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.

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

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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 guinessis [15], Annona Squamosal [16], Black pepper [17], Moringa oliferen [18], Azadirachita indica [19], Jatrophaorcas [20], citrollus coloycynthia [21], green tea [22], Pterocappossoyari [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 H₂SO₄, acetic anhydride, HCl 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 H₂SO₄ was diluted by addition of distilled
water. The inhibitors were accurately weighed
and dissolved in prepared solution of H₂SO₄ 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 x 3.0 x 1.5
cm (with a surface area of 9.0 cm²) 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 H₂SO₄ 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 (Wl) 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-weighted 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].

\[ \Delta W = W_o - W_f \]  \hspace{1cm} (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

\[ CR = \frac{\Delta W/k}{\rho A t} \]  \hspace{1cm} (2)
Where $\Delta W$ 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 ($8.75 \times 10^4$), $\rho$ 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.

$$IE(\%) = \frac{CR_1 - CR_2}{CR_1} \times 100$$

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)

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

Where $W_1$= Mild steel loss in weight in the presence of inhibitor
$W_2$ = Mild steel loss in weight in the absence of inhibitor
$\theta$ = Surface coverage

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

$$IE = 1 - \left( \frac{W_1}{W_2} \right) \times 100$$

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 and 70°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 10μm 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 leaf 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 Leave 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 the presence of heteroatoms 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

| 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) |
|--------------------------------|----------------------------|---------------------------|--------------------------|
| O-H stretch                    | Frequency (cm⁻¹)           | Frequency (cm⁻¹)          | Frequency (cm⁻¹)         |
| Hydroxyl group, H-bonded, O-H | 3268.9                     | 3306.1                    | 3291.2                   |
| C-Cl 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   | P    | 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
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
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 2 days 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.

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 the presence of heteroatoms 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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