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Evaluation of the Corrosion Inhibitive Properties of Three Different Leave Extracts on Mild Steel Iron in Sulphuric Acid Solution

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Authors' contributions

This work was carried out in collaboration among all authors. Authors GNC and COA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors CEO and ISI managed the analyses of the study. Authors CEO and ISI managed the literature searches. All authors read and approved the final manuscript.

Article Information

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ABSTRACT

The inhibitive ability of pawpaw, neem and curry leaf extracts on corrosion of mild steel in sulfuric acid solution were investigated in this work. The extracts obtained from their respective leaves, were characterized to determine their phytochemical constituents as well as functional groups present using Fourier Transform Infrared Spectroscopy (FTIR) technique. Weight loss techniques was employed to evaluate the corrosion inhibition efficiency of the leaf extracts. The Scanning Electron Microscopy (SEM) was used to study the morphology of the mild steel before and after corrosion experiments. The process factors studied was exposure time, concentration of leaf extracts (inhibitor) and temperature. The results revealed that the phytochemical constituents of the leaves are capable of inhibiting corrosion due to high concentration of tannins which is

responsible for the corrosion inhibition of mild steel. The rate of corrosion decreased with increase in the concentration of the extracts while the inhibition efficiency increased with increase in the concentration of the extracts. Scanning Electron Microscopy (SEM) revealed that the corroded mild steel in the presence of the extracts had smoother surfaces than corroded mild steel in the absence of the extracts. Also, neem leaf extract demonstrated higher potential as corrosion inhibitor than pawpaw and curry leaf extracts.

Keywords: Inhibitor; leaf extracts; curry leaf; pawpaw leaf; neem leaf; mild steel iron.

1. INTRODUCTION

In ancient history, seven metals were known to antiquity; copper, gold, iron, lead, mercury, silver, and tin. Of the ancient metals, copper may have been the most important and mercury the least. These seven metals either occur naturally in their metallic state or are easily extracted from their ores [1,2]. Metallic materials are still the most widely used group of materials particularly in mechanical, transportation and process industry. However, the usefulness of metals and alloys is constrained by one common problem known as corrosion [3].

Corrosion is a natural process, which converts a refined metal to a more stable form, such as its oxide, hydroxide or sulfide. It is the gradual destruction of materials (usually metals) by chemical reaction with their environment [4,5]. Corrosion is chemically induced on a metal which leads to deterioration of its properties. The failure of mild steel as construction materials can be dangerous to both human health and economy. As pointed out by Sheatty et al. [6]; Uwah et al. [7], mineral acids such hydrogen chloride and sulfuric acids causes harm on the surface on exposure to it by corroding majority of most alloys and metals. This causes deterioration and considerable cost and this action has lead corrosion scientists and engineers to a continuous research for a more economic viable and environmentally friendly corrosion inhibitors. Prior research by Uwah et al. [7] had already suggested leaf extracts as preferred corrosion inhibitors considering that the leaves are accessible, the extracts are eco-friendly and most importantly, they are renewable.

The corrosion inhibitive properties of these plant leaf extracts have been attributed to the presence of tannin in their chemical constituents [8,9,10]. Also associated with the presence of tannin in the extracts is the bitter taste in the bark and or leaves of the plants. The use of inhibitors is one of the best methods of preventing metals against corrosion. Most of the corrosion inhibitors are synthetic chemicals which are expensive and very hazardous to environments. Therefore, it is desirable to source for environmentally friendly inhibitors. Plants represent a class of interesting source of compounds currently being exported for use in metal corrosion protection in the systems.

The use of phytochemicals as corrosion inhibitors can be traced when tannins and their derivatives were used to protect corrosion of steel, iron and other tools [11]. A lot of extracts were from plant leaves for example Hibiscus subdariffamytruscommunis [12], Artemisa [13], *Vernonia amygdalina* [14], *Piper guinesis* [15], *Annona Squamosal* [16], Black pepper [17], *Moringa oliferen* [18], *Azardirachita indica* [19], *Jatrophacorcas* [20], *citrollus colocynthis* [21], green tea [22], *Pterocarpossoyarci* [23].

The use of naturally occurring plant extracts as inhibitors is particularly interesting because it poses little or no threat to the environment [24,25]. Therefore, this investigation aims at studying the corrosion inhibitive properties of three leaf extracts from *Murraya Koenigii* (curry leaf), *Azadirachita indica* (Neem leaf) and *Carica papaya* (pawpaw leaf) on mild steel iron.

2. MATERIALS AND METHODS

The main raw materials used in this work include: Curry leaf, pawpaw leaf and neem leaf collected from Abakpa, Enugu State, Nigeria. Others are metal coupon (mild steel) purchased from Iron steel market, Onitsha, Anambra State, Nigeria. Analytical grade chemicals such as H_2SO_4 , acetic anhydride, HCI and NaOH etc purchased from Zanzi chemicals and allied products limited, Onitsha Main Market, Anambra State, Nigeria.The purity levels of the reagents were above 99% and the entire reagents were used without further purification.

2.1 Extraction of Corrosion Inhibitors from Pawpaw, Curry and Neem Leaves

The leaf samples of pawpaw, neem and curry were first, washed separately with distilled water

to remove earthly impurities and then dried under the hot sun for three days to ensure complete removal of water. The leaves were later transformed into powdery form using mortar and pestle and sieved using a 700µm sieve. 50 g of each of the leaf sample was transferred into a Soxhlet extractor and extraction was carried out at 65°C by pouring 200 ml of ethanol in the extractor and allowing the mixture to stay at least 24hrs. Thereafter the solutions were filtered in vacuum filter and the filtrate was heated in rotary evaporator setup to expel the ethanol at 78°C for 20 min.

2.2 Characterization Extracts from Pawpaw, Curry and Neem Leaves

The phytochemical analyses, which include both qualitative and quantitative analyses of the ethanol extracts of each leaf to determine the presence of alkaloids, flavonoids, steroids, phenols, saponins, tannins and glycosides constituents were done using the method described by Raja and Sethuraman [26]. The experiments were carried out at Projects Development Institute, PRODA, Emene Enugu Nigeria.

2.3 Fourier Transform Infra-Red (Ftir) Analysis

The method described by Ayeni et al. [27] was adopted to carry out FTIR analysis of the extracts using BUCK model 500 M infra-red spectrophotometer. The sample was prepared using KBr and the analysis was done by scanning the sample through a wave number range of 500 to 4000 cm⁻¹.

2.4 Preparation of Test Solutions

1M H_2SO_4 was diluted by addition of distilled water. The inhibitors were accurately weighed and dissolved in prepared solution of H_2SO_4 to obtain different inhibitors concentrations (0.1 to 0.5 g/ml). The test solutions were prepared in different containers to carry out the corrosion experiment.

2.5 Preparation of Metal

The mild steel was prepared for corrosion experiment by adopting the method employed by Awe et al. [28]. The mild steel specimens were mechanically cut into dimension of $3.0 \times 3.0 \times 1.5$ cm (with a surface area of 9.0 cm^2) and washed

in Sodium Hydroxide. Prior to all, the mild steel coupons were mechanically polished with series of emery sand paper from 400 to 1200 grades to sufficiently remove any mill scale on the mild steel. The specimen was washed thoroughly with distilled water, degreased with absolute ethanol, dipped into acetone and dried in air. The dried specimens were stored in desiccators prior to the experiment.

The mild steel was analyzed to determine the metallic compositions using Atomic Absorption Spectrometer (AAS). The mild steel was reduced to small grains using a bench grinder (BG-6) and digested with 2M of H_2SO_4 to leach out different metallic compositions present in the mild steel. The liquid form was analyzed at the wavelength of the metal using AAS.

2.6 Corrosion Inhibition Experiment and Weight Loss (WI) Measurement

The method described by Uwah et al. [7] was adopted for this experiment. The experiments were conducted under total immersion of the metallic steel using 250 ml capacity beakers containing 200 ml prepared solution at 30°C to 70°C which was maintained in a thermostatic water bath. Samples of mild steel were measured and inserted into different concentrations of H₂SO₄ with the help of acid resistance plastic clip. The mild steel samples were retrieved at a given time interval. At the end of the exposure time, the mild steels were removed, washed thoroughly to remove the corrosion stain product with emery paper and then rinsed with distilled water and dried in acetone. The mild steel was re-weighed to determine the weight loss, in gram by the difference of mild steel weight before and after immersion. The corrosion rates (mm/yr) in the absence and presence of the inhibitors were determined.

Weight loss was calculated by finding the difference between weight of each coupon before and after immersion as reported by Uwah et al. [7].

$$\Delta W = W_o - W_f \tag{1}$$

 W_{o} is the initial weight of the metal (g); W_{f} is the final weight of the metal (g). While the corrosion rate (mm/yr) in absence and presence of inhibitors were calculated using equation 2

$$CR = \frac{\Delta Wk}{\rho At}$$
(2)

Where ΔW is the weight loss (g) after exposure time t (days), A is the area of the specimen (cm^2) , t is time of exposure in days, k is constant (8.75 x 10⁴), ρ is the metal density 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 presence of concentration of inhibitors.

The extent of adsorption of the extract on the surface of mild steel can be calculated using equation (4)

$$\theta = 1 - \left(\frac{W_1}{W_2}\right) \tag{4}$$

Where W₁= Mild steel loss in weight in the presence of inhibitor

 W_2 = Mild steel loss in weight in the absence of inhibitor

 θ = Surface coverage

Alternatively, inhibition efficiency can be evaluated using equation (5)

$$I.E = 1 - \left(\frac{W_1}{W_2}\right) \times 100$$
 (5)

By varying the concentration of extract (0.1, 0.2, 0.3, 0.4 and 0.5 g/ml), exposure time (1, 2, 3, 4 and 5days) and temperature $(30, 40, 50, 60 \text{ and } 70^{\circ}\text{C})$ the experiments was conducted.

2.7 Analysis of Metal Steel Iron Surface

The surface morphology of the mild steel metal was studied using Carl Zeiss Sigma Field Emission Scanning Electron Microscope and the images at $10\mu m$ of the mild steel surfaces at uninhibited (0%) and inhibited at optimum conditions in acidic medium.

3. RESULTS AND DISCUSSION

3.1 Phytochemical Analysis of the Raw Materials

The photochemical constituents of the leaf extracts of pawpaw, curry and neem were present in Table 1. It was observed that the leave extracts have a reasonable percentage weight of tannins; pawpaw (8.26%), Neem (8.30%) and curry (7.87%). Tannins is responsible for the corrosion inhibition of metals as opined by Ashassi and Seifzadeh [29]; Awe et al. [28]; Cang et al. [30] and hence demonstrated that pawpaw, curry and neem extracts are potential corrosion inhibitor. Tannins and flavonoids are poly phenolic compounds which contain hydroxyl group bonded to aromatic carboxyl group [7]. In solution they can undergo oxidative reaction to produce a compound derived from phenols by the loss of hydrogen ion (phenonate anion) which usually act as ligands towards metal cations [27]. Prior research by Uwah et al. [7] reported that tannin acts as physical barrier to diffusion of ions in anodic and cathodic reactions of metal ions in solution thereby resulting in decrease of corrosion rate.

3.2 FTIR Analysis of the Leaves Extracts

The Infrared spectrum (IR) of ethanol extracts of pawpaw, curry and neem leaves are represented as shown in Table 2. It was observed that, O-H stretch was present in the three leaves extracts at absorption peak between 32600-3310cm⁻¹ indicating the presence of hydroxy group, carboxylic acid and alcohol. However, C-Cl stretch was observed in curry and neem leaves extracts at absorption peak of 2120.9 and 2117.4 cm⁻¹ indicating the presence of alkane. Furthermore, C-H stretch was found out of plane at absorption peak 1077.2 cm⁻¹ in pawpaw leaf extract indicating the presence aromatic compounds. Similarly, C=O stretch observed at absorption peak between 1610 to 1650cm⁻¹ is a characteristic of conjugated ketone. These show presence heteroatoms of the in the phytochemical constituents of the leaf's extracts. The results are in concurrence with the results of the leaf extracts reported by Loto et al. [22].

3.3 Metal Composition of Mild Steel

Mild steel sheet was analyzed using AAS to determine the composition (wt %) Mn (0.31.), P (0.18), C (0.14), Si (0.06) and the rest were Fe. It could be observed that the metal highly makes up the mild steel is Iron.

3.4 Surface Morphological Studies of the Inhibition on the Surface of Mild Steel Iron

The surface appearance also known as the surface morphology of the mild steel iron, uninhibited mild steel iron and inhibited mild steel iron in acid medium after 120 hours exposure

time using Sigma Field Emission Scanning Electron Microscope (SEM) and the images at 200µm using pawpaw, neem and curry leaf extracts were presented as shown in Plate 1-4. Structural discrepancy of the mild steel was clearly observed in uninhibited and inhibited coupon with different leaf extracts with a rapidly oxidized surface in the uninhibited in acidic medium. However, it was also observed that the mild steel coupon immersed in inhibited solution of the extracts possess a smooth surface while the metal surface immersed in blank acid solutions is rough covered with corrosion products and appeared like full of pits, cracks and cavities. These results were in agreement with the report by Leelavathi and Rajalakshmi [31], which suggested that mild steel metals in acid medium without inhibitors are vulnerable to acid attack which results in corrosion. Furthermore, Plate. 2 revealed that mild steel

Table 1. Phytochemical	constituents of ethanol extracts of the leaves
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Phytochemicals	Pawpaw extract (%w/w)	Neem extract (%w/w)	Curry extract (%w/w)
Alkaloids	-	-	-
Tannins	8.26	8.30	7.87
Flavonoid	8.20	7.44	8.01
Phenols	11.51	12.2	13.01
Glycoside	8.23	7.36	8.37
Saponins	-	-	-
Steroids	-	-	-

Table 2. FTIR of ethanol extracts of the leaves

Assignment	Pawpaw leave extract (PLE)	Curry leave extract(CLE)	Neem leave extract(NLE)
Assignment	Frequency (cm ⁻¹)	Frequency (cm ⁻¹)	Frequency (cm ⁻¹)
O-H stretch	3268.9	3306.1	3291.2
Hydroxyl group, H-bonded, O- H stretch	3268.9	3306.1	3291.2
C-CI stretch Alkane group	-	2120.9	2117.4
C=O Conjugated ketone, open- chain acid anhydride	1638.3	1636.3	1636.3
Aromatic C-H in plane bend,	1077.2		-

Table 3. AAS standard composition of mild steel

Samples	Mn	Р	C	Si	Fe
Wt. (%)	0.31	0.18	0.14	0.06	99.31

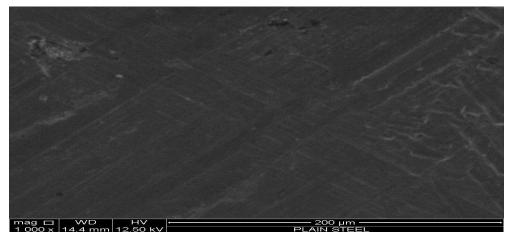


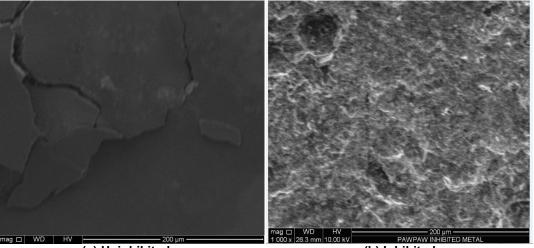
Plate 1. SEM images of mild steel coupon

inserted in neem leaf extracts (NLE) has a better smooth surface after exposure time of 120 mins. This could be attributed the high percentage of tannins in neem leaf extracts. Similarly, Plate 1-4 showed that the phytochemical constituents present in the PLE, NLE and CLE formed a protective layer of the mild steel specimen and thereby reducing the corrosion rate.

3.5 Effect of Concentration of the Leave Extracts on Corrosion Rate and Inhibition Efficiency

Figs. 1 and 2 depict the effect of the concentration of leave extracts on corrosion rate and inhibition efficiency. From Figs. 1 and 2, it

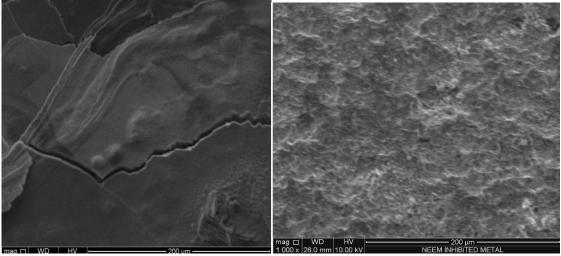
can be seen that the concentration of the leaf extracts did affect the rate of corrosion of the metal and the inhibition efficiency. First, the rate of corrosion decreased with the increase in the concentration of the extracts thereby increasing the inhibition efficiency. This could be as a result of the displacement of water molecule on the surface of mild steel metal by adsorption of more of the leaf extracts on the neutral surface through the process of chemisorption as suggested by Kesivan et al. [32]; Ismail et al. [33]. Meanwhile, as shown in Fig. 2, the inhibition efficiency increased above 80% for the three inhibitors and remained steady after 0.4 mg/ml of the inhibitor's concentration. It can be concluded that the optimum inhibition efficiency of the corrosion rate



(a) Uninhibited

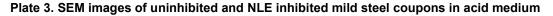
(b) Inhibited





(a) Uninhibited

(b) Inhibited



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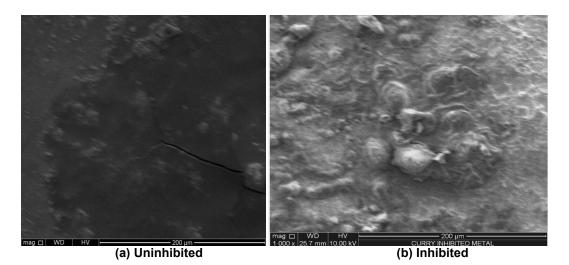


Plate 4. SEM images of uninhibited and CLE inhibited mild steel coupons in acid medium

of mild steel by the three leaf extracts occurred at a concentration of 0.4 mg/ml of inhibitor. At this concentration, the percentage inhibition of pawpaw is 82.3%, neem is 85.1% while curry is 82.01%. The higher percentage inhibition by neem leaf extracts may not be unconnected to the high tannins content as shown in Table 1. The results are in concurrence with the report by Loto et al. [22] that reported 85.9% inhibition efficiency on mild steel by neem leaf extract.

3.6 Effect of Temperature on Corrosion Rate and Inhibition Efficiency

At the same reaction conditions, the effect of temperature on the corrosion rate and inhibition efficiency were studied and results presented as shown in Figs. 3 and 4. It can be seen from Fig.

3 that the rate of corrosion (CR) increases with increase in temperature while Fig. 4 showed that increase in temperature also resulted in increase in inhibition efficiency. The increase in inhibition rate and efficiency at increase in temperature to a certain range could be attributed to the dissolution of the mild steel and rapid adsorption of the inhibitor within the surface as opined by Martinez and Stern [34]. Meanwhile, at a temperature above 65°C, the inhibition efficiency of the three leaf extracts decreased significantly as evident in Fig. 4. This could be due to partial desorption of the inhibitor from the metal surface at higher temperature as reported by Olasehinde et al. [35]. Furthermore, it can be attributed to the inactivation (denature) of the polyphenolic component of the extracts which contributes to the inhibitive action of the extracts.

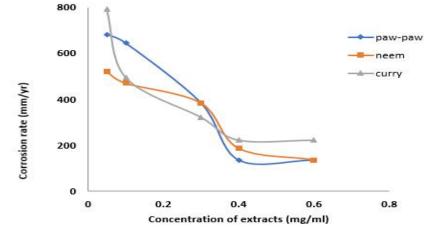
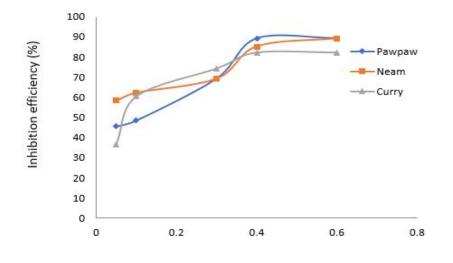


Fig. 1. Effect of concentration of the leave extracts on corrosion rate. Conditions: Time = 2days, acid conc = 1M, temperature = 70°C

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Concentration of extract(mg/ml)

Fig. 2. Effect of concentration of the leave extracts on inhibition efficiency. Conditions: Time = 2days, acid conc = 1M, temperature = 70°C

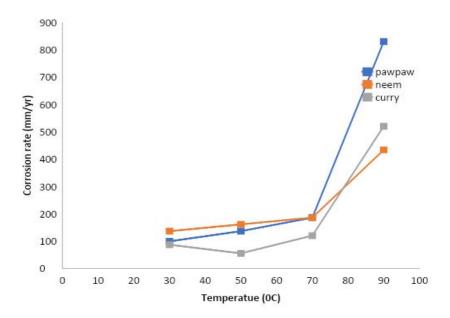
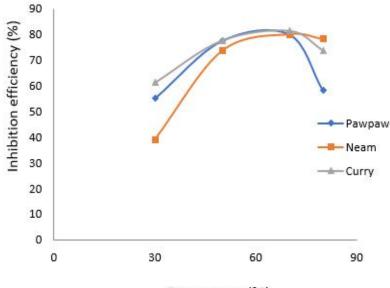


Fig. 3. Effect of temperature on corrosion rate. Conditions: Concentration of inhibitor = 0.4 mg/ml, acid conc = 1M, time = 2 days

3.7 Effect of Time on Corrosion Rate and Inhibition Efficiency

Figs. 5 and 6 depict the effect of time on corrosion rate and inhibition efficiency. The exposure time interval of 1 to 6 days was considered. It was revealed that corrosion rate and inhibition efficiency of the leaf extracts on

mild steel increases with increase in the period of contact. This could be as a result that the longer exposure of the metal to the inhibitor, the more it adsorbs inhibitor on its surface. The inhibition efficiency decreased after 2days and became steady for pawpaw and neem leaves extracts. This could be attributed to the fact that the metal surface became saturated of the polyphenol constituent of the extracts after some time. Furthermore, after exposure time of 48 hours (2 days) as evident in Fig. 6, the inhibition efficiency of neem leaf extract was found to be 69.3%, pawpaw leaf extract 68.4% while curry leaf extract was 64.6%. This further buttressed the earlier claim by Loto et al. [22] that extracts with high tannins content exhibits high inhibitive properties.



Temperature(°C)

Fig. 4. Effect of temperature on inhibition efficiency. Conditions: Concentration of inhibitor = 0.4 mg/ml, acid conc = 1M, time = 2 days

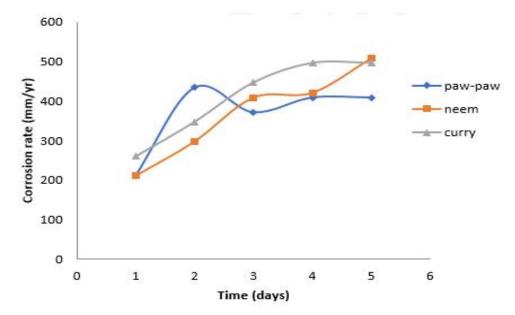


Fig. 5. Effect of time on corrosion rate. Conditions: Concentration of inhibitor = 0.4 mg/ml, acid conc = 1M, temperature = 70°C

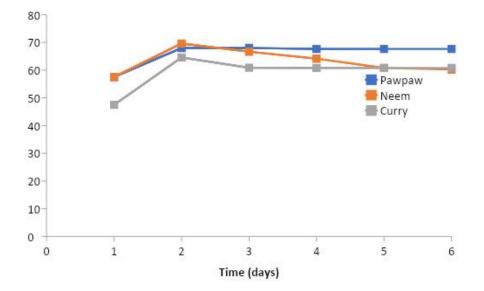


Fig. 6. Effect of time on inhibition efficiency. Conditions: Concentration of inhibitor = 0.4mg/ml, acid conc = 1M, temperature = 70°C

4. CONCLUSION

Physiochemical characterization of aqueous extracts of the leaves revealed high contents of tannin which were responsible for the corrosion inhibition of mild steel. The rate of corrosion decreases with increase in the concentration of the extracts while the inhibition efficiency increases with increase in the concentration of the extracts. On the average, neem leaf extract has higher constituents than pawpaw and curry leaf extracts. The Fourier Infrared Spectra of pawpaw, curry and neem leaf extracts revealed presence of heteroatoms the in the phytochemical constituents which make them good corrosion inhibitors. Loss in weight experiment revealed that neem leaf extract showed greater decrease in weight loss than pawpaw and curry leaf extracts. This means that neem leaf extract is a better corrosion inhibitor than pawpaw leaf and curry extract.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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