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COMPARATIVE ANALYSIS OF CORROSION INHIBITION OF MILD STEEL USING PAWPAW AND NEEM LEAVES EXTRACTS IN SULPHURIC ACID MEDIUM

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Abstract: **In this research, the corrosion inhibition of mild steel using pawpaw and neem leaves extracts in sulphuric acid medium was studied, and the inhibitive properties of the two extracts were compared. Standard characterization and Fourier Transform Infrared Spectroscopy (FTIR) techniques were used to determine phytochemical constituents and the functional groups present in the extracts respectively. The corrosion of the mild steel was studied using weight loss method, to determine the loss in weight of the mild steel when immersed in the corroding medium with and without the leaves extracts respectively. Mild steel weight loss is a measure of the extent of corrosion because the higher the weight loss the higher the rate of corrosion and vice versa. Generally, the weight loss decreased in the presence of the extracts; which indicated that the extracts were effective corrosion inhibitors. The Scanning Electron Microscopy carried out to determine the morphology of the uncorroded, corroded mild steel with and without the extracts revealed that the surface of the uncorroded mild steel has the smoothest face, while the surface of the corroded mild steel with extracts is comparatively smoother than the surface of the corroded mild steel without extracts; showing that the extracts have reduced corrosion. Comparatively, mild steel corroded in the presence of neem leaf extract has smoother surface and lower weight loss; which indicated that neem leaf extract is a better corrosion inhibitor than pawpaw leaf extract.**

Keywords: **Mild steel, Corrosion Inhibition, Sulphuric acid, Pawpaw Leaf, Neem Leaf.**

1. INTRODUCTION

Mild steel is a sort of carbon steel with a low quantity of carbon; implying it could be referred to as “low carbon steel.” The implication of the above statement is that on average, mild steel is more ductile, weldable and machinable than high carbon. Mild steels are desirable and a major construction materials, which is expansively used in chemical and chemical related industries, because they are very affordable, machinable and weldable (Sheatty et. al., 2006). Corrosion is an electrochemical phenomenon by which metallic structures are eroded slowly through anodic dissolution (Ajani et al, 2014; Asipita et al., 2014). Corrosion is an inevitable process in the industry due to the chemical nature of mild steels and because mineral acids used in various processes such as production of fertilizers, drugs, detergents, chemical cleaning, oil well acidification and acid pickling cause metal corrosion (Bentiss *et al*., 2006; Khaled and Hackerman, 2003). Corrosion is chemically induced on a metal which leads to deterioration of its properties and uses. According to Fontana (2005), sulfuric acid corrodes majority of most alloys and metals. Due to the corrosive effect of this acid on metals which causes deterioration and considerable cost, corrosion scientist and engineers are in a continuous search for a more resourceful and environmental friendly corrosion inhibitor. Uwah et al. (2013) had already opined that leaves extracts are preferred corrosion inhibitors because they are inexpensive, eco-friendly, readily available and renewable. This present study investigated the corrosion inhibition of mild steel in *Carica papaya* (pawpaw leaf) and *Azadirachitaindica* (Neem leaf) inhibitive media.

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2. MATERIALSAND METHODS

2.1 MATERIALS:

Pawpaw and Neem leaves were collected from Emene, Enugu State, Nigeria. The metal coupon (mild steel) was purchased from Coal Camp Market, Enugu State, Nigeria. The chemicals used such as H_2SO_4, acetone, HCl, distilled water, NaOH, absolute ethanol were purchased from Gerald Chemicals Ltd Ogbete Main Market, Enugu, Enugu State, Nigeria and they were of analytical grade. The major equipments used are FTIR spectrophotometer, SEM, weighing balance, beakers, conical flask, mechanical blender, thermometer, sohxlet extractor, test tube, sieve, electric oven, heating mantle and water bath.

2.2 METHODS:

2.2.1 Preparation of Pawpaw and Neem leaves extracts respectively

Fresh leaves of each were washed separately with distilled water to remove dust and other impurities. The washed leaves were oven dried at 105ºC to ensure complete removal of water. The dried leaves were crushed into powder and sieved using a 600$_{\mu}$m sieve size to get very fine particles; leaving behind the coarse ones. 100g of each ground sample was loaded into the thimble of a soxhlet extractor and extraction was carried out at 60^{o}C by pouring 300ml of absolute ethanol in the sohxlet extractor and allowing the mixture to stay at least 24hrs. The extract formed was filtered off to recover most of the ethanol. After this, the extract was heated in a water bath at 60^{o}C until most of the ethanol evaporated. .

2.2 Characterization of the Leaves Extracts

Quantitative analysis of the extracts was done to determine the presence and quantity of the phytochemical constituents, which include alkaloids, saponins, tannins, flavonoids, etc, using the method described by Harbone (1973), Sofowora (1993), Trease and Evans (1996). Here, 20g of each extract was weighed and soaked with 100mls of methane, distilled water and n-hexane respectively for 48hours.

2.3 Weight Loss Measurement

The mild steel was prepared for corrosion experiment by adopting the method employed by Awe *et al*., (2015). 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 mild steel. The specimen was washed thoroughly with distilled water, degreased with absolute ethanol, dipped into acetone and dried in air. The dried coupons were weighed (W_o) and dropped in 50ml of 1M of H_2SO_4, without addition of different concentrations of the leaves extract at a certain temperature, for 1, 2, 3, 4 and 5 days contact time respectively; with the aid of acid resistance plastic clip. Then another set up was also prepared under the same conditions and monitored at the same contact time as above but with the addition of leaves extract. Each of the weighed mild steel was suspended in a beaker with the help of a thread. 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. 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 weight loss (W_L) was determined using the equation below:

$$W_L = W_o - W_f \quad (1)$$

Where W_o = mild steel weight before immersion in the corrosive medium.

W_f = mild steel weight after immersion in the corrosive medium.

Another significant factor for measuring the extent of adsorption of the extract on surface of the mild steel is the surface coverage. It can be calculated using equation (2):

$$\theta = \{1 - \left(\frac{W1}{W2}\right)\} \quad (2)$$

W_1 = mild steel coupons loss in weight in the presence of inhibitor.

W_2= mild steel coupons loss in weight in the absence of inhibitor.

The corrosion rates in the absence and presence of the inhibitors is determined using equation 3: The inhibition efficiency (I.E) and corrosion rate (CR) are determined using equations 3 and 4:

$$CR = \frac{WL}{AT} \tag{3}$$

CR = corrosion rate (g/cm^2day).

A = surface area of the mild steel coupon (cm^2).

T = immersion time in days

W_L = weight loss (g).

The corrosion rates obtained in the absence and presence of inhibitor are used to calculate inhibition efficiency (IE %) as in equation 4:

$$IE(\%) = \frac{CR_1 - CR_2}{CR_1} x\, 100 \tag{4}$$

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 inhibition efficiency can also be calculated using the equation below:

$$\%\,\mathrm{I.E} = \{1 - \left(\frac{W1}{W2}\right)\} \times 100 \tag{5}$$

The experiment was carried out at varying concentration of extract (0.1, 0.2, 0.3, 0.4 and 0.5g/ml) and temperature (30, 40, 50, 60 and 70^oC).

3. RESULTS AND DISCUSSION

3.1: Characterization of Ethanol Extracts of Pawpaw and Neem Leaves

3.1.1 Quantitative test results

Table 1: Results of quantitative tests carried out on the extract of Pawpaw and Neem leaves

Phytochemicals	Pawpaw Leaf Extract (PLE) (%w/w)	Neem Leaf Extract (NLE) (%w/w)
Alkaloids	-	-
Tannins	7.25	7.25
Flavonoid	7.10	7.31
Phenols	12.5	12.8
Glycoside	7.2	7.3
Saponins	-	-
Steriods	-	-

Table 1 revealed that both pawpaw and neem leaves extracts contain Tannins, Flavonoids, Phenols and Glycosides as the major phytochemical constituents; with Phenols being the dominant constituents, followed by Tannins. According to Ayeni *et al*. (2012), 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. Thus, tannin is the main phytochemical responsible for inhibition of corrosion in metals. Tannins and flavonoids are poly phenolic compounds which contain hydroxyl group bonded to aromatic carboxyl group. They can generally undergo oxidative reaction in solution to yield phenonate anions (derived from phenols by the loss of H^+) which can act as ligands towards metal cations (Ayeni et al., 2012).

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3.2 Weight Loss Result and Analysis

The results of the corrosion studies carried on the mild steel in the absence and presence of the extracts is summarized in Table 2.

Table 2: Weight loss at different time intervals using Pawpaw and Neem leave extracts in 1M H_2SO_4 medium at 70°C.

Time (days)	Blank (g)	PLE (g)	NLE (g)
1	0.4	0.17	0.17
2	0.79	0.35	0.24
3	0.92	0.3	0.29
4	1.02	0.33	0.31
5	1.02	0.33	0.31
6	1.02	0.33	0.31

Table 2 is the results of the corrosion studies carried out by immersing the mild steel in 1M H_2SO_4 medium at 70°C, in the absence and presence of pawpaw and neem leaves extracts respectively, at different time of immersion. The table revealed that in the absence of the extracts the weight loss increased with immersion time but became steady after the third day. The weight loss increases as the contact time between the mild steel and the acid medium increases because the corroding medium gains more access to the surface area of the mild steel. This implies that the corrosion rate increases with the period of immersion of the mild steel on the acidic medium. After three days the weight loss became approximately the same as shown in Table 2; indicating that the mild steel has no more vacant ground for the corroding medium to cover. In other words, the corrosion process has reached saturation. The table also revealed that at any given time of immersion, the weight loss of mild steel decreased in the presence of the extracts; there is a considerable reduction in weight loss in the presence of the inhibitors. This is an indication of the effectiveness of the extracts in reducing the corrosion of the mild steel, because in the presence of the extracts, there is an increased surface coverage as well as the adsorption of phytochemical constituents on the metal surface (Fadare et al., 2015). The weight loss became steady for pawpaw and neem leaves extracts after the third day. This could be attributed to the fact that the metal surface became saturated of the polyphenol constituent of the extracts after some time. The table also revealed that neem leaf is a better inhibitor than pawpaw leaf as indicated by the relatively lower weight loss in neem leaf extract.

3.3 Fourier Transform Infra-Red Spectroscopy (FTIR)

These various functional groups present in the extracts will be revealed by this technique. The spectra will show whether the compounds contained in pawpaw and neem leave extracts make them good corrosion inhibitors.

3.3.1 FTIR spectra of Pawpaw leaf extract

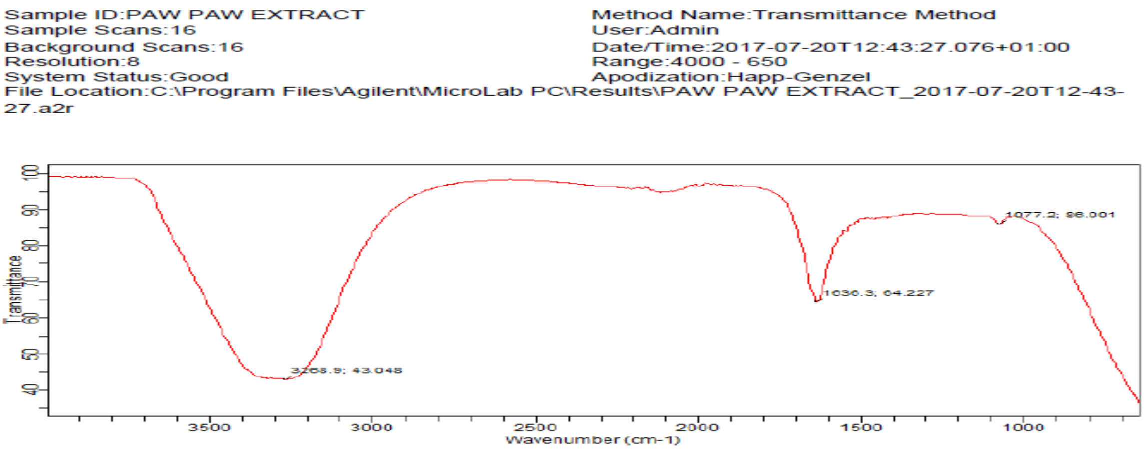


Fig 1: FTIR of Pawpaw leaf extract

3.3.2 FTIR spectra of Neem leaf extract

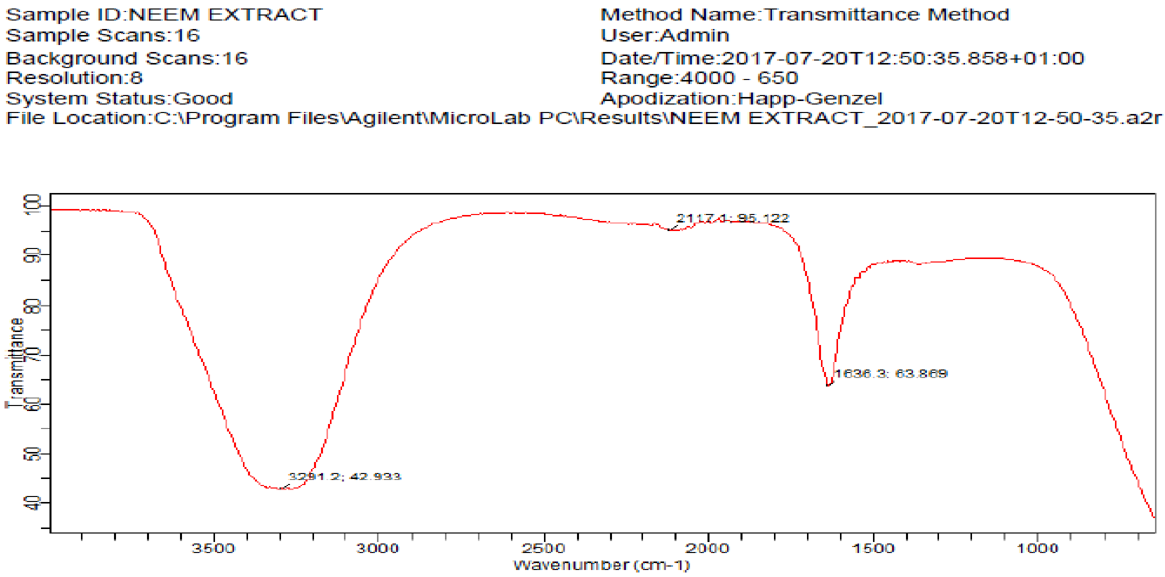


Fig 2: FTIR of Neem leaf extract

Figures 1 and 2 represent the IR spectra of ethanol extracts of pawpaw and neem leaves. From the results, it was found that the, O-H stretch and acid anhydride are commonly present in the leaves extracts. The C-Cl stretch is present only in neem leaf extract while aromatic C-H was found to be present only in pawpaw leaf extract. These show the presence of heteroatoms in the phytochemical constituents of the leaves extracts. Similar result was obtained by Ebenso *et al*. (2008).

3.4 Morphology

Scanning Electron Microscopy (SEM) was carried out at Chemical Engineering Department, Ahmadu Bello University, Zaria, Nigeria, to determine the microstructures of the uncorroded mild steel, corroded mild steel without and with pawpaw and neem leave extracts as shown in Plates1 to 3 at 200$_{\mu}$m magnification each. It was observed that there is structural distinction of the mild steel in uninhibited and inhibited with different extract, with a rapidly oxidized surface in the uninhibited in acidic medium. The SEM images also revealed that the mild steel specimen immersed in inhibited solution is in better condition having 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 (Leelavathi and Rajalakshmi, 2013). Result shows that the phytochemical constituents present in the PLE and NLE form a protective layer of the mild steel specimen and thereby reduce the corrosion rate.

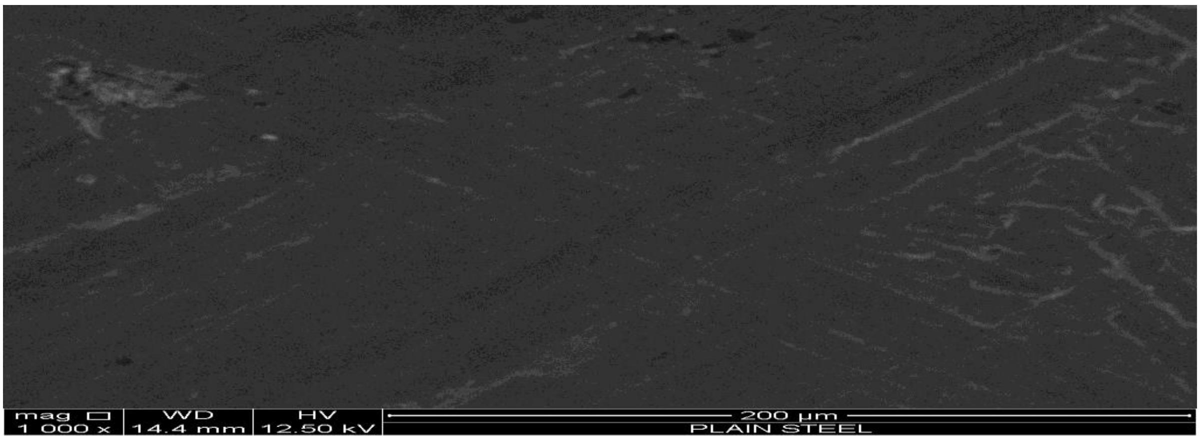
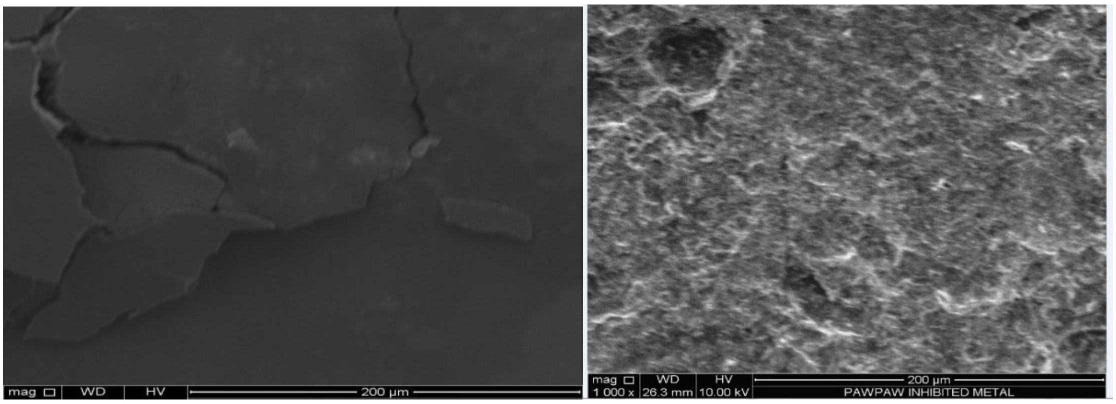
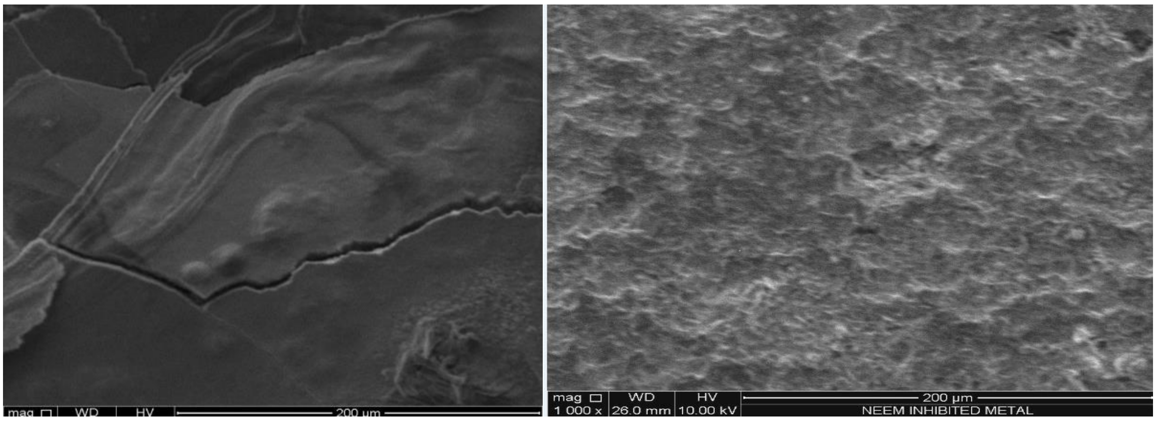


Plate 1: SEM image of uncorroded mild steel coupon



(a) Uninhibited **(b) Inhibited**

Plate 2: SEM images of uninhibited and PLE inhibited mild steel coupons in acidic medium



(a) Uninhibited **(b) Inhibited**

Plate 3: SEM images of uninhibited and NLE inhibited mild steel coupons in acid medium

4. CONCLUSIONS

The phytochemical constituents such as tannins and flavonoids present in pawpaw and neem leaves extract make them good green corrosion inhibitors because these constituents inhibit corrosion through the process of physical adsorption. On the average, neem leaf extract has higher phytochemical constituents than pawpaw leaf extract. The Infrared spectra of pawpaw and neem leaves extracts revealed that the presence of heteroatoms in the phytochemical constituents of the leaves extracts make them good corrosion inhibitors. The loss in weight of mild steel indicated that corrosion has taken place. However, in the presence of the extracts mild steel weight loss decreased; which indicated that the rate of corrosion has reduced due to the inhibitive properties of the extracts. Relatively, neem leaf extract showed greater decrease in weight loss than pawpaw leaf extract. This means that neem leaf extract is a better corrosion inhibitor than pawpaw leaf extract. This is so because it has more phytochemical constituents, and corrosion inhibition occurs as a result of the adsorption of the phytochemicals on the mild steel surface; thereby shading active corrosion site.

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