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CORROSION INHIBITION OF MILD STEEL IN NITRIC ACID MEDIUM USING EXTRACTS OF SOURSOP AND MANGO LEAVES

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Abstract - This study investigated the corrosion inhibition of mild steel using extracts of mango and soursop leaves in nitric acid medium. The extracts obtained from their respective leaves were characterized to determine their phytochemical constituents as well as functional groups present using standard characterization techniques and Fourier Transform Infrared Spectroscopy (FTIR) respectively. Gravimetric method was used to carry out the corrosion studies by determining the mild steel weight loss. Mild steel weight loss decreased in the presence of both extracts. The morphology of the corroded mild steels with and without the extracts revealed that the surface of the corroded mild steel with extracts was comparatively smoother; showing that the extracts have reduced corrosion. Comparatively, soursop leaf extract had higher average composition of the phytochemical constituents than mango leaf extract and the mild steel weight loss was lower which demonstrated that soursop leaf extract was a better inhibitor and therefore exhibited higher corrosion inhibition ability.

Keywords: Corrosion, Mild steel, Soursop leaf, Mango leaf, Nitric acid

1. Introduction

Nitric acid (HNO_3) is one of the most commonly used mineral acids in the chemical industry. The acid is used for various processes such as production of fertilizers, drugs, detergents, chemical cleaning, oil well acidification and acid pickling. As a result of this frequent use, corrosion is inevitable because many acid solutions cause corrosion in metals (Bentiss *et al*., 2006; Khaled and Hackerman, 2003; Quraishi and Sardar, 2003). According to Obot et al. (2011), though mild steel is prone to corrosion especially when exposed to acid media in the industry, it can be prevented by the use of inhibitors.

Thus metal corrosion is unavoidable but still controllable. Corrosion can be controlled through a number of ways such as material selection, design, cathodic-anodic protection (electroplating), coating and use of inhibitors. Synthetic compounds, apart from their adverse effect on the environment, have desirable properties as corrosion inhibitors. Nevertheless, the problems of environmental pollution arising from the toxicity and non-degradability posed by these compounds have generated serious concerns (Babatunde et. al 2012); and thus the need for the use of some natural products as corrosion inhibitors. Leaf extracts are chosen over inorganic substances as inhibitors because they are inexpensive, eco-friendly, readily available and renewable (Uwah et al., 2013). The phytochemical constituents of leaf extracts such as tannins, saponins and flavonoids are responsible for the inhibition properties by mechanism of physical adsorption (Ayeni, et al., 2012; Debi et al., 2013). Moreover, they can be extracted by simple procedures at low cost. In this research, the use of extracts of Soursop and Mango leaves as corrosion inhibitors of mild steel in nitric acid media was investigated.

2. Materials and Method

2.1 Equipment and materials used

The materials for this experiment include fresh leaves of Mango and Soursop, obtained from a local bush in Akegbe ugwu, mild steel coupons from coal camp Enugu. The reagents include nitric acid, Fehling's solution, potassium hydroxide with assay 97%, olive oil, distilled water, Aluminum Trichloride, Iron (iii) chloride, Sodium hydroxide, Wagner's reagent, iodine crystal, potassium iodide, acetone and ethanol (absolute) obtained from De-cliff integrated company, Enugu. Various equipment used are electronic weighing balance (Gallenkamp JA3002), beaker, tripod stand, measuring cylinder, thread, water bath, stop watch, beakers, Vernier caliper, mortar and pestle, thermometer, Sohxlet extractor, test tube, sieve and Electric oven

2.2. Method

2.2.1 Preparation of extracts of Soursop and Mango leaves

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 600μm sieve size to get very fine particles; leaving behind the coarse ones. 100g each of the ground sample was introduced into the thimble of a soxhlet extractor. 300ml absolute alcohol was later introduced and extraction carried out at 60^oC for 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^oC until most of the ethanol evaporated. 5g of the ethanol extract was dissolved in 1 litre of 2M nitric acid. Resulting solution was kept for 24hrs after which it was filtered and stored. Different concentrations of the inhibitor test solutions were then prepared from the stored solution.

2.2.2. Preparation of mild steel coupons

Mechanically, mild steel sheets were cut into coupons of dimensions of 2.9cm x 2.9cm x1cm and about 0.5g for gravimetric analysis. The coupons obtained were cleaned, degreased in ethanol; after which it was dipped in acetone and air dried. The weight of the dried coupons was obtained, and in each case was recorded as the initial weight, W_1.

2.3 Phytochemical Analysis

The phytochemical analyses, which include both qualitative and quantitative analyses of the ethanol extracts of each leaf to determine the presence of alkaloids, saponins, tannins, flavonoids, etc as well the quantity of these constituents, were done using the method described by Harbone (1973), Sofowora (1993), Trease and Evans (1996). 20g of each extract was weighed out. This was soaked differently in 100mls of methanol, distilled water and N-hexane for 48hours.

2.4 Corrosion Studies

Gravimetric method was used in this study. The mild steel size (2.9cm by 2.9cm) was cleaned, degreased in ethanol and dried with acetone. The weight of this dried coupon was weighed and recorded as initial weight (Wb). The dried mild steel coupon was then immersed in 50ml of 2M of nitric acid without of the leaves extract at a certain temperature, for 24, 48, 72, 96, 120, 144 and 168 hours contact time respectively. 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. At the end of each contact time, each sample was withdrawn from the test solution, washed twice in distilled water, dried with acetone and reweighed. The weight loss in gramme was taken as the difference in the weight of the coupon before and after immersion in different test solutions.The weight loss (W_L) was determined using Equation (1):

$$W_L = W_b - W_c \qquad \textbf{(1)}$$

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

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

2.5. Characterization of Mild Steel Coupon.

2.5.1. Fourier Transform Infra Red Spectroscopy (FTIR) Analysis

The extracts were characterized using Fourier Transform Infra Red spectroscopy to determine the functional groups responsible for corrosion inhibition.

2.5.2. Scanning Electron Microscopy (SEM)

The SEM analysis was done on the corroded and uncorroded mild steel coupon. This was done to determine the extent of corrosion inhibition by these extracts in an acid medium.

3. Results and Discussion

3.1. Characterization of Ethanol Extracts of Soursop and Mango Leaves

Table1: Results of qualitative tests carried out on the extract of Soursop leaves

Parameters	Methanol	n-hexane	Water
Alkaloids	-	+ +	+ +
Saponin	+ +	+ +	-
Flavonoid	-	+	+ +
Glycoside	+	+	+
Tannin	+ +	-	-
Quinine	-	-	-
Steroid	-	-	-
Phenol	++	-	+

3.1.1. Qualitative Test Results

Tables 1 and 2 are the results of qualitative analysis carried out on extracts of soursop and mango leaves respectively. The results revealed the phytochemical constituents present in the extracts. The respective meaning of the following signs (++, + and -) are highly present, present in trace amounts and absent respectively. In other words, the qualitative tests carried using methanol, n-hexane and water respectively revealed the presence of alkaloids, saponins, flavonoids, tannins and glycosides.

Table 2: Results of qualitative tests carried out on the extract of Mango leaves

Parameters	Methanol	n-hexane	Water
Alkaloids	-	-	+ +
Saponin	-	-	+ +
Flavonoid	+	+ +	-
Glycoside	+	+ +	-
Tannin	+ +	+ +	-
Quinine	-	-	-
Steroid	-	-	-
Phenol	+	-	+

3.1.2 Quantitative test results

The phytochemicals present in soursop and mango extracts are shown in Tables 3 and 4 respectively. The tables also revealed the percentage composition of the constituents. According to the tables, the highest constituent found in both soursop and mango leaves extract is flavonoids; followed by saponins, tannins and phenols in that order. The least constituent is alkaloids. The quantity of each phytochemical in soursop leaf extract is higher than in mango leaf extract. This could be attributed to the fact that soursop leaf extract has better applicability as corrosion inhibitor than mango leaf extract.

Table 3: Results of quantitative tests carried out on the extract of Soursop leaves

Parameters	Quantity (mg/L)	Percentage(%) composition
Alkaloids	0.863	1%
Saponins	14.4	12%
Flavonoids	74.1	64%
Glycoside	1.83	2%
Tannin	14.63	13%
Phenol	9.5	8%

Table 4: Results of quantitative tests carried out on the extract of Mango leaves

Parameters	Quantity (mg/L)	Percentage(%) composition
Alkaloids	0.42	1%
Saponins	8.4	17%
Flavonoids	31.4	64%
Glycoside	2.6	5%
Tannin	3.4565	7%
Phenol	2.79	6%

3.2 Gravimetric Method Result and Analysis

Table 5 showed the results of the corrosion studies on mild steel in 2M HNO3 medium. The studies were carried out in the absence and presence of soursop and mango leaves extract at a definite temperature with varying time of immersion. Generally, the table revealed that at any particular time of immersion, the weight loss of mild steel decreased in the presence of the extracts. The weight loss decreases more as the immersion time in both extracts increases. However, in the absence of the extracts, the weight loss increases with immersion time. This means that in the absence of suitable inhibitor, the rate of corrosion of the mild steel will increase as the duration of the immersion of the mild steel in the acid medium increases because the corroding medium gains more access to the surface area of the mild steel. Comparing the weight loss in the absence of leaves extract and in the presence of leaves extract, it was observed that at each time, there is a huge 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., 2016). The table is also useful in comparing the effectiveness of the inhibitors. For example, the table revealed that the weight loss in the presence of soursop leaf extract is lower than weight loss in the presence of mango leaf extract. This is an indication that soursop leaf extract has higher inhibition efficiency.

Table 5: Weight loss at different time intervals using soursop and Mango leave extracts in 2M HNO3

Time (hrs)	0 (g)	SSL (g)	ML(g)
24	3.3528	1.6834	2.208
48	3.6119	1.9326	2.4924
72	3.7753	2.2122	2.63
96	3.888	2.536	2.889
120	4.2889	2.8896	3.079
144	4.4716	3.116	3.2863
168	4.6878	3.2026	3.4995

SSL= Soursop leaf
ML= Mango leaf

3.3 Fourier Transform Infra-Red

Spectroscopy (FTIR) Infrared spectra are generally informative about what functional groups are present. These various functional groups help to determine a material's properties or expected behaviour by the absorption characteristics of associated types of chemical bonds. The spectra will show whether the compounds contained in soursop and mango leave extracts will make them good corrosion inhibitors.

3.3.1: FTIR Spectra of Soursop leave extract.

In Fig. 1, the peak at 831.2cm-1 represents bonded C=C alkene groups. The band at 1025.0cm-1 indicates vinyl ether C-O stretch. The peaks at 1155.5cm-1, 1244.9cm-1 and 1319.5cm-1 correspond to tertiary alcohol C-O stretch, amine group C-N stretch and Sulfone group S=O stretch. Absorption at 1375.4cm-1 1438.8cm-1 and 1617.7cm-1 are assigned to phenol group, carboxylic acid and ketone groups respectively. The peak at 1736.9cm-1 shows aldehyde group C=O stretch while the peaks at 2855.1cm-1 and 2922.2cm-1 show amine salts. The band at 3295.0cm-1 indicates carboxylic group O-H stretch. The presence of Sulphur, Nitrogen, Oxygen and alkene compound show that soursop leaf extract can effectively be used as corrosion inhibitor.

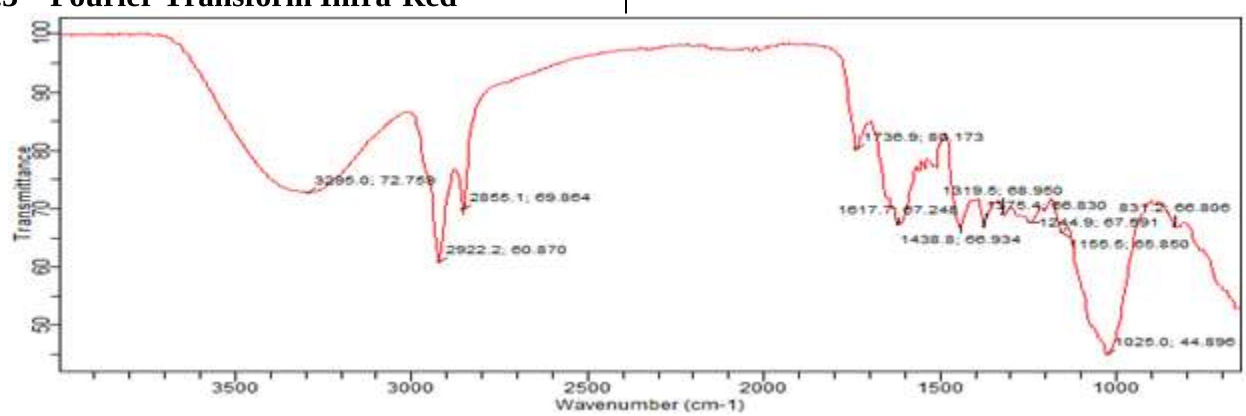

Fig 1: FTIR of Soursop leaf extract

3.3.2 FTIR Spectra of Mango Leave Extract

In fig.2, absorption band at 827.5cm-1 indicates alkene bonding C=C. The absorption band at 1028.7cm-1 indicates C-O stretch vinyl ether. The region 1200.2cm-1 indicates alkyl aryl C-O stretch while band at 1241.2cm-1 reveals C-N stretch amine group. The bands at 1293.4cm-1 and 1446.2cm-1 correspond to primary alcohol O-H stretch and alkane group C-H bond. The peaks at 1494.7cm-1 and 1699.7cm-1 correspond to aromatic ring stretch and conjugate aldehyde C=O stretch. The bands at 1617.7cm-1, 2922.2cm-1 and 3362.1cm-1 are assigned to ketone, amine salt and carboxylic acid groups respectively. Just like soursop leaf extract, mango leaf extract contains alkene, oxygen and nitrogen compounds which suggested that it has tendency to act as a good corrosion inhibitor.

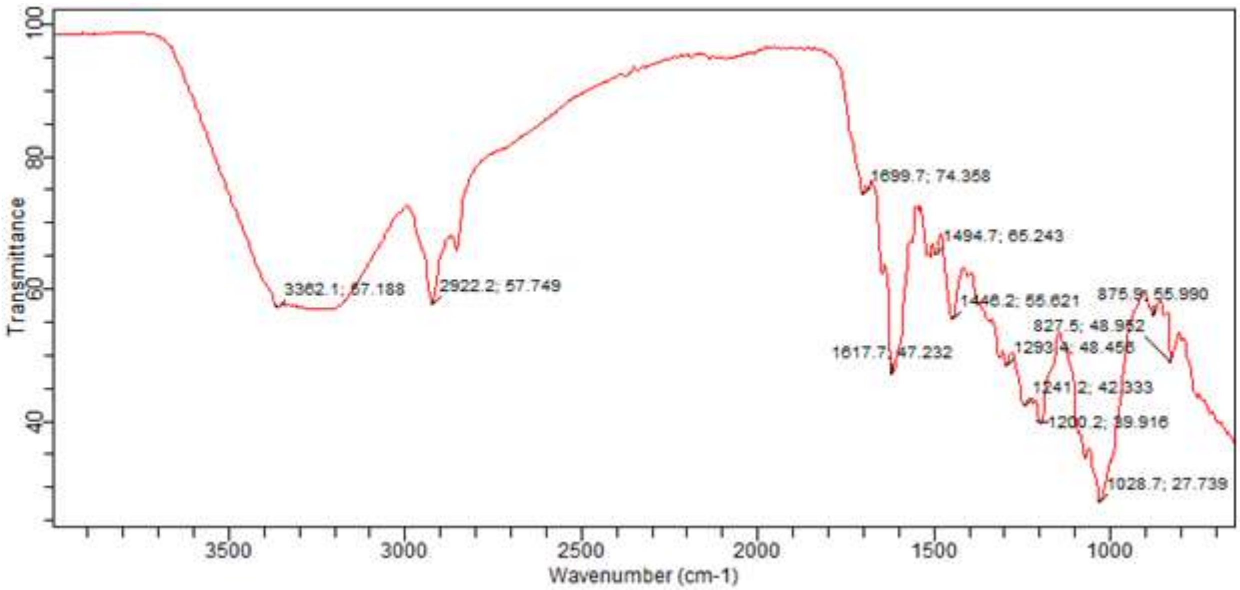

Fig 2: FTIR of Mango leaf extract

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 soursop leaf extract as shown in the Plates1 to 3

3.4.1 Uncorroded mild steel

Plate 1 represents the SEM analysis of uncorroded mild steel at 100µm magnification. The mild steel surface appear non porous and flat. A closer look at the surface of the plates show that they still have their natural smooth surface; indicating that they haven't been attacked by corrosive agents.

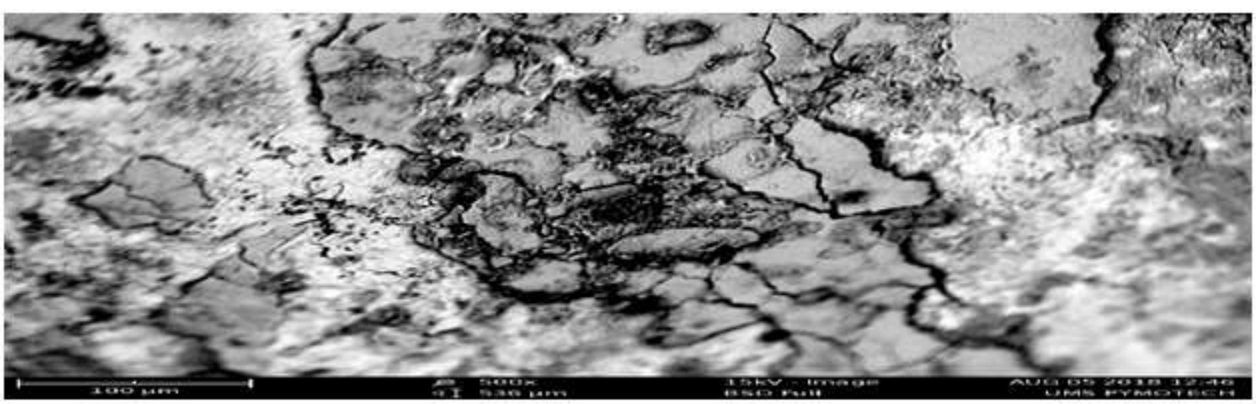

Plate 1: SEM analysis of uncorroded mild steel

3.4.2 Corroded mild steel without extract

The mild steel sheet corroded in the absence of the soursop leaf extracts. The SEM was done at 100µm magnification is represented in Plate 2. The surface is rough, and according to Prabakaran et al (2016), in the absence of the extract the corroded mild steel surface becomes rough and porous because it is directly attacked by the acids.

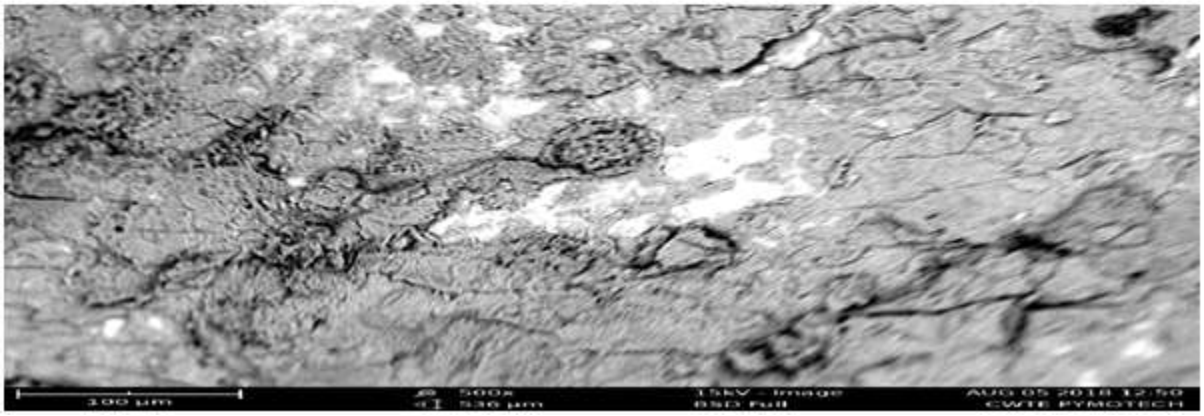

Plate 2: SEM analysis of corroded mild steel without extract

3.4.3 Corroded mild steel with extract

Plates 3 represents corroded mild steel with soursop leaf extract. The SEM was done at 100µm magnification. It was obtained after immersing corroded mild steel in tested solutions of the soursop extracts for 24hrs at 25oC. The less corrosion spots seen in the figures clearly revealed the effects of the inhibitors in reducing the level of metal deteriorations. The surface is smoother because of the formation of the adsorbed film of the extracts of the leave on the mild steel surface; and this prevents corrosion (Fiori-Bimbi etal, 2015). This implies that extracts of soursop leaves have great potentials to at least prevent or reduce corrosion of mild steels in acidic media.

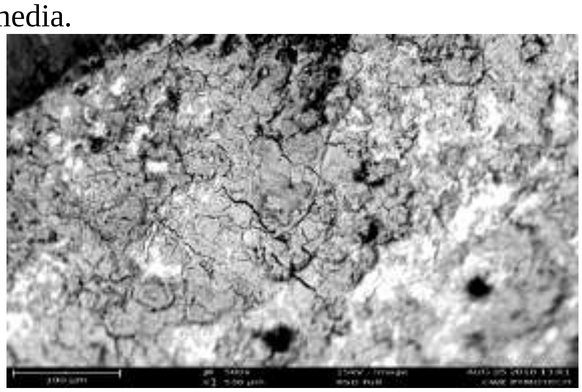

Plate 3: SEM analysis of corroded mild steel with extract

4. Conclusions

The study proved that soursop and mango leaves extracts are excellent mild steel green corrosion inhibitors due to their phytochemical. The qualitative and quantitative analysis of the extracts revealed that the average composition of the phytochemical constituents is higher in soursop leaf extract than in mango leaf extract. The Infrared spectra of soursop and mango leaves extracts revealed that their associated elements/compounds and chemical bonds make them good corrosion inhibitors. The soursop leaf extract showed better corrosion inhibitor characteristics than mango leaf extract. The morphology of the corroded mild steel in the absence and presence of soursop leaf extract revealed that the corrosion inhibition capacity of the leave extract is significant.

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