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Characterization and Optimization study of *Epiphyllum oxypetalum* extract as corrosion inhibitor for mild steel in 3 M H_2SO_4 solutions

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ABSTRACT

This study investigated characterization of *Epiphyllum oxypetalum* (EO) leaf extracts and optimization of process variables on inhibition of mild-steel in 3 M H_2SO_4 aggressive environment. Response Surface Methodology (RSM) was employed to examine the influence of process variables namely acid concentration (1.0 – 3.0 mols), inhibitor concentration (0.2 – 1.0 g/l), time (1–10 hr) and temperature (303 -343K) on inhibition efficiency. The results of phytochemical screening showed that organic components present in EO extract as a good inhibitor. Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM) studies established the presence of active compounds and adsorption of leaf extracts on metal surface. The experimental data was statistically analyzed and regression equations for corrosion rate and inhibition efficiency generated. The optimum conditions were achieved at acid concentration of 1.00 mol, inhibitor concentration of 0.055 g/l temperature of 333K, and time of 2.750 hr. The optimization results revealed that inhibition efficiency of 82.93 % was obtained at optimum combination of the extract concentration, the acid concentration, the temperature and the immersion time. The inhibitive potentials of EO extract was found to impede the localized and uniform corrosion of mild steel in acidic media, showing that the *Epiphyllum oxypetalum leaf* extract is a good inhibitor for mild steel in acidic solutions.

***Keywords*:** Acid corrosion, *Epiphyllum oxypetalum* extract, Optimization, Photochemical, Response Surface Methodology

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1. INTRODUCTION

Mild steel is widely used in engineering and industrial applications for construction and other services across the world. The destructive nature of some environments most times causes the attack of these metal surfaces by corrosion, and this has been a source of great concern globally. In order to prevent this serious attack on those metals, corrosion inhibitors are utilized to decrease the rate of corrosion as well impede the corrosion reaction [1-5].

Due to growing environmental regulations and ecological awareness for sustainable and environmentally friendly environments, attention is concentrated on developing biodegradable, nontoxic, environmentally friendly substitutes to organic and inorganic corrosion inhibitors used in the past. Subsequently, the present study in corrosion inhibition work is to find an emerging new benevolent, relatively cheap and nontoxic substitutes. As a result, attention have been drawn to developing/ investigating green products for corrosion protection [28-34].

This type of research are done by ascertaining the phytochemical properties of the compounds, electronic and molecular structures which are comparable with the conventional organic inhibitor molecules [6-10].

Again, green plant products are renewable, low-cost sources of materials and readily available. Apart from numerous species/varieties of green products and its great availability, very little have been investigated upon. This work will center on increasing the awareness of using green plant extracts as corrosion control for metals in acid solutions. Hence, the inhibiting effect of *Epiphyllum oxypetalum* extract on mild steel in 3 M H_2SO_4 solutions. The aim of this current report is to use a statistical tool known as response surface methodology (RSM) to determine, as well as optimize the corrosion inhibition efficiency of the plant extract on mild steel in acidic media

2. EXPERIMENTAL PROCEDURE

2. 1. Sample preparations

Mild steel specimen comprising of P (0.02%), Mn (0.11%), Si (0.02%), S (0.02%), Cu (0.01%), C (0.23%), Ni (0.02), Cr (0.01%) and Fe (99.56 %) were mechanically cut into coupons of 5 cm × 4 cm in size with thickness of 0.1 cm. The coupons were cleaned followed by polishing with emery paper of different grades (200, 400, 600, 800 and 1000) to expose shining polished surface.

In order to remove any oil and organic impurities, the coupons were degreased with acetone and finally washed with distilled water, dried in air and then stored in desiccators. Accurate weight of each coupon was taken in duplicate using electronic weighing balance and the initial weight was recorded. The coupons were labeled in a manner to avoid any mix up.

2. 2. Extraction of *Epiphyllum oxypetalum* extract

The leaves of *Epiphyllum oxypetalum* were collected from Awka in 2019. The botanical identification was done at Faculty of Biosciences, Nnamdi Aziliwe University Awka. The EO leaves was dried at room temperature for 2 days.

The surface area of dried EO leaves were increased into powdery form and immersed completely in ethanol for 48hrs. The solution was filtered, concentrated and prepared into different concentration of 3 M H_2SO_4 solution.

2. 3. Characterization of *Epiphyllum oxypetalum* extract

2. 3. 1. Phytochemical analysis of EO extract

Qualitative and Quantitative analysis of the plant extract were done to find out the presence and quantity of the active constituents like alkaloids, flavonoids, saponins, tannins in EO extract. Methods used by [11-12] were adopted for the phytochemical analysis of the plant extract.

2. 3. 2. FTIR analysis

FTIR spectrophotometer is a powerful instrument used in identifying the type of bonding especially functional groups existing in an organic compounds [13]. In this study, FTIR (SHIMADZU, Model: IR affinity - 1; S/N: A2137470136 SI) was used in identifying the functional groups on mild steel with inhibitor at optimal process variables and Mild Steel with inhibitor which had the highest inhibition efficiency.

2. 3. 3. Scanning Electron Microscope (SEM) analysis

Surface morphology of the mild steel with inhibitor the optimal process variables and mild steel with inhibitor having the highest inhibition efficiency were inspected using Scanning Electron
Microscope.

2. 4. Weight Loss Measurement Using Response Surface Methodology

Response surface method of design expert 10.0.0 software was used to design the experiment for the weight loss method. The process variables such as acid concentration, Inhibitor concentration, temperature and time were considered while weight loss, corrosion rate and inhibition efficiency were the expected responses of the study. The RSM was used to analyze the responses. The ANOVA and graphical analyses of the inhibition efficiencies were carried out. The mathematical models in terms of coded values were obtained. The models in terms of coded factors were used to make predictions about the response for given levels of each factor. The high levels of the factors were coded as +1 and the low levels of the factors were coded as -1 as shown on Table 1. Optimum inhibition parameters were also obtained.

Table 1. Independent variables and levels used for response surface design.

Independent variables	Symbol	Ranges and levels				
		-α	**-1**	**0**	**+1**	**+α**
Acid concentration (mols)	A	1.0	1.5	2.0	2.5	3.0
Inhibitor concentration (g/L)	B	0.2	0.4	0.6	0.8	1.0
Reaction time (hr)	C	2	4	6	8	10
Temperature (K)	D	303	313	323	333	343

2. 5. Experimental design

Response Surface Methodology (RSM) was employed to investigate the interaction among temperature, acid concentration, inhibitor concentration and reaction time on the corrosion inhibition of mild steel in 3 M H_2SO_4. The 5 level -4- factor Central Composite Design (CCD) has been employed in this study requiring 30 experiments, consisting of 2^4 factorial points, 8 star points, coded α and 6 central points were carried out. The levels of each independent variable were chosen based on the importance of experiments. The coded and corresponding uncoded values were given in Table 1.

The corresponding central composite design and their values were shown in Table 2. The experimental data obtained (weight loss, corrosion rate and inhibition efficiency) from central composite design were analyzed by response surface methodology. A mathematical model, following a second –order polynomial Equation (1) which includes interaction terms was used to calculate the predicted response [14-16].

$$Y = \beta_0 + \sum_{i=1}^{K} \beta_i X_i + \sum_{i=1}^{K} \beta_{ii} X^2{}_i + \sum_{i<j} X_i X_j + e \quad (1)$$

Equation (2) reveals the quadratic equation response of inputs variables x_1, x_2, x_3, and X_4 is;

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{14} X_1 X_4 + \beta_{23} X_2 X_3 + \beta_{24} X_2 X_4 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{44} X_4^2 \quad (2)$$

where Y is the response predicted by the Response Surface Methodology, i is the linear coefficient and j is the quadratic coefficients, regression coefficient is β, whereas k is the studied parameters, and optimized in the experiment, and the random error is e;

Table 2. RSM result of corrosion inhibition of mild steel in 3 M H_2SO_4 using *Epiphyllum oxypetalum* leaves extract.

Std	Run	Factor 1	Factor 2	Factor 3	Factor 4	Response 1	Response 2	Response 3
		Acid conc	Inhibitor conc	Time	Temperature	Weight loss	Corrosion rate	Inhibitor efficiency
		(mols)	(g/l)	(hrs)	(K)	(g)	g/cm^{2}hr)	(%)
15	1	1	0.055	6.25	333	0.45	5.17	61.63
28	2	1.5	0.04	4.5	323	0.41	6.83	69.63
25	3	1.5	0.04	4.5	323	0.28	6.83	68.58
7	4	1	0.055	6.25	313	0.98	7	68.56
21	5	1.5	0.04	1	323	0.53	5.5	49.63
11	6	1	0.055	2.75	333	0.45	6.83	83.37

20	7	1.5	0.07	4.5	323	0.41	6.83	67.63
19	8	1.5	0.01	4.5	323	0.28	14	55.88
1	9	1	0.025	2.75	313	0.98	9.8	44
10	10	2	0.025	2.75	333	0.53	8.83	72.41
24	11	1.5	0.04	4.5	343	0.59	5.9	68.29
23	12	1.5	0.04	4.5	303	0.863	8.63	46.4
26	13	1.5	0.04	4.5	323	0.41	6.83	69.63
4	14	2	0.055	2.75	313	0.42	21	40
22	15	1.5	0.04	8	323	0.86	14.33	56.3
2	16	2	0.025	2.75	313	0.33	16.5	37.74
6	17	2	0.025	6.25	313	0.41	6.83	67.63
27	18	1.5	0.04	4.5	323	0.3	3	68.37
13	19	1	0.025	6.25	333	0.24	12	50
12	20	2	0.055	2.75	333	0.31	5.17	75.4
5	21	1	0.025	6.25	313	0.45	6.83	67.63
14	22	2	0.025	6.25	333	0.41	6.83	59.63
18	23	2.5	0.04	4.5	323	0.28	7	73.58
8	24	2	0.055	6.25	313	0.98	5.5	69.56
30	25	1.5	0.04	4.5	323	0.53	6.83	69.63
9	26	1	0.025	2.75	333	0.45	6.83	73.37
17	27	0.5	0.04	4.5	323	0.41	6.83	75.63
29	28	1.5	0.04	4.5	323	0.28	14	68.88
3	29	1	0.055	2.75	313	0.98	9.8	48
16	30	2	0.055	6.25	333	0.53	8.83	62.41

$X_1 = A$; $X_2 = B$; $X_3 = C$; $X_4 = D$;

2. 5. 1. Corrosion Inhibition Efficiency and Statistical Analysis

Analysis of variance ANOVA, regression analysis and response surface plots of the interaction factors were obtained by analyzing the experimental responses (weight loss, corrosion rate and inhibition efficiency) with Design Expert 10.0.3 software. The interactive effects of the process variables which include polynomial, quadratic and linear effects on the responses were further analyzed by ANOVA test in order to determine their individual effect. The significance of the regression coefficient was tested using P-value (≤ 0.05).

The adequacy of the model and the regression model were measured by comparing the actual values of predicted R-Squared and R-squared. Hence, the relationship between the actual (experimental result) and predicted responses were assessed and analyzed by graphical representation of the predicted responses (weight loss, corrosion rate and inhibition efficiency) and actual data. Hence, the relationship between the actual (experimental result) and predicted responses were assessed and analyzed by graphical representation of the predicted responses (weight loss, corrosion rate and inhibition efficiency) and actual data.

3. RESULTS AND DISCUSSION

3. 1. Result of Weight Loss measurement using Response Surface Methodology

The responses expected from the independent variables (acid concentration, inhibitor concentration, temperature and time) during corrosion process of *Epiphyllum oxypetalum* extract as corrosion inhibitor of mild steel in 3 M H_2SO_4 solutions were weight loss, corrosion rate and inhibition efficiency and the outcome are presented in Table 2. It can be seen from Table 2 that the maximum Inhibition efficiency of 83.37% was obtained at temperature of 333K, acid concentration of 1.0 mols, inhibitor concentration of 0.055 g/l and time of 2.75hrs. This reveals that at high temperature, low inhibitor concentration of 0.055 g/l and time duration of 2.75hrs high inhibition efficiency was achieved implying that the surface coverage by the extract performed well at low conditions [17-18].

3. 2. Result of Corrosion Inhibition Efficiency and Statistical analysis

The significance and consistency of inhibition efficiency results were screened by Statistical analysis. Table 3 presents analysis of variance, ANOVA, for corrosion inhibition of mild steel in 3 M H_2SO_4 using *Epiphyllum oxypetalum* leaves extract. The table shows that from fourteen factor interactions analyzed eight were are significant using P-Value of ≤ 0.05 test. The significant factors include inhibitor concentration, temperature and other interaction factors (AB, BC, BD, C^2, D^2) showing that inhibitor concentration and temperature exert abundant effect on inhibition efficiency of the corrosion process.

The Model F-values 74.84 implies the model is significant and there is only a 0.01% chance that an F-value this large could occur due to noise. The values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case AB, BC, BD, C^2, D^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The "Pred R-Squared" of 0.9439 is in reasonable agreement with the "Adj R-Squared" value of 0.9838 that is the difference is less than 0.2. Since "Adeq Precision" measures signal to noise ratio and a ratio greater than 4 is desirable.

Therefore, we conclude that Adeq precision of 30.315 implied adequate signal. Subsequently, this model can be used to predict the process inhibition efficiency of *Epiphyllum oxypetalum* extract as corrosion inhibitor of mild steel in 3 M H_2SO_4 solutions. This implies also that the results are statistically reliable and the second order polynomial model equation in terms of the coded values of the process variables are presented in Equations (3):

$$Y = -4212.18 + 79.53A - 6138.62B + 17.71C + 260D + 500.58A*B + 1.44A*C - 0.28A*D - 264.97B*C + 20.03B*D + 0.01C*D - 5.56A + 3662.50B^2 - 1.55C^2 - 0.04D^2 \quad (3)$$

After removing p-values > 0.05 which are considered to be insignificant terms from the regression model equation. Equation (3) becomes

$$Y = 4230.51 + 7953A - 6138.62B + 21.78C + 26.25D + 500.58A*B + 1.44A*C + 0.28A*D - 264.97B*C + 20.03C*D - 5.56A^2 + 3662.50B^2 - 1.55C^2 - 0.04D^2 \quad (4)$$

where A represent acid concentration, B is inhibitor concentration, C is time and D is temperature.

3. 2. 1. Graphical analysis of the inhibition efficiency, IE (%), using RSM

The graphical analysis or 3-D surface plots was employed in this work to determine the correlation between the process factors (time, temperature, inhibitor and acid concentrations) and response (inhibition efficiency) using design expert 10.0 software. The 3-D surface plot of the inhibition efficiency of the EO plant extracts with mild steel in aggressive environments H_2SO_4 were presented in Figure 1(a-f). Plot of predicted values versus actual experimental values Figure 1a were used to test the significance of the model. The predicted versus actual plot gave linear relationship indicating that the actual experimental values are in consistent with the predicted. Corrosion inhibition of the metals in acidic environments showed that increase in concentration increases the inhibition efficiency. However, inhibition efficiency reduces as temperature increases. These observations are in agreement with previous studies of [19-21].

Table 3. ANOVA for corrosion inhibition of mild steel in 3 M H_2S0_4 using *Epiphyllum oxypetalum* leaves extract.

ANOVA for Response Surface Reduced Quadratic model						
Analysis of variance table [Partial sum of squares - Type III]						
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value Prob > F**	
Model	2950.83	13	226.99	74.84	< 0.0001	significant
A-Acid conc	37.38	1	37.38	12.32	0.0029	
B-Inhibitor concentration	184.76	1	184.76	60.92	< 0.0001	
C-Time	27.03	1	27.03	8.91	0.0087	

D-Temperature	448.68	1	448.68	147.94	< 0.0001	
AB	225.53	1	225.53	74.36	< 0.0001	
AC	25.58	1	25.58	8.43	0.0104	
AD	32.35	1	32.35	10.67	0.0049	
BC	774.09	1	774.09	255.23	< 0.0001	
BD	144.54	1	144.54	47.66	< 0.0001	
A^2	53.16	1	53.16	17.53	0.0007	
B^2	18.63	1	18.63	6.14	0.0247	
C^2	618.78	1	618.78	204.02	< 0.0001	
D^2	451.22	1	451.22	148.78	< 0.0001	
Residual	48.53	16	3.03			
Lack of Fit	31.84	11	2.89	0.87	0.6097	not significant
Pure Error	16.68	5	3.34			
Cor Total	2999.35	29				

Std. Dev.	1.74	R-Squared	0.9838
Mean	58.97	Adj R-Squared	0.9707
C.V. %	2.95	Pred R-Squared	0.9439
PRESS	168.41	Adeq Precision	30.315
-2 Log Likelihood	99.56	BIC	147.18
		AICc	155.56

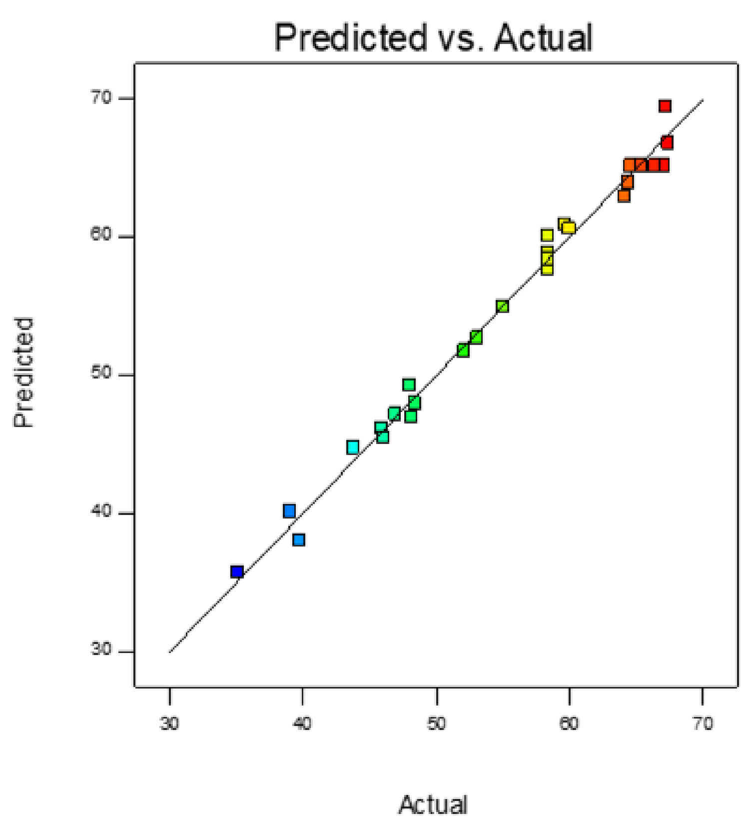
The graphs of the inhibition efficiency versus inhibitor concentrations and acid concentrations, temperature and time are in quadratic forms. Analysis of variance, mathematical model equations and optimization were also used to elucidate more on the inhibition efficiency. The analysis of variance helped in identifying the model significance terms. The powers of the variables were used to confirm the quadratic models. The 3-D graphs displayed the inhibition efficiency of the plant extract as a function of factors of acid concentration, inhibitor concentration, temperature and time. The interactive behaviour of the acid concentration, inhibitor concentration, temperature and time were equally identified.

3. 2. 2. Optimum conditions

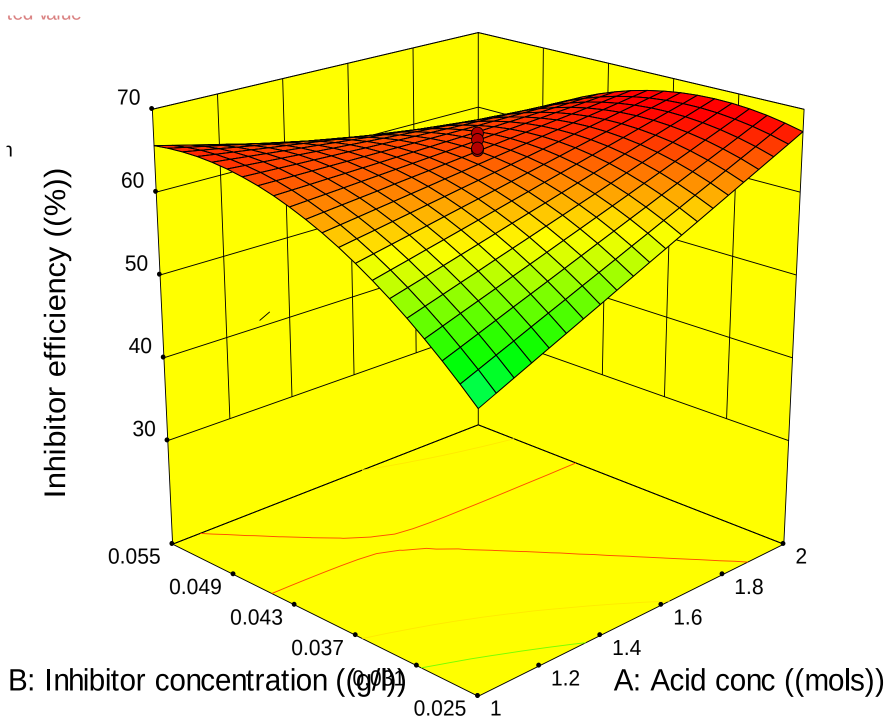
The high value of inhibition efficiency in Table 4 indicate that the plant extract is good for surface treatment of metals in corrosion control operations.

Table 4. Optimum conditions for corrosion inhibition of mild steel in acid using EO leaves extracts.

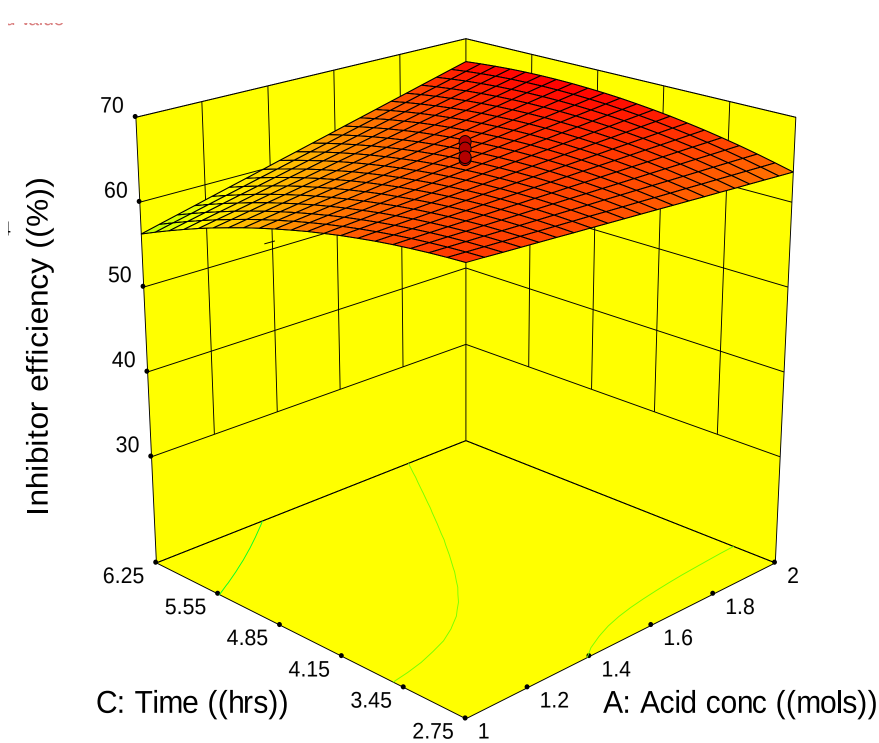
Acid concentration (mols)	Inhibitor concentration (g/l)	Temperature (K)	Time (hr)	Inhibition efficiency (%)
1.000	0.055	333.000	2.750	82.926



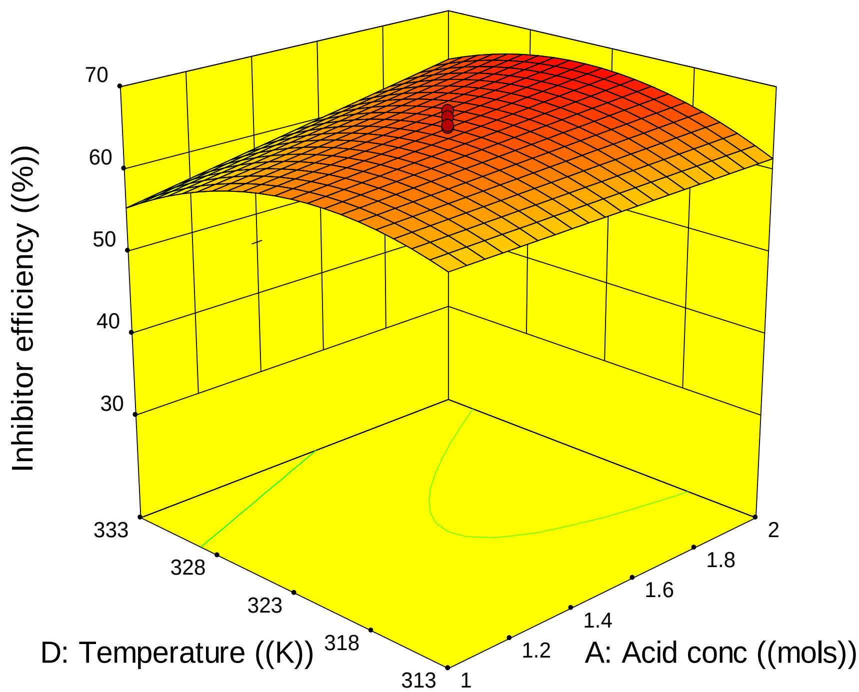
(a)


Inhibitor efficiency ((%))
70
60
50
40
30
0.055
0.049
0.043
0.037
B: Inhibitor concentration ((g/l)) 0.031
0.025
1
1.2
1.4
1.6
1.8
2
A: Acid conc ((mols))

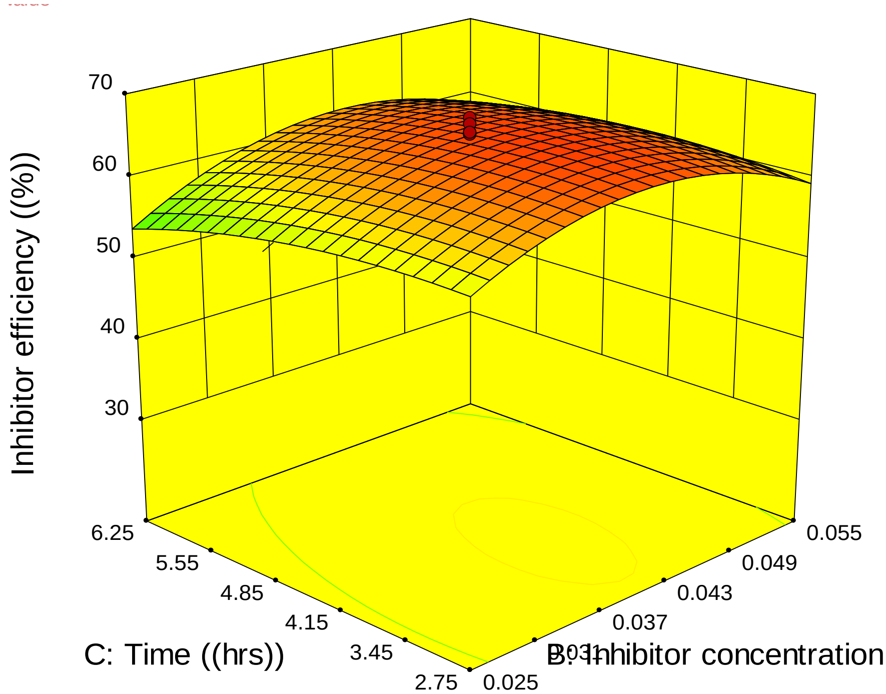
(b)


Inhibitor efficiency ((%))
70
60
50
40
30
6.25
5.55
4.85
4.15
3.45
2.75
C: Time ((hrs))
1
1.2
1.4
1.6
1.8
2
A: Acid conc ((mols))

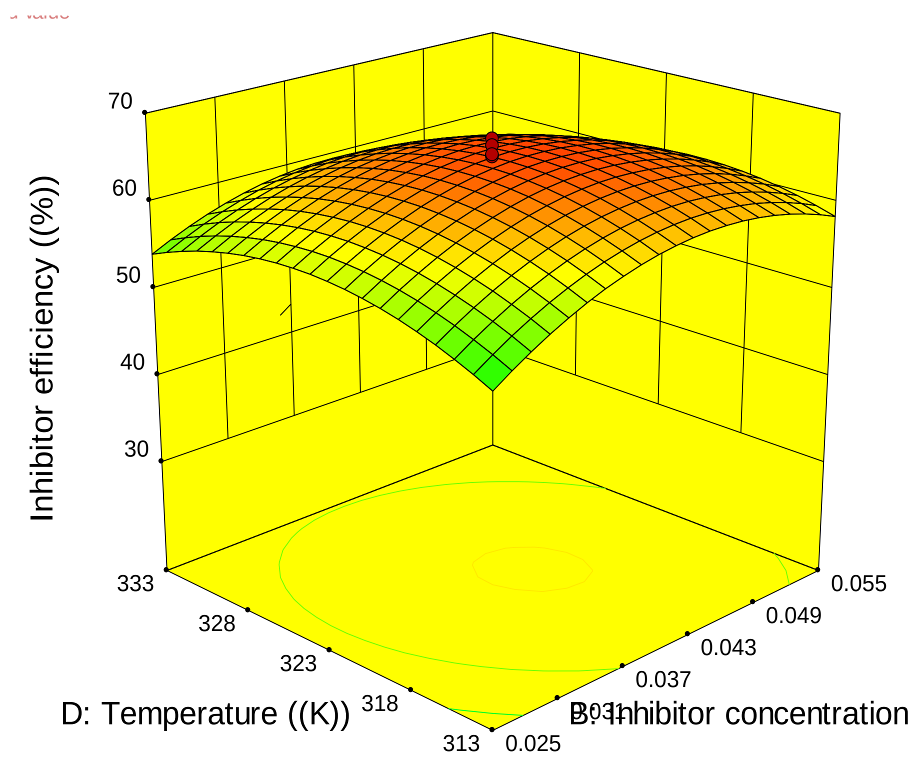
(c)


Inhibitor efficiency ((%))
70
60
50
40
30
333
328
323
318
313
D: Temperature ((K))
1
1.2
1.4
1.6
1.8
2
A: Acid conc ((mols))

(d)


Inhibitor efficiency ((%))
70
60
50
40
30
6.25
5.55
4.85
4.15
3.45
2.75
C: Time ((hrs))
0.025
0.031
0.037
0.043
0.049
0.055
B: Inhibitor concentration

(e)



(f)

Figure 1. IE (%) of *Epiphyllum oxypetalum* leaves extract as corrosion inhibitor of mild steel in H_2SO_4: **a)** Predicted versus Actual. **(b)** IE (%) versus inhibitor concentration and acid concentration. **(c)** IE (%) versus time and acid concentration. **(d)** IE (%) versus temperature and acid concentration. **(e)** IE (%) versus time and inhibitor concentration. **(f)** IE (%) versus temperature and inhibitor concentration.

3. 2. 3 Validation of results

Table 5. Validated result for corrosion inhibition of mild steel in acid using EO leaves extracts.

Acid concentration (mols)	Inhibitor concentration (g/l)	Temperature (K)	Time (hr)	Predicted inhibition efficiency (%)	Measured inhibition efficiency (%)	Percentage error (%)
1.000	0.055	333.000	2.750	82.926	84.701	2.09

Additional experiments were carried to validate the results of the selected conditions in Table 4, with predicted and measured inhibition efficiencies respectively and presented in Table 5. It was shown that measured inhibition efficiencies were close to the predicted values implying that the RSM optimizing tool was adequate appropriate for the inhibition procedure. This in accordance with [22-23].

3. 3. Result of phytochemical analysis of EO extract

Inspection of phytochemical constituents on Table 6 shows that alkaloids and flavonoids are highly present, saponin and tanins are moderately present while phenolics and phytates are present in traces and Cardiac glycosides are absent or too low to be detected. It can be inferred from table 6 that high inhibition efficiency obtained can be attributed to high amount of alkaloids and flavonoids present in the inhibitor and this agrees with [24-25]. It is noteworthy that the presence oxygen, nitrogen and aromatic ring in the molecular structures of alkaloids shows *Epiphyllum oxypetalum* as active inhibitor.

Table 6. Phytochemical screening of *Epiphyllum oxypetalum* leaf extract.

Parameters	Qualitative screening of EO extract	Quantitative screening of EO extract (mg/100g)
Alkaloids	+++	203.9
Cardiac glycosides	-	10.0
Flavonoids	+++	417.20
Phenolics	+	38.0
Phytates	+	28.3
Saponins	++	74.71
Tannins	++	76.9

3. 4. Results of Fourier transform infrared spectroscopy (FTIR) analysis

Table 7. Prominent peaks obtained from reflectance FTIR spectroscopy.

EO pure extract		Protective oxide film	
Peaks	**Band assignments**	**Peaks**	**Band assignments**
3522.47 cm^{-1}	O-H stretching	3433.18 cm^{-1}	O-H stretching
1695.34 cm^{-1}	N-H bending	1699.23 cm^{-1}	N-H bending

1068.26 cm^{-1}	C-O stretching	1088.05 cm^{-1}	C-O stretching
1423.56 cm^{-1}	C-C ring (aromatics)	1423.56 cm^{-1}	C-C ring (aromatics)
665.01 cm^{-1}	-C≡C; C-H bending vibration	623.10 cm^{-1}	-C≡C; C-H bending vibration

FTIR was employed in this study to identify the type of bonding especially the functional group(s) present in the organic compounds and possibly show that the organic compounds in the extract hinder the metal surface from aggressive corroding environment by adsorption. The peaks of FTIR from Table 7 X-Rayed the *Epiphyllum oxypetalum* extract as rich in organic acids, alkaloids and flavonoids compounds [26].

3. 5. Surface examination by Scanning electron microscope (SEM)

Figure 2 presents the SEM micrographs of Mild Steel surface during corrosion reaction in 3 M H_2SO_4 solution and in the presence of EO extract and changes that resulted in the process. From Figure 2a, the metal surface was very rough and severely damaged while in Figure 2b the surface was transformed into smoother, more uniform deposits with patches upon addition of EO leaf extract. The observed enhancement on Fig. 2b is as a result of depositions on the metal surfaces which is responsible for the inhibition [2, 27].

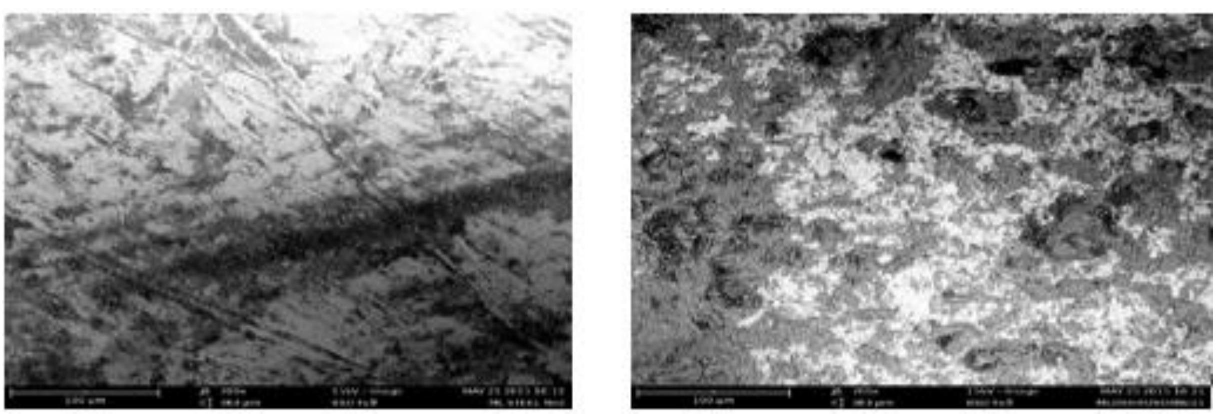


Figure 2. SEM micrographs of Mild Steel surface (a) in 3 M H_2SO_4, and (b) in presence of EO extract.

3. 6. Inhibition mechanism

The result of FTIR and SEM indicates that the mechanism of inhibition comprises obstruction of metal surface by inhibitor species through adsorption. This adsorption process is characterized by the chemical nature of the inhibitors, the nature and surface charge of the metal, type of aggressive electrolyte. The phytochemical and FTIR analysis further confirmed the presence of alkaloid and flavonoids constituents in the plant extract. Hence the adsorption of EO extract on the mild steel surface occurred electrostatically by interactions between the positively charged alkaloid content and the negatively charged metal surface.

4. CONCLUSIONS

The presence of alkaloids, flavonoids, saponins, tannins in the phytochemical screening and the FTIR result confirmed that *Epiphyllum oxypetalum* leaf extract is a good corrosion inhibitor on mild steel surface. SEM micrographs confirmed the adsorption of protective oxide film on mild steel. The established quadratic equation from CCD by applying response surface methodology showed that a combination of factors (inhibitor's concentration and time, inhibitor's concentration and temperature) significantly affected the efficiency of the inhibitor studied.

The predicted optimum inhibitor's efficiency of 82.93% was obtained from surface response methodology (CCD) modeling for the experimental design with the optimum process variables of 1.00 mols of acid concentration, 0.055 g/l inhibitor's concentration, 2.750 hours immersion time, and temperature of 333K. The validated optimum value of 82.926% was in agreement with that predicted by the regression model.

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