
FEASIBILITY OF MANGIFERA INDICA (MANGO) LEAVES EXTRACT AS A CORROSION PROTECTION ON 6061 ALUMINUM ALLOY IN HYDROCHLORIC ATTACK

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Abstract

The mango fruit (*Mangifera indica*) is one of the most abundant fruits in our country. Because of the growing demand for green chemistry in science and industry, the development of green corrosion inhibitors and green inhibition techniques has recently become extremely important. The use of plant extracts as metallic corrosion inhibitors has received a lot of interest in the last several decades. The research should also learn about corrosion control compounds and components, anti-corrosive performance, and the benefits and drawbacks of MIL extract. The *Mangifera Indica* Leaves extract includes functional groups that can neutralize oxygen during its interaction with other molecules, according to FTIR testing (Fourier-Transform Infrared Spectroscopy). The salt spray testing result is "No observable defects," indicating that the coating effectively protected the aluminum alloy against HCL damage during immersion. The Weight-Loss Method was used on the non-coated and coated samples with varying concentrations, and the results demonstrate that the sample with no coating lost a lot of weight owing to the HCL attack. The noticeable difference is that the sample with 10% concentration, Sample B, outperformed the others. One of the most important findings from the tests is that the sample with no coating deteriorates about three times quicker than the sample with coating. This study suggests using a better binder solution, experimenting with different quantities of mango leaves extract and binder solution combinations, and conducting more testing methods for more detailed results.

Keywords: *Mangifera indica*; MIL extract; Corrosion Inhibitor

Introduction

Us, being the most intelligent species that have existed and evolved in the history of mankind, learned how to utilize the resources around us. We rely on necessities to survive like food, water, and shelter. The need for food is vital for survival. But the availability of food also varies depending on the geographic placement. As humans, we both consume meat, and vegetables (plants) as a primary source of energy.

The population of a certain species might be abundant in some places. This is because there are certain factors that make them capable of living in a specific environment. Every species has its own unique characteristic. They have developed to grow in places in tropical regions. The mango is now cultivated in most frost-free tropical and warmer subtropical climates. It is cultivated extensively in South Asia, Southeast Asia, East and West Africa, the tropical and subtropical Americas, and the Caribbean (Altendorf, 2019).

One of the most abundant fruits here in our country is the Mango fruit (*Mangifera indica*). They also grow better on well-drained, and slightly higher acidity-content of the soil. These weather conditions are really suitable for our country to grow mango trees, especially in regions where there is not much rainfall or not always affected by typhoons. Mango trees do also live long. Mango trees planted from seeds will take roughly 8 years to produce fruit, while mango trees planted from saplings will take up to 5 years to produce mangoes (DeAngelis, 2022).

This just shows how rich mango trees are in terms of being a source of food or another byproduct. Highlighting its abundance, there is an estimated total equivalent area of 188, 600 hectares that has mango trees just in our country (Total land area used for mango cultivation in the Philippines from 2016 to 2020,2022). Aside from being used as a source of food. Recent studies also show that mango leaf — based products were found to be efficient in inhibiting corrosion. All plants are rich in phytochemicals which act as their defense mechanism to ward off external destructive

agents such as pathogens and predators (Thiruvananthapuram et al., 2019). Bioactive elements form an iron-polyphenol-insoluble-organometallic compound, which is responsible for the anti-corrosive property (Gopalan, 2019).

Mango is the third most important fruit crop in the Philippines. Phytochemistry studies have demonstrated that phenolic compounds are one of the most important biologically active components of *M. indica* extracts. Ultrasound- and microwave-assisted extractions and supercritical fluids have been employed to obtain bioactive molecules, such as phenolic acids, terpenoids, carotenoids, and fatty acids (Quintana et al., 2021). Additionally, there are numerous types of research that examined corrosion inhibition in solutions containing *Mangifera indica* leaves extract. Particularly, according to Ramezanzadeh et al. (2018), the mango extract decreases the iron surface damage and the inhibitor molecules' adsorption provide a hydrophobic surface.

The adaptability of aluminum goes beyond its strength and lightness. It can be worked into any shape with ease and has an astonishing range of surface finishes. Aluminum exhibits good corrosion resistance under the majority of service situations. One of the main elements affecting the low cost of finished aluminum parts is its excellent machinability. Aluminum can be joined using almost any technique, including riveting, welding, and brazing. Drawn, spun, and roll-formed aluminum sheets are all possible. Many scholars have concentrated their efforts on a studying the corrosion of aluminum in various aqueous solutions as well as study into their electrical properties and corrosion inhibition in a wide range of conducting solutions (Montijo S., 2021; Beda et al., 2017).

Corrosion is when a refined metal is naturally converted to a more stable form such as its oxide, hydroxide or sulphide state this leads to the deterioration of the material. Metal corrodes when it reacts with another substance such as oxygen, hydrogen, an electrical current, or even dirt and bacteria. Corrosion can also happen when metals like steel are placed under too much stress causing the material to crack. Several methods of corrosion prevention have been developed among which the use of synthetic corrosion inhibitors is one of the most popular and economic methods due to their ease of synthesis and application and high effectiveness at relatively low concentrations (Verma et al., 2018).

Recently, the development of green corrosion inhibitors and green inhibition strategies are highly demanded because of the increasing demand for green chemistry in the area of science and technology. In the last few decades, use of plant extracts as metallic corrosion inhibitors has attracted significant attention. Literature reports suggest that plant extracts have excellent corrosion inhibition efficiency, especially leaf extract, because of the presence of an abundant source of phytochemicals compared to other parts (Verma et al., 2018).

In this study, researchers will be examining the feasibility of *Mangifera indica* (mango) leaves extract as a natural alternative to prevent corrosion from forming on the 6061 Aluminum Alloy surface of the aircraft against HCL attack.

Materials and Methods Method of Research

The researchers employed the use of an experimental approach to be able to deal with the various procedures of the study. According to Babbie (2021), An experimental study is one that precisely follows a scientific research method. It consists of a hypothesis, a variable that the researcher may alter, and variables that can be measured, computed, and compared. Most significantly, experimental research is carried out in a controlled setting. The researcher collects data, and the findings either support or refute the hypothesis.

Research Design

This experimental research gives the researcher an idea of how feasible the mango leaves extract will perform on being a corrosion protection agent in different concentrations through a hydrochloric attack on the 6061-aluminum alloy base. To attain the necessary data needed, mango leaves will be gathered and various tests will be conducted to be able to analyze and determine the mango leaves extracts' performance.

The extract will be first studied and sent to various testing divisions to be tested for a phytochemical analysis to examine the major organic constituent. The *Mangifera Indica* (Mango) leaves extract will be characterized through

Fourier Transform Infrared (FTIR). While corrosion inhibition will be examined through Salt Spray, and Weight Loss Methods.

Materials and Equipment

The researchers utilized various consumable and non-consumable materials and equipment in order to perform this study.



Figure 2. 1kg Mango Leaves

The 1kg Mango leaves will be the key component in this research. It will be used and will undergo various procedures in order to produce the extract.



Figure 3. Beaker (250ml)

The 250ml beaker was used in soaking the samples in Hydrochloric acid.



Figure 4. 6061 Aluminum Alloy Sheet Metal (20x5x.02cm)

Before the experiment, the 6061 aluminum alloy specimens were degreased with acetone. The 6061 aluminum alloy sheet metal was sourced from an online store. The sheet metal was divided into equal parts, with a dimension of 3x5cm.



Figure 5. Hydrochloric Acid (30% concentration)

Hydrochloric acid was used to mimic the corrosion attack on the 6061-aluminum alloy.



Figure 6. Acetone

Acetone was used in our research for cleaning the 6061 aluminum alloy plates before the application of the mango leaf and epoxy primer mixture.



Figure 7. Syringe (5ml)

The syringe was used in adding the mango leaf extract to the epoxy primer.



Figure 8. Weighing Scale

The weighing scale that has 2 decimal places for accurate measurement was used to measure the weight of the metal coupons and the sample mixtures



Figure 9. Epoxy primer and Catalyst

The epoxy primer and catalyst were used as a medium in applying the mango leaf extract to the metal coupons

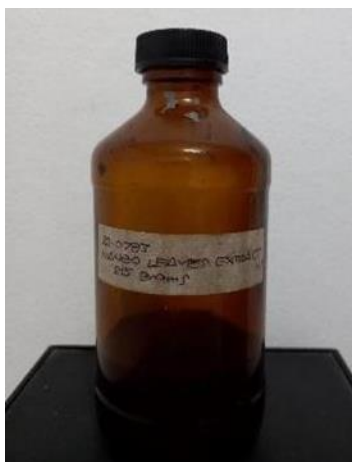


Figure 10. 225g Mangifera Indica (Mango) leaves extract

The Mangifera Indica (Mango) leaves extract is the primary material that was used in this study. The materials used for this extract were gathered by the researchers and sent to ATRDC for extraction.

Table 1. Cost of Materials, Tools, and Equipment

MATERIAL/EQUIPMENT	PRICE	QUANTITY AND MEASUREMENT	AMOUNT
MATERIAL			
6061 Aluminum Alloy	PHP 158.00	20x5x3 cm (5 pcs.)	PHP 790.00
Mango Leaf Extract (Fee for the Extraction Process)	PHP 2,000.00	215 grams	PHP 2,000.00
Primer and Catalyst	PHP 232.00	1 liter	PHP 232.00
Acetone	PHP 40.00	20ml	PHP 40.00
TOOLS AND EQUIPMENT			
Weighing Scale	PHP	1 pc.	PHP 350.00
Beaker	PHP	250ml (5 pcs)	PHP 600.0

Project Development Gathering Process



Figure 11. Mango Tree at Sucat, Muntinlupa City

To start the experiment, the researchers must collect mango leaves. 1 kilogram of mango leaves was collected to ensure that there is a sufficient amount of mango leaves for the extraction process. The mango leaves were collected in Sucat, Muntinlupa City by one of the researchers.

Acquisition of Materials, Apparatus, and Equipment

The materials, apparatus, and equipment needed for this study was acquired from October to November 2022. To start the experiment, the researchers sourced out the materials from physical and online stores. After completing of the materials needed, the researchers immediately began in conducting the experiment and chose to conduct the experiment on a house located at Sucat, Muntinlupa City.



Figure 12. Room used for weighing the materials to achieve accurate measurements.

Extraction of the Mango Leaf

The aim of this study is to determine the effectiveness of *Mangifera Indica* (Mango) leaves extract as a corrosion inhibitor on 6061 aluminum alloy against Hydrochloric attack. The researchers began the experiment by bringing the collected mango leaves to the Adamson University Technology and Research Development (AUTRDC) in order to produce the mango leaf extract.

The AUTRDC began the extraction by boiling the mango leaves in ethanol on a volumetric flask, and in order to remove the excess solvent, they used a condenser to separate the solvent from the mango leaf extract. The total amount of mango leaf extract that was collected is 225 grams.



Figure 13. Extraction Process

Addition of the Mango Leaf Extracts to the Epoxy Primer.

The researchers used epoxy primer as a medium in applying the mango leaf extract to the 6061 aluminum alloy plates. In mixing the epoxy primer and the mango leaf extract, the researchers used a hand drill in order to properly combine the two. The researchers prepared a total of four sample mixture that is composed of different concentrations, and a plain mixture that will be used to compare the effectiveness of each sample.



Figure 14. Mixing the Mango Leaf extract to the Epoxy Primer

Formulation of Samples.

After mixing the epoxy primer and *Mangifera Indica* Leaves extract at different quantities to have comparisons on different concentrations. Then the researchers added the catalyst to the mixing process. The catalyst will serve as the drying agent for the mixture. Each sample has its own labeled container. The first sample (Sample A) contains 13.39 grams of epoxy primer added with 3.35 grams of catalyst and 1.67 grams of *Mangifera Indica* Leaves extract resulting in a total weight of 18.41 grams. The second sample (Sample B) contains 13.39 grams of epoxy primer added with 3.35 grams of catalyst and 3.35 grams of *Mangifera Indica* Leaves extract resulting in a total weight of 20.09 grams. The third sample (Sample C) contains 13.39 grams of epoxy primer added with 3.35 grams of catalyst and 5.03 grams of *Mangifera Indica* Leaves extract resulting to a total weight of 21.76 grams. And the last sample (Plain Sample) contains 13.39 grams of epoxy primer added with 3.35 grams of catalyst resulting in a total weight of 16.74 grams, this sample does not contain any *Mangifera Indica* Leaves extract.



Figures 15 and 16. Total Weight of Sample A and Sample B



Figures 17 and 18. Total Weight of Sample C and Plain Sample

Table 2. Actual Measurements of the Samples
(MANGO LEAVES EXTRACT + PRIMER MIXTURE)

SAMPLE	EPOXY PRIMER	CATALYST (25%)	MANGO LEAVES EXTRACT	TOTAL WEIGHT OF MIXTURE
A	13.39 g.	3.35 g.	1.67 g.	18.41 g.
B	13.39 g.	3.35 g.	3.35 g.	20.09 g.
C	13.39 g.	3.35 g.	5.03 g.	21.76 g.
Plain	13.39 g.	3.35 g.	N/A	16.74 g.

Mangifera Indica Leaves extract are proven to be effective as a corrosion inhibitor on metal surfaces. But the MILs extract is crude oil-based which cannot be directly applied to the 6061-aluminum surface. The epoxy primer coating and catalyst are added to the MILs extract for it to bind and then applied to the metal surface.

The researchers then tested 3 different samples with different concentrations of MILs extract. The first sample which is Sample A contains 10 percent of the total weight of mixed epoxy primer coating and catalyst. Then Sample B contains 20 percent of the total weight of mixed epoxy primer coating and catalyst. Then lastly, Sample C contains 30 percent of the total weight of mixed epoxy primer coating and catalyst. The researchers kept the Plain sample as is, it does not contain any MILs extract.

Testing and Analysis

The researchers conducted testing in two parts in which they specifically tested the Mangifera Indica Leaves extract for its phytochemical composition via FTIR (Fournier-Transform Infrared Spectroscopy) testing at DOST-PTRI department. The other part of the testing is focused on corrosion inhibition and indication testing such as Salt Spray testing at DOST-MIRDC, and Weight Loss Method testing conducted by the researchers following the ASTM G31-72 guide as standard testing procedure.



Figures 19 and 20. Brought the mango leaves to AUTRDC for FTIR



Figures 21 and 22. Brought the 4 samples to MIRDC for Salt Spray



Figure 23. Soaking the samples in Hydrochloric Acid

Test Methods

The Mangifera Indica Leaves extract is tested for phytochemical composition via FTIR (Fournier-Transfer Infrared Spectroscopy testing at DOST-PTRI department). The FTIR result is then interpreted by a chemist at Adamson University.

The samples are tested after being subjected to the whole experimentation process. One of the batches that are subjected to the whole experimentation process is tested for Salt Spray testing at the DOST-MIRDC department. The other batch of sample that is also subjected to the whole experimentation process is tested for Weight Loss Method testing conducted by the researchers following the guideline from the ASTM G31-72 standard testing process. The mango leaves extract and epoxy primer combination that has been coated on the aluminum alloy coupon had undergone the standard test method of ASTM G31 – 72 which is the standard practice for laboratory immersion corrosion testing of metals. ASTM International is a global organization that sets consensus standards that are adopted and used globally across industries. Calculations of corrosion rates and the constant were adopted and strictly followed. To compute the inhibition efficiency together with the surface coverage, we need to observe the first event that the acid penetrates the coupon (Anusuya et al., 2015). In our research, the 15-minute mark indicates that the metal has been breached and the corrosion protection which is the MIL extract and epoxy primer combination has been diluted by the acid (hydrochloric acid). The succeeding minutes will greatly affect the aluminum coupon since the corrosion protector no longer protects the surface of the aluminum coupon.

Timeline of Gathering, Extraction, Testing, and Analyzing of Data
 Table 3. Timeline of the experimental study

September-October	October-November	November-December	December-January
Gathering of Materials <ul style="list-style-type: none"> • Mango Leaves • 6061 aluminum alloy sheet • Acetone • Hydrochloric • Ethanol 	Extraction of Mango Leaves.	Application of the mixture of Mango Leaf Extract and the Epoxy Primer to the 6061 Aluminum Alloy plates	Testing <ul style="list-style-type: none"> • Phytochemical Test <ul style="list-style-type: none"> ○ Fournier Transform Infrared (FTIR) • Surface Analysis <ul style="list-style-type: none"> ○ Salt Spray • Corrosion Inhibition Test <ul style="list-style-type: none"> ○ Weight Loss Method

Operation and Testing Procedure

In this study, the researchers will bring the sample subjects to the testing sites in order to get the necessary data needed.

Mangifera Indica Leaves Extract Characterization Phytochemical Test

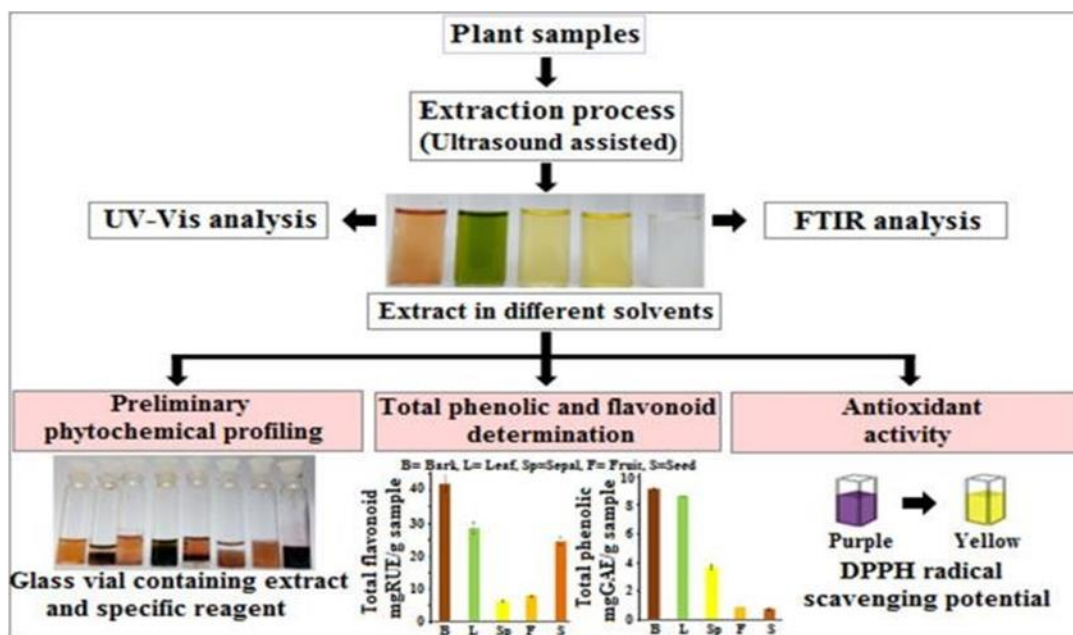


Figure 24. Process of Phytochemical Testing

In this procedure, plant samples such as leaves, stems, roots, or bark that are the reservoir of secondary metabolites are processed into aqueous and organic extracts. The presence of secondary metabolites such as alkaloids, terpenes, and flavonoids is next examined in the plant extracts. Through the use of the phytochemical test, the researchers will be able to determine the active components of the Mangifera Indica (Mango) leaves extract that is responsible for corrosion inhibition.

Fourier Transform Infrared (FTIR)

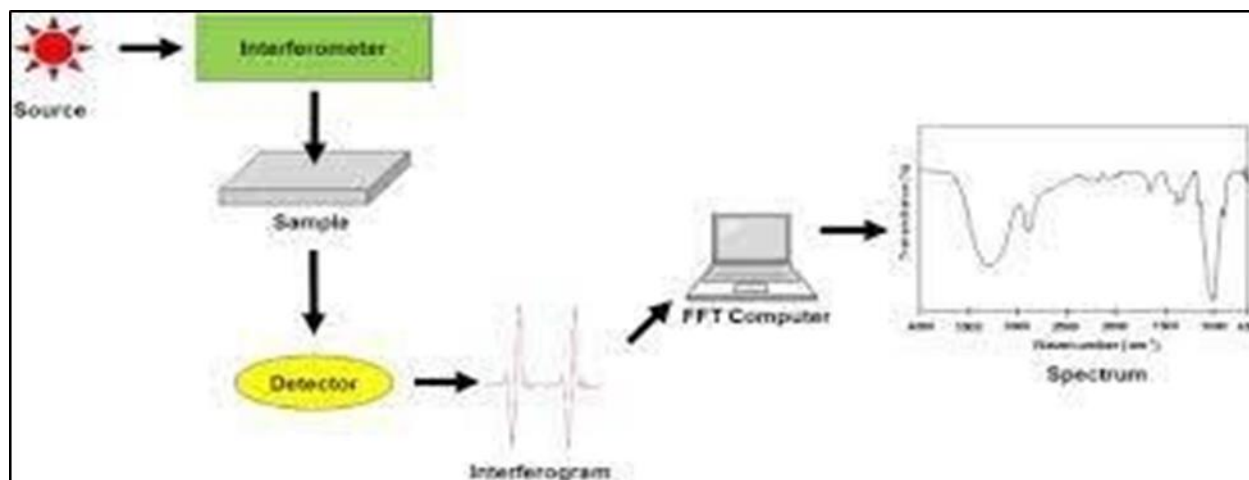


Figure 25. Fourier Transform Infrared (FTIR) process

For both organic and inorganic samples, FTIR provides quantitative and qualitative analyses. By creating an infrared absorption spectrum, Fourier Transform Infrared Spectroscopy (FTIR) can identify the chemical bonds in a molecule.

The spectra create a sample profile or specific molecular fingerprint, which can be used to screen and scan samples for a variety of components. FTIR is a useful analytical tool for identifying functional groups and describing information about covalent bonds. FTIR can also identify chemical compounds in consumer goods, paints, polymers, coatings, pharmaceuticals, foods, and other products.

Surface Analysis (Salt Spray)

In corrosive settings, metals and alloys are vulnerable to a variety of assault methods. For instance, corrosion damage to steel and aluminum alloys is frequently brought on by the chloride ion, which is naturally present in seawater. When chloride is present on metal surfaces, pitting corrosion of aluminum alloys frequently occurs when Cl⁻ ions are adsorbed on the natural oxide film during the beginning stage of corrosion. The pitting corrosion develops when the aluminum alloy is exposed to aqueous media such as saltwater. (Chanyathunyaraj, et al.)

After mixing the different samples individually, it is then applied to the bare 6061 Aluminum alloy sheet metal. After fully drying the applied coatings with different concentrations, the researchers immersed the coated samples in 30% HCL concentration for 5 minutes on the first trial of testing.

After the immersion process, the researchers then tested the immersed sample with Salt Spray testing at the DOST-MIRDC testing facility for corrosion testing. The immersed samples are exposed to the Salt Spray machine for 72 consecutive hours. The results then came out and all of the result samples indicated that there are no observable defects. The result is based on visual inspection with the use of magnifying glass. The immersion of samples to HCL for 5 minutes is not enough to penetrate the coated surface. Salt Spray testing is used to indicate corrosion on the surface of any exposed metal. Since there is no penetration on the coated surface, there is also no observable defect on the metal surface.

Actual Test Conditions	
Test Solution	5% Sodium Chloride (NaCl)
Volume of the collected solution, ml/hr	1.31-1.81
pH of collected solution	6.63-6.68
Specific Gravity of collected solution	1.030
Temperature, degree Celsius	35.4-35.6
Air Pressure, psi	17.5

Table 4. Parameters on Test Condition during Salt Spray testing.

Corrosion Inhibition Test (Weight Loss Method)

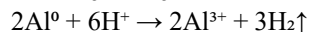
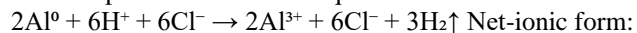
At normal temperature, aluminum and hydrochloric acid react. In hydrochloric acid, the metal dissolves, producing aluminum chloride and colorless hydrogen gas. Due to the fact that the final products won't interact with one another, this reaction is irreversible. An oxidation-reduction reaction occurs when metallic aluminum and hydrochloric acid come into contact. A salt and hydrogen gas are commonly produced in this reaction between a metal and an acid. (Truong-Son, et al.)

Reactions Mechanism

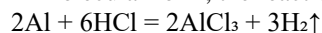
Step 1: Aluminum acts as the reducing agent, giving up electrons: $Al^0 - 3e \rightarrow Al^{3+}$

Step 2: Cations of hydrochloric acid take these electrons and are reduced to molecular hydrogen: $2H^+ + 2e \rightarrow H_2\uparrow$

The complete ionic reaction equation is as follows:



In molecular form, the reaction looks as follows:



The tests conducted on the mango leaves extract and epoxy primer combination that has been coated on the aluminum alloy coupon has been subjected to different standard formulas of the ASTM G31 – 72 and past research to assess the performance of the mango leaves extract and epoxy primer combination.

$$\text{Surface Coverage } (\theta) = \frac{W_b - W_i}{W_b}$$

Surface Coverage Formula

$$\text{Inhibition Efficiency } (\%) = \frac{W_b - W_i}{W_b} \times 100$$

Inhibition Efficiency Formula

Where:

W_b = Weight loss without inhibitor

W_i = Weight loss with inhibitor

$$\text{Corrosion Rate (CR)} = \frac{K \times W \times A \times T \times D}{\dots}$$

Where:

K = constant

T = time of exposure in hours to the nearest 0.01 h.

A = area in cm^2 to the nearest 0.01 cm^2

W = mass loss in g. D = density in g/cm^3

Given: $K=8.76 \times 10^4 mm/y$ $A=15 cm^2$

$D=2.70 g/cm^2$



Figure 26 and 27. Initial weight of Bare metal (left) and Sample B (right) before immersion to Hydrochloric Acid.



Figure 28 and 29. Initial weight of Sample C metal (left) and Sample D (right) before immersion to Hydrochloric Acid.



Figure 30. Initial weight of Plain sample before immersion to Hydrochloric Acid.

On the first 15 minutes of exposure, the bare metal has the highest weight loss of 5.91 grams. The bare metal did not have any protection at all. Sample A which has 10% concentration of MIL extract has the lowest weight loss of 0.16 grams, this means that Sample A has the highest protection against the HCL attack during the first 15 minutes of exposure. Sample B which has 20% concentration lost only 0.19 grams. Sample C which has 30% concentration lost 0.25 grams. And lastly, the plain sample which does not contain any MIL extracts.



Figure 31 and 32. Weight of Bare metal (left) and Sample B (right) after 15 minutes of immersion to Hydrochloric Acid.



Figure 33 and 34. Weight of Sample C (left) and Sample D (right) after 15 minutes of immersion to Hydrochloric Acid.



Figure 35. Weight of Plain sample after 15 minutes of immersion to Hydrochloric Acid.

After 30 minutes of exposure, the bare metal has the lowest weight loss of 0.02 grams. The bare metal still did not have any protection at all. Sample A which has 10% concentration of MIL extract lost 0.21 grams. Sample B which has 20% concentration lost 0.20 grams. Sample C which has 30% concentration lost 0.97 grams, it has the highest weight loss after 30 minutes of immersion. And lastly, the plain sample which does not contain any MIL extract lost 0.22 grams.



Figure 36 and 37. Weight of Bare metal (left) and Sample B (right) after 30 minutes of immersion to Hydrochloric Acid.



Figure 38 and 39. Weight of Sample C (left) and Sample D (right) after 30 minutes of immersion to Hydrochloric Acid.



Figure 40. Weight of Plain sample after 30 minutes of immersion to Hydrochloric Acid.

After 45 minutes of exposure, the bare metal still has the lowest weight loss of 0.01 grams. The bare metal still did not have any protection at all. Sample A which has 10% concentration of MIL extract lost 0.73 grams

Sample B which has 20% concentration lost 0.96 grams. Sample C which has 30% concentration lost 2.09 grams, it has the highest weight loss after 45 minutes of immersion. And lastly, the plain sample which does not contain any MIL extract lost 0.44 grams.



Figure 41 and 42. Weight of Bare metal (left) and Sample B (right) after 45 minutes of immersion to Hydrochloric Acid.



Figure 43 and 44. Weight of Sample C (left) and Sample D (right) after 45 minutes of immersion to Hydrochloric Acid.



Figure 45. Weight of Plain sample after 45 minutes of immersion to Hydrochloric Acid.

After 60 minutes of exposure, the bare metal still has the lowest weight loss of 0.01 grams. The bare metal still did not have any protection at all. Sample A which has 10% concentration of MIL extract lost 0.97 grams. Sample B which has 20% concentration lost 0.67 grams. Sample C which has 30% concentration lost 0.07 grams. And lastly, the plain sample which does not contain any MIL extract lost 2.12 grams, it has the highest weight loss after 60 minutes of immersion.



Figure 46 and 47. Weight of Bare metal (left) and Sample B (right) after 60 minutes of immersion to Hydrochloric Acid.



Figure 48 and 49. Weight of Sample C (left) and Sample D (right) after 60 minutes of immersion to Hydrochloric Acid.



Figure 50. Weight of Plain sample after 60 minutes of immersion to Hydrochloric Acid.

Evaluation Plan

Fourier Transform Infrared (FTIR)

FTIR testing would basically identify the composition of the mangifera indica leaves extract. Upon testing, we expect to get values lower than or more than 10% because we are using 99% ethanolic solution for the extraction process.

Our values should reach, or be lower than the following:

- Typical OH: 3334 cm^{-1}
- Carboxylic acid carbonyl (C=O) groups in phenolic compounds: 1031 cm^{-1}
- Aliphatic -CH stretching: 2936 cm^{-1}
- C-C stretching vibrations: 1612 cm^{-1}
- CH bending vibrations: 1450–1317 cm^{-1}

Corrosion Inhibition Tests Salt Spray Testing

The Salt Spray testing would indicate if there are any corrosion on the surface. The exposure of the sheet metal to saline solution would produce aluminum oxide on the surface if the coated sample is penetrated during the immersion of the sheet metal in HCL. To pass this test, the result should show that there are no observable defects on the surface.

Weight Loss Method

The weight loss method is done to show the difference in weight before and after the immersion process. The samples that contains MIL extract should have a better performance than the control sample to project its effectivity against it. Weight Loss method also shows the surface coverage and inhibition efficiency. The inhibition rate must be 90% above to show satisfactory performance.

Results and Discussions

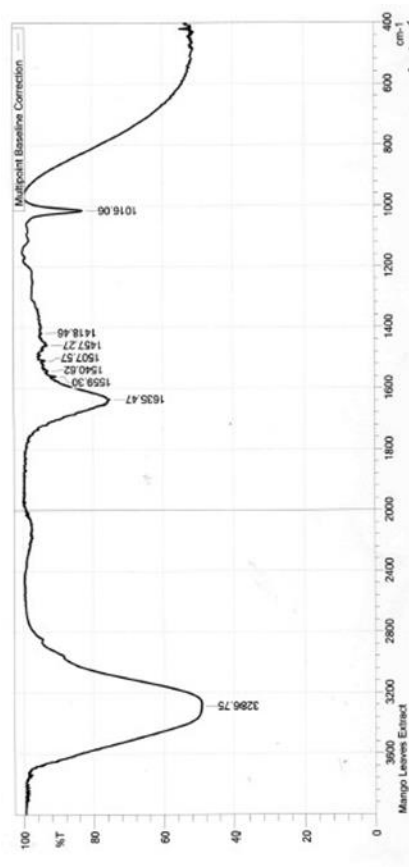


Figure 51. FTIR Test Result from DOST-PTRI department

FTIR Test

Based on the interpretation made by Mr. Kristoffer Lanze Andrew V. Molina, PGDip, RCh. FTIR spectrum results of Crude Extract of the sample compound showed that peaks at 3286 cm-1 indicates presence of OH-bond, peak at 2900 cm-1 showed presence of C-H anti-symmetric stretching, peak at 2830 cm-1, 1655 cm-1, 1450 cm-1 and 1115 cm-1 indicated presence of C-H symmetric stretching, C-O stretching, CH-CH bending and C-O bond. Peak at 1023 cm-1 showed presence of C-C stretching in the mangiferin structure. The collection of the bonds indicates functional groups that have antioxidant properties.

The figure above shows the FTIR spectrum result of the Mangifera Indica (Mango) Leaf extract. It can be observed that the figure shows different peaks, and through these peaks, the researchers will be able to determine the functional groups that correspond to the Mangifera Indica (Mango) leaf extract. The major peaks that we can observe in the figure, are the functional groups or structure of the molecules that can have an interaction with oxygen, and create chemical reactions. If oxygen will be able to have an interaction with other compounds, it will be neutralized, therefore, it will not be able to affect the surface of any metal and facilitate corrosion, because of this, the metal where the extract is applied to is being protected. Instead of the metal being corroded, the coating that was applied is the one that interacts with oxygen, and oxidizes. Some organic compounds contain antioxidant components, which are chemicals that can postpone, slow, or prevent oxidation. In other words, the presence of antioxidant chemicals in corrosive conditions can slow down the corrosion rate (Sari et al., 2018).

SALT SPRAY TESTING

Table 5. Results of salt spray testing on the different samples.

Sample	Remarks
Plain	No observable defect.
10% Concentration	No observable defect.
20% Concentration	No observable defect.
30% Concentration	No observable defect.

After mixing the different samples individually, it is then applied to the bare 6061 Aluminum alloy sheet metal. After fully drying the applied coatings with different concentrations, the researchers immersed the coated samples to 30% HCL concentration for 5 minutes on the first trial of testing. After the immersion process, the researchers then tested the immersed sample to Salt Spray testing at the DOST- MIRDC testing facility for corrosion testing. The immersed samples are exposed in the Salt Spray machine for 72 consecutive hours. The results then came out and all of the result samples indicated that there are no observable defects. The result is based on visual inspection with the use of magnifying glass. Immersion of samples to HCL for 5 minutes is not enough to penetrate the coated surface. Salt Spray testing is used to indicate corrosion on the surface of any exposed metal. Since there is no penetration on the coated surface, there is also no observable defect on the metal surface.

WEIGHT LOSS METHOD

After completely drying the samples that are coated with different concentrations, the researchers immersed all the samples in the HCL solution for a total of 1 hour. The immersion to HCL is divided into 4 equal parts, the samples are washed and weighed every 15 minutes since it was immersed. The longer exposure of the coated samples to HCL results in penetration on the coated surface. After the deterioration of the coating, the HCL starts attacking the sheet metal resulting in weight loss. With that being said, the researchers followed the ASTM G31 - 72 standard Practice for Laboratory Immersion Corrosion Testing of Metals.

Table 6. Weight Loss Data

	Initial	15 Minutes	Weight Loss	30 Minutes	Weight Loss	45 Minutes	Weight Loss	1 Hour	Weight Loss	Final Weight
Bare Metal	12.20	6.29	5.91	6.27	0.02	6.26	0.01	6.25	0.01	6.25 grams
10% Concentration	14.19	14.03	0.16	13.82	0.21	13.09	0.73	12.12	0.97	12.12 grams
20% Concentration	14.12	13.93	0.19	13.73	0.2	12.77	0.96	12.1	0.67	12.1 grams
30% Concentration	14.02	13.77	0.25	12.8	0.97	10.71	2.09	10.64	0.07	10.64 grams
Plain	14.08	13.82	0.26	13.6	0.22	13.16	0.44	11.04	2.12	11.04 grams

The table (refer to Table #) shows all the initial weight before being immersed in HCL solution. In the first 15 minutes of exposure, bare metal has the highest weight loss of 5.91 grams. The bare metal did not have any protection at all. Sample B, which has 10% concentration of MIL extract has the lowest weight loss with only a total of 2.07grams, this means that Sample B has the highest protection against the HCL attack during the first 15 minutes of exposure. While sample C (20% Concentration) also showed minor weight loss which maintained 12.1 grams of final weight. And lastly sample D (30% Concentration) and sample E (plain) had one of the highest weight loss recorded on the experiment conducted.

Table 7. Surface Coverage

	Initial	15 Minutes	Weight Loss	Surface Coverage
Bare Metal	12.20	6.29	5.91	N/A
10% Concentration	14.19	14.03	0.16	0.9729
20% Concentration	14.12	13.93	0.19	0.9679
30% Concentration	14.02	13.77	0.25	0.9577
Plain	14.08	13.82	0.26	0.9560

Table 8. Inhibition Efficiency

	Initial	15 Minutes	Weight Loss	Inhibition Efficiency (%)
Bare Metal	12.20	6.29	5.91	N/A
10% Concentration	14.19	14.03	0.16	97.29%
20% Concentration	14.12	13.93	0.19	96.79%
30% Concentration	14.02	13.77	0.25	95.77%
Plain	14.08	13.82	0.26	95.60%

The table shows the efficiency of the plain coating and the different concentrations. Based on the study conducted by Al-Amiery et al., 2020, an inhibition rate or efficiency of above 90% shows satisfactory performance for the corrosion inhibitor. This means that the combination of surface coverage and inhibition efficiency gives us an idea on how much of the surface area will be protected by the corrosion inhibitor. In our study, the sample with the 10% concentration showed the highest inhibition efficiency of 97.29%, it is followed by the 20% concentration which shows 96.79%. While the 30% concentration showed 95.77% inhibition efficiency and lastly the plain sample gave the lowest amount of inhibition efficiency of 95.60%. Although all of the samples garnered positive results, it only shows that the mango leaves extract and epoxy primer combination with 10% concentration had a greater efficiency rate than the plain epoxy primer with 1.69%. The inhibition efficiency played a crucial role in determining the performance of the mango leaves extract plus epoxy primer combination.

Table 9. Corrosion Rate (after 15 mins.)

	Initial	15 Minutes	Weight Loss	Corrosion Rate(mm/y)
Bare Metal	12.20	6.29	5.91	51,132.44 mm/y
10% Concentration	14.19	14.03	0.16	1,384.30 mm/y
20% Concentration	14.12	13.93	0.19	1,643.85 mm/y
30% Concentration	14.02	13.77	0.25	2,162.96 mm/y
Plain	14.08	13.82	0.26	2,249.48 mm/y

The table above presents the corrosion rate of the Aluminum alloy coupons after being submerged on Hydrochloric acid for fifteen minutes. The first fifteen minutes is crucial as the HCL is highly concentrated. It is very efficient in attacking the aluminum alloy's composition. The bare metal experienced a massive weight loss during the first fifteen minutes. Its corrosion rate is way high compared to the three other samples. The table also shows that the sheet metal with 10% concentration performed the best with the least corrosion rate value.

Table 10. Corrosion Rate (after 30 mins.)

	Initial	15 Minutes	Weight Loss	30 Minutes	Weight Loss	Corrosion Rate (mm/y)
Bare Metal	12.20	6.29	5.91	6.27	0.02	25,652.74 mm/y
10% Concentration	14.19	14.03	0.16	13.82	0.21	1,600.59 mm/y
20% Concentration	14.12	13.93	0.19	13.73	0.2	1,687.11 mm/y
30% Concentration	14.02	13.77	0.25	12.8	0.97	5,277.63 mm/y
Plain	14.08	13.82	0.26	13.6	0.22	2,076.44 mm/y

The table above presents the corrosion rate of the Aluminum alloy coupons after thirty minutes of being submerged on Hydrochloric acid. The corrosion rate values were way different from the first fifteen minutes. The bare metal had a slightly lower corrosion rate and the reason for this is that, unlike in the first fifteen minutes wherein the HCL was highly concentrated, it became diluted with the residues of the sheet metal. The table also shows that the sheet metal with 10% concentration still performed the best, possessing the least corrosion rate value.

Table 11. Corrosion Rate (after 45 mins.)

	Initial	15 Minutes	Weight Loss	30 Minutes	Weight Loss	45 Minutes	Weight Loss	Corrosion Rate (mm/y)
Bare Metal	12.20	6.29	5.91	6.27	0.02	6.26	0.01	17,130.67 mm/y
10% Concentration	14.19	14.03	0.16	13.82	0.21	13.09	0.73	3,172.35 mm/y
20% Concentration	14.12	13.93	0.19	13.73	0.2	12.77	0.96	3,893.33 mm/y
30% Concentration	14.02	13.77	0.25	12.8	0.97	10.71	2.09	9,545.88 mm/y
Plain	14.08	13.82	0.26	13.6	0.22	13.16	0.44	2,653.23 mm/y

The table above shows the corrosion rate of the Aluminum alloy coupons after 45 minutes of being submerged to Hydrochloric acid. The corrosion rate of the bare metal is still the highest with a value of 17,130.67 mm/y. In the coated samples, the table shows that the plain sample has the lowest corrosion rate with a values of 2,653.23 mm/y compared to other coated samples, it is then followed by the sample that has 10% concentration with a value of 3,172.35 mm/y, which is then followed by the 20% concentration that has a value of 3,893.33 mm/y. Among all of the coated samples, the 30% concentrations have the highest corrosion rate, with a value of 9,545.88 mm/y.

Table 12. Corrosion Rate (after 60 mins.)

	Initial	15 Minutes	Weight Loss	30 Minutes	Weight Loss	45 Minutes	Weight Loss	1 Hour	Weight Loss	Corrosion Rate (mm/y)
Bare Metal	12.20	6.29	5.91	6.27	0.02	6.26	0.01	6.25	0.01	12,869.63 mm/y
10% Concentration	14.19	14.03	0.16	13.82	0.21	13.09	0.73	12.12	0.97	4,477.33 mm/y
20% Concentration	14.12	13.93	0.19	13.73	0.2	12.77	0.96	12.1	0.67	4,369.19 mm/y
30% Concentration	14.02	13.77	0.25	12.8	0.97	10.71	2.09	10.64	0.07	7,310.81 mm/y
Plain	14.08	13.82	0.26	13.6	0.22	13.16	0.44	11.04	2.12	6,575.41 mm/y

The table above shows the corrosion rate of the Aluminum alloy coupons after 1-hour of being submerged to Hydrochloric acid. The corrosion rate of the bare metal is still the highest with a value of 12,869.63 mm/y, but compared to the past minutes, it can be observed that its corrosion rate is decreasing. In the coated samples, the table shows that the sample that has 20% concentration has the lowest corrosion rate with a value of 4,369.19 mm/y, which is then followed by the sample that has 10% concentration that has a value of 4,477.33 mm/y, which is then followed by the 30% concentration, with a value of 7,310.81 mm/y. It can also be observed in this table, that compared to the past minutes, the corrosion rates of the coated samples are increasing because the acid has already penetrated the coating.

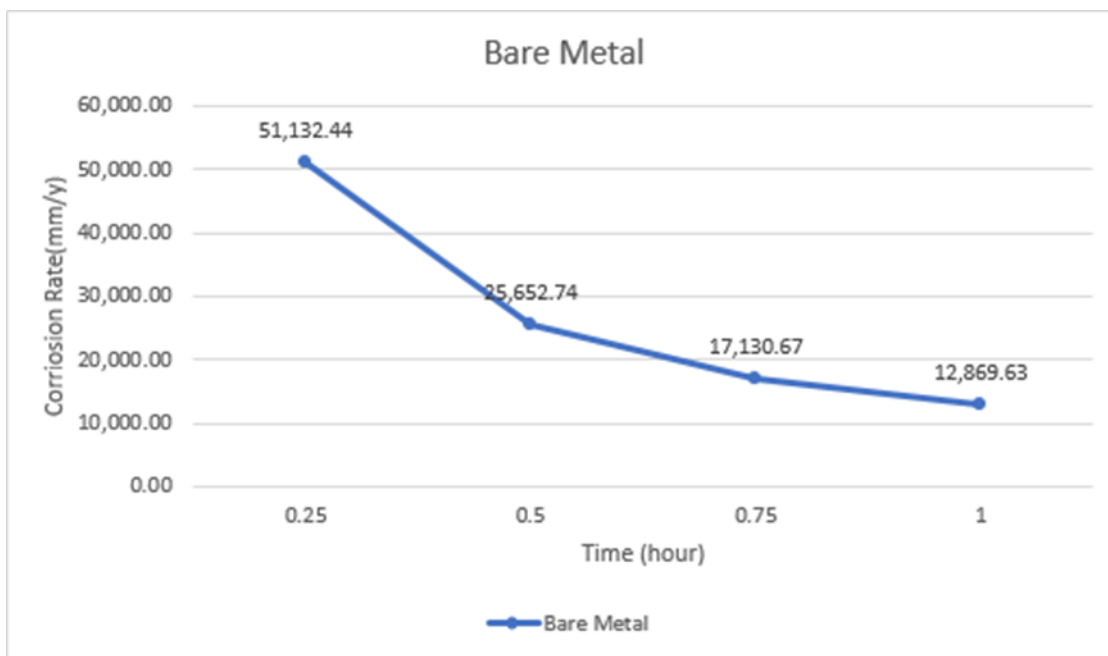


Figure 52. Corrosion Rate of Bare Metal

The figure above shows the corrosion rate of the bare metal sample in various time, specifically, 0.25 hour, 0.50 hour, 0.75 hour, and 1 hour. It can be observed that the graph is in a downtrend which means that the corrosion rate of the bare metal is decreasing. The figure also shows a significant drop in corrosion rate between 0.25 hour to 0.50 hour of submerging the aluminum alloy coupon to the hydrochloric acid

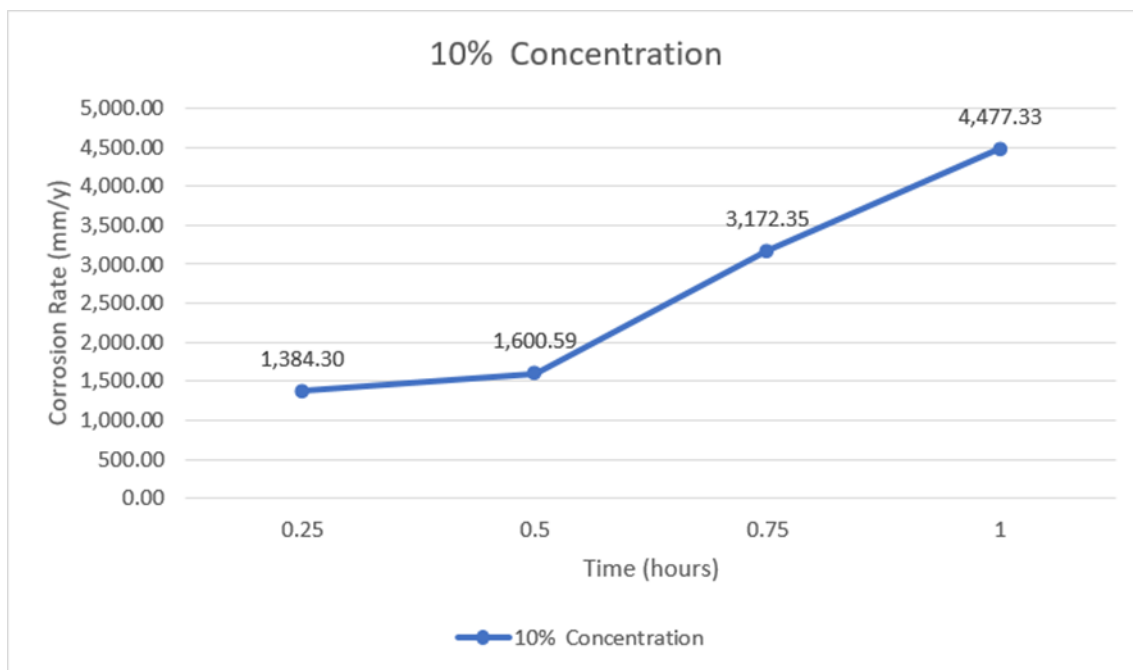


Figure 53. Corrosion Rate of 10% concentration

The figure above shows the corrosion rate of the 10% concentration sample in various time, specifically, 0.25 hour, 0.50-hour, 0.75 hour, and 1 hour. It can be observed that the graph is in an uptrend which means that the corrosion rate of the 10% concentration sample is increasing.

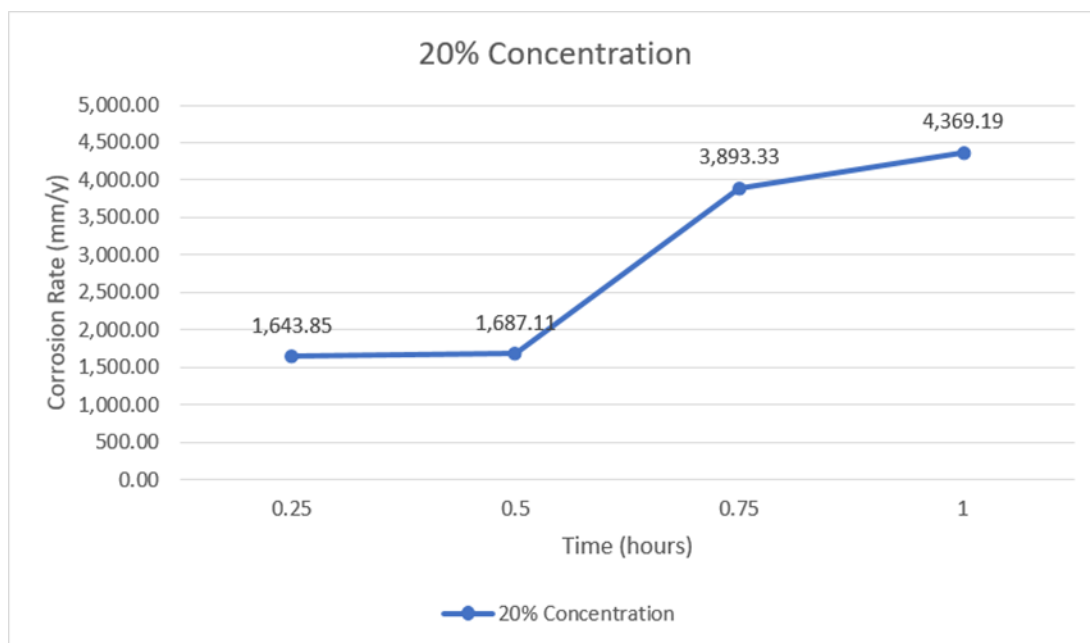


Figure 54. Corrosion Rate of 20% concentration

The figure above shows the corrosion rate of the 20% concentration sample in various time, specifically, 0.25-hour, 0.50 hour, 0.75 hour, and 1 hour. It can be observed that the graph is in an uptrend which means that the corrosion rate of the 20% concentration sample is increasing.

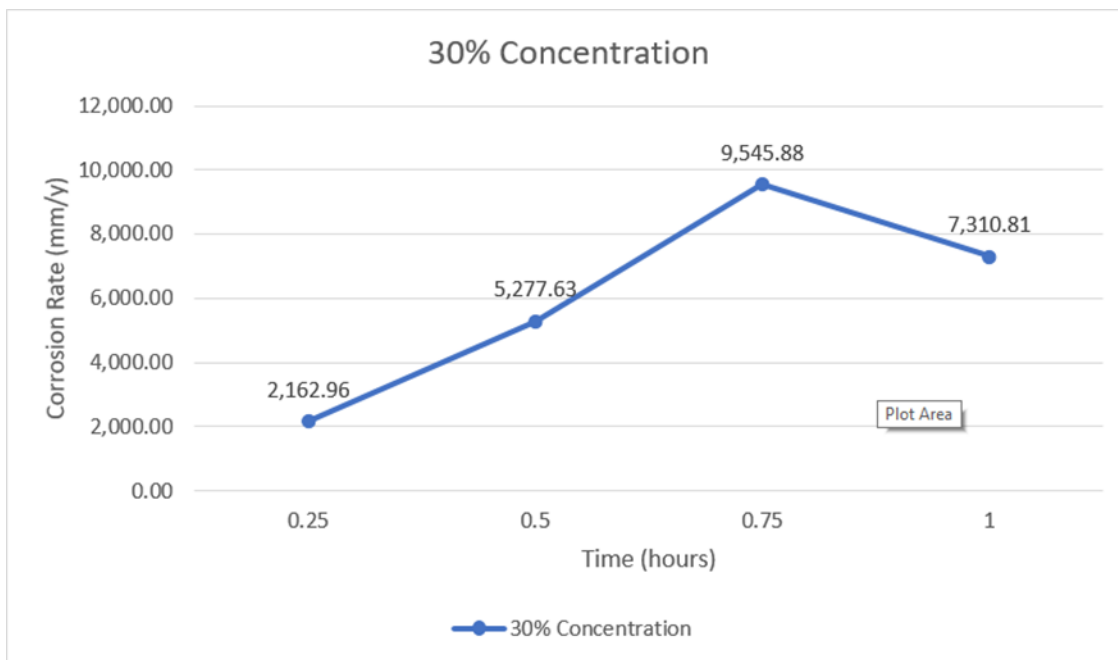


Figure 55. Corrosion Rate of 30% concentration

The figure above shows the corrosion rate of the 30% concentration sample in various time, specifically, 0.25 hour, 0.50 hour, 0.75 hour, and 1 hour. It can be observed that the graph is in an uptrend from 0.25 hour to 0.75 hours which means that the corrosion rate of the 30% concentration sample is increasing, but between the 0.75 hour to 1 hour it can be observed that there is a drop in corrosion rate.

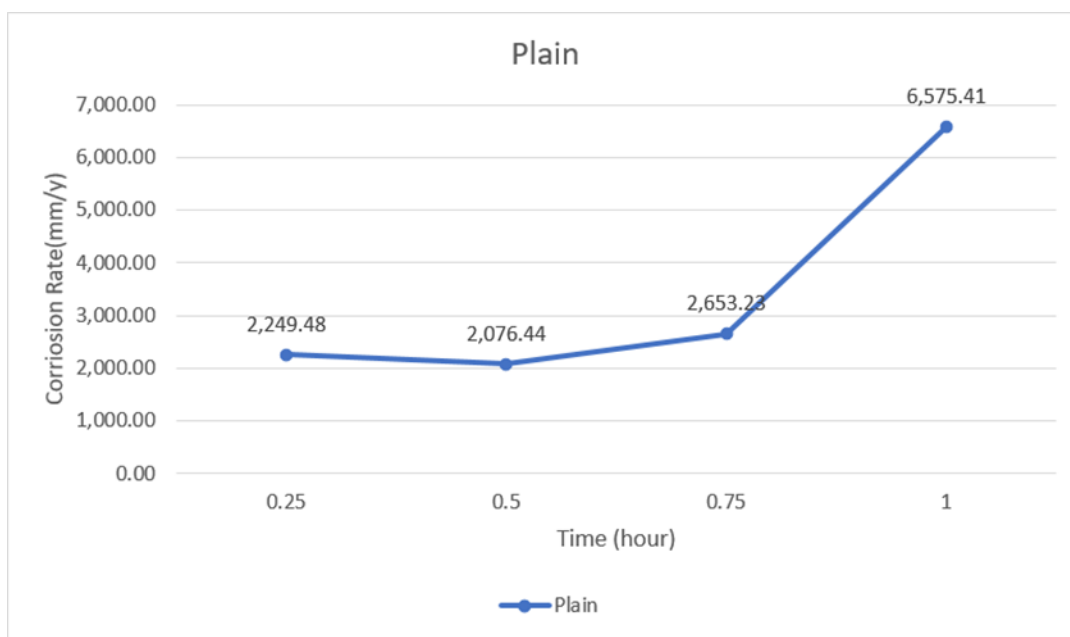


Figure 56. Corrosion Rate of Plain

The figure above shows the corrosion rate of the Plain sample in various time, specifically, 0.25 hour, 0.50 hour, 0.75 hour, and 1 hour. It can be observed that the graph is in an uptrend which means that the corrosion rate of the Plain sample is increasing.

The figures above show the corrosion rate of the Aluminum Alloy coupons that were used in this study, which are the bare metal, plain, sample with 10% concentration, sample with 20% concentration, and sample with 30% concentration in various time, specifically, 0.25 hour, 0.50 hour, 0.75 hour, and 1 hour. We can visually observe on the graphs presented above that there are different trends, for the bare metal and the sample with 30% concentration at 0.75 hour mark, we can observe that it has a downtrend which means that the corrosion rate is decreasing, and this is due to the particles that have worn off the metal and have mixed with the hydrochloric acid causing it to have a change in concentrations. We can also observe that the coated samples have an uptrend in corrosion rate, which means that their corrosion rate is increasing because the coating have only been depleted between 0.25 hour and 0.50 hour, and there is a high amount of oxidation since it is the first contact of the acid to the surface area of the Aluminum alloy coupon. We can therefore interpret that the surface area of the bare metal is being consumed at a faster rate since it has no coating and the aluminum particles have already been mixed with the acid, while for the sample with 30% concentrations it had a downtrend in corrosion rate because it is the first metal that had a contact with the acid causing its surface area to be consumed faster, and at 0.75 hour the hydrochloric acid where it is submerged already had a decrease in concentration.

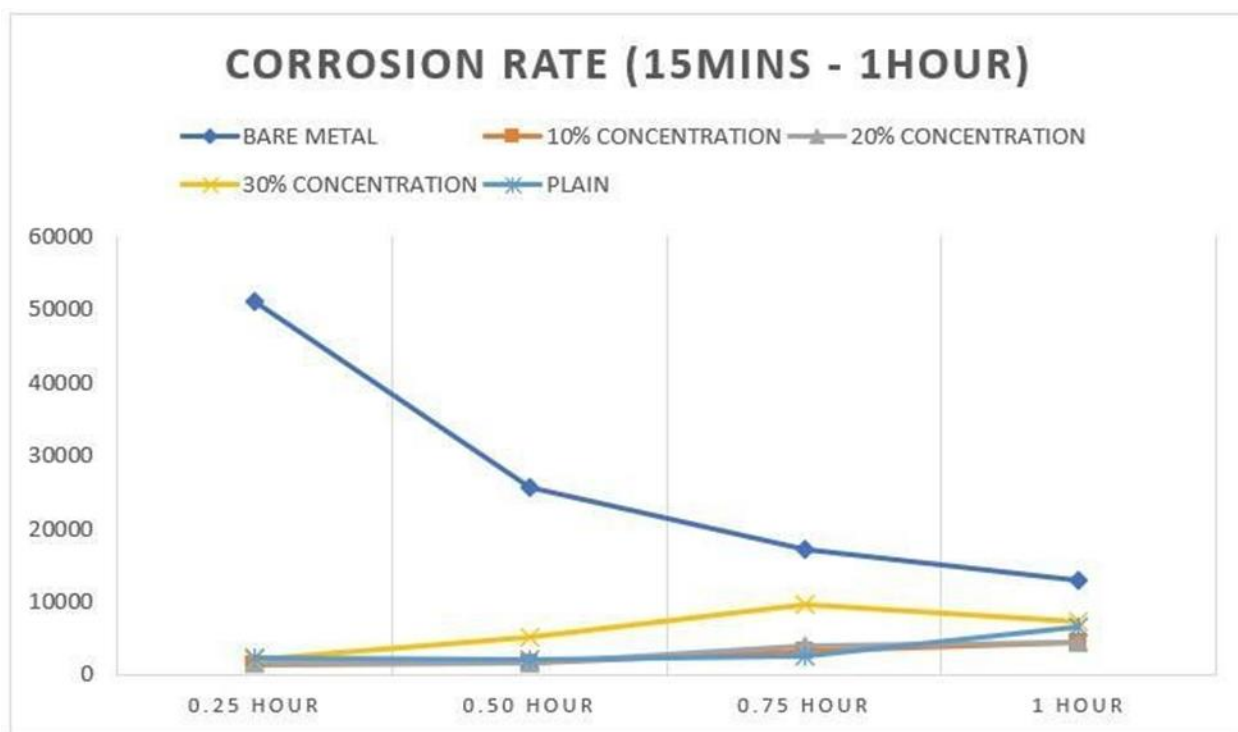


Figure 57. CORROSION RATE (15 MINS-1 HOUR)

The figure shows the corrosion rate of each sample in various times, specifically 0.25 hour, 0.50 hour, 0.75 hour, and 1 hour. We can see in the figure that during the 0.25 hour mark the corrosion rate of the sample B (10% concentration) showed the lowest corrosion rate of 1,384.30 mm/y with a significant lowered rate compared to the sample A (bare metal) which gained a corrosion rate of 51,132.44 mm/y. The 0.25th hour mark of the study gives us the crucial point on which we can determine the corrosion rate of the samples without letting the acid penetrate the surface of the aluminum alloy coupon. Between the 0.25 hour and 0.50 hour mark of submerging the aluminum alloy coupon in the hydrochloric acid, we observed that the coating were depleted and pitted resulting the acid to perforate the surface of the coupon and resulting in high amount of oxidation on the first contact of the acid on the surface area. But still at 0.50-hour, sample B (10% concentration) still showed great performance against the redox reaction with a total weight

loss of only 0.37 at 1,600.59 mm/y corrosion rate. As we proceed to the 1-hour mark of the test it still shows how sample B (10% concentration) and sample C (20% concentration) had one of the lowest corrosion rates while sample E (plain) gained a slight lead to sample D (30% concentration) in terms of corrosion rate. We can interpret here that samples B and C had a satisfactory rate and significant lead among other samples provided. We can see how far the gap between the corrosion rate of coated coupons and bare metal/coupon.

Based on the surface analysis tests that were conducted by the researchers. There is a significant difference in the anti-corrosive performance of mango leaves extract in different concentrations on 6061 alloy during HCL attack. Firstly, the researchers mixed different concentrations of the sample and applied it to the bare 6061 Aluminum alloy sheet metal. Once the coating fully dried, the researchers immersed the coated samples to 30% HCL concentration in a duration of 5 minutes on the first trial of testing. After the immersion of the sheet metal, the researchers then brought the immersed samples to DOST-MIRDC for them to conduct the Salt Spray test. The immersed samples are exposed to the Salt Spray machine for 72 straight hours. After that, the results were released, and every sample showed that there are no observable defects that can be seen. The inspection utilized a magnifying glass in order to determine the result. Five minutes of HCL immersion in the samples is insufficient to permeate the covered surface. Testing with salt spray is used to detect corrosion on any exposed metal's surface. Since there is no penetration on the coated surface, the result is that there is no observable defect on the metal surface. Moving on to the Weight loss method that was conducted by the researchers, here we can see the significant difference in anti-corrosive performance of the mango leaves extract as we weigh the different samples after immersing them on HCL for an hour. The samples are rinsed and weighed every 15 minutes after the HCL immersion, which is divided into 4 equal parts. As samples are exposed to HCL for a longer period of time, the coated surfaces become more permeable. After the coating starts to deteriorate, the HCL begins to attack the sheet metal, which causes weight loss. Having stated that, the researchers adhered to the ASTM G31 - 72 standard Practice for Laboratory Immersion Corrosion Testing of Metals.

The advantages and the disadvantage of using mango leaves extract as a natural corrosion inhibitor are listed above. The researchers have listed three of each and all of them are crucial in weighing the efficiency and utilization of the mango leaves extract for corrosion inhibition. When it comes to the advantages, first on the list is that the mango leaves extract is a natural corrosion inhibitor. This is a massive breakthrough in terms of significance in corrosion inhibition, as we all know, the aviation industry is known to use rust inhibitors a lot. The use of these synthetic materials can be harmful and toxic that's why the utilization of a natural corrosion inhibitor such as the mango leaf extract will provide a safer way of corrosion inhibition which can also help in protecting the environment and the health of personnel. Second is that, mango trees are abundant in our country. The source of the mango leaves, which is the mango tree is abundant in the Philippines. Our country is known locally and internationally to be a large producer of mangoes. Its availability won't be a problem as long as preserve these trees and plant more of them. Thirdly, mango leaves extract is an effective natural corrosion inhibitor. The feasibility of the mango leaves extract as a corrosion inhibitor was proven in our study through the different testing methods that we performed. The FTIR, Salt spray testing, and most importantly, the Weight loss method, have proven the effectivity of the mango leaves extract in terms of being an effective natural corrosion inhibitor.

Moving on to the disadvantages of the mango leaves extract, the researchers have also listed three. The first is its compatibility with the medium for application can be finicky. Since it is an oil-based extract, it doesn't easily mix with just any kind of paint. The researchers have experienced this kind of struggle during the first batch of aluminum alloy coupons that we coated. We bought a small can of unbranded epoxy primer from a paint shop and tried to mix the mango leaves extract with it. However, the two did not mix well and we reached the point of using a drill in order to mix them forcibly. We somehow succeeded but it brought us up the second disadvantage which we will discuss later on. The difficulty in compatibility was solved when we tried another epoxy primer, the brand of this epoxy primer is Guilder. Mixing the mango leaves extract with the Guilder epoxy primer became easier wherein we didn't even have to use a drill just to mix them. The second disadvantage is that the application of the mango leaves extract to the aluminum alloy coupons can be quite difficult. As the researchers were saying earlier, the compatibility brought up this disadvantage. Since the mango leaves extract did not mix well with the unbranded epoxy primer, its oil-based property made it difficult for the researchers to apply it on the aluminum alloy coupons. The mixture doesn't stick and just slides off the alloy. Our solution for this was to use our fingers in application in order to put a thin coating that will stick to the alloy, let it dry well, put another coating, and repeat the cycle. This disadvantage was also resolved when we used the Guilder epoxy primer as it was easier to apply. Lastly, the cost effectivity and feasibility of the mango leaves extract for mass production is still questionable. The reason for this is that even though the mango leaves are abundant in our country, its extraction can be quite expensive.

Conclusion

The researchers conduct this study to determine and evaluate the feasibility of Mangifera Indica leaves extract as a natural corrosion inhibitor on 6061 Aluminum alloy in HCL attack. The study also ought to know the corrosion control compound and components, anti-corrosive performance, and the disadvantage and advantage of MIL extract. One of the ideas of the study is also to promote the use of materials that is naturally around rather than using pure synthetic materials that may further damage the environment.

Based on the FTIR testing (Fournier-Transfer Infrared Spectroscopy), the Mangifera Indica Leaves extract contains functional groups that can neutralize oxygen during its reaction with other molecules. Specifically, functional groups contain antioxidants. This the key component in the phytochemical composition of the MIL extract that makes it a corrosion inhibitor.

Before exposing the coated sample to salt spray testing, it is immersed first in HCL acid for penetration test. The researchers used salt spray testing to indicate corrosion on the surface. The result of the testing is noted as “No observable defects”. Salt Spray testing result simply indicates that the coating has protected the sheet metal from corroding. Therefore, the coating successfully protected the sheet metal from the HCL attack during immersion.

After conducting the Weight-Loss Method on the non-coated and coated sample with different concentrations, the result shows that the sample that does not have any coating on it has lost a lot of weight due to the HCL attack. As what the researchers observed, the first 15 minutes of HCL attack is the key point in indicating the overall performance of the coatings. All of the coating is damaged no matter what the concentration of coating is applied on a certain sample. But the notable difference is that the sample with 10% percent concentration, which is Sample B, performs better than the sample that does not contain any MIL extract, which is the Plain Sample. Once the HCL penetrated the surface of the sheet metal, may it contain MIL extract or not, the HCL will start to corrode and dissolve the sheet metal. One of the key takeaways from the testing is that the sample that does not have any coating deteriorates almost 3 times faster than the samples that has coating. At this point, the result from the FTIR testing is relatable to the results from the surface analysis-related testing that was conducted. The components of MIL extract serve as an added protection for the first line of defense against oxidation.

Based on the findings of the study, it is therefore concluded that the Mangifera Indica leaves extract can be very good inhibitor combined with a binding solution which is the epoxy primer, can be effectively utilized as an additional layer of protection to the 6061 Aluminum alloy during HCL attacks. Having said that, the researchers agree that the study can be strengthened still more by being the topic of a more exhaustive and complete investigation.

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