)

$$ii. \text{ Inhibition efficiency (I.E) \%} = (1 - CR_{in h} / CR_{blank}) \times 100 \quad (2)$$

CR_{in h} with CR_{blank} were corrosion rates with and with no inhibitor respectively. [10].

2.4. Weight loss method using response surface technique.

We used response surface technique in design expert software in designing the test for weight loss technique. Temperature, time, inhibitor concentration plus medium concentration were the deemed factors whereas corrosion rate, inhibition efficiency and surface coverage were the anticipated replies of the learn. RSM was employed to scrutinize the responses. ANOVA with graphical studies of the inhibition efficiencies were also examined. Mathematical terms of the coded were got. Models in terms of coded factors were employed to create predictions on the response for the factor. High points of the factor were coded as + while the low points of the factor were coded as - 1. Best inhibition parameter were got.

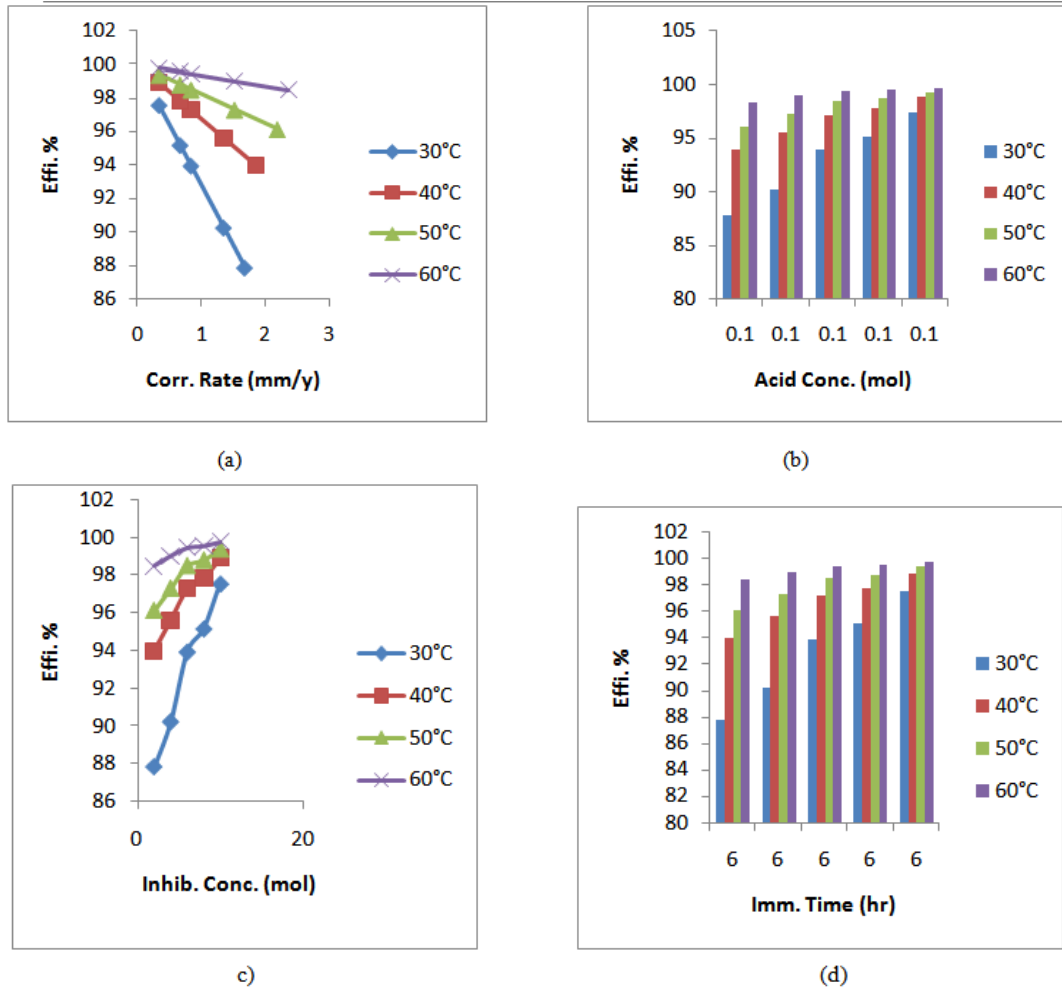
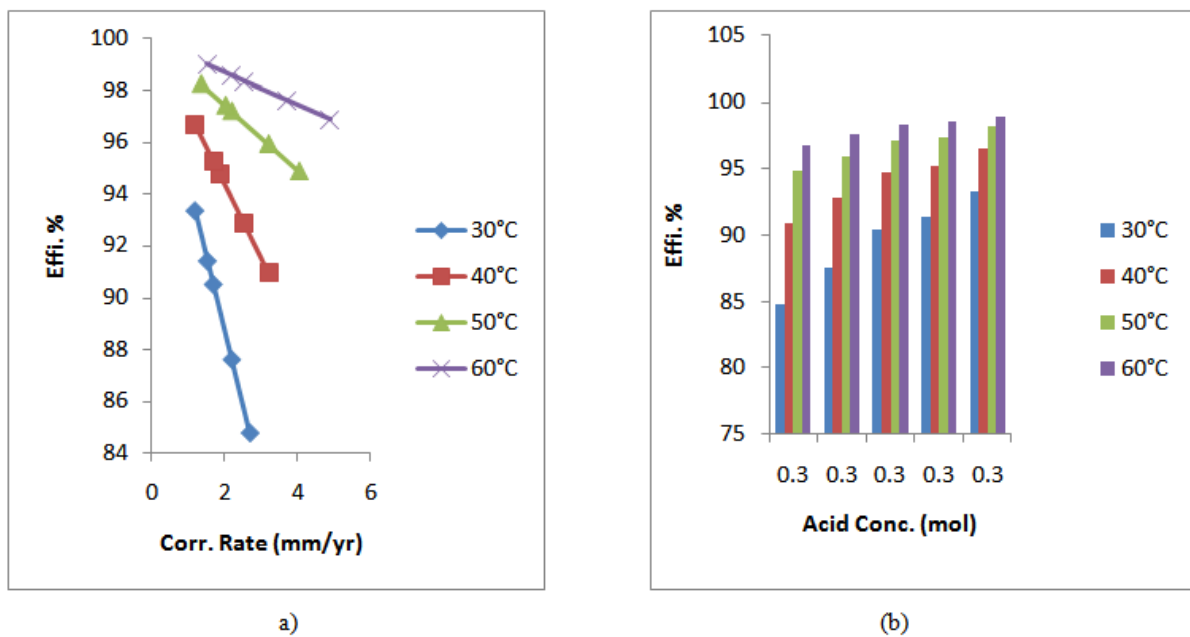


Fig. 1: Result of inhibitor concentration (a), immersion time (b), corrosion rate (c) and acid concentration (d) on the inhibition efficiency of M- steel in 0.1M HCl at different concentration of orange leaf extract inhibitor, and at varied temperatures.



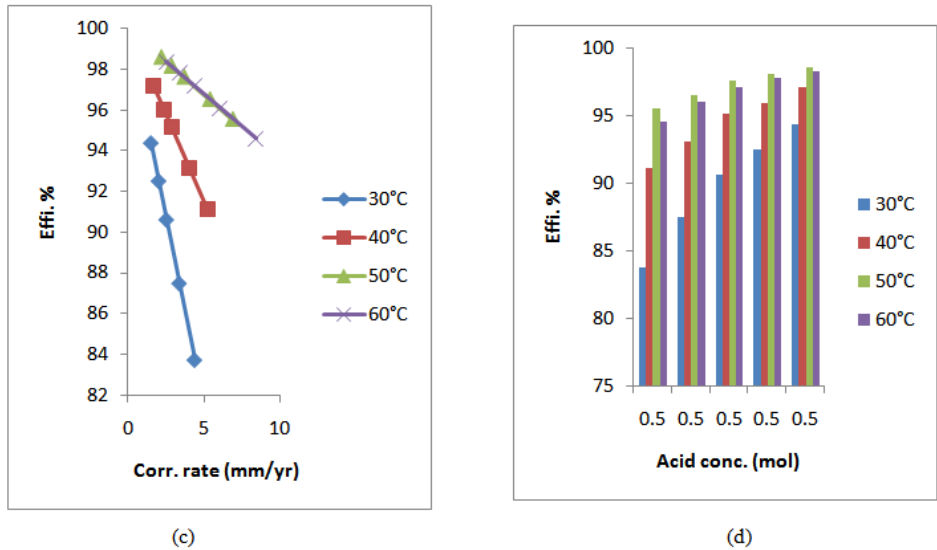


Fig. 2: Effect of inhibitor concentration (a), immersion time (b), corrosion rate (c) and acid concentration (d) on the inhibition efficiency of mild steel in 0.3M HCl at different concentration of orange leaf extract inhibitor, and at varied temperatures.

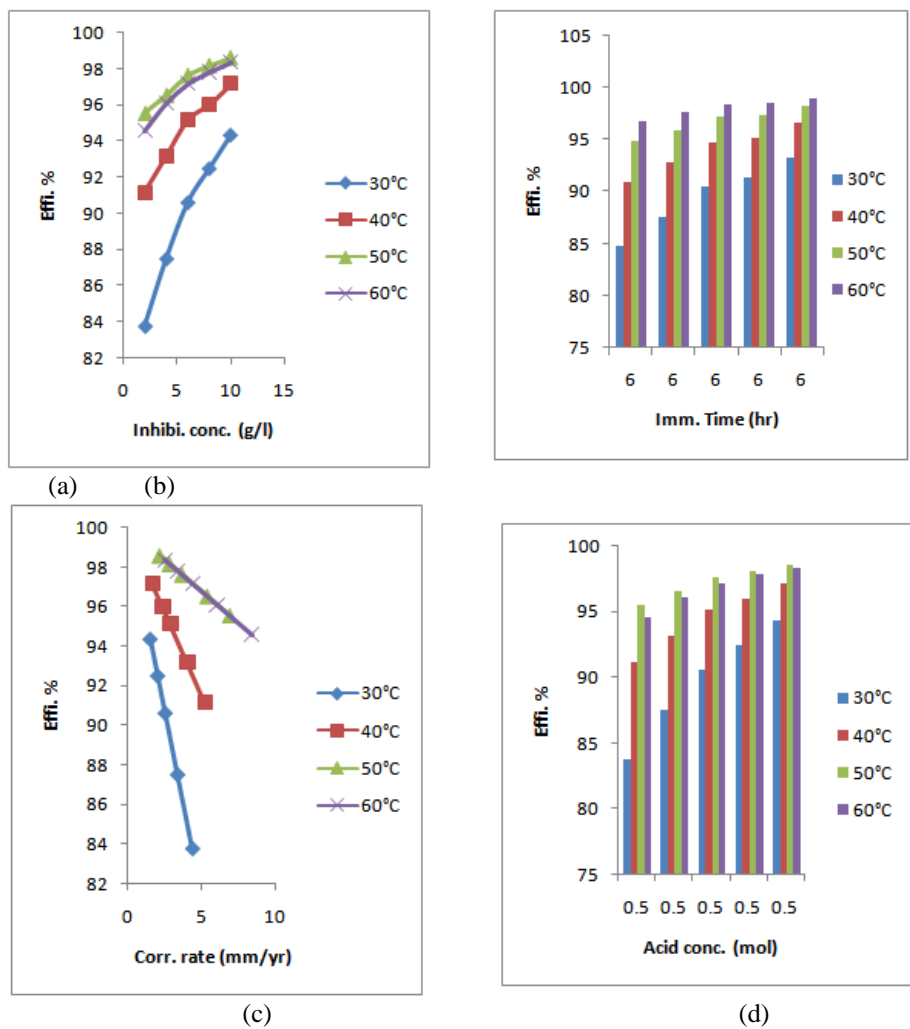


Fig. 3: Effect of inhibitor concentration (a), immersion time (b), corrosion rate (c) and acid concentration (d) on the inhibition efficiency of mild steel in 0.1M HCl at different concentration of orange leaf extract inhibitor, and at varied temperature.

III. Results And Discussion

3.1 Weight Loss Measurements

The corrosion of mild steel in 0.1 M, 0.3M and 0.3 M HCl was studied by weight loss method at 30⁰C, 40⁰C, 50⁰C and 60⁰C. The corrosion experiments was repeated at diverse concentrations of inhibitor and in each inhibitor concentration, mild steel weight loss was measured, values of corrosion rate, inhibitor efficiency and degree of surface coverage in 0.1,0.3 and 0.5 M HCl, with different concentration of inhibitor (orange leaves extract) as a role of time (hr) temperature (⁰C) concentration (g/l) efficiency (%) and corrosion rate (mm/yr) are available in table 1 below. Assessment of the figs. revealed that corrosion rate enhances with raise in the acid concentration / media concentration, raise in temperature (immersion time) and decrease in inhibitor concentration. Increase in the inhibition efficiency will lead to reduce in the rate of corrosion. Highest rate of corrosion of 8.42 (mm/yr) apart from the blank falls at the highest concentration of acid of 0.5M HCl . It's temperature was 60⁰C which was the highest temperature studied and at the list concentration of the inhibitor (orange leaf extract) of 2g/lole. This showed that the inhibitor concentration has an important role in inhibiting corrosion. The lowest corrosion rate 0.34 (mm/yr) was at the lowest acid concentration 0.1 M also at the peak concentration of the inhibitor of 10g/lole.

TABLE 1: Calculated values of corrosion rates (MM/Yr), inhibition efficiency (%) and degree of surface coverage (Θ) for mild steel corrosion in 0.1, 0.3 and 0.5 M HCl at diverse concentrations of inhibitors (orange leaf extract) and at various temperatures.

Time (Hour)	Inhibitor Conc. (g/L)	Corrosion rate (mm/yr)				Inhibition Efficiency (%I)				Degree of Surface Coverage (Θ)			
		30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
0.5 M HCl	BLANK	26.94	58.93	155.23	155.23								
	2g/LOLE	4.38	5.22	6.9	8.42	83.74	91.14	95.55	94.58	0.84	0.91	0.96	0.95
	4g/LOLE	3.37	4.04	5.39	6.06	87.49	93.14	96.53	96.1	0.87	0.93	0.97	0.96
	6g/LOLE	2.53	2.86	3.7	4.38	90.61	95.15	97.62	97.18	0.91	0.95	0.98	0.97
	8g/LOLE	2.02	2.36	2.86	3.37	92.5	96	98.16	97.83	0.93	0.96	0.98	0.98
	10g/LOLE	1.52	1.68	2.19	2.53	94.36	97.15	98.59	98.37	0.94	0.97	0.99	0.98
0.3 M HCl	BLANK	17.68	35.36	78.79	155.23								
	2g/LOLE	2.69	3.2	4.04	4.88	84.79	90.95	94.87	96.86	0.85	0.91	0.95	0.97
	4g/LOLE	2.19	2.53	3.2	3.7	87.61	92.85	95.94	97.62	0.88	0.93	0.96	0.98
	6g/LOLE	1.68	1.85	2.19	2.53	90.5	94.77	97.22	98.37	0.91	0.95	0.97	0.98
	8g/LOLE	1.52	1.68	2.02	2.19	91.4	95.25	97.44	98.59	0.91	0.95	0.97	0.99
	10g/LOLE	1.18	1.18	1.35	1.52	93.33	96.66	98.29	99.02	0.93	0.97	0.98	0.99
0.1 M HCl	BLANK	13.81	30.81	56.57	155.23								
	2g/LOLE	1.68	1.85	2.19	2.36	87.83	94	96.13	98.48	0.88	0.94	0.96	0.98
	4g/LOLE	1.35	1.35	1.52	1.52	90.22	95.62	97.31	99.02	0.9	0.96	0.97	0.99
	6g/LOLE	0.84	0.84	0.84	0.84	93.92	97.27	98.52	99.46	0.94	0.97	0.99	0.99
	8g/LOLE	0.67	0.67	0.67	0.67	95.15	97.83	98.82	99.57	0.95	0.98	0.99	1
	10g/LOLE	0.34	0.34	0.34	0.34	97.54	98.9	99.4	99.78	0.98	0.99	0.99	1

3.1 The result of the weight loss method using RSM

Replies of the corrosion rate, inhibition efficiency and surface coverage to the factors of temperature, inhibitor concentration and medium concentration for the corrosion inhibition of metals in media with orange leaves extract is accessible in Table 2. The data show the difference in corrosion rate with temperature, inhibitor concentration and medium concentration of mild steel metal in acid medium HCl. Corrosion rate increased with raise in temperature and media concentration. Decrease in corrosion rate with boost in concentration of the inhibitor indicates that inhibitor orange leaf subdued the corrosion of mild steel in acid medium. Raise in inhibitor concentration enhances the inhibition efficiency and surface coverage. Furthermore, inhibition efficiency shrinks as the temperature goes up.

Orange leaf extract has good efficiency. The high inhibition efficiency recorded in the medium may be attributed to the actions of protonated and molecular species. Some of the constituents of the inhibitors may have been adsorbed as protonated species and some molecular species [11] and [12].The presence of C-Cl stretch, N=O bend, C=C stretch, O-H stretch functional group, C-H stretch of C=O aldehydes, H-C-H asymmetric stretch, N-H symmetric and NH stretch contributed to this. This high values of the inhibition efficiencies indicated that the orange leaf used is good for the treatment of the metal surface. It has the capacity to inhibit corrosion when employed as corrosion inhibitor in metallic maintenance operations.

The disparity of inhibition efficiency with concentration, temperature and time were analyzed using mathematical models got by response surface technique (RSM) of the design expert software. RSM is an effective way to accomplish optimization by examining and modeling the effects of multiple variables and their

answers and finally optimizing the procedure. Analysis of the data includes the 3-D graphical representations of the relationship between inhibition efficiency and factors of concentration, temperature and time. The mathematical models generated shown the inhibition efficiency as a function of concentration, temperature and time. The interactions among the factors of concentration, temperature and time was identified.

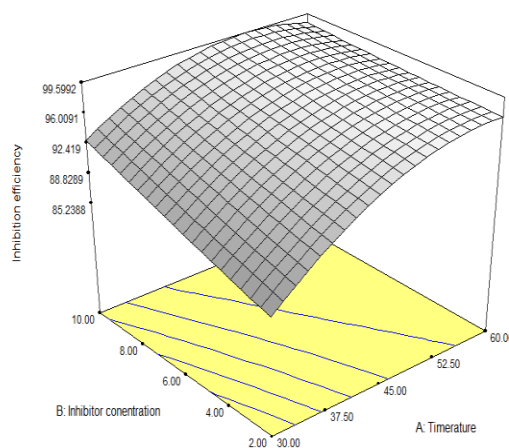
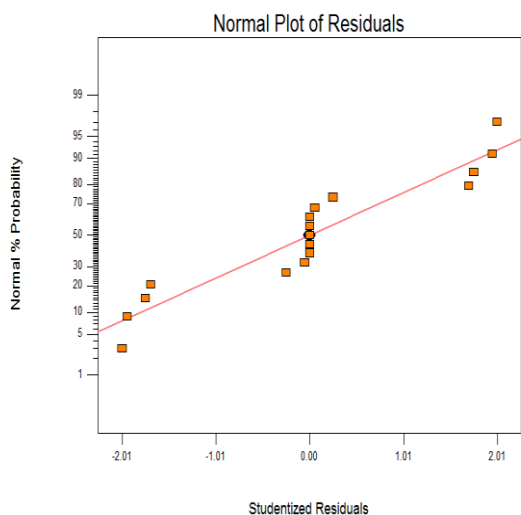
Table 2 : The RSM result of the corrosion inhibition of St. in HCl by orange leaves extract.

Std	Run	Factor 1; A: Tempt. oC	Factor 2; B: Inhibior conc. (g/l)	Factor 3; C : Medium conc.(M)	Response 1; Corrosion rate (mm/yr)	Response 2; Inhibitor efficiency (%)	Response 3; Surface coverage (K)
15	1	45.00	6.00	0.30	2.02	96.65	0.97
17	2	45.00	6.00	0.30	2.02	96.65	0.97
14	3	45.00	6.00	0.30	2.02	96.65	0.97
7	4	30.00	6.00	0.50	2.53	90.61	0.91
8	5	60.00	6.00	0.50	4.38	97.18	0.97
9	6	45.00	2.00	0.10	2.11	94.96	0.95
10	7	45.00	10.00	0.10	0.34	99.21	0.99
3	8	30.00	10.00	0.30	1.18	93.33	0.93
4	9	60.00	10.00	0.30	1.53	99.02	0.99
6	10	60.00	6.00	0.10	0.84	99.46	0.99
5	11	30.00	6.00	0.10	0.84	88.07	0.88
1	12	30.00	2.00	0.30	2.69	84.79	0.85
11	13	45.00	2.00	0.50	6.14	96.04	0.96
2	14	60.00	2.00	0.30	4.88	96.86	0.97
12	15	45.00	10.00	0.50	2.02	98.7	0.99
16	16	45.00	6.00	0.30	2.02	96.65	0.97
13	17	45.00	6.00	0.30	2.02	96.65	0.97

3.2 Graphical investigation of the Inhibition Efficiency, IE (%), as resolute with RSM

Investigation of the inhibition efficiencies of orange leaves (inhibitors) of the metal in the acidic are presented in fig. 4. The graphs of inhibition efficiency of inhibitor orange (steel in HCl), are shown in figures 4. Plots of Normal Plots of Residuals versus Studentized Residuals were used to analyzed the importance of the models order. In all the cases studied, the residuals versus studentized residuals gave linear graphs. The graphs (3-D surface plots) showed the relationship between the factors and responses of designed experiments. Factors comprised of inhibitor concentration, temperature and time of metal immersion. Response is the inhibition efficiency. Corrosion inhibition of metal steel and in acid medium HCl with plant extracts (orange leaf) showed that raise in concentration enhances the inhibition efficiency. Moreover, inhibition efficiency trims down as temperature goes up. Surveillances are in accord with earlier studies [13].

Graphs of the inhibition efficiency versus concentration, temperature and time are in quadratic forms. Further analysis of the data will addressed in terms of analysis of variance, mathematical models and optimization .The analysis of variance helped in identifying the models significance inhibitors as a terms. The powers of the variables were used to confirm the quadratic models. The models displayed the inhibition efficiency of the role of factors of concentration, temperature with time. Interactive behavior of the concentration, temperature and time were also identified.



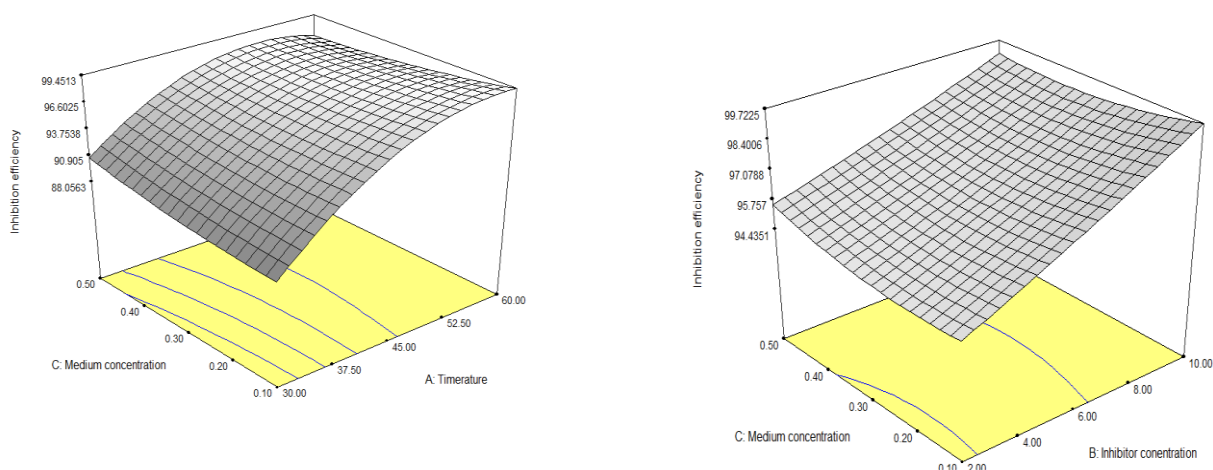


Fig.4 : IE (%) of orange leaves extracts as corrosion inhibitor of steel in HCl

a) Normal Plot of Residuals versus Studentized Residuals IE (%), b) IE (%) versus inhibitor concentration and temperature, c) IE (%) versus medium concentration and temperature, d) IE (%) versus medium concentration and inhibitor concentration.

From the ANOVA for the corrosion rate of Steel in HCl by orange, model F- value of 111.75 means that the model is noteworthy (Table 3). Here just a 0.01 possibility that an F- value this large could occur owing to noise. Values of “Prob > F” less than 0.5000 indicate model terms are noteworthy. Here A, B, C, A², AB, AC, BC are noteworthy model terms. Values larger than 0.1000 specify the model terms are noteworthy. “Pred R-Squared” of 0.8894 is in reasonable consent with the “Adj R-Squared” of 0.9842; the dissimilarity is less than 0.2. “Adeq Precision” gauges the indicator owed to noise ratio. Ratio larger than 4 is enviable. The ratio of 37.112 in designates an adequate signal. The model can be employed to steer the design space.

Table 6: ANOVA for the corrosion rate of Steel in HCl by orange.

ANOVA for Response Surface Quadratic model ; corrosion rate						
Analysis of variance table [Partial sum of squares – Type III]						
	Sum of		Mean	F	p-value	
Source	Squares	Df	Square	Value	Prob > F	
Model	36.16	9	28.94	111.75	< 0.0001	Significant
A- Temperature	2159.49	1	159.49	615.95	< 0.0001	
B- Inhibitor conc	38.76	1	38.76	149.71	< 0.0001	
C- Medium conc.	0.086	1	0.086	0.33	0.5822	
A ²	45.13	1	45.13	174.28	< 0.0001	
B ²	0.064	1	0.064	0.25	0.6331	
C ²	0.87	1	0.087	3.35	0.1100	
AB	10.18	1	10.18	39.30	0.0004	
AC	5.81	1	5.81	22.42	0.0021	
BC	0.63	1	0.63	2.44	0.1622	
Residual	1.81	7	0.26			
Lack of Fit	1.81	3	0.60			
Pure Error	0.000	4	0.000			
Cor Total	262.23	16				
Std. Dev.	0.51		R-Squared			0.9931
Mean	95.38		Adj R-Squared			0.9842
C.V. %	0.53		Pred R-Squared			0.8894
PRESS	29.00		Adeq Precision			37.112

3.3. Mathematical models of the inhibition efficiency

Mathematical models of inhibition efficiency

The mathematical models for the inhibition efficiency of orange leaves extract as corrosion inhibitor of metals in acid medium are shown in Equations 3. The model was obtained by employing RSM tools of expert design software. Each model showed the bond between the inhibition efficiency (IE), inhibitor concentration (A), temperature (T) and time or day (°C). The mathematical models in terms of coded factors were obtained using the optimization tool of the RSM. The models in stipulations of the coded factors predicted the reaction for given levels of each factor.

Positive signs in the models signified synergistic effect, while the negative signs signified antagonistic effect.

The model for the corrosion inhibition of mild steel in HCl by orange leaves extract is;

$$IE = +96.65 + 4.46 *A + 2.20 *B + 10.10 *C - 3.27 *A^2 + 0.12 *B^2 + 0.45 *C^2 - 1.59 *A*B - 1.20 *A *C - 0.40 *B *C \quad (3)$$

3.4 Optimum Parameter for Corrosion Inhibition of Mild steel Metal

Data on Table 4 show the optimum inhibition concentration, optimum temperature, optimum time and optimum inhibition efficiency for M- steel metal in HCl medium. Analysis of the optimum inhibition efficiency of dangonyaro leaf extract showed 99.46%

Table 4 : Optimum parameter for corrosion inhibition of M- steel in HCl with dangonyaro leaf extort.

Media	Inhibitor (plant extract)	Optimum inhibitor conc. (g/l)	Optimum temperature oC	Optimum medium conc. (M)	Optimum inhibition efficiency (%)
HCl	Orange leaves	10	60	0.5	99.46

3.5 Validation of the result

Confirming the validity of result, added experiment was performed. The state of the concentration, temperature and time is recorded in Table 5, along with the foreseen and calculated inhibition efficiency. Whilst revealed in the Table 5, calculated inhibition efficiency was closed to the foreseen value. It illustrates that RSM approach was suitable for optimizing the corrosion inhibition process.

Table 5 : Result validation for corrosion inhibition of M- steel in HCl medium with plant extract

Media	Innhibitor (plant extract)	Inhibitor conc. (g/l)	Temperature (o C)	Medium conc. (M)	Predicted inhibition efficiency (%)	Measured inhibition efficiency (%)	Percentage error (%)
HCl	Orange leaves	10	60	0.5	99.46	99.24	0.22

IV. Conclusion

From this investigation, the following conclusions can be drawn

1. Mild steel corrode in 0.1, 0.3 and 0.5 M HCl
2. The weight loss of mild steel in HCl depends on the concentration of acid and it corroded and even dissolve in HCl without the incorporation of the orange leaf extract as inhibitor
3. Orange leaf extract is a good reducer of corrosion in HCl and it is environmental friendly, zero cost and can easily obtained.
4. Orange leaf extract can be used to replace toxic chemical inhibitors

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