Modelling and optimization of corrosion rates of mild steel inhibited with Ficus thonningii bark extract in 1M HCl solution

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ABSTRACT
The study explores the most active and optimum effect of Ficus thonningii bark extract as corrosion inhibitor in 1M HCl medium on a mild steel using the Response surface methodology (RSM). The operation variables considered were temperature and concentration as inputs and corrosion rate (output) on mild steel inhibited with extracts of Ficus thonningii bark. A linear model developed by applying response surface methodology showed that extract concentration and temperature affect and interacts with the corrosion rate of mild steel exposed to 1M HCl solution. The predictive model equation of corrosion rate for Ficus thonningii bark extract was developed. The predictive optimum corrosion rate of 31.02082 x 10^{-3} mm/yr was obtained from RSM modelling for the experimental design with 1.0g/dm Ficus thonningii bark extract concentration at 298K. More experimentation was carried out to validate the predicted result and the validated optimum value was 31.0218 x 10^{-3} mm/yr. This value was in agreement with the predictive value generated by the regression model. The model can be used to predict the corrosion rates of mild steels exposed to varying temperatures in an acidic medium, at various using different concentration of the extract as inhibitors.
Key words: optimization, corrosion rate, Response surface methodology, Ficus thonningii bark extract.

1. Introduction

The discovery of a new bio- corrosion inhibitor from Ficus thonningii bark extract was a noble cause. However, it was nobler to establish the most active state and optimum operating conditions of this biodegradable corrosion inhibitor in actual engineering application on mild steel in 1 M HCl solution. The high level of ecological awareness and the growing environmental regulations has necessitated the drive for the development of biodegradable, renewable, readily available, affordable and effective corrosion inhibitors of mild steel. Globally corrosion is the most destructive cause of failure of mild steel. Over the years, researchers have developed different materials both organic and inorganic corrosion inhibitors. However, because of the afore mentioned properties, the demand for organic corrosion inhibitors is on the increases. [1] worked on sapium ellipticum leaf extract, the input variables used for the study were temperature and concentration. The responses were centered on inhibition efficiency and weight loss, as the optimization and modelling yielded inhibition efficiency of 89% at 1.5g/m^3 extract concentration and 303K. [2] also investigated the corrosion inhibition efficiency of Euphorbia heterophylla extracts on mild steel in HCl solution, the input variables include Extract concentration, Acid concentration, Time and Temperature, while the outputs are Corrosion rate and Inhibition efficiency. Response surface methodology (RSM) was used for the modelling and optimization of the process, which showed inhibition efficiency of 89.9% at extract concentration 1.97g/m^3, 0.5M HCl solution, temperature of 320.4 K and after 4.5 hours. [3] studied the corrosion inhibition properties of Epiphyllum Oxypetalum leaf extract and optimization of process variables on inhibition of mild steel, the input variables were temperature, time and inhibitor concentration, while the responses were weight loss, corrosion rate and inhibition efficiency using Response surface methodology (RSM). The optimum predicted inhibition efficiency yielded 82.93%, while the validated optimum value was 82.926%, this is in agreement with the predicted value by the regression model, at extract concentration of 0.055g/m^3, at 2.75 hours and 1M H_2SO_4 solution. [4] also investigated the anti-corrosion effect of Pile plant leaf extract on mild steel immersed in 1.0 M HCl solution, the input variables are concentration and temperature, while the responses were inhibition efficiency and corrosion rate. The optimized value of inhibition efficiency was 88.54% at 303K and extract concentration of 1.2g/m^3.
In this study, corrosion rate of mild steel inhibited with Ficus thonningii bark extract was modeled and optimized to bring out optimum values of inputs and outputs using RSM. The inputs are concentration of the plant extract and temperature, while the output is the corrosion rate.

2. Experimental

2.1 Sample preparation

Ficus thonningii bark was collected by scrapping the bark from the tree plant in Makurdi, Benue state Nigeria. The bark was washed, air dried under room temperature for four weeks, pulverized, sieved and stored in an air tight container ready for extraction. The 1 mm thick mild steel plate used for the production of coupons was purchased from building material market in Makurdi Benue state.

2.2 Extraction procedure (cold water maceration)

The extraction was carried out by weighing 1000 grams of the pulverized stored Ficus thonningii bark. The sample was placed in a clean 20 litres plastic bucket with an air tight cover, 10 litres of distilled water at room temperature (cold water) were poured in to the plastic bucket containing the pulverized sample. The bucket was firmly covered, agitated and left to stand for 72 hours with intermittent agitation. After 72 hours, the entire content of the plastic bucket was emptied and squeezed on a sieve, as the residue was returned to the plastic bucket again with the addition of fresh 5 litres of distilled water were added to the squeezed cake; the filtration process was repeated again. The brownish mixture of extract and water was placed in a wide dish and kept on a safe place so that the water can evaporate from the mixture naturally, leaving a flaky extract on the surface of the dish, which was scrapped, weighed and stored [5].

2.3 Weight loss test at both room and elevated temperatures

Weight loss method was carried out to determine corrosion rate as the output into the modelling and optimization software. Mild steel plate of 1mm thick was used to conduct the weight loss test at various temperatures, the plate was cut in to 600 pieces of square shape of 20mm. The coupons were filed using filing machine and polished with emery papers, the washing was done by using acetone, after which the coupons were sundried, weighed and labelled. Various grams of Ficus thonningii bark extracts, 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 respectively were placed inside the six (6) different 1000ml measuring flask containing 100ml of 1M HCl solution each. The HCl solution was further added in to the 1000ml flask until it reaches the 1000ml mark on the flask to yield 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 g/dm^3 of the extract in acidic solution. The labelled coupons were properly weighed and noted before inserting the coupons in to the individua flasks containing the required extract concentration (g/dm^3) and it was left for 48 hours. The coupons were removed from the acidic solution contained in the flask after 48 hours, then immersed into 2M NaOH solution for three (3) minutes, washed under running water, dipped in to a jar containing acetone, then dried on a clean towel. The dried coupons were all reweighed for at least three times and the average value was recorded. The coupons were all returned back to the corresponding acidic solution in the marked flasks. The same process was repeated for 96, 144, 192 and 240 hours correspondently.

The weight lost test at the following temperatures; 298, 308, 318 and 328 K was similar to weight loss test at room temperature, the major difference was the time of exposure, 2, 4, 6 and 8 hours.

The weight loss and corrosion rate were calculated using equation (1) and (2) [5].

Weight loss (Δw) =Original weight – final weight (1)

$$\text{Corrosion Rate (mm/yr)} = \frac{87.6\Delta w}{\rho AT} \quad (2)$$

Δw = weight loss of mild steel coupon (kg)

ρ= Density of mild steel (kg/cm^3)

A=Surface area of mild steel (cm^2)

T= immersion time (hrs)

2.4 Experimental design

The response surface methodology was employed to predict the optimum corrosion of mild steel in acidic medium with Ficus thonningii bark extract. The inputs considered were the inhibitors concentration and temperature, while the output was corrosion rate. Fit test was carried out on the extract using the inputs to bring out model that will fit the data provided. The model suggested based on the inputs/outputs for the extract was linear model.

Results and discussion

3.1 Model and ANOVA of the process variables

The model summary and ANOVA of the input variables (temperature and extract concentration) and the corresponding response (corrosion rate) of mild steel in 1 M HCl solution, inhibited with Ficus thonningii bark extract is presented in Table 1, it shows a significant model P-value ≤ 0.0001. This indicates the significance and the adequacy of the models. The adequacy is also tested by comparing the model validation parameters [2]. The high R-Squared value obtained shows consistency and that the process parameters explain 99 % of the variance of the corrosion rate.

ANOVA regression analysis and response surface plot of the interaction factor were obtained by analyzing the experimental responses (corrosion rate) with design Expert 8.06 software. The interactive effects of the process variable (concentration and temperature) which include linear effects on the responses were further analysed by ANOVA test in order to determine their individual effect. The significance of the regression coefficient was tested using P-value (<0.05).

Hence the relationship between the actual (experimental result) and predicted responses were assessed and analysed by graphical representation of the predicted response (Corrosion Rate) and actual (inputs) data.

The model F-value 45.64 implies that the model is significant and there is only a 0.01% chance that F-value this large could occur due to noise. The value of Prob > F less than 0.0500 indicated model terms are significant, while values greater than 0.1 indicate model not significant

The Pred. R-Square of 0.7428 is in reasonable agreement with the Adj R-Square value of 0.7951, the difference is less than 0.2. Adequate precision measure signal to noise ratio and a ratio greater than 4 is desirable, the ratio is 21.903. We can conclude that adequate precision of 21.903 implied adequate signal, hence this model can be used to predict the corrosion rate of mild steel in 1M HCl solution in the presence of Ficus thonningii bark extracts as corrosion inhibitors. These results are statistically reliable and Linear model equation in coded values of the process variables are presented in equation (3).

$$CR = A + Bx_1 + Cx_2 \quad (3)$$

Where: CR = Corrosion Rate, x_1 = Temperature, x_2 = Concentration

Table 1: Model Summary and ANOVA of Ficus Thonningii Bark Extract

Model Summary Statistics

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	8.51	0.8130	0.7951	0.7428	2091.72	Suggested
2FI	8.63	0.8167	0.7892	0.6931	2496.04	Aliased

Analysis Of Variance Table

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	6610.86	2	3305.43	45.64	< 0.0001	significant
A-Temperature	2926.26	1	2926.26	40.40	< 0.0001	
B-Extract Conc.	3684.60	1	3684.60	50.87	< 0.0001	
Residual	1521.01	21	72.43			
Cor Total	8131.86	23				

Analysis of variance table [Partial sum of squares - Type III]

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Residual	1521.01	21	72.43			
Cor Total	8131.86	23				

Std. Dev.	8.51	R-Squared	0.8130
Mean	63.97	Adj R-Squared	0.7951
C.V. %	13.30	Pred R-Squared	0.7428
PRESS	2091.72	Adeq Precision	21.903

Coefficient Factor	Estimate	df	Standard Error	95% CI Low	95% CI High	VIF

Intercept	63.97	1	1.74	60.36	67.59	
A-Temperature	14.81	1	2.33	9.97	19.66	1.00
B-Extract Conc.	-18.14	1	2.54	-23.43	-12.85	1.00

3.2 The predictive model equation

The model equation of corrosion rate for Ficus thonningii bark was also presented in Table 2, the equation is peculiar to Ficus thonningii bark, the model equation is used to predict corrosion rates of a system at a given temperature and concentration.

Table 2: Model Equation for Ficus thonningii bark

Final Equation in Terms of Actual Factors for Ficus thonningii bark
Corrosion Rate = -227.01721 +0.98763 * Temperature -36.27571 * Concentration

3.3 Actual and predicted corrosion rates

The experimental data are analysed to check the correlation between the values of the experimental and the predicted corrosion rate and the result of the plot is shown in Figure 1. The R^2 values of 0.8130 shows that the data points are distributed reasonably near the straight line, the closer the R^2 values gets to 1.0, the more reasonable the data points. Higher values of R^2 indicates that there is good relationship between experimental and predicted values of response as was explained by [2] with R^2 value of 0.931. This indicates a good relationship between the actual and the predicted corrosion rate. The result suggests also that the selected model is adequate for predicting the process corrosion rate, this is accordance with [6].

The graph shown in Figure 2, clearly shows the relationship between the corrosion rate and extract concentration as obtained from experimental and predictive results. Corrosion rate reduces with increase in inhibitors concentration for the predicted and experimental [7], this can be seen for both the experimental and predictive results, the trend is the same. The result is also in agreement with the R^2 value 0.8130, which tends to 1.0, it shows that there is a good relationship between experimental and predicted values, hence the model is adequate for predicting corrosion rates for Ficus thonningii bark extract inhibited 1 M HCl solution.

The result shown in Figure 3, explains the effect of temperature on corrosion rates of mild steel exposed to 1 M HCl solution with 0.6 g/dm^3 of Ficus thonningii bark extract as inhibitor. Corrosion rate increases with increase in temperature [4] and [8], the trend was the same for both experimental and predictive results, the model can be used effectively in making corrosion rates predictions for mild steels exposed to varying temperatures in an acidic medium, using Ficus thonningii bark extract as inhibitors

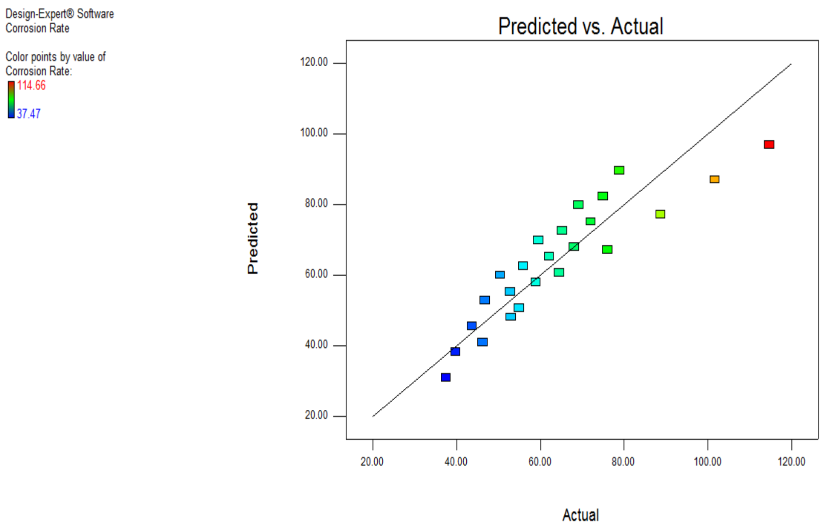
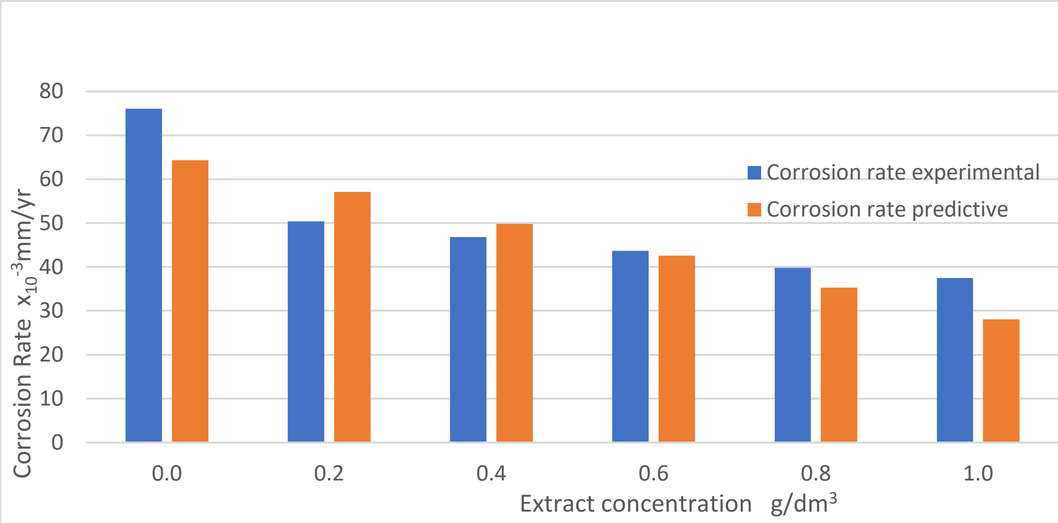


Figure 1: Plot of Predicted Versus Actual Corrosion Rate of Ficus Thonningii Bark Extract

Figure 2: Corrosion rate at 298K



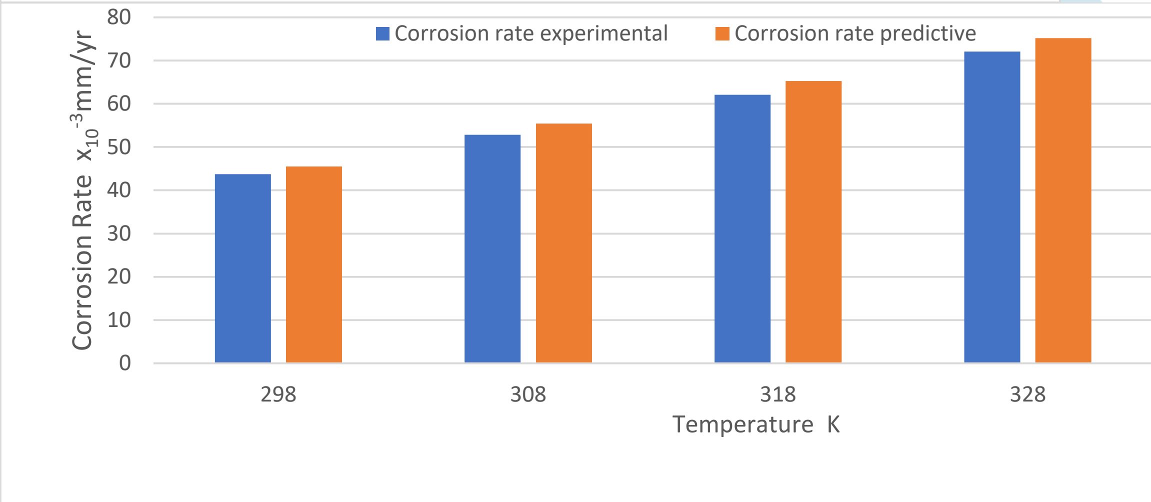


Figure 3: Corrosion rate at extract concentration of 0.6g/dm^3

3.4 Optimisation of corrosion rate

The graph shown in Figure 3 is a 3-D which shows the corrosion rate of the mild steel at optimum operational conditions of extract concentration and temperature. The graph display corrosion rate of the mild steel exposed in 1M HCl medium in the presence of the Ficus thonningii bark extract as a function of factors of extract concentration and Temperature.

The interactive behaviour of the extract concentration and temperature on the corrosion rate of the mild steel was displayed and the optimal condition of the process was also determined.

Predicted corrosion rate (31.02082 x 10^{-3}) mm/yr was optimum at 298K and 1.0g/dm extract concentration.

Additional experiments were carried out to validate the result obtained from the selected variables (temperature and concentration), the measured corrosion rate (31.0218 x 10^{-3}) obtained, as shown in Figure 4. The measured value was close to the predictive values (31.02082 x 10^{-3}); this validates the RSM optimizing tools used for the procedure in accordance with [3], who validated the predicted inhibition efficiency.

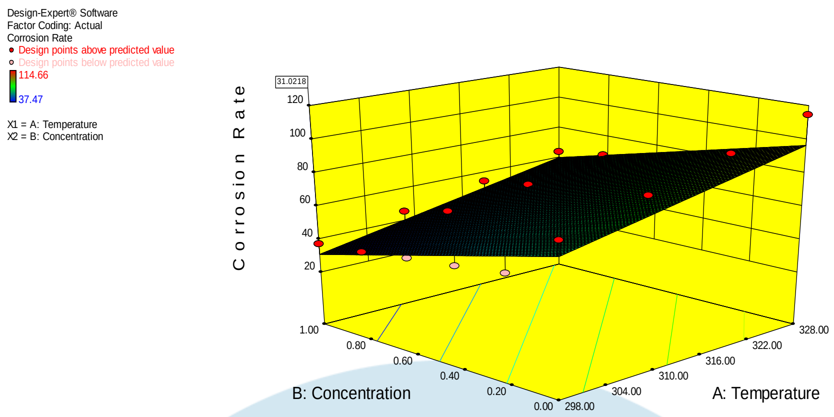


Figure 4: 3-D Surface Plot for optimum Corrosion Rate

Figure 5: Optimization Result of Ficus Thonningii Bark Extract

Corrosion rate = $x10^{-3}$

Conclusion

The Linear equation developed by applying response surface methodology showed that extract concentration and temperature affects the corrosion rate of mild steel exposed to 1M HCl solution. The predictive model equation of corrosion rate for Ficus thonningii bark is:

Corrosion rate = -227.01721 + 0.98763 * Temperature – 36.27571 * concentration

The predictive optimum corrosion rate of 31.02082 x 10^{-3} mm/yr was obtained from RSM modelling for the experimental design with 1.0g/dm Ficus thonningii bark extract concentration at 298K as the optimum process variables, the validated optimum value is 31.0218 x 10^{-3} mm/yr, which is in agreement with the predictive value generated by the regression model. The model can be used to predict the corrosion rates of mild steels exposed to varying temperatures in an acidic medium, using different concentration of Ficus thonningii bark extract as inhibitors

5. Credit author statement

All authors contributed to the study conception and design. Olisakwe H.C. and Ikpambese K.K: Conceptualization; Formal analysis; Investigation; Resources; Software; Validation; visualization; Writing-original draft; Writing-review and editing. Tuleun L. T: Conceptualization; Methodology, Supervision; Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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