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OPTIMIZATION OF KATEMFE SEED EXTRACT AS A CORROSION INHIBITOR FOR MILD-STEEL IN 0.5M HCL

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ABSTRACT

Optimization of the inhibiting effect of the Katemfe Seed extract on the mild steel in 0.5M HCl was investigated using gravimetric method. Phytochemical analysis was done to determine the qualitative tests of the extract. The factor variables used for the Central Composite design were Temperature 30-60, Time: 1-5 h and Concentration: 0.2-1.0g/L respectively. The analysis showed that a linear effect of the process variables was highly significant. The optimum conditions predicted were Temp: 54.65^oC, Time: 2.54 h and Concentration: 0.33g/l; and the optimum corrosion rate condition was predicted 1.0($\frac{mg}{cm^2h}$). The experimental value of 1.5($\frac{mg}{cm^2h}$) agreed closely with that obtained from the regression model. This study revealed that Katemfe seed extract is a good inhibitor for the corrosion of Mild steel in 0.5M HCl and the performance can be improved by moderating the process variables.

Keywords: Corrosion, Corrosion rate, Inhibition, Optimization, Weight loss,

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1. INTRODUCTION

Corrosion is the deterioration or destruction of metals and alloys in the presence of an environment by chemical or electrochemical means. It has been a major challenge that had attracted much investigations and researches. Mild steel is used for constructions because it has low cost when compared with steels. However according to Camila and Alexandra, 2013; the most effective method for prolonging the lifespan of industrial parts made from alloys and metals is the use of inhibitors. Furthermore, inhibitors are generally toxic and environmentally destructive hence there is need for environmentally friendly, cost effective and sustainable replacements [1,2]. Currently, a large amount of research has focused on organic extracts that can replace synthetic compounds. Green corrosion inhibitors such as extracts from many plants had been investigated and reported by several authors as being biodegradable and not containing heavy metals or other toxic compounds. Plant products are organic in nature, and contain certain photochemical including tannins, flavonoids, saponins, organic and amino acids, alkaloids, and pigments which could be extracted by simple less expensive procedures. Extracts from different parts of plant have been widely reported as effective and good metal corrosion inhibitors in various corrosive environments. A lot of research had been done on the use of plant sources / extracts as effective inhibitors. Groundnut leaves [3]. Jatroha [4]; Cashew stem [5] Vernonia amygdalina extract [6] Banana peels [7]; Potato peels [8]. The aim of this research is to determine the optimal process levels for katemfe seed extract determining the corrosion rate on katemfe as a corrosion inhibitor on mild steel in 1M HCl solution

2. MATERIALS AND METHODS

2.1. Material Preparation

Mild steel coupons were collected from Mechanical Engineering Department, Landmark Universtity, Omu-Aran, Kwara State, Nigeria. Each coupon was cut into 2cm x 2 cm with 2cm diameter. A hole of 0.1mm was drilled inside the coupons. The coupons were degreased, dried and then stored in a moisture free dessicator before use. Katemfe seed extract was collected from Ekiti State, Nigeria.

2.2. Preparation of Plant Extract

Katemfe seed extract powder of 320g was completely soaked in ethanol solution for 24 hours with moderate stirring, and then filtered to obtain a high yield concentration of the filtrate. The filtrate was subjected to evaporation to obtain the pure extract and was stored in a dessicator. The fine powder obtained was refluxed with HCl at 90 ℃ for 3 hrs in the water bath. Thereafter, the beaker was removed from the water bath and left to cool for 2hrs. After cooling, the reflux of different acidic media was filtered out to get the filtrate by passing it through filter paper and the pure extract from the acidic media was collected and stored. Acetone was used to dry and preserve the extract in a desiccator to prevent it from reacting with the environment.

2.3. Phytochemical Analysis of the Extract

Phytochemical analysis was done on the Katemfe Seed Extract (KSE) to determine the presence of active compounds such as saponins, alkaloids, tannins and flavonoid which made an extract as a reactive inhibitor

2.4. 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 inhibition on mild steel using KSE. 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 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 [9].

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.

Table 1. Experimental range of the independent variables, with factor levels for the inhibition of Katemfe Seed Extract on Mild-Steel in HCl solution

Independent variable	Symbols	Range and levels

		-1	0	+1
Time of exposure h	X_1	1	3	5
Temperature of, The solution °C	X_2	30	45	60
Inhibitor concentration in the extract	X_3	0.2	0.6	1.0

Table 2. Central Composite Design factors and levels.

Std	Run	Block	Factor 1 A:Time(h)	Factor 2 B:Temp(°C)	Factor 3 C:Conc(g/l)
11	1	Block 1	5.00	45.00	0.60
9	2	Block 1	3.00	45.00	0.60
19	3	Block 1	1.00	45.00	0.60
13	4	Block 1	5.00	60.00	0.60
3	5	Block 1	3.00	60.00	0.20
6	6	Block 1	5.00	60.00	0.60
5	7	Block 1	5.00	60.00	1.00
12	8	Block 1	3.00	30.00	0.20
4	9	Block 1	1.00	45.00	0.60
15	10	Block 1	3.00	60.00	0.20
17	11	Block 1	5.00	45.00	0.20
10	12	Block 1	3.00	30.00	0.20
18	13	Block 1	3.00	45.00	0.60
20	14	Block 1	3.00	30.00	1.00
2	15	Block 1	1.00	30.00	1.00
14	16	Block 1	3.00	30.00	0.60
7	17	Block 1	5.00	45.00	0.60
16	18	Block 1	3.00	60.00	0.60
1	19	Block 1	3.00	45.00	1.00
8	20	Block 1	3.00	30.00	1.00

2.5. Gravimetric (Weight Loss) measurement

The Mild steel specimens were immersed in 100 ml test solutions. Weight loss measurements were conducted under total immersion using 250 ml capacity beakers containing various concentration of test solution at temperature range of 30-60 °C. The mild steel coupons were weighed and suspended in the beaker. Weight loss Experiment was carried out by weighing the specimens before and after immersion in 0.5M HCl in the presence and absence of inhibitor. At the appropriate time, the coupons were taken out, immersed in acetone, scrubbed with a bristle brush under running water, dried and reweighed. The weight loss was calculated as the difference between the initial weight and the weight after the removal from the corrosion medium. The experimental readings were recorded, however the weight loss (Δw) and, corrosion rate (CR) were determined using the equation given below;

$$\Delta Wu = (\Delta Wu - \Delta Wi) \quad (1)$$

$$C.R. = \frac{\Delta W}{A \times T} = \frac{W_1 - W_2}{A \times T} \qquad (g/cm^2 h) \qquad (2)$$

Where, W_1= Weight of Mild steel coupon before immersion.

W_2= Weight loss of Mild steel coupon after immersion.

$CR1$= Corrosion rate of the Mild steel coupons in the absence of inhibitors.

$CR2$= Corrosion rate of the Mild steel coupons in the presence of inhibitors.

A= Surface area of the coupon in cm^2

ΔW= Change in weight

3. RESULTS AND DISCUSSION

3.1. Result on phytochemical analysis (Qualitative Test)

The presence of each active compound showed that Katemfe Seed Extract is a good inhibitor. The phytochemical components result of KSE analysis is as shown in Table 3.

Table 3. Phytochemical Component result of KSE

Test	Reagent	Color Change	Confirmation
Alkaloids	Wager	Brown Precipitate (PPT)	+++ positive
Tannin	FeCl3	Greenish PPT	++ positive
Flavonoid	NaOH+AlCL3+ H2SO4	Yellow PPT	++ positive
Saponins	Distilled Water	Formed a persistent foam	+++positive
Steroid	Acetic anhydride& 2ml Conc .H2SO4	Reddish brown	++ positive

+++ =Highly present;

++moderately present

3.2. Results on Weight Loss Measurement

The data obtained from the weight loss measurements of the Mild steel coupons showed that the rate of corrosion decreases with increase in the concentration of inhibitors as shown in Table 2. Furthermore, as the concentration of inhibitor increases, corrosion rates reduce as shown in Table 4.

Table 4. Central-Composite Design factor levels of independent variables with Response

Run	Factor1; X_1: Time (h)	Factor 2; X_2: Temp (°C)	Factor 3; X_3:Inhibitor concen. (g/l)	Response 1:weight loss (g)	Response 2: corrosion rate $(\frac{mg}{cm^2 h})$	
1	5.00	45.00	0.60	0.07	3.5	
2	3.00	45.00	0.60	0.03	2.5	
3	1.00	45.00	0.60	0.01	2.5	
4	5.00	60.00	0.60	0.06	3	
5	3.00	60.00	0.20	0.07	5.8	
6	5.00	60.00	0.60	0.12	6	

7	5.00	60.00	1.00	0.08	4
8	3.00	30.00	0.20	0.01	0.83
9	1.00	45.00	0.60	0.03	7.5
10	3.00	60.00	0.20	0.07	5.8
11	5.00	45.00	0.20	0.02	1
12	3.00	30.00	0.20	0.01	0.83
13	3.00	45.00	0.60	0.03	2.5
14	3.00	30.00	1.00	0.01	0.83
15	1.00	60.00	1.00	0.03	7.5
16	3.00	30.00	0.60	0.02	1.7
17	5.00	45.00	0.60	0.04	2
18	3.00	30.00	0.60	0.07	1.7
19	3.00	45.00	1.00	0.02	1.7
20	3.00	30.00	0.20	0.01	0.83

3.3. 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:

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4{X_1}^2 + B_5{X_2}^2 + B_6{X_3}^2 + B_7X_1X_2 + B_8X_1X_3 + B_9X_2X_3 \quad (3)$$

Was fitted between the response (Corrosion rate (Y)) and the process variables: time (A), temperature (B), and inhibitor concentration (C). The final response equation for in terms of coded is as given

$$Y = +0.037 + 0.010A + 0.029B + 1.899C \quad (4)$$

From Table 5, 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 analysis of variance indicated that the linear 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

Established. Furthermore, as recorded by [10], the increase in temperature with reduction in time made the concentration to be more effective because the availability of the presence of the phytochemicals (Alkaloids, Tannin, Flavonoid, Saponin and Steroid) as presented in Table 3; means more of the heteroatoms were adsorbed on the surface of the mild steel an then decrease the corrosion rate. The rate of corrosion with reference to low temperature confirmed result obtained by [11].

Table 5: Analysis of Variance (ANOVA)

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	0.013	3	4.296	13.20	0.0001	significant
A	*7.393*	*1*	*7.393*	*2.27*	*0.1513*	
B	*8.274*	*1*	*8.274*	*25.42*	*0.0001*	
C	*1.392*	*1*	*1.392*	*0.43*	*0.5224*	
Residual	5.207	16	3.255			
Lack of Fit	*2.757*	*9*	*3.064*	*0.88*	*0.5838*	*not significant*
Pure Error	*2.450*	*7*	*3.500*			
Cor Total	0.018	19				

R-Squared	0.7122
Adj R-Squared	0.6583
Pred R-Squared	0.5489

Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case B are significant model terms. The "Pred R-Squared" of 0.5489 is in reasonable agreement with the "Adj R-Squared" of 0.6583."Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 9.681 indicated an adequate signal. This model can be used to navigate the design space.

3.4. Surface Response Plots for Mild Steel with Katemfe Seed Extract

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 1 – Fig 3. It showed in Fig 1 showed that corrosion rate increased with increase in temperature and also increased with increase in time;

However; Fig 2 showed that corrosion rate decreased with increase in inhibitor concentration and also increases with an increase in inhibitor concentration and also increases with an increase in time as shown in Fig 3; but decreases with an increase in inhibitor concentration .This can be attributed to adsorption. Fig 4 showed the Plot of the predicted versus experimental.it showed that the Plots were reasonably distributed near to the straight line showing the underlying assumptions of the analysis were appropriate while Fig 5 showed the Plot of the normal versus Residuals.

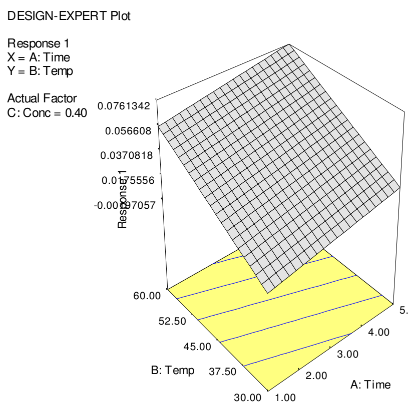

Figure 1. Effect of Time and Temperature on corrosion r ate of KSE on Mild Steel

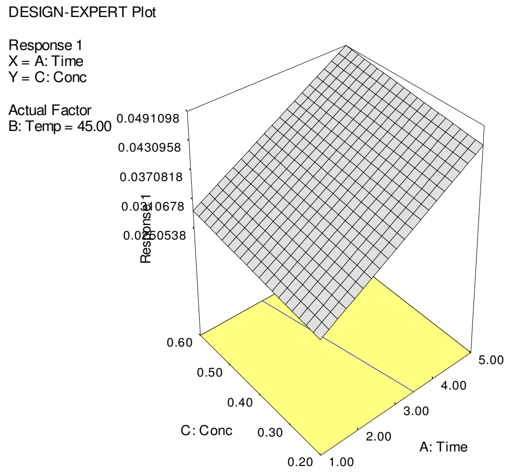

Figure 2. Effect of Time and Inhibitor on Corrosion rate of KSE on Mild steel

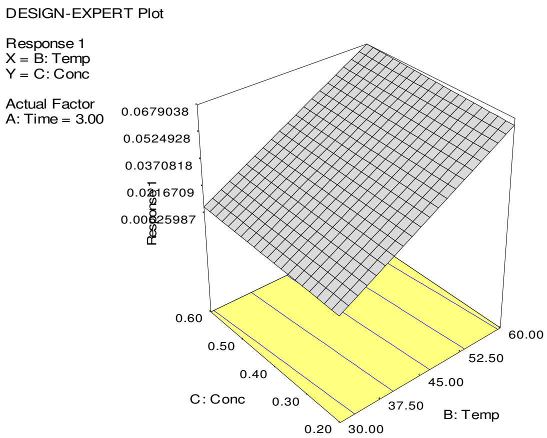

Figure 3. Effect of Temperature and Inhibitor concentration on corrosion rate of KSE on Mild Steel

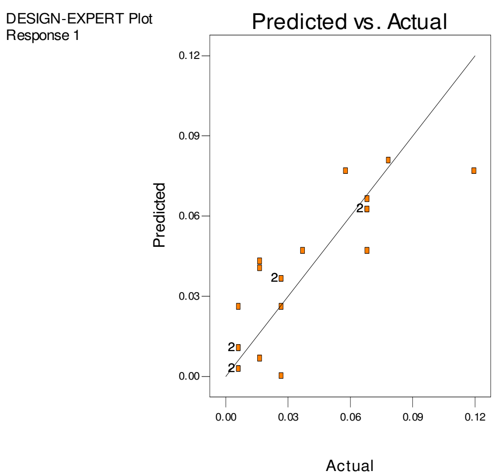

Figure 4. Plot of predicted versus Actual **Figure 5**. Normal versus Residuals

3.5. Experimental Validation

The optimum conditions predicted from the software with: Temp: 54.65 Deg, Time: 2.54 h and Conc: 0.33g/l; and the optimum corrosion rate condition was predicted to be $1.0(\frac{mg}{cm^2h})$Experiment was carried out at this optimum conditions to validate the predicted optimum values. The experimental value of $1.5(\frac{mg}{cm^2h})$ agreed closely with that obtained from the regression model.

4. CONCLUSION

The central Composite Design and the response surface methodology enabled the determination of the optimal operating conditions for corrosion rate percentage. The validity of the model was proven by fitting the values of the variables of the model equation and by carrying out experiments using these values. The optimum conditions predicted were Temp: 54.65^{o}C, Time: 2.54 h and Concentration: 0.33g/l; and the optimum corrosion rate condition was predicted $1.0(\frac{mg}{cm^2h})$. The experimental value of $1.5(\frac{mg}{cm^2h})$ agreed closely with that obtained from the regression model.

REFERENCES

[1] Loto et al. Int. J. Electrochem. Sci.2013, 8, 11087 – 11100.

[2] Wiersema, John Harry; León, Blanca. World Economic Plants: A Standard Reference. CRC Press. 1999, 661. ISBN 0-8493-2119-0

[3] Olawale, O. et al. Evaluation of Ground nut leaves extract as corrosion inhibitor on mild steel in 1M Sulphuric acid using Response Surface Methodology (RSM) 2018,9,11, 829-841.

[4] Olawale et al. Corrosion Inhibition of Mild Steel in Seawater using Jatropha Stem.Analele Universitatii "Eftimie Murgu" Resita. 2016, 23, 1, 228-238.

[5] Olawale, O et al.(2015) A Study on Corrosion Inhibitor of Mild-Steel in Hydrochloric Acid using Cashew Waste. Inter. J. Moder. Eng. Res.2015, 5, 8, 25-30.ISSN:2249-6645

[6] Olawale et al. Inhibitory action of Vernonia amygdalina extract (VAE) on the corrosion of carbon steel in acidic medium.2017, J. Electrochem. Sci. Eng. l: 7, 3 145-152.

[7] Sangeetha,et al. Eco friendly extract of banana peel as corrosion inhibitor for carbon steel in sea water.J.Nat.Prod. Plant Resour 2012, 2,601-610.

[8] Ibrahim et al. Corrosion inhibition of mild steel using potato peel extract in 2M HCl solution .Int.J. Elctrochem.Sci. 2011, 6, 6542-6556

[9] Raymond et al.

[10] Khaled, K.F Molecular simulation, quantum chemical calculations and electrochemical studies for the inhibition of mild steel by tirazoles.Electochim.Acta. 2009, 53, 3484-3492.

[11] James; A.O. and O. Akaranta.O. Studies on the corrosion inhibition of acetone extract of red onion skin for mild steel in hydrochloric and sulphuric aicd solutions.Sceintal Africana.2009, 8,1, 23-34