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EVALUATION OF GROUNDNUT LEAVES EXTRACT AS CORROSION INHIBITOR ON MILD STEEL IN 1M SULPHURIC ACID USING RESPONSE SURFACE METHODOLOGY (RSM)

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ABSTRACT

The inhibiting effects of Groundnut Leaves Extract (GLE) on corrosion of Mild Steel in 1M H_2SO_4 acidic media was investigated in this study. The effect of the concentration of inhibitor (0.2 - 1.0 g/l), time (3 – 5 days) and temperature (40−60 ℃) on corrosion rate and Inhibition efficiency were investigated using Response Surface Methodology (RSM). The Phytochemical analysis of the GLE was performed; the result showed that organic constituents were present which made the Groundnut Leaves Extract a good inhibitor. Mathematical models were derived for the groundnut leaves extract. The optimum conditions obtained were temperature 49.72 ^{O}C, time 5 days and inhibitor concentration of 0.29 g/l while the optimum Inhibition Efficiency at this optimum condition was predicted to be 85.9%. The outcome of the ANOVA test confirmed a confidence level of 95%. Gravimetric experiment was carried out at these optimum conditions to validate the predicted optimum values. The obtained experimental value of 86.3% agreed closely with that obtained from the regression model. The micrographs result of Scanning Electron Micrographs analysis showed that passive layer of film was formed on the surface. This study has revealed that Groundnut Leaves Extract is a good inhibitor for the corrosion of Mild steel in 1M H_2SO_4 solution.

Keywords: Corrosion Inhibition, Groundnut Leaves Extract, Inhibition Efficiency, Optimization.

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1. INTRODUCTION

Corrosion is the destruction of material by a reaction with its environment. The petroleum industry is one of the most affected by corrosion due to the presence of numerous corrosive substances in the crude oil, which affect equipment and pipelines from the extraction of crude oil to the transportation of final products [1].

Corrosion Inhibitors are chemicals that react with a metallic surface or the environment the surface is exposed to; resulting in giving the surface a certain level of protection [2]. Inhibitors slow corrosion processes by either increasing the anodic or cathodic polarization behavior [3] reducing the diffusion of ions to the metallic surface or increasing the electrical resistance of the metallic surface [4]. This practice have exploration has been centered on the utilization of green chemistry; which incorporates plant extracts that contain many organic compounds such as amino acids, alkaloids, pigments and tannins used as green alternatives for the toxic and hazardous compounds. Furthermore, due to the biodegradability, eco-friendliness, low cost and easy availability of the extracts of some common plants and plant products they have been studied as corrosion inhibitors for various metals and alloys under different environmental conditions. The environment may be liquid, gas or mixture of solid and liquid [5]

The use of inhibitors has been widely explored to decrease corrosion of mild steel used in acidic environments because they form various types of films in several ways; by adsorption, by the formation of bulky precipitates, and by the formation of a passive layer on the metal surface. The inorganic salts and organic compounds, which are proven as good inhibitors have limited solubility and toxic properties, because they contain nitrogen, sulfur or oxygen. Aware of that, researchers orient their corrosion inhibition studies toward human health and safety consideration awareness. These environmentally friendly inhibitor sources are natural products which are non-toxic, cheaply processed, biodegradable and readily available in plenty and are sometimes regarded as agro-waste. In this regard, plant-based inhibitors such as Red Peanut skin [6] Jatropha stem [7]; Rice husk [8], Bitter Kola Stem [9] and Pawpaw leaves [10] among several others have recorded varied levels of success; research efforts are still ongoing to search for other green inhibitor alternatives. This present work is tailored to the use of Groundnut Leaves Extract as eco-friendly source to produce green corrosion inhibitor for mild steel. Furthermore, using this agricultural waste will provide two advantages to the environment. The volume of agro waste could be reduced and the low cost inhibitor could be easily produced for reducing the pollution problems arising from structural steel corrosion. The aim of this research is to determine the inhibition efficiency of the groundnut leaves extract as a corrosion inhibitor on mild steel in Sulphuric $acid$ (H_2SO_4) solution.

2. EXPERIMENTAL

Response Surface Methodology (RSM) is a statistical tool used to investigate the interaction between several illustrative variables and one or more response variables. The process of RSM includes designing of a series of experiment sufficient and reliable measurement of the response and developing a mathematical model of the second order response surface with the best fittings. The Software Design Expert (6.0.8) was used to analyze the data.
The mathematical empirical model is defined as:

$$Y = B_0 + B_1X_1 + B_2X_2 + B_{11}X^2{}_1 + B_{22}X^2{}_2 + B_{12}X_1X_2$$

Where Y: is the response or dependent variable: X_1 and X_2 are the independent variables B_0,B_1,B_2, B_{11}, B_{22}, B_{12} are the regression coefficients. The theory and applications of RSM are highlighted in the literature [11].

3. EXPERIMENTAL DESIGN

A central composite design of 20 experimental runs which included 3- operating variables was established for the experiments used for studying the effect of: temperature of the solution, Time of exposure and Inhibition Concentration of extract on corrosion parameters using GLE.

The Factor levels with the corresponding real values are shown in Table 1 while the Design Matrix is shown in Table 2 The matrix for the three variables was varied at 3 levels (-1, 0 and +1). As usual the experiments were performed in random order to avoid systematic error
The following steps in RSM considered for this study

1. Design of experiment using Central Composite Design (CCD) to obtain the points where the experimental run was performed.
2. Experimental observation of the corrosion inhibition effects of the various factors at the design points.
3. Obtaining a mathematical model expressing the relationship between the process factors and the percentage inhibition efficiency.
4. Prediction of the Optimum values of the process for the maximum inhibition efficiency using RSM.
5. Experimental verification.

The RSM was used to analyze the responses. The ANOVA and graphical analyses of the inhibition efficiency and corrosion rate were carried out. The mathematical models in terms of coded factors were obtained. The models in terms of coded factors were used to make predictions about the response for given levels of each factor. Optimum inhibition parameters were also obtained.

3.1. Preparation of Mild Steel Coupon

Mild Steel, obtained from the Mechanical Engineering laboratory was cut into Coupons of 2 cm by 2 cm in dimension with thickness of 0.2 cm and a hole 0.2cm drilled in the middle, cleaned with emery paper to expose the shiny surface, degreased with acetone to remove any oil impurities, washed with distilled water and then dried in air and stored in a desiccator., The coupons were weighed and recorded as the initial weight of the mild steel before being exposed to the corrosion media containing inhibitor.

3.2. Preparation of Plant Extract

The groundnut leaves (GL) used was collected from Landmark University Teaching and Research farm and dried for five (5) days under a shield, then grinded to fine powder and kept for further analysis. For every extraction process conducted 30grams of the ground groundnut leaves was measured and extracted using the Soxhlet extractor for 4hours using 180g of ethanol. After extraction, the samples were then re-extracted to remove the ethanol. The filtrates obtained were ~~be~~ used to prepare inhibitor concentrations in sulphuric (H_2SO_4) corrodents.

3.4. Phytochemical Analysis

The Groundnut Leaves Extract (GLE) was first grinded; then subjected to phytochemical analysis to check the secondary metabolites: alkaloids, flavonoids, tannins, steroids and saponins present. The result of the phytochemical analysis is shown below in Table 3.

3.5. Weight Loss (gravimetric)

Weight loss measurements was conducted under total immersion using 250 ml capacity beaker containing prepared solution at 40°C to 60°C which was maintained in a thermostatic water bath. The Mild Steel Coupons were weighed and dropped in the corrodent. The coupons were retrieved with time variables of (3 -5 days); exact Temperature of the solution and the inhibitor concentrations (0.2 – 1.0 g/l) in extract for each run were followed using the design matrix format in Table 3. After each exposure time, the mild steel coupons were removed, washed thoroughly to remove the corrosion product with emery paper, rinsed with distilled water and then dried in acetone. The mild steel was re-weighed to determine the weight loss, in gram by the difference of mild steel weight before and after immersion. The corrosion rates (g/cm^2h) in the absence and presence of the studied inhibitors were determined. Weight loss was calculated by finding the difference between the weight of each coupon before and after immersion;

$$\Delta W \ = W_b - \tag{1}$$

Where W_b is the weight before immersion; $W_a$is the weight after immersion. While the corrosion rate (g/cm^2h) in absence and presence of inhibitors was calculated using equation (2).

$$CR=\frac{\Delta w}{At} \tag{2}$$

Where Δw is the weight loss (g) after exposure time t (days), A is the area of the specimen (cm^2) and t is the time of exposure in days, and CR is the corrosion rate at each exposure time. Inhibition efficiency was calculated as

$$IE\% = \frac{w_o - w_1}{w_o} \times 100 \tag{3}$$

Where, w_1 and w_0 are the weight loss values in presence and absence of inhibitor, respectively. A is coupon; t is time of exposure, IE % are the inhibition efficiency.

Morphological observations of the corroded Mild Steel samples were carried out using Scanning Electron Microscopy (SEM).

4. RESULTS AND DISCUSSION

4.1. Evaluation of Regression Model for Inhibition Efficiency (%I.E)

The correlation between the experimental process variables and the inhibition efficiency was evaluated using the Central Composite Design (CCD) modeling technique.

The effect of inhibition on inhibition efficiency in 0.1M H_2SO_4 is presented in Table 4 which showed the weight loss of the coupons, corrosion rate and inhibition efficiency. It showed that inhibition efficiency has increased since the concentration of the extract (inhibitor) is not directly visible in it.

The correlation between the experimental process variables and the inhibition efficiency was evaluated using the Central Composite Design modeling technique. A polynomial quadratic regression equation of the form was derived

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4{X_1}^2 + B_5{X_2}^2 + B_6{X_3}^2 + B_7X_1X_2 + B_8X_1X_3 + B_9X_2X_3 \tag{4}$$

Where fitted between the response (% inhibition efficiency(Y)) and the process variables: time (X_1), temperature (X_2), and inhibitor concentration (X_3). The final response equation for in terms of coded is as given:

$$Y = 54.94 + 2.10X_1 - 10.42X_2 + 2.51X_3 + 12.95{X_1}^2 - 28.55{X_2}^2 + 8.20{X_3}^2 + 4.19X_1X_2 - 15.79X_1X_3 + 9.34X_2X_3 \quad (5)$$

Furthermore, Table 5 presented the analysis of variance (ANOVA).The degree of freedom (DF), Fisher test (F-test) and probability values (p-value) are 9, 4.22 and 0.0173 respectively. The p-value is less than 0.05, which implied that the model is significant. The ANOVA results showed that the quadratic model is suitable to analyze the experimental data. To develop a statistically significant regression model, the significance of the regression coefficients was evaluated based on the p-values. The coefficient terms with p-values more than 0.05 are insignificant and are removed from the regression model. The analysis in Table 5 showed that the linear, quadratic and interaction effects of temperature and inhibition concentration are significant model terms. The model reduces to Equation 6 after eliminating the insignificant coefficients

$$Y = 54.94 - 10.42X_2 - 28.55{X_2}^2 - 15.79X_1X_3 \quad (6)$$

The analysis of variance indicated that the quadratic model was significant and adequate to represent the actual relationship between inhibition efficiency and the significant model variable as predicted by small p-value. This means that the model is a good representation of variation in the experimental data. The adequate correlation between the experimental values of the independent variable and predicted values further showed the accuracy of the model. The significant and adequacy of the established model was further collaborated by high value of the coefficient of determination of 79.17% as shown in Table 5 that gives the reason to conclude that the model is appropriate.

However; in Table 5, shown as ANOVA for the corrosion inhibition of Mild Steel in H_2SO_4 by Groundnut Leaves Extract; the “Pred R- squared” implies that the overall mean is a better predictor of the inhibition efficiency than the current model. Since “Adeq Precision” measures the signal to noise ratio; a ratio greater than 4 is desirable and from the ANOVA we have a ratio of 8.279. Furthermore; Regression Coefficient and significance of Response Surface Quadratic is shown in Table 6

4.2. Experimental Validation

The optimum level of 85.97%. Predicted by the software was validated, to confirm if significant or not as seen in Table 6. However; experimental value of 86.30% agreed closely with that obtained from regression model. Furthermore, Table 7 showed the experimental value, predicted value with the residual for the 20 experimental runs obtained from the CCD.

Table 6 Optimum parameters for the corrosion inhibition process

Optimum parameters		Optimum inhibition efficiency (%)
Inhibitor concentration (g/l)	0.29	85.97
Temperature (°C)	49.72	
Time (days)	5	

Table 7 Diagnostic case statistics of inhibition efficiency

Run	Coded variables			Experimental value	Predicted value	Residual
	X_1	X_2	X_3			
1	-1	0	-1	39.20	51.10	-11.90
2	1	0	-1	86.30	78.50	7.80
3	-1	1	0	15.60	3.21	12.39
4	1	1	-1	42.60	47.36	-4.76
5	-1	0	1	70.60	69.02	1.58
6	1	0	1	17.70	33.27	-15.57
7	-1	1	1	47.50	58.48	-10.98
8	1	1	1	48.20	39.48	8.72
9	-1	0	0	74.70	65.79	8.91
10	1	0	1	73.80	69.99	3.81
11	0	-1	1	54.90	36.81	18.09
12	0	1	0	10.60	15.97	-5.37
13	0	0	-1	57.10	60.63	-3.53
14	0	0	1	81.90	65.65	16.25
15	0	0	0	50.70	54.94	-4.27
16	0	0	0	50.70	54.94	-4.27
17	0	0	0	50.70	54.94	-4.27
18	0	0	0	50.70	54.94	-4.27
19	0	0	0	50.70	54.94	-4.27
20	0	0	0	50.70	54.94	-4.27

4.3. Surface Response Plots for Inhibition on Mild Steel

The interactive effects of the process variables on the IE (%) were studied by plotting a three dimensional surface curve against any two independent variable, while keeping the other variable constant. The 3D curves are shown in Fig 1 – Fig 3. It showed in Fig 1 that at a given inhibitor concentration inhibition efficiency increased with decrease in time, but also decreased with increase in temperature.

However; Fig 2 showed that inhibition efficiency increases with increase in inhibitor concentration but decreases with an increase in time

Nevertheless; increase in temperature with increase in inhibitor concentration but decrease in inhibition efficiency was observed in Fig 3, this could be attributed to the fact that the longer the metal stay in the acid the more it corrodes

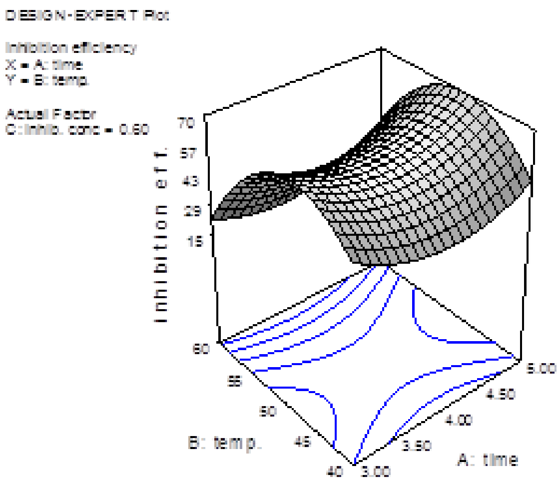

Figure 1 Effects of time and temperature on inhibition efficiency of GLE on Mild Steelat constant inhibitor concentration of 0.60g/l

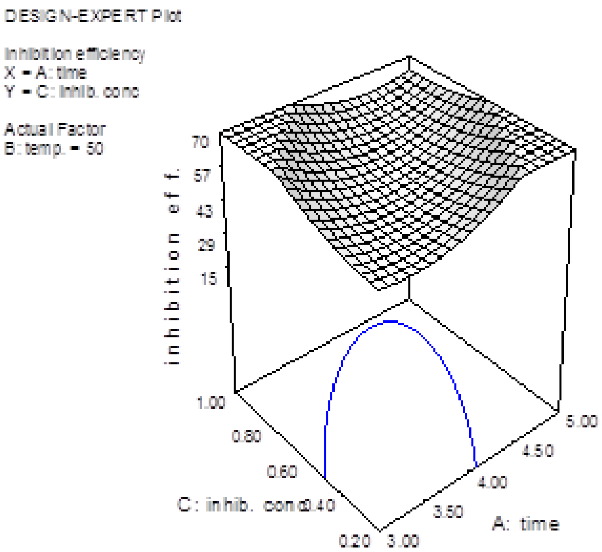

Figure 2 Effects of time and inhibitor concentration on inhibition efficiency of GLE on Mild Steel at constant temperature 50°C

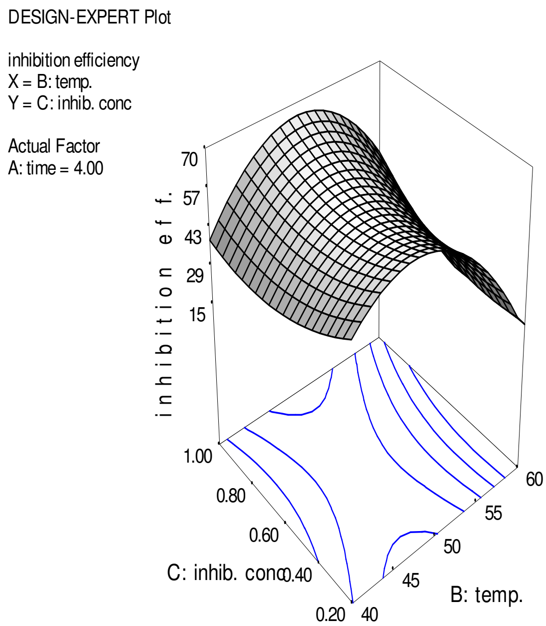

Figure 3 Effects of temperature and inhibitor concentration on inhibition efficiency of GLE on Mild Steel at constant time of exposure 4 days.

5. EVALUATION OF REGRESSION MODEL FOR CORROSION RATE

The correlation between the experimental process variables and the corrosion rate was evaluated using the Central Composite Design (CCD) modeling technique. A polynomial quadratic regression equation of the form:

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4{X_1}^2 + B_5{X_2}^2 + B_6{X_3}^2 + B_7X_1X_2 + B_8X_1X_3 + B_9X_2X_3 \quad (7)$$

Was used, where fitted between the response (Corrosion rate (Y)) and the process variables: time (X_1), temperature (X_2), and inhibitor concentration (X_3). The final response equation for in terms of coded is as given:

$$Y = 59.45 - 11.52X_1 + 23.93X_2 - 4.65X_3 - 8.58{X_1}^2 + 0.17{X_2}^2 - 16.73{X_3}^2 - 8.73X_1X_2 + 8.02X_1X_3 - 5.69X_2X_3 \quad (8)$$

Table 5 showed: degree of freedom (DF), Fisher test (F-test) and probability values (p-value) are 9, 7.16 and 0.0025 respectively.

From Table 8, the ANOVA result showed that the quadratic model is suitable to analyze the experimental data, develop a statistically significant regression model; the significance of the regression coefficients was evaluated based on the p-values. The coefficient terms with p-values more than 0.05 are insignificant and are removed from the regression model. The model reduces to Equation (9) after eliminating the insignificant coefficients

$$Y = 59.45 - 11.52X_1 + 23.93X_2 \quad (9)$$

The analysis of variance indicated that the quadratic model was significant. This means that the model is as a good representation of variation in the experimental data. The adequate correlation between the experimental values of the independent variable and the predicted variables further showed the adequacy of the model. The significance and adequacy of the established model was further collaborated by the high value of the coefficient of determination (R^2 is 86.57%) in Table 8; it showed that it is close to the "Adj R-Squared" of 0.7448 as one might normally expect. Furthermore, since “Adeq Precision” measures the signal to noise ratio;

a ratio greater than 4 is desirable and from the ANOVA with a ratio of 10.521 and this therefore makes the use of groundnut leaves extract as a corrosion inhibitor on Mild Steel in 1M H_2SO_4 very effective and the model appropriate.

The analysis in showed that the linear, quadratic and interaction effects of temperature and inhibition concentration are significant model.

Table 8 ANOVA for the corrosion inhibition of Mild Steel in $\boldsymbol{H_2SO_4}$ by Groundnut Leaves Extract

ANOVA for Response Surface Quadratic model; corrosion rate						
Analysis of variance table [Partial sum of squares]						
Source	Sum of squares	Degree of freedom (DF)	Mean of squares	F- values	P-value Prob > F	
Model	11253.65	9	1250.41	7.16	0.0025	Significant
X_1(time)	1326.41	1	1326.41	7.60	0.0203	
X_2(temperature)	5724.53	1	5724.53	32.79	0.0002	
X_3(inhibitor concentration)	216.60	1	216.60	1.24	0.2914	
$X_1{}^2$	202.60	1	202.60	1.16	0.3067	
$X_2{}^2$	0.081	1	0.081	4.650E-004	0.9832	
$X_3{}^2$	769.54	1	769.54	4.41	0.0621	
X_1X_2	609.70	1	609.70	3.49	0.0912	
X_1X_3	514.56	1	514.56	2.95	0.1168	
X_2X_3	258.78	1	258.78	1.48	0.2514	
Residual	1745.96	10	174.60			
Lack of fit	1745.96	5	349.19			
Pure error	0.000	5	0.000			
Cor. Total	12999.61	19				
Std. Dev.	13.21		R- squared			0.8657
Mean	46.88		Adj R- squared			0.7448
C.V%	28.19		Pred. R-squared			0.3858
Press	7984.12		Adeq. Precision			10.521

5.1. Surface Response Plots for corrosion rate on Mild Steel

The interactive effects of the process variables on the corrosion rate were studied by plotting a three dimensional surface curve against any two independent variable, while keeping the other variable constant. The 3D curves are shown in Fig 4 – Fig 7 respectively. Fig 4 showed the plot of predicted versus experimental. It showed that the plots were reasonably distributed near to the straight line indicating a good relationship between the experimental and predicted values of the response, and that the underlying assumptions of the analysis were appropriate. It also showed that the selected quadratic model was adequate in predicting the response variables for the experimental data. The predicted versus experimental plot gave a linear graph. The graph (3-D surface plot) in Fig 5 to Fig 7 showed the relationship between the factors and corrosion rate response of the designed experiment.

Fig 5, showed that corrosion rate increased with increase in temperature and also increased with increase with time; while Fig 6 showed that corrosion rate decreased with increase in inhibitor concentration and also increases with an increase in time, as shown in Fig 7, but decreases with an increase in inhibitor concentration. This assumed physical adsorption.

Furthermore; the Groundnut Leaves Extract obeys the mechanism of physiorption with the inhibition efficiency increasing with increase in inhibition concentration but decreases with time and temperature.

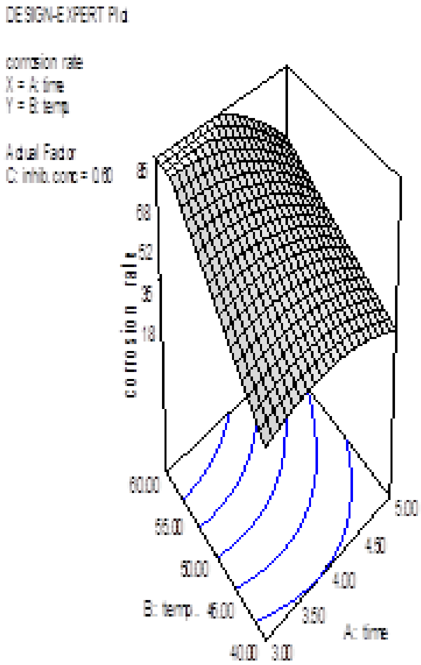

Figure 4 Plot of Predicted values versus the Actual Experimental values for corrosion rate of GLE on Mild Steel

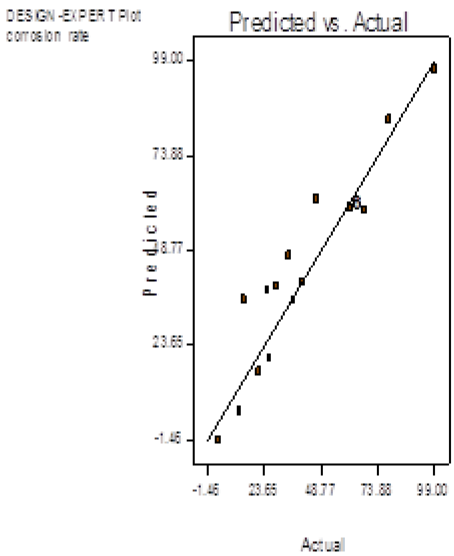

Figure 5 Effects of time and temperature on corrosion rate of GLE on Mild Steel

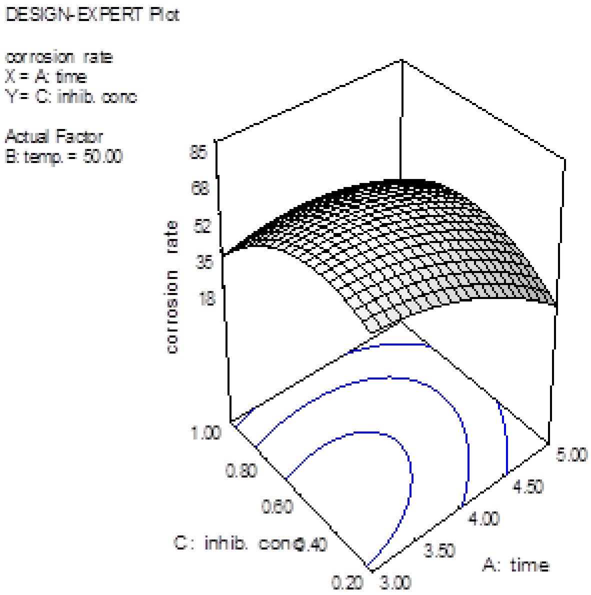

Figure 6 Effects of time and inhibitor concentration On corrosion rate of GLE on Mild Steel at Constant temperature'

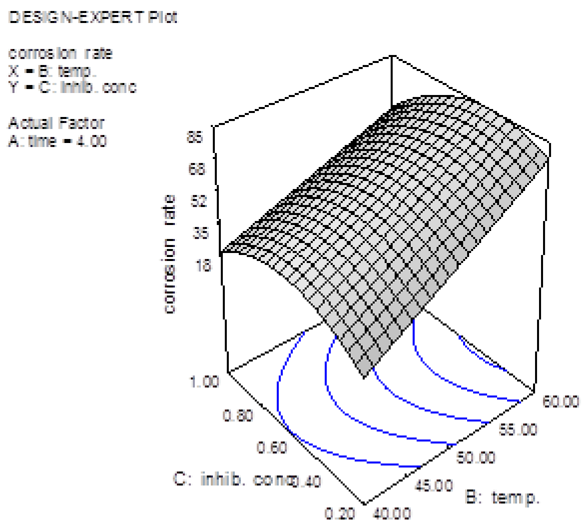

Figure 7 Effects of temperature and inhibitor concentration on corrosion rate Rate of GLE

6. DISCUSSION ON SCANNING ELECTRONS MICROGRAPHS (SEM) ANALYSIS

The micrographs revealed that in the absence of inhibitor, the surface was strongly damaged, due to corrosion while in the presence of inhibitor there was a much smaller damage on the surface as shown in Fig 8. This was attributed to the formation of a good protective film on the Mild Steel surface. Furthermore, in Fig 9, it was observed that the damage was very minute which showed a better protective film

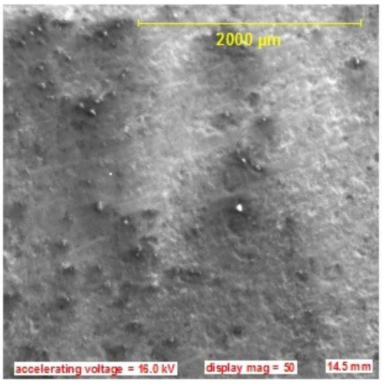

Figure 8 The SEM micrographs for mild steel In 1.0M in the absence of corrosion Inhibitor (blank)

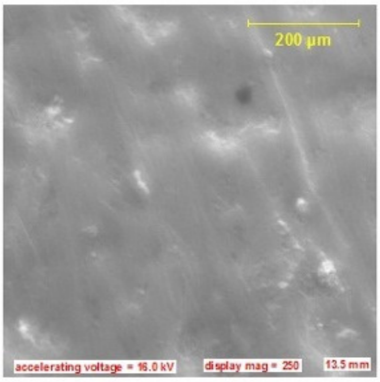

Figure 9 SEM micrograph of Mild steel mild steel exposed to 1.0M with corrosion inhibitor 0.29. G/l extract

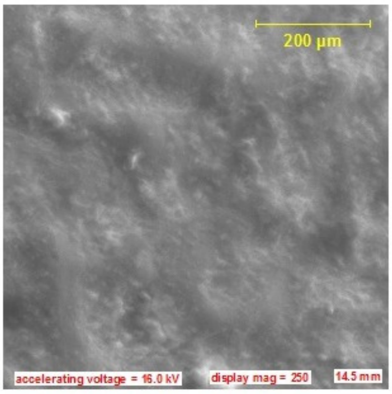

Figure 10 SEM micrographs for mild steel exposed to 1.0M H_2SO_4 with corrosion inhibitor GLE; validated after prediction.

7. CONCLUSION

Groundnut Leaves Extract (GLE) shows an outstanding ability to provide improved corrosion resistance of Mild Steel in acidic medium (1.0M of H_2SO_4). An optimal operating condition for inhibition efficiency of 85.9% was observed. The validation of the model was carried out at this

optimal process conditions; time: 5 days, temperature: 49.72 ℃ and inhibitor concentration: 0.29 g/l to validate the predicted optimum value.

The experimental value of 86.3% agreed closely to that gotten from the Response Surface Methodology. The inhibitive ability of the GLE depends on protective oxide layer formed on the mild steel by adsorption due to the presence of alkaloids, tannins, and steroids. It can be concluded be used to reduce metal dissolution in acidic media

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