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Characterization and optimization study of *Ficus exasperata* extract as corrosion inhibitor for mild steel in seawater

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ABSTRACT

This study investigated the characterization of *Ficus exasperata* extract and the optimization of the process variables on inhibition of mild steel in seawater environment. Box Behnken Design was employed to examine the influence of three process variables: Temperature: 25-29 °C; Time: 3 – 6 days; Inhibitor concentration: 1-5 v/v. Phytochemical screening of the extract was done. The physicochemical constituents of the seawater were also determined. The experimental data was statistically determined and Scanning Electron Microscope (SEM) was used to characterize the mild steel. The result of the phytochemical screening of the *Ficus exasperata* plant extract (FEPE) showed the presence of contain inhibitive constituents: alkaloids, tannins, saponins, flavonoids and glycosides. The highest inhibition efficiency of 86.31% at a temperature of 29 °C for 6days at an inhibition concentration of 3v/v was observed from the experimental run. The optimal process levels of time: 5.74 days, temperature: 27.95 °C and inhibitor concentration: 2.90v/v, gave 87.52% as its inhibition efficiency. The result of the SEM from the optimal process level validated showed that more passive film was formed which can be attributed to the adsorption of the *Ficus exasperata* extract. It can be concluded that the *Ficus exasperata* was a good eco-friendly inhibitor.

***Keywords*:** Corrosion, Ficus exasperata, inhibition efficiency, optimization, weight loss

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1. INTRODUCTION

Corrosion degrades the useful properties of materials and structures including strength, appearance and permeability to liquid and gases. It is important to note that most metal structures used in aqueous environment (sea water, salt water and rain) like ships, oil platforms and pipelines piers are made from mild steel, mild low-carbon and low alloy steels as well as copper-based alloys. Corrosion and abrasion in piping, tanks and process equipment occur as a result of the salt content in water. Therefore, protecting these valuable assets from the effect of sea water exposure, amongst others, is a major challenge for corrosion engineers in these environments [1].

The use of corrosion inhibitors, is among the numerous methods of corrosion control and prevention and it is very popular. Organic compounds containing nitrogen, sulfur or oxygen atoms are the most efficient inhibitors. Unfortunately, some chemical inhibitors used for corrosion control have been limited because of some reasons which amongst others includes their synthesis which is very often expensive and their very toxic and hazardous nature for human beings and the environment [2].

This has prompted the search for green technology and also eco-friendly corrosion inhibitors as an alternative to replace inorganic and organic inhibitors to foster sustainable greenness of the environment. These nontoxic, benign, inexpensive, renewable and readily-available alternative corrosion inhibitors have been found from different parts of plant extracts [3].

Phytochemical compounds' presence in these plant products justified their use as corrosion inhibitors [4]. Some of the plants that had been used as eco-friendly inhibitors are, bitter leaf stem [5]; jatropha bitter kola stem [6]; *Apium graveolens*, *Punica, granatum* and *Camelia sinensis* extract [7]; pawpaw leaves [8]; *Mangerifa indica* peel [9]; *Luffa cylindrica* peel [10]; *Jatropha* stem [11]; *Vernonia amygdalina* [12]; *Camellia sinensis* [13]; *Poupartia birrea* [14]; mango bark and leaf [15]; *Juglans regia* green fruit shell [16]; *Nauclea latifolia* [17]; *Ginkgo* leaf [18]; *Rosa carun* fruit [19]; *Ocimum gratissium* [20]; Groundnut leaves [21]; phytic acid [22]; waste *Musa paradisiaca* peels [23]; *Epiphyllum oxypetalum* extract [24]; *Picralima nitida* leaves [25], *Erigeron floribundus* [26], *Spondias mombin* [34] and *Gmelina arborea* [35].

This research investigated the use of *Ficus exasperata* extract as corrosion inhibitor on mild steel in seawater.

2. MATERIALS AND METHODS

2. 1. *Ficus exasperata* Plant Extract (FEPE)

The *Ficus exasperata* plant extract was collected from Omu-Aran community sun-dried in open sunlight for about a week, and then milled into powder. The fine powder obtained was completely soaked in ethanol solution for about 24 hours with intermittent stirring to have a homogeneous solution and the extract was collected through filtering in form of paste.

The filtrate was then subjected to a process of evaporation for the removal of excess alcohol. The filtrate obtained was inhibitor in pure form. The extracts were used for the preparation of inhibitor test solutions in the concentration range of 1 -5 v/v of the seawater.

2. 2. Preparation of Medium

Seawater of 10 litres was used for this study. Furthermore, its physicochemical constituent was determined. Warmest seawater in Nigeria is 27.1 °C while the coldest is 25.7 °C. Some literature also reported warmest seawater is 29 °C while the coldest is 28 °C. This research used the temperature variation between 25 and 29 °C.

2. 3. Phytochemical Screening

Phytochemical screening was done on the extract to determine the presence of tannis, alkaloids, flavonoids and saponins which are constituents of a good extract.

2. 4. Preparation of Specimen

Mild steel used for this research was 2 mm thick with 25 mm × 25 mm × 2 mm dimension. A hole of 0.1 mm was drilled at the middle of the mild steel. These coupons were degreased with acetone, washed with distilled water and then emery paper was used to polish the surface to make it clean and then stored in a desiccator. The number of mild steels was predicted by Experimental Design via Design Expert Software.

2. 5. Design of Experiment

Design Expert software was used to predict the number of experimental runs. The variables used are volume of seawater, amount of extract, time and temperature. The experimental design used was Box Behnken Design. 17 experimental runs were generated from the Box Behnken design. The Factor levels with the corresponding real values is as shown in Table 1; while the interaction of the variables in coded form is as presented in Table 2. The variables in their real form as presented in the Design Expert software is as shown in Table 3.

Table 1. Experimental range of the independent variables, with factor levels for the inhibition of FE Plant Extract on Mild-Steel in seawater

Independent variable	**Symbols**	**Range and levels**		
		-1	0	+1
Time (days)	A	3	4.5	6
Temperature (°C)	B	25	27	29
Inhibitor concentration (v/v)	C	1	3	5

Table 2. Matrix interactions of the variables in coded form

Std	**Run**	**Block**	**Time**	**Temp**	**Inh. Conc**
7	1	Block 1	-1.00	0.00	1.00

15	2	Block 1	0.00	0.00	0.00
13	3	Block 1	0.00	0.00	0.00
1	4	Block 1	-1.00	-1.00	0.00
16	5	Block 1	0.00	0.00	0.00
10	6	Block 1	0.00	1.00	-1.00
2	7	Block 1	1.00	-1.00	0.00
17	8	Block 1	0.00	0.00	0.00
6	9	Block 1	1.00	0.00	-1.00
5	10	Block 1	-1.00	0.00	-1.00
14	11	Block 1	0.00	0.00	0.00
8	12	Block 1	1.00	0.00	1.00
12	13	Block 1	0.00	1.00	1.00
4	14	Block 1	1.00	1.00	0.00
11	15	Block 1	0.00	-1.00	1.00
3	16	Block 1	-1.00	1.00	0.00
9	17	Block 1	0.00	-1.00	-1.00

Table 3. Interactions of the three variables

Std	**Run**	**Block**	**Time (days)**	**Temp (°C)**	**Inh. Conc (v/v)**
7	1	Block 1	3.00	29.00	3.00
15	2	Block 1	6.00	25.00	3.00
13	3	Block 1	3.00	27.00	1.00
1	4	Block 1	3.00	27.00	5.00
16	5	Block 1	4.50	27.00	3.00
10	6	Block 1	4.50	25.00	1.00
2	7	Block 1	4.50	27.00	3.00

17	8	Block 1	4.50	27.00	3.00
6	9	Block 1	4.50	27.00	3.00
5	10	Block 1	3.00	25.00	3.00
14	11	Block 1	6.00	27.00	1.00
8	12	Block 1	4.50	29.00	5.00
12	13	Block 1	6.00	27.00	5.00
4	14	Block 1	4.50	25.00	5.00
11	15	Block 1	4.50	27.00	3.00
3	16	Block 1	6.00	29.00	3.00
9	17	Block 1	4.50	29.00	1.00

2. 6. Measurement by Weight Loss Method

The gravimetric weight loss measurement method is the most widely used method for assessment of corrosion inhibition based on findings from the literature. The weight loss was done for all the 17 experimental runs gotten from the Box Behnken Design in the Design Expert Software. The coupons were cleaned with distilled water, dried, weighed and then re-weighed for calculating the weight loss using equation (i)

Corrosion rates was calculated using the expression in equation (ii):

$$Weight\ Loss\ (g) = wt_1 - wt_2 \quad \text{(i)}$$

$$Corrosion\ Rate\ (C_r)\left(\frac{\frac{g}{cm^2}}{day}\right) = \frac{\Delta m}{A \times t} \quad \text{(ii)}$$

where: wt_1 is the initial weight before immersion; wt_2 is the final weight after immersion C_r is the corrosion rate, Δm is the weight loss, A is the sectional area and t is the exposure time of the mild steel coupon.

The inhibition efficiency (%) of the *Ficus exasperata* plant extracts was evaluated using equation (iii) while surface coverage was calculated using equation (iv)

$$IE\ (\%) = \frac{C_{r1} - C_{r2}}{C_{r1}} \times 100 \quad \text{(iii)}$$

$$Surface\ Coverage\ (\theta) = \frac{C_{r1} - C_{r2}}{C_{r1}} \quad \text{(iv)}$$

where C_{r1} and C_{r2} are the corrosion rate in the presence and absence of the inhibitors.

2. 7. Validation of the Optimal Process Levels

The optimal process level predicted was validated and compared with the result of the coupon with the highest inhibition efficiency as observed from the experimental run.

2. 8. Surface Characterization

Surface Analysis: Scanning Electron Microscopy (SEM) was used for surface morphology of the blank mild steel, mild steel with the highest inhibition efficiency and the mild steel obtained via the optimal process levels which was validated.

3. DISCUSSION OF RESULTS

3. 1. Result of the physicochemical properties of the seawater

The result of the physicochemical properties of the seawater is as shown in Table 4. Furthermore, acording to effluent water quality standards, the pH is 5.5 -9.0, COD 250 mg/L and the tolerance limits for inland surface waters. Class C shows a standard pH of 6.5 to 8.5, iron is maximum 50 mg/L, DO is minimum 4 mg/L.

Table 4. Physicochemical Properties of Seawater.

S/N	Parameters	Results
1	Temperature	28.70 °C
2	pH	7.28
3	Iron	6.00 mg/L
4	Chlorine	3.70 mg/L
5	Sulohate	94.00 mg/L
6	Potassium	42.00 mg/L
7	Calcium	128.00 mg/L
8	Zinc	3.80 mg/L
9	Nickel	4.30 mg/L
10	BOD	2.80 mg/L
11	COD	3040.00 mg/L
12	EC	20.60 mg/L
13	TDS	10.30 mg/L
14	DO	6.10 mg/L
15	Magnesium	360 mg/L

3. 2. The Result for Phytochemical Analysis of *Ficus exasperata* plant extract

The result of the phytochemical constituents of the *Ficus exasperata* plant extract indicated the presence of bioactive constituents of a good corrosion inhibitor as shown in Table 5. The phytochemical screening conducted showed the presence of tannins, saponins, flavonoids, alkaloids and glycosides while phenol is in negligible quantities. This result showed that the FEPE is a good inhibitor which confirmed the report by [27].

Table 5. Phytochemical Analysis of *Ficus exasperata* plant extract.

Phytochemical Test	Reagents	Colour changes	Results
Saponins	Distilled water	Persistent foam	++
Tanins	Ferric chloride ($FeCl_3$)	Greenish ppt	+
Flavonoid	Sodium hydroxide (NaOH)	Yellow ppt	++
Glycosides	Sulphuric acid (H_2SO_4)	Reddish brown ppt	++
Alkaloid	Wagner	Brown ppt	+++
Phenol	Iron III chloride ($FeCl_3$)	None	-

+++ = *highly present*, ++ =*Moderately Present*, - = *absent or present in negligible quantity*

3. 3. RSM Result of the Corrosion Rate, Inhibition Efficiency and Surface Coverage of Mild Steel in Seawater by FEPE

This study used *Ficus exasperata* plant extract (FEPE) as corrosion inhibitor for mild steel in seawater medium. Table 6 shows the RSM results of the Corrosion rate, Inhibition efficiency, surface coverage of the mild steel in seawater by FEPE. It was observed from the Box Behnkne design that the maximum inhibition efficiency of 86.31% was obtained at: Time of 6 days, Temp of 29 °C, and Inhibition Concentration of 3v/v from experimental run :16.

Table 6. RSM Result of the Corrosion rate, Inhibition efficiency and surface coverage of Mild Steel in Seawater Medium by *ficus exasperata* plant extract

S/N	Time (days)	Temp (°C)	Inh.Conc (v/v)	Weight Loss (g)	Corrosion Rate(CR) $\frac{g}{cm^2.day}$	Inhibition Efficiency (IE) %	Surface coverage (Ɵ)
1	3.00	29.00	3.00	2.6250	0.14	83.55	0.8355
2	6.00	25.00	3.00	12.750	0.34	30.58	0.3058
3	3.00	27.00	1.00	3.5625	0.19	31.22	0.3122

4	3.00	27.00	5.00	3.5625	0.20	73.22	0.7322
5	4.50	27.00	3.00	5.3438	0.19	67.45	0.6745
6	4.50	25.00	1.00	5.3438	0.19	67.45	0.6745
7	4.50	27.00	3.00	5.3438	0.19	67.45	0.6745
8	4.50	27.00	3.00	5.3438	0.19	67.45	0.6745
9	4.50	27.00	3.00	5.3438	0.19	67.45	0.6745
10	3.00	25.00	3.00	3.5625	0.19	65.77	0.6577
11	6.00	27.00	1.00	5.6250	0.15	82.31	0.8231
12	4.50	29.00	5.00	3.3750	0.12	84.22	0.8422
13	6.00	27.00	5.00	10.875	0.29	63.99	0.6399
14	4.50	25.00	5.00	8.7188	0.31	65.99	0.6599
15	4.50	27.00	3.00	8.3438	0.19	67.45	0.6745
16	6.00	29.00	3.00	3.0000	0.08	86.31	0.8631
17	4.50	29.00	1.00	4.7813	0.17	81.32	0.8132

3. 4. Result of Corrosion Inhibition Efficiency and Statistical Analysis

The ANOVA for corrosion inhibition of mild steel in sea water using *Ficus exasperata* extract is as presented in Table 7.

Table 7. ANOVA Analysis of variance table [Partial sum of squares]

Source	Sum of Squares	DF	Mean square	F Value	Prob > F
Model	0.065	6	0.011	39.58	< 0.0001 significant
A	2.813	1	2.813	10.23	0.0095
B	0.034	1	0.034	122.97	< 0.0001
C	5.512	1	5.512	20.06	0.0012
AB	0.011	1	0.011	40.11	< 0.0001
AC	4.900	1	4.900	17.83	0.0018
BC	7.225	1	7.225	26.29	0.0004

Residual	2.749	10	2.749		
Lack of Fit	2.749	6	4.581		
Pure error	0.000	4	0.000		
Core Total	0.068	16			
Std. Dev.	0.017	R-Squared	0.9596		
Mean	0.19	Adj R-Squared	0.9354		
C.V%	8.51	Pred R-Squared	0.7989		
PRESS	0.014	Adequate precision	22.090		

The Model F-value of 39.58 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Furthermore, the values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, BC are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The R-squared value of 0.9596 (95.96 %) showed that there is a probability of 95.96 that the model can be replicated.. The adjusteed R-squared value of 0.9354 is in agreement with the predicted R-squared value of 0.7989, that is, the difference is less than 0.2. Since Adequate Precision measures signal to noise ratio and a ratio greater than 4 is desirable, therefore, we conclude that the Adequate Precision of 22.090 implied adequate signal.

This model can be used to navigate the design space. Subsequently this model can be used to predict the corrosion inhibition of *Ficus exasperata* extract as corrosion inhibtior of mild steel in seawater. This implies that the results are statistically reliable and the second order polynomial model equation in terms of coded values of the process variables are presented in Equation (v).

Final Equation in Terms of Coded Factors:

$$(Y)\ \ IE = +65.98 + 1.40\,A + 17.33B + 7.04C + 9.49A\,B - 15.08AC - 7.16BC \qquad (v)$$

3. 5. Discussion of Graphical Analysis (3D) of the Inhibition Efficiency using RSM

The 3D surface plots were employed to determine the correlation between the process factors (Time, temperature and inhibitor concentration) and the response (Inhibition Efficiency) using the Design Expert software. The 3D Plots were presented from Figs 1-3. Corrosion inhibition of the mild steel in seawater showed that an increase in concentration increases the inhibition efficiency. Furthermore; inhibition efficiency reduces as temperature increases. This confirmed the report from [24, 28, 29].

The plot of normal vs residuals is as presented in Fig. 4 while the plot of Predicted Vs Actual gave linear relationship indicating that the actual experimental values are in consistent with the predicted as shown in Fig. 5.

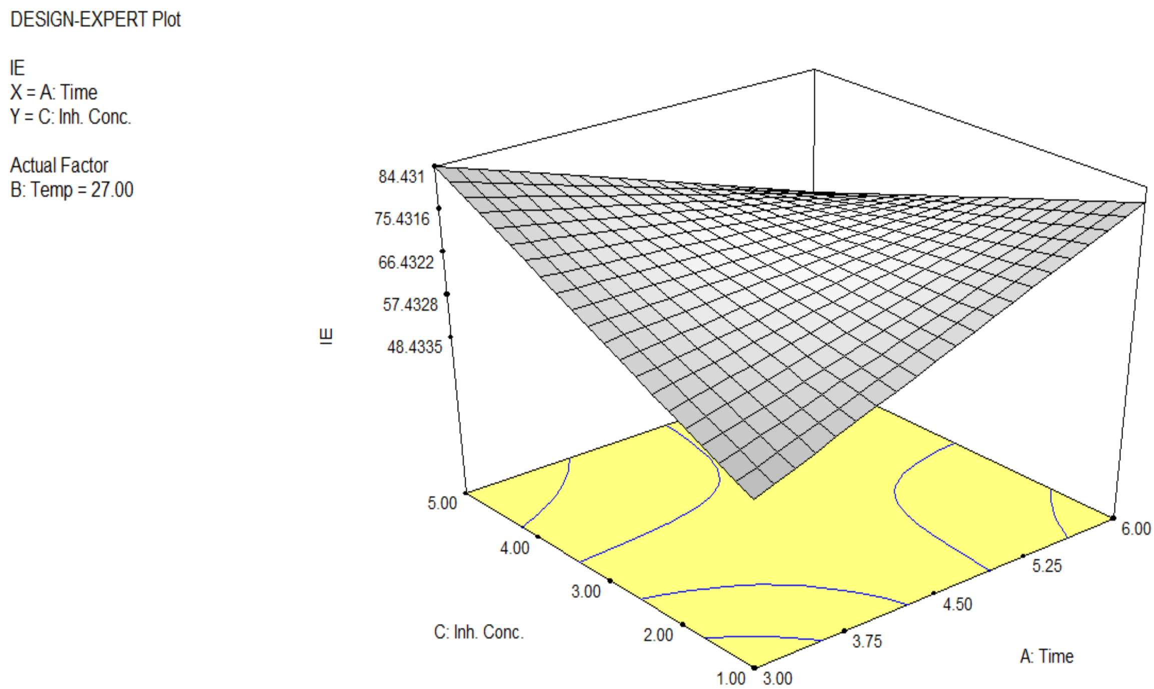

Figure 1. IE (%) Vs. Inhibition Concentration and Time

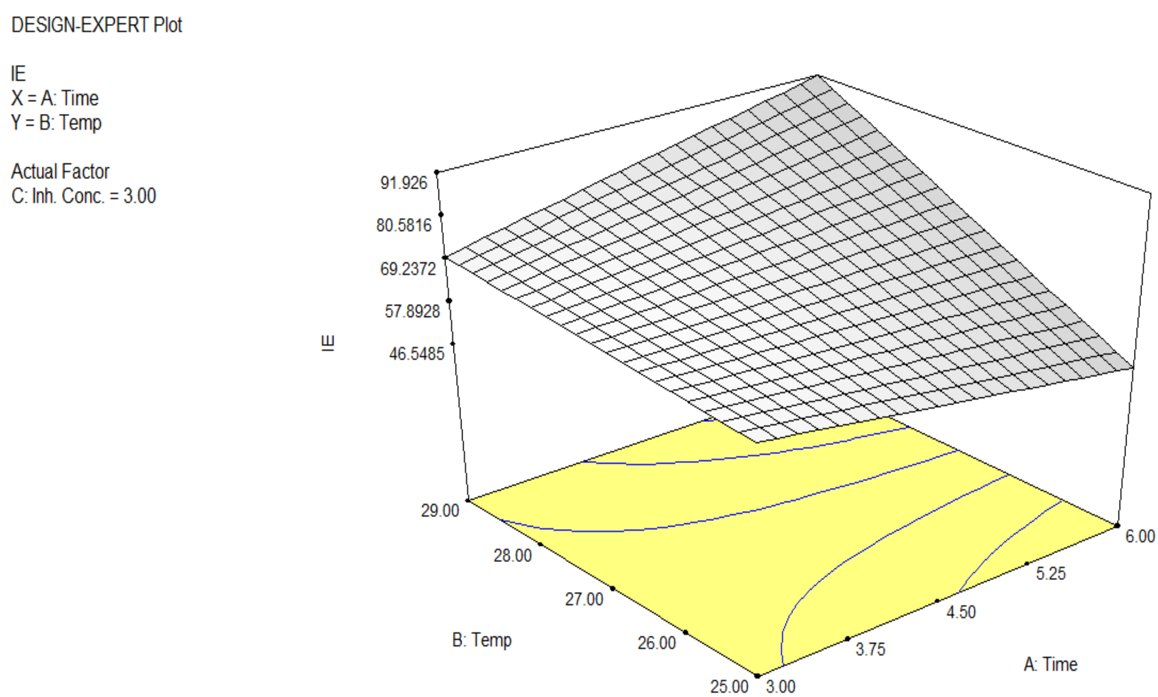

Figure 2. IE (%) Vs Temperature and Time

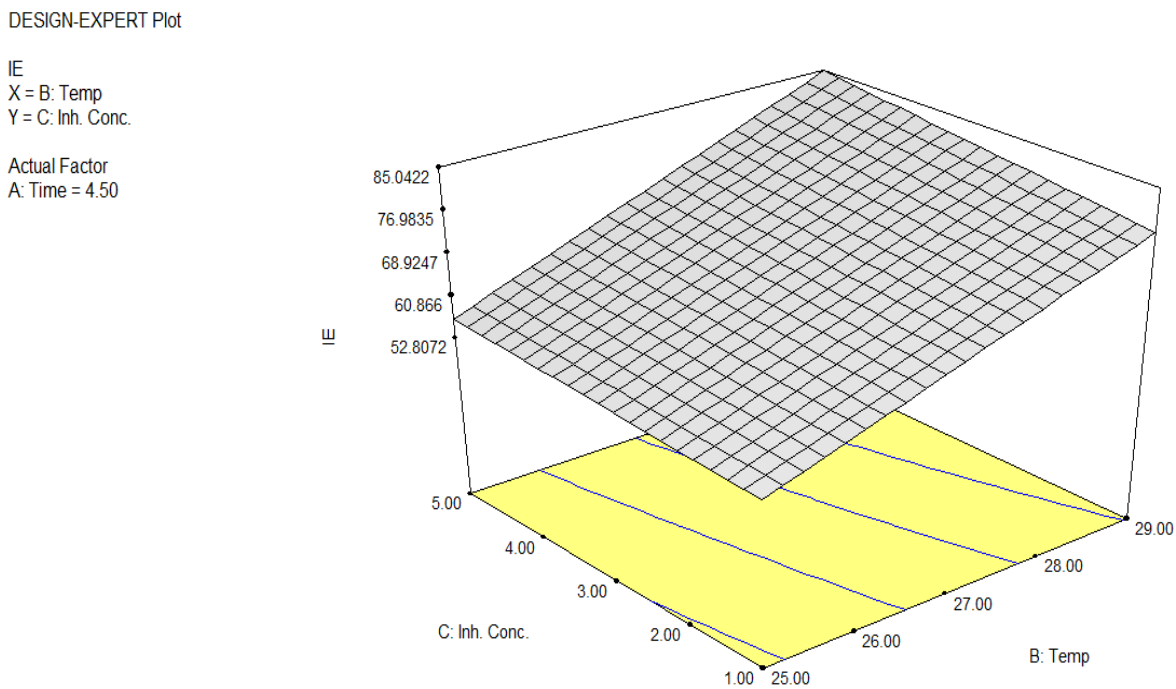

Figure 3. IE (%) Vs. Inhibitor Concentration and Temperature

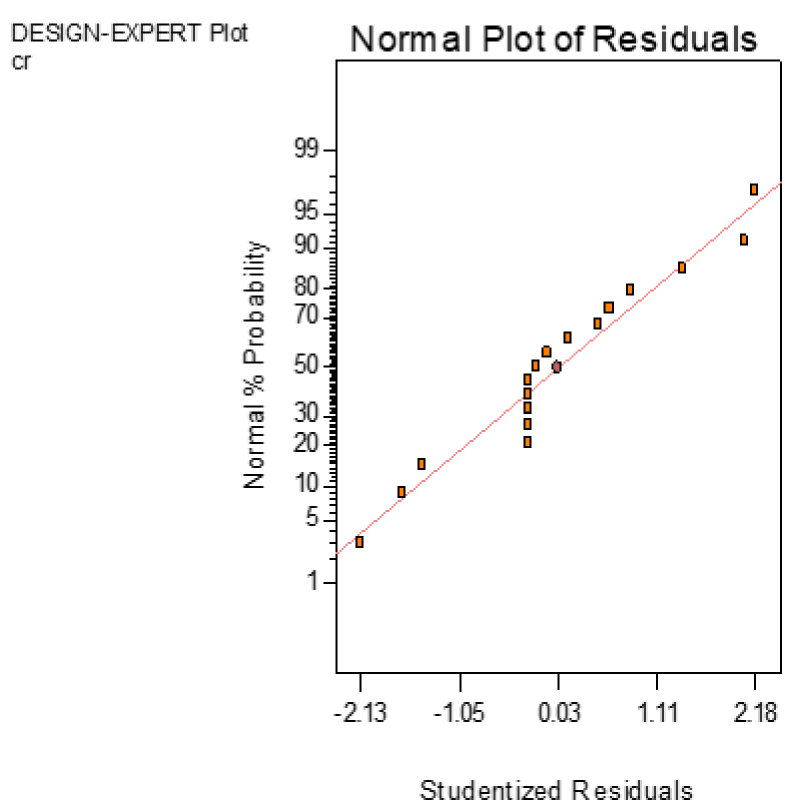

Figure 4. Plot of Normal plot vs Residuals.

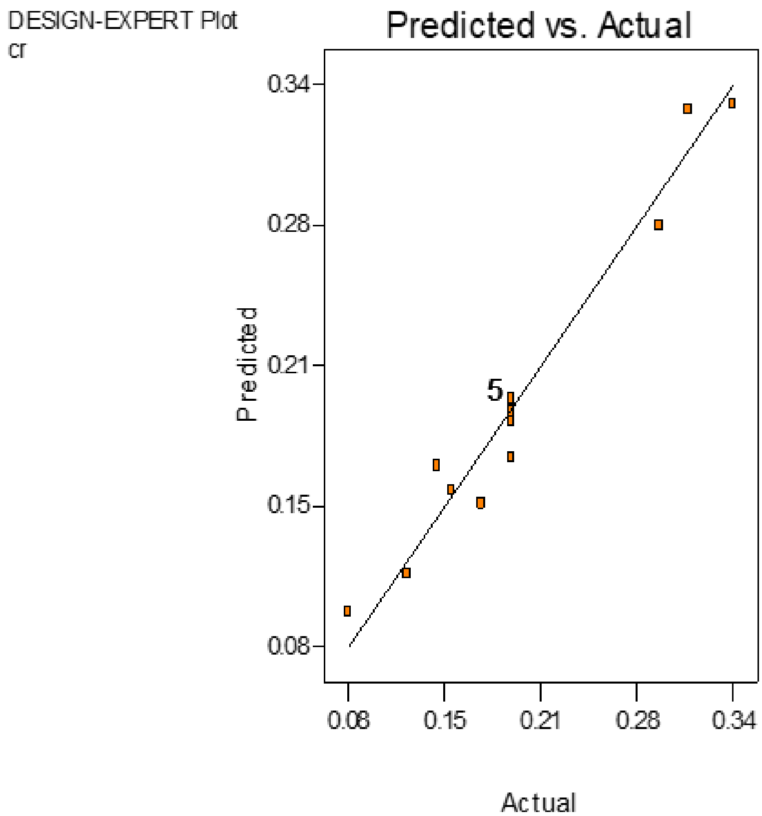

Figure 5. Plot of Predicted Vs Actual

3. 6. Result of the Validated Experiment

The result of the optimal process validated is as shown in Table 8. It showed that the inhibition efficiency observed via the optimal process level was close the one observed form the experimental design. This showed that the optimization was adequate and appropriate for the inhibition in accordance with report of [25, 30].

Table 8. Validated result for corrosion inhibition via optimal process level.

Temp (^oC)	**Time (days)**	**Inh.Conc (v/v)**	**IE (%)**
27.95`	5.74	2.90	87.52

3. 7. Result of Scanning Electron Microscope (SEM) Analysis

The surface morphology of the blank mild steel, mild steel with the best process levels observed from the experimental design and the mild steel via the optimal process level which was validated are as shown in Figures 6-8 respectively. The SEM analysis result showed that the blank mild steel without inhibitor was highly corroded with cracks as seen in Figure 6.

Furthermore, it was observed from Figure 7 that the mild steel showed formation of plane layer of protective film as a result of the *Ficus exasperata* plant extract (FEPE). Furthermore, Figure 8 showed that more protective film was formed which could be attributed to the adsorption of the FEPE on the surface of the mild steel. This is in agreement with previous research done by [21, 24, 30-35].

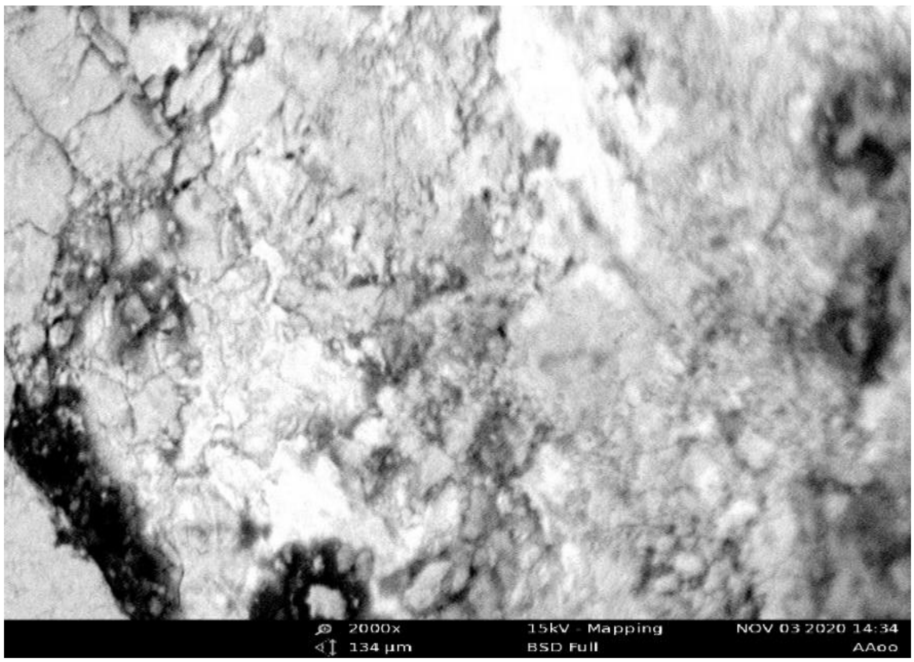

Figure 6. SEM micrograph for Blank MS coupons.

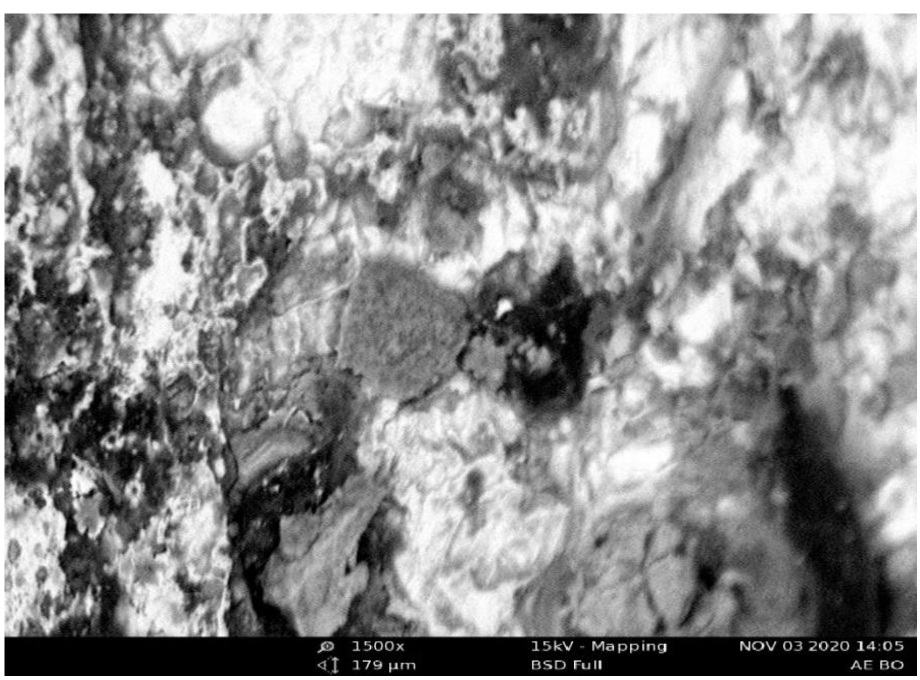

Figure 7. SEM micrograph for best process level.

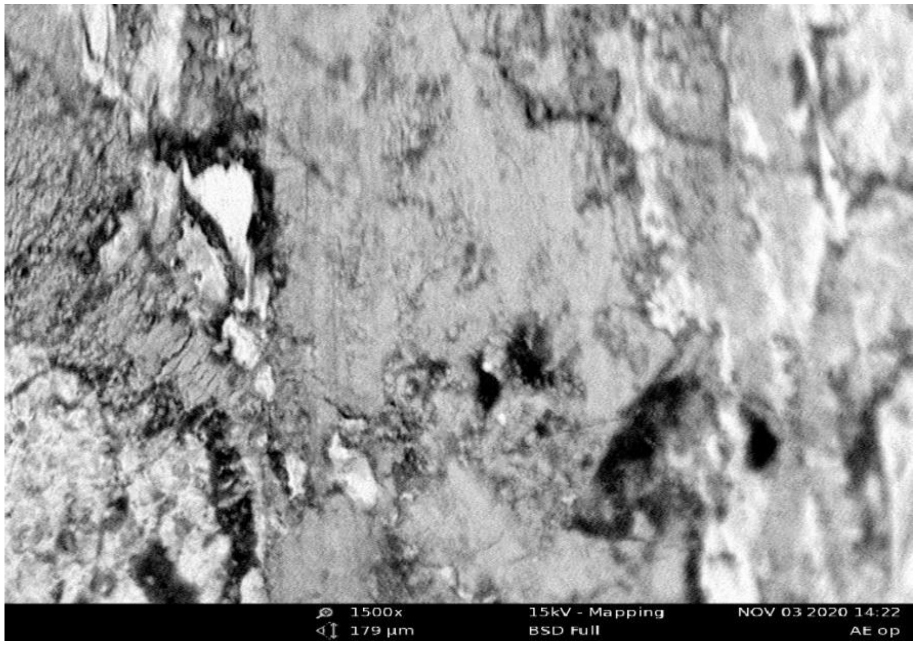

Figure 8. SEM micrograph for optimal process level

3. 8. Inhibition Mechanism

The result of the SEM indicated that the mechanism of inhibition was via the obstruction of the mild steel surface by inhibitor species though adsorption. This adsorption process was characterized by the inhibitor's chemical nature, surface change of the metal and type of aggressive electrolyte. The result of the phytochemical analysis further confirmed the presence of alkaloids, flavonoids, tannins constituents of the extract which affirmed the adsorption process of the FEPE on the mild steel surface [31-33].

4. CONCLUSION

The result of the phytochemical screening of the *Ficus exasperata* plant extract (FEPE) showed the presence of inhibitive constituents: alkaloids, tannins, saponins, flavonoids and glycosides. The highest inhibition efficiency of 86.31% at a temperature of 29 ℃ for 6days at an inhibition concentration of 3v/v was observed from the experimental run. The optimal process level predicted were time of 5.74 days, temperature of 27.95 ℃ and inhibitor concentration of 2.90v/v, which was validated and inhibitor efficiency of 87.52% was observed.

The result of the SEM from the optimal process level validated showed that more passive film was formed which can be attributed to the adsorption of the *Ficus exasperata* extract. It can be concluded that the *Ficus exasperata* extract was a good eco-friendly inhibitor.

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