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APPLICATION OF RESPONSE SURFACE METHODOLOGY USING CENTRAL COMPOSITE DESIGN FOR THE EVALUATION OF CORROSION INHIBITION PERFORMANCE OF *Pilliostigma thoningii* ON MILD STEEL IN ACIDIC MEDIA

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ABSTRACT
Response Surface Methodology (RSM) based on Central Composite Design (CCD) was employed to examine the corrosion inhibition efficiency of pilliostigma thoningii stem bark extract on mild steel in hydrochloric acid solution using gravimetric method. The interactive effects of the inhibitor concentration, temperature and time were optimized for maximum response. The optimum inhibition efficiency of 93.75% at 1.0g/L of inhibitor concentration, temperature of 333K and immersion time of 1h was accomplished. The effectiveness of the inhibitor was also supported using scanning electron microscopy (SEM). Analysis of the results revealed that the adsorption of pilliostigma thoningii extract on the surface of the mild steel was spontaneous and occurred according to physisorption mechanism. The data obtained were fitted into various adsorption isotherms and the mechanism of the interaction was found to conform best to the Langmuir isotherm.
Keywords: Optimization; Mild steel; Corrosion; Pilliostigma thoningii; Response Surface Methodology

INTRODUCTION

Mild steel is a useful metal in industries. But its usefulness is often threatened by corrosion phenomena. Corrosion is simply explained as the deterioration of metal, alloy or other properties due to adverse reaction with the environment (Umoren *et al.,* 2019). These processes are common in oil and gas refineries and most of the petrochemical plant failures are caused by corrosion, and this promotes economic, environmental and human life losses (Verma, 2006). Hence the prevention of metal corrosion is of paramount importance.

The use of organic compounds as corrosion inhibitors is one of the most practical methods of protecting metals against corrosion and it is increasingly becoming popular (Abeng *et al.,* 2017). The organic inhibitors act by adsorption and protection of the metal through the formation of film bearing heteroatoms with high electron density such as phosphorous, sulphur, nitrogen and oxygen or those containing multiple bond which are considered as the centre of adsorption that are effective as corrosion inhibitors (Döner *et al.,* 2011).

Inhibitors are chemical substances or combination of substances which when added in very low concentrations in a corrosive environment effectively prevents or reduces corrosion without any significant reaction with the components of the environment. The studies of plant extract as cost effective and non-harmful corrosion inhibitors have attracted huge interests of researchers (Geethamani *et al.,* 2019). Green corrosion inhibitors owing to their viability and absence of heavy and toxic substances in their molecules are gaining increasing interest in recent time. Plants constituents such as tannins, alkaloids, phenolics, amino acid are organic in nature which have been used as corrosion inhibitors. These inhibition effectiveness of plant extract is credited to the presence of complex organic compounds containing oxygen, nitrogen and sulphur heteroatoms.

The presence of aromatic rings, conjugated double bonds as well as triple bonds have also been identified to provide adsorption centres with which they interact with the active sites on the metal (Wei *et al.*, 2020). Plant extracts due to their potential benefits such as *picralima nitida* extract gave 86.78% efficiency for zinc in 1.0M HCl at inhibitor concentration of 1.2g/L(Ezeugo *et al.,* 2017), pawpaw leaves extract gave 80.29% efficiency for mild steel in HCl medium at inhibitor concentration of 1.0g/L (Omotioma and Onukwuli 2016),*epiphyllum oxypetalum* extract gave 82.93% efficiency for mild steel in 3M H_2SO_4solution at inhibitor concentration of 0.055g/L (Emembolu *et al.,* 2020), blended guava and fluted pumpkin extract gave an efficiency of 93.70% and 78.14% on mild steel in 0.5M HCl acid at an inhibitor concentration of 0.2g/L and 0.487g/L respectively(Nwachukwu *et al.,* 2021).

Response surface methodology (RSM) was adopted in this study through the usage of CCD as a statistical tool and was used to obtain the experimental schedules in which a response of interest is affected by different variables and the aim is to obtain the optimal response (output) which is also affected by different independent variables (input). A systematic investigation to obtain an insight onto the inhibition performance of ethanolic extract of *pilliostigma thoningii* stem bark on mild steel in an acidic environment was accomplished by establishing a mathematical model showing the existence of relationship between the various process factors like the inhibitor concentration, temperature and time and the response variable (Inhibition Efficiency) (Lavanya 2021).

MATERIALS AND METHODS

Preparation of the Plant Extract

The bark of *Piliostigma thoningii* were collected from Janguza town, Kano state, Nigeria. The sample was washed with distilled water and air-dried for 21 days and ground to powder. Consequently, 250g of the powder was soaked in 95 % ethanol (2L) for 72hours and then filtered. The filtrate was evaporated to afford crude ethanol extract.

Mild Steel Specimen

Mild steel with composition of 67.79% Fe was used for the investigation and was mechanically cut into coupons with the dimension (2cmx2cmx0.36cm). The coupons were cleaned followed by polishing with silicon carbide emery paper (120, 400, 800, and 1000grade) to expose shining polished surface. To remove oily and organic impurities, the coupons were degreased with acetone and finally washed with distilled water, dried in air and then stored in a desiccator. The weight of each coupon was taken using electronic weighing balance and the initial weight was recorded (Revie and Uhlig, 2011).

Inhibitor Preparation

0.2g of the ethanol crude extract was dissolved in $250cm^3$ of 1M HCl solution. The solution was then transferred into $1000cm^3$ volumetric flask and made up to the volume with 1M HCl solution. Similar other concentrations of the inhibitor 0.4, 0.7, 1.0, and 1.2g/L were then prepared by dissolving 0.4, 0.7, 1.0 and 1.2g of the inhibitor respectively. The 1M HCl prepared also functioned as the blank solution in all the experiment (Ajeigbe *et al.*, 2017)

Weight Loss Measurement

The pre-weighed metal coupons were drilled to create holes and then immersed with the aid of a sewing thread in 50ml test solution with and without an inhibitor varying the concentrations at different temperatures and time intervals. All the weight loss experiments were performed in unstirred aerated hydrochloric acid solutions containing various concentrations of inhibitors as shown in the experimental set up in Table 1. At the end of the immersion time, the mild steel specimens were removed from the test solution, carefully washed with distilled water, then with acetone and dried. The masses of the metal coupons were taken, recorded and the differences in mass was used to estimate the corrosion rates, surface coverage and the inhibition efficiency using the following equations 1 to 3 respectively (Ajeigbe *et al.*, 2017; Anadebe *et al.,* 2018).

$$R_C = \frac{\Delta w}{AT} \quad (1)$$

$$IE_{WL}\% = \left(1 - \frac{\Delta Winh}{\Delta wblank}\right) x\ 100 \quad (2)$$

$$\theta = \frac{\Delta wblank - \Delta winh}{\Delta wblank} \quad (3)$$

Where, Δw is mass loss (mg), A is surface area of metal (cm^2), T is time (h), R_L is corrosion rate ($mgcm^{-2}h^{-1}$), % IE_{WL} is IE (%) and θ is surface coverage respectively.

Optimization of Inhibition Efficiency using Response Surface Methodology

A three-factor-five level central composite design (CCD) involving the effect of inhibitor concentration, temperature and exposure time was adopted to examine their combined effects on the inhibition efficiencies as response. The experimental design range showing the independent variable levels for the corrosion inhibition is shown in table 1.

The experiment were randomly performed to avoid systematical errors. A total of twenty runs were generated in the design matrix. The result were analyzed using the DX7 Design Expert 716 software.

TABLE 1: Experimental Design Range and Independent Variable Levels for Corrosion Inhibition of *Piliostigma thoningii*

Independent variables	Units	Range				
		Lowest $-\alpha$	Low -1	Centre 0	High +1	Highest +α
A: Concentration	g/L	0.2	070	0.7	1	1.2
B:Tempreture	K	303	318	318	333	333
C:Time	h	0.045	2.5	2.5	4.0	5.02

Surface Morphology Examination

A scanning electron microscope was used to evaluate the surface morphological modifications of mild steel immersed in inhibited and uninhibited acid solutions. The mild surface was well polished before immersion into the test solutions for 5 hours at the room temperature after which they were retrieved, rinsed with distilled water, dried and examined by scanning. The imageries were taken from the segment of specimen from where the information was obtained.

RESULTS AND DISCUSSION

Optimization of the Inhibition Process Using Response Surface Methodology

Central composite design (CDD) was applied to optimize the inhibitive reactions in order to see the effect of the interaction between the independent variables: temperature, concentration and time and also to explore how the interaction affect the inhibition efficiencies.

Table 2: Result of Corrosion Inhibition of ethanol extract of *pilliostigma thoningii* stem bark

Standard Order	Factor 1 A: Concentration (g/l)	Factor 2 B: Temperature (K)	Factor 3 C: Time (h)	Actual Inhibition Efficiency (%)	Predicted Inhibition Efficiency (%)
1	0.70	318.00	2.50	89.90	84.29
2	1.00	333.00	1.00	93.75	90.44
3	0.40	333.00	4.00	70.97	68.71
4	0.40	303.00	1.00	77.70	77.84
5	0.70	318.00	2.50	93.30	84.90
6	1.00	303.00	4.00	91.55	85.55
7	0.40	303.00	4.00	90.69	85.74
8	0.40	333.00	1.00	92.00	89.36
9	0.70	318.00	2.50	63.70	72.33
10	0.70	318.00	2.50	77.50	80.55
11	1.00	303.00	1.00	75.60	85.49
12	1.00	333.00	4.00	73.80	75.59
13	0.70	318.00	2.50	83.30	85.89
14	0.70	318.00	5.02	87.00	96.09
15	0.70	292.77	2.50	69.62	71.17
16	0.70	318.00	0.45	71.00	71.17
17	1.20	318.00	2.50	72.50	71.17
18	0.70	318.00	2.50	73.00	71.17
19	0.70	343.23	2.50	70.00	71.17
20	0.20	318.00	2.50	72.90	71.17

Optimization of the process variables and inhibition efficiencies of mild steel in 0.1M HCl in the presence and absence of PT extract concentration (0.4-1.2) with varying temperatures (303-343k) are represented in table 2. It is clear from the table that the experiment carried out at these optimum conditions relate closely with those predicted. The inhibition efficiency increases to 85.74 at 303k and decreases to the lowest inhibition efficiency of 71.17% at the highest temperature 343k. These findings are in agreement with that of (Edoziuno *et al.,* 2020; Jimoh and Usman, 2021).

Statistical Model of the Inhibition Process and Analysis

A well-defined general second-order model were determined by the interactions on the optimization process conditions of concentration, temperature and immersion time as represented in equation 4.

$Y=\beta 0+\Sigma\beta iXi+\Sigma\beta iiX^2i+\Sigma XiXj+ei<jKi=1Ki=1$(4)

Where xi and xj represent the design variable and β the turning parameters.

The interactive significance of the process variables to the corrosion inhibition process was evaluated using ANOVA. The p-value was used to determine the significance of the model while the model adequacy is tested by the coefficient determination (R^2). The model is coded and the actual values showing the relative impact of the factors for the inhibition process is given respectively in equation 3 and 4 as follows:

$IE(\%) = +71.17 + 2.44 * A - 2.94 * B + 3.03 * C + 0.74 * A * B - 1.38 * A * C + 4.10 * B * C + 1.86 * A^2 + 3.31 * B^2 + 7.01 * C^2$(5)

Final equation in terms of actual factors

$$IE(\%) = +1817.66542 - 65.66308 * Concetration$$
$$-10.13195 * Temperature - 69.38853 * Time$$
$$+0.16500 * Concentration * Temperature$$
$$-3.06111 * Concentration * Time$$
$$+0.18233 * Temperature * Time$$
$$+20.70396 * Concentration^2$$
$$+0.014724 * Temperature^2$$
$$+3.11447 * Time^2 \quad (6)$$

From the ANOVA, the model F-value of 3.06 implies that the model is significant. There is only a 4.80% chance that a "MODEL F-VALUE" this large could occur due to noise. Values of "Prob> F" less than 0.0500 indicate model terms are significant. If there are many insignificant model terms (not counting those required to support hierarchy). The lack of "Fit F-value" of 40.94 implies the lack of fit is significant. There is only a 0.05% chance that a lack of "fit F-value this large could occur due to noise. Non-significant lack of fit is desirable. The "pred R-squared" of -0.9740 agrees with the "Adj R-squared" of 0.4941. A negative "pred R-squared" implies that the overall mean is a better prediction of the response than the current model. "Adequate" precision measures the signal to noise ration. A ratio greater than 4 is desirable. The ratio of 5.638 indicates an adequate signal and hence this model can be used to navigate the optimization of the inhibition process.

Plot of Predicted and Actual Experimental Values of IE and the Rest

Experiments carried out at this optimum conditions relate with those predicted. The plot showing the correlation between actual and predicted inhibition efficiency is shown in figure 1a while the plot of residual and predicted values as well as the internal normal plots of residual are shown in figure 1b and 1c respectively. The optimum inhibition efficiency of the ethanol extract of stem bark of *pilliostigma thoningii* is 93.75% at the optimal concentration of the inhibitor of 1.0g/L, temperature of 333K and time of 1h. The inhibition process is therefore, shown to be concentration, time and temperature dependent (Dominic and Monday, 2016).

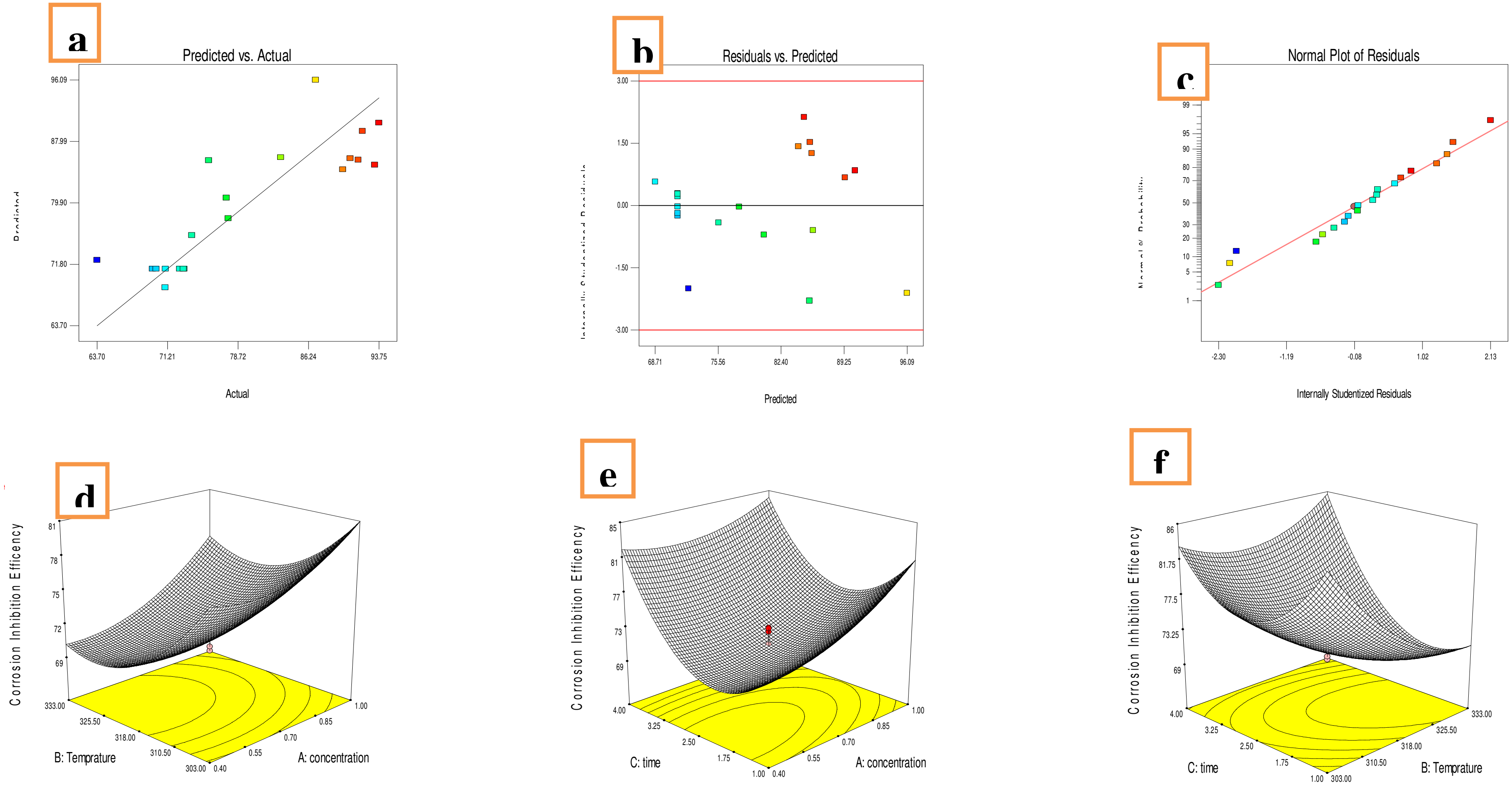

Fig 1: Plot of (a) predicted vs actual (b) residual vs predicted (c) Normal probability of residual and 3-D surface plot for the (d) Inhibition Efficiency, temperature and acid concentration (e) Inhibtion Efficiency, immersion time and extract concentration (f) Inhibition Efficiency, immersion time and temperature

Figure 1d shows the 3-D surface plot for the inhibition efficiency, the immersion time and the extract concentration, keeping the acid concentration and the temperature at their mean value of (1.0g/L) and (303K) respectively. The inhibition efficiency increases with both the immersion time and the increase in the extract concentration. For figure 1e, it depicts the 3-D surface plot for the inhibitor efficiency, the temperature and the extract concentration, while the other two factors, the acid concentration and the immersion time are kept at their mean values of 0.7g/L and 2.5hrs respectively. The maximum inhibition efficiency is indicated at the maximum extract concentration (1.0g/L) and the lowest temperature of (303k). Figure 1f shows the 3-D surface plot for the inhibition efficiency, the acid concentration and the extract concentration keeping the temperature and the immersion time at their mean value of 318k and 2.5h respectively. The plot indicates that the inhibition efficiency increases slightly with the increase in the extract concentration. However, the maximum inhibition efficiency is predicted at the lowest acid concentration and the highest extract concentration.

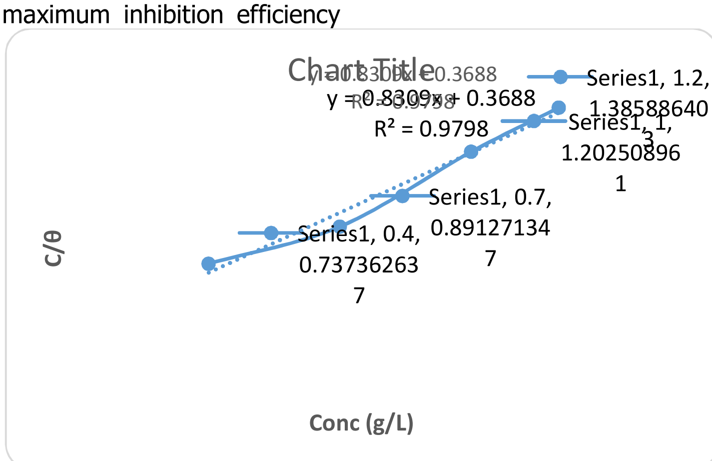

Figure 3: Adsorption Isotherm model for the inhibition of mild steel using crude ethanol extract of *pilliostigma thoningii* stem bark

3.4. The Adsorption Isotherms and its Application

Adsorption isotherm gives an information about the interaction among absorbed molecules as well as their interactions with the metal surface (Anadebe *et al.*, 2018).

Basically, all existing isotherms are available in the form of equation 7 as follows:

$f(\theta, x)exp(-a\theta) = KC$(7)

Where a represents the molecular interaction parameter which depends on the molecular interaction of the absorbed layer, and also the heterogeneity level of the surface. The $f(\theta, x)$ denotes the configurational factor which depends on the physical interaction of the model (Dominic and Monday 2016).

In this study, the type of adsorption testing was done using the theoretical surface coverage (θ) data which gives the different adsorption isotherms. The adsorption data obtained from these experiment fitted well into the Langmuir adsorption isotherm and is stated in equation 8 as

$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh}$ (8)

Where θ is the surface coverage, C_{inh} is concentration of the inhibitor and K_{ads} is the equilibrium constant for the adsorption process (Onukwuli *et al.,* 2021).

Table 3: Adsorption isotherm parameters for the inhibition process on mild steel

Adsorption isotherm	**Slope**	**Intercept (g/L)**	**R^2**	**K (L/g)**	**ΔG_{ads} KJ/mol**
Langmuir	0.8309	0.3688	0.9798	2.711	-12.628

The standard free energy of adsorptionΔG_{ads}, is calculated using the expression in equation 9

$\Delta G_{ads} = \frac{1}{55.5} exp\left(\frac{-\Delta G_{ads}}{RT}\right)$(9)

On rearranging the equation, the following equation was obtained

$\Delta G_{ads} = -RTln(55.5K_{ads})$(10)

Equation 9 is used to calculate the adsorption parameters at temperature 333k for *pilliostigma thoningii* at optimal conditions. The adsorption parameters obtained from the isotherm (figure 3) for the inhibitor is listed in table 3.
However, many adsorption isotherm were tested at the optimum condition of temperature and time, but the Langmuir adsorption isotherm were the best fitted for the inhibition of the molecules of the *pilliostigma thoningii* stem bark extract on mild steel surface. The straight line observed reveals that the main process of inhibition is adsorption, and it also shows that increase in inhibitor efficiency with increase in the inhibitor is an indication of an increase in number of components of the inhibitor adsorbed over the mild steel surface blocking the active site, thereby protecting the metal from corrosion (Anadebe *et al.,* 2018).The closeness of the slope to unity (0.8309) and the non-zero intercept is due to the interaction between the inhibitor molecule on the metal surface and also the changes in the adsorption heat with the increasing surface coverage.
The values of K_{ads} gives a pointer to the force of adherence that exist between an adsorbate and an adsorbent. Larger values of K_{ads} suspects adsorption and therefore improved inhibition efficiency (Lavanya 2021). From the values of ΔG_{ads} calculated as presented in table 3, negative value implies the spontaneity of the adsorption process and stability of the adsorbed layer on the mild steel surface. The ΔG_{ads}value is lower than -20KJ/mol which is consistent with physisorption.

Scanning Electron Microscopic Analysis

The SEM images were taken to study the surface morphology of mild steel under the influence of HCl medium and the corrosion inhibitor. This helps to initiate the interaction between the inhibitor molecules and the metal surface. These analysis are carried out on both the corroded and the protected metal surfaces. Figure 2b and 1c shows the SEM micrographs of mild steel surfaces for both the uninhibited and inhibited system upon exposure for 6hours at room temperature. Figure 1a shows unvarying smoothness of polished mild steel surface before immersion. Generally, it was deduced that the inhibition observed was as a result of formation of insoluble stable protective coverage by adsorption process (Ebenso *et al.,* 2008).

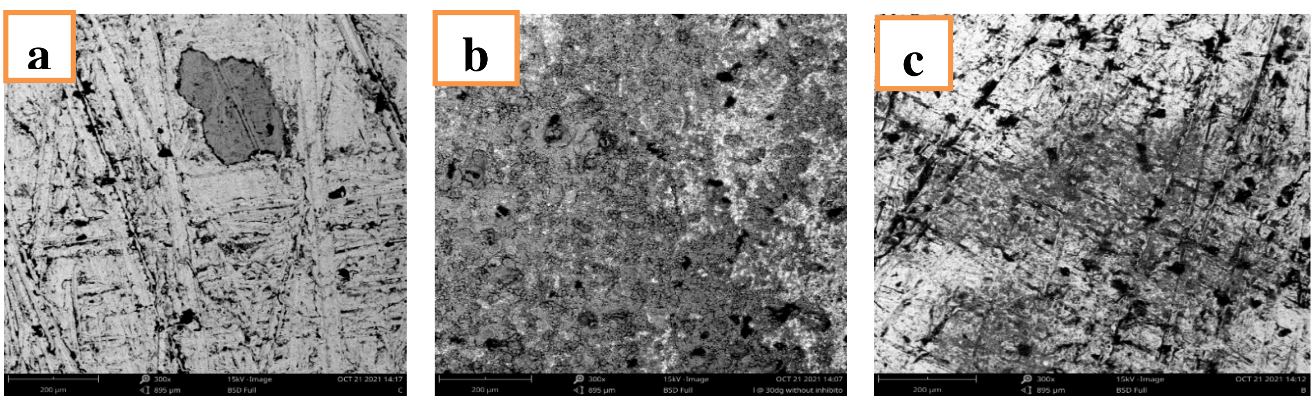

Fig 2: SEM images of (a) Fresh mild steel (b) Corroded mild steel and (c) Mild steel with inhibitor

CONCLUSION

The Response Surface Methodology (RSM) used in this research has accurately predicted the process parameters for maximum corrosion inhibition efficiency of *Pilliostigma thoningii* stem bark extract on mild steel in 1M HCl. The optimum inhibition efficiency of 93.75% at 1.0g/L of inhibitor concentration, temperature of 333K and reaction time of 1h was accomplished. Similarly, the effectiveness of the inhibitor was also supported using scanning electron microscopy (SEM). The data obtained were fitted into various adsorption isotherms and the mechanism of the interaction was found to conform best to the Langmuir isotherm. The low negative value of ΔG_{ads} is an indication of physical interaction between the inhibitor and mild steel. This also signifies the spontaneity of the adsorption process and the stability on mild steel surface.

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