

Effect of *Citrus aurantifolia* Seed Extract on the Mechanical Properties of Mild Steel in 0.1M Hydrochloric Acid

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ABSTRACT

Citrus aurantifolia (lime) seed extract was evaluated as corrosion inhibitor for mild steel in 0.1M Hydrochloric acid, using weight loss measurements at room temperature. *Citrus aurantifolia* (lime) seed extract inhibited the corrosion of mild steel significantly with 88.4% efficiency at the highest concentration of 45mls of the inhibitor. Tensile strength, hardness, Surface profiling and micro structural tests were carried out on the as- received and inhibited coupons. The tensile tests revealed an increase in tensile strength, demonstrating that the extract not only protects the steel from corrosion but also improves its ductility and strength. Hardness measurements showed a noticeable increase in surface hardness while the Surface analysis confirmed the formation of a protective film on the mild steel surface, attributed to the adsorption of active compounds from the seed extract. This study offers valuable contributions to the field of corrosion science and materials engineering.

Keywords:

Citrus aurantifolia,
Hydrochloric acid,
Mild steel,
Tensile strength.

INTRODUCTION

The need to develop corrosion inhibitors that are environmentally friendly and with specific actions on substrate has led to a wide-range study on natural inhibitors (Avwiri and Igbo, 2003). Metals represent some of the most widely employed materials in both ornamental and structural applications due to their strength, malleability, and aesthetic appeal. Among these, mild steel stands out as a preeminent choice because of its cost-effectiveness, ease of fabrication, and favourable mechanical properties. It is particularly prominent in the petroleum industry, where it is routinely used in the construction of pipelines and related infrastructure. However, the aggressive and often acidic nature of the fluids transported through these pipelines presents a significant challenge, as it leads to accelerated corrosion and material degradation over time (Eziaku, 2017). Despite its advantageous properties—such as high tensile strength, ductility, and versatility—mild steel remains highly susceptible to corrosion when exposed to harsh chemical environments, especially those involving acids.

This vulnerability limits its long-term performance and can result in costly maintenance, repair, or replacement in industrial systems. As a result, corrosion mitigation remains a critical area of research and engineering focus. Ongoing efforts are directed toward improving the

corrosion resistance and overall durability of mild steel under diverse operational conditions through protective coatings, alloying, and the use of corrosion inhibitors (Angst, 2019). These strategies aim to extend the service life of mild steel components while maintaining safety and reducing environmental and economic impacts associated with material failure. Given the huge demand for environmentally acceptable corrosion inhibitors, plant-based inhibitors have drawn a lot of interest. Derived from many sections of plants, these inhibitors are to offer a non-toxic, environmentally friendly replacement for traditional synthetic inhibitors. Several plant extracts have been investigated in many research to help lower metal corrosion (Avwiri and Osarolube, 2010).

Mild steel is extensively utilized in numerous industrial sectors due to its desirable mechanical characteristics, and affordability, for manufacturing. In spite of these identified advantages, it is highly vulnerable to corrosion, especially when exposed to acidic environments like hydrochloric acid (HCl). This corrosion can seriously weaken the structural integrity of mild steel, leading to major financial losses and safety risks in industries that employ processes such as acid cleaning, pickling, and descaling. In order to address this issue, synthetic chemical-based corrosion inhibitors have traditionally been used. However, concerns related to their

environmental impact, toxicity, and disposal have driven the search for more sustainable and environmentally friendly alternatives. In this regard, natural plant extracts have gained attention as effective eco-friendly corrosion inhibitors, owing to their biodegradability, low environmental risk, and wide availability. In this context, plant-based extracts have gained significant attention as green alternatives for corrosion inhibition, Ajani *et al* (2014). These natural substances are not only biodegradable and non-toxic, but they are also readily available, making them a promising solution for environmentally conscious industries. Among the various plant species investigated for their corrosion-inhibiting properties, *Citrus aurantifolia*—commonly known as lime—has emerged as a potential contender (Asipita, *et al* 2014). The plant is rich in organic compounds, including alkaloids, flavonoids, and essential oils, which have demonstrated the ability to reduce the corrosive effects of acidic environments on metal surfaces. The paper of Chuka *et al* (2014) investigated the effect of corrosion on the mild steel in five different environments reported that the rate of corrosion of mild steel in the various media decreases in the following manner; 0.1M hydrochloric acid, underground(soil), salt water, fresh water, and the atmosphere.

While much of the research on *Citrus aurantifolia* has focused on the peel, leaves, and fruit, there has been limited investigation into the efficacy of the seeds of the plant as a corrosion inhibitor. The seeds of *Citrus aurantifolia* are known to contain a unique array of bioactive compounds, yet their specific role in corrosion inhibition remains largely unexplored. This study aims to fill that gap by assessing the abilities of *Citrus aurantifolia* seed extract as a corrosion inhibitor for mild steel, particularly when exposed to 0.1M hydrochloric acid.

The research will focus on evaluating the mechanical properties of mild steel—such as tensile strength, hardness, and ductility after exposure to the extract. These parameters will help determine the effectiveness of the seed extract in preserving the structural integrity of mild steel in an acidic environment. The results of this study could contribute significantly to the development of green corrosion inhibitors that are not only environmentally friendly but also cost-effective for industrial use. Furthermore, it could open the door to new applications for *Citrus aurantifolia* seed extract in a wide range of industries, from construction to manufacturing, where metal corrosion is a persistent problem.

MATERIALS AND METHODS

Citrus aurantifolia Seed Extraction Process

The *citrus aurantifolia* fruits were bought from the oil mill market of Port Harcourt in Rivers state. The fruits were cut to remove the seeds from it, the seeds were

washed in clean water and rinsed in air distilled water to eliminate any pulp, sand, and pollutants. The seeds were then dried in a clean, dust-free room which has its temperature to be 25-35 degrees Celsius, till the absence of moisture content was felt. The seeds were grounded into a fine powder. 130 grams of the seed powder was poured directly into a large beaker and then mixed together with N-hexane. The mixture was kept in a controlled environment at room temperature for 48 hours to allow evaporation to take place, it was stirred at 12-hours intervals. The extract was next run through filter paper to eliminate any solid left overs. To increase surface area for evaporation, the filtered extract was laid in a shallow evaporation dish. It took 32 hours for the evaporation process to reach total evaporation devoid of any N-hexane residue.

Phytochemical Test for *Citrus Aurantifolia* Seed Extract

Phytochemical test was carried out on the dried grounded citrus aurantifolia seeds at the Phytotherapy Laboratory of the Department of Pharmacognosy, University of Port Harcourt. The results of the test showed the presence of flavonoids, alkaloids, terpenoids, tannins and glycosides. This shows that citrus *aurantifolia* seeds can serve as a good corrosion inhibitor.

Mild Steel Preparation

The mild steel sheet used in this study was obtained from the mechanical workshop, Choba campus, University of Port Harcourt, Rivers State. It was cut into coupons with dimensions 40 mm by 40 mm with a thickness of 1.5 mm. A 2 mm hole was drilled on the mild steel coupon, this hole was created to enable threading and labelling, the threading aided with insertion and removal of the mild steel coupons from the corrosive medium. The mild steel was properly cleaned in ethanol and distilled water and wiped with dried lint-free piece of fabric. A spark analysis was carried out to ascertain the standard and component of the mild steel, and the analysis grades was C-4120(1.29), C-4140(1.53) and with elemental composition of Fe (90.50%), Pb (1.52%), Mn (2.25%), Co (4.50%), Cr (0.63%), and Ag (0.61%) respectively. Also, the surface characterization was carried out at Turret Engineering Services, Port Harcourt, with the use of Inverted metallurgical microscope.

Gravimetric Analysis with *Citrus Aurantifolia* Seed Extract

The cleaned mild steel coupons were weighed using analytical balance and weights were recorded. Beakers filled with the corrosive media of 0.1 M of HCL contained each labelled and threaded coupons immersed in each beaker and the already extracted citrus aurantifolia seed extract was added as the inhibitor at different concentrations of 5, 10, 15, 20, 25, 30, 35, 40,

45 millilitres and were taken out at the intervals of 24, 48, 72, 96, 120, 144, 168, 336, 504, 672 hours respectively. With the help of the threads attached to each coupon, the coupons were taken off the solution following the exposure time and were quickly cleaned with distilled water, ethanol and then air dried. In another separate beaker, a coupon was immersed in a solution of HCL without an inhibitor, the readings of this particular coupon were taken before and during every withdrawal of other coupons and were also recorded.

Mechanical Properties Testing

The mechanical testing of the mild steel coupon was carried out at Turret Engineering, along East West Road, Rumuosi, Port Harcourt. The coupons were arranged separately on the testing platform under clamping machine secured to prevent moving during testing. The indenter was aligned with the already-prepared mild steel samples; load was then applied using the Brinell hardness testing equipment; this load was especially applied for 10 to 15 seconds.

The Brinell Hardness formula is given by;

According to Bhaduri and Bhaduri (2018), $BHN = \frac{P}{\pi D (D^2 - D^2 - d^2)}$

Where P is the applied load.

D is the diameter of the indenter.

And d is the diameter of the indentation.

Surface profiling technique fit for mild steel was employed. In this instance, contact profiling was applied. The surface profiling test was conducted using an SRT5000 profilometer, whereby the stylus was moved over the surface and data points were gathered to produce the surface profile.

Microstructure and Surface Characterization Testing

The C-4120 mild steel coupon samples were cleaned using 80, 180, 320 and 600 grits emery papers, then polished with 1200 grit and diamond paste. The mild steel coupon samples were then etched with 5% Nital etchant. The samples were examined using an inverted metallurgical microscope before and after the immersion into the solution containing hydrochloric acid only and hydrochloric acid with citrus aurantifolia seed extract.

Tensile Strength Testing

The mild steel was positioned on the Universal Testing Machine (UTM) in a suitable manner. To guarantee a correct alignment and prevent any bending or off-axis loading, the machine was calibrated to tightly match the UTM. Measurement of the initial dimensions of the steel, including the cross-sectional area and gauge length, were recorded at the same dimensions of the specimen. Though the strain rate ranged from 0.2 to 2.0 mm/min, the UTM was programmed to apply a consistent rate of

strain—crosshead speed. The test began when the UTM progressively tensely forced the specimen. The data on the applied force were recorded constantly till the steel fractured.

RESULTS AND DISCUSSION

Analysis on the Effects of Inhibitor on Weight Loss, Inhibitor Efficiency and Corrosion Rate

Weight Loss Analysis

Figure 1 shows the lowest and highest weight loss noted correspondingly for the 24 hours corrosion time with 5ml inhibitor concentration and the 672 hours corrosion time with 45ml inhibitor concentration, the weight loss ranged from 0.021g to 0.1671g. Weight loss decreased with increase in concentration of *Citrus aurantifolia* seed extract. Accordingly, the corrosion rate shows a notable decrease as the inhibitor concentration rises, therefore suggesting that the citrus aurantifolia has a good corrosion inhibition.

Inhibitor Efficiency and Corrosion Rate

Figure 2 shows the corrosion rate ranging from 1.84×10^{-5} gcm²hr⁻¹ to 1.2×10^{-4} gcm²hr⁻¹. Figure 3 indicate the inhibitor efficiency. With the lowest and highest inhibitor efficiency recorded for the 168 hours corrosion time with 15ml inhibitor concentration and 24 hours corrosion time with 45ml respectively, the efficiency which was calculated through weight loss measurement process for different inhibitor concentration and corrosion time ranged from 42.59% to 88.4%. Effective corrosion inhibition is shown by a notable drop in the corrosion rate as the inhibitor concentration rises. The findings are in agreement with research of Oguzie (2008) and Verma et al., (2025) showing corrosion-inhibiting effects of plant extracts including flavonoids, alkaloids, tannins, and other phytochemicals. This implies that the active chemicals in the seed extract efficiently adsorb onto the steel surface to provide a protective barrier lowering metal dissolution.

Mechanical Properties

Figures 4, 5 and 6, display the Brinell Hardness, tensile Strength, and Surface Profile at different times respectively. The Brinell Hardness, Strength, Surface Profile values recorded at all times of measurement fell below their respective control levels. Figure 3.4 shows increase in hardness values with the addition of the seed extract. This indicates a harder and more resilient surface, which is advantageous for applications requiring wear resistance. Figure 3.5 shows that adding *Citrus aurantifolia* seed extract gradually increases tensile strength, suggesting that the inhibitor not only guards against corrosion but also maintained the mechanical strength of the mild steel.

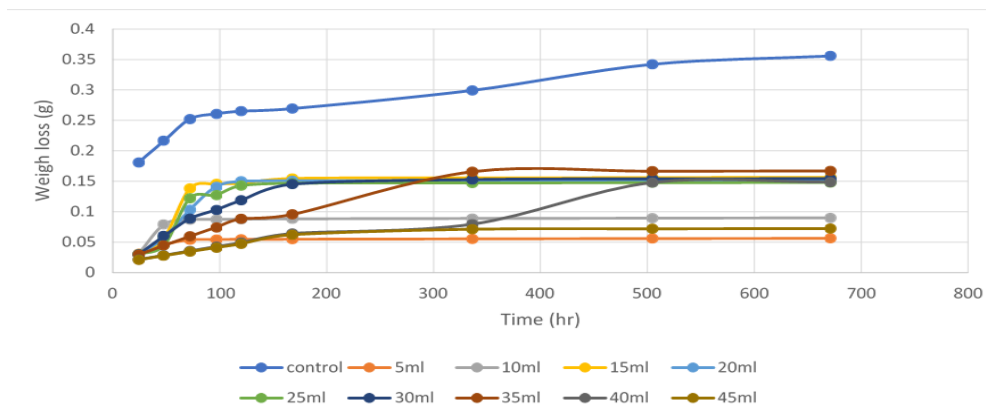


Figure 1: Variation of Weight loss (g) with Time (hr)

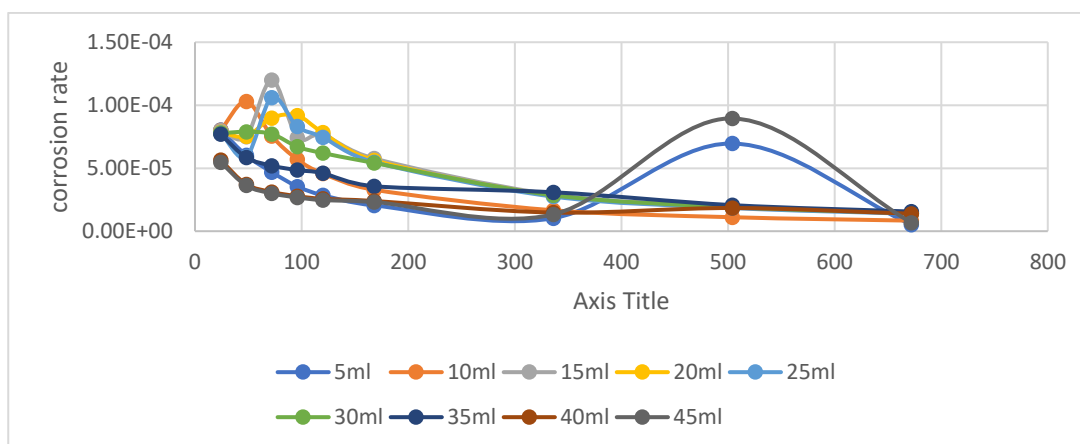


Figure 2: Corrosion rate at different concentration and time of the mild steel coupon

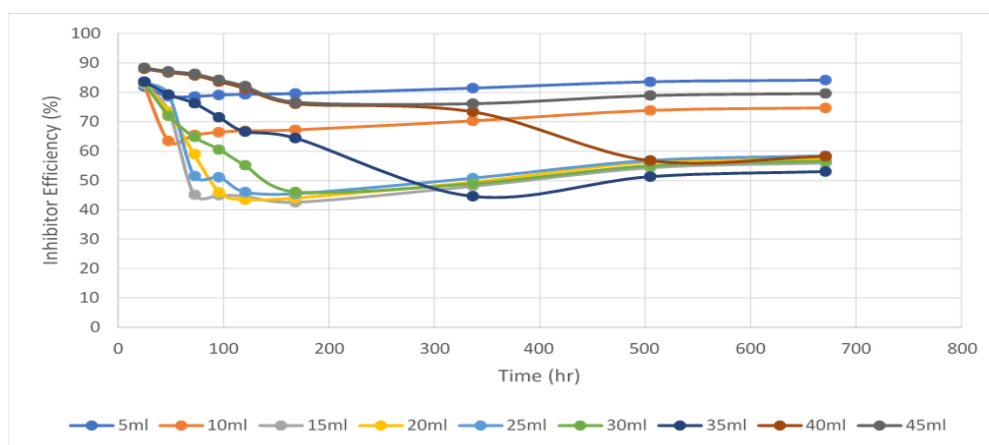


Figure 3: Variation of Inhibitor efficiency with concentration and time of mild steel after corrosion

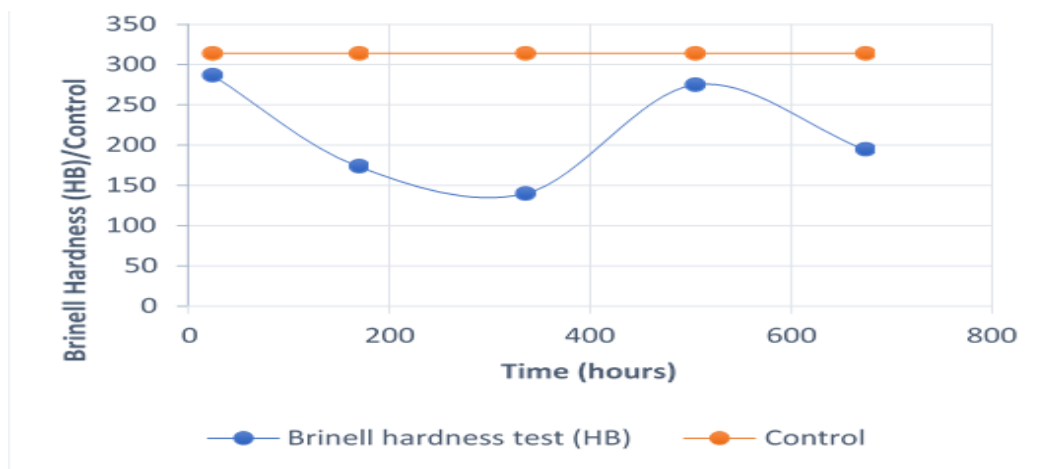


Figure 4: Variation of Brinell Hardness with time for the control and inhibited coupon

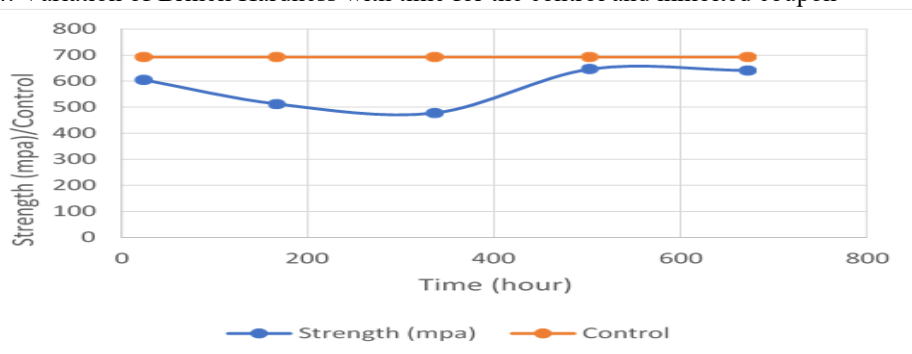


Figure 5: Variation of Tensile Strength with Time for control and inhibited coupon

Surface Profiling Analysis

The Scanning electron microscopy (SEM) reveals the surface morphology of mild steel coupons before and after immersion in 0.1 M. of HCl, with and without *Citrus aurantifolia* seed extract. Reflecting the pre-corrosion state, SEM pictures of the unexposed mild steel surface exhibit a smooth, polished appearance with few surface flaws. Plates 2, 3, 4, 5 and 6 demonstrate mild steel's surface morphology following acid immersion. Surface damage in SEM images of the inhibited samples is rather decreased. A reasonably smooth surface with less pits and cracks indicates that the extract retards corrosion successfully. A protective coating developed on the blocked surfaces also helps to explain the low corrosion rate. The microstructure reveals mostly Pearlite, the darker tones in the picture, and also Ferrite, the lighter variants of the structure.

The Day 1 and Day 7 samples correspondingly show more clearly the ferrite. This implies that the microstructure test shows a mostly ferrite structure which can be ascribed to numerous elements, including low carbon content of the steel. The Days 14, 21, and 28 accordingly have more of the pearlite form. There are

Micrographs of Mild Steel Coupons before and after Corrosion

various reasons why a microstructure test shows a predominance of pearlite structure. Typically, between 0.8% and 0.02%, pearlite develops in steels with intermediate carbon content. The formation of pearlite is highly sensitive to how quickly the material cools. When cooling is slow or the material is held at temperatures within the “pearlite nose” of the Time-Temperature-Transformation (TTT) diagram, pearlite forms. Over longer periods—such as at Days 14, 21, and 28—the steel, maintained or slowly cooled at specific temperatures, allows more austenite to convert into pearlite. The transformation becomes more complete with time. Steels with higher carbon content tend to favour the formation of pearlite. If the alloy composition supports pearlite, extended time periods enable carbon atoms to diffuse and segregate into cementite layers.

Pearlite is a microstructure consisting of alternating layers of ferrite (a soft, ductile phase) and cementite (a hard, brittle phase). It develops when austenite, which exists at high temperatures, cools at a moderate pace, allowing carbon atoms to diffuse and form these characteristic layered structures.

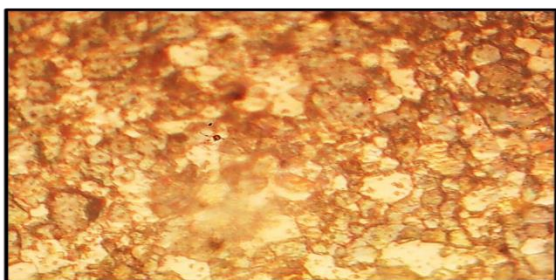


Plate 1: Micrograph before corrosion exposure x400

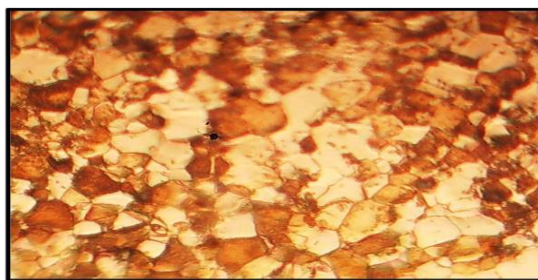


Plate 2: Micrograph after day 1

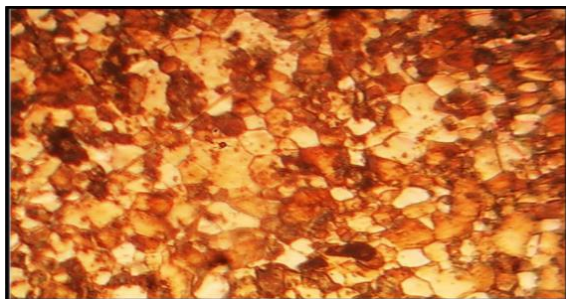


Plate 3: Micrograph after 7 days

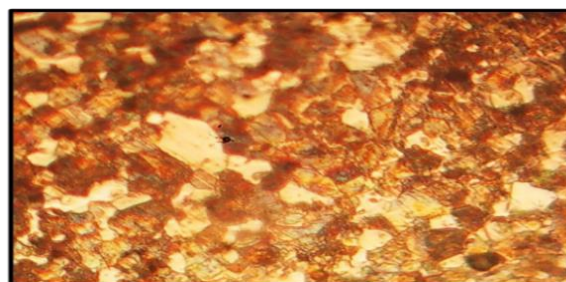


Plate 4: Micrograph after 14 days

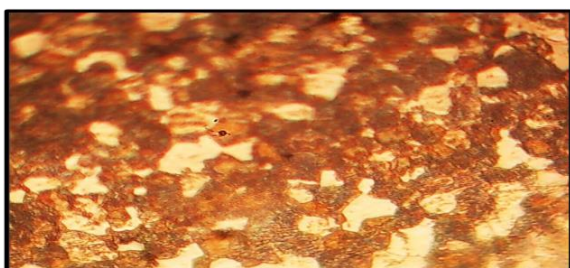


Plate 5: Micrograph after 21 days

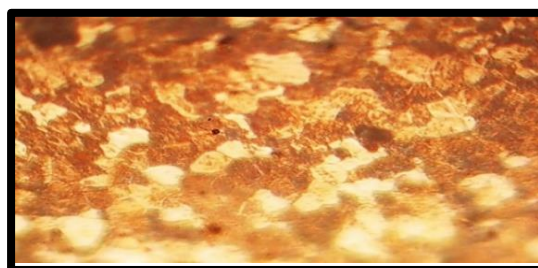


Plate 6: Micrograph after 28 days

All Magnifications of Samples after Corossion are X200

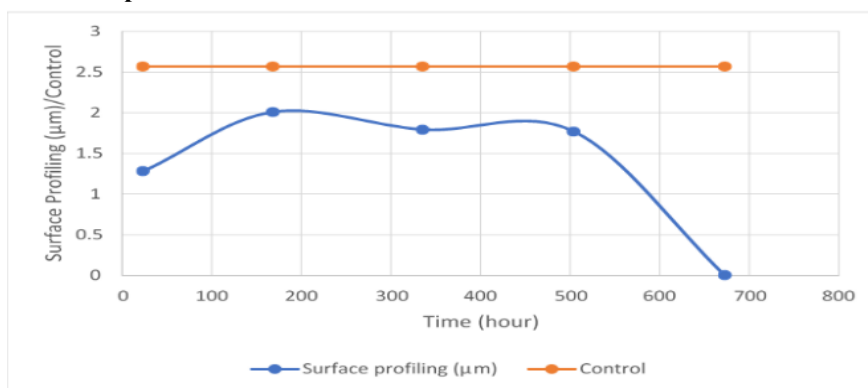


Figure 6: Surface Profiling of mild steel before and after corrosion

The clearer presence of ferrite observed in the samples for the Day 1 and Day 7 suggests that the microstructure is primarily composed of ferritic phases. Ferrite, which is a relatively soft and ductile form of steel, has a body-centered cubic (BCC) crystal structure and is typically associated with steels that have low carbon content.

However, several factors contribute to the predominance of ferrite in the microstructure, these include; Low Carbon Content: Ferrite remains stable at low carbon levels, as it can only dissolve a minimal amount of carbon (approximately 0.02% at room temperature). The dominance of ferrite implies that the steel likely has a low

carbon composition, which restricts the formation of other microstructures such as pearlite or martensite. Cooling Rate and Heat Treatment, indicating the thermal history of the samples also plays a significant role. Slow cooling or particular heat treatment methods encourage the formation of ferrite by allowing carbon atoms to diffuse out of austenite and precipitate as cementite, leaving ferrite behind. The alloying elements, like manganese, silicon, and chromium in addition to carbon, can help stabilize the ferrite phase or affect the kinetics of phase transformations, thereby promoting a higher ferrite content, and this in turn leads to Time-Dependent Changes, the difference in ferrite visibility between the Day 1 and Day 7 samples may reflect microstructural changes over time, such as tempering effects. Nevertheless, ferrite remains the dominant phase in both, indicating the fundamental ferritic nature of the steel is maintained

CONCLUSION

The effectiveness of *Citrus aurantifolia* seed extract as a corrosion inhibitor for mild steel is comprehensively assessed through a combination of analytical techniques. Weight loss analysis is employed to determine the rate of corrosion over time, providing quantitative data on the extract's ability to reduce metal degradation. In addition, surface profiling techniques, including microscopic examination of the mild steel's microstructure, are used to visually evaluate surface damage and morphological changes induced by acid exposure. Furthermore, mechanical property tests—such as tensile strength, hardness, and ductility measurements—are conducted to examine whether the extract has any effect on the structural integrity of the steel. Together, these methods offer a thorough evaluation of the extract's protective efficiency and its potential as a sustainable alternative to conventional corrosion inhibitors. The inhibition efficiency of the mild steel coupon had its lowest efficiency to be 42.59% at 168 hours period with the concentration of 15 ml of inhibitor, and the highest efficiency was 88.49% which was recorded at 24 hours period with the concentration of 45 ml of inhibitor. The protective layer formed by the binding of extract molecules to the steel surface acts as a barrier, significantly slowing down the corrosive action of the acid on the underlying metal. This layer hinders the direct interaction between the acid and the steel, thereby reducing the rate of corrosion and extending the material's lifespan. Notably, this inhibitor, derived from natural sources, is environmentally benign and biodegradable, making it a sustainable and ecologically responsible alternative to conventional synthetic inhibitors. Its effectiveness in mitigating corrosion, coupled with its low toxicity and renewable origin, positions it as a promising candidate for widespread

industrial applications where both performance and environmental impact are critical considerations.

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