Optimization and modelling of Aluminium corrosion inhibition using Almond (Prunus amydgdalus) fruit leaves extract as green inhibitor in HCl acidic medium

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OPTIMISATION AND MODELLING OF ALUMINIUM CORROSION INHIBITION USING ALMOND (PRUNUS AMYDGDALUS) FRUIT LEAVES EXTRACT AS GREEN INHIBITOR IN HCL ACIDIC MEDIUM

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ABSTRACT

The addition of inhibitors is a standard practice in industries to control corrosion. However, no research had been done on the use of Almond fruits extract as a corrosion inhibitor.This study investigated the optimisation of Almond leaves extract as corrosion inhibitor on Aluminum in 1M HCl. The effect of temperature (30-50^oC), inhibitor concentration (0.2 g/l - 0.6 g/l) and time (2-6 h) on inhibtion efficiency was investigated using Response Surface Methodology (RSM). The results revealed that Almond leaves extract effectively inhibits the corrosion rate of Aluminum immersed in the acidc medium. The concentration of 0.38 g/l, temperature of 36 ^oC and time 5 h gave the optimum conditions to validate the predicted values. The experimental value of 97.9% agreed closely with that obtained from the predicted model. This study revealed that Almond fruit extract could be recommedned as a low cost and a good inhibitor for acid corrosion of Aluminum.

Keywords: Almond fruit leaves, Aluminum, Inhibitor, Corrosion,Optimisation, Weight loss.

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1. INTRODUCTION

Corrosion of metals is a major industrial problem that has attracted much investigation and research. It is the deterioration of a material by its environment (Chigoziri et al., 2015) [1]. Aluminum is a metal that corrodes as a result of electrochemical reaction to environment. It has been reported that passive oxide film formed on aluminum surface is responsible for its resistance to corrosion. However, the surface film is atmospheric and dissolves substantially when the metal is exposed to high concentrations of acids or bases. Aluminum is used extensively in industry as well as for domestic applications. Furthermore, it is used as reaction vessels, pipes, machinery and chemical batteries because of its advantage which are its low, lightness and good corrosion resistance at moderate temperatures (El-Maghraby, 2009; Omotioma and Onukwuli, 2017)[2-3]. It is important to state that most metals corrode on contact with water (moisture in the air), acids, bases, salts, oils, aggressive metal polishes and other solid and liquid chemicals (Aggarwal, 2010). Aluminum dissolves readily in hydrochloric acid to form aluminum chloride and hydrogen. The reaction is more vigorous as the acid becomes hotter and more concentrated as shown in Eq. (1).

$$2Al + 6HCl \rightarrow 2AlCl_3 + 3H_2 \quad (1)$$

Aluminum structures corrode as a result of electrochemical reaction to environment. Unueroh et al. (2010) reported that reported that corrosion is one of the major challenges facing oil and gas industries. Corrosion inhibitors are chemicals that react with a metallic surface, or the environment the surface is exposed to; which result to giving the surface a certain level of protection. Corrosion inhibitors normally have the ability to control the corrosion through forming different kinds of films such as adsorption via formation of precipitates or through forming of inactive layer on the surface of the metal (Obayes et al., 2017) [6]. One of the most practical methods of protection against corrosion especially in acidic media is inhibitor (Trabanelli, 1991)[7]. According to Hughes et al. (2016) and Odewunmi et al. (2015), inhibitors slow corrosion processes by either increasing the anodic or cathodic polarization behavior; reducing the movement or diffusion of ions to the metallic surface, or cause an increase in the electrical resistance of the metallic surface. The environmental toxicity of some inhibitors has led researchers to investigate the use of green and agro-based materials as the alternatives to inorganic substances. Based on the environmental regulations that require toxic chemicals to be replaced with the recently used term "Green chemicals", this has increased research in this area. These organic materials are non-toxic, biodegradable, cheaper and eco-friendly, hence they are considered more important and attractive. According to reports by Al-Turkustani et al. (2012); Benali et al. (2013); Buchweishaija and Mhinzi (2008); Pehlivan et al. (2012); Singh et al. (2013); Gadow et al. (2017); Raja and Sethuraman (2008); Zarrok et al. (2013); Ngobiri et al., 2015; Umoren et al. (2015); Chakravarthy and Mohana (2014), extracts from agro-based sources, such as tannins, amino acids, alkaloids, and pigments; were investigated and indicated that they can be utilized as inhibitors. Other plant extracts are Jatropha curcas (Olawale et al., 2015); Rice husk (Adediran et al., 2017) [9]; Barley grass extract (Ammar et al., 2017)[10]; Water hyacinth (Singh et al., 2011)[11]; Moringa oleifera (Kairi and Kassim, 2013) [12]; Vernonia amygdalima extract (Loto et al., 2013) [13]; Cashew waste (Olawale et al., 2017) [14].

Almond (Terminalia catappa), a large tree grows mainly in the tropical regions of Africa, Asia and Australia. It is a tree that provides shade because of its large leaves. It has edible but slightly acidic fruit. The leaves contain several flavonoids, tannins, saponines and phytosterols. Due to this chemical richness, herbal medicines for various purposes can be obtained from its leaves. In line with this chemical richness, it is important to investigate the corrosion inhibition efficiency from the extract from its leaves. Therefore, this present study focuses on the Response Surface Optimization of the inhibitor efficiency of Almond Fruit Leaves Extract (AFLE) as an inhibitor for aluminum corrosion in HCl solutions. The effects of the process parameters namely; the extract concentration, temperature, time and acid concentration on the corrosion process were investigated using a Box Behnken design.

Materials and Methods

The interaction between several illustrative variables and one or more response variables can be investigated with the utilisation of a statistical tool called Response Surface Methodology (RSM). For sufficient and reliable measurement of response, RSM process involves the designing of series of experiments and developing a mathematical model of the second order response surface with the best fittings. In analysing the data, the Software Design Expert (6.0.8) was used. The mathematical empirical model is defined as:

$$Y = B_0 + B_1X_1 + B_2X_2 + B_{11}X_1^1 + B_{22}X_2^2 + B_{12}X_1X_2 \quad (2)$$

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. Raymond et al. (2016) discussed the theory and application of RSM.

Experimental Design

A box Benhken design of 17 experimental runs included 3-operating variables was established for the experiments used for studying the effects of temperature, time and inhibition concentration on corrosion parameters was performed on the AFLE. 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). The experiments were performed in random order to avoid systematic error. In the study, the design of experiment using Box Behnken to obtain the points where the experimental run was performed. Thereafter, experimental observation of the corrosion inhibition effects of the various factors at the design points. A mathematical model was obtained which expresses the relationship between the process factors and the percentage inhibition efficiency. The prediction of the optimum values of the process for the maximum inhibition efficiency using RSM was done and by experimentation, verification was carried out.

Table1. Experimental range of the independent variables, with factor levels for the inhibition of Almond leaves extract on Aluminium in HCl solution

		Range and levels		
Independent variable	**Symbols**	-1	0	1
Time	X_1	2	4	6
Temperature	X_2	30	40	50
Inhibition concentration	X_3	0.2	0.4	0.6

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

Table 2: Design matrix for the corrosion inhibition of Aluminum in HCl by the AFLE

	Coded variables			Actual Factors		
Run	**a**	**b**	**c**	**Factor 1; a: time (days)**	**Factor 2; b: temperature (Deg. C)**	**Factor 3; c: inhibitor concentration (g/l)**
1	0	0	0	4	30	0.6
2	-1	1	1	6	50	0.4
3	0	0	0	6	40	0.2
4	1	-1	1	2	40	0.6
5	0	1	0	4	40	0.4
6	0	-1	0	4	50	0.2
7	1	1	1	6	40	0.6
8	0	0	1	4	40	0.4
9	-1	-1	1	4	40	0.4
10	0	0	0	4	30	0.4
11	1	0	0	6	40	0.2
12	-1	-1	-1	2	30	0.2
13	0	0	0	4	50	0.4
14	0	0	0	2	30	0.4
15	0	0	0	2	50	0.6
16	-1	1	-1	4	40	0.4
17	1	-1	-1	4	40	0.4

*a = X_1 (Time (days)), b = X_2 (Temperature (^oC), c = X_3 (Inhibitor concentration (g/l))

Specimen preparation

An analytical grade of 1.0 M HCl was used in this study. AFLE was collected from Omu-Aran, Kwara State, Nigeria (**Latitude:** 8° 08' 18.85" N, **Longitude:** 5° 06' 09.36" E). Sheet of Aluminum was obtained within Omu-Aran and was cut into 17 coupons ($2\ cm \times 3\ cm$). Its chemical composition was determined using an Optical Emission Spectrometer and the results are presented shown in Table 3

Table 3 Percentage chemical composition of the Aluminum sheet

Fe	Si	Mn	Zn	Cr	Mg	V	Ti	Cu	Al
0.02	0.255	0.04	0.05	0.02	0.03	0.04	0.02	0.03	99.5

The samples were cleaned using emery paper to expose the shiny surface, degreased with acetone to remove oil impurities, washed with distilled water and then dried in the air and later stored in desiccators.

Preparation and extraction from the Almond leaf extract

The solvent used for the extraction of AFLE was ethanol. The AFLE leaf samples were washed and dried then grinded into fine powder. 20 g of leaf extract were extracted with Soxhlet extractor for 6 hours using 800 mg/L of ethanol. After extraction, the samples were allowed to cool for 6 hours and were filtered. The filtrates obtained were used to prepare inhibitor concentration ranges between 0.2-0.6 g/l solutions using the box Behnken design. The Coupons were weighed before and after immersion in the inhibitor.

Weight loss (gravimetric) measurement method

Weight loss measurements were conducted under total immersion using 250 ml capacity beakers containing solution to temperature ranges of 30-50^{o}C in a thermostatic water bath. The Aluminum coupons were weighed and dropped in the inhibitor. The coupons were retrieved with time variables of 2-6 days, exact temperature and the inhibitor concentrations (0.2 – 0.6 g/l) as shown in Table 2. After each exposure time, the aluminum coupons were removed and washed so as to remove the corrosion product with the emery paper, rinsed with distilled water and then dried in acetone. The samples were re-weighed to determine the weight loss (g) by the difference of aluminum weight before and after immersion. This can be obtained using Eq. (3).

$$\Delta W = W_b - W_a \qquad (3)$$

where W_b is the weight before immersion; W_a is the weight after immersion.

The inhibition efficiency was calculated using Eq. (4):

$$IE\% = \frac{W_b - W_a}{W_b} \times 100 \qquad (4)$$

Surface Characterization

Scanning Electron Microscope (SEM) was used to determine the surface morphology of the aluminum coupons of the (i) blank, the coupon; (ii) the highest inhibition efficiency (iii) optimized coupon, respectively.

2. RESULTS AND DISCUSSION

RSM Analysis

The RSM analysis of aluminum corrosion in 1M HCl is displayed in Table 4. Box Behnken gave 17 experimental runs as predicted from the software used.

Table 4 RSM result of corrosion inhibition of Al in HCl by Almond fruit extract

Block	Temp (^{o}C)	Conc (g/l)	Time(h)	Wt loss	Inh.Eff(%)
Block 1	30	0.6	4	0.01	96.6
Block 1	50	0.4	6	0.04	90.11
Block 1	40	0.2	6	0.07	81.05
Block 1	40	0.6	2	0.03	77.5
Block 1	40	0.4	4	0.02	93.3
Block 1	50	0.2	4	0.06	85.36
Block 1	40	0.6	6	0.02	93.8
Block 1	40	0.4	4	0.02	94.3
Block 1	40	0.4	4	0.02	94.5
Block 1	30	0.4	4	0.01	99
Block 1	40	0.2	6	0.04	10.2
Block 1	30	0.2	2	0.04	89.2
Block 1	50	0.4	4	0.01	2.7
Block 1	30	0.4	2	0.01	96.3
Block 1	50	0.6	2	0.02	5.6
Block 1	40	0.4	4	0.03	90.9
Block 1	40	0.4	4	0.02	94.5

From Table 5, the ANOVA results showed that the quadratic models were suitable to analyze the experimental data. A statistical significant regression model was developed and was also evaluated based on p-values ($p < 0.05$). The coefficient terms with p-values more than 0.05 are insignificant and are removed from the linear, quadratic and interaction effects of temperature and time are significant model terms.

Table 5: ANOVA for analysis of % inhibition efficiency and their significant tests and effects of AFLE on Aluminum

Source	Coefficent Estimate	DF	Mean of Square	F-Value	P-Value (Prob>F)
Model	4.092	9	4.547	3.91	0.0429 Significant
A	4.324	1	4.324	3.72	0.0951
B	4.799	1	4.799	4.13	0.0817
C	6.602	1	6.602	0.057	0.8184
A^2	1.613	1	1.613.	1.39	0.2773
B^2	3.116	1	3.116	2.68	0.1456
C^2	3.392	1	3.392	0.029	0.8692
AB	8.68	1	8.68	7.469	0.979
AC	7.466	1	7.466	0.64	0.4492
BC	4.896	1	4.896	0.42	0.537
Residual	8.13	7	1.162		
Cor.Total	4.906	16			

The model reduces to Eq. (5) after eliminating the insignificant coefficients.

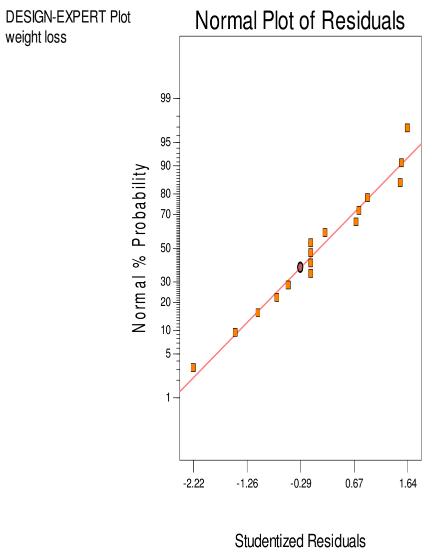
$$Y = 0.037346 + 4.22892\,T - 0.39815\,IC - 0.014083t - 7.32869T^2 + 0.49398IC^2 - 7.35128T^2 - 1.61898T \times IC + 6.51167T \times t - 0.013441IC \times t \quad (5)$$

where T is temperature, IC inhibitor concentration and t is time.

Figure 1 shows the plots of normal versus studentized residual which showed a linear relationship. Figure 2 depicts the plot of studentized residual versus predicted value. The analysis of the Almond fruit extract (inhibitor) is presented in Figure 3(a-b). Moreover, the graph (3-D surface plot) which showed the relationship between factors and response of the designed experiment are as shown in Figures 3-5. It showed that inhibition efficiency increased with increase in concentration but decreased with increase in temperature.

Validation of the predicted levels

The optimum condition inhibition efficiency of 96.6% was observed. The predicted levels from the software are: Temperature: 36°C; Time: 5 h and Inhibition concentration: 0.38 g/l. Experiment was carried out at these out optimum conditions to validate the predicted values. The experimental value of 97.9% of inhibition efficiency strongly agreed closely with that obtained from the regression model.



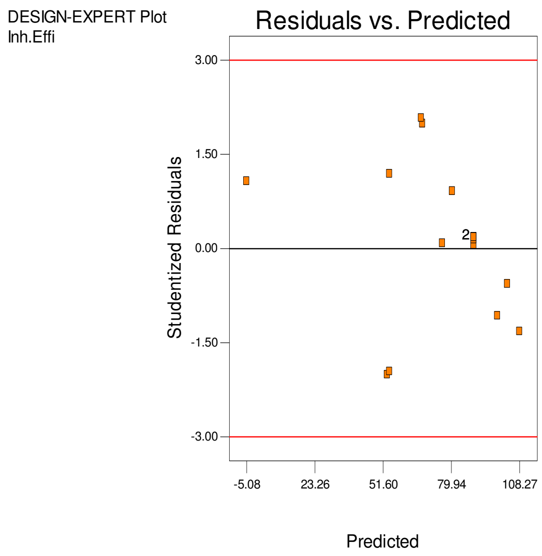
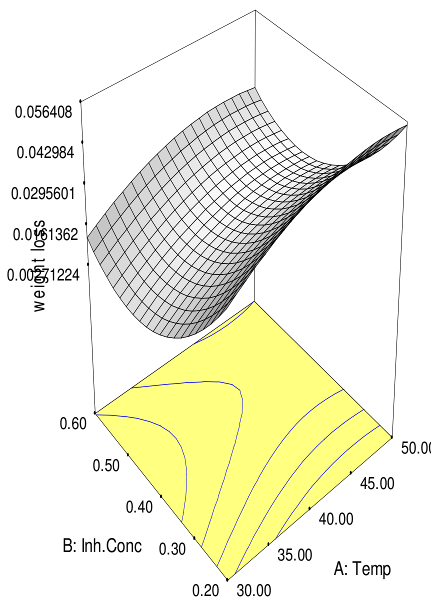


Fig. 1: Normal versus Studentized residuals **Fig. 2.** Studentized versus Predicted



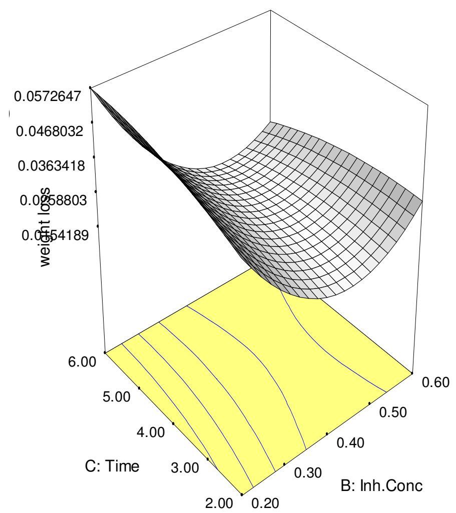


Fig. 3: 3-D Plot of Inh. Conc. against Temp. **Fig. 4: 3-**D Plot of Inh. Conc. against Time

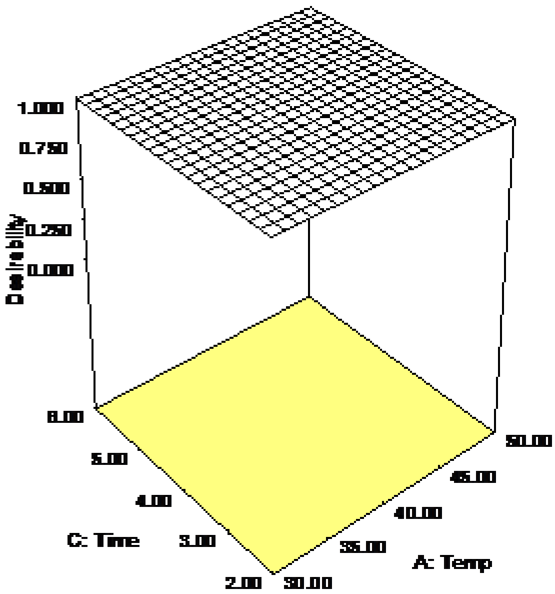


Fig. 5: 3-D plot of Time against Temperature

SEM Analysis

The SEM images and EDX of blank, maximum inhibition and the optimal are as presented in Figs. 6a (i) and (ii) respectively. A significant damage was observed on the aluminum which was immersed in 1M HCl solution indicated that the surface was highly corroded. The surface of the corroded area of the aluminum in Figs. 6b (i) and (ii) was protected and the corrosion reduced based on the appropriate inhibitor concentration as it is evident from the morphology where a protective film was formed. Fig. 6c (i) showed the presence of the inhibitor at the optimum concentration in which the elemental composition is as shown in Fig. 6c (ii). Furthermore, the surface was remarkably protected because of the formation of protective layer of the adsorbed extract molecules of the aluminum surface by almond fruit extract which indicated more restricted corrosion unlike Fig. 6b (i). These are in good agreement with the other method like electrochemical measurements.

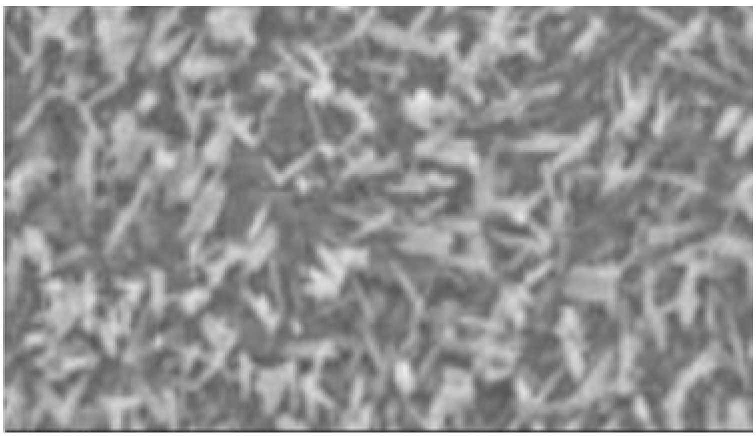


Fig. 6a (i): SEM of Blank Aluminum

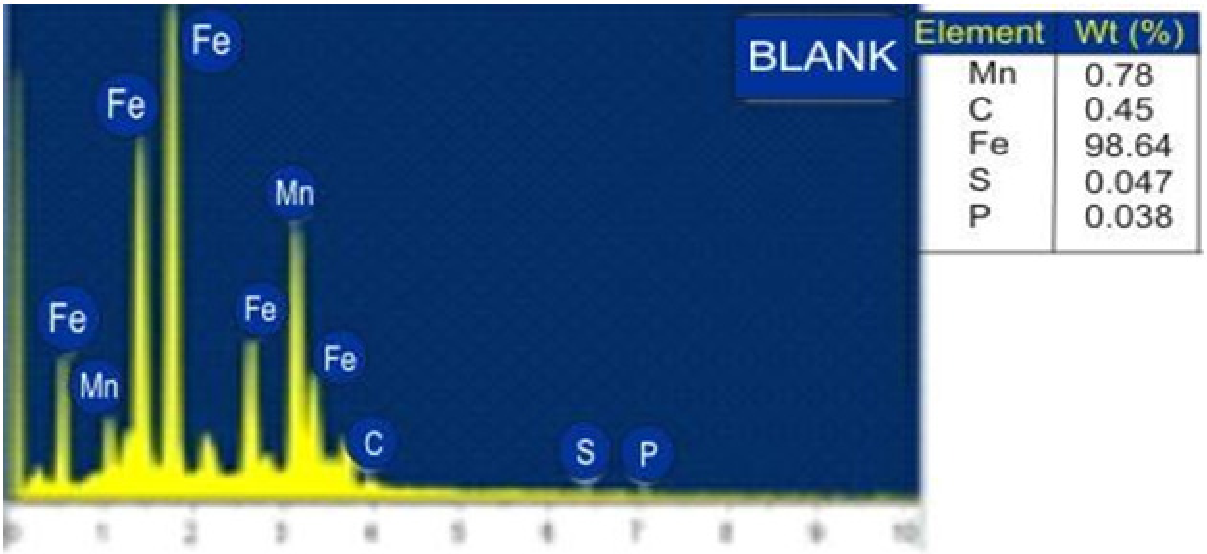


Fig. 6a (ii): EDX of Blank Aluminum

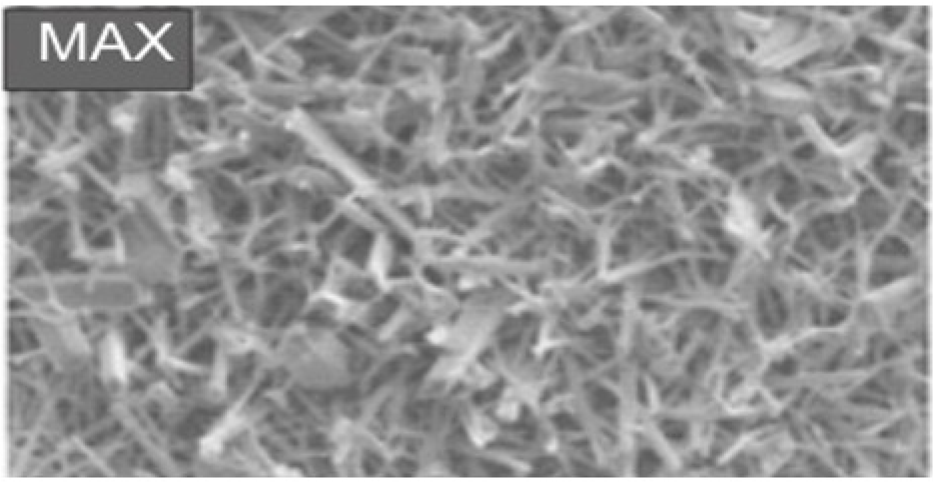


Fig. 6b (i): SEM of maximum inhibitor

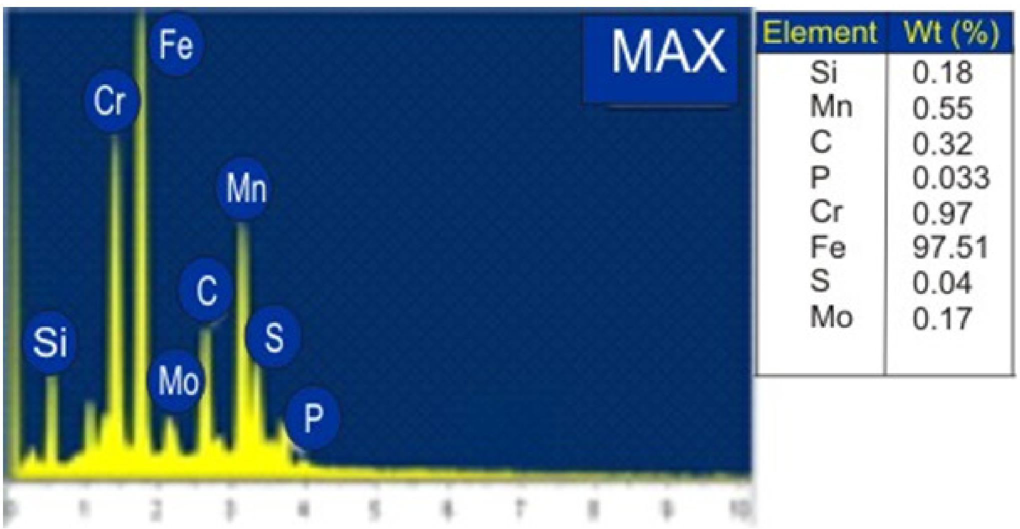


Fig. 6b (ii): EDX of maximum inhibitor

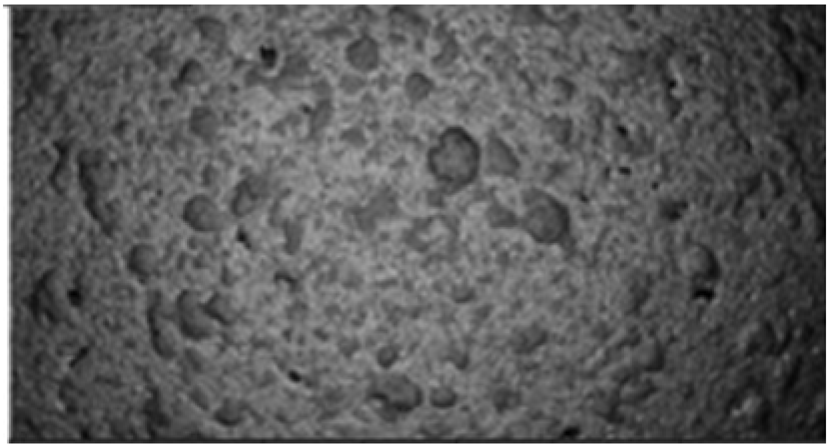


Fig. 6c (i): SEM at Optimal level

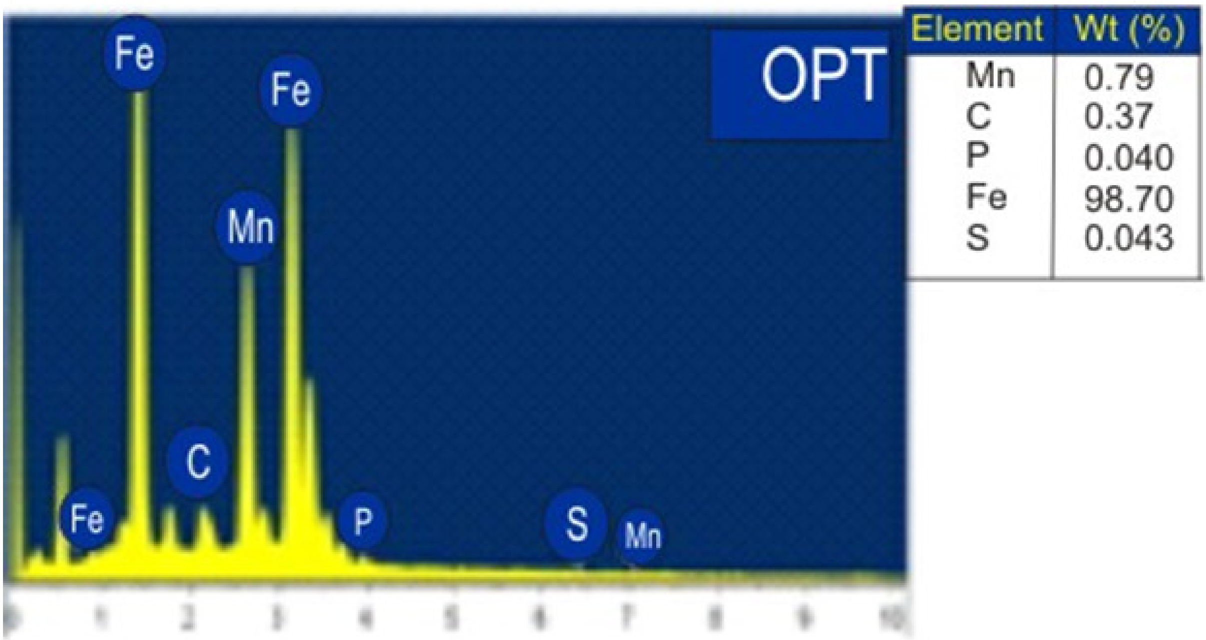


Fig. 6c (ii): EDX at Optimal level

3. CONCLUSION

BoxBehnken design and RSM enabled the determination of optimal operating conditions of percentage inhibition efficiency. The validity of the model was proved by fitting the values predicted. Experiments carried out at these optimum conditions validated the predicted optimum values. The experimental value of 97.9% agreed closely with that obtained from the regression model. The result further showed that increase in concentration increases the inhibitor efficiency. SEM images also supported the formation of film on the aluminum surface. ALFE can be recommended as an eco-friendly corrosion inhibitor on Aluminum.

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