Optimization and Effect of Leaves Extracts on Corrosion of Mild Steal in Acidic Medium

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Abstract: Optimization of inhibitive effects of leaves extracts of carica papaya (pawpaw) and veroniniaamigdanina (bitter) on corrosion of mild steel within acidic medium was studied by weight loss tmethod using central composite design of response surface methodology for optimization. Atomic Absorption Spectrophotometer was used to study and determine its elemental compositions. The extract was obtained through liquid extraction using ethanol, as well as analyzed to determine phytochemical components and functional groups using American Society of Testing Materials and Fourier Infra red Spectrometer respectively. The effect of process variables such as concentration of inhibitors, time, temperature and concentration of medium on corrosion rate in the acidic (HCl) medium was studied. The result obtained showed that the major composition of the mild steel is iron and the presence of phytochemical components and functional groups made the extracts perform as good inhibitors. It was observed that the corrosion rate decrease while there was an increase in concentration of the inhibitor, time and concentration of the medium and increase with increase in temperature. The optimal conditions are: concentration of inhibitor 16.36mg/100ml, time, 6.55h, temperature 48oC, concentration of medium 2.2M in pawpaw leaf extract inhibited acidic medium; concentration of inhibitor 18.94mg/100ml, time, 5.5h, temperature 48oC, concentration of medium 1.74M in veroniniaamigdanina (bitter) extract inhibited medium; The optimal conditions obtained gave inhibition efficiencies of 88.4% and 96.4% in pawpaw leaf extract and better leaf extract inhibited acid media respectively. It shows that leaves extracts can inhibit corrosion of mild steel in acidic medium.

Key words: Carica papaya (pawpaw), veroniniaamigdanina (bitter), HCl, inhibitors, optimization.

1. Introduction

Over the years, much money are been spent trying to avert control of mild steel in a structures. Corrosion can be defined as an irreversible reaction of a material with the environment, which usually (but not always) results in a degradation of the material or its properties [1], [2]. It is the inevitable deterioration of materials by the chemical interaction with their environments. It is the returning of the materials to its original form (stable state) to the mother earth. So there are several aspects of corrosion: the material, the environment, and the material properties [3]-[5]. The degradation of metals has a negative influence on the populace and is apparently an issue of concern as it influences the economic cost and also creates safety awareness from the damages such as in pipelines, building, bridges, waste water system, and even our residence [6], [7]. A recent study in USA of industrial sectors, predicted that the cost of corrosion would rise over $1.1 trillion in 2016. These estimates were based on a landmark study by NACE (National Association of Corrosion Engineers) that estimated (direct) corrosion costs were $276B in 1998 as reported in the NACE Corrosion Costs Study.

A number of methods are available in the controlling of corrosion in the structures.. The most frequently used methods include organic and metallic protective coating; corrosion resistant alloys, plastics as well as polymers; corrosion inhibitors as well as cathodic production method which are used on piping, subversive storage cistern and offshore facilities [8], [9]. Though, there is no accord concerning which method evaluate corrosion levels of structures most accurately, that is why the method for corrosion control will remain a problem no matter how corrosion inhibition is being carried out.

Corrosion Inhibitors are some chemical substance added to the corrosive environment in small quantities to substantially reduce the rate of corrosion. Over the years, a lot of researches have been done on the area of corrosion inhibition. Several authors have carried out research on corrosion in acidic medium like hydrochloric acid, sulphuric acid etc but sufficient work is yet to be done on the optimization and effect of process parameter of selected factorsaffecting corrosion of mild steal. Therefore, this paper aims to achieve and promote the use of locally sourced plants as corrosion inhibitors and provide optimum conditions and effect of process parameters [1].

2. Materials and Methods

Carica papaya (pawpaw) and*veroninia amigdanina (bitter)* leaves were collected from Amokwe, Udi, Enugu State, Nigeria. The metal coupon (mild steel) was purchased from Kenyatta Market, Achara Layout, Enugu, Enugu State, Nigeria. The chemicals used such as HCletc were purchased from Gerald Chemicals Ltd Ogbete Main Market, Enugu, Enugu State, Nigeria and they were of analytical grade.

2.1. Extraction of Juice from the Leaves

The plants leaves were washed thoroughly with water to remove unwanted material. The samples were shaded dried, pulverized, and weighed. The weighed inhibitors were stored in desiccators prior to use. 60g each of the ground samples was mixed with ethanol tightly covered to prevent evaporation and kept for 48 hours. Then the extracts were filtered to obtain high yields of the concentration. The filtered solutions were heated in rotary evaporator setup to expel the ethanol at 70°C for 20 min.

2.2. Characterization of the Extracts

The phytochemical analysis of the extracts acquired were carried out at Projects Development Institute, PRODA, Enugu, Nigeria The laboratory analysis was done using AmericanSociety for Testing Methods, ASTM (D4903). The instrumentation tests were:

2.3. Preparation of Test Solution

A known concentration of a solution of HCl was prepared by dilution using distilled water. The inhibitors were each accurately weighed with weighing balance dissolve in prepared solution of HCl to obtain different inhibitors concentrations (5, to 25 mg/100ml). The test solutions were prepared in different containers to carry out the corrosion experiment.

2.4. Preparation of Metal Specimen

The mild steel obtained was analyzed to determine the metallic compositions using AAS. It was prepared for corrosion experiment by adopting the method employed by [10]. The mild steel specimens were mechanically cut into dimension of 3.0 x 3.0 x 1.5 cm (with a surface area of 9.0 cm^2). Prior to all, the mild steel coupons were mechanically polished with series of emery paper from 400 to 1200 grades to sufficiently remove any mill scale on the sample of the mild steel. The specimen was washed thoroughly with distilled water, degreased with absolute ethanol, dipped into acetone to avoid corrosion and dried in air. The dried specimens were stored in desiccators before use.

3. Experimental Procedure

Weight loss measurements were conducted under total immersion using 250 ml capacity beakers containing 100 ml prepared solution at 30°C to 70°C which was maintained in a thermostatic water bath using method employed by [10]. The mild steel coupons were weighed and dropped in different concentrations of HCl with the aid of acid resistance plastic clip at the required conditions. The coupons were retrieved at a certain time. After each exposure time, the mild steel coupons were removed, washed thoroughly to remove the corrosion product (Rust Stain) with emery paper, rinsed with distilled water and dried in acetone as previously described. The mild steel was re-weighed to determine the weight loss, in gram. The procedure was repeated with different concentration of inhibitors in the solution. The corrosion rates (g/cm^{2}h) in the absence and presence of the inhibitors were determined. The variations of factors used in the experiment are shown in Table 1 below. Weight loss was calculated by finding the difference between weight of each coupon before and after immersion as reported by [10].

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

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

$$CR = \frac{\Delta W}{At} \tag{2}$$

where ΔW is the weight loss (g) after exposure time t (h), A is the area of the specimen (cm^2), t is time of exposure in hours and CR is the corrosion rate at each exposure time. The corrosion rate obtained in the absence and presence of inhibitor were used to calculate inhibition efficiency (IE %) as in equation 3.

$$IE(\%) = \frac{CR_1 - CR_2}{CR_1} x\ 100 \tag{3}$$

where IE (%) is inhibition efficiency, CR_1 is the corrosion rate of mild steel in absence of inhibitors; CR_2 is the corrosion rate of mild steel coupons in the presence of inhibitors.

Table 1. Test Matrix for the Corrosion Rate Experiment

Parameters	**Conditions**
Time	2 hrs, 4 hrs, 6hrs, 8hrs and 10 hrs
Temperature	30°C, 40°C, 50°C, 60°C, 70°C
HClsolution concentration	1M, 1.5M, 2M, 2.5M, 3M
Inhibitor concentration	5%v/v, 10%v/v, 15%v/v, 20%v/v and 25%v/v

Design Expert software was used in this study to design the experiment and to optimize the inhibition conditions. The experimental design employed in this work is a two-level-four factor full factorial design, including 30 experiments. Concentration of the extract ($\%v/v$), exposure time ($hour$), temperature (oC), and concentration of the medium (M) were selected as independent factors for the optimization studyThe response chosen was inhibition efficiency, IE (%) obtained from corrosion inhibition of mild steel using *carica papaya* (pawpaw) and veroninia amigdanina (bitter) leave extracts. Six replications of centre points were used in order to predict a good estimation of errors and experiment were performed in a randomized order. The actual and coded levels of each factor are shown in Table 2 and Table 3. The coded values were designated by -1 (minimum), 0 (centre), $+1$ (maximum), $-\alpha$ and $+\alpha$. Alpha is defined as a distance from the centre point which can be either inside or outside the range.

Table 2. Range of Each Factor in Actual and Coded Form for Corrosion Inhibition of Mild Steel Using CaricaPapaya(pawpaw) and VeroniniaAmigdanina (bitter), Leaves Extracts in Acidic Medium

Factor	Units	Low level	High level	-α	+α	0 level
Conc. of Extract (A)	(mg/100ml)	10(-1)	20(+1)	5(-2)	25(+2)	15
Exposure time , (B)	Hours	4(-1)	8(+1)	2(-2)	10(+2)	6
Temperature (C)	°C	40(-1)	60(+1)	30(-2)	70(+2)	50
Conc. of media (D)	Mol/dm^3	1.5(-1)	2.5(+1)	1(-2)	3(+2)	2

Table 3. Experimental Design Matrix for Corrosion Inhibition of Mild Steel Using *Carica Papaya* (pawpaw) and *Veroninia Amigdanina (*bitter *)*Leaves Extracts in Acidic Medium

Run order	**Conc. of extract (%v/v) A**		**Exposure Time (Hours) B**		**Temperature (°C) C**		**Conc. of medium (mol/dm^3) D**		**IE of *carica papaya* (pawpaw) leaf extract (%)**	**IE of veroniniaamigdanina(bitter) leaf extract (%)**
	Coded	**Real**	**Coded**	**Real**	**Coded**	**Real**	**Coded**	**Real**		
1	-1	20	-1	4	-1	40	-1	1.5		
2	+1	20	-1	4	-1	40	-1	1.5		
3	-1	10	+1	8	-1	40	-1	1.5		
4	+1	20	+1	8	-1	40	-1	1.5		
5	-1	10	-1	4	+1	60	-1	1.5		
6	+1	20	-1	4	+1	60	-1	1.5		
7	-1	10	+1	8	+1	60	-1	1.5		
8	+1	20	+1	8	+1	60	-1	1.5		
9	-1	10	-1	4	-1	40	+1	2.5		
10	+1	20	-1	4	-1	40	+1	2.5		
11	-1	10	+1	8	-1	40	+1	2.5		
12	+1	20	+1	8	-1	40	+1	2.5		
13	-1	10	-1	4	+1	60	+1	2.5		
14	+1	20	-1	4	+1	60	+1	2.5		
15	-1	10	+1	8	+1	60	+1	2.5		
16	+1	20	+1	8	+1	60	+1	2.5		
17	-2	5	0	6	0	50	0	2		
18	+2	25	0	6	0	50	0	2		
19	0	15	-2	2	0	50	0	2		
20	0	15	+2	10	0	50	0	2		
21	0	15	0	6	-2	30	0	2		
22	0	15	0	6	+2	70	0	2		
23	0	15	0	6	0	50	-2	1		
24	0	15	0	6	0	50	+2	3		
25	0	15	0	6	0	50	0	2		
26	0	15	0	6	0	50	0	2		
27	0	15	0	6	0	50	0	2		
28	0	15	0	6	0	50	0	2		
29	0	15	0	6	0	50	0	2		
30	0	15	0	6	0	50	0	2		

4. Result and Discussion

4.1. Yield of Extract from the Leaves

30g of CaricaPapaya (pawpaw) leave extract and 42g of*veroninia amigdanina (bitter)* leave extract were obtained respectively from 60g each of the dried leaves *carica papaya* (pawpaw) and veroniniaamigdanina (bitter) leaves. These represent 50 and 70% yield respectively showing that all leaves have good quantity of extract which can serve as green inhibitors.

4.2. Phytochemical Analysis

Table 4: presents the photochemical constituents of ethanol extracts of *carica papaya* (pawpaw) and veroninia amigdanina(bitter) leave. The results indicate that saponin, tannin, terpenes, flavanoid, perpenes, glycoside and alkaloids were present while anthraquinone is absent in the leaves ethanol extracts. The chemical structures of most of these phytochemicals contained electron rich bond or hetero atoms that facilitated their electron donating ability; hence the inhibition of the corrosion of mild steel by ethanol extracts of these leaves may be attributed to the phytochemical constituents of the extracts. Similar results have been reported by other researchers for the inhibition of the corrosion of mild steel and aluminium by ethanol extract of some plants [11], [12].

Table 4. Phytochemical Constituents of Ethanol Extracts of Leaves

S/N	Phytochemicals	Pawpaw leave Extract (%w/w)	Better leave Extract (%w/w)
1	Saponins (%w/w)	5.86	6.71
2	Terpenes (%w/w)	7.22	9.69
3	Tannins (mg/100g)	7.22	9.69
4	Flavonoid (%w/w)	2.26	5.83
5	Phlobatannins (%w/w)	-	-
6	Anthraquinones (%w/w)	-	-
7	Glycoside (mg/100g)	0.13	1.58
8	Alkaloids (%w/w)	3.27	6.33

4.3. Metal Composition of Mild Steel

Mild steel sheet was analyzed using*Atomic absorption spectroscopy (*AAS) to determine the composition (wt %).It could be observed from Table 5 that iron is the dominant metal that make up the mild steel.

Table 5. Metal Composition of Mild steel Using *Atomic Absorption Spectroscopy (AAS)*

Metals	Composition (Wt %)
Magnesium	0.90
Phosphorus	0.70
Carbon	0.50
Silicon	0.04
Iron	97.86

4.4. Effect of Time on Corrosion Rate of Mild Steel

Fig. 1 depicts the effect of time on corrosion rate of mild steel in presence and absence of inhibitors. From the figure, it can be observed that corrosion rate was rapid and progressive in absence of inhibitor. The increase in corrosion rate in the medium may be adduced to the loss of electrons from the coupon. It was also observed that the metal corroded more in uninhibited acid solution with progressive colour change from transparent solution to brownish solution due to the release of various forms of corrosion products suspected to be Fe_2O_3, Fe_2O_4 and $Fe(OH)_3$ that were formed on the surface of the corroded surface. This may be as a result of corrosive nature of acid.

The corrosion of metal coupons smeared with inhibitors *carica papaya* (pawpaw) leaf extract and veroninia amigdanina (bitter) leaves extracts initially increased and then decreased. This may be as a result of the adsorptions of the inhibitors which were gradually increasing initially and on long exposure of the metal in the inhibitor solution which thereby inhibited the corrosion. The corrosion rate of mild steel in uninhibited medium was higher than when in an inhibited medium.

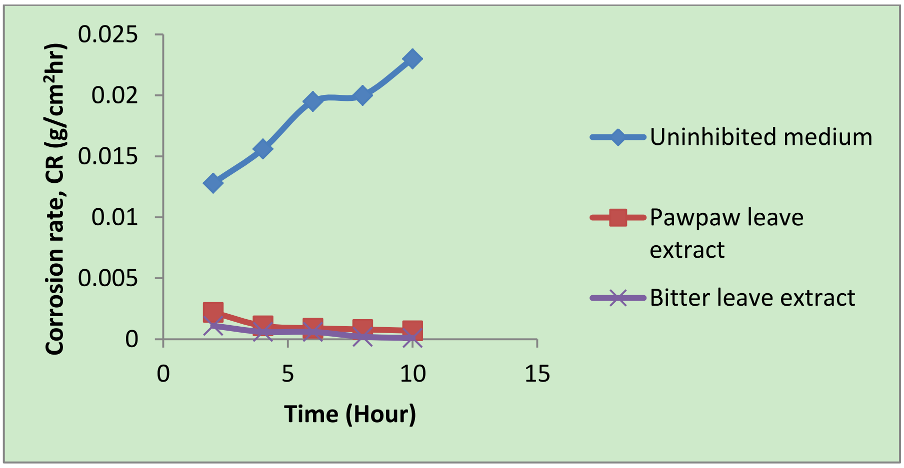

Fig. 1. Effect of time on corrosion rate of mild steel in the presence and absence of different inhibitors.

4.5. Effect of Concentration of the Medium on Corrosion Rate

Fig. 2 presents effect of concentration of medium on corrosion rate of mild steel in the presence and absence of different inhibitors. From the figure, it's obvious that corrosion rate of uninhibited medium continually increases as concentration of the medium increased. While the corrosion rate of inhibited medium slightly increased and later decreased. This could be that the quantity of inhibitor in the solution was enough to counter the corrosive nature of high concentrated acidic medium.

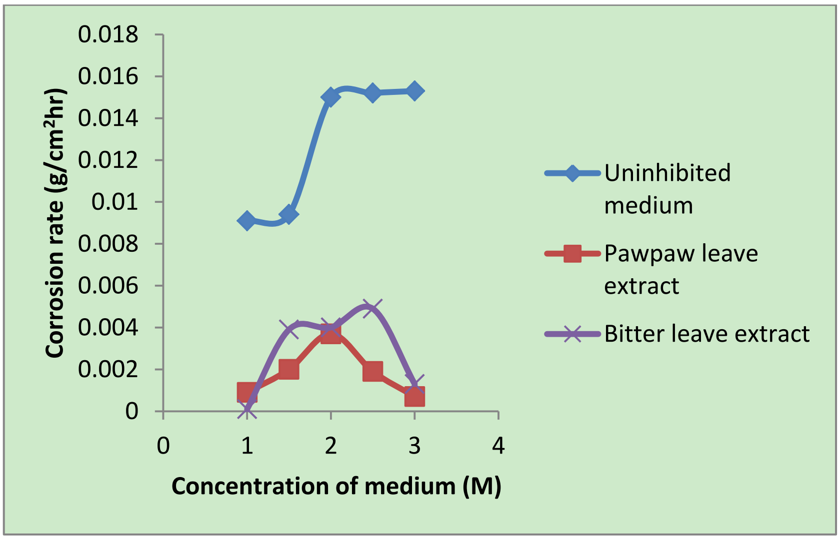

Fig. 2. Effect of concentration of medium on corrosion rate of mild steel in the presence and absence of different inhibitors.

4.6. Effect of Concentration of Inhibitor on Corrosion Rate

The variation of corrosion rate of mild steel in acidic medium in the absence and presence of various concentrations of ethanol extracts of *carica papaya* (pawpaw) leaf extract andveroniniaamigdanina (bitter)leaves were studied at certain conditions as shown in Figure 3. It was observed from the figure that the corrosion rate of mild steel in the medium decreased with increase in the concentration of the extract. This could be that as the concentration of the extract increase, there is was increase in the number of adsorption of the extract constituents on the surface of the mild steel which makes a barrier for mass transfer and prevents further corrosion. This result is in consonance with the findings [13].

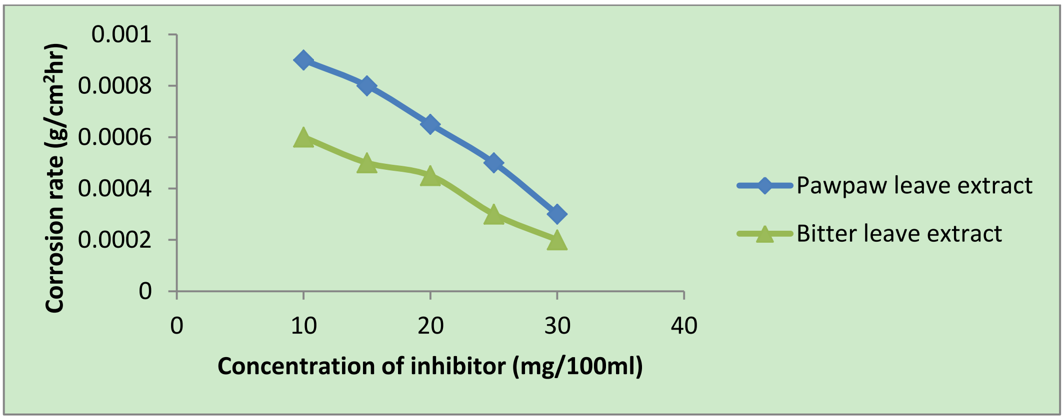

Fig. 3. Effect of concentration of inhibitor on corrosion rate of mild steel in the presence and absence of different inhibitors.

4.7. Effect of Temperature on Corrosion Rate

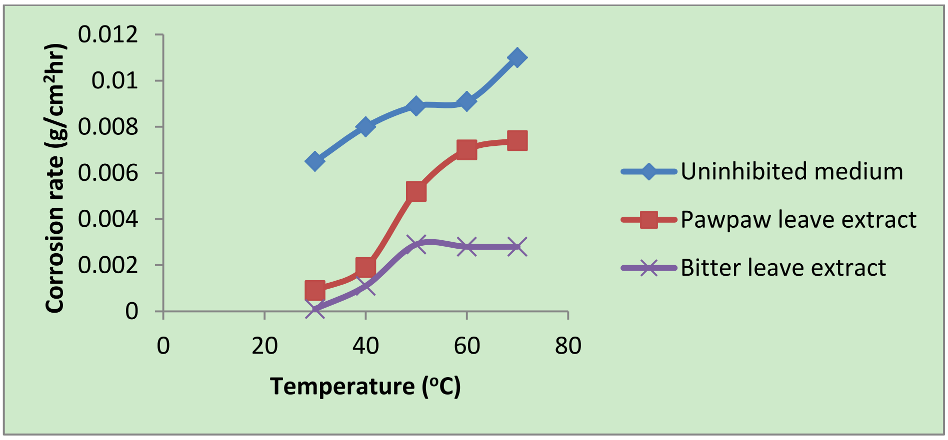

Fig. 4. Effect of temperature on corrosion rate of mild steel in presence and absence of different inhibitors.

The effect of temperature on the corrosion rate of mild steel in free acid and in the presence of different concentrations of the inhibitor (plant extract) was studied in the temperature range of 30°Cto 70°C as shown in Fig. 4. It was found that the rates of corrosion of mild steel in free acid solution and in the presence of inhibitor increased with increase in temperature. This is expected because as temperature increase, the rate of corrosion of mild steel also increased as a result of increase in the average kinetic energy of the reacting molecules. However, the corrosion rate decreased more for inhibited acid solution than the uninhibited acid solution. The decrease in the corrosion rate for the inhibited acid solution compared to the uninhibited was as a result of the mitigating effect of the plant extract on the corrosion rate of the mild steel.

4.8. Optimal Conditions for Corrosion Inhibition of Carica Papaya (Pawpaw) Leaf Extract

The inhibitive effect of pawpaw leave extract was optimized using CCD of RSM and the following optimal conditions were obtained: concentration of inhibitor is 16.37mg/100ml, time is 6.55hr, temperature is 48^{o}C and acid concentration is 2.2M. The inhibition process under the obtained optimum operating conditions was carried out in order to evaluate the precision of the model; the experimental value and predicted values are shown in Table 6. Comparing the experimental and predicted results, it can be seen that the error between the experimental and predicted is o.5%, therefore it can be concluded that the generated model has sufficient accurancy to predict the corrosion inhibition efficiency.

Table 6. Results of the Model Validation (Xxperiment 1 Indicates the Optimum Inhibition Process Conditions and Corrosion Inhibition Efficiency of Pawpaw Leave Extract in Acidic Medium)

Experiment	Concentration.of inhibitor/extract (mg/100ml) A	Exposure time (Hour) B	Temperature (^{o}C) C	Conc.of medium(mol/dm^{3}) D	Experimental inhibition efficiency (%)	Predicted inhibition efficiency (%)
1	16.37	6.55	48	2.2	87.9	88.4

4.9. Optimal Conditions for Corrosion Inhibition of Veroniniaamigdanina(Better) Leave Extract

The inhibitive effect of veroniniaamigdanina (better) leave extract was optimized using CCD of RSM and the following optimal conditions were acquired: concentration of inhibitor is 18.94mg/100ml, time is 5.50hr, temperature is 48^{o}C and acid concentration is 1.74M. The inhibition process under the acquired optimum operating conditions was carried out in order to evaluate the precision of the quadratic model; the experimental value and predicted values are shown in Table 7 Comparing the experimental and predicted results, it can be seen that the error between the experimental and predicted is 1.3%, therefore it can be concluded that the generated model has sufficient accurancy to predict the corrosion inhibition efficiency.

Table 7. Results of the Model Validation (Experiment 1 Indicates the Optimum Inhibition Process Conditions and Corrosion Inhibition Efficiency of VeroniniaAmigdanina (better) Leave in Acidic Medium)

Experiment	Concentration of inhibitor/extract (mg/100ml) A	Exposure time (Hour) B	Temperature (^{o}C) C	Conc. of medium (mol/dm^{3}) D	Experimental inhibition efficiency (%)	Predicted inhibition efficiency (%)
1	18.94	5.50	48	1.74	95.1	96.4

5. Conclusion

From the study it can be concluded that ethanol extract of *carica papaya* (pawpaw) and of veroninia amigdanina (bitter) leaves are adsorption inhibitors for the corrosion of mild steel in acidic medium. The inhibition efficiency of ethanol extracts of the leaves was due to the phytochemical constituents of the extracts. The optimal conditions are: concentration of inhibitor 16.37mg/100ml, time, 6.55h, and temperature 48°C, concentration of medium 2.2M in pawpaw leave extract inhibited acidic medium; concentration of inhibitor 18.96mg/100ml, time, 5.50h, temperature 48°C, concentration of medium 1.74M inveroniniaamigdanina (bitter) leave inhibited medium. The optimal conditions obtained gave inhibition efficiencies of 88.4% and 96.4% % in pawpaw leave extract andveroninia amigdanina (bitter) leave inhibited acid media respectively. This indicates that leaves extracts can inhibit corrosion of mild steel in acidic medium.

Conflict of Interest

The authors have no conflict of interest.

Author Contributions

The first author was involved deply in the laboratory analysis, the second author was the content reader of the work, while the third author was deply involved in research analysis of the work. The three authors were involved in the final approval version before sending the work for publication..

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