



INVESTIGATION OF PUMPKIN POD EXTRACT AS CORROSION INHIBITOR FOR CARBON STEEL IN HCL SOLUTION

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ABSTRACT

This work focused on the use of natural, and eco-friendly waste material as an inhibitor for inhibiting carbon steel corrosion. In order to obtain minimum rate of corrosion on carbon steel, optimization of the process factors that affect carbon steel corrosion was undertaken using the Response Surface Methodology (RSM). Three parameters were fluctuated viz; temperature, pumpkin pod extract concentration and time of exposure and their corresponding effects on rate of corrosion of carbon steel were ascertained. The data obtained was fitted to a model that is quadratic which was subsequently validated. The predicted lowest rate of corrosion by the model is 2.5427mm/yr with optimal conditions of 178.07ppm of pumpkin extract concentration, exposure time of 2.26hrs, and 35.28°C of temperature. The conditions were validated in three replicates and corrosion rate of 2.75mm/yr was obtained.

Keywords: corrosion, optimization, pumpkin pod, response surface methodology.

1. INTRODUCTION

The driving force that causes metals to corrode is a natural consequence of their temporary existence in metallic form [1]. Corrosion is thus inevitable for metals and alloys. Corrosion is the most predominant cause of metal failures today, surpassing other failure modes like fatigue, creep, impact, and others. The cost of corrosion is not only the cost of replacement but additional costs as well such as; Loss of production due to shut down or failure, High maintenance costs, Compliance with environmental and consumer regulations, loss of product quality due to contamination from corrosion of the materials, a high fuel and energy costs as a result of leakage from corroded pipes, extra working capital and larger stocks. The various corrosion prevention techniques available are based on two working principles; alter the component or alter the environment. The alteration of the component can be through design changes which are especially effective where the corrosion is affected by fluid flow characteristics (erosion corrosion, cavitation), presence or absence of gaseous phases (cavitation, pitting), dissimilar metal contact (galvanic corrosion), and solution stagnation (crevice corrosion). Alteration of component also

includes alteration of the material of construction, i.e. choosing a different alloy grade for the same component, and the application of coatings. On the other hand alteration of the environment would include the use of inhibitors, or the application of electrochemical protection (anodic or cathodic) [2]. Previous studies have used extract of plant materials on various metals and their alloys in the presence and absence of many inhibitors for corrosion control. Stainless steel [3; 4] using pomegranate husk extract, extract of Rhizophora mangle leave, extracts of pomegranate, Alkaloids extracted from isertiacoccinea plant. Mild steel [5 – 13] using the extract of phyllanthusfraternus extract, extracts of Anacylis pyrethrum leaf, Retamaretam extracts, Saracaindica (Asoka) leaf extracts. Ethanol extracts from leaves, bark, and root of Nauclealatifolia, extract of dodonaeaviscosa leaves, ethanol extracts of Andrographispaniculata (king bitter) Vernoniaamygdalina (bitter leaf). Carbon Steel [14 – 16] using Reuterallutea (Desf) Maire) Punica plant extract, oil of Thymus Pallidus plant from Morocco. Aluminium and its alloy [17 – 19] using green tea extract, Solanumtrilobatum leaves extract, and Morindacitrifolia leave, M. citrifolia leave extract

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respectively. Uwah, *et al* [20] used copper with extract of Cannabis plant as an inhibitor Pumpkin (*Telfairia occidentalis*) Pod is an agricultural waste readily available in Nigeria, it is biodegradable, and contain phytochemical constituents such; tannins, flavonoid, steroid, terpenoids, saponin, carbohydrates, amino acids and proteins [5, 21]. These organic compounds contain polar functions with N, S, O atoms as well as conjugated double bonds or aromatic rings in their molecular structure, which are the major adsorption centre. The use of Pumpkin pod extract as corrosion inhibitor and the statistical analysis of the inhibition process on carbon steel is not available in any open literature to the best of our knowledge, hence the need for this research work. In this work, three parameters namely; temperature, pumpkin pod extract concentration, and time of exposure were optimized. In order to minimize the rate of corrosion of carbon steel, RSM was used using the Box – Behnken experimental design to determine the effects of three factors (pumpkin pod extract concentration, temperature, and time of exposure) and their reciprocal effect on rate of carbon steel corrosion. This work established the process conditions in achieving minimum rate of corrosion on carbon steel in acidic environment which could be applied in industrial scale.

2. MATERIALS AND METHODS

2.1. Materials

Pumpkin pods (*T. occidentalis*) were obtained from Agbarho Community in Effurun, Delta State, Nigeria. Carbon steel was procured from accredited iron sheet dealer in Effurun and machined in Mechanical Workshop at Federal University of Petroleum Resources, Delta State. HANNA model pH – 211 (pH meter), Genlab oven model Mino/75/f (oven), weighing balance of model (BH – 600), and beakers, were employed for this corrosion study.

Hydrochloric acid, acetone, and ethanol solutions used were of analytical grades and were procured from a qualified chemical dealer in Effurun, Delta State, Nigeria. Distilled water was procured from the Department of Chemical Engineering Laboratory, Federal University of Petroleum Resources, Effurun Delta, State, Nigeria for sample preparation and solutions.

2.2. Methods

2.2.1. Pre-treatment of sample and sample characterization

The samples were thoroughly washed thereafter sun dried and pulverized into powdery form with the aid of

laboratory blender. It was then sieved with a sieve of 0.143 μ m mesh. The sample was later stored in a desiccator prior to use.

2.2.2. Fourier Transform Infrared Spectroscopy (FTIR)

Pumpkin pods (*T. occidentalis*) 0.143 μ m particle size was observed with FTIR spectroscopy (Buck Scientific model 530) with the range 500 - 4000 cm^{-1} (wavelength). The background material used in the analysis is potassium bromate (KBr).

2.2.3. Extraction of Pumpkin pods (*T. occidentalis*) extract

The Pumpkin pods (*T. occidentalis*) were washed thoroughly with running water to remove debris. The washed samples were sun dried for 14 days and ground to a particle size of 0.143 μ m. The sample was stored in a desiccator before used. 50g of the dried Pumpkin pods (*T. occidentalis*) powder was transferred into a 500mL Soxhlet extractor and 500mL of 70% ethanol reflux continuously for 3 hours at 78°C. The set-up was placed on a heating mantle and the Pumpkin pods (*T. occidentalis*) extract was extracted exhaustively by heating the solution. The extract was obtained after recovering ethanol in a Rotary evaporator (model R-210) at 40°C.

2.3. Procedure of the experiment

The gravimetric or weight loss method was used. The corrosion study was carried out using the method described by [22]. The carbon steel was mechanically polished with silicon carbide abrasive paper, degreased with ethanol, washed in distilled water and dried in acetone. Each carbon steel coupon was sized 40mm \times 20mm \times 2mm. Before polishing, a hole of 0.1cm was drilled on each coupon. The coupon was suspended with the aid of a nylon thread in a 100ml beaker with 100ml of 1.5M HCl at three; different pumpkin pod extract concentrations, exposure time, and temperature.

2.3.1. Optimization studies of corrosion inhibition process on carbon steel

Optimization of the process variables affecting the rate of corrosion was carried out using Response Surface Methodology (RSM). The three factors varied were temperature, pumpkin pod extract concentration, and exposure time and their corresponding effects on rate of carbon steel corrosion in acidic medium was investigated. 17 runs of experiments were produced

using Box Behnken Design (BBD). The model fitness was evaluated using the test of significance and analysis of variance (ANOVA) that is type III. The selected variables pumpkin pod extract concentration, exposure time, temperature represent X_1 , X_2 , and X_3 respectively. This is shown in Table 1.

The coefficient of the polynomial model was determined using the multiple regressions as shown in Equation (1).

$$Y = b_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j} \beta_{ij} X_i X_j + e \quad (1)$$

Where, Y is the weight loss, b_0 is the intercept, b_{ij} is the interaction effect, and b_{ii} denotes the quadratic coefficients of X_i , and e is the random error [23 – 24]. Design Expert 7.00 software was used to design and analyze the data from the experiment. This is a statistical software package that does design of experiments, comparative tests and optimization of the process variables. It is also used to study the parameters on the yield of a process using the graphical tool.

2.4. Corrosion rate determination

The expression for measurement of corrosion rate (C.R) in millimeters penetration per year (mm/yr) was

used to measure the rate of corrosion rate for the specimens, which was expressed in equation 2.

$$C.R. = \frac{87.6w}{at\rho} \quad (2)$$

Where, w is corrosion weight loss of carbon steel (mg), a is the total surface area of the specimen in (cm²), t is the exposure time in hours (hr), and ρ is the density of the specimen (g/cm³).

3. RESULTS AND DISCUSSION

3.1. FTIR analysis of Pumpkin pod (*T. occidentalis*) extract

The FTIR spectrum of Pumpkin pods (*T. occidentalis*) extract is shown in figure 1. At broad band of 3324cm⁻¹ corresponds to an alcoholic –OH stretching group. The peak at 2974 cm⁻¹ can be assigned to alcoholic C-H group. 1639 cm⁻¹ wavelength indicates an aromatic (C=C) stretching while the value of 1728 cm⁻¹ suggest (C=O) stretching frequency. Aromatic rings due to aromatic skeletal vibrations is noticed at 1508 cm⁻¹ and 1421cm⁻¹ can be assigned to aromatic rings due to aromatic skeletal vibrations. The broad band of 1329 cm⁻¹ seen in the spectrograph is due to bending vibrations of OH group while 1209 cm⁻¹ is due to guaicynl ring in conjunction with C-O stretching group. The presence of aromatic, and carbonyl groups in the extract suggest that it can be used as an inhibitor.

Table 1: Experimental factors codes and level

Variables	Symbol	Low Factor (-1)	Mid-point Factor (0)	High Factor (+1)
Pumpkin pod extract concentration, (ppm)	X_1	50	150	250
Exposure time, (hr)	X_2	2	7	12
Temperature, (°C)	X_3	35	45	55

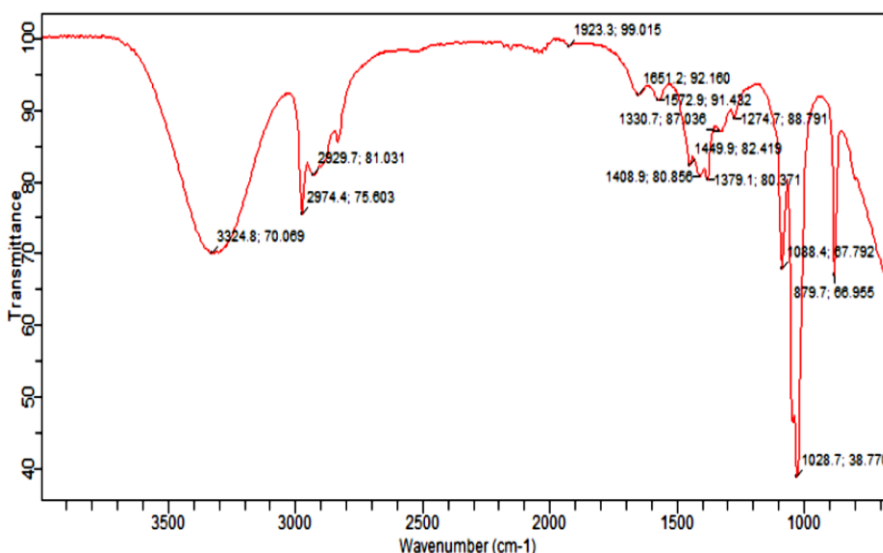


Fig. 1: FTIR spectra of Pumpkin pod extract

Table 2: ANOVA of Regression Equation Results for Quadratic Model

Sources	Sum of squares	DF	Mean square	F – Value	P - value
Model	2242.23	9	249.14	519.78	<0.0001
Conc. of extract	106.30	1	106.30	221.78	<0.0001
Exposure time	897.94	1	897.94	1873.39	<0.0001
Temperature	946.78	1	946.78	1975.28	<0.0001
Concentration and time	26.31	1	26.31	54.90	0.0001
Conc. and Temp.	16.35	1	16.35	34.11	0.0006
Time and Temp.	167.40	1	167.40	349.25	<0.0001
Conc. of extract ²	15.14	1	15.14	31.60	0.0008
Exposure time ²	10.02	1	10.02	20.91	0.0026
Temperature ²	48.68	1	48.68	101.55	<0.0001
Residual error	3.36	7	0.48		
Lack of fit	3.34	3	1.11	256.23	<0.0001
Pure error	0.017	4	0.004342		
Corelation Total	2245.59	16			

Table 3: Optimization objective

Constraints	Goal	Lower limit	Upper limit
Concentration of pumpkin extract	in range	50	250
Contact time	in range	2	12
Temperature	in range	35	55

3.2. Statistical analysis of the Response Surface Methodology Quadratic Model of BBD

Statistical testing of the quadratic model was done using analysis of variance (ANOVA) as depicted in Table 2. The F – value was used to check the significance of the model. The high F – value of 519.78 and p – value < 0.0001 indicated that the quadratic model used is significant. Thus, the data obtained experimentally is well represented by the quadratic model. The standard deviation value of 0.69 is realized as seen in Table 4. The coefficient of determination (R²) is defined as the ratio of the explained variation to the total variation, and is a measure of the degree of fitness of the model [3].

[25] suggested that a good model fit should yield a coefficient of correlation (R²) of at least 0.8. Coefficient of correlation (R²) value of 0.9985 was obtained for the model which is closer to 1 suggesting that the predicted values are close to the actual values used in the experimental design, signifying that the model is accurate [26]. The results from this investigation showed that the selected factors were sufficiently represented by the obtained model. This also described that there is an actual correlation among the factors selected in the experiment. It showed that 99.85% of the total variation in the corrosion rate of pumpkin pod extract can be connected experimentally to the studied variables. The p – values was used to check the significance of each of the coefficient in the model. An adequate precision

ratio of 80.880 is obtained in this work this indicates an adequate signal to noise ratio. It can also be suggested that the model can be used to navigate the design space. The precision ratio value gotten confirmed the adequacy of the model signal. The goodness – of – fit of the regression equation was measured using the adjusted coefficient of determination (R²). The predicted R² value of 0.9762 which indicate the model power in response prediction. The coefficient of variation (CV) for the standard deviation of the mean and experimental data is 4.48%. This showed a better reliability and precision of the experiments carried out [27 – 28]. The reproducibility of the data value was confirmed from the coefficient of variation value obtained which is lesser than 10%. A p-value lower than 0.05 indicates that the model is statistically significant, whereas a value higher than 0.1000 indicates that the model is not significant [6]. The significance of each coefficient is also established using the Probability value (P – value) and the ratio of the mean square (F – value) at 5% confidence level. It can be seen that corrosion of stainless steel using pumpkin pod extract as inhibitor was significantly affected by (X₁, X₂, and X₃, p<0.0001) linear terms but exposure time and temperature has a good synergistic effect on the rate of corrosion. It is obvious that all the interaction terms (X₁X₃, X₁X₂ and X₂X₃) were also significant at the probability level of (p<0.05) but it can be seen that only exposure time and temperature that has positive synergistic effect

on the rate of carbon steel corrosion likewise all the quadratic terms (X_1^2 , X_2^2 , and X_3^2) were also significant at the probability level of ($p < 0.05$). This suggest a positive interaction between all the process variables studied which were noted to be the core factors influencing the rate of corrosion, and weight loss of the carbon steel. The Response Surface Methodology (RSM) model for the rate of corrosion of carbon steel using pumpkin pod extract as an inhibitor was optimized using the desirability function of the Response Surface Methodology. The optimum conditions predicted from the quadratic model as shown in Table 3 were pumpkin pod extract concentration (178.07 ppm), exposure time (2.26 hours), and temperature (35.28 °C) that correspond to the corrosion rate of 2.5423mm/yr. These values were validated experimentally with an average corrosion rate of 2.75 mm/yr from three replicates, this was in good agreement with the optimum result predicted by the regression model. The statistical estimates of the Box Behken design was shown in Table 4.

The experimental testing of full quadratic model is expressed by the second order polynomial equation in term of actual values below:

$$Y = 12.24 - 3.65x_1 + 10.59x_2 + 10.88x_3 - 2.56x_1x_2 - 2.02x_1x_3 + 6.47x_2x_3 + 1.90x_1^2 + 1.54x_2^2 + 3.40x_3^2 \quad (3)$$

where, Y is the rate of corrosion (mm/yr), x_1 is concentration of pumpkin pod extract (ppm), x_2 is exposure time (hr), and x_3 is the temperature (°C).

Table 4: Statistical estimates of BBD

Parameters	Value
Standard deviation	0.69
Mean	15.46
Coefficient of variation (%)	4.48
Coefficient of determination (R^2)	0.99985
Predicted Coefficient of determination (R^2)	0.9762
Adjusted Coefficient of determination (R^2)	0.9966
Adequate precision	80.880

The predicted and experimental data of the rate of corrosion (mm/yr), were investigated also to determine their correlation this is shown in figure 2. The data points on the plot were discreetly distributed

and very close to the horizontal point line. This suggests a very good relationship between the predicted values and experimental values obtained for the response, this also implied the strong predictive power of the model used. This further elucidate the assumptions made that the data can be used to navigate the design space. In addition, a normal plot of residuals between the normal probability (%) and the internally studentized residuals was also obtained as seen in figure 3. In this way, the residuals was checked to determine how well the model satisfies the assumptions of analysis of variance (ANOVA), and the internally studentized residuals can be used to measure the standard deviations separating the experimental and predicted values [7]. This suggests a very good relationship between the predicted values and experimental values obtained for the response, this also implied the strong predictive power of the model used. This further elucidate the assumptions made that the data can be used to navigate the design space.

3.3. Surface Response Plots

3-D surface response plots were presented in Figures (4 – 6) based on the analysis of the full quadratic model. It was used to give an important evidence on the system behaviour within the experimental design on the corrosion rate of stainless steel. Figure 4 described the effect of pumpkin pod extract concentration (x_1) and time of exposure (x_2) on the rate of corrosion, it can be seen that the corrosion rate is reduced as pumpkin pod extract concentration is increased with time of exposure. This also affirmed the negative synergy of the time of exposure in the model obtained for carbon steel corrosion. Figure 5 depicted the effect of temperature (x_3), and pumpkin pod extract concentration (x_1), on the rate of corrosion, it was observed that lower temperature and higher pumpkin pod extract concentration favoured a decreased in rate of corrosion on carbon steel. This also confirmed the synergetic effect of these two terms on rate of carbon steel corrosion. Figure 6 showed the effect of exposure time (x_2), and temperature (x_3) on rate of corrosion, it was revealed that the higher temperature with increased in exposure time favoured a higher rate of carbon steel corrosion studied. This also confirmed the synergistic effect of these two variables of the rate of corrosion.

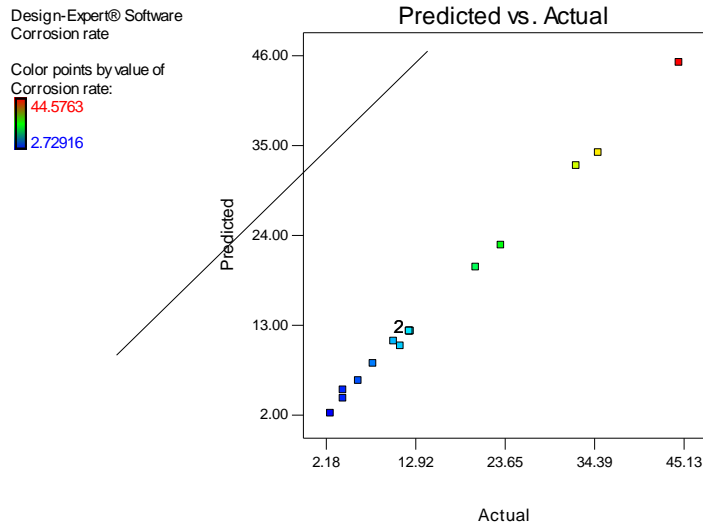


Fig. 2: Predicted values of the model versus the experimental data values.

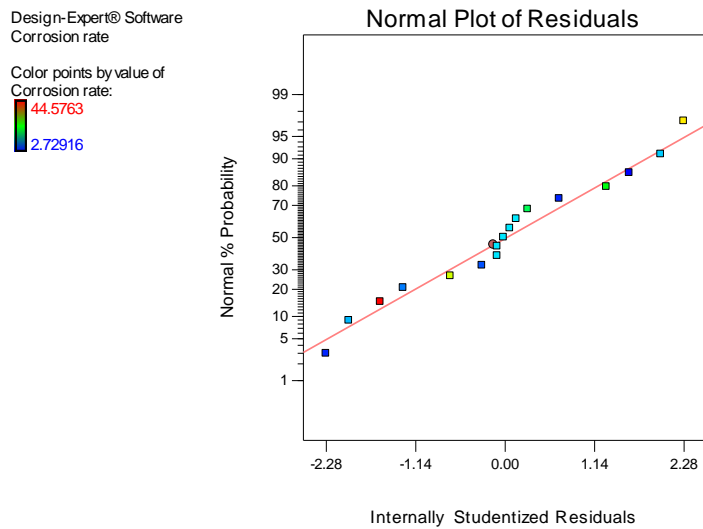


Fig. 3: Normal values of the residuals versus the internally studentized residuals

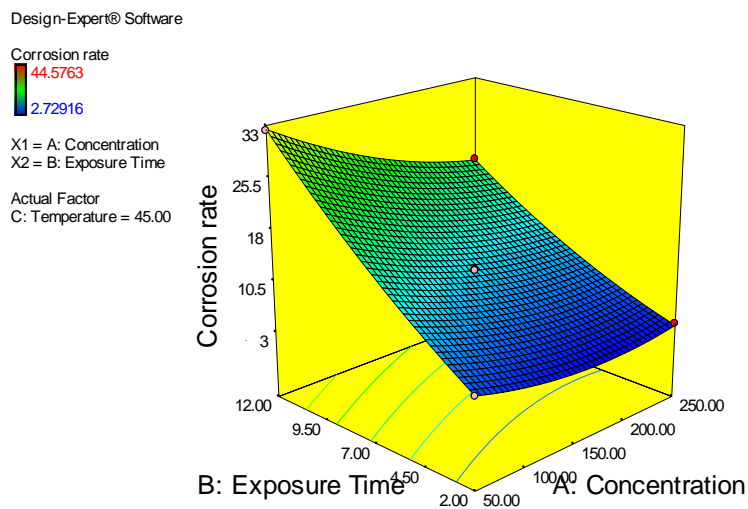


Fig. 4: Surface Plot for the influence of pumpkin pod extract concentration, time, and interface with corrosion rate.

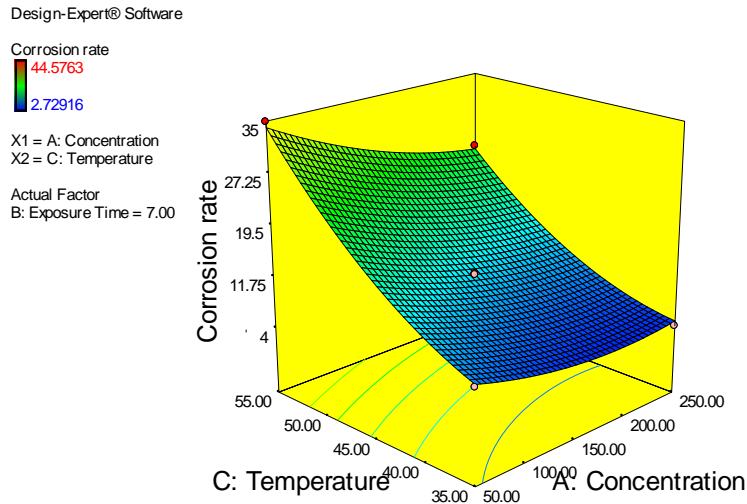


Fig. 5: Surface Plot for the influence of pumpkin pod extract concentration, temperature, and interface with corrosion rate.

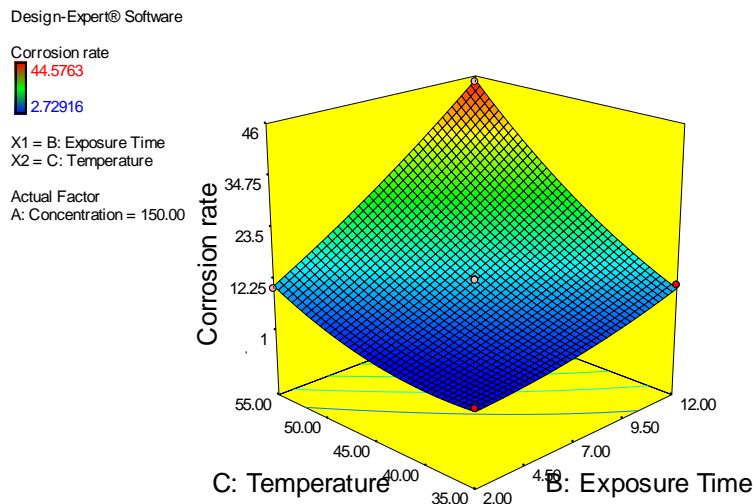


Fig. 6: Surface Plot for the influence of exposure time, temperature, and interface with corrosion rate.

4. CONCLUSION

Statistical analysis and optimization of corrosion rate of pumpkin pod extract on carbon steel were carried out in this work. The effects of pumpkin pod extract concentration, exposure time, and temperature on the corrosion rate of carbon steel was determined using Box Behnken design of the Response Surface Methodology (RSM). It was confirmed that all the three process factors considered were all significant but exposure time and temperature showed positive synergy on the rate of corrosion. It was established in this study that pumpkin pod extract inhibit the rate of corrosion on carbon steel as evidenced in the reduced weight loss obtained from the analysis. The second order mathematical model is well fitted to the obtained experimental data. The optimum conditions

ascertained was, pumpkin pod extract concentration of 178.07 ppm, 2.26 hrs of exposure time and temperature of 35.28 °C. This condition was validated in three replicates in the laboratory and rate of corrosion of 2.75 mm/yr was achieved. This extract developed for carbon steel corrosion inhibition can be scaled up for industrial application.

5. ACKNOWLEDGEMENTS

The authors would like to thank, Mrs. Effi Evelyn, Head, Chemical Engineering Laboratory, Federal University of Petroleum Resources, Effurun, for providing the enabling environment for this work, Mr. Akpeji Honest, Technologist, Chemistry Department, Federal University of Petroleum Resources, Effurun for his technical assistance.

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