International Journal of Engineering Science Invention (IJESI)
ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
www.ijesi.org ||Volume 8 Issue 01 Ver. I || Jan 2019 || PP 60-71

Exploiting the Extract Constituents of Pentaclethra Macrophylla Bentham (Ugba) Leaves in the Corrosion Inhibition of Mild Steel in Acidic Media

* Chinonso B. Adindu [1, 2a], Ujupaul J. M. Ikezu [1], Uche G. Nwokeke [1],
[1]*Department of Chemistry, Imo State University, P M B 2000 Owerri, Imo State Nigeria.*
[*2]*Electrochemistry and Material Science Research Laboratory, Department of Chemistry, Federal University of Technology, P M B 1526 Owerri, Imo State Nigeria.*
Corresponding Author: Chinonso B. Adindu

Abstract: *The chemical constituents of Pentaclethramacrophylla (PM) were investigated by phytochemical, GC-MS and FTIR analysis and exploited in the corrosion inhibition of mild steel in acidic media. The phytochemical results revealed the presence of saponin, alkaloid, flavonoid, tannin and phenol which shows that PM extract is a prospective corrosion inhibitor. Also the GC-MS and FTIR results showed that the extract contained some functional groups that are expected to aid corrosion inhibition. Weight loss and electrochemical results indicated that PM extract functioned as a good corrosion inhibitor for mild steel via the adsorption of its extract constituents on the mild steel surface which was confirmed by the scanning electron microscopy results.*
Key words*; extract constituents, corrosion, inhibitor, Pentaclethramacrophylla, gravimetric analysis,*

Date of Submission: 26-12-2018 Date of acceptance: 11-01-2019

I. Introduction

The problem of corrosion has persisted over the years despite many data on corrosion control. Corrosion inhibition is one of the cost effective and safe methods of controlling corrosion. Corrosion inhibitors are organic and inorganic additives that function by protecting the metal surface from corrosion attack [1-5]. Inorganic inhibitors work by oxidizing the metal surface thereby creating an impermeable layer which helps isolate the metal from the corrosive environment while organic inhibitors possess features such has hetero atoms, large surface area and double bond which upon adsorption, blanket the metal surface and isolate it from the corrosive attack [6-10]. The toxic nature and high price of most synthetic inhibitors [11-16] has led to the search for materials of plant origin as corrosion inhibitors. This is because these materials contain phytochemicals which bear close resemblance with those of conventional inhibitors yet ecologically friendly, non-toxic, cheap and readily available [17-22].

Regarding our continuous interest in the use of plant extracts as corrosion inhibitor for mild steel, we herein report the corrosion inhibition of mild steel in acidic solutions using extract from Pentaclethramacrophylla as the inhibitor. Pentaclethramacrophylla (African oil bean) is a tropical tree crop belonging to the Leguminosae family andMimosoideae sub-family [23].The plant is found in Nigeria West Africa, both the seed, leaves, bark, stem and root of PM have been found to contain chemical constituents which are medically useful [24].The corrosion inhibiting properties of PM will be investigated using weight loss and electrochemical methods of corrosion monitoring. The surface morphology of the mild steel will be investigated by SEM imaging while phytochemical screening, GC-MS and FTIR will be used to investigate the extract constituents of PM.

II. Experimental Procedures

2.1 Preparation of plant material

The leaves of Pentaclethramacrophylla were obtained from the garden of Imo state University Owerri and identified by Dr. Mbagwu of the department of plant science and Biotechnology. The leaves were dried to a low moisture content, ground weighed and dipped in absolute ethanol for 24-h. The resulting solution was cooled and filtered to obtain the stock solution. The stock solution was quantified by comparing the weight of the dried residue with the weight of the plant material before extraction. Test solutions were prepared in the concentration range of 200- 1000 mg/L.

2.2 Preparation of the metal specimen

The mild steel specimens used for the experiments have the weight percentage composition: C-0.05, Mn-0.6, P-0.36, Si-0.3 and the balance FE [25], the metal was obtained from a commercial source and press cut into the desired dimensions and stored in a moisture free desiccator for use when desired.

2.3 Phytochemical screening

Phytochemical screening of the plant material was carried out by the method described by Okwu 2001 [26]. It involved investigating the composition of alkaloids, flavonoids, saponins, tannins and phenols present in the PM extract.

2.4 GC-MS Analysis

Gas chromatography-mass spectroscopy experiment was performed on the PM extract to determine its organic constituents. The experiment was performed using SHIMADZU, JAPAN GCMS-QP2010 PLUS GC-MS apparatus. The experimental conditions are as described in our previous work [27].

1.5 FTIR Experiment

Todetermine the functional groups present in PM ethanol extract, FTIR analysis was carried with a FTIR (KBr) Nicolet Magna-IR 560 spectrophotometer. The analysis was performed by mixing the PM extract with KBr, making the pellet and presenting the sample for FTIR analysis.

2.6 Weight loss experiment

Weight loss experiments were performed on mild steel of dimension 3 cm x 3cm x 0.14 cm. The metals were wet-polished using silicon carbide abrasive paper from #200 to1000 [28], washed in distilled water and dried using acetone and air. The gravimetric apparatus was set up by suspending the coupons in 300 ml of the test solutions with rod and hooks. To ascertain the weight loss of the mild steel specimen with time, the coupons were retrieved at 24 h interval continuously for 120 h. When retrieved, the coupons were immersion in a solution containing sodium hydroxide (20%) and zinc dust to momentarily quench the corrosion reaction [29], washed with clean water re-weighed and re-immersed in the test solutions. The weight loss was taken as the difference between the weight of the coupon before immersion and the weight after a given time.

2.7 Electrochemical experiment

Mild steel specimens of dimension 1 cm x 1 cm x 0.14 cm were used for the electrochemical analysis. Prepared as described in the gravimetric experiment and fixed in polytetrafluoroethylene (PTFE) rods with epoxy resin. The coupon was fixed by leaving one side (area $1cm^2$) uncovered; the analysis was performed with a VERSASTAT 400 complete DC Voltammetry and corrosion system with a V^3 software [30]. The mild steel specimen was the working electrode while the counter electrode was a graphite rode and saturated calomel electrode was used as the reference electrode. The reference electrode was connected via a luggins capillary and the system was allowed to stand in an unstirred and aerated condition for 1hour at 30^oC. The conditions for the electrochemical impedance analysis was a corrosion potential (E_{corr}) over a frequency range of 100 KHz -10 MHz and a signal amplitude of perturbation of 5 mV. Potential range of ±250 mV versus corrosion potential at a scan rate of 0.333 Mv/s was used for the potentiodynamic polarization analysis [31].

2.8 Scanning electron microscopy experiment

Morphological analysis of the mild steel surface prior to and after immersion in the text solutions was carried out using SHIMADZU SSX-550 scanning electron microscope. Mild steel specimens of dimension 15 × 10 × 2 mm were washed with distilled water, dried with acetone and submitted for SEM examination. The surface of the plain metal was examined, also examined was the metals dipped in 1 M HCl and 0.5 M H_2SO_4 without and with PM extract.

III. Results And Discussion

3.1 Phytochemical and GC-MS Results

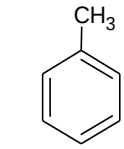
To ascertain the chemical constituents of PM, phytochemical analysis was performed; the result is presented in Table 1. From the result, it is clear that PM contains considerable amount of phytochemicals which makes it a prospective candidate as a corrosion inhibitor. The GC-MS result shows 24 compounds (Fig.1a & b). Some of these compound containing atoms that have been reported to support corrosion inhibition [32-33] are listed in Table 2.

Tables 1Phytochemial result of PM extract

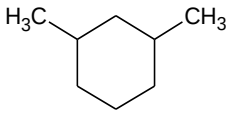
Phytochemical	% abundance
Alkaloids	0.02
Saponins	0.28
Flavonoids	0.15
Phenols	2.45ppm
Tannins	8.36 ppm

Table II Some chemical constituents of PM extract

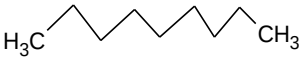
Line	Name of compound	Molecular formula	Molecular weight	Retention time	Area (%)
1	Methylbenzene	C_7H_8	92	3.766	2.06
2	1,3-Dimethylcyclohexane	C_8H_{16}	112	3.929	2.36
6	Nonane	C_9H_{20}	128	5.572	7.88
7	1-Ethyl-3-methylbenzene	C_9H_{12}	120	6.407	4.75
19	Haxadecanoicacd	$C_{16}H_{32}O_2$	256	21.216	5.83
21	13-Decosenoate	$C_{23}H_{44}O_2$	352	23.130	2.63
22	3,7,11,15-Tetramethyl-2-Hexadecen-1-ol	$C_{20}H_{40}O$	296	23.410	1.78



methylbenzene



1,3-dimethylcyclohexane



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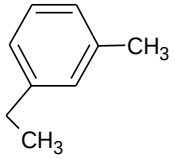
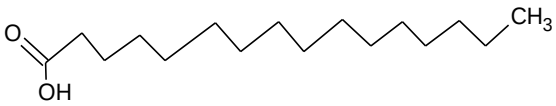
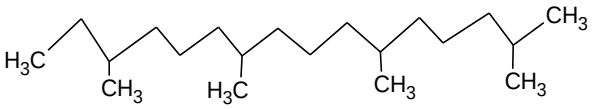


1-ethyl-3-methylbenzene



hexadecanoic acid

13_decosenoate



3,7,11,15_Tetramethyl_2_hexadecen_1_ol

Fig. 1(a) Structures of some chemical constituents of PM extract

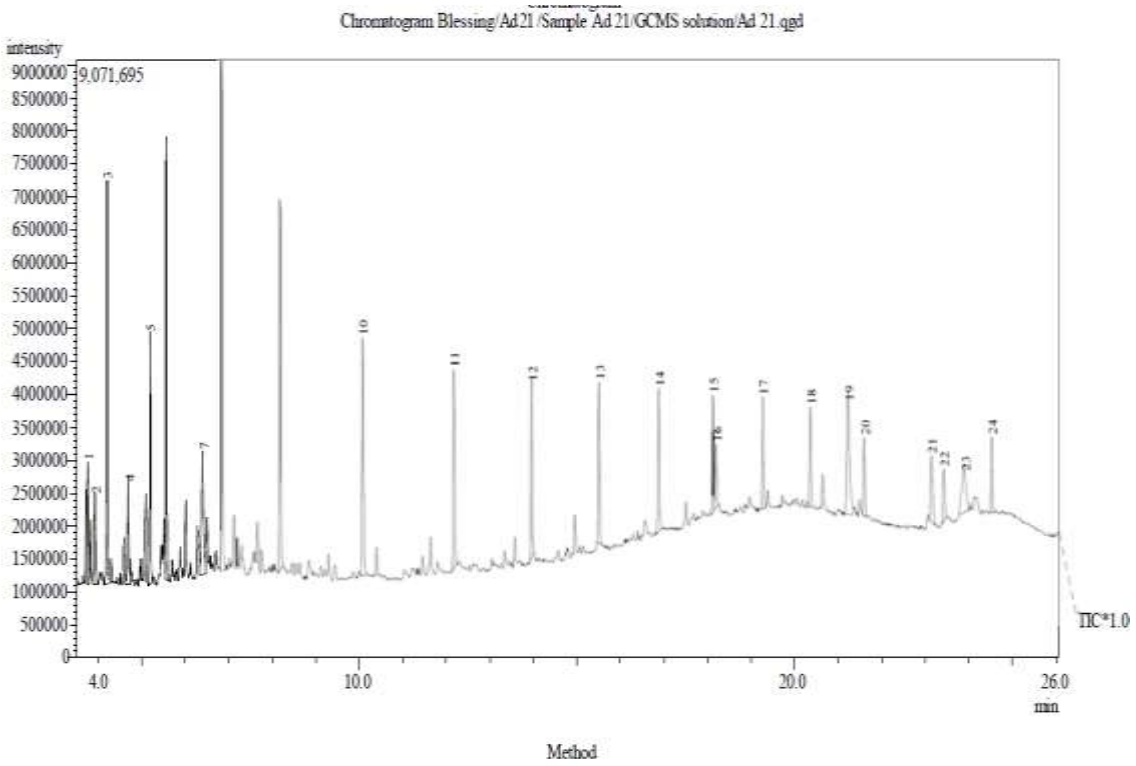


Fig. 1(b) GC-MS microgram of PM extract

3.2 Fourier Transform Infrared spectroscopy Result

FTIR analysis was performed to detect the functional groups present in the PM extract. The FTIR spectrum is presented in figure 2. The result revealed these functional groups; wide rounded RO-H(Alcohol) broad band at the frequency of 3250.2 cm^{-1}, carboxylic acid C=O bond at a frequency of 1513.3,1610.2 and 1703.2 cm^{-1}, Methylene C-H bend at a frequency of 1349.3 cm^{-1} and 1446.2 cm^{-1}, Cyclohexane ring vibrations Methyl (CH−) at a frequency of 1036 cm^{-1}, Primary amine, CN stretch at 1088.4 cm^{-1}, Aromatic C-H in-plane bend at a frequency of 1222..6 cm^{-1} and aromatic C-H at 764.1 and 808.1, the result was in agreement with the GC-MS analysis result.

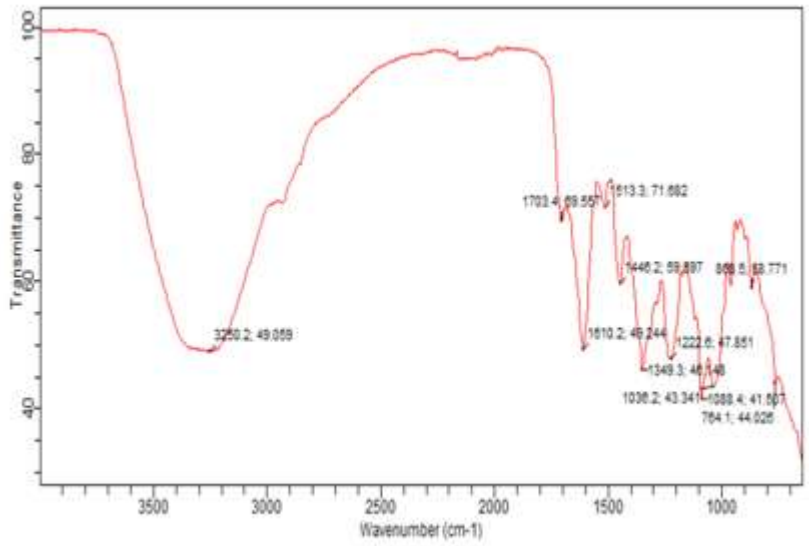


Fig. 2 FTIR spectra of PM extract

3.3 Gravimetric Procedure

The corrosion rate of mild steel in both 1 M HCl and 0.5 M H_2SO_4 without and with PM extract as an anticorrosion agent was evaluated using the gravimetric technique of corrosion monitoring. Fig. 3 shows the plots of weight loss against concentration obtained for (a) 1 M HCl and (b) 0.5 M H_2SO_4 .The results show that PM inhibited the corrosion of mild steel in both acidic environments. The weight loss was seen to increase with time and decreased with concentration of PM extract. The effectiveness of PM extract as an anticorrosion agent was quantified by calculating the inhibition efficiency with the expression below:

$$IE\ (\%) = \left[1 - \frac{\Delta W_{inh.}}{\Delta W_{uninh.}}\right] X\ 100 \qquad (1)$$

Where ΔW_{inh} Represents the weight loss in the presence of PM as the anticorrosion agent and ΔW_{uninh} is the weight loss in the uninhibited acid solutions. Fig. 4 is the plots of inhibition efficiency against time for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H_2SO_4 solutions. The result shows that PM extract functioned effectively as an anticorrosion agent in both acid solutions as the inhibition efficiency is seen to increase with both concentration and time. The result is in line with that reported elsewhere [34-35].

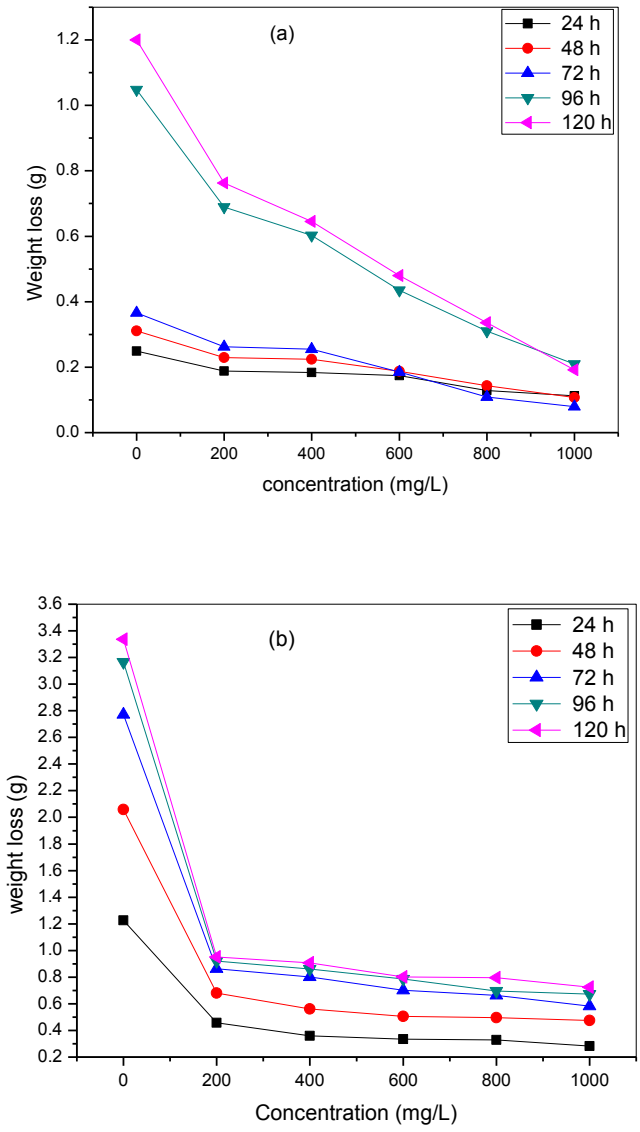


Fig. 3 Weight loss vs concentration for mild steel corrosion in the absence and presence of (a) 1m HCl and (b) 0.5 MH_2SO_4 solutions.

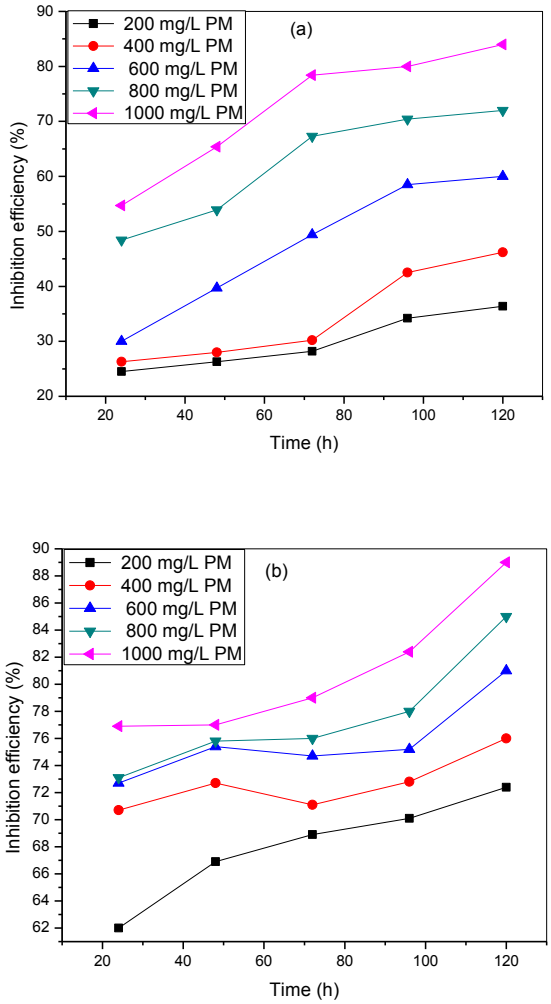


Fig. 4 variation of inhibition efficiency with time for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H_2SO_4 solutions in the presence of PM as the anticorrosion agent.

3.4 Potentiodaynamic Polarization Results

Potentiodynamic polarization analysis was undertaken to study the effect of PM extract on the anodic and cathodic half reactions [36]. The potentiodynamic polarization curves for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H_2SO_4 solutions without and with PM extract are presented in Figure 5 while the potentiodynamic polarization data derived from the curves are presented in Table 3. The results show that the addition of the extract affected both the anodic dissolution of the mild steel and cathodic hydrogen gas evolution [37]. Addition of PM extract shifted the corrosion potential E_{corr} slightly towards the more positive values while both the anodic and cathodic current densities and also the corrosion current density i_{corr}were reduced showing that the extract functioned via mixed corrosion inhibition mechanism in both 1 M HCl and 0.5 M H_2SO_4. The current densities in the absence ($i_{corr.\ bl}$) and presence ($i_{corr.Inh}$) of PM extract where used to estimate the inhibition efficiency from the polarization data as below;

$$IE(\%) = \left(\frac{I_{corr\ (bl)} - I_{corr\ (inh)}}{I_{corr\ (bl)}}\right) \text{x } 100 \qquad (2)$$

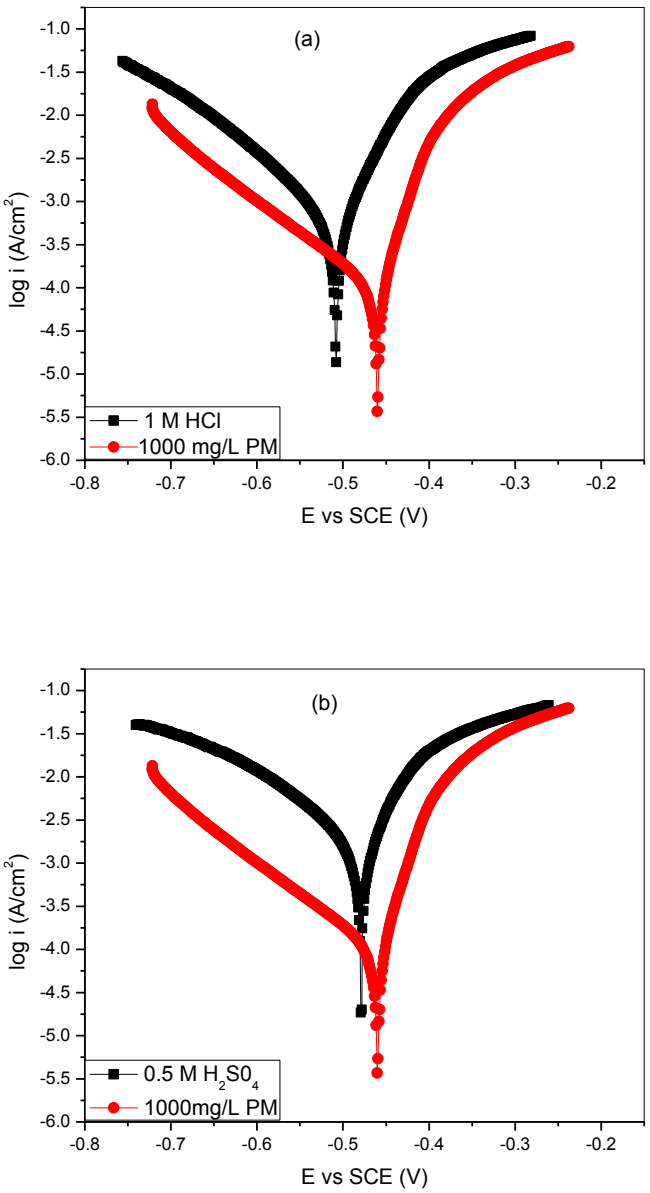


Figure 5 Potentiodynamic polarization plots for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H_2SO_4 without and with PM extract.

Table. III Potentiodynamic polarization data for mild steel corrosion in 1 M HCl and 0.5 M H_2SO_4 in the absence and presence PM extract.

System (mg/L)	EmV (vs SCE)	i_{corr} (µA/cm^2)	IE%
1 M HCl	-508.3	456.8	
1000 PM	-477.5	31.507	93.1
0.5 M H_2SO_4	-639.2	3570	
1000 PM	-476.5	0.003264	99.9

3.5 Electrochemical Impedance Spectroscopy Results

To give insight into the kinetics of the electrochemical reactions at the metal/acid interface, electrochemical impedance spectroscopy experiments were undertaken. Electrochemical Nyquist plots for mild steel corrosion without and with PM (1000 mg/L) in (a) 1 M HCl and (b) 0.5 M H_2SO_4 solutions are presented

in Figure 6 while the electrochemical data from the polarization curves are shown in Table 4. Figure 6 show only one depressed capacitive semicircle in the presence and absence of PM extract over the frequency range examined. The size of the semicircle is seen to increase with the addition of PM extract. The high frequency intercept with the real axis in the curves represents the solution resistance (R_s) while the low frequency intercept with the real axis represents the charge transfer resistance (R_{ct}) [38]. From table 4 it can be seen that the addition of PM extract reduced the size of the double layer capacitance (Q_{dl}), this decrease shows that the constituents of PM extract are adsorbed on the mild steel surface protecting it from the corrosion attack. The value of the charge transfer resistance (R_{ct}) is seen to increase with the addition of PM extract this caused the increase in the size of the capacitive semi-circle of the Nyquist plot showing that the PM extract exhibited inhibitive effect on the mild steel. The values of the charge transfer resistance in the absence of PM extract ($R_{ct.\ bl}$) and in the presence of the extract ($R_{ct.inh}$) where used to estimate the inhibition efficiency from the impedance data as follows;

$$IE(\%) = \left(\frac{R_{ct,inh} - R_{ct,bl}}{R_{ct,inh}}\right) x\ 100 \qquad (3)$$

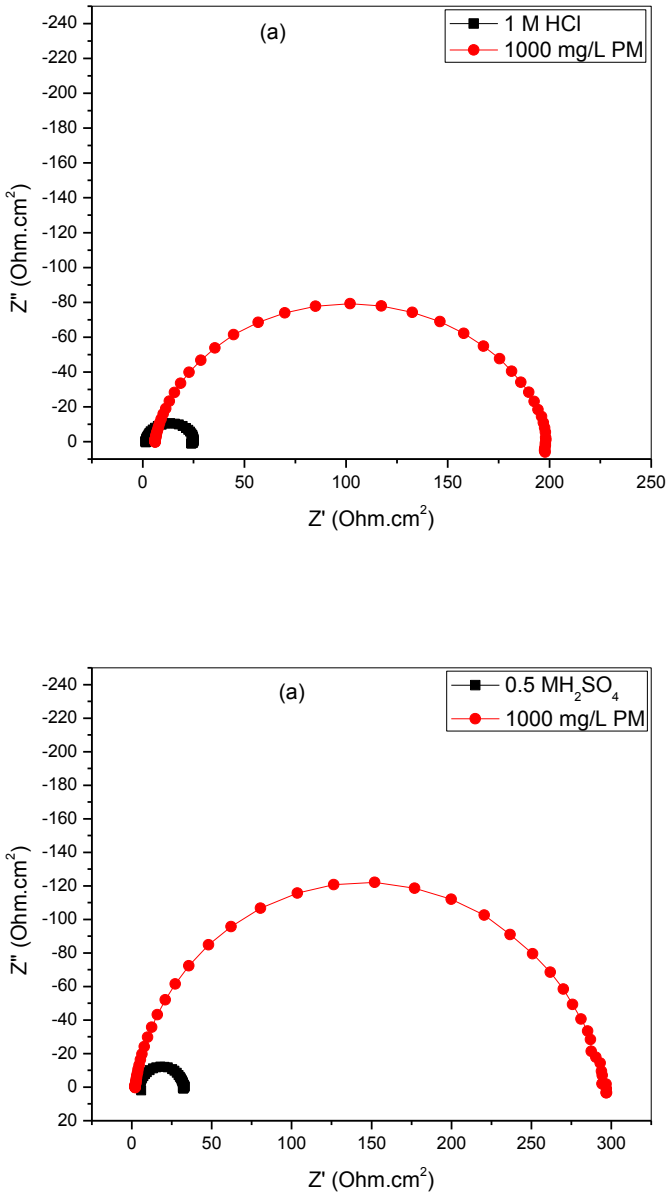


Fig. 6 Nyquist plot for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H_2SO_4 solutions in the absence and presence of 1000 mg/L PM extract.

Table IV Impedance data for mild steel Corrosion in 1 M HCl and 0.5 M H_2SO_4 without and with PM extract.

System (mg/L)	$R_{ct}(\Omega cm^2)$	$Q_{dl}(\mu\Omega^{-1}S^n cm^{-2})$ x10^{-6}	IE(%)
1 M HCl	23.46	13.5	
1000 PM	192.3	3.29	87.8
0.5 M H_2SO_4	9.788	12.9	
1000 PM	111.8	5.57	91.2

3.6 Scanning electron microscopy examination results

Morphological examination was undertaken on the mild steel surface prior to and after immersion in the acidic solutions containing 1000 mg/L of PM for 24 h to determine the effect of the adsorbed surface constituents on the mild steel surface. Figure 7 shows the microgram of mild steel before immersion in the acidic solutions, Figure 8 show the micrograms of the metal before (8a) and after immersion (8b) in 1 M HCl solution containing 1000 mg/L of PM while Figure 9 shows the images before (9a) and after immersion (9b) in 0.5 M H_2SO_4 containing 1000 mg/L of PM after 24 h. The metal surfaces can be seen to have been severely corroded in the blank solutions while the images of the mild steel specimen immersed in the acid solutions containing the PM inhibitor present smoother surfaces, this can be due to the serious dissolution of the mild steel in the blank solutions.

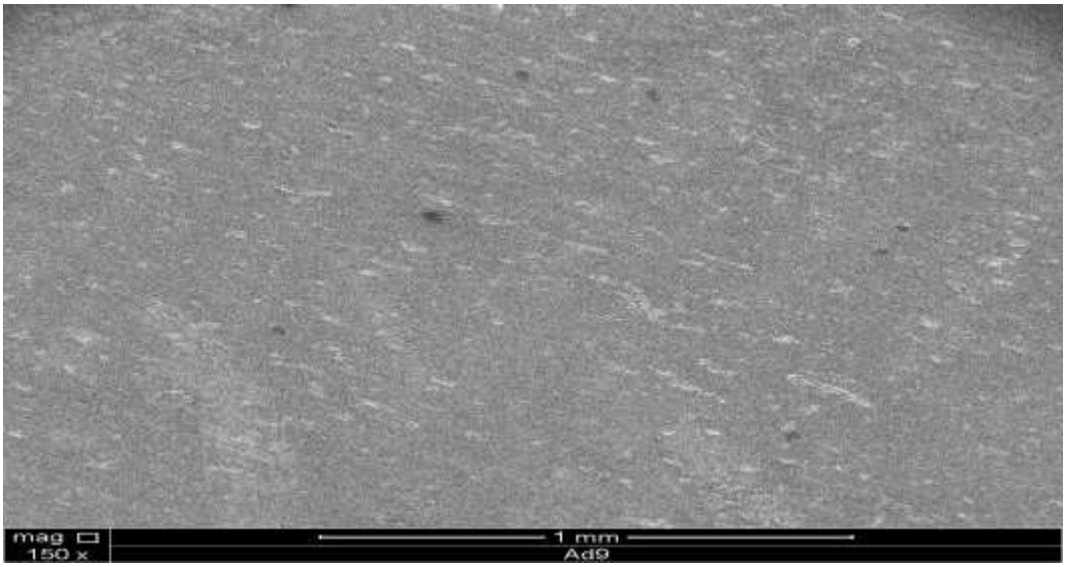
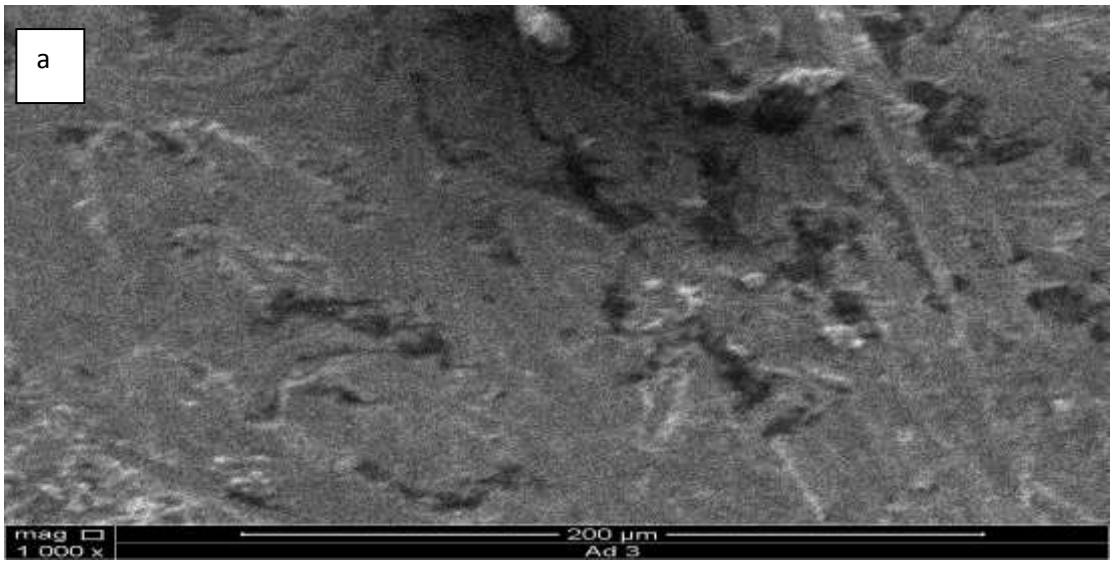


Fig. 7 SEM image of the un-corroded mild steel surface



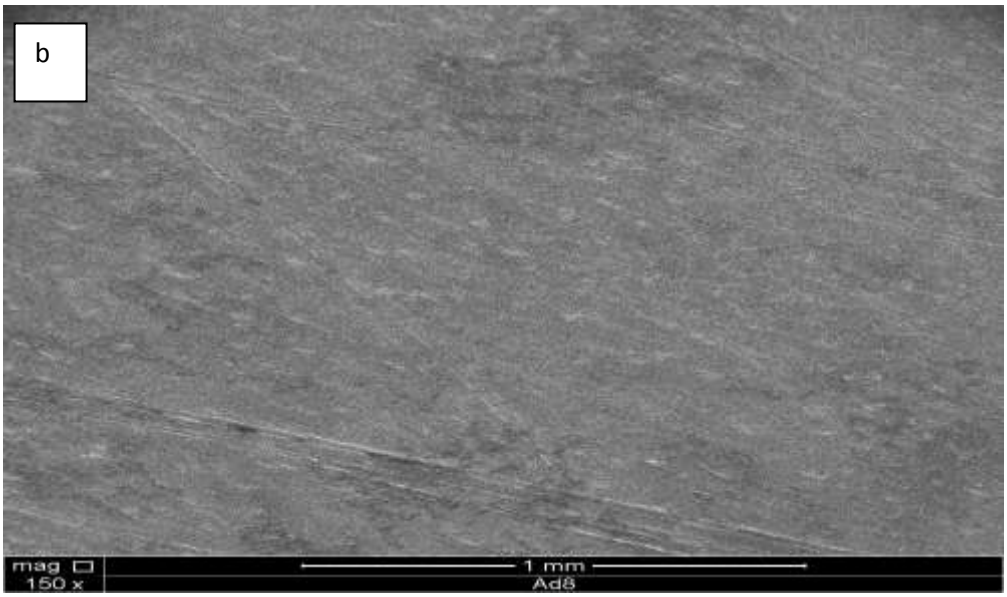
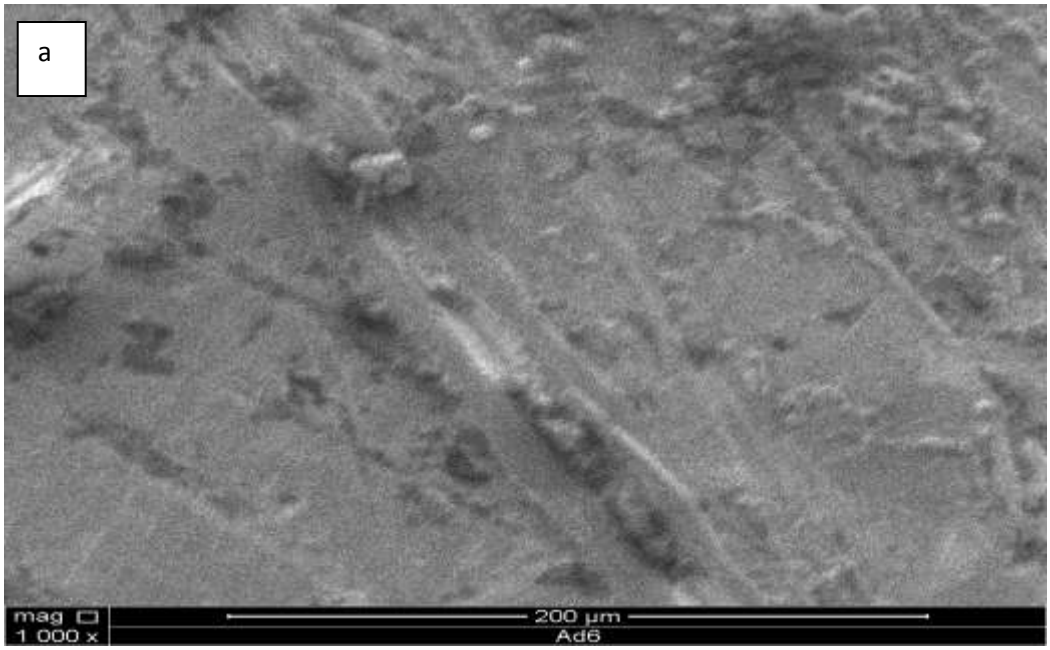


Fig. 8 SEM images of the mild steel surface before (a) and after (b) immersion in 1 M HCl solution containing 1000 mg/L of PM extract.



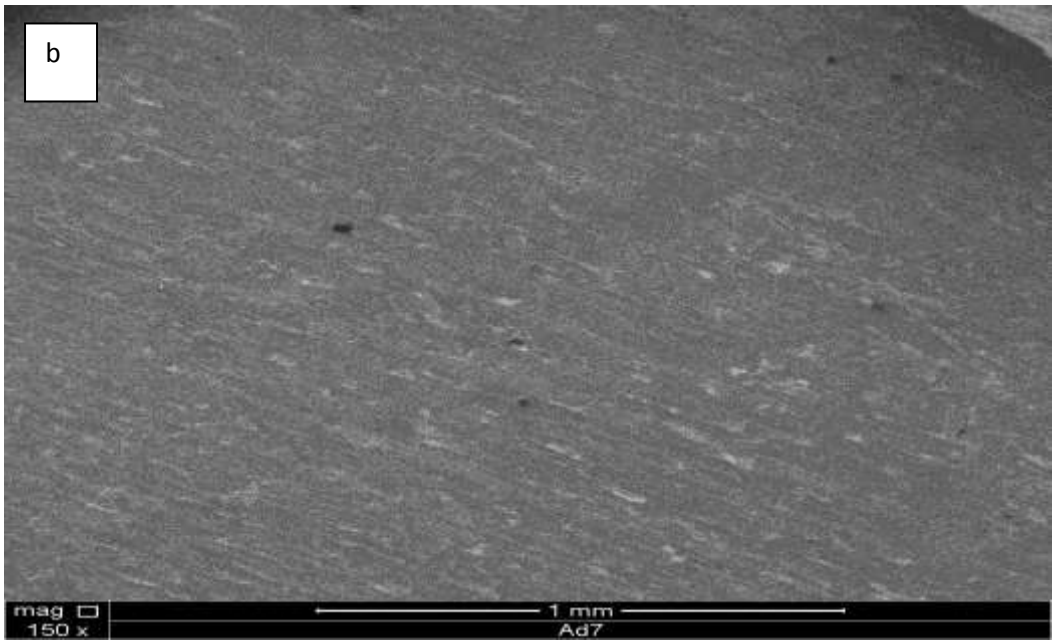


Figure 9 SEM images of the mild steel specimen before (a) and after (b) immersion in a 0.5 M H_2SO_4 solution containing 1000 mg/L of PM extract.

IV. Conclusion

This research involved the determination of the chemical constituents of Pentechlethramicrophyta (PM) leaves and their application in the corrosion inhibition of mild steel in acidic environments. The phytochemical results showed the presence of saponins, alkaloids, flavonoids, tannins and phenols which suggest PM extract to be a good candidate for corrosion inhibition. The GC-MS and FTIR results revealed that PM extract contained some active constituents which have been reported to aid corrosion inhibition of mild steel. Weight loss and electrochemical methods of corrosion monitoring were used to study the corrosion inhibition ability of Pentechlethramicrophyta leaves. The weight loss results revealed that weight loss increase with time and decreased with concentration of PM extract, the inhibition efficiency increased with both concentration and time. The potentiodynamic polarization results showed that PM extract is a mixed-type corrosion inhibitor mild steel inhibiting both the anodic and cathodic half reactions. The impedance and SEM results both indicate that the inhibition of mild steel was achieved via adsorption of the chemical constituents of PM on the metal solution interface.

Conflicting interest

The authors declare no conflict of interest

Funding

This project was sponsored by TETFund under the TETFund IBR research grant with grant number TETFUND/DESS/UNI/OWERRI/IBR/2016/VOL1./23

Acknowledgment

The authors would like to thank Professor E. E. Oguzie for his contributions to the success of this project.

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Chinonso B. Adindu" Exploiting the Extract Constituents of Pentaclethra Macrophylla Bentham (Ugba) Leaves in the Corrosion Inhibition of Mild Steel in Acidic Media" International Journal of Engineering Science Invention (IJESI), vol. 08, no. 01, 2019, pp 60-71