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The Corrosion Inhibition Potential of Carbonized Orange Peel (COP) Extract as an Inhibitor for Mild Steel in Acid Solution

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ABSTRACT

[This study explores the corrosion inhibition potential of Carbonized orange peel (biomass) extract as an inhibitor for mild steel in acid solution, offering a low-cost, renewable alternative to conventional synthetic inhibitors. The extract, rich in polyphenols and oxygenated compounds. 0.1M of Hydrochloric HCl. acid solutions at 50°C was prepared and investigated via weight loss technique. UV–Visible spectroscopy showed progressive absorbance reduction with immersion time, indicating adsorption of active molecules, FTIR confirmed the preservation of O–H, C=O, and C–O–C groups in the surface film, suggesting stable interfacial attachment without structural degradation. Electrochemical analyses revealed mixed-type inhibition behaviour, with significant reductions in corrosion current density and increased charge transfer resistance, consistent with barrier film formation. SEM imaging confirmed surface protection, showing reduced pitting and smoother morphology compared to the uninhibited steel. The results indicate a predominantly physisorption mechanism, possibly enhanced by weak chemisorption. Comparable to literature-reported biomass inhibitors, this system offers environmental compatibility and economic advantages, demonstrating the potential of agricultural waste valorization for industrial corrosion control in acidic environments.]

1.0 INTRODUCTION

Corrosion normally occurs at a rate determined by an equilibrium between opposing electrochemical reactions. One reaction is the anodic reaction, in which a metal is oxidized, releasing electrons into the metal. The other is the cathodic reaction, in which a solution species (often O_2 or H^+) is reduced, removing electrons from the metal. When these two reactions are in equilibrium, the flow of electrons from each reaction is balanced, and no net electron flow (electrical current) occurs (Olumide, *et al.,* 2024).

Corrosion of mild steel is a complex electrochemical process influenced by various factors, including the presence of corrosive agents, such as acids. In acidic environments, corrosion typically involves the dissolution of iron (Fe) and the formation of iron ions (Fe^{2+}) along with the release of hydrogen gas (H_2). The process is influenced by factors like pH, temperature, and the concentration of corrosive species. Understanding the fundamental corrosion mechanisms in mild steel provides the basis for developing effective corrosion inhibition strategies (Al-Baghdadi and Al-Amiery, 2021). Corrosion inhibition methods aim to mitigate the destructive effects of corrosion by introducing chemical compounds known as inhibitors. These inhibitors form protective layers on the metal surface, reducing the rate of corrosion. Various types of inhibitors, including organic compounds, inorganic compounds, and mixed inhibitors, have been explored. In particular, organic corrosion inhibitors have gained attention for their ability to form stable adsorption layers on metal surfaces, hindering the corrosion process (Hanoon and Resen, 2021).

Mild steel was initially developed without a primary focus on corrosion resistance. However, as its use in manufacturing and construction has grown, one of the most significant challenges encountered is controlling its corrosion rate when exposed to various corrosive environments. Corrosion is a natural process that weakens metals by reducing the binding energy between atoms, leading to oxidation and electron loss. As the metal loses electrons, they flow through the material to another site, where they are consumed in a reduction reaction (Al-Amiery, 2021).

Mild steel is widely used in various industries, including construction, automotive, and manufacturing. However, one of the major challenges faced by mild steel is corrosion, particularly in acidic environments, which are common in industrial processes such as cleaning, pickling, and in acidic rain. Corrosion leads to degradation of the steel, compromising its structural integrity, efficiency, and service life. The use of corrosion inhibitors offers a potential solution to mitigate this issue. Corrosion inhibitors are chemical substances that, when added to a corrosive medium, reduce the corrosion rate by forming a protective film on the metal surface. This project aims to explore the effectiveness of orange peels corrosion inhibitors in preventing the corrosion of mild steel in acidic solutions.

2.0 Material and Method

2.1 Materials

The materials and reagent used for the analysis are presented in Table 1.

Table 1: Material and Reagents Used for the Research

S/N	Material/Reagents	Description	Grade	Source
1	Fresh orange peels	Inhibitor source	Organic	Local vendors
2	Mild steel	Corrosion substrate	Commercial grade	Auchi axis
3	Hydrochloric acid (HCl)	Corrosive medium (37% w/w)	Analytical	Chemical shop,Onitsha
4	Ethanol (C_2H_5OH, 95%)	Solvent for extraction	99.9% analytical grade	Chemical shop, Onitsha
5	Distilled water	Solution preparation and rinsing	Laboratory grade	Chemical shop, Onitsha
6	Acetone	Cleaning and drying agent	Analytical grade	Chemical shop, Onitsha

The Equipment and Apparatus used for the present study are also shown in Table 2.

Table 2: Equipment and Apparatus Used for the Study

S/N	Equipment	Description	Grade	Source
1	Weighing Balance	Measuring weights (±0.0001 g)	Analytical	Unit operations laboratory
2	Emery paper (grades 220–800)	Surface polishing of coupons	Commercial	Hardware store
3	Soxhlet Apparatus	Extracting orange peel compounds	Borosilicate glass	Megacorp
4	Rotary Evaporator	Solvent removal	Digital	Unit operations laboratory. Auchi poly
5	Oven	Drying peels and samples	0–250°C	Unit operations laboratory, Auchi poly
6	pH Meter	pH monitoring		Unit operations laboratory, Auchi poly
7	Crucible	For carbonizing	Nalgen 6000-30	Local vendors Auchi

8	Muffle furnace	For heating at a very high temperature	**Carbolite CWF 11/13**	Unit operations laboratory, Auchi poly
9	Water Bath	Temperature-controlled immersion	0–100°C	Unit operations laboratory, Auchi poly
10	Desiccators	Storage under dry conditions	Glass Pyrex	Unit operations laboratory, Auchi poly
11	UV-Vis Spectrophotometer	Inhibitor analysis	190–1100 nm	-
12	FTIR Spectrometer	Functional group detection	4000–400 cm^{-1}	-
13	Magnetic Stirrer	Homogenizing inhibitor-acid mix	Digital	Unit operations laboratory, Auchi poly
14	Beakers	Mixing vessel	Borosilicate glass	Unit operations laboratory, Auchi poly
15	PDP (Potentiodynamic Polarization):	for electrochemical corrosion rate and inhibition efficiency	Gamry Reference 600+, Scan rate: 1 mV/s,	-
16	SEM	surface morphology	Model: Phenom	-

2.2 Methods

2.2.1 Sample Collection and Processing of Orange Peels

Collection of fresh orange peels

Fresh orange peels were manually removed from ripe fruits collected from local vendors at Sabo axis Auchi, Edo State. Only healthy, unfermented, pesticide-free peels were selected.

Preparation and processing of orange peel inhibitor

The collected peels were first rinsed with distilled water to eliminate surface dirt and contaminants. They were then air-dried under shade for 7 days to minimize degradation of natural constituents. The air-dried peels were subsequently carbonized in a muffle furnace at **500 °C for 2 hours and 30 minutes** to enhance surface activity and improve adsorption characteristics. The carbonized material was allowed to **cool for 2 hours,** then **pounded into fine powder** using a clean mechanical mortar and pestle. The resultant powder was **sieved using a 0.5 mm mesh** to ensure particle size uniformity. To eliminate residual moisture, the sieved carbonized peel was further **oven-dried at 115 °C** for complete dehydration. The dried powder was stored in airtight containers to prevent moisture reabsorption and oxidation before extraction.

2.2.2 Soxhlet extraction of orange peel inhibitor

The Soxhlet extraction technique was adopted to isolate corrosion-inhibiting compounds from the carbonized orange peel powder. 20 g of the carbonized orange peel powder was weighed and placed in a thimble within the Soxhlet apparatus. 300 mL of 95% ethanol was poured into a 500 mL round-bottom flask serving as the solvent reservoir. The extraction was carried out for 6 hours at 78 °C **using** a controlled heating mantle. The resulting extract was then heated using a temperature controlled heating mantle at 45 °C to evaporate solvent from the solvent-oil mixture followed by gentle desiccator drying to eliminate any residual ethanol. The yield was recorded, and the final extract was kept for use in corrosion tests.

2.2.3 Preparation of mild steel coupons

The mild steel was obtained from local metal vendor in Auchi. Coupons were cut to 2.5 cm × 1.5 cm × 1.0 cm using a hacksaw. The mild steel was polished sequentially with sand paper, rinsed with de-ionized water and allowed to dry at room temperature.

After drying, the coupons were soaked in acetone for 45 minutes to remove residual contaminants, then sun-dried. Each was weighed using an analytical balance and stored in a desiccator before the corrosion test.

3.0 RESULTS AND DISCUSSION

3.1 Weight-Loss Measurements:

The traditional weight-loss measurements were performed. At temperatures ranging from 303 K to 333 K, cleaned, polished, and weighed mild-steel specimens were immersed in a 1.0 M HCl environment in the absence and presence of varied doses of the tested inhibitor for various exposure times (1, 5, 10, 24, and 48 h). To ensure that the tests could be replicated, each measurement was carried out five times. The corrosion rate and inhibition efficiency were calculated from Equations (1)

$$\mathrm{CR} = \frac{weight\ loss}{area \times density \times time} \tag{1}$$

Time (h): 1,5,10,24, and 48 respectively.

Density = 1.19g/ml

$$\mathrm{CR} = \frac{weight\ loss}{area \times density \times time}$$

At 1h; $\mathrm{CR1} = \frac{0.21}{3.75\times1.19\times1} = 0.047$

At 5h; $\mathrm{CR2} = \frac{0.13}{3.75\times1.19\times5} = 0.006$

At 10h; $\mathrm{CR3} = \frac{0.39}{3.75\times1.19\times10} = 0.009$

At 24h; $\mathrm{CR4} = \frac{0.56}{3.75\times1.19\times24} = 0.005$

At 48; $\mathrm{CR5} = \frac{0.67}{3.75\times1.19\times48} = 0.003$

Table 1: Weight Loss Summary

Specimen	Initial weight (w_1)	Final weight (w_1)	Weight loss	Corrosion rate (CR) $\mathrm{CR} = \frac{weight\ loss}{area \times density \times time}$ ($\mathrm{gcm}^{-3}h$)
1	99.80	99.38	0.21	0.047
2	100.28	100.15	0.13	0.006
3	100.42	100.03	0.39	0.009
4	102.58	102.02	0.56	0.005
5	101.02	101.35	0.67	0.003

Where W is the weight loss of the tested mild steel (gram), a is the tested mild-steel surface area (cm^2), t is the exposure time (h), and d is the tested mild-steel density (gcm^{-3});

The corrosion rate decreased as the inhibitor concentration increased, and the inhibition improved. This shows that, when the concentration rises, inhibitor molecules are essentially adsorbed to a higher extent onto the metal substrate, which results in broader surface coverage. As a result, it was clear that the performance of the inhibition was concentration dependent. Hence, the metal contact with the acid solution was limited. The PMBMH showed that the highest inhibition efficacy is at 48h, and the inhibition effectiveness was found to be 67%.

3.2 UV-Vis Analysis

The UV-Vis spectrum was obtained using a Shimadzu UV-180 UV/Visible scanning spectrophotometer to assess the potential inhibitory effect of the extract on the metal surface. This analysis involved monitoring the light absorption behavior of the extract solution in contact with the metal, to evaluate how effectively the extract reduced the corrosion rate.

Following the immersion of the metal specimen in the extract solution, the mixture was analyzed with the UV-Vis spectrophotometer to identify any chemical interactions between the extract's components and the metal. As illustrated in Figure 1, distinct absorption peaks appeared at various wavelengths, corresponding to both saturated and unsaturated compounds present in the extract. Notably, peak intensities gradually decreased over time, suggesting a continuous depletion of extract components from the solution due to their adsorption onto the metal surface. Furthermore, Figure 1 showed no emergence of new peaks in the spectrum, indicating the absence of any new chemical species. This suggests that the interaction between the extract and the metal surface was physical in nature, supporting the conclusion that the adsorption process was physisorption rather than chemisorption.

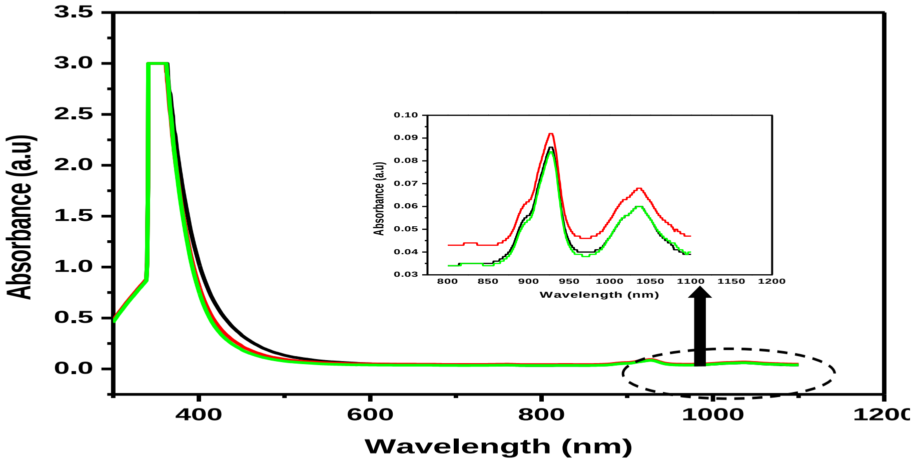

Figure 1: UV-Vis Spectral of Solution Extract of Metal Coupon

3.3 Electrochemical analysis

Electrochemical measurements were performed using a single-chamber cell setup comprising a mild steel working electrode with an exposed area of 1 cm², a saturated calomel electrode (SCE) as the reference, and a platinum rod serving as the counter electrode. The tests were carried out at 25 °C in a 0.5 M HCl solution containing 100 ppm of the inhibitor extract, using a Metrohm Autolab system controlled by Nova 2.1.1 software.

Table 2: Electrochemical Parameters Obtained from the Electrochemical Studies of Orange Peel Inhibitor Extracts at 303K.

Conc. (mg/L)	**Ecorr. (mv)**	**Icorr. (µA/cm2)**	**βa**	**βc**	**C.R (mmpy)**	**I.E %**
Blank	-472.78	20.349	155.463	268.462	14.339	NA
50	-413.05	20.296	140.868	250.288	10.626	25.89
100	-480.05	20.264	90.314	179.282	8.362	41.54
150	-468.07	8.813	97.408	172.158	6.210	56.69
200	-457.27	4.029	67.958	164.084	2.839	80.20

Figure 2 and Table 2 showed that the cathodic and anodic reactions are reduced as the concentration of the inhibitor increases by decreasing the polarization of the current densities of both side of the curve. Corrosion rate decreased steadily from 14.339 mmpy to 2.839 mmpy as the concentration of the inhibitor increased from 50 mg/L to 200 mg/L while the inhibition efficiency increased from 25.89% to 80.20%. The cathodic and the anodic slopes were altered showing reduction in metal dissolution and decrease in hydrogen reaction dissolution confirmed that orange peel has a mixed inhibitor.

After immersing the metal specimen in the solution for one hour, polarization studies were conducted by sweeping the potential from the open circuit potential (OCP) at a rate of 1 mV/s. Electrochemical Impedance Spectroscopy (EIS) was also performed, spanning frequencies from 100 kHz to 0.01 Hz with an applied AC amplitude of 0.005 V. Corrosion current densities were obtained by applying the corrosion potential to the linear Tafel regions of the anodic and cathodic curves. EIS data were analyzed using ZSimWin 3.60 software to extract impedance parameters and model the system with appropriate equivalent electrical circuits.

The corrosion rates of mild steel in aggressive solutions can be obtained using potentiodynamic polarization curve was thus applied to examine the effect of inhibitor extract concentration. The percentages of inhibition efficiency and values of associated electrochemical parameters were highlighted in the Table 2. Investigation of the obtained data disclosed that the Icorr values decrease steadily in the presence of orange peels extract with increasing inhibitor concentration. The Ecorr values tilted to more positive potentials in the presence of inhibitor concentration. The inhibition of mild steel orange peel extract on mild steel in 1M HCl was monitored by measuring the cathodic and anodic behavior change of coupon which is equivalent to hydrogen reduction and metal oxidation respectively and the sweep is conducted at the rate of 2mV/s.

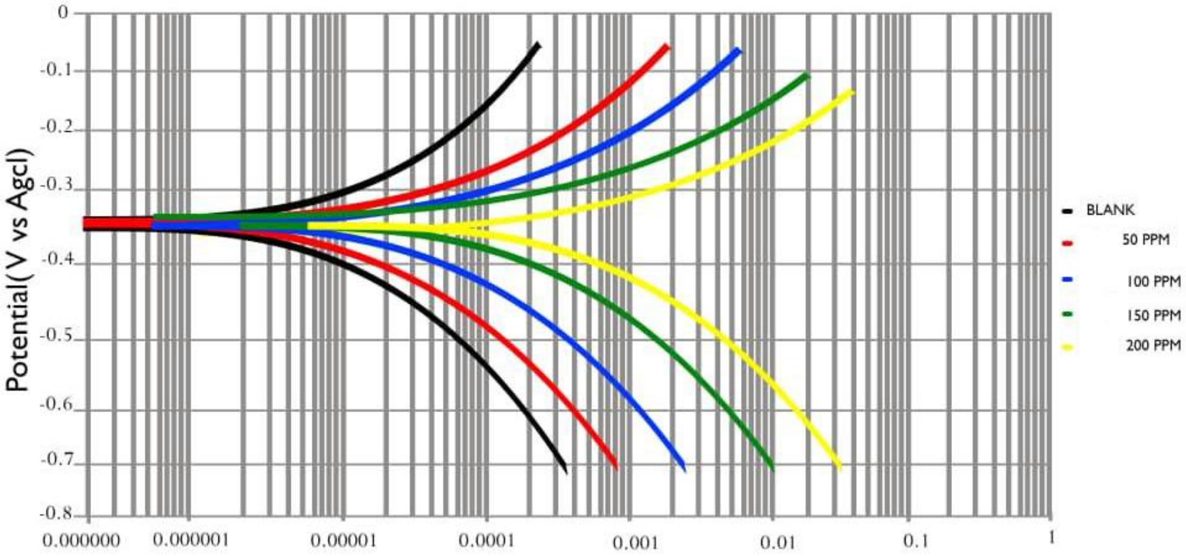

Figure 2: Potentiodynamic Polarimetry Sweep Curve of Mild Steel Immersed in 1M HCl in the Presence and Absence of Orange Peel Inhibitor at Room Temperature.

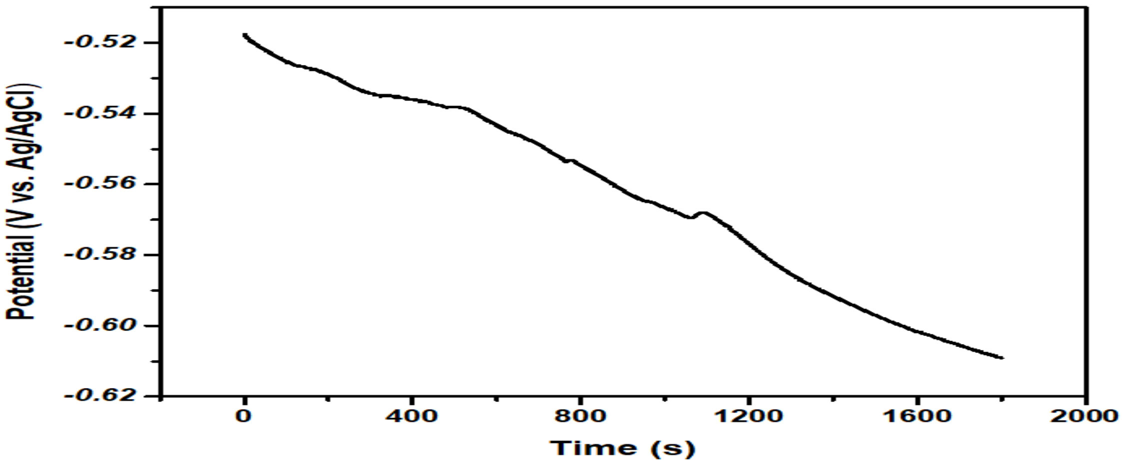

Figure 3: Open Circuit Potential (OCP) Plot of Metal in the Solution Extract

Figure 3 shows the open circuit potential (OCP) for the metal specimen in the extract solution at 100 ppm. The figure shows that the average OCP tended towards more negativity implying that the metal was corroding in the extract solution medium with the trend achieving stability after about 1900 s. Figure 3 equally presents the potentiodynamic polarization (PDP) curve for the metal specimen in the extract solution medium. This plot also called Tafel curve for the metal specimen immersed in the extract medium illustrates the cathodic and anodic currents generated in the course of the electrochemical corrosion reaction which is ordinarily a redox reaction that occurred when the metal was immersed in the extract medium. Essentially, the left and right hand sides of the curve represents the anodic and cathodic activities of the metal specimen in the extract medium. The movement of the curve to higher corrosion current density is an evidence of the metal specimen corrosion in the extract medium.

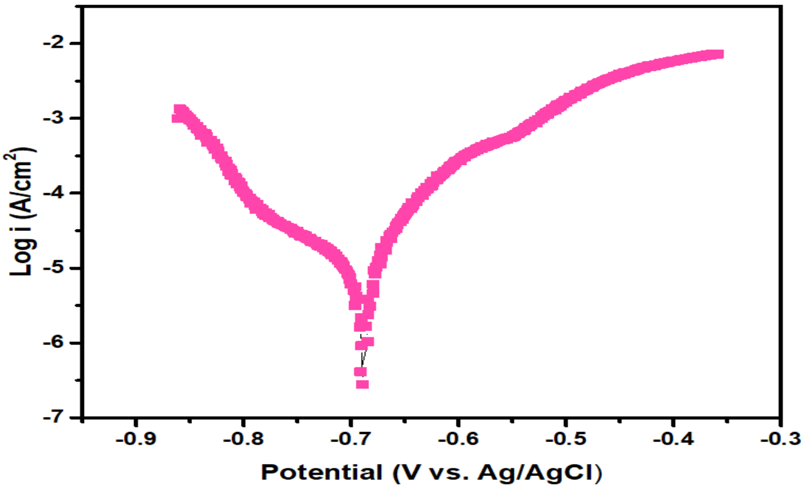

Figure 4: Potentiodynamic Polarization (PDP) Plot of Metal in the Solution Extract.

Figure 4 and 5 shows the Nyquist plot for electrochemical impedance (EIS) data for the metal specimen in the extract medium at the investigated condition. EIS measurement was conducted to examine the corrosion behaviour of the metal specimen in the extract medium. Figure 4 equally shows that the metal specimens corrodes in the extract medium and also reveals the corrosion behaviour of the metal specimen. The semicircle is the capacitive loop showing evidence of corrosion product accumulation at the metal/extract medium interface with the curve exhibiting irregular suppressed semicircle form due to frequency dispersion and is therefore an indication of the non-ideal nature of the metal specimen surface reflecting non-homogeneity and surface roughness.

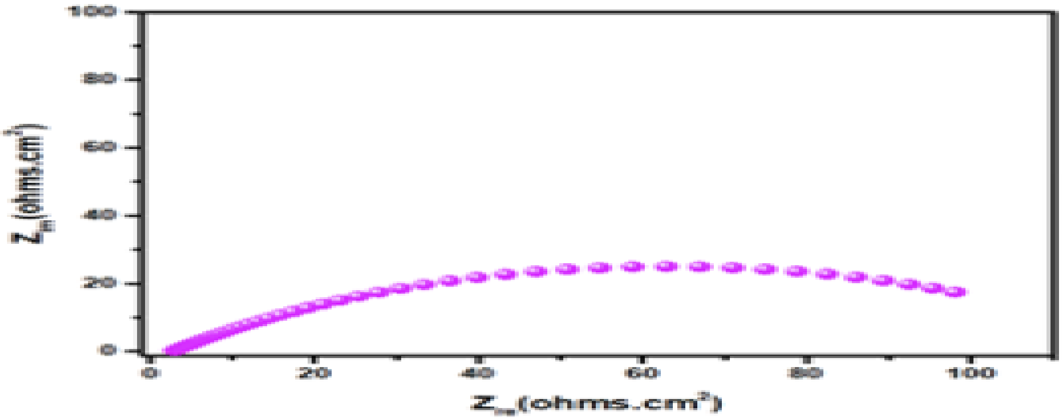

Figure 5: Electrochemical Impedance Spectroscopy (EIS) Plot of Metal in Solution Extract

3.4 FTIR Analysis

Fourier Transform Infrared (FTIR) spectroscopy was employed to investigate the type of bonding interactions involved in the adsorption of organic inhibitors onto the metal surface. The analysis was carried out using a PerkinElmer Spectrum 2 FTIR Spectrometer, with spectra recorded over the wavelength range of 4000 to 450 cm^{-1}. To verify the adsorption of the extract components onto the corroding metal surface, FTIR was first used to identify the functional groups present in the pure extract. Subsequently, the analysis was repeated on the film formed on the metal surface after exposure to the extract at the specified concentration. As illustrated in Figure 6, several peaks observed in the extract's spectrum also appeared in the spectrum of the film adsorbed on the metal, confirming that constituents of the extract had adhered to the metal surface. Additionally, the FTIR results provided insight into the chemical composition and molecular structure of the extract based on the position of absorption bands in the spectra. These bands corresponded to specific functional groups, helping to identify and analyze the chemical compounds present in the extract solution. Fourier transform infrared (FTIR) spectroscopy analysis was used to determine the kind of bonding for organic inhibitors adsorbed on the metal specimen surface. FTIR analysis was performed using FTIR spectra in the 4000 – 450/cm wavelength range using a PerkinElmer Spectrum 2 FTIR Spectrometer.

In order to confirm the adsorption of the extract constituent on the corroding metal specimen surface, FTIR analysis was carried out to first identify the functional group present in the extract and then on the adsorbed film obtained from the surface of the metal at the investigated concentration. The FTIR spectra showed clearly that some of the peaks detected in the spectra of the extract as shown in Figure 4.6 were also present in the spectra of the adsorbed film formed on the surface of the corroding metal specimen, thus confirming the adsorption of the extract constituent on the metal specimen surface. The FTIR analysis further provided information about the extract composition and structure as identified by the peak position in the spectra of Figure 4.6. Absorption bands associated with functional groups could be identified, aiding in the detection and analysis of chemical compounds present in the extract solution.

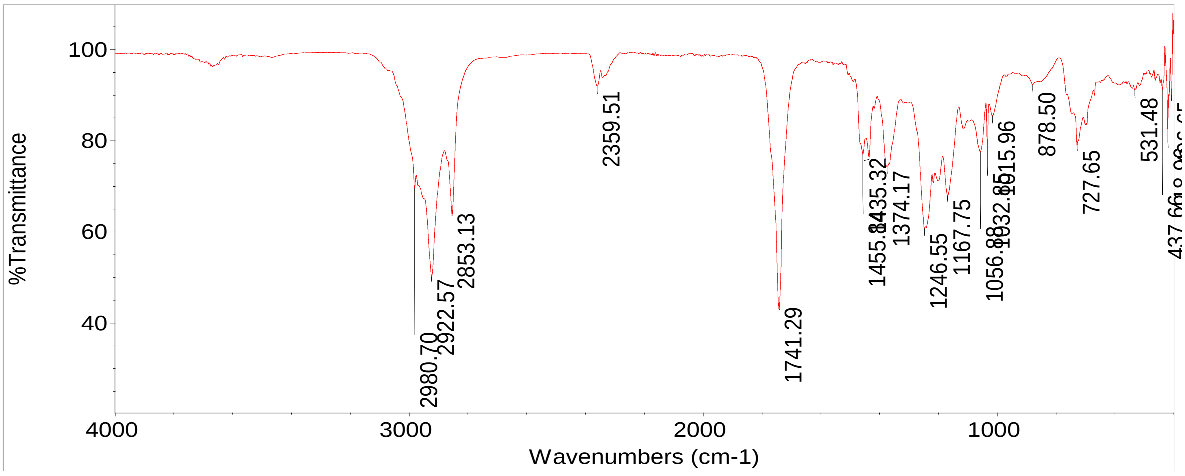

Figure 3.6: FTIR Spectra of Solution Extract of Metal Coupon.

3.5 Surface Investigation

Scanning Electron Microscopy (SEM) is commonly used to analyze the microstructural and morphological changes on metal surfaces resulting from corrosion. In this study, the mild steel specimen was immersed in 0.5 M HCl containing 100 ppm of the extract for one hour. After the exposure period, the specimen was examined using a JEOL JSM-IT100 SEM to assess surface morphology, microstructural changes, and the effectiveness of the extract in forming a protective film. As shown in Figure 7, the SEM image reveals the surface characteristics of the metal after treatment with the extract solution. The micrograph displays a rough and uneven surface with signs of localized corrosion, such as pits and corrosion products, indicating significant surface deterioration caused by the acidic environment. Despite this, the image also shows the presence of a protective layer formed on the metal surface, suggesting that the extract provided a degree of surface coverage. This film appears to act as a barrier, limiting the penetration of corrosive ions and thus helping to mitigate the overall corrosion rate.

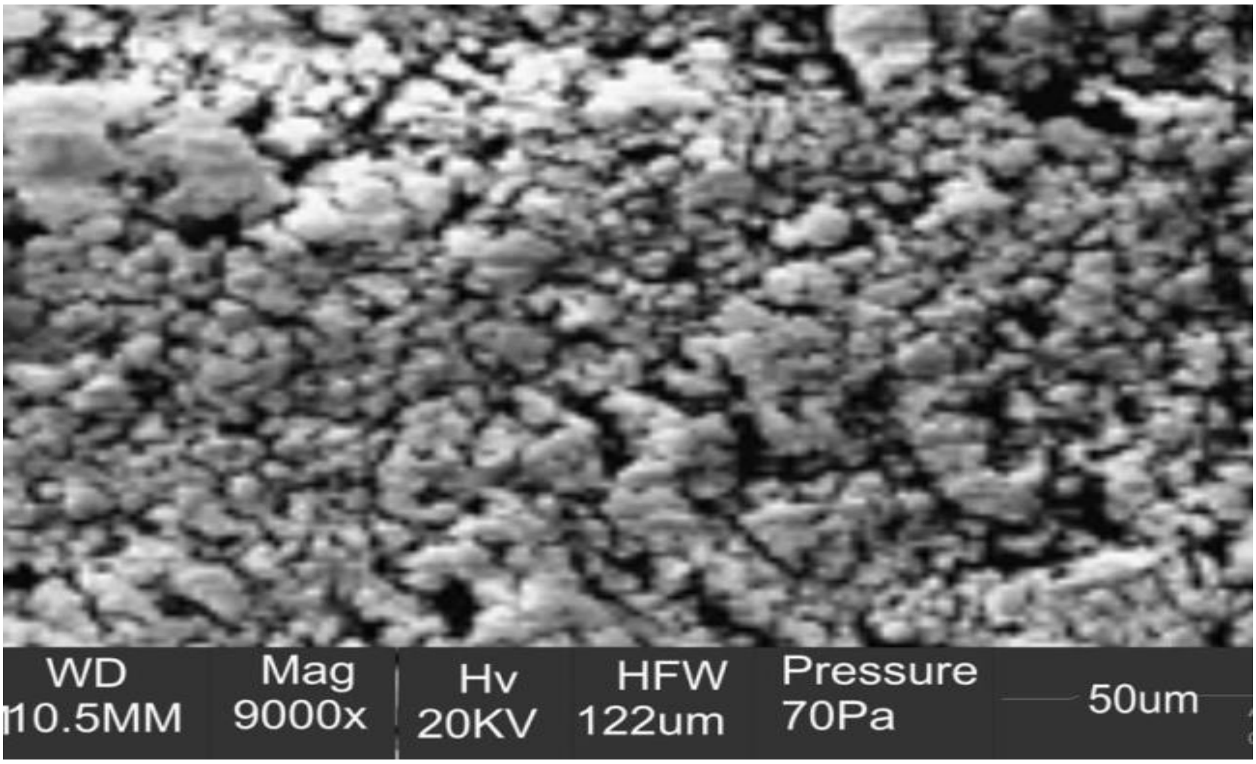

Figure 3.6: SEM Microstructure of Exposed Metal Coupon Surface

CONCLUSION

This study confirmed that carbonized orange peel extract serves as an effective and eco-friendly corrosion inhibitor for mild steel in 0.5 M HCl solution. Both spectroscopic and electrochemical evaluations indicated that the inhibition process is largely physical in nature (physisorption), involving hydrogen bonding, π-electron interactions, and dipole forces between the extract's functional groups and the steel surface. UV–Vis spectroscopy showed no new absorption peaks during metal immersion, suggesting the inhibitor molecules remained chemically unchanged, while FTIR analysis confirmed the presence of key functional groups (–OH, C=O, and C–O–C) on the metal surface after adsorption. Electrochemical techniques, including OCP, potentiodynamic polarization (PDP), and electrochemical impedance spectroscopy (EIS), consistently demonstrated lower corrosion current densities and higher charge transfer resistance in the presence of the extract. These results indicate that the extract effectively slows down both anodic and cathodic corrosion reactions. Supporting this, SEM images revealed the formation of a protective surface film that partially covered the steel, acting as a barrier against aggressive chloride ions. In summary, the combination of analytical and surface characterization methods confirmed that the carbonized orange peel extract forms a stable, organic-rich layer on mild steel, significantly lowering corrosion rates compared to an uninhibited system. Its high inhibition efficiency, low cost, biodegradability, and environmental friendliness make it a promising alternative to conventional synthetic inhibitors, especially in acidic environments like cleaning and pickling processes. These results also support ongoing research on plant-based inhibitors and emphasize the potential of converting agricultural waste into valuable materials for industrial corrosion protection.

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