

## RESEARCH ARTICLE

## EXPERIMENTAL SYNTHESIS, CHARACTERISATION, AND INHIBITION EVALUATION OF GREEN CORROSION INHIBITORS USING BIO-EXTRACTS

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## ARTICLE DETAILS

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## ABSTRACT

The growing environmental concerns associated with synthetic corrosion inhibitors have also heightened demand for environmentally friendly substitutes derived from natural sources. This paper assesses the synthesis and inhibition of green corrosion inhibitors from bio-extracts of Carica papaya leaf (CPL), Vernonia amygdalina leaf (VGL), and Annona muricata leaf (AML) on aluminium, mild steel, and zinc in HCl and H<sub>2</sub>SO<sub>4</sub>. Phytochemicals such as alkaloids, flavonoids, phenols, saponins, tannins, steroids, phytates, and glycosides were confirmed by phytochemical analysis. It was determined that the Bio-Extracts were described by Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography-Mass Spectrometry (GC-MS). Thermometric analysis and scanning electron microscopy (SEM) were used to evaluate corrosion inhibition. An optimal inhibition efficiency of 75.42 -84.56% was obtained with the extracts, which was due to the formation of protective Fe<sup>2+</sup>-extract complexes by adsorption on metal surfaces. FT-IR spectra have shown shifts in peak positions, indicating synergistic interactions between functional groups. These results demonstrated the possibility of using bio-extracts as non-toxic, sustainable industrial activity inhibitors.

## KEYWORDS

green corrosion inhibitors; bio-extracts; physisorption; spectroscopy; density functional theory

## 1. INTRODUCTION

Corrosion has remained a common problem in most markets, including the chemical, manufacturing, and allied industries, especially in acidic environments. Conventional synthetic corrosion inhibitors, such as chromates, nitrites, and organic amines, have been popular due to their efficiency. Their impacts on ecosystems have included bioaccumulation capacity and acute toxicity to aquatic organisms, resulting in strict regulatory limitations in directives such as the REACH directive in Europe and EPA guidelines in the United States. This reaction has sparked interest in green inhibitors of renewable plant origin that offer biodegradability, cost-effectiveness, and inherent heteroatomic binding properties (e.g., N, O, S) that are conducive to metal surface adsorption. Past reviews highlight the significance of plant leaves in controlling the corrosion of both ferrous and non-ferrous metals in chloride- and sulphate-containing environments (Ifediorah E. I et al., 2025). The inhibition efficiencies of extracts of Carica papaya (papaya), Vernonia amygdalina (bitter leaf), and Annona muricata (soursop) from sub-Saharan Africa have shown potential for corrosion inhibition. For example, Carica papaya leaf extract (CPL) prevents mild steel corrosion in HCl due to the presence of alkaloids and flavonoids in the chelate complex. A. amygdalina protects copper through multi-techniques, and A. muricata protects copper in produced water through adsorption isotherms. Despite these developments, gaps have been identified in evaluations that combine synthesis optimisation, multi-technique validation, and quantum-chemical understanding, especially for multi-metal systems in dual acids (HCl/H<sub>2</sub>SO<sub>4</sub>). This study will address these gaps through the synthesis of ethanol-based bio-extracts of CPL, VGL, and AML and the rigorous testing of their inhibitory effects on aluminium (Al), mild steel (MS), and

zinc (Zn) in 1 M HCl and H<sub>2</sub>SO<sub>4</sub>. Objectives include: phytochemical and spectroscopic characterisation to determine active constituents; quantitative evaluation using thermometry and SEM. The article is useful for sustainable corrosion mitigation in chemical engineering.

## 2. MATERIALS AND METHODS

## 2.1 Materials and Plant Extract Synthesis

HCl, H<sub>2</sub>SO<sub>4</sub>, Acetone (Analytical grade), Mild steel, Emery Paper, Beaker, Philtre paper, Thermometer, Thermostatic bath, FTIR Spectrophotometer Shimadzu, Model IR affinity GCMS-QP2010 plus Shimadzu, Japan SEM (Model: Rhenom Prox, Phenom World Eindhoven, Netherlands), Potentiostat/galvanostat 263 electrochemical work station.

The leaves of Carica Papaya were collected in Ihembosi and Okija, Anambra State, Nigeria. The leaves were dried in the sun for four days. The leaves were dried, ground to increase surface area, and stored in an airtight container. In each extraction process, 30g of ground leaves was weighed and impregnated with 1000 ml of ethanol for 48 hrs. After 48 hours, the two plant mixtures were filtered. A mixture of the ethanol and the plant extract is obtained as the filtrate. The ethanol extract was concentrated, and the solvent was removed by distillation. The plant extract was weighed and used in the corrosion inhibition study.

Mild steel and Zinc coupons and sheets were washed and polished with emery paper to create a shiny surface. The coupons were degreased in acetone to remove all oil and organic impurities, then rinsed in distilled water, left to dry in air, and stored in desiccators. The electronic weighing balance was used to weigh all coupons, and the initial weight for each was recorded.

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## 2.2 Phytochemical Analysis of the Extracts

The solvent-free extract obtained was subjected to qualitative tests to identify the various plant constituents present in the sample. The characterisation of genotypes is qualitative using phytochemicals; data were gathered on the presence or absence of plant metabolites: essential oils, anthraquinones, base alkaloids, weakly basic alkaloids, phenols and tannins, quaternary salts, flavonoids, and saponins. Determinations are done on materials that are shadow-dried and reduced into a dust form by passing through a laboratory mill. Therefore, these processed products are subjected to hexane 10 times to remove fats. Following this process, the materials are stored in hexane, filtered, and left to stand for 5 to 7 days to allow the remaining hexane to evaporate. This processed material was covered with ethanol and held overnight. Several filtrations were then conducted until the liquid material changed to a hyaline appearance. It is with the help of this liquid that assays are done to establish the presence or absence of each of the above-mentioned metabolites.

For base and weakly basic alkaloids and quaternary salts, the ethanol extract was evaporated to dryness, then 10% hydrochloric acid was added. A separation funnel was used to pass through mixed material. The initial isolation is done using chloroform. The aqueous volume left in the funnel was treated with a specified amount of ammonium hydroxide until the solution became basic, and the mixture was washed with chloroform to obtain the second phase. The third stage had been abandoned in the funnel. A small amount of each of the three phases was applied to silica gel plaques using a capillary tube, then sprayed with Dragendorff's reagent. Tests in which the phase was positive for alkaloids developed a black spot. Under this test, it was not difficult to identify the kind of alkaloids on each step (first, second, or third) that reacted to the Dragendorff compound. The actions were confirmed by subjecting the three stages to reaction with Meyer's reagent, which resulted in a white precipitate and a small portion of the raw extract.

The Quantitative Tests for Phytochemicals were carried out to determine the Terpenoids, Flavonoids, Tannins, Alkaloids, Steroids, Cardiac Glycosides, Phytates, Phenols/Phenolics/Phenolic Acids, Saponins

## 2.3 FT IR Analysis of Plant Extracts

The metal samples were immersed in the acidic media in the presence of the plant extracts. At the end of the corrosion study, the corrosion products in the beakers were collected using sample bottles. A SHIMADZU FT-IR spectrophotometer (model IR Affinity-1, 5/NA 2137470136 SI) was used to determine the functional groups of the extracts (pure) and corrosion products. A comparative analysis of the various FTIR peaks was carried out to determine the exact functional groups involved in the corrosion inhibition process.

The analysis of the extracts shows variations in the peaks used to determine their functional groups (Omotioma and Onukwuli, 2015). This analysis was carried out at the National Centre for Renewable Energy at the University of Nigeria.

## 2.4 GC-MS Analysis of Plant Extracts

GC-MS analysis was carried out on a Mass Spectrophotometer Model No

QP2010 plus Shimadzu, Japan. The carrier gas used was Helium at a flow rate of 0.5ml/min. 1µl sample injection volume was utilised. The inlet temperature was maintained at 250 °C. The oven temperature was initially programmed to 80°C for 4 min, then increased to 240°C. And then programmed to increase to 280°C. Total run time was 90 min. The MS transfer line was maintained at 200°C. The source temperature was maintained at 180°C. The peaks in the chromatogram were integrated and compared with the GC-MS library database.

## 2.5 Corrosion Inhibition Evaluation

Thermometric Method of the Corrosion Inhibition Study

The measurements were carried out using a thermostat set at 30 °C for aluminium, mild steel, and zinc in free and inhibited HCl and H<sub>2</sub>SO<sub>4</sub>. The temperatures of the system containing the metals and the test solution were recorded regularly until a steady temperature was obtained. The reaction number (RN) was evaluated using Equation (1)

$$RN = \frac{T_m - T_i}{t} \quad (1)$$

Where T<sub>m</sub> and T<sub>i</sub> are the maximum and initial temperatures (in °C), respectively, and

It is the time in minutes elapsed to reach T<sub>m</sub>.

The inhibitor efficiency was determined using Equation (2)

$$IE\% = \left(1 - \frac{RN_{add}}{RN_{free}}\right) \times 100 \quad (2)$$

Where RN<sub>free</sub> and RN<sub>add</sub> are the reaction numbers for the metal dissolution in free and inhibited corrosive medium, respectively.

## 2.6 Scanning Electron Microscopy Analysis of Surface Metals.

The test specimens were immersed in 100 mL of 0.5 M H<sub>2</sub>SO<sub>4</sub> with extract concentrations of 0.2 g L<sup>-1</sup> and 1.2 g L<sup>-1</sup> for 15 hrs. After the experiment, the specimens were washed with distilled water, dried, and examined using a surface electron microscope (SEM), Phenom Prox, MVE 016477830, manufactured by Phenom World, Eindhoven, the Netherlands.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Phytochemical Analysis of Plant Extracts

Results of the qualitative and quantitative analysis of phytochemicals of the leaves are presented in Table 1. The *Annona muricata* leaf is found to be rich in terpenoids and alkaloids, the *Carica papaya* leaf is rich in tannins and flavonoids, and *Vernonia amygdalina* leaf is rich in Flavonoids, terpenoids and alkaloids. The presence of the phytochemicals indicates that the leaf extract possesses corrosion-inhibiting properties. This observation is consistent with previous reports, indicating that corrosion control of metals is achieved through the presence of corrosion-inhibiting features (Ifediorah E. I et al., 2025). So, the *Annona muricata*, *Carica papaya*, and *Vernonia amygdalina* leaf extracts are appropriate green corrosion inhibitors.

**Table 1:** Phytochemicals of *Annona*, *Vernonia* and *Carica papaya* leaves

Phytochemicals	<i>Annona muricata</i>	<i>Carica papaya</i> (Qualitative value and sign)	<i>Vernonia amygdalina</i>
Alkaloids (mg/100g)	785	630	175
	765 ++	660 +++	150 ++
	785	645	185
Cardiac Glycosides (mg/100g)	10 -	10 +	75 -
	10	5	60
	15	5	55
Flavonoids (mg/100g)	405 ++	985 +	640 +++
	420	960	660
	415	975	635

**Table 1 (cont):** Phytochemicals of *Annona*, *Vernonia* and *Carica papaya* leaves

Phenolics (GAE/g)	67.1 +++	70.3 +++	75.4 +++
	66.8	70.5	75.7
	66.6	70.2	75.3
Phytates (mg/100g)	50 +	10 +	40 -
	45	20	35
	55	15	40
Saponins (mg/100g)	85 +	130 -	125 +
	85	125	105
	80	140	130
Terpenoids (mg/100g)	960 +++	515 ++	495 ++
	990	545	485
	985	520	480
Steroids (mg/100g)	265	95	140
	280 ++	80 ++	165 +
	255	90	125
Tannins (mg/100g)	490 ++	1165 ++	160 +++
	475	1155	140
	495	1130	185

-. (too little to be observed qualitatively), + (in traces), ++ (concentrated) and +++ (highly concentrated)

### 3.2 Results of the FTIR analysis of the Plant leaves extract

Functional group of the plant extracts. A comparative analysis of the FTIR peaks was performed to identify the functional groups involved in the corrosion process. The

Figure 1 - 3 shows the results of the FTIR analysis of the leaf extracts.

In Figure 1 (*Carica papaya* FTIR analysis result), wave bands 3543.24 $\text{cm}^{-1}$  to 3852.0 $\text{cm}^{-1}$  represent strong and broad stretch bond of aliphatic and aromatic 3261.46 to 3477.62 $\text{cm}^{-1}$  are for medium and often broad stretch of amines and amides.

The wave bands at 3053.02 $\text{cm}^{-1}$  and 3142.8 $\text{cm}^{-1}$  are variable stretches of alkyl and aldehyde groups. The wave band at 2971.96 $\text{cm}^{-1}$  corresponds to a strong, very broad stretch of the carboxylic acid (free alcohol bond). Wave bands of 2751.94 $\text{cm}^{-1}$ , 2829.14 $\text{cm}^{-1}$  are two-peaked medium-stretched bonds of aldehyde. 2458.58 $\text{cm}^{-1}$  waveband range of 2674.74 $\text{cm}^{-1}$  is strong and very sharp stretch bond of carboxylic acid (alcohol). The waveband from 2404.54 $\text{cm}^{-1}$  to 2030.12 $\text{cm}^{-1}$  represents a variable, sharp stretch bond of alkyne and nitrite. The waveband from 1837.48 $\text{cm}^{-1}$  to 1658.56 $\text{cm}^{-1}$  is strongly representative of the stretch bond of acids, esters, anhydrides, and aldehydes. The wave bands at 1597.8 $\text{cm}^{-1}$  and 1439.54 $\text{cm}^{-1}$  are composed of multiple sharp, medium peaks, indicating stretching of the aromatic bond.

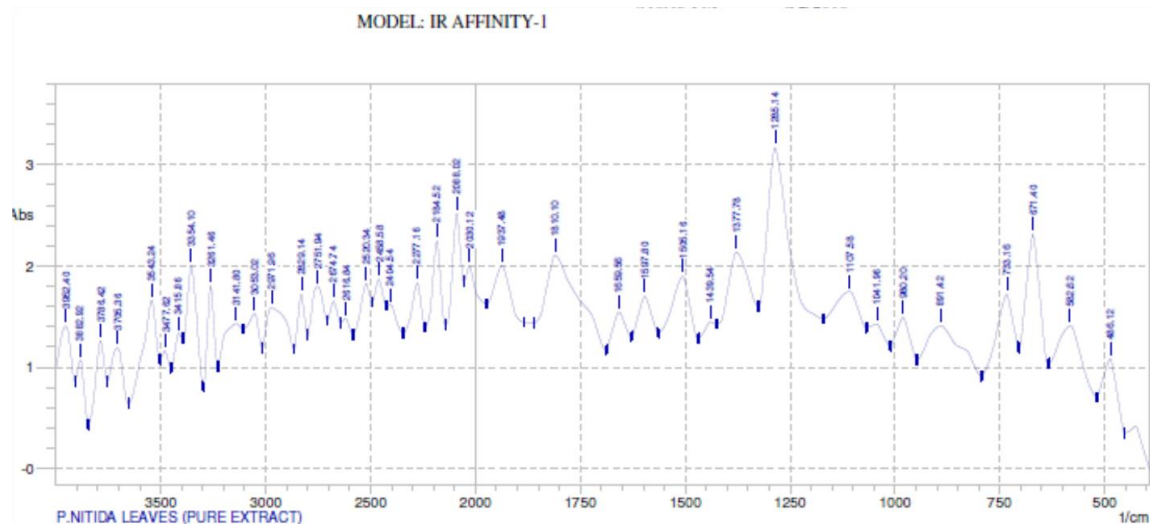
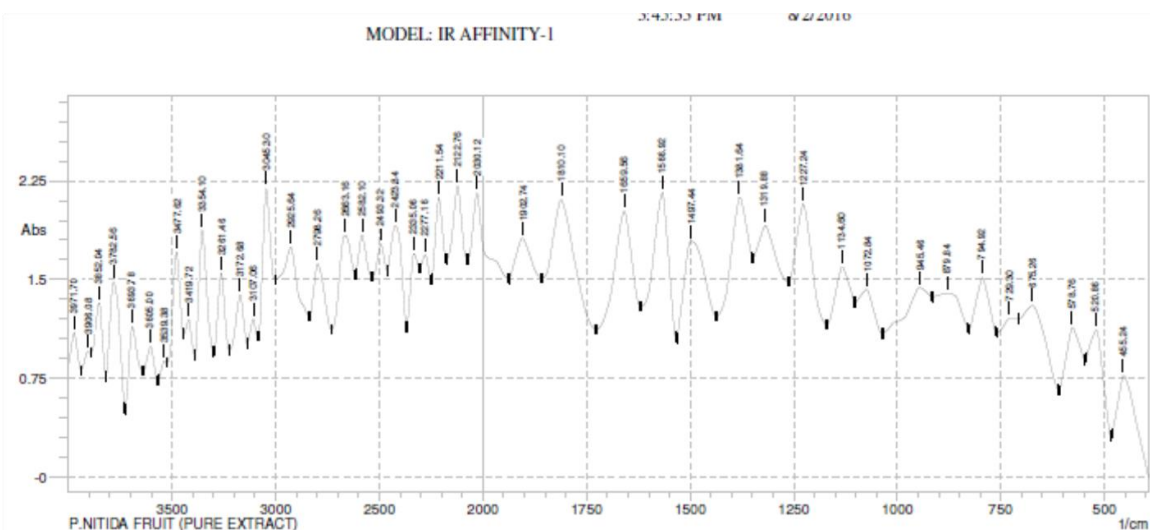
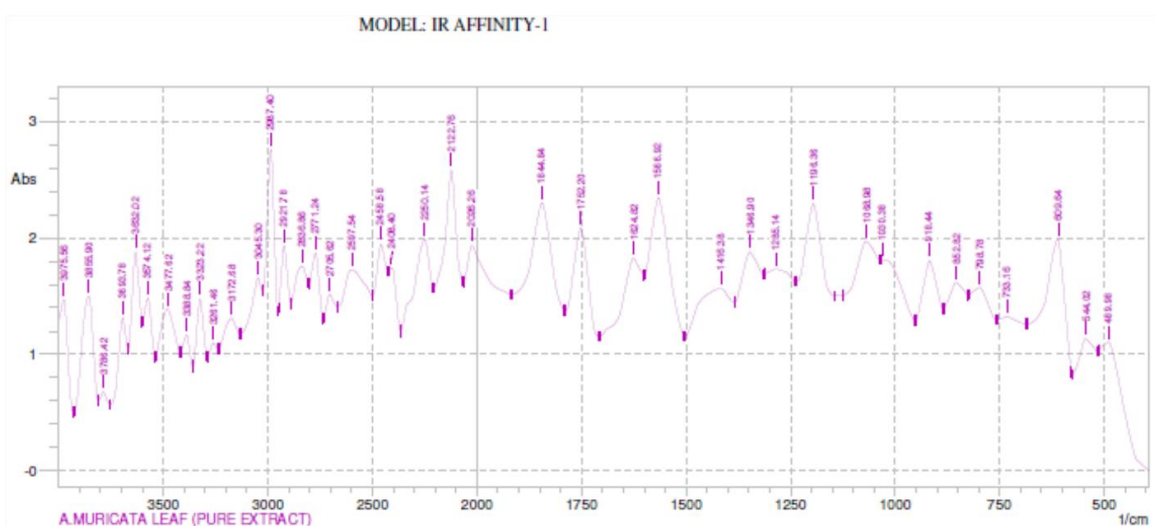
In Figure 2 (FTIR analysis result of *Vernonia amygdalina*), wave bands of 3539.38 $\text{cm}^{-1}$  to 3971.7 $\text{cm}^{-1}$  represent the strong, sharp and broad free bond of alcohol. The wave band at 3354.1 $\text{cm}^{-1}$  to 3477.62 $\text{cm}^{-1}$  represents a medium-to-broad stretch of the amine or amide group, and the band at 3261.46 $\text{cm}^{-1}$  represents a strong, sharp bond of an alkyne. The 2925.64-3172.68 waveband range represents a variable stretch of alkyl and aromatic groups. The wave band from 2582.1 $\text{cm}^{-1}$  to 2798.26 $\text{cm}^{-1}$  represents a very broad signal centred near the 3,000 $\text{cm}^{-1}$  free bond

wave band of alcohol. Wave band 7211.44 $\text{cm}^{-1}$  to 2493.32 $\text{cm}^{-1}$  represents a variable and sharp stretch of alkenes and nitrite. The 2030.12 $\text{cm}^{-1}$  and 2122.76 $\text{cm}^{-1}$  bands correspond to the variable- and sharp-stretch bonds of alkynes. That of 1659.56 $\text{cm}^{-1}$  to 1902.74 $\text{cm}^{-1}$  stands for that of a very strong stretch of acids, esters, and anhydrides. The wave band at 1566.92 $\text{cm}^{-1}$  represents a medium-to-strong bond in amines and amides, while the wave bands at 1319.24 $\text{cm}^{-1}$  to 1497/44 $\text{cm}^{-1}$  represent strong alkyl stretching. The range of wave bands that stretched from 1072.84 $\text{cm}^{-1}$  to 1227.24 $\text{cm}^{-1}$  is a strong stretch of acids, esters, and anhydrides. PNS pure extract contains some essential organic compounds with strong inhibitory properties, capable of replacing O<sub>2</sub>, H<sub>2</sub>S, and O<sub>2</sub> with amines, nitrite, hydroxyl, and benzene, etc.

In Figure 3 (FTIR analysis result of *Annona muricata*), Wave bands at 3975.56 $\text{cm}^{-1}$  to 3388.84 $\text{cm}^{-1}$  represent strong, sharp stretches of free bonds of alcohol and phenol. 3261.4 $\text{cm}^{-1}$  to 3045.30 $\text{cm}^{-1}$  represents a broad stretch of acids and carboxylic groups. Wave band 2987.40 $\text{cm}^{-1}$  to 2836.85 $\text{cm}^{-1}$  is a variable stretch bond of the alkyl Sps C-H group. The band from 2771.2 $\text{cm}^{-1}$  to 2705.62 $\text{cm}^{-1}$  is a medium-intensity stretch of aldehydes. Sharp, variable wave bands at 2122.76 $\text{cm}^{-1}$  to 2024.6 $\text{cm}^{-1}$  are due to the stretch bond of alkynes and nitrite. The wave band at 1844.84 $\text{cm}^{-1}$  is very strong and represents an anhydride bond. The wave band at 1596.92 $\text{cm}^{-1}$  is for the medium- to strong-stretch bond of amines and

amides. The wave bands at 1416.38 $\text{cm}^{-1}$  are for medium- to strong-stretch bonds of alkenes, while the bands at 1285.14 $\text{cm}^{-1}$  and 1030.38 $\text{cm}^{-1}$  are strong and represent stretches of acids, esters, and ethers, respectively.

The identified functional groups include O-H stretch, N-H symmetric stretch, C-H stretch, CH<sub>3</sub> C-H bend, and C-F stretch. The presence of heteroatoms (N and O) indicates that the *Annona muricata*, *Carica papaya*, and *Vernonia amygdalina* leaves extracts are suitable inhibitive additives. (Ifediorah, E. I. et al., 2025, Omotioma and Onukwuli, 2016 and 2017).

Figure 1: FTIR spectrum of *Carica papaya* leafFigure 2: FTIR spectrum of *Vernoniaamy gladina* leafFigure 3: FTIR spectrum of *Annona muricata* leaf

### 3.3 GC-MS analysis of the plant leaves extract

#### 3.3.1 *Carica papaya* Leave

Peak 1 present toluene, formular  $C_7H_8$ , molweight 92, peak 2 assigned to cyclohexane, formular  $C_6H_{10}$ , molweight 98, peak 3 represent Hexane, formular  $C_6H_{14}$ , molweight 114, peak 4 indicates 1,3-cyclopentadiene, formular  $C_5H_8$ , molweight 82, peak 7 stands for undecene, formular  $C_{11}H_{20}$ , molweight 154, peak 8 indicates tridecane, formular  $C_{13}H_{26}$ , molweight 184, peak 9 stands for octane, formular  $C_8H_{18}$ , molweight 114, peak 10 represents tridecane, formular  $C_{13}H_{26}$ , molweight 184, peak 11 shows

hexadecane, formular  $C_{16}H_{34}$ , molweight 226, peak 12 assigned to tridecane, formular  $C_{13}H_{26}$ , molweight 184, peak 13 presents dodecene, formular  $C_{12}H_{22}$ , molweight 170, peak 14 shows tridecane, formular  $C_{13}H_{26}$ , molweight 184, peak 15 indicates tetradecene, formular  $C_{14}H_{28}$ , molweight 198, peak 16 shoes pentadecane, formular  $C_{15}H_{30}$ , molweight 210, peak 17 indicates tridecane, formular  $C_{13}H_{26}$ , molweight 184, peak 18 represents dodecene, formular  $C_{12}H_{22}$ , molweight 170, peak 19 assigned to octadecane, formular  $C_{18}H_{36}$ , molweight 254, peak 20 indicates tridecane, formular  $C_{13}H_{26}$ , molweight 184, peak 21 shows eicosane, formular  $C_{20}H_{42}$ ,



**Table 2:** Inhibition Efficiency of the Plant Extracts for Al in HCl Medium

Inh. conc. (g/l)	IE (%); CPL	IE (%); VGL	IE (%); AML
0.2	50.43	42.19	51.78
0.45	65.29	56.89	69.1
0.7	74.14	68.41	74.11
0.95	81.37	75.94	84.26
1.2	85.27	79.93	88.21

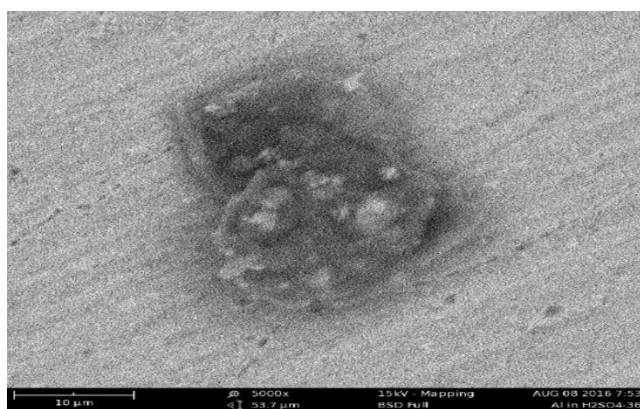
**Table 3:** Inhibition Efficiency of the Plant Extracts for Al in H<sub>2</sub>SO<sub>4</sub> Medium

Inh. conc. (g/l)	IE (%); CPL	IE (%); VGL	IE (%); AML
0.2	52.45	44.92	54.33
0.45	67.37	61.96	67.59
0.7	76.14	71.72	83.51
0.95	84.99	80.29	86.4
1.2	87.04	83.07	88.73

### 3.5 Scanning Electron Microscopy

Optical microscope tests of the metals aluminium, mild steel, and zinc revealed that when a smooth metal surface is immersed in an acidic medium for hours, in the absence of plant extract, corrosion pits form due to the acid's aggressive attack. A rough surface with flakes of dark colour was observed in (figure 5), which indicates deposition of corrosion products and chloride ions on the surface. But in the presence of an

inhibitor, smooth layers were recognized due to the adsorption of organic species present in the extract at most active sites. The image's brightness indicates good interaction between the inhibitor and the metal. Based on the information provided by the optical microscope, it can be concluded that plants extract protective metals in acidic environments, forming a protective film on the metal surface, leading to a considerable reduction in corrosion.

**Figure 5:** Al in HCl in absence of inhibitor

## 4. CONCLUSIONS

In synthesising and evaluating ethanol-based bio-extracts from Carica papaya leaves (CPL), this study establishes CPL as an eco-friendly corrosion inhibitor, achieving inhibition efficiencies (IE%) of 75%-85% across aluminium, mild steel, and zinc substrates in HCl and H<sub>2</sub>SO<sub>4</sub> media. This performs better than conventional synthetic inhibitors in various conditions, it is safe and does not pose any environmental liabilities such as bioaccumulation and aquatic toxicity, as mentioned in past literatures on plant-derived green corrosion inhibitors (GCIs), the efficiency stems from physisorptive synergy, wherein bioactive constituents predominantly alkaloids, flavonoids, phenols, and tannins facilitate electrostatic adsorption onto protonated metal surfaces, displacing solvated water molecules and forming a compact, hydrophobic barrier that impedes anodic dissolution and cathodic hydrogen evolution. GCMS, FTIR, and SEM analysis confirm the corrosion-inhibitive properties of the plant extracts. This dual analytical framework not only mitigates empirical trial-and-error but also fosters eco-efficient corrosion control. By reducing reliance on hazardous chromates or amines, estimated to contribute to industrial wastewater pollution, these bio-extracts will mitigate economic losses from corrosion while minimizing ecological degradation.

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