

**ELECTROCHEMICAL STUDIES OF THE CORROSION INHIBITORY POTENTIAL OF *Annona muricata* LEAVES EXTRACT ON ALUMINUM IN HYDROCHLORIC ACID MEDIUM**

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ABSTRACT

Owing to high toxicity and cost of most corrosion inhibitors, the search for inexpensive environmental friendly inhibitors is receiving attention. The influence of *Annona muricata* (AM) leaves extract on aluminum corrosion in HCl medium was studied using Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization (PDP). EIS results revealed substantial increase in charge transfer resistance (R_{ct}) from 2762 Ωcm² (devoid of AM) to 4719 Ωcm² (in company of 1000 ppm of AM). Percentage inhibition efficiency improved with upsurge in AM concentration. Potentiodynamic polarization measurements results showed tremendous decrease in corrosion current density from 1641 μAcm⁻² (in the absence of the inhibitor) to 920 μAcm⁻² when 1000 ppm of the inhibitor was introduced. The shift in corrosion potential in company of AM (in comparison with corrosion potential obtained without AM) was less than 85 mV, suggesting that AM showed mixed type performance. Percentage inhibition efficiency increased from 32 to 40 % in the presence of 500 ppm and 1000 ppm respectively. Gas chromatography mass spectroscopy (GCMS) analysis of AM leaves extract revealed the presence of diverse compounds such as 5-Tetradecene, Caryophyllene, Neophytadiene, Pentadecanoic acid and 9-Octadecenoic acid (Z)-, methyl ester. High concentration of *Annona muricata* leaves extract could be utilized as an environmental friendly inhibitor for aluminum deterioration in hydrochloric acid, based on the findings in this study.

Keywords: Corrosion inhibitor, Aluminum, *Annona muricata*, Electrochemical studies, Hydrochloric acid

INTRODUCTION

Aluminum is widely used in various industries for diverse application such as production of vessels, pipes, machinery and batteries. The utilization of aluminum for diverse applications could be attributed to its unique and interesting attributes such as light weight, affordability, high strength, excellent electrical conductivity and resistance to corrosion (Thacker and Ram, 2021). According to Bilgiç (2023), the most utilized metal after Fe is aluminum owing to its small mass (atomic), corrosion resistance and standard electrode potential. However, despite its natural corrosion resistance, aluminum is susceptible to corrosion in certain aggressive environments, particularly in acidic mediums like hydrochloric acid (which is often employed for chemical cleaning of metals). Corrosion of metals (i.e the weakening of metals due to interface between the metal and its environment) has caused huge economic loss, equipment breakdown and accidents (Bilgiç, 2023; Festus *et al.*, 2022; Mchihi *et al.*, 2024a). Since it is an undesirable occurrence, several mitigation techniques such as anodic and cathodic protection, utilization of corrosion resistant materials, coatings, and the usage of inhibitors have been employed for reducing or preventing corrosion. Among the explored mitigation techniques, the use of corrosion inhibitors appears to be gaining substantial attention. Research across various fields indicates their potential as a straight forward and efficient approach to protect metal surfaces from attack by corrosive substances. Inhibitors reduce or prevent corrosion when added to corrosive environments in small concentration (Thacker and Ram, 2021). Corrosion inhibitors can minimize or prevent corrosion by creating a protective film on the surface of metal which obstructs direct metal – corrosive agents connection. However, many corrosion inhibitors are

synthesized compounds that are toxic and expensive to produce. Therefore, there is a rising attention in the development of green inhibitors (especially extracts of plants) that are inexpensive, readily available and environmentally friendly (Alexander *et al.*, 2024; Mchihi *et al.*, 2023). Natural corrosion inhibitors are more biodegradable and available compared to synthesized inhibitors (Alexander *et al.*, 2024). *Annona muricata* (AM) is a typical fruit-bearing tree native to the Caribbean, Central America, and part of South America that belongs to the Annonaceae family. It is commonly known as soursop. It is extensively cultivated in India and is also cultivated in Nigeria. It is a plant known for its various medicinal properties. Different parts of *Annona muricata* such as fruit, leaves, seeds, bark and roots have been used for treatment of cough, intestinal parasites, liver ailments, inflammation, arthritis and diabetes, among other uses (Naik and Sellappan, 2019). According to a previous study, phytochemical screening of AM revealed that it contains saponins, alkaloids, proteins, amino acids, carbohydrates, phytosterols, quinines, lipids, phenolic, coumarins and flavonoids (Naik and Sellappan, 2019). Preliminary studies suggest that extracts of this plant have potential as green corrosion inhibitor (Vimala *et al.*, 2012). The inhibitory efficacy of AM leaves extract on mild steel in 1 N hydrochloric acid has been reported (Vimala *et al.*, 2012). The researchers established that AM is an outstanding inhibitor for mild steel in 1 N HCl and showed mixed type behavior. The use of AM as environmentally friendly inhibitor for protection of API 5L Grade A from corrosion attack under produced water has also been reported (Ayende *et al.*, 2023). The maximum inhibition efficiency recorded when 2 mL of the inhibitor was introduced (weight loss measurements) was 52.62 % (Ayende *et al.*, 2023). They also

reported that *annona muricata* leaves acted as a mixed type inhibitor which agrees with Vimala *et al.* (2012). Alexander *et al.* (2024) also reported leaves extract of AM as inhibitor for Zn corrosion in NaCl medium. They concluded that alkaloids from AM leaves could be utilised as effective inhibitors for corrosion of Zn in sodium chloride. The use of other plants as inhibitors for Aluminum deterioration in diverse media has also been reported (Obi-Egbedi *et al.*, 2012; Obot *et al.*, 2011; Ukpe, 2019; Sharma *et al.*, 2015; Singh *et al.*, 2012; Ennouri *et al.*, 2017; Fouda *et al.*, 2020). The aim of this study is to investigate the efficacy of AM leaves extract as environmental friendly corrosion inhibitor for aluminum in 1 M HCl medium using electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP).

MATERIALS AND METHODS

Sampling and extraction procedure

Leaves of AM were collected from Lufuwape community, Ogun state Nigeria. The leaves were identified at Department of Botany, University of Lagos Nigeria and the number 10100 was assigned. The leaves were dried for three days in the laboratory, followed by pulverization. The powdered sample obtained was stored in an air-tight container. 20 g of the powder was transferred to a sample bottle containing 200 mL of ethanol. The mixture was then placed in a sonicator for 10 minutes. After sonication, the mixture was filtered and centrifuged to remove debris. A portion of the extract obtained at this stage was used for Gas Chromatography Mass Spectrometry (GC-MS) and the remaining larger portion was concentrated with the aid of rotary evaporator to obtain the extract that was used for electrochemical studies. The extraction procedure described in this work is a modification of the procedure reported by Jayachandran *et al.* (2021).

Gas Chromatography Mass Spectrometry (GC-MS)

GCMS scrutiny of the ethanolic AM leaves extract was achieved with an Agilent 5977B GC/MSD scheme coupled with Agilent 8860 auto-sampler, a Gas Chromatograph interfaced to a Mass Spectrometer (GC-MS) furnished with an Elite-5MS (5% diphenyl/95% dimethyl polysiloxane) fused capillary column (30 × 0.25 μm ID × 0.25 μm df). 99.9 % He gas was utilised as carrier gas and an injection volume of 1 μl was utilized (a split ratio of 10:1). The He gas was utilized at steady flow rate of 1 mL/min. The injector temperature was maintained at 573 K, while 523 K was the ion-source temperature. The temperature of oven was programmed from 373 K (isothermal for 0.5 min), with an increase of 293 K/min to 553 K (2.5 min). Mass spectra were

taken at 70 eV. The solvent delay was 0 -180 seconds, and the total GC/MS running time was 21.33 min.

Electrochemical Investigation

The electrochemical assessments were carried out using a cell assembly featuring three electrodes: a 1cm² exposed aluminum surface as the working electrode (WE), Ag/AgCl as the reference electrode (RE) with reported potentials relative to Ag/AgCl, and a platinum wire serving the counter electrode purpose. The corrosive medium used for the investigation was 1 M HCl solution. To ensure the stability of the system and minimize the impact of charging current, the initial step involved determining the free dissolution potential of aluminum. This was achieved by recording the open circuit potential (OCP) for 1800 seconds (30 minutes). The dissolution process continued until a stable OCP was attained, indicating the metal's equilibrium potential, and subsequently, electrochemical tests were conducted under OCP conditions. For electrochemical impedance spectroscopy (EIS), tests were performed with a signal amplitude perturbation of 10 mV across a frequency range of 100 kHz to 10 MHz. Potentiodynamic polarization investigation was carried out within potential range of -250 mV and +250 mV, at a scanning rate of 0.2 mVs⁻¹. Subsequently, the obtained data were analyzed using Zsimpwin 3.2 model and EC-lab. Equation 1 and 2 were utilized to calculate inhibition efficiency using PDP and EIS data respectively (Haque *et al.*, 2023).

$$\%IE = \frac{I_{\text{corr}}^{\circ} - I_{\text{corr}}}{I_{\text{corr}}^{\circ}} \times 100 \quad (1)$$

$$\%IE = \frac{R_{\text{ct}} - R_{\text{ct}}^{\circ}}{R_{\text{ct}}} \times 100 \quad (2)$$

Where R_{ct} = resistance to charge transfer in company of AM, R_{ct}° = charge transfer resistance devoid of AM, I_{corr}° = corrosion current density in the absence of AM while I_{corr} = corrosion current density in the presence of AM.

RESULTS AND DISCUSSION

Open Circuit Potential

Open circuit potential (OCP) versus time profile of aluminum in bare HCl and AM inhibited HCl is presented in Figure 1. Introduction of 500 ppm of AM resulted to slight OCP shift in the negative direction. On the other hand, introduction of 1000 ppm of AM resulted to shift of OCP towards positive direction. This observed shift in OCP suggests that AM has an influence on the corrosion of aluminum in 1 M HCl (Mchihi *et al.*, 2024a). This influence could be a result of adsorption of AM on the surface of aluminum, resulting to creation of protective film.

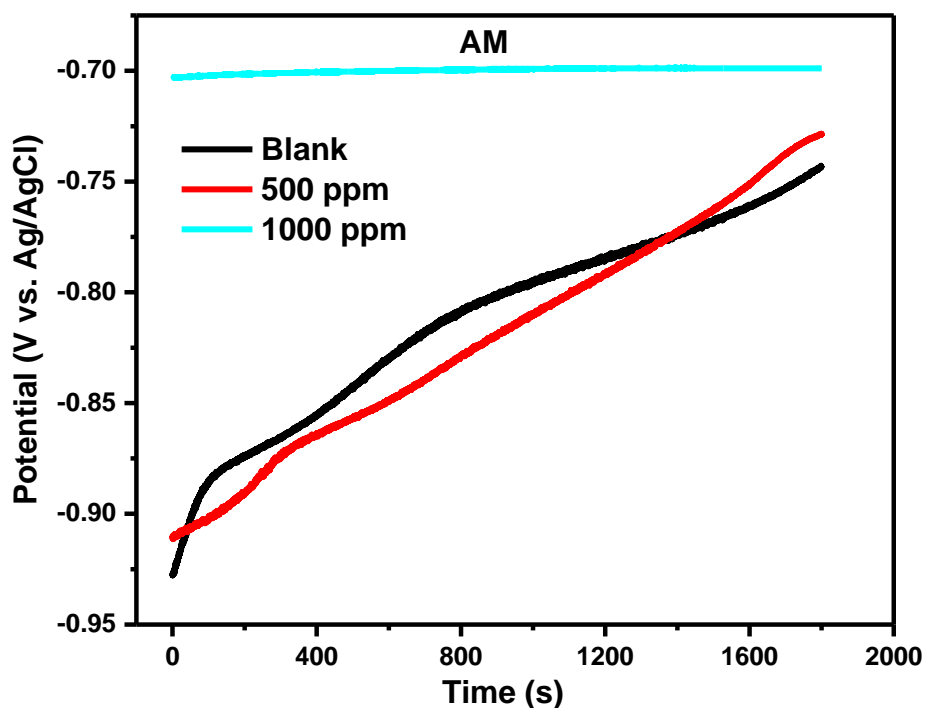


Figure 1: OCP versus time plot for aluminum in 1 M HCl devoid of AM and in company of different concentrations of AM

Potentiodynamic Polarization

Potentiodynamic plots (also known as tafel plot) for aluminum in 1 M HCl in the presence and absence of AM leaves extract is shown in Figure 2. Parameters obtained from polarization studies such as corrosion current density (I_{corr}), anodic and cathodic tafel slopes (β_a and β_c), corrosion potential (E_{corr}) and the inhibition efficiency are presented in Table 1. It is obvious that I_{corr} values reduced consistently with upsurge in concentration of AM, suggesting the establishment of a shielding surface film by AM molecules on aluminum (Mchihi *et al.*, 2024b). Variation in the E_{corr}

values was also noticed when AM was introduced. A displacement exceeding 85 mV indicates predominantly cathodic or anodic inhibitor, while values below 85 mV suggest a mixed-type inhibitor. In this study, the maximum shift in the E_{corr} value is below 85 mV, suggesting that AM operated as a mixed-type inhibitor (Odozi *et al.*, 2019; Odozi *et al.*, 2021; Shalabi *et al.*, 2014; Thacker and Ram, 2021). The inhibition efficiency computed from PDP measurements increased with upsurge in AM concentration which could be due to upsurge in adsorbed AM molecules. This observation is consistent with other reports (Odozi *et al.*, 2021)

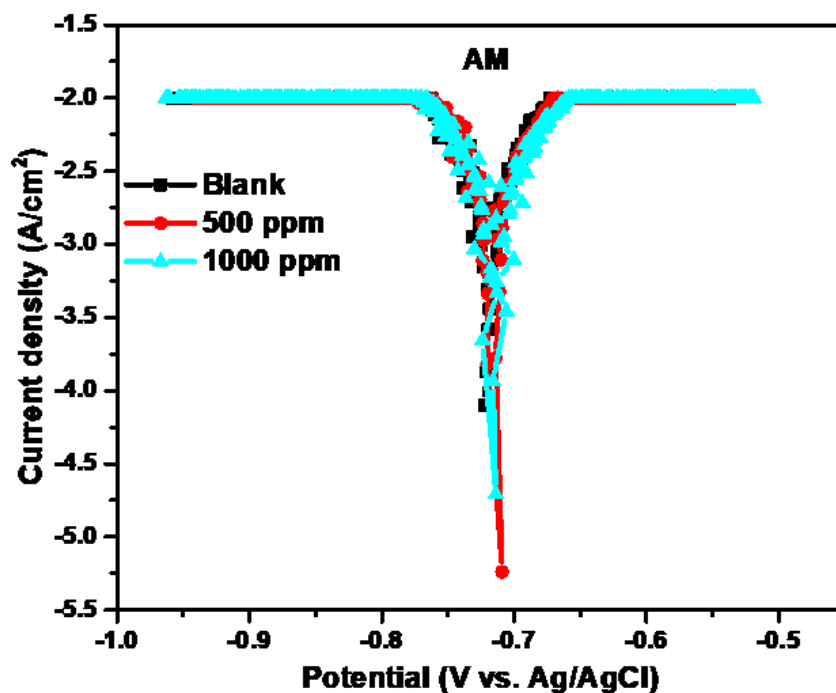


Figure 2: Tafel plots for aluminum in 1 M HCl devoid of AM and in company of AM.

Table 1: PDP parameters for aluminum in 1 M HCl devoid of AM and in company of AM.

Inhibitor concentration	E_{corr} (mV/SCE)	I_{corr} (μAcm^{-2})	β_a (mVdec ⁻¹)	β_c (mVdec ⁻¹)	θ	IE (%)
Blank	-720	1641	42	98		
500 ppm	-721	1122	53	71	0.32	32
1000 ppm	-720	920	78	63	0.40	40

Electrochemical Impedance Spectroscopy

Nyquist plot (also known as impedance plot) for aluminum in 1 M HCl devoid of AM and in company of AM is shown in Figure 3. It can be clearly observed that nyquist diagrams are

larger in the presence of the AM compared to nyquist plot for aluminum in the bare acid, which indicate the inhibitory effect of AM.

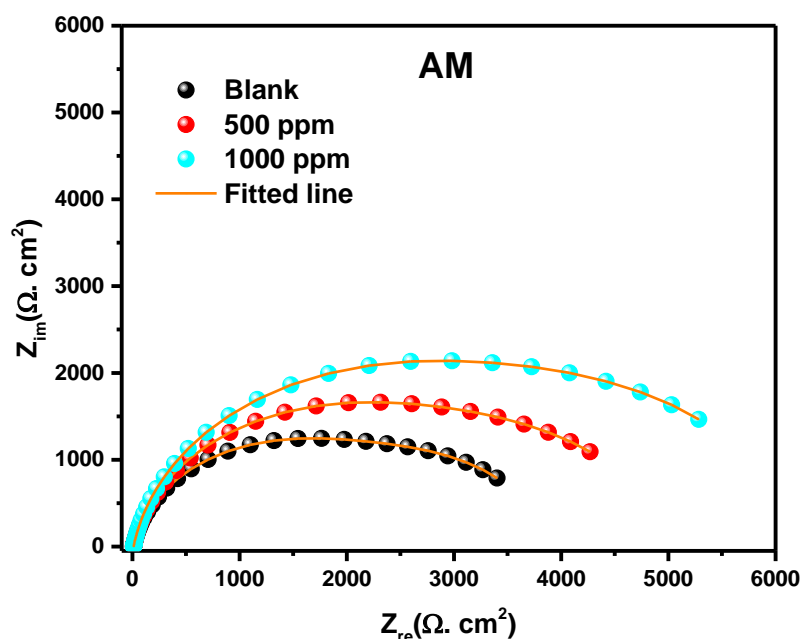


Figure 3: Nyquist plot for aluminum in 1 M HCl devoid of AM and in company of AM

The impedance diagram size (Figure 3) improved with corresponding upsurge of AM concentration, consequently increasing protection efficiency. Furthermore, the observed imperfection in semicircle shape of the impedance diagrams could be attributed to the non-homogeneity of the metal surface (Mchihi *et al.*, 2024a). The similarity in shape of semicircles suggest that the presence of AM did not alter the corrosion mechanism of aluminum in hydrochloric acid

(Eziuka *et al.*, 2023). The circuit that was utilized for fitting impedance data is shown in Figure 4, where R_s stands for solution resistance, R_{po} is pore resistance and CPE is the constant phase element. Notice that the data revealed two non-ideal capacitances owing to surface heterogeneity between the aluminum and the surrounding film (i.e CPE 1 and CPE 2) (Mchihi *et al.*, 2024b).

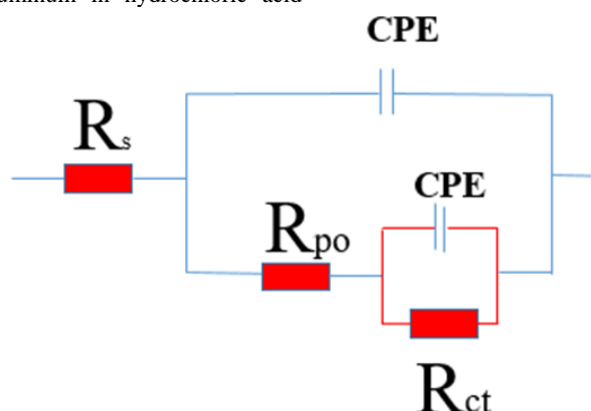


Figure 4: Electrical equivalent circuit employed to fit the EIS data

The parameters obtained from EIS study are presented in Table 2. The presence of AM improved the resistance to charge transfer, R_{ct} which could be a result of adsorption of AM on aluminum surface (Mchihi *et al.*, 2024b). The increase

in R_{ct} was AM concentration dependent. The upsurge in inhibition efficiency with AM concentration suggests a dose-dependent effect which could be due to upsurge in surface coverage (θ).

Table 2: EIS parameters for aluminum in 1M HCl in the presence and devoid of AM

System	R_s (Ωcm^2)	R_{po} (Ωcm^2)	CPE1 ($\mu\Omega^{-1}\text{s}^n \text{cm}^{-2} \times 10^{-5}$)	n1	R_{ct} (Ωcm^2)	CPE2 ($\mu\Omega^{-1}\text{s}^n \text{cm}^{-2} \times 10^{-5}$)	n1	θ	IE %
Blank	10.49	1113	5.63	0.89	2762	6.61	0.88		
500 ppm	10.5	1392	5.32	0.89	3623	5.94	0.83	0.23	23
1000ppm	10.48	1465	5.11	0.89	4719	5.55	0.90	0.37	37

Bode and phase angle plots for Aluminum in 1 M HCl devoid of AM and in the presence of different dosages of AM are presented in Figure 5. The plots shows increased area under

the curves when AM was introduced compared to bode and phase angle plots recorded for Aluminum in the bare HCl which supports the observation in nyquist plots.

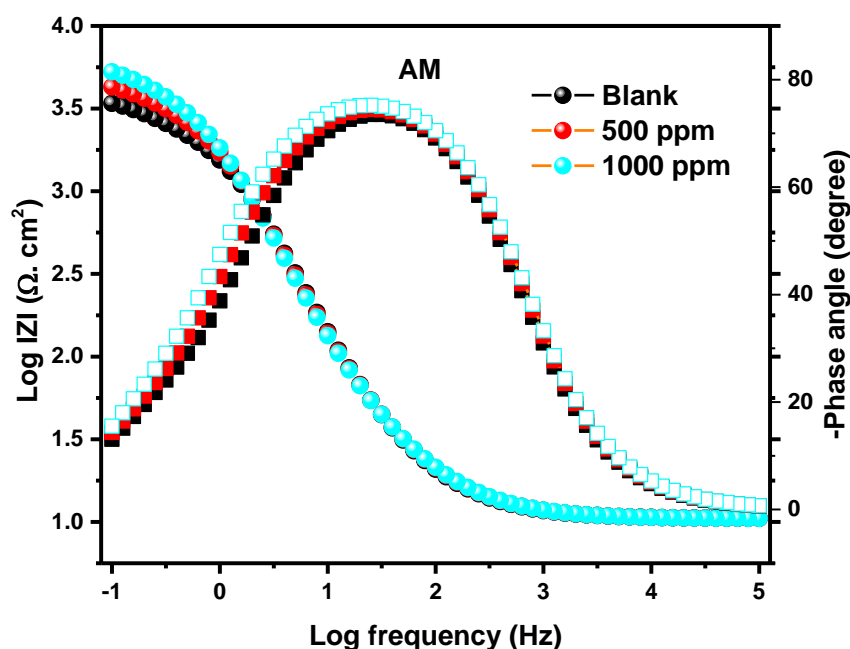


Figure 5: Bode and phase angle plots for Aluminum in 1 M HCl devoid of AM and in company of different dosages of AM.

GC-MS

The ethanolic extract of AM leaves chromatogram is presented in Figure 6. Several peaks were generated and characterized. Based on their retention times (RT), the compounds that were found in the extract include 5-Tetradecene, (E)- (RT = 5.542 min), Cyclotetradecane (RT = 7.877 min), Cyclohexane, 1-ethenyl-1-methyl-2, 4-bis(1-methylethenyl)- (RT = 7.957 min), Caryophyllene (RT = 8.283 min), alpha-Muurolene (RT = 9.118 min), Naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)- (9.347 min), 9-Octadecene, (E)- (RT = 9.982 min), 1-Heptadecene (RT = 11.813 min), Neophytadiene (RT = 12.214 min), 3-Eicosyne (RT = 12.426 min), 1-Tridecyne (RT = 12.580 min), Pentadecanoic acid, 14-methyl-, methyl ester (RT = 12.981), Pentadecanoic acid,

14-methyl-, methyl ester (RT = 12.981), Tetradecyl trifluoroacetate (RT = 13.484 min), Octadecanoic acid, ethyl ester (RT = 13.524 min), 9-Octadecenoic acid (Z)-, methyl ester (RT = 14.371 min), 13-Octadecenal, (Z)- (RT = 14.486 min), Methyl stearate (RT = 14.549 min), 9-Octadecenoic acid (RT = 14.549 min), 9-Octadecenoic acid (RT = 14.863 min), 2-Methyl-Z,Z-3,13-octadecadienol (RT = 14.915 min), Octadecanoic acid, ethyl ester (RT = 15.046 min), Carbonic acid, hexadecyl prop-1-en-2-yl ester (RT = 17.495 min) and 1-Octadecanesulphonyl chloride (RT = 17.553 min). The inhibitory effect of *Ammonia muricata* could be attributed to the presence of these phytochemicals. Some of these compounds contain hetero atoms that are known to facilitate adsorption of inhibitors on surface of metals which reduces or prevents corrosion (Alexander et al., 2024; Chioma et al., 2022)

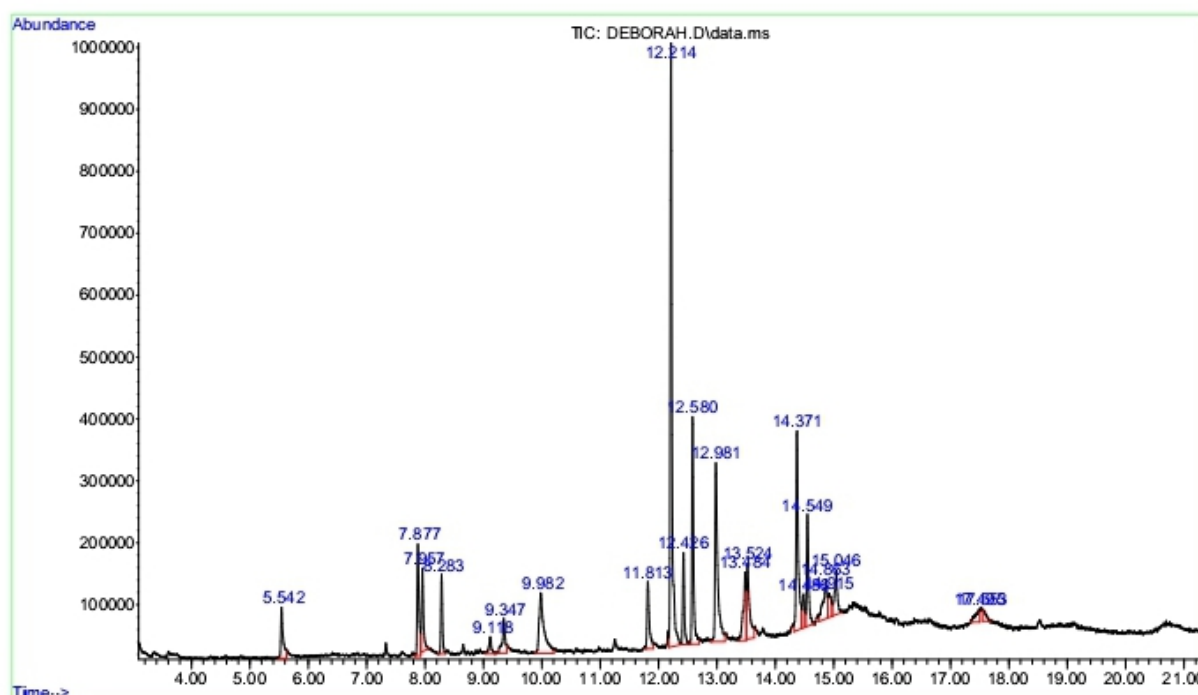


Figure 6: Chromatogram of ethanolic extract of *Annona muricata* leaves

CONCLUSION

Spectroscopy and polarization techniques were employed to probe the corrosion inhibitory effect of *Annona muricata* for aluminum in HCl medium. Electrochemical impedance spectroscopic studies revealed upsurge in charge transfer resistance values in the presence of *Annona muricata*. This increase in charge transfer depended on *Annona muricata* extract dosage. Potentiodynamic polarization outcomes exposed that corrosion current density declined when *Annona muricata* was introduced. Values of corrosion potential suggests that *Annona muricata* exhibited mixed type compartment. GCMS analysis revealed diverse phytochemicals present in the extract utilized for this study. The highest inhibition efficiency (40 %) was achieved when 1000 ppm of extract of *Annona muricata* was introduced. The concentration dependency of inhibition efficiency on concentration suggest that higher efficiency would be achieved by increasing inhibitor concentration.

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