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# **Optimization of the corrosion inhibition process of mild steel in acid medium using natural Jatropha Curcase and Costus Afar Leaves**

**Ekweze M.A. and Mbah J.C.**

*1,2 Department of Chemical Engineering. Enugu State University of Science and Yechnology PMB 01660 Agbani Enugu State Nigeria*

*\*Mbah J.C, Email address: [chinenyembahj@gmail.com](mailto:chinenyembahj@gmail.com)*

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**Abstract:** Corrosion inhibition of mild steel in acid medium (Nitric and Hydrochloric acids) was studied using naturally available Jatropha Curcase and Costus Afar leave extracts. Gravimetric method of corrosion monitoring was employed in achieving this aim. The Surface morphology of the two extracts were studied using scanning electron microscopy (SEM). The results from effect of process parameters reveals that inhibition efficiency increased with increase in extract concentration but decrease with increase in temperature. The ethanol extract of Jatropha Curcase and Costus Afar leave has the highest inhibition efficiencies of 90.12% and 85.89% respectively. Corrosion rate was found to increase with time but decrease in the addition of Jatropha and Costus leave extracts. Response surface methodology (RSM) was applied to predict the optimum control of mild steel corrosion in acid medium with Jatropha Curcase and Costus Afar leave extracts as inhibitor. Applying Central composite design in this study was found to be good in predicting the optimum range for controlling the metal from corroding thereby reducing the number of experimental runs. From the results it can be deduced that extract from Jatropha Curcase and Costus Afar leaves are good corrosion inhibitor of mild steel in acidic medium at increased inhibitor concentration and decreased acid concentration and temperature.

#### **1. Introduction**

Mild steel according to metal supermarket (2016) is low carbon steel which contains 0.05- 0.25% carbon and has relatively low amount of alloy. Its low carbon content contributes to its malleability ductility, machinability and weldability, its low alloy content speech to its subjection to oxidation but cheaper when compared to other steel. Most industries use these steel materials in constructing their equipment, because of its specific properties. Petroleum industries for example use steel in constructing their equipment such as the distillator, pump, storage tank and others. The minerals like salt particles and other cordial element contained in crude oil get adsorbed on the surface of the tank when pumped into storage tanks. Also, chemical industries use mild steel to construct their hold up tanks where chemicals are stored. The steel is chemically reactive and because of its reactive nature with their respective environment will react with the chemical and it is destroyed. The destruction of the metal as a result of chemical reaction is called corrosion (Fontana 2005).

The mechanism of any corrosion process is that, when the metal come in contact with atmospheric moisture it will be oxidized to metal ion and electron, when the electrons are transferred from metal,

oxygen is converted to oxide ion. When the oxide ion react with the metal ion it forms metal oxide film, which is the corrosion product. Corrosion can be classified based on its occurrence as the nature of its corrodent, some dry, some wet. The mechanism of corrosion can neither be in an electrochemical form nor with direct chemical reaction, and as appearance of corroded metal which can either be consistence or localized (Ahmed, 2006). Corrosion can also be classified based on environment as dry or chemical corrosion and wet or electrochemical corrosion (Fontana, 2005). In industries corrosion is one of the main factors affecting the reliability of the system. (Kochi *et al.,* 2012). It not only affects the equipment but also the surrounding environment. To overcome the atrocities caused by corrosion, there is need for corrosion monitoring or measurement. Reports reveals that corrosion measurement employs a variety of techniques to determine how corrosive the environment is and at what rate metal loss is being experienced. Corrosion measurement is the quantitative method by which the effectiveness of corrosion control and prevention techniques can be evaluated and provides the feedback to enable corrosion control and prevention methods to be optimized. (Fontana, 2005: Ahmad 2006: Pierrer, 2006). The methods include; Corrosion coupons (Weight loss measurement), Electrical Resistance (ER), Linear Polarization Resistance (LPR), Galvanic (ZRA), Hydrogen Penetration.

Researchers have came up with an ideas on how to minimize corrosion problem with the use of a substance called inhibitors. Inhibitors are chemical substances which when added in small quantity decreases the rate of corrosion without changing the concentration of the corrosive agent (BS. ENISO 8044 2000). It has been a reported that compounds containing Nitrogen, sulphur oxide and phytochemical (Saponin, tannin and flavonoid) are excellent inhibitors (Jothi *et al.,* 2013). The mechanism happens when the inhibitors it is introduced into the environment it adsorbed itself at the surface of the corroding materials with an electrostatic bonding type to prevent the reaction between the metal and the chemical. Inhibitors can be organic and inorganic inhibitors. The efficiency of the organic inhibitors depends on the polar functional group with sulphur, oxygen or nitrogen atom in the molecule. Pi electrons and heterocyclic compounds have hydrophobic or hydrophilic ionizable parts. The establishment centre of the adsorption process is usually the polar function (Gentil., 2003, Apreal *et al.,* 2013). Those organic inhibitors that contains oxygen, nitrogen and or Sulphur, is adsorbed on the surface of the metal blocking the active corrosion site. Compounds that have pi bonds are the most efficient organic inhibitor but it is highly toxic and environmentally harmful. (Mahmoud., 2013). The concentrations of the inhibitor in the medium are critical because the metal surface covered is proportional to the inhibitor concentration. (Roberge., 1999 and ElSayed., 2006). Examples of organic inhibitor are extracts of natural substances Sulphur containing compounds with heterocyclic nitrogen compounds, acetylene compounds, Amines (Gentil., 2003, Apreal *et al.,* 2013). The main functional group responsible for forming a physisorbed bond with metal surface are: Amine (-NH2), carboxyl (- COOH) and phosphonate (-PO3H2) (Hammonds, 1989).

Survey literature indicated that *Jatropha curcase* and *Costus afer* were used ad corrosion inhibitors of metals in aggressive media (Ikpeseni *et al.,* 2021; Mokhtari *et al.,* 2014; Odoemelm *et al.,* 2009). The natural extracts are widely used in corrosion inhibition of metallic materials due to the biomolecules rich in aromatic rings as well as heteroatoms (O, N, S…) and double and triple bonds (Wan Nik *et al.,* 2017; Fekkar *et al.,* 2020). The inhibitory process is generally explained by the synergistic intermolecular effect of the various molecules of the natural extract (Khadom *et al.,* 2022; Lrhoul *et al.,* 2022).

Inorganic inhibitors and some of the organic inhibitors are not environmentally friendly and biodegradable, but some organic inhibitors like green inhibitors are ecofriendly and biodegradable. Plant extracts has been reported to be a good organic inhibitor. (Onen 2004, Ekpe *et al.,* 1994). The

present study is to investigate on the use of extract from *Jatropha curcase* and *Costus afer* leaves as a corrosion inhibitor for mild steel in acid medium.

#### **2. Materials and methods**

Some of the materials used for the work include; mild steel coupon, *Jatropha curcase* leave and *Costus afar* leave.



*jatropha curcase*: Germplasm Resources Information Network



*costus afar:* <https://botanicimage.com/> List of reagents used; nitric acid, hydrochloric acid, ethanol, n-hexane, distilled water, and acetone.

# **2.1 Phytochemical analysis of the samples**

Qualitative and quantitative analysis was done on *J.curcase and C.Afar* leaves, to determine the presences and the quantities of phyto-constituents in the samples. Test For Alkaloid, Saponin, Quinone, Glycosides Test, Tannins, Phenol, Flavonoids, Steroids, Saponin Determination (Modified Getsetner Method) And Alkaloid Determination by Modified Harbone Method

# **2.2 Scanning Electron Microscopy (SEM) Analysis**

SEM analysis was done to confirm the presence of a protective adsorbed film of the inhibitor on the mild steel. After exposure to the nitric acid solution in the absence and presence of J.curcase leave

extract for 3hrs in a corrosion bath at a temperature of  $60^{\circ}$ C, the mild steel surface morphologies were tested using Hitachi S3400N SEM.

#### **2.3 Experiment design for optimization processes**

The first step of research surface methodology (RSM), is to introduce a required approximation that will be used to find true relationship between the dependent variable and the set of independent variables. The second step is, forming a mathematical model in the form of a second-order polynomial used to predict the response as a function of independent variables involving their interactions. Generally, the behavior of the system is explained by the following quadratic equation;

$$
Y = b_0 + \sum_{i=1}^n b_i x_i + \sum_{i=1}^n b i i x_{ii}^2 + \sum b i j x i x j \ (2.1)
$$

Where Y is the predicted response,  $b_0$  the offset term, bi the linear effect, bij the squared effect, bij the interaction effect and xi and xj represent the coded independent variables.

Optimization of inhibition process involved three numeric factors of temperature, dosage and acid concentration, and one categorical factor of type of inhibitor with two levels namely; Jatropha Curcase and Costus Afar leave extracts. The number of levels of the categorical factor multiplied the number of experiments (20) giving Forty (40) experiments for the process.

The factors and levels and the design matrix for the optimization of process factors are shown in the Tables 2.1 below with Type of Inhibitor as Jatropha Curcase and Costus Afar

S/N	<b>FACTORS</b>	<b>JNITS</b>	-α	-		$+$	$+\alpha$
	Temperature	$0\sigma$	30	40		60	$70\,$
	Acid concentration	M	0.5				ل و ک
	Dosage	g/mol	0.2	9.4	0.6	0.8	

**Table 1.** Factors and levels for inhibition process using CCD

# **3. Results and Discussion 3.1 PHYTOCHEMICAL ANALYSIS OF THE SAMPLES**

Phytochemical screening was done on the Jatropha Curcase and Costus Afar leaves using qualitative method and the result is shown on the table 4.1. The result revealed the presence of Alkaloid, flavonoid, Glucoside, Tannin, Phenol, Saponins and Steroid in both leaves. Also the quantity analysis was done on the leave of both extracts and the result is also presented on Table 2. From the result, Jatropha curcase leave extract was found to contain more of alkaloid, Tannin, Phenol and Saponin, when compared to Costus Afar leave extract. Also, Costus Afar leave extract if found to contain more of Flavonoid and Glycosides when compared to Jatropha Curcase leave extract. The chemical structures of most of these phytochemicals revealed the presence of electron rich bond or hetero atoms which facilitate their ability to donate electrons (Fadare *et al,* 2015). Therefore, the inhibition of the corrosion of mild steel by ethanolic extract of Jatropha curcas and Costus Afar leaves is attributed to the phytoconstituents of the extract. Similar conclusion has been drawn by some researchers for the inhibition of the corrosion of mild steel by ethanol extract of some plant (Bendahou *et al,* 2006; Ebenso *et al*  2008; Loto, 1998; Anuada *et al* 2005, Nnnanna *et al* 2010). Research has also proven that plant extract that contains amino acids are indeed effective for corrosion inhibition (Jothi *et al,* 2016).

Parameters		J.Curcase		Qty		C.Afar		Qty
	Water	Ethanol	$n-$		Water	ethanol	$n-$	
			hexane				hexane	
Alkaloid	$++$	$\overline{\phantom{0}}$	$++$	4.63%	$+$		$^{+}$	5.61%
Flavonoid	$++$	$++$	$+$	3.97%	$++$	$^{+}$	$\overline{\phantom{a}}$	0.52%
Tannin	$^{+}$	$+++$	$++$	$1.8$ mg/l	$+$	$++$	$^{+}$	$2.93$ mg/l
Glycoside	$+++$		$++$	0.18%	$+++$		$++$	0.15%
Phenol	$++$	$+$	-	0.17%		$^{+}$	$\overline{\phantom{a}}$	1.31%
Saponin	$++$	$++$	$\overline{\phantom{a}}$	2.38%	$\overline{\phantom{0}}$	$++$	$\overline{\phantom{a}}$	3.30%
Steroid	$++$	$\overline{\phantom{0}}$			$++$			

**Table 2**: Phytochemical screening of Jatropha Curcase and Costus Afar leave extracts.

 $+++$  = Abundantly present,  $++$  = Moderately present,  $+$  = Insignificantly present,  $-$  = Absent.

# **3.2 Result on xrf done on the metal**

**Table 3.** is the composition of the mild steel used. From the table, the metal has following composition (in wt%) 0.076 C, 0.2612 Ca, 0.2603 Si, 0.8504 Mn, 0.250 Cr, 0.085 Co, 0.0350 P, 0.0283 S.



# **3.3 Optimization of corrosion inhibition process using CCD**

Corrosion inhibition process was optimized using central composite design (CCD). It involved three numeric factors which were temperature  $({}^{0}C)$ , Acid concentration (M), dosage (g) and one categorical factor which is type of inhibitor. The categorical factor has two levels of Jatropha Curcase leave extract, and Costus Afar leave extract. Weight loss after corrosion of the mild steel coupon was calculated as the response.

Std order	Run Order	Temp $(0^{\circ}C)$	Dosage(g)	Acid Conc. (M)	$\sigma f$ Type Extract	Weight loss (g)
17	$\mathbf{1}$	50.00	0.60	1.50	J.Curcase	3.1209
26	$\sqrt{2}$	60.00	0.40	2.00	$C$ <i>Afar</i>	0.72978
18	3	50.00	0.60	1.50	J.CURCASE	
40	$\overline{4}$	50.00	0.60	1.50	C.AFAR	0.6418
12	$\overline{5}$	50.00	1.00	1.50	J.Curcase	2.9496
33	6	50.00	0.60	0.50	C.AFAR	0.3792
25	$\overline{7}$	40.00	0.40	2.00	C.AFAR	0.99426
9	8	30.00	0.60	1.50	J.Curcase	3.3138
$\overline{4}$	9	60.00	0.80	1.00	J.CURCASE	2.5101
21	10	40.00	0.40	1.00	C.AFAR	0.64134
$\overline{2}$	11	60.00	0.40	1.00	J.CURCASE	2.4633
36	12	50.00	0.60	1.50	C.AFAR	0.65988
30	13	70.00	0.60	1.50	C.AFAR	0.39852
24	14	60.00	0.80	1.00	C.AFAR	0.50202
13	15	50.00	0.60	0.50	J.CURCASE	1.896
22	16	60.00	0.40	1.00	C.AFAR	0.97266
11	17	50.00	0.20	1.50	J.CURCASE	6.0141
31	18	50.00	0.20	1.50	C.AFAR	1.59882
19	19	50.00	0.60	1.50	J.CURCASE	3.0222
28	20	60.00	0.80	2.00	C.AFAR	0.3384
$\overline{5}$	21	40.00	0.40	2.00	J.CURCASE	4.9713
35	22	50.00	0.60	1.50	C.AFAR	0.01032
8	23	60.00	0.80	2.00	J.CURCASE	1.692
3	24	40.00	0.80	1.00	J.CURCASE	1.7958
34	25	50.00	0.60	2.50	C.AFAR	0.63258
10	26	70.00	0.60	2.50	C.AFAR	1.9926
16	27	50.00	0.60	1.50	J.CURCASE	3.2994
21	28	30.00	0.60	1.50	C.AFAR	0.66276
$\mathbf{1}$	29	40.00	0.40	1.00	J.CURCASE	3.2067
23	30	40.00	0.80	1.00	C.AFAR	0.35916
38	31	50.00	0.60	1.50	C.AFAR	0.59298
20	32	50.00	0.60	1.50	J.CURCASE	2.9649
37	33	50.00	0.60	1.50	C.AFAR	0.61296
14	34	50.00	0.60	2.50	J.CURCASE	3.1629
$\overline{7}$	35	50.00	0.80	2.00	J.CURCASE	4.1316
39	36	50.00	0.60	1.50	C.AFAR	0.6044
27	37	40.00	0.80	2.00	C.AFAR	0.82632
32	38	50.00	1.00	1.50	C.AFAR	1.21992
15	39	50.00	0.60	1.50	J.CURCASE	3.06480
6	40	60.00	0.40	2.00	J.CURCASE	3.6489

**Table 4** Design structure for weight loss on the mild steel

#### **3.4 Analysis of variance of corrosion inhibition process**

The analysis of variance is used to analyse the variation of the values gotten after experiment, and tell if the factors are significant or not. The analysis of variance table for corrosion inhibition process is shown on Table 4.9. The Model F-value of 48.06 implied the model was significant. There was only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicated model terms were significant. In this case temperature (A), dosage (B), acid concentration (C), type of inhibitor (D), interaction of temperature and acid concentration (AC), temperature and type of inhibitor, dosage and type of inhibitor, acid concentration and type of inhibitor and quadratic effect of dosage were significant model terms. The "Lack of Fit F-value" of 0.64 implied the Lack of Fit was not significant relative to the pure error. There was 81.28% chance that a "Lack of Fit F-value" this large could occur due to noise.

The "Pred R-Squared" of 0.9351 was in reasonable agreement with the "Adj R-Squared" of 0.9157. "Adeq Precision" measures the signal to noise ratio. A ratio of 24.197 indicated an adequate signal.

Source	Sum of	Df	Mean square	F-values	P-values
	Squared				Prob>F
Model	76.08	9	8.45	48.06	< 0.0001
A-Temperature $(^0C)$	1.64	$\mathbf{1}$	1.64	9.31	0.0047
$B$ -Dosage $(g)$	4.77	1	4.77	27.14	< 0.0001
C-Acid concentration	1.96	1	1.96	11.15	0.0023
(M)					
D-Type of inhibitor	56.22	$\mathbf{1}$	56.22	319.67	< 0.0001
AC	1.54	1	1.54	8.74	0.0060
AD	0.99	1	0.99	5.63	0.0243
<b>BD</b>	2.11	1	2.11	12.00	0.0016
CD	1.16	1	1.16	6.57	0.0156
$B^2$	5.69	1	5.69	32.35	< 0.0001
Residuals	5.28	30	0.18		
Lack of Fit	2.96	20	0.15	0.64	0.8128
Pure Erro	2.32	10	0.23		
Cor Total	81.36	39			

**Table 5** ANOVA table for corrosion inhibition process

# **3.5 Quadratic model equation for the corrosion process**

#### **The model equations was represented both in coded and actual form as shown below;**

*Jatropha curcase (J.Curcase);*

Weight loss (g)=+1.60-0.23A-0.39B+0.25C-1.19D-0.31AC+0.18AD+0.26BD-0.19CD+0.32B<sup>2</sup>

*Costus Afar Leave (C.Afar);*

Weightloss(g)=+3.66871+0.052781A+12.91765B+3.97489C-0.061994AC+8.08510D

# **Concluding calculation in relations of concrete elements:**

*Jatropha curcase (J.Curcase);*

Weightloss(g)=+3.66871+0.052781Temperature( $^{\circ}$ C)12.91765Dosage(g)+3.97489Acid concentration(M)-0.061994Temperature( ${}^{0}C$ )xacidconcentration(M)+8.08510Dosage(g)<sup>2</sup>

*Costus Afar Leave (C.Afar);*

 $Weightloss(g) = -0.86179 + 0.087948$ Temperature(°C)-10.34898Dosage(g)+3.21472Acid concentration(M)-0.061994Temperature( ${}^{0}C$ )xacidconcentration(M)+8.08510Dosage(g)<sup>2</sup>

#### **3.6 The 3D plots**

Model equation was validated using residual plot hence the normal plot of residual shows that the residuals follow anordinal dispersion and there might be some moderate even disperse. The S-molded bend means a change of reaction



**Figure 1. Normal Plot of Residuals**

#### **a. Residuals vs Predicted Plot**

Figure 2 is a plot of the residuals versus the ascending predicted response values that tests the assumption of constant variance which shows that the developed model is adequate because the residuals for the prediction of responses are less than 3% and the residuals tend to be close to the diagonal line. It also proves that the predicted response from the empirical model is in good agreement with the actual values based on the R2 value of 0.9351 for the corrosion inhibition of mild steel in acidic medium using Jatropha Curcase leave extract and Costus Afar leave extract.

# **b. Residuals vs Run**

This is a plot of the residuals versus the experimental run order. It allows you to check for lurking variables (process factors) that may have influenced the response during the experiment which we are not in control of. If there is, the plot should show a random scatter. Trends indicate a time related variable lurking in the background. Blocking and randomization takes care of those trends ruining the analysis (Figure 3).

Design-Expert® Software Weight loss (g)

Color points by value of Weight loss (g): 6.0141

.<br>0.0132



**Figure 2. Residuals v Predicted plot**





**Figure 3: A Plot of Residuals v Run**

#### **c. Predicted vs Actual**

A graph of the actual response values versus the predicted response values helps to detect a value, or group of values that are not easily predicted by the model. In Figure 4 the data was split evenly by 45 degree line which indicated that the model equation can be used to predict the process. The figure illustrates the empirical data points on the weight loss. These data points are also compared with the values that are predicted. The quantified response data which are for a specific run are actual values. The approximating functions were used to generate the predicted values which are evaluated from the model. It also proves that the empirical results have a fine agreement when they are compared to the values that are predicted



**Figure 4:** Plot of Predicted vs. Actual.

#### **3.7 Effect Of Inhibitor Type**

From the ANOVA table, it was observed that inhibitor type is significant. Figure 5 shows a factor plot showing the effect of using two inhibitors. From the plot, it is observed that the weight loss with *C. Afar* leave extract is higher than that with *J. Curcase*. This is an indication that *J. Curcase* leave extract is more efficient in the inhibition process. The higher values recorded in *C. Afar* leave extract is attributed to its lower content of phyto-constituents when compared with *J. Curcase* leave extract.



**Figure 5.** Model Graph Showing One Factor Effect.

Figure 6 shows the 3D plot for the interaction effect. It can be seen from the plot that decrease in temperature and acid concentration in the presence of the two inhibitors decreases the weight loss. The decrease in weight loss is attributed to the adsorption of the phyto-constituent on the surface of mild steel coupon.

#### Design.



**Figure 6:** 3D Surface Plot Showing Interaction Effect.

#### **3.8 Optimal conditions for corrosion inhibition process**

Numerical optimization was used to obtain the optimum conditions for the corrosion inhibition process. The aim is to minimize the weight loss on the corrosion of mild steel coupon. Table 4.10 shows the optimum conditions for the corrosion inhibition process. It can be seen from table 4.10 that the deviations between the predicted values and experimental values were less as evidenced by the low errors recorded. This actually showed that the generated model equations can predict the corrosion inhibition very well.





# **3.9. Scanning Electron Microscopy (SEM) Analysis**

The structural analyses of mild steel in the presence and absence of Jatropha Curcase Leave extract in 2M nitric acid (HNO3) was done and the results are shown in the plates below. The micrograph of the mild steel revealed that more pits were formed on the surface of metal without inhibitor, this is due to acid attack which result to the deposition of corrosion products. Presence of Jatropha Curcase inhibitor retards the corrosion rate because plain layer was observed at the surface of the metal with inhibitor which indicates that there is occurrence of a protective film layer by the active components of the extract. This is in agreement with research done by Loto *et al.* 2011, Alaneme *et al.* 2015 and Onkwuli *et al.,* 2018.



**Plate 1.SEM** photograph of the mild steel in 2M HNO<sub>3</sub> for 24 h in the presence of 0.4%w/v of Jatropha Curcase extract



Plate .2. SEM photograph of the mild steel in 2M HNO<sub>3</sub> for 24 h in the absent of extract

#### **Conclusion**

Response surface procedure is the best methodology for streamlining the hindrance cycle and lessening the quantity of trial runs. In addition, the SEM examination uncovered the event of a film layer adsorbed on the metal surface, while FTIR considers uncovered the practical gatherings existing in the adsorbed defensive layer. From the outcomes it tends to be derived that extricate from Jatropha Curcase and Costus Afar leaves are acceptable consumption inhibitor of mild steel in acidic medium at expanded inhibitor focus and diminished corrosive fixation and temperature.

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