# EFFECTS OF ADDITIVE ON THE CORROSION BEHAVIOUR OF ALUMINUM METAL UPON EXPOSURE TO PALM BIODIESEL AT ROOM TEMPERATURE



## EFFECTS OF ADDITIVE ON THE CORROSION BEHAVIOUR OF ALUMINUM METAL UPON EXPOSURE TO PALM BIODIESEL AT ROOM TEMPERATURE

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JANUARY 2025

## DECLARATION

I declare that this project report entitled "Effects Of Addititve On The Corrosion Behaviour Of Aluminum Metal Upon Exposure To Palm Biodiesel At Room Temperature" is the result of my own work except as cited in the references



## APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature	:
Name of Supervisor	: <u>Dr Nurhidayah Binti Ismai</u> l
Date	. 21 Feb 2025

#### **DEDICATION**

The writing of this report is dedicated to my parents, family, supervisor, and colleagues, provided that their support, love and encouragement have been my foundation throughout this journey. Everyone contributed to my inspiration and determination for the completion of this writing. I am truly grateful for all the sacrifices, patience and for the belief in me which had made the completion of this work possible. Additionally, I would love to thank my friends and mentors that have been providing me all the guidance and emotional support. Finally, I hope this work of mine will help any students in inspiring them to keep pushing and not giving up

until they have exceed their limit.

#### ABSTRACT

This study purposely conducted to investigate the effects of an additive (Pyrogallol, PY) on the corrosion behaviour of aluminum metal when exposed to palm biodiesel (B20) at room temperature by the compliance ASTM G31 static immersion test. The primary objective is to identify the influence of additive on the corrosion rate, pitting formation and the oxidation stability of the aluminum coupon over time. The results of the static immersion test within 30 to 150 days provide significant colour changes in the biodiesel solution particularly in B20+PY. The findings indicate the presence of unsaturated fatty acid methyl ester (FAME) of the biodiesel reaction with the trapped oxygen and the PY. The corrosion rate of the aluminum coupon in B20+PY was consistently lower than the pure B20 throughout the immersion test. The condition supports the suggesting that PY enhances the formation of a protective aluminum oxide layer. Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis post immersion confirmed the formation of this protective oxide layer which was more significant in B20+PY. The findings also show the effectiveness of PY in improving the corrosion behaviour of the aluminum coupon by reducing the oxidative stress and enhancing the natural passivation layer. All of those mentioned play an important role in minimising localized corrosion. It is suggested that further research should include more detailed methodologies for cross sectional analysis in order to asses the corrosion at higher precision. اونيۈمرسىتى تيكنىكل مليسيا

## ACKNOWLEDGEMENT

I would like to show my appreciation to my supervisor Dr Nurhidayah Binti Ismail for providing me the opportunity to conduct my final year project with her. She never has been supportive and giving me the proper guidance whenever I encountered difficulties throughout the year. I am thankful for her patience and commitment in making sure of the success for my final year project.

Besides, I would like to thank the lab assistant, Puan Nurhidayah and Encik Azhar for their help during the lab session. Their contribution is significant for the completion of this final year project. My coursemates, family and everyone involved with this final year project deserve my gratitude.



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## LIST OF ABBREVIATIONS

- PY Pyrogallol
- FAME Fatty Acid Methyl Ester
- ASTM American Society for Testing and Materials



#### **CHAPTER 1**

#### INTRODUCTION

### 1.1 Background

Biodiesel as a renewable and sustainable alternative fuel is getting more significant to replace diesel fuel completely or partially. It is derived from natural sources such as vegetable oils, animal fats and waste cooking oil (Kugelmeier et al., 2021). However, the adoption of biodiesel particularly within the automotive sector has caused us to encounter both advantages and disadvantages that require careful examination. The advantages offered by the biodiesel is it has the potential to mitigate the environmental pollution and reduce the dependence on finite petroleum resources. These traits of the biodiesel are aligned with the growing concerns over the need for energy security. Nevertheless, the extensive use of biodiesel has led us to challenges such as metallic corrosion particularly significant in automotive applications. This type of corrosion can affect engine longevity due to biodiesel's incompatibility with certain metallic and polymeric materials (Kugelmeier et al., 2021). In addition, biodiesel presents several challenges such as high density, low heating value and high fuel consumption. These issues can be addressed through the use of additives which play a crucial role in enhancing engine performance, combustion efficiency, emission levels and overall physicochemical properties. Additives are chemical compounds employed to improve the characteristics of fuel including diesel, biodiesel and gasoline (Yadav et al., 2020).

### **1.2 Problem Statement**

As we progress towards the use of pure biodiesel B100 from the current B10 blend, it is significant for us to go through the intermediary stages such as the adoption of B20, B30 and so forth. For example, there are many countries around the world that have successfully embraced higher biodiesel blends including the United States (B5 to B20), Indonesia (B20) and Brazil (B11). However, the adoption of biodiesel comes with an issue regarding the high oxidizing tendency that cause corrosion on automotive materials. The high oxidizing tendency is due to the increasing water content that carry oxygen (Kugelmeier et al., 2021).

The use of biodiesel as a cleaner alternative to diesel has its advantages and disadvantages in the automotive sector. While biodiesel helps reduce pollution and dependence on limited oil resources, it also brings challenges especially for vehicles. Its usage presents significant challenges such as causing metallic corrosion on materials used in automotive industry. Some of the materials used are carbon steel, stainless steel, aluminum and copper. Stainless steel is used in fuel injector pumps and exhaust systems. Aluminum in fuel feed pump, fuel pump, cylinder and piston. Copper and its alloys in gasket, housing and bearing. Carbon steel is mainly used in the manufacture of tanks for fuel storage (Kugelmeier et al., 2021).

The challenge poses a significant threat to automotive materials concerning their oxidation stability which is crucial for engine longevity. The resistance to oxidation could potentially lead to a shortened lifespan of the engine. Moreover, the challenge extends beyond operational implications such as affecting storage conditions over prolonged periods. The formation of slush worsens the situation by potentially damaging the engine components and affecting the engine performance. Furthermore, the effects of this issue are not limited to the engine component but also impact other metal parts within the engine.

These complexities show the need for comprehensive prevention strategies within the field of automotive engineering. In order to prevent this issue and make biodiesel more practical, an effective solution is required to ensure the reliability of biodiesel as a fuel source. Additive emerges as a promising solution to address this issue. Additive such as the Pyrogallol (PY) is applied to improve the properties of biodiesel. By incorporating additive, it becomes possible to tackle the limitation associated with renewable fuels and decrease the reliance on conventional petroleum-based counterparts (Yadav et al., 2020).

## 1.3 Objective

The objectives of this study are as follows:

- 1. Investigate the effects of additive on the corrosion behaviour of aluminum metal in palm biodiesel at room temperature.
- 2. Evaluate the role of antioxidant additive in protecting aluminum metal against corrosion in palm biodiesel.

### 1.4 Scope of Study

1.

The scopes of this study are:

Biodiesel used is B20 and B20 + additive.

2. Metal used is an aluminum coupon that replicate the automotive part (fuel feed pump, fuel pump, cylinder and piston). Aluminum is selected as the sample material because of the limited research conducted on this material compared to other automotive materials. In one of the previous studies involving corrosion test in biodiesel, aluminum has shown a good compatibility and did not show any compound formed on its surface without presenting any corrosion

(Kugelmeier et al., 2021).

- Static immersion test at room temperature following the ASTM G31 standard.
   Range of immersion between 30 150 days.
- 4. Corrosion rate determined by using weight loss measurement.
- Metal surface examination using 3D Non-Contact Profilometer and Scanning Electron Microscope (SEM) with Energy Dispersive Xray (EDX)

## **CHAPTER 2**

#### LITERATURE REVIEW

### 2.1 **Biodiesel Properties**

Biodiesel derived from animal fats and vegetable oils exhibits several challenges in terms of corrosion and oxidation. This renewable and sustainable biodiesel has properties that jeopardize the stability and durability of biodiesel. The triglycerides in these feedstocks are prone to oxidize which lead to the formation of peroxides and acids over time. Thus, degrading the fuel quantity and engine performance. The oxidation instability is due to the presence of unsaturated fatty acids that vulnerable to react with oxygen. The process also results in the formation of corrosive by products that can damage fuel system components such as fuel injectors, pump and storage tanks. The animal fats contribute to the corrosiveness due to their higher saturation levels that cause the formation of deposits and blockage in fuel lines (Kugelmeier et al., 2021).



Figure 2.1 Colour of biodiesel before and after contact with CS, SS, Al and Cu (Kugelmeier et al., 2021)

## 2.2 Automotive Materials

Automotive compartments that come into direct contact with fuel such as fuel tank and fuel pump are made of aluminum due to its mechanical properties and corrosion resistance (Kugelmeier et al., 2021; Miller et al., 2000). Its corrosion resistance makes it a reliable choice in ensuring longevity and durability of the compartment. Besides, the non-corrosive nature of aluminum helps to ensure the fuel remain uncontaminated. Thus, maintaining the purity and efficiency of the combustion process. It also had the ability to dissipate heat efficiently which helps in maintaining optimal operating temperatures for fuel pump. This shows that aluminum as the selected material provide the ability to prevent overheating potential failure. The use of aluminum in automotive industry reflects a balance in fuel efficiency, durability and performance (Miller et al., 2000; Fazal et al., 2011)



Figure 2.2 Aluminum 5052 fuel tank plate Source: https://www.mt-aluminum.com

## 2.3 Effect of temperature on the corrosion behaviour of material

The effect of temperature on the corrosion behaviour of metals in biodiesel is significant because higher temperature generally accelerates the corrosion rate. The increase in temperature boosts the kinetic energy of the reactant molecule that led to an increased in the rate of chemical reactions including those that cause the corrosion. In the context of biodiesel, higher temperature can exacerbate the oxidative degradation of the fuel which produce more acidic by-products such as peroxides and organic acids. These acidic compounds are highly corrosive and can aggressively attack the metal surface for a faster material degradation. Metals such as aluminum, copper, and various steel alloys are vulnerable to the accelerated corrosion at higher temperature. Furthermore, increased temperatures can cause the biodiesel to absorb more water. This will further facilitate the corrosion processes like pitting and crevice corrosion. This thermal effect is concerning in the automotive applications because compartment may be exposed to fluctuating temperatures during operation (Fazal et al., 2011)



Figure 2.3 Corrosion rate of mild steel after exposure to B0, B50 and B100 for 1200h at different temperatures (Fazal et al., 2011)

## 2.4 Effects and properties of antioxidant additives

Additives play an important role in enhancing the properties of biodiesel especially in terms of combination, deposit formation and corrosion during long-term storage. Antioxidants such as tert-Butylhydropquinone (TBHQ), butylated hydroxylanisole (BHA), diphenylamine (DPA), butylated hydroxyl toluene (BHT), prophy gallate (PG) and pyrogallol (PY) commonly added to biodiesel to prevent oxidation degradation. Table 2.1 below provides the common list of additives used in specific biodiesel based on previous research. The application of these antioxidants reduces the formation of acidic by products that can cause corrosion. They stabilize the fuel by neutralizing free radicals and slowing down the oxidation process. By effectively preventing or slowing down oxidation, antioxidant additives help maintain the fuel's quality which prevent engine deposits and ensure reliable engine operation over extended period of storage or use. Thus, improving the overall stability and performance of biodiesel fuels (Yadav et al., 2020).

Table 2.1 List of additives commonly us	ed in specific biodiesel and comparative
analysis of performance and emission ef	fects of additives on different biodiesels
(Yadav et	al., 2020)

S. no	Fuel	Additives	Brake power	BTE	BSFC	со	нс	NOx	References
11.	H2 biodiesel dual-fuelled jatropha methyl ester (JME) biodiesel	Zinc oxide nanoparticle fuel additive	-	-	-	Increase	Decrease	Increase	[27]
12.	Jatropha methyl ester	Antioxidant additives, ethylenediamine, butylated hydroxytoluence, a tocopherol acetate, p- phenylenediamine and L ascorbic acid	-	-	Decrease	Increase	Increase	Decrease	[28]
13.	Jatropha biodiesel	N'-diphenyl-I, 4- phenylenediamine antioxidants	-	Same	Same	Decrease	Decrease	Decrease	[29]
14.	Jatropha biodiesel	Nano-aluminium oxide (n-Al2O3)	Increase	Increase	-	-	-	-	[30]
15.	Jatropha- palm blended biodiesel	Blended biodiesel	-	-	-	Increase	-	Increase	[31]

16.	Jatropha biodiesel	Pentanol, di- tertiary butyl peroxide (DTBP), and 2-ethylhexyl nitrate (EHN)	Decrease	Decrease	Decrease	Increase	Increase	Decrease	[32]
17.	Water- biodiesel emulsion fuel (Jatropha)	Carbon nanotubes and diethyl ether	-	Increase	-	-	-	Decrease	[33]
18.	Callophyllum inophyllum methyl ester (CIME) biodiesel	Zinc Oxide (ZnO) and ETHANOX	-	Increase	-	Increase	Increase	Decrease	[34]

### 2.5 Selection of aluminum for corrosion test

Aluminum has emerged as preferred material in the automotive industry particularly for application that required weight reduction and corrosion resistance. This literature review focuses on the selection of aluminum for an experiment that aimed to replicate the fuel tank of a vehicle. Generally, vehicle fuel tank is a critical component that needs high corrosion resistance and weight efficiency.

The primary objectives for selecting aluminum in automotive application is its good compatibility with the corrosive biodiesel and its ability to reduce the overall weight of the vehicle. This reduction is crucial as manufacturers strive to improve fuel efficiency. According to previous study, the use of aluminum can lead to weight savings which directly impacts the vehicle performance and fuel efficiency.

Aluminum corrosion resistance is another factor that makes it suitable for automotive application. For instance, the aluminum alloys 5000 series are known for their excellent resistance to marine and industrial atmospheres. This characteristics makes them ideal for the inner panel of vehicle including fuel tanks. However, the 6000 series aluminum alloys are typically used for the outer panels of vehicles due to their higher strength and machine formability. More examples that demonstrate the difference in the application of aluminum alloys 5000 series and 6000 series provided in the Table 2.2.

HANV		
Outer Panels	HANV6016-T4	
	HANV6016-T4P (super-lite)	
Inner Panels	HANV5051 A-O	
	HANV5182-O (inner-lite)	
	HANV6016A-T4	
Structural sheet	HANV5754-O	
	HANV5454-O	
	HANV6016A-T4	

Table 2.2 Product range-automotive aluminium sheet from HANV (Miller et al.,2000)

The automotive industry has faced challenges in reducing vehicle weight while simultaneously enhancing the safety features. The application of safety systems such as antilock systems braking (ABS) and airbags has led to the increasing weight of vehicle. Previous effort to mitigate this increased weight through design improvements and power train efficiency enhancement have proven insufficient. Therefore, the shift towards lightweight materials like aluminum 5000 series has been identified as a proper solution. The transition of materials used for the industry varied to specific regions as every region required different materials to optimise the performance due to environmental factors. Another example of different choice of materials in different regions is provided in Table 2.3.

Moreover, weight reduction achieved through the use of aluminum has a direct correlation with increased fuel efficiency. The decrease in vehicle weight reduces the overall energy required for propulsion. Thus, improving the fuel efficiency and at the same time reducing the emissions. The selection of aluminum 5000 series for the replication of vehicle fuel tanks in corrosion test is well-justified. Its excellent corrosion resistance and weight reduction capabilities makes in ideal choice for enhancing fuel efficiency (Miller et al., 2000;Mittelbach & Schober, 2003)

		Europe	North America
	Outer Panels		
	Alloy	6016-T4	6111-T4
	Surface texture	EDT or EBT	MF
	Pre-treatment	Pickling + Zr/Ti conversion	None
	Lubrication	Oil or dry-lubricant	Oil
WIA 2	Inner Panels		
	Alloy	5051/5182/6181A	6111/2008/5182
1119	Surface texture	MF or EDT	MF
9	Pre-treatment	Pickling + Zr/Ti conversion	None
5	Lubrication	oil or dry-lubricant	Oil
	Structure/sheet		<i>J.</i> , <i>J</i>
N	VERAlloy TEK	NIKAL 6xxx-T4	LAK 5754-O
	Surface texture	EDT	MF
	Pre-treatment	Pickling + Zr/Ti conversion	conversion
	Lubrication	Oil or dry-lubricant	oil
	Structure/extrusion		
	Alloy	6xxx	6xxx

Table 2.3 Alloy choice: Europe vs North America (Miller et al., 2000)

#### 2.6 Antioxidant additive concentration

The selection of an effective antioxidant concentration is critical in enhancing the stability and performance of biodiesel fuels. Pyrogallol (PY) has been identified as a potent antioxidant because it demonstrates superior performance in various studies compared to other additives. This literature review focuses on determining the optimal concentration of pyrogallol for corrosion resistance in a replicated vehicle fuel tank.

In previous study by Mittelbach and Schober (2003) and Muhammad Nur Islamuddin Bin Amran (2023), they investigated the effects of different additives on biodiesel stability using the transesterification process. The additives tested included pyrogallol (PY), prophyl gallate (PG), butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA) and tertbuthylhydroquinone (TBHQ) at a concentration of 1000ppm. Their results indicated that pyrogallol consistently emerged as on of the most effective antioxidants in enhancing the oxidation stability of biodiesel. Following these these initial findings, they further tested the most effective additives at concentrations of 1000 ppm, 250 ppm and 500 ppm. Across these screenings, pyrogallol maintained its superior performance. Thus, reinforcing its potential as a leading antioxidant for biodiesel applications.

Another relevant study by Muhammad Nur Islamuddin Bin Amran (2023), he explored the effect of temperature on the tribological properties with pyrogallol as an additive. In this study, biodiesel (B10) was mixed with 600 ppm of pyrogallol to evaluate its performance under varying temperature conditions. The findings revealed that pyrogallol significantly improved the tribological properties of biodiesel. The result also suggested that higher concentrations might further enhance these benefits.

He concluded by recommending an increase in the concentration of pyrogallol to optimize the additive properties. The recommendation made indicate a positive correlation between the concentration of pyrogallol and its effectiveness. The collective findings from these studies suggest that the concentration of pyrogallol plays a crucial role in determining its efficacy as an antioxidant additive. Higher concentrations appear to enhance the beneficial properties of pyrogallol including improved oxidation stability and tribological performance. Consequently, it is essential to determine the optimal concentration of pyrogallol for specific applications such as corrosion resistance in automotive fuel tanks.



Figure 2.4 Influence of antioxidant concentration on the oxidation stability of rapeseed oil methyl esters; (a) undistilled; (b) distilled. PY, Pyrogallol; PG, n-prophyl gallate (Mittelbach &Schober, 2003)



## 2.7 Metal Surface Examination

The metal surface examination is one of the analysis phases in the static immersion test of aluminum coupons in pure biodiesel B20 and biodiesel B20 mixed with pyrogallol. This examination aims to observe and characterize the types of corrosion that occur on the aluminum coupon surface including corrosion pits, surface roughness and other uniform or localized corrosion features,

In an experiment for the analysis of AISI 1020 steel corrosion conducted by Rios et al. (2014), optical microscopy is the primary method used for this surface examination with magnification levels at maximum 200x. This range enable detailed inspection of finer features such as the depth and distribution of corrosion pits and the characterization of microstructural changes due to corrosion. The 200x magnification level demonstrated its effectiveness in providing detailed and accurate observations of corrosion features on metal surfaces.

In addition to the magnification, the image acquisition parameters are crucial for ensuring high-quality documentation and analysis. According to Rios et al. (2014), images shoud be captured at a resolution of 1280x1024 pixels with one frame taken every two seconds. This high-resolution imaging technique allows for comprehensive documentation of the corrosion progression. It provides detailed visual records that facilitate precise analysis of changes in surface morphology over time. Such detailed documentation is essential for understanding the dynamics of corrosion processes and the effectiveness of corrosion inhibitors like pyrogallol.

The expected results from the metal surface examination include various forms of corrosion. Corrosion pits are typically small, localized holes that form on the metal surface where the protective oxide layer has been disrupted. These pits can vary in size and depth which indicate the severity and progression of localized corrosion. Surface roughness changes can signal uniform corrosion where the entire surface is uniformly attacked. This leads to a matte or rough appearance. Additionally, localized corrosion features such as intergranular corrosion or crevice may manifest as cracks along grain boundaries or deep localized corrosion within confined spaces or joints.

Understanding these corrosion features is vital for assessing the performance of aluminum 5052 in biodiesel environments. The findings from these examinations will provide the insights into the suitability of aluminum alloys for automotive applications and the effectiveness of pyrogallol as a corrosion inhibitor in biodiesel fuels. By employing standardized magnification levels and high-resolution imaging techniques, this study ensures a thorough and accurate analysis of corrosion phenomena on aluminum surfaces.



Figure 2.5 Schematic diagram of simultaneous optical and electrochemical measurements (Rios et al., 2014)

## 2.8 Summary

The literature review provides crucial insights into the selection of materials, methodology and analytical approaches for conducting corrosion tests aimed at replicating vehicle fuel environments. By the contribution of comprehensive examination of existing research, significant findings have emerged that inform the design and execution of corrosion experiments. This comprehensive review covered various aspects including the suitability of different materials for simulating automotive components, the effectiveness of specific methodologies for corrosion testing and the most effective analytical approaches for evaluating corrosion behaviour and performance. By combining these key findings, a solid foundation can be established for experimental approach and ensuring that the chosen materials, methods and analytical techniques align with the objectives of the study and yield meaningful insights into corrosion phenomena within vehicle fuel environments.

The experiment utilization of B20 biodiesel as a test fuel is chosen to reflect a transitional step towards the broader adoption of B100 which represents 100% biodiesel content. This transitional approach aligns with evolving global and national trends. It is also the initiatives aimed at reducing reliance on fossil fuels and promoting the use of renewable energy sources in transportation. As highlighted by Kugelmeier et al. (2021), countries like the United States and Indonesia have already embraced B20 biodiesel blends in their fuel supply chains. This demonstrates the feasibility and benefits of incorporating higher biodiesel concentrations. In contrast, the current usage is predominantly limited to B10 biodiesel blends. Therefore, conducting experiments with B20 biodiesel is crucial for Malaysia gradual transition towards higher biodiesel blends such as B100.

By studying B20 biodiesel performance and compatibility in automotive applications, valuable insights can be gathered into its effectiveness, feasibility and potential challenges. This experimental approach not only supports Malaysia's commitment to sustainability and energy independence but also lays the groundwork for future developments in renewable fuel technologies and policies. Eventually, the research findings can inform decision-makers and stakeholders in shaping policies, regulations and strategies for optimizing biodiesel utilization and accelerating the transition towards more environmentally sustainable transportation fuels.

The selection of aluminum 5052 to replicate the fuel tank which is a critical component of the car's inner panel is based on a thorough evaluation process guided by recommendations from research and consultations with experts. As suggested by Miller (2000), aluminum from the 5000 series is estimated suitable for applications requiring corrosion resistance and structural integrity. This makes it an ideal candidate for simulating the fuel tank environment. Following this recommendation, a survey was conducted among aluminum suppliers to identify the available options in the local market. Three types of aluminum , namely 6061, 5052 and 5083 were identified as potential candidates. Afterwards, consultations were held with the supervisor and material experts from the university to determine the most suitable aluminum alloy for replicating the fuel tank. After carefully considering factors such as corrosion resistance, formability, and mechanical properties, aluminum 5052 emerged as the preferred choice. It is known for its excellent corrosion resistance and formability. Aluminum 5052 offers the ideal combination of properties required for simulating the fuel tank environment effectively.

This decision highlights a systematic and collaborative approach in material selection. This is to ensure that the chosen alloy meets the specific requirements of the experiment and aligns with established recommendations and expertise in the field. By opting for aluminum 5052, the fuel tank conditions can be confidently replicate and corrosion tests can be done with a material that accurately represents real-world applications. All the consideration in material selection contribute to the validity and relevance of the experimental findings.

The concentration of PY for this study is 800 ppm. In previous studies of Mittelbach & Schober (2003) and Amran (2023), it was observed that the usage of PY was between 100 ppm – 1000 ppm. Specifically, 100 ppm, 250 ppm, 500 ppm, 600 ppm have been tested. Thus, the optimum concentration range for PY is determined by referring to those studies. The optimum concentration is in the range of 600 ppm – 1000 ppm. Since 600 ppm and 1000 ppm have been tested in previous studies, 800 ppm is by far the most suitable choice which also optimized the relevance of this study.

#### RSITI TEKNIKAL MALAYSIA MELAK/

The experiment will employ a static immersion test following to the ASTM G31 standard. This method involves immersing the metal specimens in biodiesel to evaluate their corrosion resistance over a specific period. In order to validate the result, two analysis conducted which are the weight loss measurement and metal surface examination. These analysis provide quantitative and qualitative data on the extend of corrosion and the integrity of the metal surface after exposure to B20 biodiesel. This structured approach ensures a comprehensive evaluation of the corrosion resistance of aluminum 5052 in a biodiesel environment.

## **CHAPTER 3**

### METHODOLOGY

## 3.1 Introduction

This chapter outlines the methodology used to conduct the FYP project following the flowchart provided in Figure 3.1. The approach ensures the achievement of the established aims to produce exceptional results. Detailed procedures including the preparation of the metal coupon sample, blending process, weight measurement, immersion of the sample into the biodiesel and the experimental procedure with the equipment utilized. A homogenizer employed to blend the biodiesel with additive, PY. After the homogenization, the metal coupon sample immersed in both the B20 biodiesel blend and pure B20 biodiesel without the additive. In order to evaluate the corrosion rate of the metal coupon post-experiment, a specific formula using the weight loss of the metal coupon as per ASTM G31 is applied. Then, a 3D Non-Contact Profilometer used for the metal surface examination.



Figure 3.1 Methodology flowchart

#### **3.2** Material Preparation

Prior to conducting the experiment, it was necessary to carry out the sample preparation. In this experiment, the sample used was aluminum coupon 5052 which later on immersed in two different types of biodiesel solution, pure biodiesel B20 and biodiesel B20 blended with PY.

### 3.2.1 Aluminum coupon 5052

From the previous literature review, Kugelmeier (2021) prepared the aluminum coupon by referring to the ASTM G31 standard. In ASTM G31, the aluminum coupon size preferred is 25mm x 20mm x 3mm and a 2mm diameter hole. After the determination of the required size, the aluminum 5052 was obtained from the local aluminum supplier through a purchase order. 25 pieces of aluminum 5052 specimen were supplied according to the provided dimension except the 25mm width and 2mm diameter hole. Machining process was done to fulfil the required dimension by the ASTM G31.



Figure 3.2 ASTM G31 Aluminum Coupon (25mm x 20mm x 3mm)



Figure 3.3 Actual Aluminum Coupon (25mm x 25mm x 3mm)

The machining of the aluminum coupon involved three processes which were grinding, drilling and deburring. Firstly, the aluminum coupon was grinded into the required size following the ASTM G31. Then, a 2mm diameter hole was drilled to suspend aluminum coupon with Teflon afterwards followed by deburring to remove residual burrs for a better surface cleanliness.



Figure 3.4 Aluminum coupon before and after drilling

## 3.2.2 Biodiesel

In this experiment, there were two types of solution which were pure biodiesel B20 and biodiesel B20 blend with 800 ppm of PY. Initially, the amount of biodiesel B20 for each beaker was determined by referring to the ASTM G31 standard. The determination of volume for biodiesel B20 required for each beaker with the metal specimen was done by considering the preferred ratio of solution volume to specimen area which is  $0.2mL/mm^2$ . The ratio suggested that for every  $mm^2$ , 0.2mL of biodiesel B20 is necessary. The total surface area of the specimen is  $1282.57mm^2$ , so the surface area is multiplied by the solution volume-to-area ratio in order to determine the required volume of biodiesel for each beaker. By calculation, it was calculated that the volume of biodiesel needed for each beaker is 256.51mL of biodiesel B20. It is the minimum required volume to adequately immersed the entire surface area of the specimen. The calculation ensures that the specimen is fully immersed in the biodiesel solution and allow for a more accurate experimentation as intended. Consequently, the total volume of biodiesel B20 required for all 8 beakers was calculated and the resulting calculation yielded a total volume of 2052.08 mL.



Figure 3.5 Biodiesel B20

#### 3.2.3 Blending

Since there were two types of solution which were pure biodiesel B20 and biodiesel B20 mixed with 800 ppm of PY, a blending process was needed for the biodiesel B20 with PY solution. Before blending, 800 ppm concentration was converted into mass (g) using the formula as below:

Mass of  $PY = (Concentration of PY \times Volume) \times 10^{-\#}$  (1)

In order to reduce the time taken for the blending process, two 500mL beakers were used to conduct the blending twice instead of four 300mL beakers.



Figure 3.6 Pyrogallol (PY)

The blending involved two mixtures since it was done twice. The first mixture contains 513.02 mL of biodiesel B20 with 0.4005g of PY. The first mixture was poured into beakers 2 and 4. Meanwhile, the second mixture contains 513.02mL of biodiesel B20 with 0.4030g of PY. The second mixture was poured into beakers 1 and 3 for the static immersion test.







Figure 3.8 Mixture of biodiesel B20 and Pyrogallol (PY)



Figure 3.9 Beaker with 500mL biodiesel B20 placement at Ultrasound Homogenizer

After the homogenizing process run for 10 minutes, 5 minutes rest was necessary to observe whether it has been fully homogenized or not before proceesing for another 10 minutes of homogenizing. If PY has been fully homogenized, there was no need to proceed for another 10 minutes of homogenizing. At the same time, the condition of the beaker also needs to be monitored from time to time. If the beaker was too hot, then it needed to be submerged into a basin of water to cool down the temperature of the beaker. If it was not effective to cool down the beaker with water, a basin of ice was another preferable option. This action was significant to ensure that the beaker did not break during the homogenizing at elevated temperatures. During the homogenizing process of the biodiesel B20 with PY using Ultrasound Homogenizer, the first and second mixture had the same duration for the whole process which was 20 minutes of homogenizing and 5 minutes of rest. Both mixtures were cooled down in a basin of water during the 5 minutes rest due to the increasing temperature of the beakers.

Table 3.1 Time taken for homogenizing process

Mixture	Time Taken
1 (Beaker 2 & 4)	20 min + 5 min rest
2 (Beaker 1 & 3)	20 min + 5 min rest

After the homogenizing has been completed, the mixture of biodiesel B20 and PY from the 500mL beaker was transferred into two 300mL beakers with their respective label. The same step was repeated for another 500mL biodiesel B20 with 0.4g of PY.



Figure 3.10 B20 + PY; Beaker 1 (30days), Beaker 2 (50 days), Beaker 3 (100 days), Beaker 4 (150 days)



Figure 3.11 Pure biodiesel B20; Beaker 1 (30 days), Beaker 2 (50 days), Beaker 3 (100 days), Beaker 4 (150 days)

## 3.2.4 Weight Measurement

Weight measurement procedure was conducted for the aluminum coupon prior to the static immersion test. This is due to the weight loss being one of the parameters for the result of the experiment. Initial weight of the aluminum coupon before immersion test and the final weight of the aluminum coupon after the immersion test will be used in the formula to calculate corrosion rate.

Aluminum coupon was cleaned with acetone before measuring the weight. Acetone removed contaminants on the surface of the aluminum coupon such as oils, grease and dust from handling of the previous process. Acetone is an effective solvent that can ensure the surface cleanliness of the aluminum coupon. Besides, it prevented any extra weight from these substances from affecting the measurement. Accuracy in weight measurement is crucial for precision in this experiment whereas contaminants can lead to inaccuracies. Additionally, cleaning the aluminum coupon standardised the preparation procedure by ensuring consistency and reproducibility in experimental processes. In this case, consistency was made possible by reducing variability in the results afterwards. Furthermore, a clean surface is vital for the following treatment such as corrosion testing because contaminants can interfere with chemical reactions or physical interactions. Thus, cleaning the aluminum coupon with acetone ensures the accuracy, consistency and reliability in weight measurement and the corrosion test.



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### **3.3** Static Immersion Test

for storage.

Static immersion test was done by immersing the cleaned aluminum coupon into the pure biodiesel B20 and biodiesel B20 mixed with PY. There were four different durations of immersion for the corrosion test which were 30 days, 50 days, 100 days and 150 days. The aluminum coupon was suspended using Teflon through the 2 mm diameter hole. The aluminum coupon was suspended in vertical position so that the entire surface area of the aluminum coupon was fully exposed with the pure biodiesel B20 and biodiesel B20 mixed with PY. Then, the Teflon was attached to the beaker using tape and labelling was made to ease the analysis procedure afterwards. Finally, each of the beakers was wrapped using wrapping paper to provide a vacuum condition for the experiment at room temperature. All the beakers were being kept inside a box



Figure 3.12 Teflon used to suspend the aluminum coupon inside the beaker



Figure 3.14 Biodiesel B20+PY; Beaker 1 (30 days), Beaker 2 (50 days), Beaker 3 (100 days) and Beaker 4 (150 days)



Figure 3.15 All beakers wrapped with wrapping paper

## 3.4 Weight Loss Measurement

The analysis of this experiment will be conducted once the duration of the immersion reached the due date. There were four due dates for each of the immersion which were 30 days, 50 days, 100 days and 150 days. The first analysis of this experiment was to determine the corrosion rate of the aluminum coupon. This was done by weighing the aluminum coupon once it reached its due date of immersion. The difference between the initial and final weight after immersion test was indicated the weight loss, W. The weight loss was then applied into the formula to determine the corrosion rate of the aluminum the formula of the corrosion rate following the ASTM G31 standard as follows.

Corrosion rate (mpy) = 
$$\frac{W x 534}{D x T x A}$$
 (2)

Where corrosion rate "mpy" satnds for mils (0.001 inch) per year, W is the weight loss (mg), D is the density  $F_{\underline{cm^3}}^{\underline{g}}$ G, A is the exposed surface area (*inch*<sup>2</sup>) and T is the exposure time (h).

## 3.5 Metal Surface Examination

Upon reaching the due date for the immersion test, metal surface examination of the aluminum coupon was conducted using 3D Non-Contact Profilometer. This analysis aimed to assess the extent and nature of the corrosion that has occurred on the aluminum surface. The expected results include observations of corrosion pits, surface roughness and any uniform or localised corrosion features.

Magnification levels utilised during the metal surface examination were between 50x to 100x. This range was chosen as the parameter based on the previous study by Rios (2014) where magnification levels from 100x and above was used for a more detailed inspection. These range of magnifications enable the observer to focus on specific areas of interest identified at lower magnifications which allow for the precise measurement of pit depth, characterization of fine surface features and identification of microstructural changes. In this experiment, examination of microstructure was not the primary objectives. So, metal surface examination for this experiment at lower magnification was sufficient to fulfill its functionality to support the main objectives of the experiment.

A lower magnification at 50x, the 3D Non-Contact Profilometer was able to capture a broad overview of the surface. This allowed for the identification of largescale features such as widespread surface roughness, major pitting sites and extensive areas of uniform corrosion. These initial observations were essential for understanding the general extent of corrosion and identifying areas that require more detailed examination.

In addition to visual inspection, image acquisition was performed at a resolution of 1280x1024 pixels that captured one frame every 2 seconds. This high-resolution imaging allowed for a detailed and continuous observation of the surface changes over time. It also provide a comprehensive view of the corrosion progression and the effectiveness of any protective measures in place.



## **CHAPTER 4**

## **RESULTS AND DISCUSSION**

## 4.1 Biodiesel Condition

After the biodiesels were used throughout the static immersion test at 30 days, 50 days, 100 days and 150 days, there was a significant change in the colour of the biodiesel solution which homogenised with PY compared to the pure biodiesel solution. The physical condition of the two types of biodiesel can be observed in Figure



Figure 4.1Solution (a) Pure B20 before immersion (b) B20+PY before immersion (c) Pure B20 after immersion (d) B20+PY after immersion

Based on Figure 4.1 (a) and (b), the initial physical condition of the two types of solution can be observed in terms of their colour. Both of the solutions had a similar yellowish colour prior to the immersion test. Meanwhile, B20+PY most probably has a greater density in terms of its properties due to the presence of PY compared to pure B20. In Figure 4.1 (c) and (d), the two solutions had a significant difference in terms of their colours after the immersion test. The pure B20 still maintained its yellowish colour of solution as per the initial condition before immersion. However, the B20+PY had turned into a darker and chalky condition compared to its initial condition.

The significant change in the colour of the solution is obviously influenced by the presence of the additive PY in the biodiesel solution. This is because PY is known to have a low solubility and stability in biodiesel (Varatharajan et al., 2018). Low oil solubility of the PY is also dependent on the long-chain hydrocarbons of fatty acid methyl ester (FAME) presence in the biodiesel that inhibited the PY from dispersing in biodiesel solution (Hery Sutanto et al., 2019).

There are two factors that could affect the intensity of the colour changes of the solution. The two factors are FAME, which is present in the biodiesel itself supported by oxygen and the degradation of the PY. FAME particularly in unsaturated condition is the cause of oxidation in biodiesel as it offers a high level of reactivity with oxygen (Gaurav Dwivedi et al., 2014).

The solution setup of the static immersion test according to ASTM G31 has allowed the presence of trapped oxygen in the biodiesel solution. The trapped oxygen became one of the mediums that supported the occurrence of oxidation in biodiesel. This analytical assumption is strongly supported by a solubility test performed in the previous research (Subroto et al., 2013). The solubility test has shown that the PY saturation point is the lowest as its absorbance value is the first to not provide any increasing value after 1000ppm concentration (Hery Sutanto et al., 2019). The solubility test result is also verified and aligned with the fact that among all of the commonly used phenolic antioxidants, PY has the lowest solubility in biodiesel (Subroto et al., 2013).

Even though a homogenizing process of the biodiesel and PY was carried out during the sample preparation, it did not solve the low solubility issue of the PY in the long term as this experiment period was 150 days in total. The homogenizing process at high temperatures did help to dissolve the PY in the biodiesel but only for that particular time. As the static immersion test started, the temperature started to drop at room temperature to comply with the parameter set up for this immersion test. The relevance of the room temperature condition was to mimic the condition of a fuel tank in a vehicle when the engine is not operating.

The temperature drop has caused the PY to return to its naturally low oil solubility after a period of time and started to concentrate at the bottom part of the beaker used rather than remaining uniformly dispersed. During this particular time, the PY reaction with the unsaturated FAME of the biodiesel mixed with oxygen has caused it to degrade over time. Additionally, the degradation intensity became worse since the condition is localized where it is high in concentration. This has caused the reaction between the unsaturated FAME of biodiesel and degradation of PY to release dark-coloured and chalky by products which then slowly diffuse into the solution over time. Thus, producing a noticeable colour changes particularly within the B20+PY solution as observed in Table 4.1. The explanation of the colour changes indicate that the initial homogenizing process in the early stage of the of the immersion test only temporarily mask the issue of low oil solubility in PY. However, it does not have the capability to mitigate the issue of low oil solubility to the fundamental. Despite the

issue with the colour changes, it does not reflect on the effectiveness of the PY as the best antioxidant additive for biodiesel. This is due to PY being very sensitive to biodiesel stability as compared to other antioxidant. The stability of the PY is the highest as compared to other natural and synthetic antioxidant (Gaurav Dwivedi et al., 2014).

## 4.2 Corrosion Mechanism

In this part of the analysis, discussion on how the corrosion takes place over the aluminum metal coupon throughout the immersion test is the focal point.

#### 4.2.1 Weight loss measurement

Prior to the determination of corrosion rate that occurred on the aluminum coupon, mass loss of the aluminum coupon was initially identified in order to calculate the corrosion rate using formula provided by ASTM G31. The mass loss of the aluminum coupon is tabulated in Table 4.1.

Aluminum coupon		Mass (g)			
		Initial	Final	W, mass loss (initial – final)	
1		4.0676	4.0695	-0.0019	
2	Beaker 1	3.8166	3.8157	+0.0009	
3	(30 days)	4.1962	4.1921	+0.0041	
Average	B20+PY	4.0268	4.0258	+0.0010	
4	Beaker 2	4.1964	4.1962	+0.0002	
5	(50 days) B20+PY	3.9885	3.9883	+0.0002	

Table 4.1 Weight data result of aluminum coupon

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6		4.1226	4.1223	+0.0003
Average		4.1025	4.1023	+0.0002
7		4.1518	4.1552	-0.0034
8	Beaker 3	4.0687	4.0711	-0.0024
9	(100 days)	4.1387	4.1407	-0.0020
Average	B20+PY	4.1197	4.1223	-0.0026
10		4.1215	4.1217	-0.0002
11	Beaker 4	4.0861	4.0865	-0.0004
12	(150 days)	4.0591	4.0598	-0.0007
Average	B20+PY	4.0889	4.0893	-0.0004
13		4.0798	4.0763	+0.0035
14	Beaker 1	4.2502	4.2497	+0.0005
15	(30 days)	4.0138	4.0134	+0.0004
Average	B20	4.1146	4.1131	+0.0015
16	00 00 00	4.1087	4.1082	-0.0895
17 🗆	Beaker 2	4.0939	4.0933	+0.0006
18	(50 days)	3.9647	3.9640	+0.0007
Average	B20	4.0559		
10		4.0558	4.0552	+0.0006
19	Beaker 3	4.0930	4.0552	-0.0004
20	Beaker 3 (100 days)	4.0930 4.0964	4.0552 4.0934 4.0982	-0.0006 -0.0004 -0.0018
19       20       21	Beaker 3 (100 days) B20	4.0930 4.0964 3.9284	4.0552 4.0934 4.0982 3.9310	-0.0006 -0.0004 -0.0018 -0.0026
20 21 Average	Beaker 3 (100 days) B20	4.0930 4.0964 3.9284 4.0393	4.0552 4.0934 4.0982 3.9310 4.0409	-0.0006 -0.0004 -0.0018 -0.0026 -0.0016
19 20 21 Average 22	Beaker 3 (100 days) B20 Beaker 4	4.0930 4.0964 3.9284 4.0393 4.0729	4.0552 4.0934 4.0982 3.9310 4.0409 4.0730	+0.0006 -0.0004 -0.0018 -0.0026 -0.0016 -0.0001
19           20           21           Average           22           23	Beaker 3 (100 days) B20 Beaker 4 (150 days)	4.0930 4.0964 3.9284 4.0393 4.0729 3.9789	4.0552 4.0934 4.0982 3.9310 4.0409 4.0730 3.9790	+0.0006 -0.0004 -0.0018 -0.0026 -0.0016 -0.0001 -0.0004
19           20           21           Average           22           23           24	Beaker 3 (100 days) B20 Beaker 4 (150 days) B20	4.0930 4.0964 3.9284 4.0393 4.0729 3.9789 4.1173	4.0552 4.0934 4.0982 3.9310 4.0409 4.0730 3.9790 4.1161	+0.0006 -0.0004 -0.0018 -0.0026 -0.0016 -0.0001 -0.0004 +0.0012
19           20           21           Average           22           23           24           Average	Beaker 3 (100 days) B20 Beaker 4 (150 days) B20	4.0930 4.0964 3.9284 4.0393 4.0729 3.9789 4.1173 4.0564	4.0552 4.0934 4.0982 3.9310 4.0409 4.0730 3.9790 4.1161 4.0560	+0.0006 -0.0004 -0.0018 -0.0026 -0.0016 -0.0001 -0.0004 +0.0012 +0.0004



Figure 4.2 Graph of mass loss (g) vs Immersion time (days)

Figure 4.2 shows that the aluminum coupon pure B20 solution continuously has mass loss for up to 150 days. Meanwhile, aluminum coupon in B20+PY only had a significant mass loss within the 30-50 days duration. This timeframe of mass loss indicates that PY has stabilised the oxidation rate occurring in B20+PY which inhibited the mass loss for aluminum coupon once its oxidation was stabilised after 50 days duration. This is agreed by Hery Sutanto et al. (2019) in previous research where degradation occurred more intensively towards biodiesel without additive compared to biodiesel mixed with additive.

#### 4.2.2 Corrosion Rate

By indicating the mass loss earlier in the analysis, the corrosion rate can be calculated using the formula provided by the ASTM G31 for static immersion test. The formula was mentioned in Chapter 3 as follows.

Corrosion rate (mpy),

$$\frac{W x 534}{D x T x A} \tag{2}$$

Where corrosion rate "mpy" stands for mils (0.001 inch) per year, W is the weight loss (mg), D is the density  $(g/cm^3)$ , A is the exposed surface area (*inch*<sup>2</sup>) and T is the exposure time (h).

KNI.	Solution	W, mass loss (g)	D, density $F \frac{g}{cm^3}G$ ,	A, exposed surface area (inch <sup>2</sup> )	T, exposure time (hour)
TI TE	B20 (30 days)	+0.0015			720
	B20 + PY (30 days)	+0.0010			
5	B20 (50 days)	+0.0006			1200
	B20 + PY (50 days)	+0.0002	2.68	1.988	9
U	B20 (100 days)	-0.0016	MALAYS	IA MELAI	CA 2400
	B20 + PY (100 days)	-0.0026			
	B20 (150 days)	+0.0004			3600
	B20 + PY (150 days)	-0.0004			

Table 4.2 Corrosion rate data



Figure 4.3 Graph of corrosion rate (mpy) vs Immersion time (days)

As observed in Figure 4.3, corrosion rate in B20+PY (0.1392mpy) is lower than the B20 (0.2088mpy) at 30 days immersion period. This is due to the speed up formation of protective layer (aluminum oxide) known as passivation process which involved the oxidation and reduction of biodiesel, PY and enhanced by the trapped oxygen in the beaker. The oxidation process known to be dependent to the level of reactivity with oxygen. It is agreed by Gaurav Dwivedi et al. (2014) in previous research involving the mechanism of oxidation. Even though the data shows that the corrosion rate in B20+PY is higher than the pure B20 within the 30 days, it is not necessarily a negative impact caused by the PY in the long term. From Figure 4.3, a significant drop of the corrosion rate in B20+PY is observed at 50 days immersion time compared to the pure B20. This is because the ability of PY to stabilised the oxidation and reduction rate during the 30 days period has allowed the aluminum coupon to have a greater proportion of protective layer quicker than the aluminum in pure B20. Y.H. Yoo et al. (2011) also mentioned that corrosion product from the reaction of biodiesel and aluminum could be transform into white-coloured  $Al(OH)_3$  in the presence of  $H_20$ which is from the trapped moisture in the beaker for this immersion test. Generation of the protective layer was done by formation of crystalline aluminum oxyhydroxite (Boehmite, AlOOH) at the beginning of the process (Norouzi et al., 2012). The strong protective layer which knownly as  $Al_2O_3$  oxide film formed on the surface of the aluminum metal gives the metal a greater protection since it cannot be easily broken by the acid presents in the blends (Norouzi et al., 2012). Thus, fitting the role in protecting the aluminum metal against corrosive compounds.

The corrosion rate data of pure B20 at 50 days shows the evidence that corrosion activity continuously occurred at a consistent rate compared to B20+PY. It was due to the normal rate of protective layer formation which was slower than the enhanced protective layer formation provided by the aid of PY. Although the type of aluminum metal used was not specified, he corrosion rate data were comparable to the previous study by A.Shehzad et al. (2023) because of the blend composition similarity. The relatively small difference between the pure B20 and B20+PY corrosion rate at 50 days in this immersion test compared to the previous study strengthens the opinion on the superior tailored chemical properties in terms of corrosion resistance provided by the PY in B20+PY. The protective layer managed to stabilise the corrosion activity that attacked the specimen throughout the immersion over time until the corrosion attack is considered negligible. This condition where mass loss is less than  $1x10^{-4}g$  was observed to a similar immersion test conducted by Kugelmeier et al. (2021)

#### 4.3 Surface Characterization

Aluminum coupon specimens were taken for surface examination using 3D Non-Contact Profilometer and SEM-EDX after the immersion test. Corrosion pit and uniform corrosion are the most commonly identified on the surface of the specimen post-immersion test under 3D Non-Contact Profilometer.



Figure 4.4 3D Non-Contact Profilometer imaging of aluminum coupon specimen (a) B20+PY (b) B20

Corrosion pit observed on the specimen surface was resulted from the corrosion attack that took place in the early stage of the immersion test. It is the visual result of the oxidation and reduction process that necessarily happened prior to the formation the protective layer on the surface of the specimen. The corrosion pit formation identified on the surface of the aluminum specimen was due to oxide protective layer has a positive surface charge in its natural state and has the ability to absorb negative charge ion (Mccafferty, 2003).

In the context of immersion test, highly reactive hydroperoxide ( $ROO^{-}$ ) as the byproduct of biodiesel oxidation is attracted to the positive surface charge which eventually oxidized the aluminum metal underneath the oxide film. Gaurav Dwivedi et al. (2014) found that the primary oxidation product of double bonds are unstable allylic hydroperoxides which are unstable and easily form a variety of secondary oxidation products. The attraction of the negative ion hydroperoxide has caused the formation of void or defects on the surface of the specimen. Presence of the negative ion also allow for further degradation of the oxide layer that exposed the aluminum surface to localized corrosion and makes it highly vulnerable. The negatively charged ions have a small radius and capable to penetrate through the oxide film which cause the pitting corrosion at the metal substrate or oxide film interface (Mccafferty, 2003). The hydroperoxides are not within the detection range of the SEM-EDX as it is an organic ions from the result of biodiesel oxidation.





Figure 4.5 SEM-EDX results of aluminum coupon (a) B20+PY (b) B20 (c) AL5052 sample

SEM-EDX results in Figure 4.5 shows the presence of Al, Mg and oxygen was identified. However, no compound was found to have formed on the surface of the specimen. These results are aligned with the ones reported by Kugelmeier et al. (2021). In particular, Al wt% in B20+PY (34.3wt%) is significantly lower compared to the B20 (59.8 wt%). The enhanced protective layer through the aid of PY keep the bare aluminum unexposed from the environment and simultaneously masked the aluminum detection. Natural formation of an oxide film on aluminum is estimated to be a few nanometers thick as mentioned by Joao Victor De Sousa Araujo et al. (2023). This results to the relatively lower Al wt% of the B20+PY compare to the B20 where the bare aluminum is more exposed. Validation on this result is made through the determination of consistency in Al wt% trend for multiple specimens throughout the 150 days immersion time.

Oxygen wt% found on the surface of the aluminum coupon specimen showed differences in B20+PY (10.2 – 12.7 wt%), B20 (9.0 – 9.8 wt%) and sample (7.0 wt%). Enhanced formation of a protective oxide layer in B20+PY allows the oxide layer to remain intact with the aluminum surface due to a reduction in corrosion activity. This is significant in providing the higher amount of oxygen wt% found on the aluminum coupon specimen. Meanwhile, the oxygen wt% in B20 does not exceed the minimum oxygen wt% found in B20+PY. Oxidation that leads to the formation of a protective layer for the aluminum coupon specimen did not occurred as much as in the B20+PY. However, the lower oxygen wt% in the sample evidently shows that oxidation does occurred in the B20 at lower rate than the B20+PY. This supports the agreement by Joao Victor De Sousa Araujo et al. (2023) that aluminum is a passive metal that capable to protect itself through a naturally formed protective layer.

#### **CHAPTER 5**

#### **CONCLUSION AND RECOMENDATIONS**

#### 5.1 Conclusion

This study evaluated the effects of additive on the corrosion behaviour of aluminum metal upon exposure to palm biodiesel at room temperature. Based on the experimental results, the following conclusions can be drawn:

Pure B20 and B20+PY solutions both managed to demonstrate corrosion activity in terms of mass loss and pitting formation on the aluminum coupon specimens even though not significantly affect the specimens.

The immersion test after 50 days did not provide any mass loss detection on the aluminum coupon specimens and corrosion rates in B20+PY. Meanwhile, B20 showed a continuous mass loss and corrosion rate up to 150 days. A more apparent pitting found on the surface of the aluminum specimens compared to the condition prior to the 50 days period.

PY managed to improve the corrosion behaviour of the aluminum coupon specimen by reducing the oxidation rate in the biodiesel blends. It also maintained the oxide protective layer throughout the immersion test and minimizing aggressive localized corrosion attack through uniform corrosion stabilization.

PY has proven to have supported in maintaining the stability of the passivation layer on the aluminum surface by reducing the oxidative stress that destabilized the passivation layer in the biodiesel blend without PY.

Further studies can be carried out to determine the corrosion attack on the whole specimen surface by conducting methodologies that can implement both overall

metal surface examination and cross-section analysis which are evidently an effective method to analyse corrosion attack at higher precision especially on aluminum that has proven its high compatibility with biodiesel blends based on previous research.

### 5.2 **Recommendations**

Cross section analysis and material composition by SEM-XRD (Xray Diffraction) are recommended for further studies of the corrosion activity. This is because cross section analysis can provide a deeper insight on the corrosion attack depth and profile. Cross section analysis allow for a better understanding of the corrosion internally which is not visible in surface analysis. SEM-XRD recommended due to its ability to cover larger surface area compared to SEM-EDX where the scope of the analysis is limited to a pinpoint location.

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA Tajuk Projek : Effects Of Additive Or Exposure To Palm Bio	UNIVERSITI TEKNIKAL MALAYSIA MELAKA FAKULTI KEJUTERAAN MEKANIKAL BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA II n The Corrosion Behaviour Of Aluminum Metal Upon diesel At Room Temperature				
Sesi Pengajian . Semester 1 2024/2025					
Saya Muhammad Hafiq Bin Abdul Ha ini disimpan di Perpustakaan Laman	ikim. mengaku membenarkan laporan Projek Sarjana Muda Hikmah dengan syarat-syarat kegunaan seperti berikut:				
<ol> <li>Laporan adalah hakmilik Universiti Teknikal Malaysia Melaka.</li> <li>Perpustakaan dibenarkan membuat salinan untuk tujuan pengajian sahaja.</li> <li>Perpustakaan dibenarkan membuat salinan laporan ini sebagai bahan pertukaran antara institusi pengajian tinggi.</li> <li>Sila tandakan (✓):</li> </ol>					
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