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Research Article

Utilization of Extract from Bushcane (Costus Afer) as Green Corrosion Inhibitor for Mild Steel

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Abstract

Corrosion has adverse environmental consequences since it affects the manufacturing sector resulting in enormous economic loss. The corrosion characteristics and mechanism of mild in the solution of hydrochloric acid was studied. Extract of bush cane (Costus afer) was screened for physicochemical and phytochemical properties and utilized as corrosion inhibitor for mild steel, the process was optimized using Response Surface Methodology (RSM). The presence of OH, NH of alcohols, phenols, or substituents connected to aromatic rings, C=O for amides and ketones, C-N of aliphatic amines, and C=C of alkenes and nitriles were shown using Fourier Transform Infrared Spectroscopy (FTIR). The optimum condition was found by minimizing time, temperature, inhibitor concentration and corrosion rate while maximizing weight loss, and inhibitor efficiency. The results obtained revealed that the bush cane extract contain saponin, flavonoid, alkaloid and tannin, which suggests the potency of the extract as a good inhibitor as it contains a considerable amount of phytochemical with basic heteroatom. The statistical significance of the weight loss, corrosion rate and inhibitor efficiency were evaluated using the analysis of variance (ANOVA). it was observed that the regression was statistically significant at the F-value of 94.60, 25.87 and 4.72 respectively. P-value of > 0.0235, > 0.0001 and 0.0265 respectively. The optimum temperature, inhibitor concentration and contact time were found to be 27.9 ℃, 20.0% and 5 hours respectively at desirability value of 0.637. At this optimum condition, the weight loss was found to be 0.102%, corrosion rate was 1.583mg/cm^{2}hr and inhibitor efficiency was 62.9%. From the results obtained in this experiment, It is concluded that the bushcane extract can be used as a corrosion inhibitor for the protection of mild steel.

Keywords

Mild Steel, Plant Extract, Corrosion Inhibitor, Hydrochloric Acid (HCl)

1. Introduction

Corrosion is a natural phenomenon which inevitably have huge impact on the economy. Corrosion is a natural process that happens when metals are exposed to corroding environment, this chances of corrosion can be reduced or checked by by the applications of inhibitors [1, 2]. For most industrialized nations; spends up to 3.5 – 5.4% on corrosion control [3]. About one-quarter of the world's annual steel production which corresponds to about 150 million tons per day has been reported to be destroyed by corrosion [4]. 25 to 30% of these losses can be saved using proper knowledge of corrosion prevention [4].

Mild steel (MS) is an alloy of 0.15-0.30% C, 0.4% Si,

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0.7-0.9% Mn, 0.04% S, 0.04% P used for constructional and structural applications. The outstanding mechanical properties, ease of fabrication, excellent weldability, and low purchasing cost make mild steel more preferrable as construction material [5]. Although it is less resistant to corrosion in acidic medium [6]. MS is prone to corrosion, which may be avoided by a difficult process that comes at a significant financial expense. According to estimates, the cost of corrosion worldwide amounts to 3-4% of GDP, of which 15–35% may be avoided by applying current corrosion prevention techniques [7, 8].

Different method and materials have been used to prevent the corrosion of mild steel, some of which are cathodic protection and the use of inhibitors [9]. One useful strategy to avoid corrosion, particularly in acidic conditions, is to apply inhibitors to halt unexpected dissolving and acid consumption. [3]. unfortunately, the limited employment of many chemical inhibitors have is as a result of their costly manufacturing and potential toxicity and environmental hazards to humans [10]. In order to achieve sustainable environmental greenness, this has prompted the quest for environmentally acceptable corrosion inhibitors as an alternative to inorganic and organic inhibitors. Plant extracts have various sections that contain these alternative corrosion inhibitors that are non-toxic, benign, affordable, renewable, and easily accessible [11].

Plants that produce natural compounds with alkaloids have an inhibitory effect. They are affordable and ecologically beneficial. Shrubs and herbs are abundant in the natural world [12]. Green corrosion inhibitors, which are biodegradable and free of heavy metals and other harmful substances, are very interesting since organic corrosion inhibitors are poisonous. Utilizing plant extracts, often known as "green corrosion inhibitors," one can successfully slow down the pace of corrosion [13, 14]. Generally, green Inhibitors have hetero atoms. Because of their greater basicity and electron density, O, N, and S are shown to hinder corrosion. The active centers for the adsorption process on the metal surface are O, N, and S. The order of the inhibition efficiency should be O < N < S < P. The chemical makeup and physical characteristics of the compound, such as the electron density at the donor atom, the porbital nature of the molecule, and its electronic structure, all affect how well a green inhibitor works [12, 14]. The inhibition may result from molecules or ions adhering to the metal surface, from an increase or decrease in anodic or cathodic reactions, from a decrease in the rate at which reactants diffuse onto the metal surface, or from the creation of a barrier film that protects the metal [3, 27].

2. Materials and Method

2.1. Preparation of Specimen (Mild Steel)

Mild steel was used for the study, the thickness of the mild steel was 1.5mm, and was pressed-cut mechanically into coupons of sizes 30mm x 30mm. These coupons were used as procured from the material and metallurgical engineering workshop of the University of Uyo, Akwa Ibom State, Nigeria. And was polished, degreased using ethanol, dried in acetone, weighed and stored in a desiccators to be used for the experiment.

2.2. Preparation of The Bush Cane (Costus afer) Extract (Inhibitor)

The fresh stems of bush cane *(Costus afer*) was harvested, washed to remove dirt, peeled to get the fleshy part, blend and filtered to get the extract. With varying% concentration of the data generated from the design expert in Table 1, the filtrate was used to prepare a stock solution. Thereafter, it was used to prepare an acid solution using Equation (1) below;

$$V_{extract} = \frac{C_{dil} \times V_{350ml}}{C_{extract}} \quad (1)$$

2.3. Preparation of the Electrolyte

The electrolyte used for the experiments was 1.0M HCl. The electrolyte was prepared in the Chemical Engineering Research laboratory, at University of Uyo, Akwa Ibom State, Nigeria. The concentrated HCl acid used for this research has a density of 1.18g/cm^3. A percentage purity of 35% and molar mass of 36.5g/mol.

2.4. Corrosion Rates Determination

Gravimetric Mass-Based Loss

Gravimetric experiments were conducted on test coupons of dimension 30mm x 30mm x 1.5mm, under total immersion conditions at room temperature. Using an analytical balance, the weight of the mild steel (MS) specimen was measured both before and after it was submerged in the corrosive solution, with a 4-digit accuracy. After a given immersion time in the solution with different concentrations of inhibitors, the specimen (coupons) was retrieved from corrosion medium (1M HCl) at different time intervals. Washed with ethanol, dried in acetone and oven dried at 100 ℃ for 10 minutes and was cooled in a desiccator before taking the final weight [15].

The difference in weight is taken as metal loss resulting from corrosion. The corrosion rate (CR, mg/cm^2/h) is calculated by dividing the weight loss (W, mg) that was measured during the tests by the immersion period (Δt, h) and the metal surface area in contact with the solution (A, cm^2). The corrosion rate was calculated using Equation (2) as reported [16].

$$CR = \frac{87.6\Delta W}{DAT} \text{ (mm/yr)} \quad (2)$$

Where;

W = weight loss in mg.

D = density of the mild steel g/cm^3,

A = area in cm^2 and

T = exposure time in hours.

The Percentage Inhibitor Efficiency (IE)

The inhibitor efficiency, (IE%) was calculated using the Equation (3) as reported [17].

$$IE\% = 1 - \frac{CR_O - CR}{CR_O} \quad (3)$$

Where; CR_O = corrosion rate in the absence of an inhibitor,

CR = corrosion rate in the presence of an inhibitor.

2.5. Design of Experiment and Statistical Analysis

Table 1 shows the summary of the range and levels of each of the variables. The responses are the weight loss, corrosion rate and inhibitor efficiency.

Table 1. *Independent variables and levels for central composite design (CCD).*

Independent variables	Low level (-1)	High level (+1)
Temperature ℃	27	35
Concentration%	10	100
Time hr.	3	9

2.6. Process Optimization

The process was optimized to get maximum yield (Y%) of inhibitor efficiency. The inhibitor efficiency was conducted using response surface methodology where the input variables (Temperature = A, Concentration =B, Time =C) were varied to observe its impact on preselected responses of yield (Y%) of finally extracted green inhibitor. Regression (R^2), adjusted R^2, predicted R^2, and lack of fit were the analysis of variance (ANOVA) methods used to support the significance and sufficiency of the regression model created [20].

3. Results

3.1. Evaluation of Regression Model for Weight Loss, Corrosion Rate and Inhibitor Efficiency

The temperature (A), Inhibitor concentration (B) and time of exposure (C) were considered as the independent variables while the weight loss, percentage inhibition efficiency, and corrosion rate were the response. Table 2 shows the result of the experimental schedule.

Table 2. *Experimental Factors and the Corresponding responses for Bushcane inhibitor.*

Std	Run	Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3
		A: Temperature	B: Concentration	C: Time	Weight Loss	Corrosion Rate	Inhibitor Efficiency
Cc		C	%	Hrs.	G	mg/cm^2hr	%
5	1	27	55	3	0.1175	2.9793	52.4291
13	2	31	55	6	0.1632	2.0691	43.8596
9	3	31	10	3	0.1357	3.4408	47.1161
17	4	31	55	6	0.1792	2.2719	39.5412
6	5	35	55	3	0.1401	3.5524	42.9095
7	6	27	55	9	0.1281	1.0827	62.5329
16	7	31	55	6	0.1404	1.7800	51.7028
2	8	35	10	6	0.1164	1.4757	60.7287

Std	Run	Factor 1 A: Temperature	Factor 2 B: Concentration	Factor 3 C: Time	Response 1 Weight Loss	Response 2 Corrosion Rate	Response 3 Inhibitor Efficiency
8	9	35	55	9	0.1993	1.6845	33.1881
1	10	27	10	6	0.0668	0.8469	89.4843
3	11	27	100	6	0.1208	1.5315	53.8579
4	12	35	100	6	0.1534	1.9461	48.2119
15	13	31	55	6	0.1405	1.7851	51.5652
10	14	31	100	3	0.1356	3.3850	47.9735
12	15	31	100	9	0.1632	1.3790	48.5656
14	16	31	55	6	0.1552	1.9676	46.6116
11	17	31	10	9	0.1387	1.1723	56.2736

The experimental result of the different models evaluated using the Design Expert Software, are presented in Table 2. The experimental values obtained were fitted using model-fitting technique in the Design Expert Software version 13.0.5.0. Fitting of the data to various models (linear, two factorials, quadratic, and cubic) and their subsequent ANOVA showed that the weight loss, corrosion rate and inhibitor efficiency models for the utilization of bush cane extract as green inhibitor for mild steel was suitably described by quadratic model as shown in Tables 3 and 4.

Table 3. *Fit statistics for weight loss model.*

Std. Dev.	0.0162	R^2	0.8639
Mean	0.1408	Adjusted R^2	0.6889
C.V.%	11.50	Predicted R^2	-0.0290
		Adeq Precision	9.5993

Table 4. *Fit statistics for corrosion rate model.*

Std. Dev.	0.0162	R^2	0.8639
Mean	0.1408	Adjusted R^2	0.6889
C.V.%	11.50	Predicted R^2	0.0290
		Adeq Precision	9.5993

Table 5. *Fit statistics for inhibitor efficiency model.*

Std. Dev.	0.2177	R^2	0.9708
Mean	2.02	Adjusted R^2	0.9333
C.V.%	10.78	Predicted R^2	0.7503
		Adeq Precision	15.8312

ANOVA was used to assess the interactive importance of the process variables to the corrosion rate inhibitor efficiency and weight loss. The model's significance is measured by the p-value [19].

It was discovered that the regressions were statistically significant at F-value of 4.94, 25.87 and 4.72 for the weight loss, corrosion rate and inhibitor efficiency respectively to Table 8 and that the chance of having these F-value due to noise is 2.35, 0.01, and 2.65%, these models were also significant at P-values of 0.0235, > 0.0001 and 0.0265 for the weight loss, corrosion rate and inhibitor efficiency respectively as shown in Table 6, these were less than 0.0500 which indicates that the models are significant, since p-values less than 0.05 indicates a significant model, this agrees with the findings of [18, 19].

Table 6. ANOVA for quadratic model for weight loss model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0117	9	0.0013	4.94	0.0235	S
A-Temperature	0.0039	1	0.0039	14.75	0.0064	
B-Concentration	0.0017	1	0.0017	6.34	0.0399	
C-Time	0.0013	1	0.0013	4.80	0.0646	
AB	0.0001	1	0.0001	0.2753	0.6160	
AC	0.0006	1	0.0006	2.25	0.1773	
BC	0.0002	1	0.0002	0.5764	0.4725	
A^2	0.0016	1	0.0016	5.91	0.0453	
B^2	0.0021	1	0.0021	7.87	0.0263	
C^2	0.0004	1	0.0004	1.53	0.2567	
Residual	0.0018	7	0.0003			
Lack of Fit	0.0008	3	0.0003	0.9478	0.4974	NS
Pure Error	0.0011	4	0.0003			
Cor Total	0.0135	16				

S = Significant, NS = Non significant

Table 7. ANOVA for quadratic model for corrosion rate model.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	11.04	9	1.23	25.87	0.0001	s
A-Temperature	0.6151	1	0.6151	12.97	0.0087	
B-Concentration	0.2132	1	0.2132	4.50	0.0717	
C-Time	8.08	1	8.08	170.40	< 0.0001	
AB	0.0115	1	0.0115	0.2421	0.6378	
AC	0.0002	1	0.0002	0.0044	0.9492	
BC	0.0172	1	0.0172	0.3632	0.5657	
A^2	0.3118	1	0.3118	6.58	0.0373	
B^2	0.2686	1	0.2686	5.67	0.0489	
C^2	1.63	1	1.63	34.37	0.0006	
Residual	0.3319	7	0.0474			
Lack of Fit	0.1607	3	0.0536	1.25	0.4023	NS
Pure Error	0.1712	4	0.0428			
Cor Total	11.37	16				

S = Significant, NS = Non significant

Table 8. ANOVA for quadratic model for inhibitor efficiency model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2029.77	9	225.53	4.72	0.0265	S
A-Temperature	670.99	1	670.99	14.04	0.0072	
B-Concentration	378.04	1	378.04	7.91	0.0261	
C-Time	12.83	1	12.83	0.2684	0.6203	
AB	133.51	1	133.51	2.79	0.1386	
AC	98.26	1	98.26	2.06	0.1948	
BC	18.34	1	18.34	0.3837	0.5552	
A^2	212.17	1	212.17	4.44	0.0731	
B^2	365.42	1	365.42	7.64	0.0279	
C^2	151.07	1	151.07	3.16	0.1187	
Residual	334.61	7	47.80			
Lack of Fit	226.59	3	75.53	2.80	0.1729	NS
Pure Error	108.01	4	27.00			
Cor Total	2364.38	16				

S = Significant, NS = Non significant

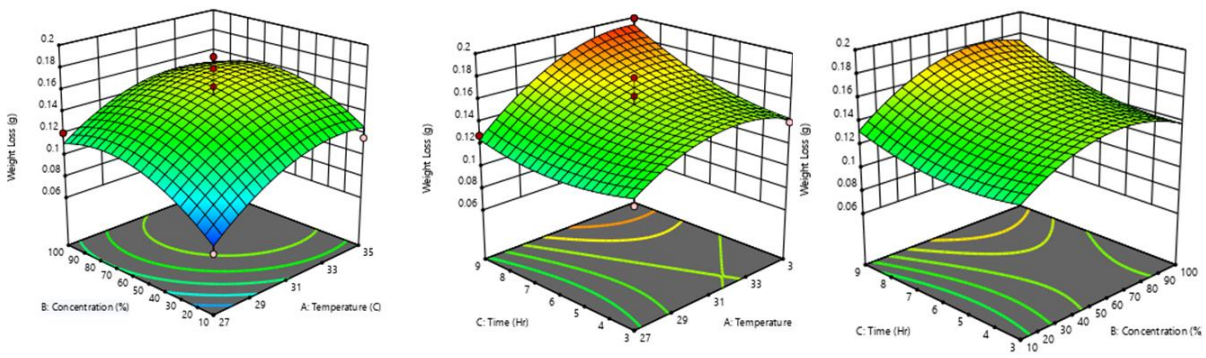
The regression Equation between the weight loss and the coded variables can be expressed as

$$\text{Weight Loss} = 0.1557 + 0.022A + 0.014425B + 0.01255C - 0.00425AB + 0.01215AC + 0.00615BC - 0.0192A^2 - 0.02215B^2 + 0.00975C^2 \quad (4)$$

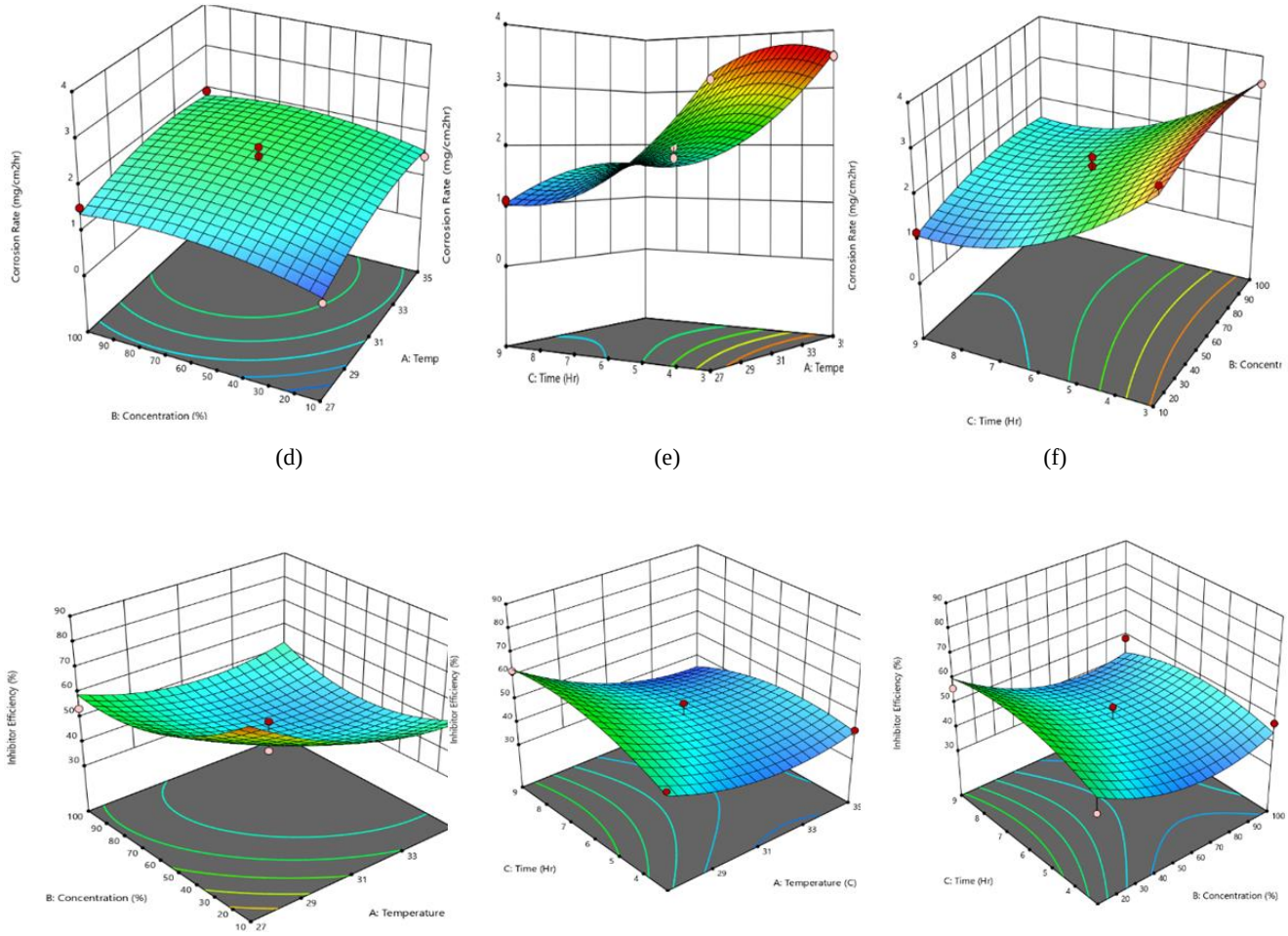
$$\text{Corrosion Rate} = 1.974735042735 + 0.27727979582146A + 0.16323005698006B - 1.004895C - 0.0536AB + 0.00718AC + 0.0656BC - 0.272A^2 - 0.252B^2 + 0.622C^2 \quad (5)$$

$$\text{Inhibitor Efficiency} = 46.656 - 9.158A - 6.874B + 1.266C + 5.778AB - 4.956AC - 2.141BC + 7.099A^2 + 9.32B^2 - 5.9899C^2 \quad (6)$$

3.2. Response Surface Plot for Green Inhibitor Models



(a) (b) (c)



(d) (e) (f)

(j) (h) (i)

Figure 1. *3D plot for weight loss, corrosion rate and inhibitor efficiency models.*

Figure 1a to 1c shows 3D plots for the weight loss model, from the 3D surface plot for the effect of concentration and temperature on the weight loss of the mild steel in the inhibitory medium. it is observed that there is an initial increase in the weight loss as from 10 to 60% and then it decreases as the concentration gets to 10, also, weight loss increased initially as the temperature increases from 27 ℃ to 33 ℃ and then decreases as the temperature keeps increasing slight increase. This corresponds with the findings of [20]. The effect of time and temperature on the mild steel weight loss is represented by the 3D plot in Figure 1b, it is observed that an increase in contact time resulted in an initial decrease in weight loss While an increase in temperature results in an initial increase in weight loss. It is also observed that the maximum weight loss was obtained at the extreme of both temperature and time. This same patern is obsered in Figure 1c. this is in line with the observation of [21].

Similarly the effect of the process parameters on the corrosion rate is show in Figure 1d to 1f. it is observed from figure 1d that there is an initial increase in the corrosion rate as the inhibitor concentration increases from 10% to about 60% and later reduces from about 60% to 100%, this is also observed with as the temperature of the inhibitor is increased, the corrosion rate increases as the temperature of the medium increases from 27 ℃ to about 32 ℃ and stops increasing as the temperature is increased to 35 ℃. Also figure 1e shows the effect of time and temperature on the corrosion rate of the mild steel, from the curvature of the graph is observed that the corrosion rate decreases as the time of contact is increased from 2hours to 9hours while corrosion rate increases rapidly with increase in temperature from 27 ℃ to 35 ℃. This shows that increase in temperature affects the rate of corrosion of mild steel in the inhibitory medium. This agrees with the findings of [20], who also found out in his study that an increase in temperature reduces inhibitor efficiency the effect of contact time and the inhibitor concentration is shown in the in Figure 1f. It is observed that an increase in the contact time from 3hours to 9hours reduces the corrosion rate of the mild steel from about 3.5mg/cm^2 hr to about 0.8mg/cm^3 hr as shown in the 3D plot below. Also it is observed that an initial increase in the inhibitor concentration from 10% about 60% resulted in an initial increase in the corrosion rate and then drops as the concentration increases to 100%. As reported by [20].

Lastly the effect of the process parameters on the Inhibitor efficiency is show in Figure 1g to 1i. Figure 1f shows the 3D

surface plot of inhibitor concentration against the temperature of the medium. It is observed that increase in concentration initially reduces the inhibitor efficiency and later increased as the concentration increases to 100%. It is observed that an increase in temperature reduces the efficiency of the inhibitor. Figure 1g shows the effect of time and temperature on the inhibitor efficiency, from the curvature of the graph is observed that the inhibitor efficiency increases with increase in contact time from 2 hours to 9 hours while inhibitor efficient decreases rapidly with increase in temperature from 27 ℃ to 35 ℃. This shows that increase in temperature affects the efficiency of the inhibitor of bush cane extract for mild steel [22]. Figure 1i shows that an increase in the contact time from 3 hours to 9 hours increases the inhibitor efficiency of the extract for the mild steel from about 60% as shown in the 3D plot below. Also it is observed that an initial increase in the inhibitor concentration from 10% about 60% resulted in an initial decrease in the inhibitor efficiency of the medium and then increases as the concentration increases to 100%. This is in accordance with the finding of [23, 24].

3.3. Phyto-chemical Characteristics of Costus afer Extract

The Costus afer infrared spectroscopy result is clearly evidence that the sample included certain functional groups. The aliphatic amine's CN stretch was shown by the vibration peak at 1106.21cm^{-1}, whereas the nitro compound's NO stretch was disclosed by the peak at 1531.53 cm^{-1}. The presence of the NH bend of primary amines was indicated by the peak at 1647.26 cm^{-1}, whereas the C=C stretch of alkenes and nitriles was shown by the absorption peak at 2345.52 cm^{-1}.

Table 9 shows that the NH stretch of alcohols, phenols, or substituents in an aromatic ring is indicated by absorption peaks at 3439.19 cm^{-1}, whereas the O-H stretch of alcohol, esters, and amides is indicated at 3847.15cm^{-1} [25]. The chemical compounds that are presence in the plant extract after the phytochemical analysis was successfully carried-out and are presented in Table 10. The result shows the presence of active compound in the plant extract of *Costus afer* [26].

Table 9. *FTIR Result of bush cane extract.*

Wave Band Cm	Description	Type of Vibration
1106.21	CN	aliphatic amine stretch.
1531.53	NO	Nitro compound Stretch.
1647.26	NH	Primary amine Bend.
2345.52	C=C	alkenes and nitriles stretch.
3439.19	NH	stretch of alcohols, phenols or substituent on aromatic rings.
3847.15	O-H or NH	Stretch of alcohol, esters and amides.

Table 10. *Phytochemical analysis of plants extract.*

Chemical Constituents	Bush cane Extracts
Tannin	+
Alkaloids	+
Flavonoids	+
Saponins	+

Key: + … Presence _... Absence

3.4. Optimization

The numerical optimization technique (using Design Expert 13 software) was used to get the optimum values of the independent variables i.e. temperature, time and inhibitor concentration. The response variables selected for optimization were time, temperature and inhibitor concentration. The optimum condition was found by minimizing time, temperature inhibitor concentration and corrosion rate while maximizing weight loss, and inhibitor efficiency. The optimum temperature, inhibitor concentration and contact time were found to be 27.954 ℃, 20.031% 5.272hours respectively at desirability value of 0.637. At this optimum condition, the weight loss was found to be 0.102%., corrosion rate was 1.583mg/cm^2 hr and inhibitor efficiency was 62.938%. [20]

4. Conclusion

The results obtained revealed that the bush cane extract contain saponin, flavonoid, alkaloid and tannin, which suggests the potency of the extract as a good inhibitor as it contains a considerable amount of phytochemical with basic heteroatom. Also, the FTIR results indicated the presence of the following: Stretch of aliphatic amine, stretch of nitro compound, Bend of primary amine, stretch of alkenes and nitriles, stretch of alcohols, aromatic rings, Stretch of alcohol and esters and amides groups. The analysis of variance (ANOVA) was used to evaluate the statistical significance of the weight loss, corrosion rate and inhibitor efficiency quadratic model, it was observed that the regression was statistically significant at the F-value of 94.60, 25.87 and 4.72 respectively. P-value of > 0.0235, >0.0001 and 0.0265 respectively. The optimum temperature, inhibitor concentration and contact time were found to be 27.9 ℃, 20.0% and 5 hours

respectively at desirability value of 0.637. At this optimum condition, the weight loss was found to be 0.102%, corrosion rate was 1.583mg/cm^2 hr and inhibitor efficiency was 62.9%. From the results obtained in this experiment, it is concluded that bush cane extract is a good corrosion inhibitor for mild steel.

Conflicts of Interest

The authors declare no conflicts of interest.

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