

# Optimization and Predictive Modelling of Gravimetric Corrosion Characteristics of *Irvingia Gabonensis* Leaves Extracts as Anti Corrosion Inhibitor of Mild Steel in HCl Solution



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**ABSTRACT:** In this research, the optimization and predictive modeling of gravimetric corrosion characteristics of *Irvingia gabonensis* leaf extracts (IGLE) as an anti-corrosion inhibitor of mild steel in hydrochloric acid were investigated. Design expert software version 11 were used to analyze the corrosion inhibition-related process characteristics, such as inhibition efficiency, corrosion rate, and weight loss, and their relationships. An effort were made to obtain the optimal conditions for these corrosion inhibition-related process characteristics. Weight loss measurement and design methodology were used for the evaluation of the inhibition efficiency of IGLE for mild steel in HCl. The corrosion inhibition process variables were optimized and predictive regression models were developed using Box-Behnken tool of the Response Surface Methodology (RSM). The findings showed that there were a good fit between the model predictions and the experimental results. The quadratic models developed were significant with P value less than 0.05. The research established an inhibition efficiency of 88.9%, a corrosion rate of 0.143mm/yr, and a weight loss of 0.02 g, which were obtained at the optimum conditions of an extract concentration of 0.6 g/L, an immersion time of 16 hrs, and a temperature of 298K. Therefore, the models were considered ideal for prediction with a confidence level of 95%, and the optimal combination is suitable for the corrosion inhibition process design. Hence these models can be recommended for applications such as oil well acidizing and pickling pipelines.

**Key words:** Inhibition efficiency, optimization, *irvingia gabonensis*, response surface methodology, predictive modelling

## 1. INTRODUCTION

Corrosion is an electrochemical process that attacks materials in aggressive environments such as acids and seawater [1, 2]. Due to its low-cost and mechanical strength, mild steel is widely used in industries and various domestic applications; however, it is easily corrode in aggressive media. Various engineering fields and other industrial applications rely on acid pickling for cleaning metal surfaces and removing old scale layers, despite making the material prone to corrosion [3]. The addition of inhibitors to acid solutions during this process tends to mitigate the corrosion effect [4]. Also, during the oil recovery process, wells receiving acid treatments require inhibitors to prevent corrosion [5, 6]. Therefore, corrosion prevention is less costly than repairing or replacing damaged systems.

Corrosion inhibitors are being employed in the midst of several approaches to mitigating and preventing corrosion in the petrochemical processing industries [7]. The majority of inhibitors demonstrated to be effective are natural complexes containing nitrogen, sulfur, or oxygen atoms. It has become increasingly important to use plant-based extracts as corrosion inhibitors due to their environmental acceptability, cost-effectiveness, availability, and biological compatibility [8–16].

Plant photochemical constituents present in the plant-

base extract can limit steel corrosion [17–21], but how to optimize the process is scarcely studied. This method can be used to optimize experimental results obtained from gravimetric measurements, making it an important tool for any research in this field of study. RSM application reduces the number of experiments that will be required to evaluate the performance of inhibitors, thus minimizing the cost and duration of the experiment [22–28]. RSM is utilized for planning experiments, conducting analyses, developing models, and optimizing process parameters. Utilizing Response Surface Methodology (RSM), statistical studies, and analyses of corrosion inhibition for metals in acidic conditions using *Irvingia gabonensis* extracts as anti-corrosion inhibitors is the main focus of this study.

According to Odejobi and Akinbulumo [29], RSM was used to increase the efficiency of *Euphorbia heterophylla* as a corrosion inhibitor for mild steel in HCl solutions. At the optimal combination of extract concentration, acid concentration, temperature, and immersion time of 1.97 g/l, 0.50 mol, 47.40 °C, and 4.5 h, respectively, the researchers observed an inhibition efficiency of 89.8%.

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Also Saigaa et al. [30] discovered that when the temperature was set to 200 °C and the extract concentration was set to 700 ppm, the inhibition efficiency of *Asphodelus ramosus* reached a maximum of 90.52%.

Salam et al. [31] investigate and optimize microbiologically induced corrosion of mild steel in crude oil environments using a response surface approach. The results indicate that pH is the most influential factor in determining the reaction and that the predicted values and actual data are reasonably in agreement ( $R = 0.9660$ ,  $Adj-R = 0.9516$ ). Their result demonstrates that RSM design, which considers all relevant factors, can accurately predict the optimal operating conditions in crude oil environments required to reduce mild steel corrosion in oil pipelines. Also, Okewale and Adebayo [32] conducted research on the effect of pumpkin pod extract on the inhibition of mild steel corrosion in HCl solution. Response Surface Methodology (RSM) is used to determine the optimal combination of process and experimental variables influencing corrosion rate. Under ideal conditions of 1.78.07 ppm extract concentration, 2.26 hr of exposure duration, and 35.280 °C, the corrosion rate was determined to be 2.54 mm/yr, which is the lowest corrosion rate attained. Emembolu et al. [33] describe the optimization of *Epiphyllum oxypetalum*'s inhibitor efficiency against mild steel corrosion in 3M H<sub>2</sub>SO<sub>4</sub>. On the basis of these process variables, the optimal extract concentration, acid concentration, temperature, and immersion time were determined to be 0.055 g/l, 1.00 mol, 333 K, and 2.750 h, with a predicted inhibitor's efficiency value of 82.93%. Even though *Irvingia gabonensis* has been acknowledged for its medicinal and anti-corrosion properties [34,35]. Okuma et al. [9] examined the inhibitive properties of *Irvingia gabonensis* leaf extract as a corrosion inhibitor in an induced hydrochloric acid environment. The results revealed that as the concentration of the extract increases, the corrosion rate decreases. In a similar investigation, Okuma et al. [44] studied the kinetic and thermodynamic properties of the *Irvingia gabonensis* leaf extract as a corrosion inhibitor in an acidic environment. The results suggest that *Irvingia gabonensis* leaf extract effectively prevented acid-induced corrosion of mild steel. Kinetic and thermodynamic analysis revealed that the inhibited species displayed greater resilience in the acidic environment. The Phytochemicals organics compounds of *Irvingia gabonensis* is presented in Table 1. As shown in the Table, the plant extract contains alkaloids, tannins, flavonoids and saponins, which as shown in literature to exhibit high inhibition performance against corrosion [9,34,45]. However, no study has demonstrated the optimization of the plant extract to examine the optimal inhibition efficiency, corrosion rate and weight loss of the plant extract. Therefore, this study aims to address these aspects.

## 2. EXPERIMENTAL

### 2.1 Material

The chemical composition of the mild steel plate was determined by X-ray fluorescence spectroscopy, which is a technique that uses the interaction of X-rays with a sample to

identify the elements present and determine their concentrations [46]. The composition of the steel is summarized in Table 2. Before the corrosion process, the mild steel was mechanically cut into its desired shape. As described in [26], the coupon was polished with emery paper to enhance its lustre and degreased with acetone (99.5% purity) to remove any oil residue.

**Table 1** Phytochemicals present in IGLE inhibitor [9].

Phytochemical	Occurrence
Alkaloids	++
Tannins	+++
Flavonoids	++
Saponins	++
Resins	+

+++ = Present in appreciable quantity

**Table 2** Mild steel composition .

Element	Composition ( % )
C	0.16
P	0.04
Mn	0.8
S	0.05
Fe	98.95

### 2.2 Preparation of Extract

The *Irvingia gabonensis* leaves (IGL) used was identified and purchased from Effurun market in Delta State, Nigeria. The fresh leaves were dried for five (5) days was then pulverized and kept for extraction. To create the extracts, 20 g of powdered desiccated leaves were refluxed for four hours in 1.0 M HCl (35% grade) and allowed to remain overnight. The products' filtrates were used as the stock solution. Using the filtrate, extract concentrations of 0.2, 0.4, and 0.6g/L were then obtained.

**Table 3** Variables level of independent parameter for Box- Behnken Design.

Independent Parameters	Low level(-1)	High level(+)
Temperature, (K)	298	318
Immersion time, (hours)	8	24
Inhibitor Concentration (g/L)	0.2	0.6

### 2.3 Weight Loss Experiment

The weight loss method was performed at 298K, 308K, and 318K temperatures. As indicated previously [37], the difference between the initial and final weights of an unprotected and protected test solution was analysed. After the corrosion testing was finished, the coupons were

**Table 4** Box-behnken factor-response design layout

Std	Run	Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3
		A:Temp	B:Immersion Time	C:Inhibitor Conc.	Weight Loss	Corrosion Rate	Inhibition Efficiency
		K	hr	g/l	g	mm/yr	%
23	1	298	8	0	0.13	0.942	-
21	2	298	8	0.2	0.052	0.621	60.4
24	3	298	8	0.4	0.038	0.296	70.8
17	4	298	8	0.6	0.025	0.078	80.8
5	5	308	8	0	0.12	0.864	-
27	6	308	8	0.2	0.061	0.621	49.2
22	7	308	8	0.4	0.042	0.384	65
8	8	308	8	0.6	0.034	0.108	71.7
19	9	318	8	0	0.14	1.082	-
2	10	318	8	0.2	0.08	0.824	42.9
6	11	318	8	0.4	0.06	0.642	57.1
25	12	318	8	0.6	0.04	0.421	71.4
13	13	298	16	0	0.18	0.621	-
15	14	298	16	0.2	0.1	0.437	44.4
14	15	298	16	0.4	0.08	0.31	55.5
11	16	298	16	0.6	0.02	0.143	88.9
4	17	308	16	0	0.2	0.681	-
26	18	308	16	0.2	0.08	0.224	60

removed and rinsed in acetone before being reweighed. The experimental data were recorded and analysed using equations (1)-(3) respectively as adopted from previous study [26].

$$\Delta W = w_i - w_a \quad (1)$$

$$CR = \frac{w_{bl} - w_{inh}}{\text{Area (m}^2) \times \text{Time (day)}} \quad (2)$$

$$IE(\%) = \frac{w_{bl} - w_{inh}}{w_{hl}} \times \frac{100}{1} \quad (3)$$

$W_i$  and  $W_a$  represent the coupon's initial and final weights, respectively.  $w_{bl}$  and  $w_{inh}$  represent, respectively, the weight loss values for the blank and inhibited conditions.

#### 2.4 Optimization and Modelling Technique using Response Surface Methodology

In preliminary studies, weight loss, corrosion rate, and inhibitor efficiency were found to be substantially affected by inhibitor concentrations, temperatures, and immersion time [38]. Using Box-Behnken tool of RSM, the IE and other corrosion experimental results were modelled and optimized. The investigation was conducted using a quadratic design model. Response variables (Responses 1–

3) included WL, CR, and IE; independent variables (Factors 1–3) included 298K–318K temperature, 8–24h immersion period, and 0–0.6 g/l inhibitor concentration alongside the factors levels is presented in Table 3. The Box-Behnken Design employs only two points (low and high levels) for each independent parameter to strike a balance between experimental efficiency and accuracy. This design allows for the creation of response surfaces and facilitates the estimation of main effects and interaction effects while minimizing the number of experimental runs needed. It provides sufficient information for fitting a quadratic model without the excessive resource demands of a full factorial design.

### 3. RESULT AND DISCUSSION

#### 3.1 Response Surface Analysis

The Response surface analysis of mild steel corrosion in 1M HCl is presented in Table 4. A total of 27 experimental runs were analysed from the design tool.

#### 3.2 Design Variance Analysis and Mathematical Model of the Corrosion Characteristic

Table 5 displays the results of the analysis of variance (ANOVA), which indicates that quadratic models are suitable for analysing experimental data.

**Table 5** Variance analysis for inhibition efficiency on mild steel in 1.0 HCl with IGLE

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	24265.26	9	2696.14	40.22	< 0.0001	significant
A-Temperature	1.74	1	1.74	0.0259	0.8741	
B-Immersion time	388.15	1	388.15	5.79	0.0278	
C-Inhibitor Concentration	2707.72	1	2707.72	40.39	< 0.0001	
AB	113.96	1	113.96	1.7	0.2097	
AC	47.55	1	47.55	0.7093	0.4114	
BC	62.3	1	62.3	0.9294	0.3485	
A <sup>2</sup>	17.16	1	17.16	0.256	0.6194	
B <sup>2</sup>	198.86	1	198.86	2.97	0.1031	
C <sup>2</sup>	2435.03	1	2435.03	36.33	< 0.0001	
Residual	1139.58	17	67.03			
Cor Total	25404.84	26				
Std. Dev.	8.19		R <sup>2</sup>	0.9551		
Mean	48.48		Adjusted R <sup>2</sup>	0.9314		
C.V. %	16.89		Predicted R <sup>2</sup>	0.889		
			Adeq Precision	17.8652		

**Table 6** Variance analysis for corrosion rate of mild steel in 1.0 HCl with IGLE

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2	9	0.2224	46.98	< 0.0001	significant
A-Temperature	0.0005	1	0.0005	0.0997	0.7561	
B-Immersion time	0.1327	1	0.1327	28.02	< 0.0001	
C-Inhibitor Concentration	0.1793	1	0.1793	37.87	< 0.0001	
AB	0.0712	1	0.0712	15.03	0.0012	
AC	0.013	1	0.013	2.75	0.1155	
BC	0.0582	1	0.0582	12.29	0.0027	
A <sup>2</sup>	0.0423	1	0.0423	8.94	0.0082	
B <sup>2</sup>	0.002	1	0.002	0.4277	0.5219	
C <sup>2</sup>	0.0425	1	0.0425	8.97	0.0081	
Residual	0.0805	17	0.0047			
Cor Total	2.08	26				
Std. Dev.	0.0688		R <sup>2</sup>	0.9613		
Mean	0.445		Adjusted R <sup>2</sup>	0.9409		
C.V. %	15.46		Predicted R <sup>2</sup>	0.8995		
			Adeq Precision	24.5615		

**Table 7** Variance analysis for weight loss of mild steel in 1.0 HCl with IGLE

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0968	9	0.0108	34.67	< 0.0001	significant
A-Temperature	0.0009	1	0.0009	2.96	0.1034	
B-Immersion time	0.0001	1	0.0001	0.4286	0.5214	
C-Inhibitor Concentration	0.0143	1	0.0143	46	< 0.0001	
AB	0	1	0	0.0453	0.8339	
AC	1.28E-07	1	1.28E-07	0.0004	0.9841	
BC	0.0067	1	0.0067	21.65	0.0002	
A <sup>2</sup>	0.0004	1	0.0004	1.34	0.2638	
B <sup>2</sup>	0.0006	1	0.0006	1.99	0.1761	
C <sup>2</sup>	0.0073	1	0.0073	23.54	0.0001	
Residual	0.0053	17	0.0003			
Cor Total	0.102	26				
Std. Dev.	0.0176		R <sup>2</sup>	0.9483		
Mean	0.0904		Adjusted R <sup>2</sup>	0.921		
C.V. %	19.47		Predicted R <sup>2</sup>	0.8513		
			Adeq Precision	18.7818		

Statistically, the model is significant, with an F-value of 40.22. This "model F-value" has a stochastic occurrence probability of 0.01%. Additionally, model terms are significant if their "Prob > F" values are less than 0.0500. In this instance, the letters A, B, C, AB, AC, BC, A<sup>2</sup>, B<sup>2</sup>, and C<sup>2</sup> are significant model terms. If a model term's value is greater than 0.1000, it is irrelevant to the model. With an R<sup>2</sup> of 0.9551 (95.51%), the likelihood of replicating the model successfully is high. The variance is less than 0.2, as indicated by the consistency between the adjusted R-squared value of 0.9314 and the predicted R-squared value of 0.89. Utilizing this model, you can investigate additional potential layout options [39–40]. This model can also predict the corrosion inhibition efficiency of mild steel in 1.0 HCl when treated with *Irvingia gabonensis leaf extract* (IGLE). This indicates that the findings can be relied upon statistically. The model equation is represented by Equation (4) in terms of the coded values of the process

$$\text{variables. Inhibition Efficiency (IE)} = 73.0505 - 0.487305A + 11.4091B + 16.2804C + 5.3375AB - 1.49961AC + 2.36844BC - 1.79375A^2 + 8.52484B^2 - 9.71522C^2 \text{ (4)}$$

Also, from Table 6, the p-value for the regression model for corrosion rate is < 0.05 (0.0001), so it is a significant model. The F-value of 46.98 also reaffirms the significance of the model. This indicates the input parameters such as temperature, immersion time, and extract concentration have a significant effect on the corrosion rate. The second-order polynomial model obtained for corrosion rate is expressed in Eq. (5).

$$\text{Corrosion Rate} = 0.16719 + 0.00803463A - 0.210923B - 0.132475C - 0.133375AB + 0.0248193AC +$$

$$0.0723896BC + 0.0890625A^2 - 0.0272013B^2 + 0.0405764C^2 \text{ (5)}$$

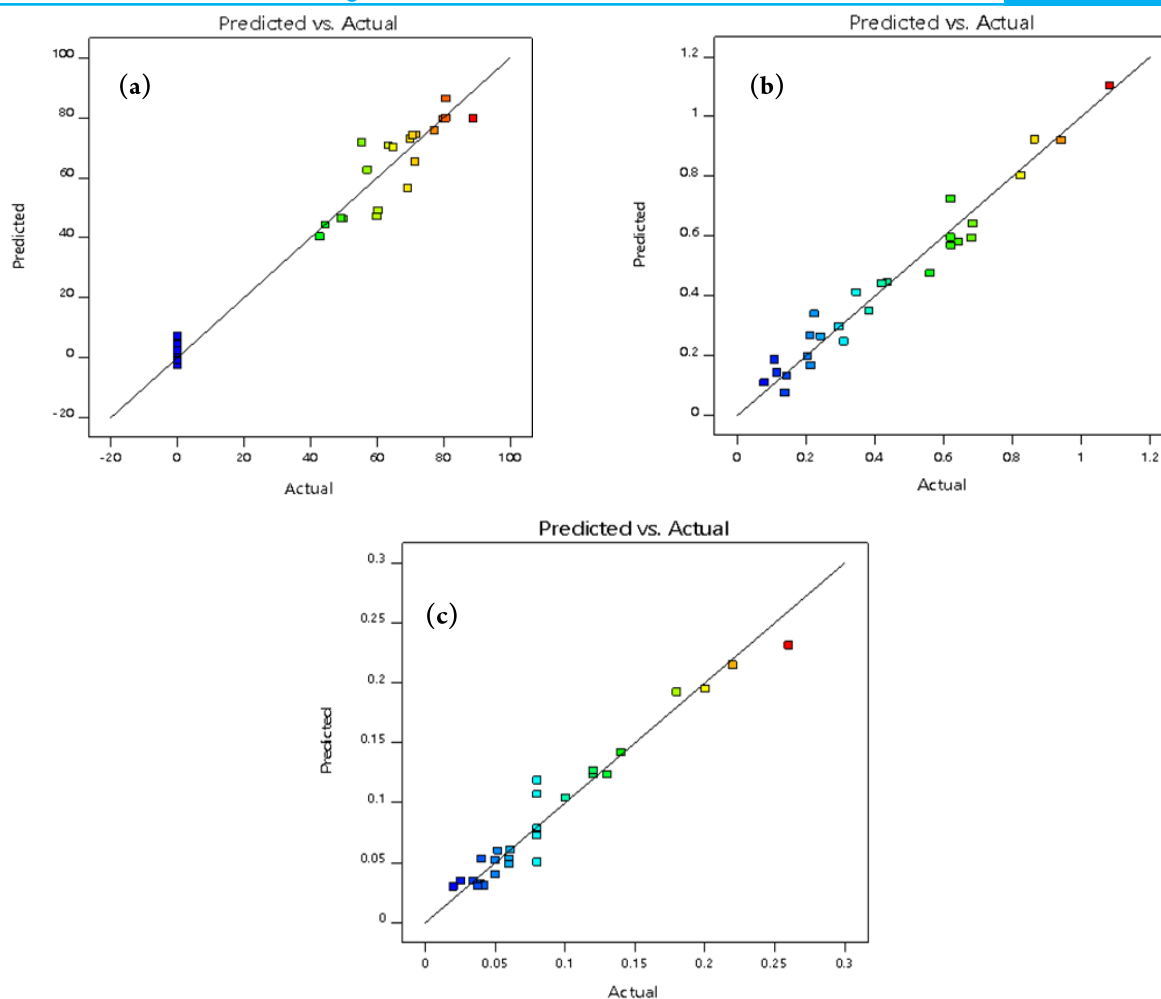
Table 7 highlights the ANOVA results on the response of weight loss to the process variable at the 95% confidence level and 5% significance level. As indicated, the probability for the linear model is 0.0001, which is < 0.05. This is evidence to show that the parameters direct influence on weight loss is significant. The F-value of 34.67 also shows that the model is significant since there is only a 0.01% possibility that such a large F-value could be obtained as a result of noise.

The R<sup>2</sup> values predicted (Tables 5, 6, and 7) are conclusively in agreement with the adjusted R<sup>2</sup> values since their variance is less than 0.2, respectively, and the R<sup>2</sup> is close to 1, so the fitted model is considered ideal for the process [41].

Adeq Precision measures the signal to noise ratio. A ratio of 4 or higher is considered adequate and desirable. In Tables 3, 4, and 5, the ratios of 17.87, 24.56, and 18.78 indicate adequate signals. These models can therefore be successfully used to navigate the design space [42].

$$\text{Weight Loss} = 0.0527064 + 0.0112112A + 0.00667665B - 0.0373707C + 0.001875AB - 7.76694e-05AC - 0.0245893BC + 0.0088125A^2 - 0.0150287B^2 + 0.0168222C^2 \text{ (6)}$$

The validation model is illustrated in Fig. 1(a–c), which presents the relationship between the actual and predicted results of inhibition efficiency, corrosion rate, and weight loss of mild steel in 1.0 HCl with IGLE. The validation model assess the level of fit between the model's predicted and experimental values. As can be observed, a good fit result was obtained from the data points as the plot



**Fig. 1.** Model graph of predicted *versus* actual for (a) inhibition efficiency; (b) corrosion rate (c) weight loss

clustered around the line of best fit [40]. These trends show that the achieved design model is adequate to predict the studied corrosion characteristics of mild steel in the inhibited environment. There is also a strong correlation between the predicted and actual values of the IE, corrosion rate, and weight loss.

Figures 2(a–c) provide a three-dimensional surface plot that showcases the surface interaction between the dependent variables (inhibition efficiency, corrosion rate and weight loss) and some of the independent variables (temperature and inhibition concentration). It can be seen from the figures that the response variables are influenced by the independent factors. The surface plot depicts that increasing both the extract concentration of IGLE and temperature promotes inhibition efficiency and also has a significant impact on the expected response due to the increase in environmental conditions. Thus electrochemical reaction would likely occur when the average atmospheric temperature is high [43]. The model surface 3D results were discovered to be consistent with the experimental results, suggesting IGLE is a suitable plant extract for surface treatment of mild steel in aggressive environments.

### 3.3 Numeric Optimisation Solution

The numerically optimised solution of the dependent

factors and independent factors was obtained from the statistical analysis. This is a procedure carried out to determine the predicted optimal level of input parameters out of many numerical optimization [44–46]. The optimal values help in establishing the level of performance of the independent parameters on the corrosion characteristics [2, 43]. The optimisation method involved establishing target criteria and goals for both factors and response variables. The independent variables were set to be in range, while the response variables were set at maximum, as presented in Table 8. The factor settings that result in the highest desirability value suggest an acceptable conclusion of the numerical optimisation, despite the fact that the ultimate purpose of numerical optimisation is not to maximise the desirability value. There may be several islands with acceptable results (locally optimal), which is a possibility. The optimal responses of the corrosion characteristics are 0.02 g, 0.143 mm/yr, and 88.9% for weight loss, corrosion rate, and inhibition efficiency under optimal conditions of 298K for temperature, immersion time of 16 hrs, and inhibitor concentration of 0.6g/l. The optimum conditions were reliable in determining the significance of individual responses.

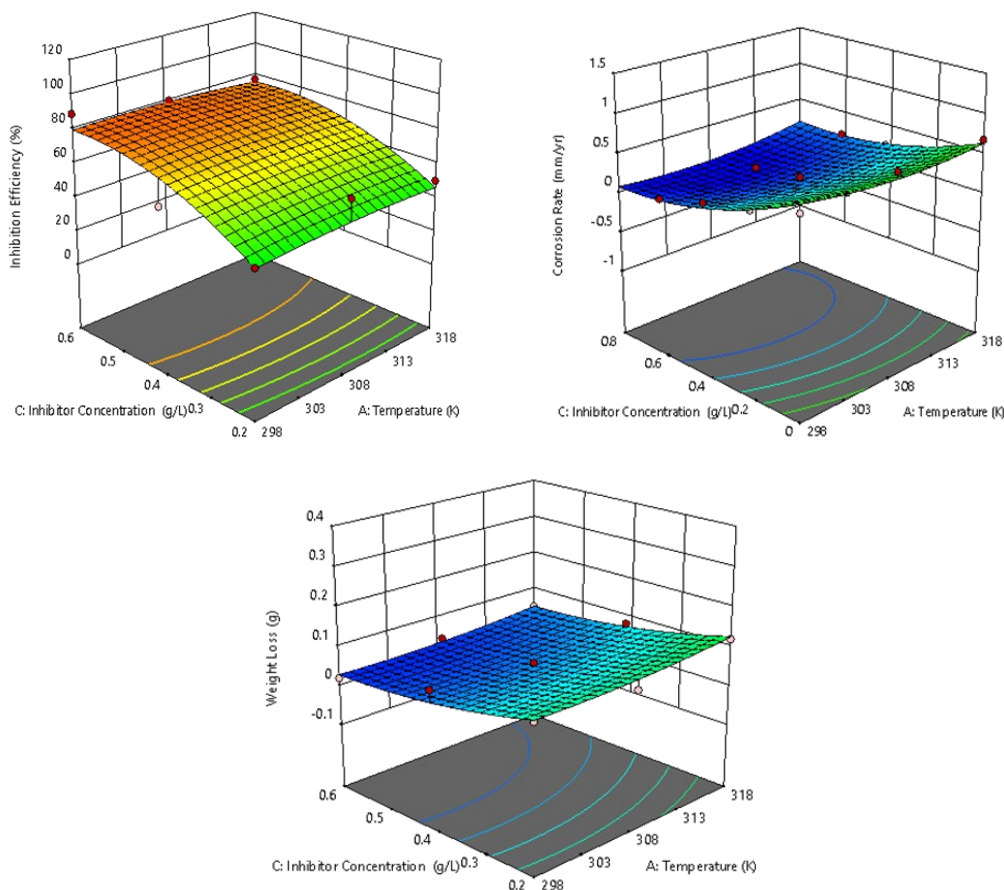


Fig. 2. 3D surface interaction plots for (a) Inhibition efficiency (b) Corrosion rate; (c) weight loss

Table 8 Pre-set optimisation goals for desirability

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Temperature	is in range	298	318	1	1	3
B:Immersion time	is in range	8	24	1	1	3
C:Inhibitor Concentration	is in range	0.2	0.6	1	1	3
Weight Loss	maximize	0.02	0.26	1	1	3
Corrosion Rate	maximize	0.078	1.082	1	1	3
Inhibition Effi-	maximize	0	88.9	1	1	3

Table 9 Comparative validation of predicted and experimental values of IGLE corrosion parameters

Parameters	Inhibitor Con-	Immersion time	Temperature	Predicted values	Experimental Values	Uncertainty error
	centration		(K)			
	g/l	hours				%
	0.6	16	298			
WL				0.02	0.027	0.7
CR				0.143	0.149	0.6
IE				88.90	90.4	2

### 3.4 Validation of Predict Models

An additional experiment to validate the model's prediction was done with the predicted optimum conditions. Duplicate samples of mild steel were machined for each test, and the mean result obtained was recorded. The results are shown in Table 9. As presented in this table, the predicted and experimental values are close to each other, and the uncertainty errors between them are 0.7, 0.6, and 2% for WL, CR, and IE, respectively. Therefore, we establish that the models are statistically fit in the prediction of the corrosion parameters responses, since the uncertainty errors are less than 5%, the models are therefore validated to be statistically fit for the prediction of corrosion characteristic responses. Hence these models can be recommended for applications such as oil well acidizing and pickling pipelines.

## 4. CONCLUSION

This study focused on optimizing and predicting the corrosion inhibiting properties of IGLE for mild steel in HCl solution. The quadratic model used demonstrated strong correlation ( $R^2$ , adjusted  $R^2$ , predicted  $R^2$ ) and significance ( $p$ -value < 0.0001) in predicting inhibition efficiency, corrosion rate, and weight loss. The extract concentration, immersion time, and temperature positively influenced these parameters. The research achieved 88.9% inhibition efficiency, 0.143mm/yr corrosion rate, and 0.02g weight loss under optimal conditions. The validation experiment done at the optimal conditions established 88.9% for IE, 0.143mm/yr for CR and 0.02g for WL respectively. Since the uncertainty error is less than 5%, the models are therefore validated to be statistically fit for the prediction of corrosion characteristic responses.

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## DECLARATION OF CONFLICTING INTEREST

The authors confirm that there is no potential conflict of interest involved in the research, the authoring, or the publication of this article.

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