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Research article

Optimization of chicken nail extracts as corrosion inhibitor on mild steel in 2M H_2SO_4

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ABSTRACT

The inhibiting effects of Chicken Nails Extract (CNE) on Mild Steel corrosion in 2M H_2SO_4 were investigated in this study. The effect of the concentration of inhibitor (0.5–1.5 g/l), time (5–8 h) and temperature (40-70^{o}c) on Inhibition efficiency were investigated using Response Surface Methodology. The Physiochemical analysis and proximate analysis of the CNE were investigated; the result showed that organic constituents were present which made the Chicken nails extract a good inhibitor. The rate of corrosion increases as time and temperature increase while the Inhibition efficiency was discovered to increase as the inhibitor concentration increases. The optimum conditions obtained were temperature 63.63 °C, time 5 h and inhibitor concentration of 0.1 g/l. The optimum Inhibition Efficiency at these optimum conditions was predicted to be 74.04%. The micrographs result of Scanning Electron Micrographs analysis showed that in the presence of the inhibitor, there was a passive layer of a film formed on the surface. This study revealed that Chicken Nails Extract is a potentially good green inhibitor for Mild steel corrosion in 2M H_2SO_4.

1. Introduction

The word "corrosion" is used to describe the electrochemical degradation of metals and alloys in the atmosphere. It is a gradual destruction of materials via their chemical reactions with their surroundings. This major challenge has been estimated at 3–5 percent in the U.S. and European nations (Bhaskaran et al., 2005) and has severe implications in industries. For instance, the presence of numerous corrosive substances in crude oil contributes to the corrosion observed in pipelines and in the petroleum industry in general (Obot et al., 2009). Corrosion engineers and scientists are now showing more research interests in the green technology approach via the adoption of eco-friendly materials as corrosion inhibitors (Adindu et al., 2017; Hamadia et al., 2018; Qiang et al., 2018). The most widely used ferrous alloy for a broad spectrum of applications is Mild Steel. This is because of its weldability and use in the construction of pipelines and other related materials. Corrosion inhibitors are chemical compounds which when added to a metal or an alloy reduces the corrosion rate of the material.

Corrosion inhibition can therefore be defined as a process of alleviating or reducing corrosion. It is known to be the most economical and pragmatic approach to reducing corrosion attack on metals (Palou et al., 2014; Evrim et al., 2016). In recent times, researches on Inhibitors have focused on the use of environmentally friendly materials. The corrosion inhibitory effects of various plant extracts had been reported with promising inhibitory efficiency. Some of these plants are: Katemfe (Olawale et al., 2018a, b); Cashew waste (Olawale et al., 2015); Bamboo (Li et al., 2012, 2014); Gentiana *olivieri* (Evrim et al., 2016); Melon and Groundnut peels (Ita et al., 2016); Pawpaw leaves (Omotiowa and Onukwuli, 2017); Bitter kola leaf (Anadebea et al., 2018); *Ocimum gratissium* (Udunwa et al., 2017); *Thevetia Peruvianna* (Fouda et al., 2016); Mango extract (Onukwuli and Omotioma, 2016); phyllantus amarus extract (Okafor et al., 2008) *Vernonia amygdalina* (Loto et al., 2013), Katemfe fruit leaves (Olawale et al., 2018a, b) Almond fruit extract (Olawale et al., 2018a, b) Waterlemon (Odewunmi et al., 2015) Bitter leave root (Awe et al., 2015); Extract of Murraya koenigii Leaves (Quraishi et al., 2010); Gnetum Africana leaves (Nnanna et al., 2013); Gentiana olivieri extract (Evrim et al., 2016); Sida acuta (Umeron et al., 2016); Origanum majorana Extracts (Challouf et al., 2016) *Nauclea latifolia* (Uwah et al., 2013); *Slavia aucheri mesatlantica* (Znini et al.,2012); Pigeon pea leaf (Anadebe et al.,2019). The inhibiting activity of these

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plant/animal extracts is as a result of the phytochemical/physiochemical compositions such as tannins, alkanoids and amino acids. This made it exhibit good inhibiting action. Klodian and Matjaz (2016); McCafferty, 2010 stated that corrosion-related costs can be estimated as up to 3–5% of the Gross National Product in most developed countries. This concern has led to new initiate with the aim of reducing the impact of corrosion. Although, Manikandan et al., 2015, Anadebe et al. (2019) and Stefania et al. (2019) have used Chicken feathers and amino acids as corrosion inhibitors respectively, Chicken nails have not been used in this regard. This study is therefore tailored for dual purposes; the first purpose is the establishment of the effectiveness of chicken nail extract as corrosion inhibitor and also the use of chicken nails extract as a form of anti-corrosion agent for mild steel in acidic media using optimization approach.

2. Experimental procedure

2.1. 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 0.2 cm drilled in the middle was cleaned with emery paper to expose the shiny surface, degreased with acetone to remove any oil impurity, washed using distilled water and then dried in air before being stored in a desiccator.

2.2. Preparation of chicken nails extract

The chicken nails (CN) used were collected from Landmark University Teaching and Research farm and dried for five (5) days. Dried CN was then pulverized and kept for extraction. For each extraction process conducted, 30 g of the chicken nails powder was placed in the Soxhlet extractor with 180 cm^3 of ethanol for 4 h. The extract solution was afterwards concentrated and employed for the preparation of inhibitors concentrations in 2M H_2SO_4.

2.3. Gravimetric or weight loss method of the corrosion inhibition study

Weight loss measurements were conducted under total immersion using 250 ml capacity beaker containing prepared solution at a temperature range from 40 °C to 70 °C which was maintained in a thermostatic water bath. The Mild Steel Coupons were weighed and dropped in the inhibitor concentration media of (0.5–1.5 g/l) with time variation from 5-8 h for each run using a design matrix format for variables interaction.

Furthermore, after each exposure time, the mild steel coupons were removed, washed thoroughly to remove the corrosion product with emery paper, rinsed properly with distilled water and then dried in acetone. The mild steel was weighed again to determine the weight loss, in grams by finding the difference between the weight of the mild steel before and after immersion. The corrosion rates (g/cm^2h) in the absence and presence of the understudied inhibitors were determined. The Weight losses were calculated by finding the difference between the weight of each coupon before and after immersion;

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

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

Table 1
Experimental range of independent variables with different levels for the CN. Inhibition of Chicken nails Extract on mIld Steel in H_2SO_4 solution.

Independent Variables	Factor Levels		
Time(hrs)	5	6.5	8
Temperature(°c)	40	55	70
Inhibition Concentration (g/l)	0.5	1.0	1.5

Table 2
Central Composite Design factors and levels#.

Run	Factor 1 A.Time(hrs)	Factor 2 B. Temp (°c)	Factor 3 C.Inhi (g/l)
1	5	40	1.5
2	8	40	0.5
3	5	40	0.5
4	5	70	0.5
5	8	70	0.5
6	5	70	1.5
7	8	70	1.5
8	5	55	1
9	8	55	1
10	6.5	55	0.5
11	6.5	55	1.5
12	6.5	55	1
13	6.5	55	1
14	6.5	55	1.5
15	8	40	0.5
16	8	70	0.5
17	6.5	55	1
18	8	55	1
19	6.5	55	1
20	6.5	55	0.5

Table 3
Proximate analysis.

Proximate	Result
Protein	Present
Moisture	Present
Ash	Present
Glyceride	Present
Lysine	Present
Nitrogen	Present
Amino acids	Present
Fat	Present

Table 4
Qualitative Determination of Physiochemicals present in the methanol and ethanol extract of chicken nails.

Reagent	Color change	Result
Wagner	Brown ppt.	+
10% KOH	Dirty white ppt.	-
10% NaOH	Yellow ppt.	++
Distilled water	A persistent foam	+

NB: +++ = highly ++ = moderately present; + = low present; - = Not present.

$$IE\% = \frac{w_0 - w_1}{w_0} \times 100 \tag{3}$$

where, $w_1 w_0$ and are the weight loss values in the presence and absence of inhibitor, respectively. A is the total area of the mild steel sample; t is the immersion time, IE% which is the inhibition efficiency.

2.4. Proximate analysis

Proximate analysis was done on the chicken nails to determine the presence of some active constituents that make a good inhibitor.

Table 5
RSM result of the corrosion inhibition of Mild Steel in H_2SO_4 by Chicken Nails Extract (CNE).

Run	Factor 1 A.Time(hrs)	Factor 2 B. Temperature (°c)	Factor 3 C. Inhibition Conc (g/l)	Weight Loss(g)	Response 1 Corrosion rate (mg/mm^{2}h)	Response 2 Inhibition efficiency (%)
1	5	40	1.5	0.144	0.072	2.30
2	8	40	0.5	0.758	0.236	13.03
3	5	40	0.5	0.222	0.111	3.50
4	5	70	0.5	4.774	3.467	68.08
5	8	70	0.5	4.232	1.322	60.4
6	5	70	1.5	4.560	2.28	69.61
7	8	70	1.5	4.422	1.38	67.8
8	5	55	1	2.166	1.083	31.24
9	8	55	1	4.890	1.528	74.06
10	6.5	55	0.5	4.512	1.735	70.67
11	6.5	55	1.5	2.622	1.008	39.28
12	6.5	55	1	3.426	1.317	54.74
13	6.5	55	1	3.574	1.374	53.93
14	6.5	55	1.5	2.622	1.008	39.28
15	8	40	0.5	0.758	0.236	13.03
16	8	70	0.5	4.232	1.322	60.4
17	6.5	55	1	3.426	1.317	54.74
18	8	55	1	4.890	1.528	74.06
19	6.5	55	1	3.426	1.317	54.74
20	6.5	55	0.5	4.512	1.735	70.67

Table 6
ANOVA for the corrosion inhibition of Mild Steel in H_2SO_4 by Chicken Nails 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	10.78	9	1.20	15.66	<0.0001	Significant
A(time)	0.12	1	0.12	1.56	0.2398	
B(temperature)	4.34	1	4.34	56.73	<0.0001	
C(inhibitor concentration)	0.10	1	0.10	1.35	0.2725	
A^2	9.357	1	9.357	0.12	0.7338	
B^2	0.038	1	0.038	0.50	0.4965	
C^2	1.352	1	1.352	0.018	0.8969	
AB	1.93	1	1.93	25.30	0.0005	
AC	0.60	1	0.60	7.79	0.0191	
BC	0.37	1	0.37	4.83	0.0526	
Residual	0.76	10	0.076			
Lack of fit	0.76	2	0.38			
Pure error	3.249	8	4.061			
Cor. Total	11.55	19				
Std. Dev.	0.28			R- squared		0.9338
Mean	1.27			Adj R- squared		0.8741
C.V%	21.75			Pred. R-squared		0.6686
Press				Adeq. Precision		18.679

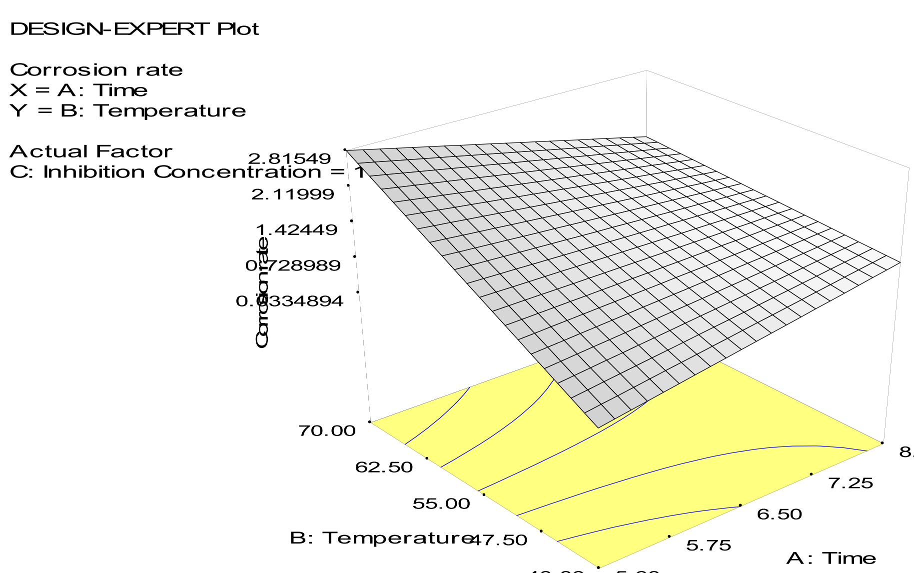

Fig. 1. Effects of time and temperature on corrosion rate of CNE on Mild Steel at constant inhibitor concentration.

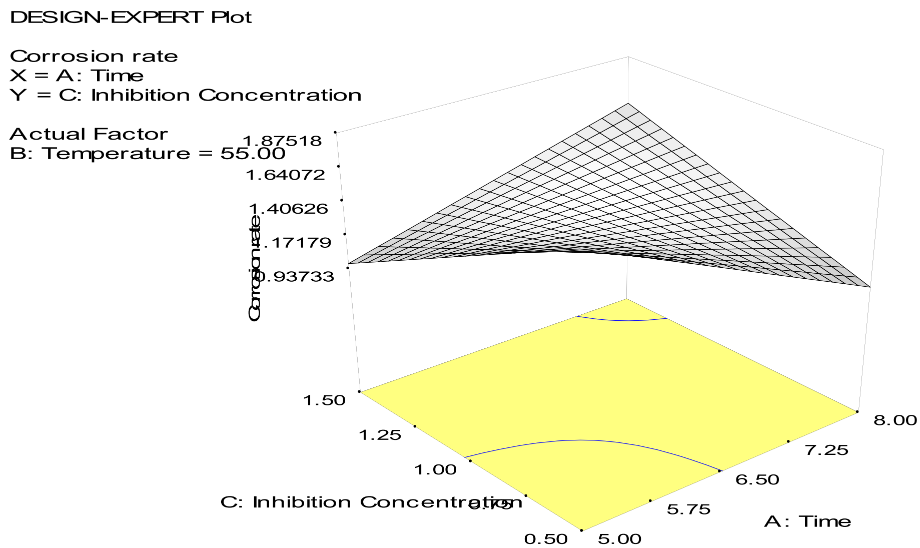

Fig. 2. Effects of time and inhibitor concentration on corrosion rate of CNE on Mild Steel at constant temperature.

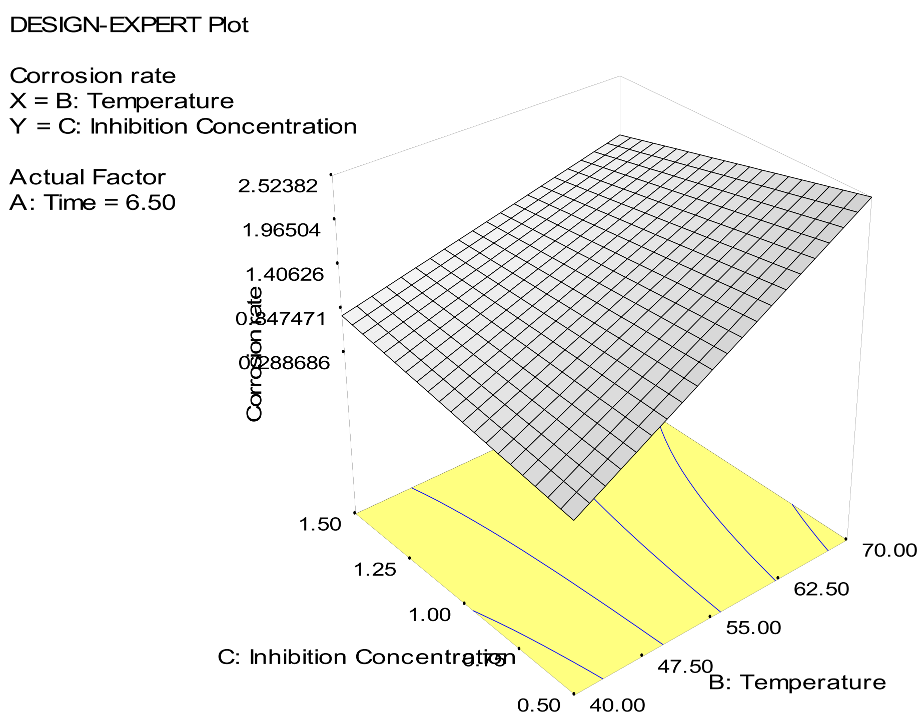

Fig. 3. Effects of temperature and inhibitor concentration on corrosion rate of CNE on Mild Steel at constant time.

2.5. Physiochemical analysis of the extract

Physiochemical analysis of the Chicken nails extract was carried out to determine the presence of active metabolites such as saponins, alkaloids, tannins and flavonoid.

2.6. Experimental design

A Central Composite Design (CCD) of 20 experimental runs which included three (3) operating variables was established for the experiments used for studying the influence of temperature of the solution, time of exposure and inhibition concentration of CNE as the variables for corrosion inhibition on mild steel. The Factor levels with the corresponding real values are presented in Table 1 while the Design Matrix is as presented in Table 2. The matrix for the three variables was varied at 3 levels (-1, 0 and +1); while the experiments were performed in random order to avoid a systematic error.

Software Design Expert (6.0.8) was utilized for data analysis. Methodology adopted by Olawale et al. (2018a, b).

The following steps in RSM considered for this study:

1. Establishment of a CCD to get the points where the experimental runs were performed.
2. Experimental observation of the corrosion inhibition effects of the various factors at the design points.
3. Obtaining a mathematical model that expresses the relationship between the process factors and the percentage inhibition efficiency.
4. Prediction of the process optimum values for the maximum inhibition efficiency via the use of RSM.

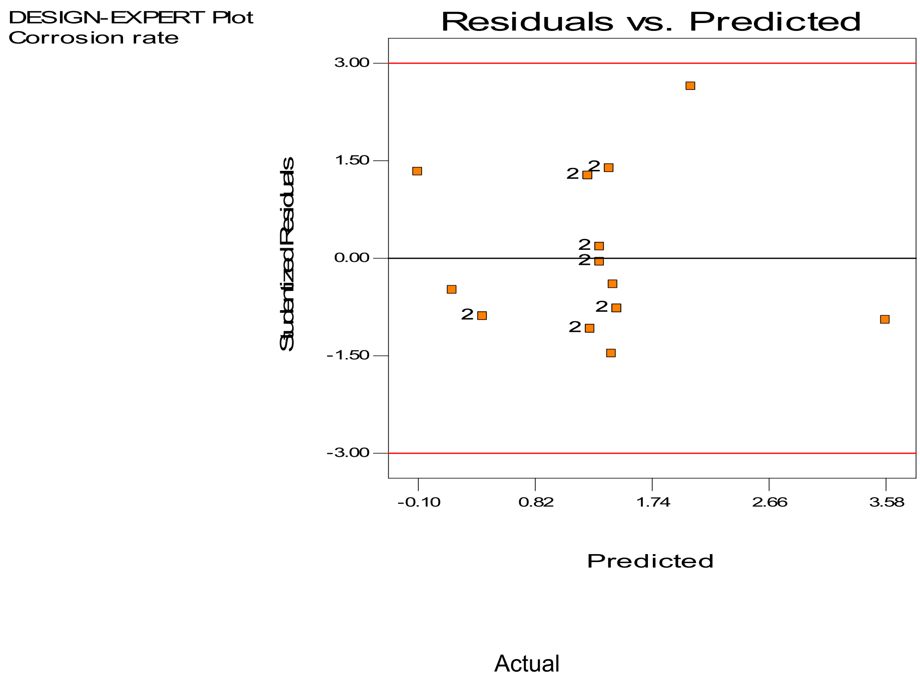

Fig. 4. Plot of residuals values versus the predicted experimental values for corrosion rate of CNE on Mild Steel.

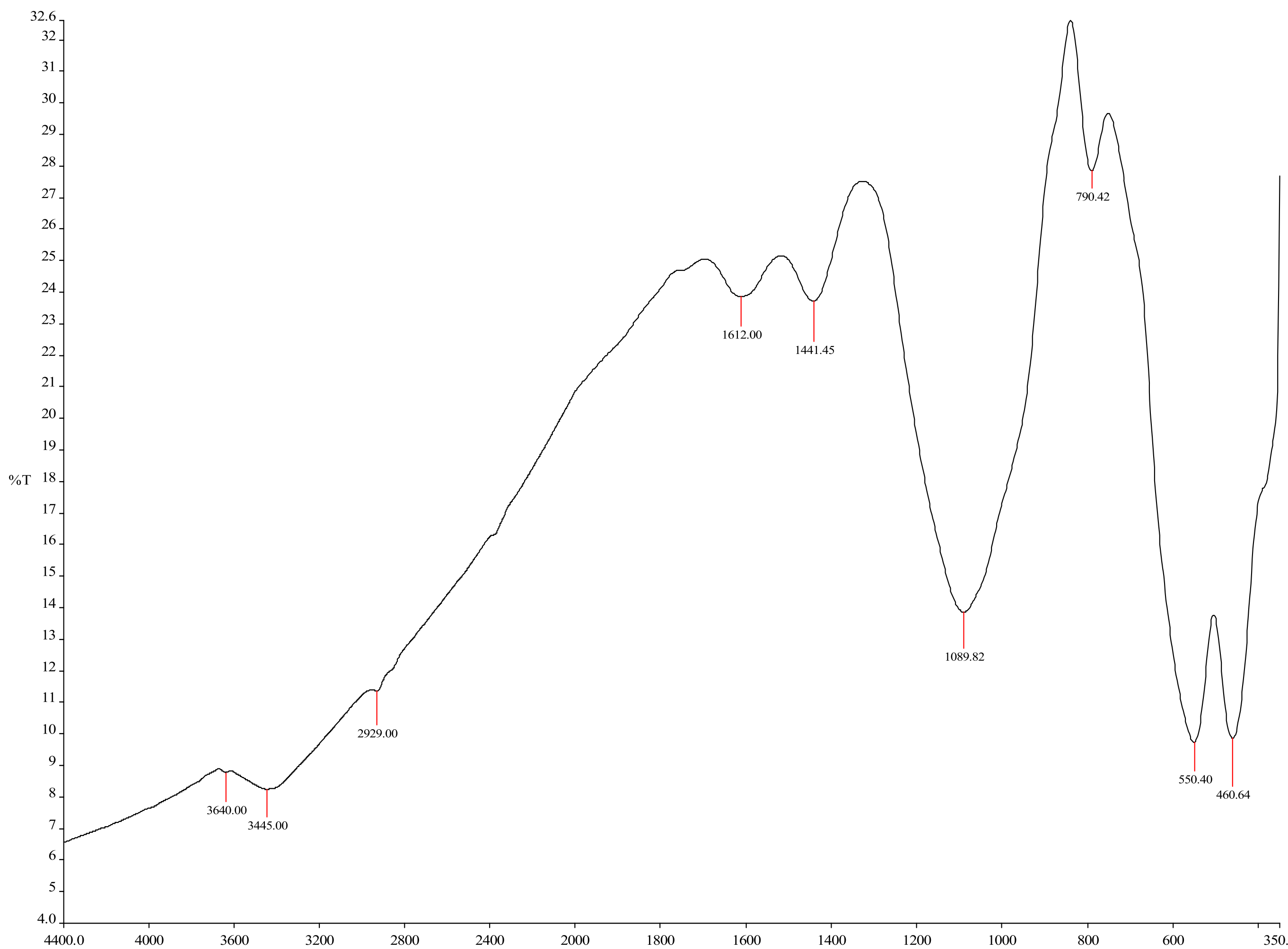

Fig. 5. Coupon without inhibitor (blank metal).

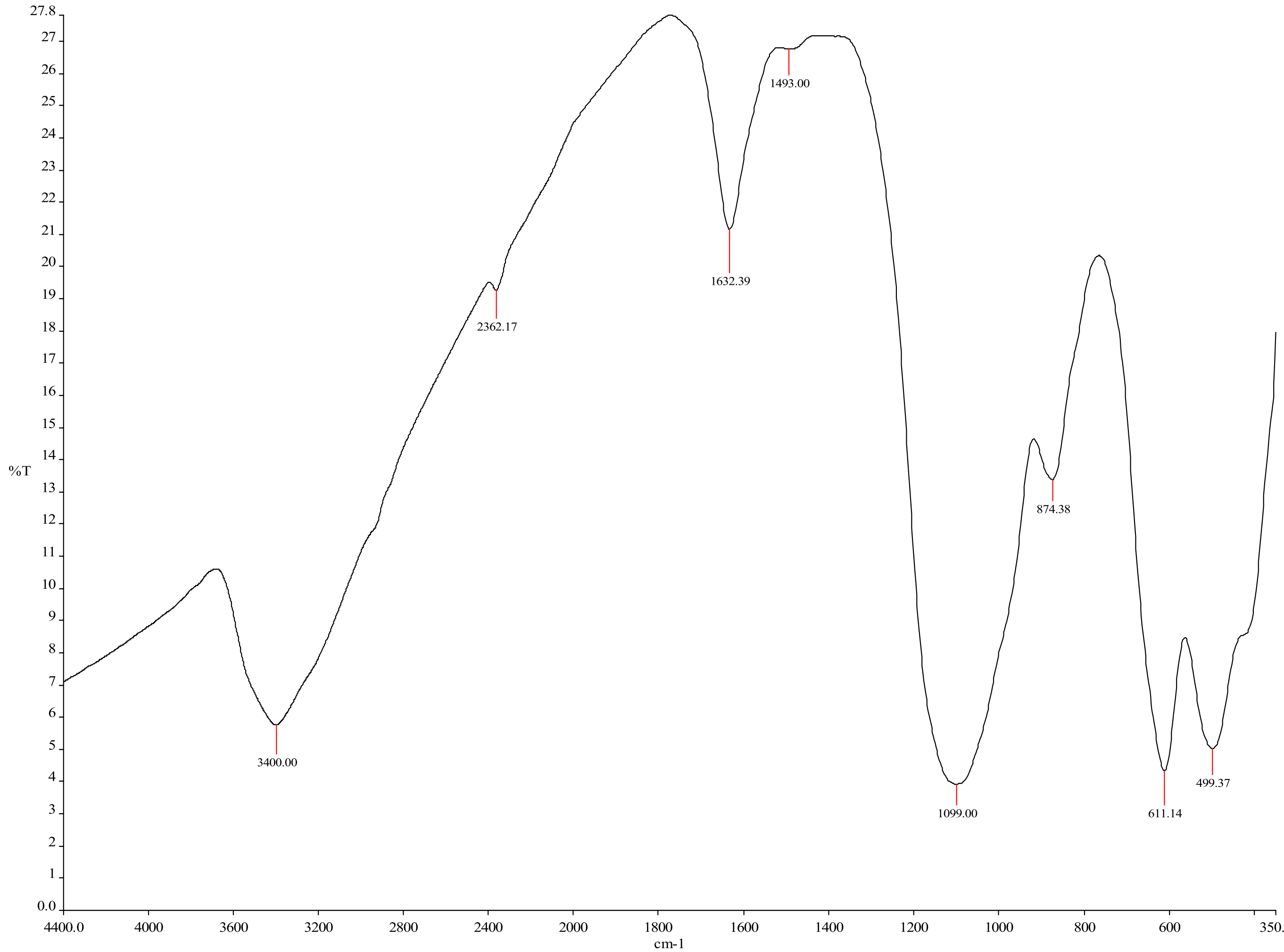

Fig. 6. Coupon with inhibitor that gave highest inhibition efficiency.

5. Experimental verification (Methodology adopted by Olawale et al., 2018a, b).

2.7. FTIR characterization

FTIR analysis was used to identify the active functional groups on the: blank Mild Steel; Mild Steel with inhibitor which had the highest inhibition efficiency and the Mild Steel with inhibitor at optimal process variables were carried out with the use of Perkin-Elmer-1600 Fourier transform infrared spectrophotometer. The sample was prepared using KBr.

2.8. Scanning Electron Microscope (SEM) analysis

The morphology of the blank Mild Steel; Mild Steel with inhibitor having the highest inhibition efficiency and the mild steel with inhibitor via the optimal process variables were examined using Scanning Electron Microscope.

3. Discussion of results

3.1. Proximate analysis results

Results of proximate analysis of the CNE are as shown in Table 3.

The inclusion of amino acids in the description of natural corrosion inhibitors is often related majorly to the presence of nitrogen in all of their molecular structures. Furthermore, some of them such as tyrosine, tryptophan, and phenylalanine contain aromatic rings that are one of the major factors reducing the corrosion rate by blocking sites on the metal due to adsorption. Amino acids have been reported widely in the literature as good and safe corrosion inhibitors for many metals in different corrosive media.

Latifa et al. (2018) reported that Amino acids are effective in the inhibition of metals in different corrosive solutions. The efficiency of those compounds is greatly affected by several factors such as the metal (surface state, chemical nature), the medium (concentration, pH, and temperature), inhibitor (concentration, structure molecular, solubility, the inhibitor-metal surface bonding), and the immersion time. Furthermore, Wu (2013) also stated that one of the encourager compounds which can be utilized as a safe corrosion inhibitor is an amino acid. They are environmentally-friendly compounds, completely soluble in aqueous media. These properties justify their use as corrosion inhibitors. The presence of amino acid in chicken nails made it organic. This property allows it to exhibit a good inhibiting action as confirmed from a report by Ambrish et al. (2012). Furthermore, Fawzy et al. (2018) stated that the corrosion mechanism was via adsorption of the amino acids (glycine, alanine, and leucine as aliphatic amino acids and histidine as basic amino acid) on the active corrosion sites. The proximate analysis result confirmed that the presence of amino acid in the chicken nail extract made it a good inhibitor for Mild Steel.

3.2. Results of the physio-chemical constituents

The result of the physiochemical constituents of chicken nail is as

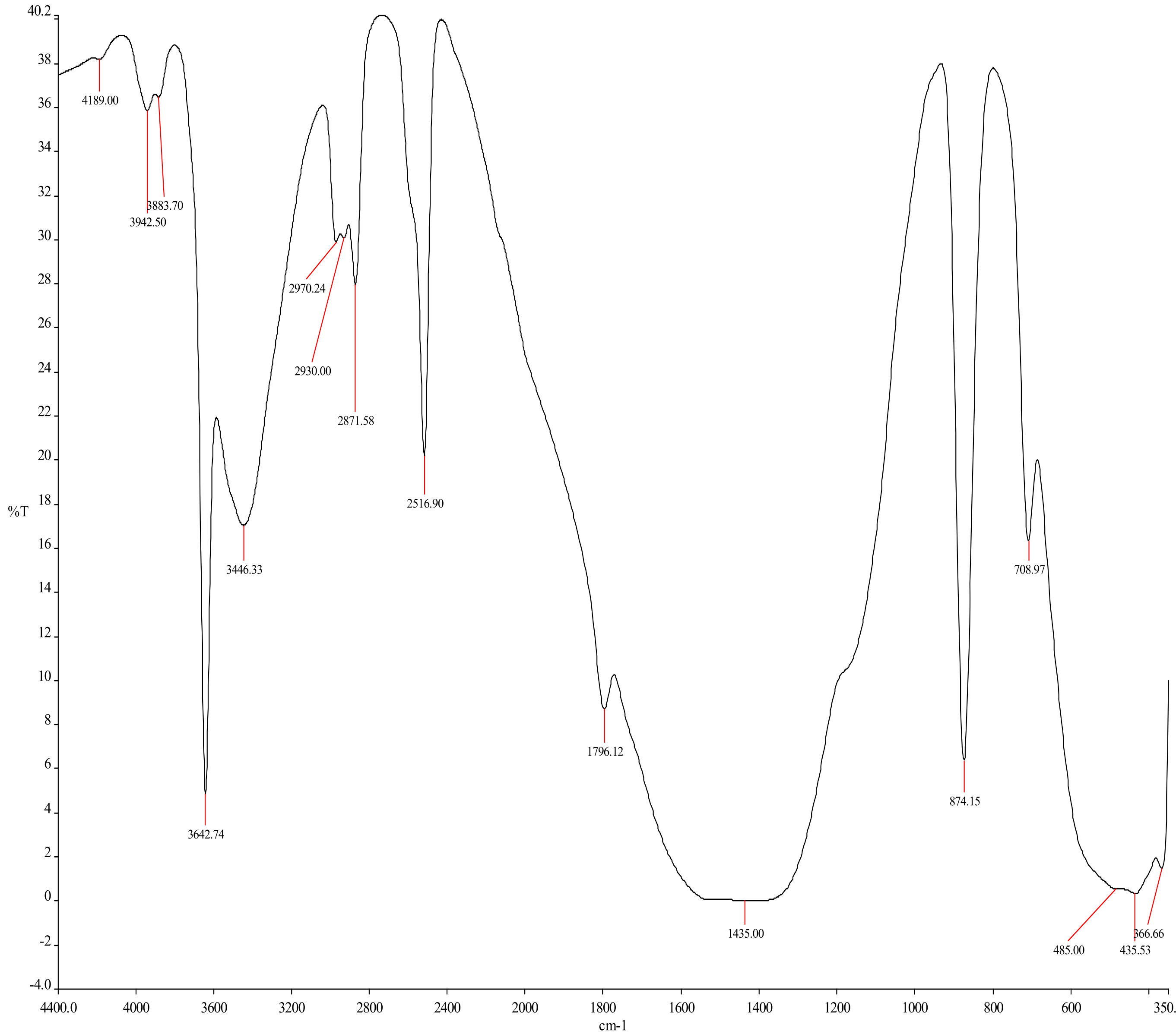

Fig. 7. Coupon with inhibitor via the optimal process variables.

shown in Table 4. It showed that saponins are highly present alkaloids and flavonoids which are major constituents of a good inhibitor. Lebrini et al. (2011) reported that natural inhibitors are often non-toxic or at least have lower toxicities to species in the environment when compared to synthetic organic inhibitors. This showed that Chicken Nail extract is a potential good inhibitor.

Table 4 above showed a high amount of flavonoid was present while Alkaloids and saponins were moderately present. The high quality of flavonoids made it an active corrosion inhibitor.

The results observed were confirmed from a report by El-Etre et al. (2005) that inhibition efficiency is characterized by the presence of complex organic species like tannins, carbohydrates, alkaloids, protein, and their acid hydrolysis products.

3.3. *Statistical analysis and modeling of the inhibition efficiency*

Analysis of the experimental data was carried out using Design Expert 10 version software to obtain the analysis of variance (ANOVA), regression analysis and the response of the interaction plots.

The result of CCD with observed inhibition efficiency and corrosion rates is as shown in Table 5; while ANOVA Table is as shown in 6 with R-squared value of 0.9338; and the Adjusted R-squared (0.8741) respectively. The values of the Adjusted R-squared and the Predicted R-squared are found to be in reasonably closed because the difference is less than 0.2. This showed that the experimental data obtained for the inhibition efficiency are statistically consistent and the second-order polynomial model selected was suitable for modeling. The second-order polynomial equation in final equation terms of the coded values of the process parameters is as given below: (see Table 6)

$$\text{Regression equation} = +54.74 + 1.02A + 28.93B - 2.96C + 4.04A^2 - 17.87B^2 - 4.01C^2 4.0AB + 1.21AC + 1.59\,BC$$

where:

A represents:(time), B represents: temperature;
while C represents: inhibitor concentration.

3.4. *Results of 3D surface response plots for corrosion rate on mild steel*

The 3D curves showed the relationship between variables and

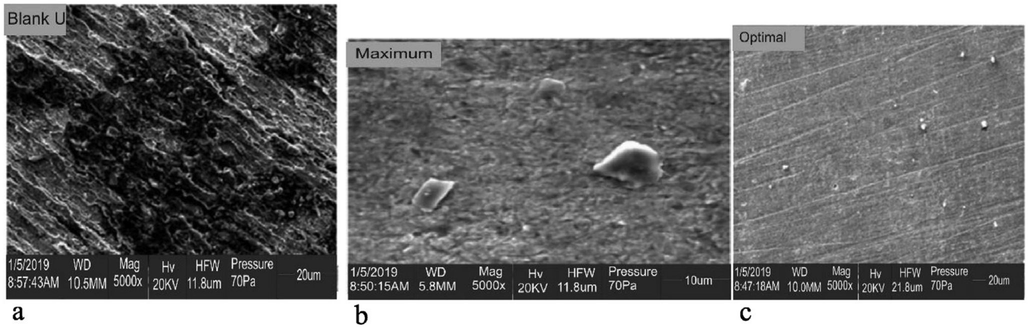

Fig. 8. (a–c) SEM macrographs of the surface morphologies of: (a) bank mild steel; (b) mild steel obtained via the highest inhibition efficiency and (c) mild steel obtained at the optimal predicted level which was validated.

response (inhibition efficiency) of the designed experiment in Fig: 1–3 respectively. However, Fig. 1, showed that corrosion rate increased with increase in temperature and also increased with increase with time; while Fig. 2 showed that corrosion rate decreased with increase in inhibitor concentration and also increases with an increase in time, also an increase in temperature as corrosion rate increases, as shown in Fig. 3, but decreases with an increase in inhibitor concentration. This assumed physical adsorption. Fig. 4 Plot of residuals values versus the predicted experimental values for the corrosion rate of CNE on Mild Steel**.**

3.5. *Predicted optimal levels*

The optimal process conditions observed were time: 5 h, temperature: 63.63 °C and inhibitor concentration: 1.0 g/l respectively.

3.6. *Results of fourier transform infrared spectroscopy (FTIR) analysis*

Coupons without inhibitor, the coupon with the highest inhibition efficiency and the coupon with inhibitor via the optimal process variables are presented as in Figs. 5, 6, and 7 respectively; were investigated to identify the functional group present using FTIR –Perkins Elmer Spectrum BX II model.

Furthermore; Fig. 5 showed the spectra of the blank metal. Fig. 6 showed the spectra of the coupon with the highest inhibition efficiency with the formation of a protective film. The analysis result showed the presence of C–H bond; C=O, C = = C, C–H while Fig. 7 revealed the presence of N–H; C=O; C=C, C–H; O–H were more pronounced which predominantly was responsible for the corrosion process which confirmed what was reported by Omotioma and Onukwuli (2016); Alaneme et al. (2015); Wang et al. (2011) respectively. The wavelength differs for the functional groups because of the different strengths that are bond with each of the coupons. The best from the FTIR results was the one with the optimal process variables as shown in Fig. 7. According to Benali et al. (2013), compound or extracts with amines, alkenes, carboxylic acids, alkyl halides primary and secondary amines, and alcohol functional groups are usually effective corrosion inhibitors. This was confirmed by the report obtained in Figs. 6 and 7 respectively.

3.7. *Result of SEM analysis*

The micrographs in Fig. 8a revealed lines and serrated edges in the absence of inhibitor, due to Sample preparation which made the surface to be strongly damaged while in Fig. 8b; with the presence of inhibitor, the mild steel was favored with sulphuric ions ionized on the surface and pitting was scarcely evident. Furthermore, Fig. 8c showed more effectiveness of the chicken nails extract which formed more protective films on the mild steel by reducing the dissolution of the steel in the acidic media. The result confirmed what was reported by Evrim. et al. (2016).

3.8. *Mechanism of corrosion*

The mild steel surface of the corrosion product in the absence of the inhibitor was porous and as a result, it showed no corrosion protection. The mild steel via the highest inhibition efficiency with CNE had minimal corrosion with the evidence of the inhibiting via CNE on the surface while the mild steel with the optimal predicted variables showed smoother surface due to the formation of a more compact protective film. The SEM result via optimal process variables showed white patches due to the presence of protective oxide which showed an appreciable resistance to corrosion.

4. Conclusion

The result obtained from the physiochemical analysis showed that alkaloids, flavonoids, and saponins are present. Furthermore, the results of the proximate analysis showed the presence of amino acid which is a good constituent of a corrosion inhibitor. The FTIR results affirmed the existence of redox reactions for the tested Chicken nails extracts and as a result, acted as a mixed-type inhibitor. The optimal process conditions observed were; time: 5 h, temperature: 63.63 °C and inhibitor concentration: 1.0 g/l respectively. The result of Scanning Electron Micrographs analysis showed that the passive layer of film was formed on the surface of the mild steel which confirmed the high performance of the inhibiting effect of the Chicken Nails Extract which subdued corrosion. It can be concluded that Chicken nails extract is a good inhibitor.

Declarations

Author contribution statement

Olawale O: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Bello J O: Analyzed and interpreted the data; Wrote the paper.

Ogunsemi B T: Conceived and designed the experiments; Wrote the paper.

Uchella U C: Performed the experiments.

Oluyori A P & Oladejo N K: Contributed reagents, materials, analysis tools or data.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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