CORROSION EFFECT OF CEIBA PENTANDRA BLENDS WITH COMMERCIAL DIESEL ON ALUMINIUM ALLOYS

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RESEARCH REPORT SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF ENGINEERING

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CORROSION EFFECT OF CEIBA PENTANDRA BLENDS WITH COMMERCIAL DIESEL ON ALUMINIUM ALLOYS

ABSTRACT

Corrosion effect of aluminium alloys in ceiba pentandra biodiesel at various blends with B5 commercial diesel (10%,20%,30%) were investigated. Static immersion test was carried out at ambient temperature for each grade of AA3003, AA5052 and AA6061 for 1200 hours. The compatibility of ceiba pentandra blends with B5 commercial diesel were analysed for its density and viscosity. Microstructure changes of aluminium alloys were observed by both SEM and EDS analysis. The results of corrosion rate showed that the corrosion reaction surges with the increase in concentration of ceiba pentandra biodiesel. The surface morphology reveals the presence of oxide layer as the initial stage of passivation on both B5 commercial diesel and CP10. However, the corrosiveness looks vulnerable for aluminium alloys immersed in both CP20 and CP30 due to the biodiesel concentration whereby both carbon and oxygen element are presence on the EDS analysis.

Keywords: Biodiesel, Ceiba Pentandra, Blending, Corrosion behaviour

KESAN KAKISAN CAMPURAN CEIBA PENTANDRA DENGAN DIESEL KOMERSIAL TERHADAP ALOI ALUMINIUM

ABSTRAK

Kesan kakisan aloi aluminium dalam campuran biodiesel ceiba pentandra dengan pelbagai diesel komersial B5 pada konsentrasi (10%, 20%, 30%) diselidiki. Ujian rendaman statik dilakukan pada suhu ambien bagi setiap gred AA3003, AA5052 dan AA6061 selama 1200 jam. Keserasian ceiba pentandra bercampur dengan diesel komersial B5 dianalisis untuk ketumpatan dan kelikatannya. Perubahan mikrostruktur aloi aluminium dianalisis dengan bantuan SEM dan EDS. Hasil kadar karat menunjukkan bahawa tindak balas kakisan bertambah dengan peningkatan konsentrasi ceiba pentandra biodiesel. Morfologi permukaan mendedahkan kehadiran lapisan oksida sebagai tahap awal pasifasi pada kedua-dua larutan diesel komersial B5 dan CP10. Bagaimanapun, kakisan kelihatan mudah terdedah pada aloi aluminium yang direndam dalam CP20 dan CP30 masing-masing kerana kehadiran unsur karbon dan oksigen.

Kata kunci: Biodiesel, Ceiba Pentandra, Larutan diesel, Kakisan

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LIST OF SYMBOLS AND ABBREVIATIONS

ASTM	:	American Society for Testing and Materials
EN	:	European standard
B5	:	biodiesel with 95% diesel fuel
CP	:	Ceiba pentandra
CCPO	:	crude Ceiba pentandra oil
CPBD		Ceiba pentandra biodiesel
CP10	:	Ceiba pentandra biodiesel blending 10%
CP20	:	Ceiba pentandra biodiesel blending 20%
CP30	:	Ceiba pentandra biodiesel blending 30%
B5	:	commercial diesel
AA	:	Aluminium alloy
mpy	:	mils per year
SEM	:	scanning electron microscopy
EDS	:	energy dispersive spectroscopy

CHAPTER 1: INTRODUCTION

1.1 Background

The demand of energy is growing rapidly due to tremendous progress of economy and people's mobility. Energy consumption of countries like China and India alone have surpasses half of the global needs (Joshi, Pandey, Rana, & Rawat, 2017). The global population are at the peak right now and this also correlated to the energy consumption (Shuba & Kifle, 2018). Transportation service have increased whereby more cars are on the road now. The dependency for fossil fuel have created huge environmental concern where climate change and greenhouse effects are haunting us back.

Renewable energies are seen as alternative option for energy sources. The fossil fuel is leading the energy source for most of industry's needs. The global price drop in the crude oil further hampered the efforts to improve the utilisation of renewable energy. Paris agreement was vital in reviving renewable energy agenda as countries came along with the commitment to prioritise the climate change and emission control. Besides, European Union have also supporting the efforts to significantly increase renewable energy productions (Galanopoulos, Yan, Li, & Liu, 2017).

Biofuel are considered as potential source of energy which are able to replace or reduce the consumption of fossil fuel in short term (Chisti, 2007). This is largely due to the compatible of biodiesel whereby there is no engine modifications are required. Most of the countries have initiated the efforts to incorporated biofuel in the commercial diesel as a mandatory action. For instance, commercial diesel sold in fuel station comprises from 5% to 20 % blending of biofuel depending on the country.

The introduction of biofuel in automotive industry leads to compatibility issues with metals. Aluminium and its alloys being a primary material selection for automotive, transportation and pipeline industry have exhibits excellent properties. It also exhibits excellent corrosion behaviour compared to other metals. However, it is still susceptible to corrosion particularly pitting corrosion. This occurs when the oxidation layer of aluminium alloys breaks apart.

1.2 Problem Statement

Numerous research has been conducted on the performance of various blends of biofuel on diesel engine but research on corrosion behaviour are still limited. It was reported that the biofuel possesses greater corrosiveness compared to commercial diesel due to presence of free water and free fatty acids (Haseeb, Masjuki, Ann, & Fazal, 2010). The continues pressure by EU nations to impose ban for palm oil pushed us in a dilemma whereby market plunge may be occurred due to this sanction. Hence diversifying our biodiesel feedstock option are seen as an ideal approach.

The study of *Ceiba pentandra* for its compatibly as biodiesel feedstock are investigated thoroughly. Despite various research on the characteristic of *Ceiba pentandra* as a biodiesel, the corrosion behaviour of it on aluminium alloy remains unanswered. Hence, this study will contribute to significant findings.

1.3 Research Aim and Objective

The research aim is to study the corrosion behaviour of different grades aluminium alloys under different composition of CP blend with the commercial diesel. The objectives of this study are: -

- To study the corrosion characteristic of different biodiesel feedstock (*Ceiba pentandra*) in favour to palm oil biodiesel.
- To investigate the appropriate biodiesel blend which has the lowest corrosion rate of aluminium alloys with respect to different grades.
- To characterise the morphology of microstructural and metallurgical defects on the aluminium alloys after the static immersion test.

1.4 Scope of Studies

The scope of this research is to investigate and understand corrosion behaviour over different blends of biodiesel (Ceiba pentandra) on aluminium alloys. This investigation also covers the weight reduction observation of different grades of aluminium alloys (AA3003, AA5052 and AA6061) due to corrosion after the static immersion. Besides, oxidation layer formation and pitting corrosion are evaluated on the course of this evaluation. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) are used to conduct an extensive study on the corroded surface of aluminium alloys.

CHAPTER 2: LITERATURE REVIEW

This chapter gives a comprehensive study on the aluminium, corrosion effect of biodiesel, current studies related to the topic and findings including methodological contribution towards aluminium alloys characteristic on biodiesel. This literature review is also vital in justifying the novelty of the research topic.

2.1 Aluminium

2.1.1 Background

Aluminium and its alloys have wide-ranging application in automotive, aerospace and transportation industry (Dursun & Soutis, 2014). It is the most widely used metal only after to steel and it is categorised into heat treatable, non-heat treatable and casting alloys (Olakanmi, Cochrane, & Dalgarno, 2015) . The heat treatment process capable to load relatively high level of stress to produce exceptional performance material (Dursun & Soutis, 2014). Aluminium are also preferred material selection due to its good corrosion resistance, machinability and unique combination of properties.

The properties of aluminium are subject to complex interface of morphology and chemical composition which occurred during the heat treatment process, solidification and for wrought alloys through deformation processing (Sverdlin, 2003). Physical properties of aluminium vary respective to the alloyed material whereas the pure aluminium (Al) itself is a silvery white metal with atomic number of 13 and atomic weight of 26.98 g/mol. Aluminium also exhibits very good electric conductivity. High tension power lines are now dependent to aluminium alloy as an ideal material (Mondolfo, 1976).

Despite being light material, aluminium has excellent mechanical attributes. Heat treatment, quenching and alloying can improve the tensile strength up to 700 MPa from only 90 MPa on pure aluminium (Embury, 1996). Aluminium comprises of one-third of Young Modulus compared to steel at 70000 MPa. Thus, the characteristic of aluminium under static and dynamic stress are more elastic than steel. This elasticity offers the ability for aluminium to return back its shape and size. A summary of physical properties of aluminium are shown in Table 2.1.

Table 2.1: Properties of Aluminium (Embury, 1996)

Property	Value
Atomic Number	13
Atomic Weight (g/mol)	26.98152
Valency	3
Crystal Lattice	Face centred cubic
Boiling Point (°C)	2480
Melting Point (°C)	660.2
Electrical Resistivity at 20°C (μ.cm)	2.69
Thermal Conductivity (0-100°C) (cal/cms. °C)	0.57
Mean Specific Heat (0-100°C) (cal/g.°C)	0.219
Co-Efficient of Linear Expansion (0-100°C) (x10-6/°C)	23.5
Density (g/cm3) 2.	2.6898
Electrical Resistivity at 20°C (μΩcm)	2.69
Modulus of Elasticity (GPa)	70
Poisson Ratio	0.34

Excellent corrosion characteristic of aluminium is obtained from its chemical properties. Aluminium forms an oxidation layer when it contacts with oxygen. This layer of aluminium oxide creates a barrier which helps to protect from corrosion. Crystal structure arrangement of face center cubic (F.C.C) makes aluminium with enhanced properties of ductility.

2.1.2 Microstructure and Surface Morphology

Corrosion behaviour of aluminium is heavily dependent on the microstructure and surface morphology. Different microstructural may be developed during the solidification of aluminium castings (Wislei Riuper Osório, Freire, & Garcia, 2005). Grain structure of aluminium are subject to take many forms during the solidification process which will be influencing the corrosion properties (Wislei Riuper Osório et al., 2005; C. A. Santos, Quaresma, & Garcia, 2001). Numerous studies have been conducted on the correlation of grain morphology and its influence towards the mechanical properties (W. R. Osório, Santos, Quaresma, & Garcia, 2003; Siqueira, Cheung, & Garcia, 2002) . The relationship of grain size is well reported by Quaresma, Santos, & Garcia (2000) whereby the grain size is inversely proportional to the strength of aluminium but with much improving corrosion resistance.

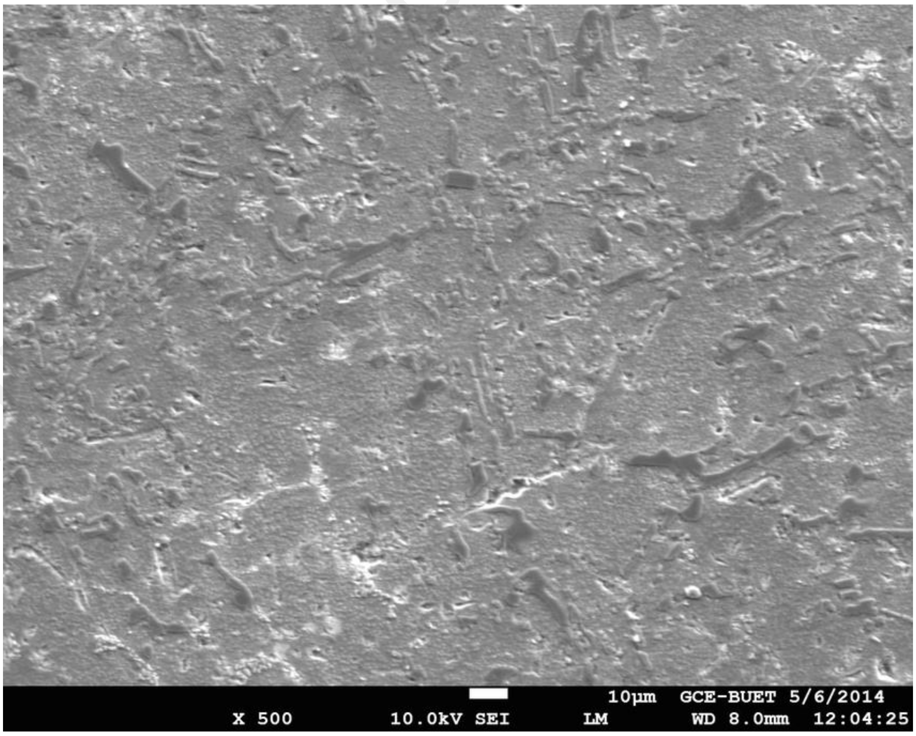


Figure 1: SEM image of aluminium alloy manufactured as engine piston (Kaiser, Qadir, & Dutta, 2015)

SEM image of aluminium alloy is shown in Figure 1. Kaiser et al. (2015) also reported that the plate and needle like microstructure are mainly Al_3Ni intermetallic. There are Ni particles inside Al-Si matrix and it is distributed evenly (Kaiser et al., 2015). Typical corrosion topographic are shown in Figure 2 (a,b) reveals the occurrence of trenching of matrix next to cathodic second phase particle corrosion ring and intergranular attack are visible in Figure 2(c) (Donatus et al., 2017). Pitting are well observed in the Figure 2(d) image whereby degradation occurs under the surface (Donatus et al., 2017).

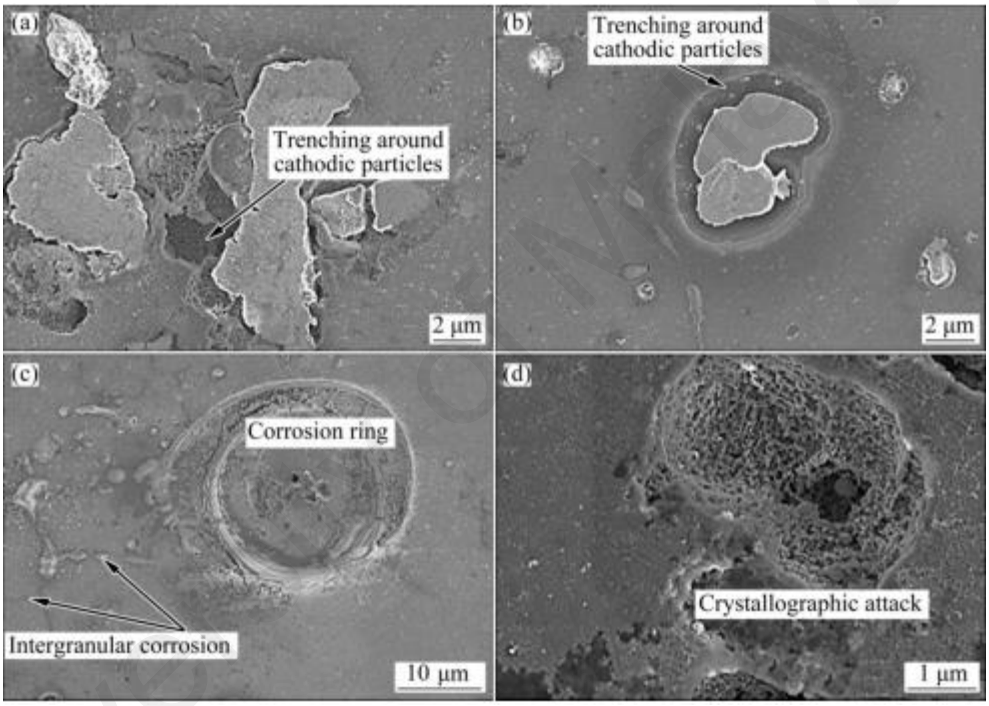


Figure 2: SEM of as-polarised AA2024-T3 (Donatus et al., 2017)

2.1.3 Aluminium Alloys and Its Properties

Aluminium alloys are commonly divided into two classes of wrought and cast composition (J.R. Davis, 1998). Both of these classes differs from chemical composition temper designation (Mathers, 2002). Most wrought composition of aluminium alloys are dependent to the work hardening for the mechanical properties which usually involved annealing process. The casting alloys are principally not age treatable and used only in solution or precipitation modified conditions (J.R. Davis, 1998).

The 4-digit system of aluminium alloy classification is used and it was introduced by American Aluminium Association. Series of AA1XXX which are unalloyed and pure composition followed by AA2XXX series and up to AA9XXX series are within the grades of aluminium alloys. The first digit of the series represents the major alloying elements with second digit identifies the impurity limit of allotting element (Shuba & Kifle, 2018). The properties for each aluminium alloy series are as following (J.R. Davis, 1998):

- AA1XXX series alloys

Pure aluminium of 99% or higher. It exhibits excellent corrosion resistant, electric and thermal conductivity but has poor mechanical properties. If the alloying content increases, the corrosion properties will have minor degradation.

- AA2XXX series alloys

The primary alloying element is cooper and magnesium as the secondary element(Davis, 1999; Pierre R. Roberge, 2012) . This series of alloys requires solution heat treatment to enhance its mechanical properties but do not have a good corrosion characteristic compared to other series of aluminium alloys whereby it is subject to intergranular corrosion under certain conditions.

- AA3XXX series alloys

The primary alloying element is manganese. This series alloys usually cannot be heat treated but the strength is approximately at 20% higher than AA1XXX series. The addition of manganese and silicon can improve the mechanical properties and volume fraction dispersoid (Li, Zhang, & Chen, 2016) . It is reported that it also has relatively good corrosion resistance properties. The mostly used grade is AA3003 where it is used in many applications due to its workability.

- A4XXX series alloys

Silicon acts as the principle alloying element. Despite being a non-heat treatable series of aluminium alloy, there present of silicon, controlled amount of magnesium and cooper makes it to respond in the improving strength through heat treatment. This series of alloy also exhibits good corrosion resistance.

- A5XXX series alloys

Magnesium is the major alloying element for this series of aluminium alloys. It has high corrosion resistance properties due to precipitate of Al_8Mg_5 particle when magnesium is present in solid solution or partially.

- AA6XXX series alloys

Both silicon and magnesium element take proportion required for the formation of magnesium silicide (Mg_2Si) precipitates. It makes this series alloys heat treatable. Besides, it has good formability, weldability and corrosion resistance. The intergranular corrosion is minimal due to the proportion of precipitates but if the silicon level beyond the needed to form Mg_2Si, the corrosion will increase (Zahavi, 1982).

- AA7XXX series alloys

Zinc is the major alloying element ranging from 1% to 8% of the composition. This series of aluminium alloy exhibits high strength properties but compromised in corrosion characteristics. Cooper, magnesium, chromium and zirconium may be present as different composition within the same grade. Whenever cooper is involved as the alloying element, the corrosion resistance become vulnerable. It is reported that Al-Zn-Mg-Cu alloys has superior strength but compromised on the corrosion. Heat treatment without specific measures of solution heat treating temperature causing eutectic melting (Vander Voort, 2006).

- AA8XXX series alloys

For this series of aluminium alloys, the alloying element may include tin and lithium. The dispersion strengthened Al-Fe-Ce alloys or from Al-Fe-V-Si alloys is used to improvise the temperature performances. These grade of alloys is developed for bearing application whereby the lubricity is needed and it is achieved with the present of Tin element.

2.1.4 Application of Aluminium Alloys

Alloying is a process of combining one or more elements to improve its properties. Besides, alloying is to make sure application based grades are developed. Each application or product requires the certain properties which need to be fulfilled and vital. Hence, different grade of aluminium alloys available can fit the product's or application's requirements. Alloying element determine the properties of each grade of alloys. Selected aluminium alloy from each series are filtered out with respect to its applications as in Table 2.2.

Table 2.2: Selected Application of Aluminium Alloys (Davis, 2001)

Series	Grade	Applications
1XXX	1100	Consist of pure aluminium which are highly resistance to corrosion and chemical attack. Widely used for chemical processing, fan bland, nameplates and etc.
2XXX	2024	Generally used for application which required high strength. The corrosion properties are only considered as fair and this grade alloys widely used for truck wheel, fasteners, machine gears and etc.
3XXX	3003	General purpose alloy used as sheet metal works, stamping, fuel tanks, container, chemical equipment and etc. It also has good formability and weldability.
5XXX	5052	Despite having strength higher than 3003, this grade alloys exhibits excellent formability. It also has good corrosion resistance and often used in application such as pressure vessel, automotive parts, marine application, kitchen equipment, train cars and etc.
6XXX	6061	This grade alloys exhibits formability and weldability and also excellent corrosion resistance. It is used automotive parts, aircraft equipment, machine parts and etc.
7XXX	7075	Mainly used in application which requires highest strength such as aircraft. This grade also exhibits expectational corrosion resistance properties which are close to pure aluminium.

Series 3XXX, 5XXX and 6XXX are generally used for automotive industry and also in fuel tanks constructions. It will be ideal if further literature narrowed on only this series. Research and studies on these grades are essential since both of the applications are related for fuel handling and may lead to new findings.

Typical composition of different grades of aluminium alloy with 3, 5, and 7 series are tabulated in Table 2.3. It will be useful to identify major alloying element and the roles of other elements on the properties.

Table 2.3: Properties of Selected Aluminium Grades

Grades	Mn	Zn	Si	Cu	Al	Fe	Mg	Cr	Ti
3003	1.2	0.1	0.6	0.1	Bal	0.7	0	0	0
5052	0.1	0.1	0.25	0.1	Bal	0.4	2.8	0.25	0.15
6061	0.15	0.04	0.71	0.19	Bal	0.35	0.96	0.08	0.03

2.2 Corrosion on Aluminium and Its Alloys

Aluminium in general has excellent corrosion properties and poses outstanding resistance in neutral atmosphere due to the present of alumina Al_2O_3 film which forms on the surface as a protective barrier. Oxidation reaction is blocked by the passive oxide film as the electron will not go through. The alumina (Al_2O_3) film can be describe by the Equation 1.

$2Al + H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^-$ …………………………………….. **Equation 1**

Metal will exhibit three reactions when it is dissolved in aqueous solution. Those reactions are either corrosion, immunity or passivation (Coleman, Scott, & McEnaney, 1994) . The corrosion behaviour of aluminium alloys significantly dependent to the acidity or alkalinity environment. Numerous factors are involved in corrosion behaviour of aluminium and its alloys. Those are primarily on the metal composition if it involves its alloys. Besides, environment plays a vital role in the corrosion resistance as well. Temperature and the joining methods will also influence the corrosion behaviour.

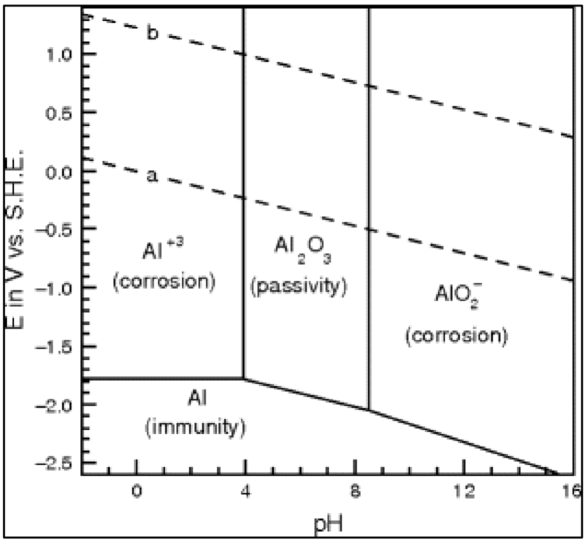


Figure 3: Pourbaix Diagram of Aluminium (McCafferty, 2010)

Pourbaix diagram is useful in distinguishing the stable phases of an aqueous electrochemical system. Figure 3 represents the thermodynamic stability of the passive oxide film on aluminium alloy. The dotted line Figure 3(a,b) signifies the water stability region. The area below Figure 3(a) line represents the condition whereby the water is unstable and decomposes to hydrogen H_2 with alkalisation of OH^-. Water is unstable and decomposes to oxygen O_2 and acidification of H^+ for the condition above the dotted line Figure 3(b).

Corrosion can be observed from Figure 3 at both acid and alkaline condition whereas in the neutral solution, aluminium surface is observed as passive. This is due to the hydroxide in insoluble state. The governing equation for the corrosion of aluminium alloys are described in Equation 2,3 and 4. Equation 2 represent the anodic reaction condition.

$Al \rightarrow Al^{3+} + 3e-$……………………………………..……………….……**Equation 2**

$O_2 + 2H_2O + 4e- \rightarrow 4OH$…………………………………………………..…**Equation 3**

$2H + 2e- \rightarrow H_2$…………………………………………………………**Equation 4**

If there is a presence of oxygen, the cathodic reaction will occur and the reduction of oxygen to hydrogen can be observed. It is observed that the single phase aluminium alloys typically have superior corrosion resistance compared to aluminium alloys which contains second phase intermetallic particles (Burleigh, 2003). This is due to the fast reduction of oxygen at the cathode when it involves impurities such as cooper and iron precipitates (Burleigh, 2003). Equation 4 derives the cathodic reaction without the present of oxygen.

2.2.1 Influence of Composition and Morphology on Corrosion

Aluminium possess excellent corrosion resistance and its alloys have the similar characteristic despite the alloying element. Nevertheless, it is still vulnerable to corrosion particularly the localised (Baer et al., 2000; Boag, Hughes, Glenn, Muster, & McCulloch, 2011; Boag et al., 2010). Localise corrosions which often lead to failures including pitting corrosion, stress corrosion cracking and crevice corrosion are difficulty to detect (Xiao & Chaudhuri, 2011). The microstructure and environment changes dynamically leading to complexity for any earlier detection (Xiao & Chaudhuri, 2011).

For 3XXX series, the major alloying element of Manganese has a relatively low solubility in aluminium but when it remains in solid solution, corrosion resistance are vastly improved (Polmear, 2005). The approximate addition of 1% of manganese forms the good corrosion characteristic with sensible strength of non-heat treatable alloys (Polmear, 2005). It is reported by Nisancioglu (1990) that the manganese present have effectively reduced pitting exposure predominantly when adjusting Fe intermetallic particles. Manganese also have exhibits the influence of reducing the concentration of Fe. Thus, this results in reduced corrosion level with the similar level of Al matrix despite having Al_6MnFe composition (Koroleva, Thompson, Hollrigl, & Bloeck, 1999).

Manganese present will only helpful for the corrosion resistance properties if the within the range of solubility limit (Liu & Cheng, 2010). Cathodic reaction will increase when the manganese quantity in aluminium is above 1.25% (Liu & Cheng, 2010). It was reported that the increase in both manganese and silicon during the intermetallic phases results in a decreased rate of localised reduction activity (Nis¸ancioğlu, 1990, 1992). It was also reported by Nisancioglu (1983) that the corrosion properties of 3XXX series aluminium alloys may be improved by using small size and low volume of intermetallic phases or with an increase content of manganese in the matrix.

The 5XXX and 6XXX series aluminium alloy's magnesium element which constitute respectively at 2.8% and 0.9%. The alloy density is decrease with the presence of magnesium whereby it stabilises the Guinier–Preston zone and also has high solubility in aluminium. It was reported by Muller & Galvele (1977) that there is no significant effect on the pitting corrosion when magnesium is in solid solution. Besides, cathodic reaction is reduced with the present of magnesium in solid solution thus improving the alloys corrosion properties but these alloys are prone to localised corrosion if the amount of magnesium is found to be in excess (John S. Vetrano, 1998). Searles, Gouma, & Buchheit (2001) reported that this is largely due to the present of precipitation of either Al_8Mg_2 or Al_3Mg_2 which will form along the grain boundaries when the excess of magnesium or continuous exposure at raised temperature (Baer et al., 2000).

The 6XXX series aluminium alloys share almost equal proportion of both magnesium and silicon composition. Precipitation of Mg_2Si particles occurs due to the present of both magnesium and silicon. These particles are catalyst to the improvement of the alloys strength but the drawback are localised corrosion may occur (Eckermann, Suter, Uggowitzer, Afseth, & Schmutz, 2008).

The excess of silicon on from grades of aluminium alloys within the 6XXX series increases the cathodic reaction as reported by Eckermann et al. (2008) whereby the silicon will be present along the grain boundary. Hence, this may cause stress corrosion cracking and intergranular corrosion (Guillaumin & Mankowski, 2000; Larsen, Walmsley, Lunder, Mathiesen, & Nisancioglu, 2008; Zeng et al., 2011)

2.2.2 Pitting Corrosion

Pitting corrosion is localised corrosion whereby small holes are visible in the material and it is often occurs for the metals with the passive protective layer (Moore, Sykes, & Grant, 2008). Microstructure plays vital role in the pitting corrosion formation since it is mainly caused by the grain structure between opposite are which are in contact with corrosive atmosphere. Intermetallic element has been reported by Szklarska-Smialowska (1999) as the pitting corrosion initiation factor. Several factors are key in pitting corrosion such as alloy compositions, solution concentrations and temperature.

Pitting corrosion usually develops in the presence of chloride ions whereby the ions are absorbed on the oxide layer (Toit, 2011). The drop in pH occurs due to anodic dissolution in cations, Al^{3+}. This is followed by hydrolysis. The positive chloride ions then are drawn in to the pit area and balance charge occurs as shown in Figure 4.

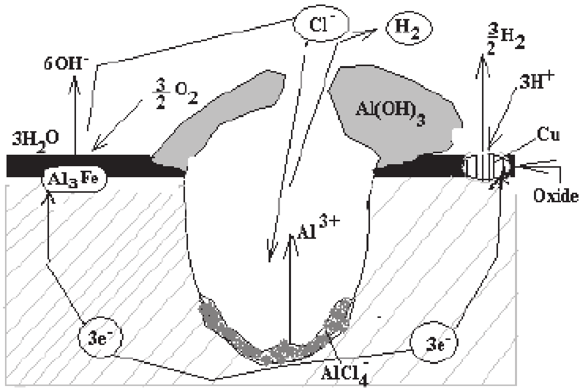


Figure 4: Pitting corrosion mechanism (Toit, 2011)

Re-passivation happens over dissolution since both low pH and high chloride concentration condition.

2.3 Biodiesel

2.3.1 Background

Biodiesel are commonly defined as mono-alkyl esters of animal fats or vegetable oils. Transesterification process is involved in the derivation of long chain fatty acids like lauric, palmitic, stearic and oleic which are obtained from renewable biolipids ("Biodiesel," 2008; Demirbas, 2007). Viscosity of the oil is lowered down by this transesterification process to make sure the standard is achieved. The chemical reaction for this process is derived as Equation 5.

Triglycerides + Monohydric alcohol → Glycerine + Mono Alkyl esters…**Equation 5**

The conversion into biodiesel is basically the same whereby oil or fat will react with methanol or ethanol with the presence of sodium hydroxide or potassium hydroxide as the catalyst to form the product, biodiesel and glycerine. The chemical name for biodiesel are either methyl esters or ethyl esters ("Biodiesel," 2008). Generally, the chemical and physical properties of either methyl ester or ethyl esters are almost the same and it exhibits the same heat content. Ethyl ester's viscosity have been observed slightly higher that methyl ester. This effects the results of engine power and torque favouring methyl ester slightly ("Biodiesel," 2008).

Table 2.4: Technical Properties of Biodiesel ("Biodiesel," 2008)

Common name	Biodiesel (bio-diesel)
Common chemical name	Fatty acid (m)ethyl ester
Chemical formula range	$C_{14} - C_{24}$ methyl ester or $C_{15-25}H_{28-48}O_2$
Kinematic viscosity range (mm2 /s, at 313 K)	3.3−5.2

Table 2.4: continued,

Density range (kg/m3, at 288 K)	860−894
Boiling point range (K)	>475
Flash point range (K)	430−455
Distillation range (K)	470−600
Vapor pressure (mm Hg, at 295 K)	<5
Solubility in water	Insoluble in water
Physical appearance	Light to dark yellow, clear liquid
Odour	Light musty/soapy odour
Biodegradability	More biodegradable than petroleum diesel
Reactivity	Stable, but avoid strong oxidizing agents

Table 2.4 describes the properties of biodiesel. The biodiesel derived from natural sources such as rapeseed, soya , palm and etc. meets the specification of ASTM D 6751 (Mofijur et al., 2012). Marchetti, Miguel, & Errazu (2007) reported that numerous studies have been conducted using different type of oil as raw material, various alcohol types and catalysts. The production of biodiesel from EU are mainly from rapeseed whereas from US are from soybean. Figure 5 represents the flow chart biodiesel processing. The base-catalysed production commonly follows steps where it involves mixing of both alcohol and catalyst, transesterification process, separation, biodiesel washing, alcohol elimination, glycerine neutralisation and quality control ("Current Technologies in Biodiesel Production," 2008).

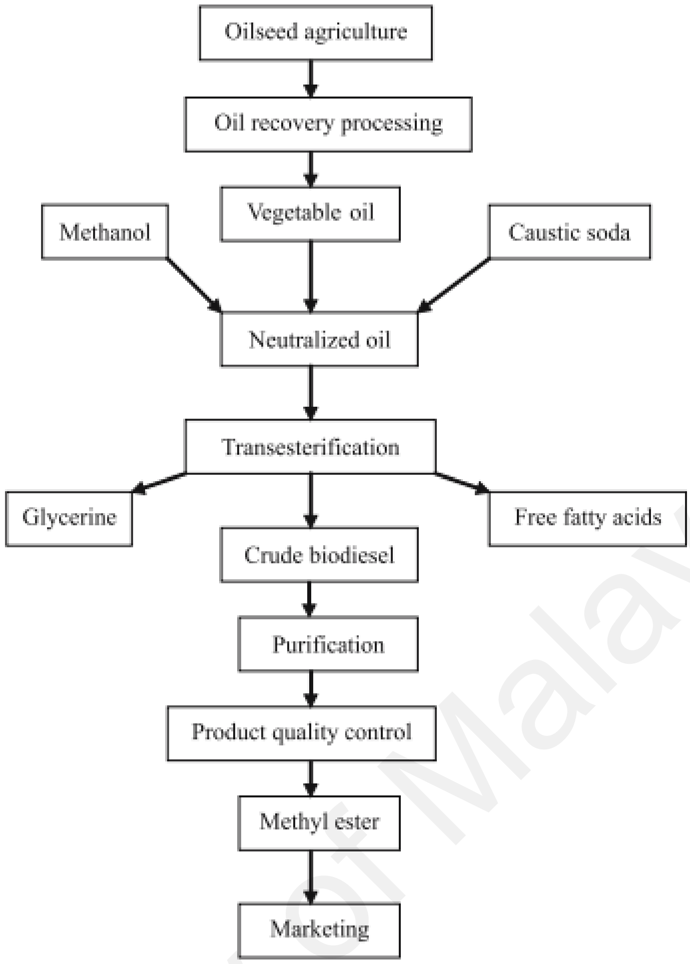


Figure 5: Process of base catalysed biodiesel ("Current Technologies in Biodiesel Production," 2008)

2.3.2 Potential of Exploring Biodiesel Options

The demand for biodiesel production was growing at remarkable rate of 23% per annum between 2005 and 2015 (Naylor & Higgins, 2017). The global price for crude oil was increasing significantly leading to potential expanding the biodiesel production. However, this does not sustain long as the crude of price drop drastically by early of 2015. Biodiesel being alternative fuel was expected to take a dip in the production but fortunately the decline in the production does not happen.

This is mainly due to affirmative action taken by respective biodiesel production countries whereby incentives and policy made available to prioritise exploration of this renewable energy.

Governments around the globe have also prioritised and planned to increase the mandate on biodiesel composition. As current mandate in Malaysia, it was reported by Naylor & Higgins (2017) that biodiesel blending is at the B5 and expending to B7 and planned to introduce B10 by year 2017. Most of the countries are inline to increase their respective biodiesel blending requirement mandate at up to B30 similar Indonesia government's intention for an increase by 2020. This creates an opportunity to explore more types of biofuel as the raw material.

As reported earlier, EU are reviewing plan to scrap off the usage of palm oil biofuel. If this action is materialised in future, countries which are heavily dependent for biofuel production from palm feed will severely impacted. Malaysia and Indonesia's export of biodiesel will be reduced drastically due to this embargo. Hence, alternative biofuel material source must be explored. This is to avoid any economic impact and to create more diversified options.

Besides, expanding the research on different raw material for biodiesel will also reduce the country's dependency on the imported oil. Local feedstock can be used for biodiesel production. As of current situation, the biodiesel feedstock comprises of soybean oil at 30%, rapeseed oil at 25%, palm oil (18%) , recycled vegetable oil at 10%, animal fats at 6% and other unknown source of vegetable oil at 11% (USDA).

2.3.3 Issues and Limitation of Biodiesel as Renewable Energy

Despite being one of the potential alternative to fossil fuel, biodiesel also poses issues and limitation that need to be addressed. Various issues were reported by on the challenges of utilising biodiesel in Malaysia (Anuar & Abdullah, 2016). Those issues are diverging from environmental concern, social and sustainability. Currently the production cost for biodiesel is higher compared to petrol diesel and this become one of the issues that slowing down the usage ("Biodiesel," 2008). It was reported by Bala (2005) that the maximum production of vegetable oil or animal fats is not sufficient to meet the demand.

Apart from that, technical issues arise with the consumption of biodiesel. Performance, emission, wear, and corrosion are some of the issues within the technical limitation of biodiesel (Hasan & Rahman, 2017). These are the obstacles why biodiesel still unable to use as the complete replacement of petrol-diesel. On this literature, the significance of biodiesel's corrosion behaviour are included in extensive manner.

2.4 Corrosion of Metals in Biodiesel

Corrosion of metals in biodiesel are one of the factors which hinders the commercialisation of these products. It remains a concern predominantly for automotive industry whereby it is reported by Fazal, Haseeb, & Masjuk (2013) that biodiesel exhibits more corrosiveness than diesel. It was reported by Sankara (2011) that the corrosion of metals in biodiesel are dependent to the raw material of the fuel. Adding to that, the water accumulation increases the bacterial growth thus increasing the corrosiveness (Sankara Papavinasam, 2011). The study of corrosion of metals in biodiesel are important to address the growing concern on its effectiveness but there are very limited paper on this issue (Fazal, Haseeb, & Masjuki, 2010).

Corrosion performance of different metals such as cast iron, carbon steel, cooper, brass and etc. was studies in several papers (Fazal et al., 2013). It was report by Geller, Adams, Goodrum, & Pendergrass (2008) that cooper are more prone to corrosion in fat based biodiesel compared to ferrous alloys. The corrosiveness of cooper is in great extend compared to other metals (Fazal et al., 2013). Metals such as aluminium, copper, zinc brass and bronze are suggested for biodiesel non-compatible material (*Biodiesel Handling and Guidelines*, 2006).

Storage and handling of biodiesel must be delicate as the it has poor oxidation stability. (N. A. Santos et al., 2012). The sensitivity of biodiesel's storage can leads into significant degradation of its properties (Dennis Y. C. Leung, Wu, & Leung, 2010). It is due to the insoluble gums, aldehydes and water contamination which are found on the degraded biodiesel (D. Y. C. Leung, Koo, & Guo, 2006). These external materials are the main reason for the corrosion as reported by Deyab (2016). Oxidation happens rapidly once water and air contaminant are found. The oxidation by-product can lead into formation of deposit and corrosion which eventually stall the engine or system (Knothe, 2002). The tank storing condition have also influences corrosion and microbiologically influenced corrosion (Sankara Papavinasam, 2011). This mainly happens due to the presence of moist air on the storage and also water contamination (Sankara Papavinasam, 2011).

Quality of the extracted oil are still considered a major problem. It was observed that oil with high concentration of poly-saturated fatty acids are more prone in decrease of oxidation stability (Borsato, Maia, Dall'Antonia, Silva, & Pereira, 2012). Study by Rodrigo (2012) reveals that each of the oil source material exhibits its own corrosion resistance characteristic. Soybean oil extract shows in decreased oxidation stability but Jatropha has exhibit slight better (Rodrigo A. A. Munoz, 2012). This is due the composition of 40.3 % linoleic acid and 37% linolenic acid which lower the amount of unsaturated material which lead into oxidation stability (Amit et al., 2010). Besides, Moringa oleifera oil extract also exhibits excellent oxidation resistance with the potential shelf life of 4 to 5 years (Rashid, Anwar, Moser, & Knothe, 2008). Based from the overall studies, the corrosion behaviour of biodiesel is heavily dependent on the type of raw material.

Cooper are subjected to severe degradation due to corrosion. This is reported in various papers and even reported that cooper is not recommended to be used in biodiesel (Fazal, Suhaila, Haseeb, Rubaiee, & Al-Zahrani, 2018). Microstructure variations on the metal surface are evidence of corrosion presence whereby layer of black material are found on the cooper strip (Hu, Xu, Hu, Pan, & Jiang, 2012). These changes are observed by Hu (2012) where rapeseed extract was used as biodiesel. However, Hu (2012) also reported that only some organic salt was found on the surfaces of mild steel and aluminium. These organic salts are possible by-product for the reaction of free fatty acids with biodiesel which acts as ions. Corrosion rate on cooper are higher than mild steel followed by aluminium as reported by Thangavelu, Ahmed, & Ani (2016). Corrosion of cooper are due to the formation of cupric oxide (CuO) leaving black colour layer on the surface (Haseeb et al., 2010).

Fazal (2014) reported that oxidation of biodiesel has the capabilities to produce several types of fatty acids. This is critical for the formation of different metal oxide which eventually accelerate the corrosion. Acceleration of corrosion rate are observed by Fazal (2011) when further investigation carried out on the effect of temperature on corrosion behaviour. Both diesel and palm oil origin biodiesel have exhibits with the increase of corrosion rate with the rise of temperature (Fazal, Haseeb, & Masjuki, 2011b). Moreover the oxidation instability occurs during the immersion of mild steel on the elevated temperature due to increase of oxygen in biodiesel (Fazal et al., 2011b).

2.4.1 Corrosion of Aluminium Alloy in Biodiesel

The shift of automotive industry to aluminium alloy due to the stiff competition on weight reduction which eventually reduces drag and will improve fuel consumption. Currently, light weight metal such as Aluminium and Magnesium alloy are making its move towards replacing the conventional iron blocks particularly in high performance vehicle (Chew, Haseeb, Masjuki, Fazal, & Gupta, 2013). The investigation of corrosion behaviour on these light weight non-ferrous metals such as aluminium and magnesium alloys remains inadequate (Chew et al., 2013).

Weight loss method are one of the widely used methodology to study the corrosion behaviour. Investigation reveals that the corrosion rate for aluminium alloy 5086 is at 3.0910 mpy when it is immersed in 100% palm biodiesel at room temperature for 1440 hrs (Chew et al., 2013). However, the similar studies on the weight loss reveals that the corrosion rate is only at 0.66 mpy when it is immersed in waste biodiesel with 0.1% of rosemary extract at same room temperature (Deyab, 2016) . The corrosion rate also decreases proportional to the increase of rosemary extract concentration. It was reported that rosemary plant exhibits antioxidative properties due to the presence of phenolic acid, flavoid diterpenoids and triterpenes which decreases the corrosion reaction of the biodiesel on aluminium (Chen, Shi, & Ho, 1992).

Similar investigation using 100% sunflower biodiesel on pure aluminium results in corrosion rate of 0.2710 mpy for room temperature and 0.540 mpy for an elevated temperature of 60 C (Cursaru, Brănoiu, Ramadan, & Miculescu, 2014). It was observed by Cursaru (2014) that corrosion rates doubled for not only aluminium but cooper and mild steel as well when it was immersed in higher temperature. Further studies on using different percentage of composition of rapeseed methyl esters (RME) biodiesel reveals presence of corrosion as well (Norouzi, Eslami, Wyszynski, & Tsolakis, 2012). Norouzi

(2012) reported that the corrosion rate rises with the increase of RME concentration biodiesel as well. Aluminium alloy 6060 was used in this investigation and the corrosion rate was approximately at 0.325 mpy for immersion in 100% RME biodiesel. Unsaturated fatty acids especially oleic acid and linoleic acid are found to be higher with the increase of concentration of biodiesel (Norouzi et al., 2012). Hence, corrosion reaction increases with the higher concentration of biodiesel (Norouzi et al., 2012).

Almost all the papers have reported that the metals are prone to corrosion reaction when the concentration of biodiesel increases. Adding to that, studies on using biodiesel-diesel-ethanol (BDE) blends also have resulting to increase of corrosion proportional to higher concentration and raised temperature (Thangavelu et al., 2016). Corrosion rate of approximately 0.06 to 0.07 mpy was recorded when pure aluminium was immersed with BDE blend at room temperature and 0.21-0.22 mpy for the same blends at 60 C. The influence of temperature on the corrosion rate of aluminium are also significant in the study by S.K Fasogbon (2016). The corrosion rate was found at 0.003644 mm/year for room temperature and increases tremendously to 0.319652 mm/year at 100 C with the same 100% cooking oil based methyl ester biodiesel (S. K. Fasogbon, 2016). This correlation is due to the presence of methyl ester as an oxygenated fuel which have a tendency to be ready more quicker at higher temperature of the fuel (S. K. Fasogbon, 2016).

Kaul et al. (2007) have conducted corrosion investigation of aluminium in different types of biodiesel such as Salvadora, Karanja, Mahua and Jatropha Curcas. The studies reveal that aluminium was more susceptible to corrosion in Jatropha Curcas and Salvadora compared to another biodiesel and diesel. For the studies of corrosion of aluminium exposed to palm oil biodiesel, the surface morphology does not exhibit any significant changes or defects (Chew et al., 2013).

Besides, oxidation or other corrosion product is not found on aluminium leading to possibility of XRD technique's inefficiency to detect such a thin layer (Chew et al., 2013).

The surface morphology studies found to be limited as well. Further investigation by Chew (2013) on the surface of aluminium found that no formation of oxide was identify. This may be due to very thin layer of formation which unable to detect by XRD technique (Chew et al., 2013). However, the TAN values increase significantly to 0.902 mg KOH/g shows that the palm oil biodiesel has been degraded due to oxidation of unsaturated components of aluminium surface (Chew et al., 2013). The surface investigation by Norouzi et al. (2012) using RME blends found that the protective film were generated on aluminium surface due to passivation which influences the corrosion behaviour. TAN figures have also showed in converging sequence ranging from 1.5 to 3 mg KOH/g with respect to an increase of RME blends composition in biodiesel. The TAN of 1.68 mg KOH/g was found in B100 palm oil which was immersed with aluminium reported by Fazal, Haseeb, & Masjuki (2012). This figure was relatively same with cast iron and relatively lower compared to brass and cooper (Fazal et al., 2012). However, these figures were way above the ASTM D6751 TAN limit. Increased TAN number are primary indication of the presence of corrosive acids which eventually lead to higher corrosion. Kaul (2007) also reported similar TAN trend on his investigation. The increase of TAN after the immersion test indicates that oxidation due to contact with metal surface. Oxidation also increases with the addition of water content which leads to increase of corrosiveness (Tsuchiya, Shiotani, Goto, Sugiyama, & Maeda, 2006).

Pitting corrosion was found by Fazal et al. (2010) at 18% for B100 palm oil and 10% for B0 diesel. Besides, the EDS analysis found that higher percentage of carbon and oxygen on the oxide layer proves that passivation occurs. The growth of oxide layer may occur by metal affecting in and out (Goldstein, 1960).

As conclusion based on the several studies related to corrosion behaviour of aluminium and its alloy in biodiesel, the biodiesel's raw material plays huge role in determining the corrosion rate. In addition, the concentration and temperature also effect the corrosiveness. Concentration of biodiesel also reduces the corrosion resistance. Hence, further study on identifying new raw material for biodiesel are essential.

2.4.2 Recent Studies on Ceiba Pentandra (CP) as Biodiesel Blends

Ceiba pentandra (CP) commonly known as kapok or its scientific name as *Ceiba pentandra (L.) Gaertn* are generally found in Sri Lanka, India, South East Asia, East Asia and Afrika (Abdullah, Rahmah, & Man, 2010). The fibres are mainly used as stuffing material for beds and pillows in Malaysia (Abdullah et al., 2010). CP fibres are lightweight, fluffy and has rich oiliness (T.-T. Lim & Huang, 2007) . The oil is extracted from the seeds which usually occupy around 25-28% of each fruit (Y.K. Walia, 2009). The CP seeds contains considerable amount of oil which is 27.5%, 35.0% of protein and 19.0% of fibres (Anwar, 2014).

Figure 6: Ceiba pentandra seeds (T. K. Lim, 2012; Ong et al., 2014)

Ong (2014) states that the CP biodiesel satisfies both EN14214 and ASTM D6751 standards whereby the vital properties such as density, viscosity, flash point and calorific values are met. The CP10 produces the best engine performances with acceptable emission characteristic (Ong et al., 2014).

Table 2.5 provides information on the both physical and fatty acid's properties. Fatty acids properties are influencing factor in determining the corrosion behaviours.

Table 2.5: Properties of crude Ceiba pentandra, (Silitonga, Ong, Mahlia, Masjuki, & Chong, 2013)

Kinematic viscosity at 40 °C (mm2/s)	34.45
Density at 15 °C (kg/m3)	905.2
Acid value (mg KOH/g)	16.80
Flash point (°C)	170.5
Fatty acid composition	**Percentage (wt.%)**
C12:0 (Lauric acid)	0.1
C14:0 (Myristic acid)	0.1
C16:0 (Palmitic acid)	0.1
C16:1 (Palmitoleic acid)	0.3
C18:0 (Stearic acid)	2.6
C18:1 (Oleic acid)	17.4
C18:2 (Linoleic acid)	39.7
C18:3 (Linolenic acid)	1.5
18: CE[a] (Malvalic acid)	18.5

Oxidation stability is vital for determining the quality of fuel and it is governed by the EN14214 standard for biodiesel. This oxidation may occur when biodiesel in contact with ambient air, temperature, water or moist in fuel tank and dust particle (Pullen & Saeed, 2012). Oxidation stability often classified as the fuel's tendency to react oxygen at ambient temperature (Pullen & Saeed, 2012). The value of the oxidation stability for CP is recorded at 4.42 hrs (Silitonga, Ong, et al., 2013). However, the CP biodiesel blends are above the minimum standard of 6 hours whereby CP10, CP20 and CP 30 are respectively at 20.82 hrs, 15.82 hrs and 11.82 hours (Silitonga, Masjuki, et al., 2013).

[a] CE: cyclopropane ester.

The investigation by Silitonga (2013) proves that CP are potential non-edible biodiesel feedstock. Several studies have also observed on the compatibility of CP blends as biodiesel whereby it satisfies both standardisation and performance (Ong et al., 2014; Silitonga, Ong, et al., 2013). Nevertheless, the most appreciate CP blends are yet to be determined from the corrosion perceptive.

CHAPTER 3: METHODOLOGY

3.1 Materials

The surface preparation of aluminium alloys is vital in making sure no foreign material influences the corrosion reaction. This is due to the characteristic of corrosion hugely dependent on the surface preparation. On this investigation, the AA3003, AA5052 and AA6061 was procured from local supplier. The sample was cut using a mechanical saw machine at 2.54 cm by 2.54 cm (1 in x 1 in) with the thickness of 0.30 cm. Different grades of aluminium alloys exhibits different corrosion behaviour due to its alloying composition. The detailed composition with the major alloying element are shown in Table 3.1.

Table 3.1: Chemical composition of AA3003, AA5052 and AA6061

Aluminium Alloy Grades	Element Composition, %									
	Mn	**Zn**	**Si**	**Cu**	**Al**	**Fe**	**Mg**	**Cr**	**Ti**	**Others**
3003	1.5	0.1	0.6	0.2	96.75	0.7	0	0	0	0.15
5052	0.1	0.1	0.25	0.1	95.75	0.4	2.8	0.35	-	0.15
6061	0.15	0.25	0.8	0.4	95.85	0.7	1.2	0.35	0.15	0.15

3.1.1 Preparation of samples

Firstly, the samples were washed using water to remove the cutting excess. It is then mechanically grinded using silicon carbide abrasive paper ranging from grade 400 to 1200 to make sure the surface starches are removed and to obtain smooth surface. The samples were then degreased with acetone and rinsed with distilled water before it was left dried in desilicated environment for 24 hours. Weight of those samples were then measured using electronic weight balance machine with accuracy of 0.01mg before exposing in various biodiesel blending composition.

3.1.2 Biodiesel – Commercial Diesel Blending

The Ceiba pentandra biodiesel was produced in the University Malaya's fuel technology lab whereas the commercial diesel was acquired from Petronas gasoline station. The characterisation of this Ceiba pentandra biodiesel properties was tested in compliance of both EN 14214 and ASTM 6751 as published in paper (Silitonga, Ong, et al., 2013). However, no papers have reported on this blended biodiesel properties. Nevertheless, this study was carried out to investigate its properties limited to the density, kinematic and dynamic viscosity only.

It is noted that the commercial diesel already has 5% of palm oil biodiesel as its composition in line with government mandatory requirements. The preparations of biodiesel-diesel were blended using electric magnetic stirrer at a speed of 2000 rpm in order to achieve homogenous mixtures. In this research, the ratio of CP10 represents the blending of 10% biodiesel with respect of 90% commercial diesel. Consistently, others were categorised at CP20 and CP30.

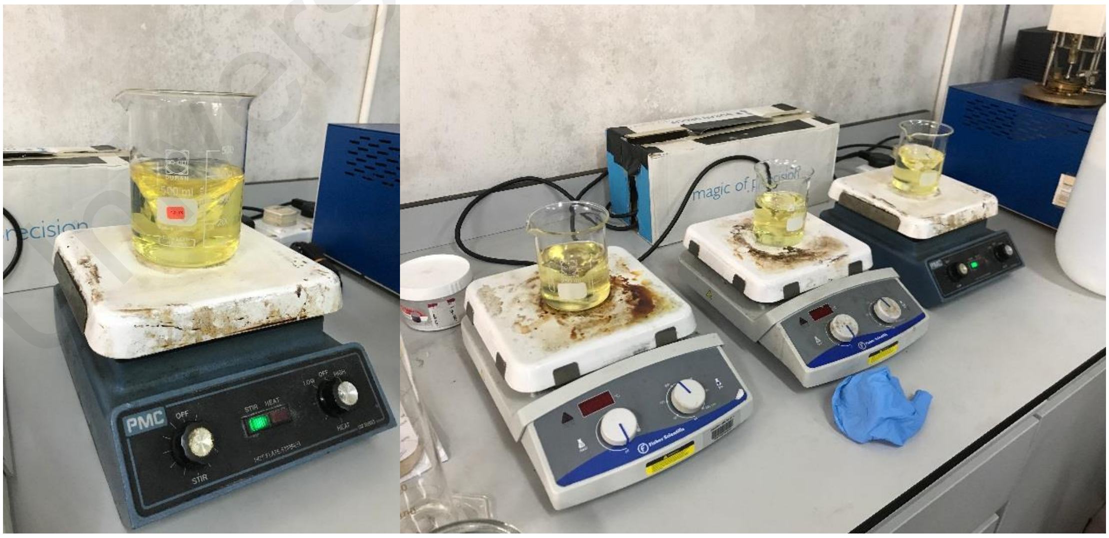


Figure 7: Biodiesel – commercial diesel blending at different ratio

3.2 Experiment Setup

3.2.1 Static immersion test

Samples were then immersed in different biodiesel blending starting from B5, CP10, CP20 and CP30. Small airtight containers were used to immersed those samples and it was left at room temperature ranging from (24-27 °C) for 1200 hours. Figure 8 represent one sample of AA immersed in CPBD blends and this step was repeated for 3 samples within the same grade.

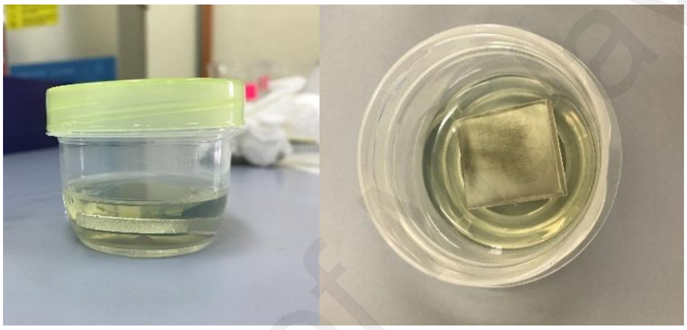


Figure 8: Immersed AA samples with CPBD blend

Upon completion of immersion, the samples were then removed and was cleaned with reference to ASTM G1-03 chemical cleaning procedure standards. The cleaning preparation of the samples were made by brushing with soft toothbrush to remove oxidation layer and any loose deposit on the surface. Samples were then immersed in diluted 65%wt nitric acid solution and left in ultrasonic cleaner for approximately 3 minutes to completely remove the corrosion products. Nitric acid was diluted so that no excessive base metal is removed during the cleaning process. The samples were then rinsed with distilled water before left dried under room temperature. Glass desiccator with silica gel was used to place those AA samples in order to make sure any moisture content is completely removed. Samples were left in desiccator for 7 days. The post static immersion cleaning process are well documented in Figure 9.

Figure 9: Process of cleaning corrosion product

Figure 9 (a,b) represents the usage of ultrasonic cleaner whereby cavitation bubbles are developed by the high frequency sound to remove the impurities within the surfaces of the alloy. In addition, silica gel in Figure 9 (c) is used as moisture absorbent and it is wrapped with the samples which finally left in desiccator as shown in Figure 9 (d).

3.2.2 Weight loss

After completion of immersion test, the mean weight loss of 3 samples was measured for each condition using the same electronic weight balance machine. The weight loss results was used to calculate the corrosion rate in accordance to ASTM D 2688-5 which also being referenced by Norouzi et al. (2012) as in Equation 6.

$$\text{Corrosion rate} = \frac{W \times 3.45 \times 10^6}{D \times T \times A} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ \textbf{Equation 6}$$

The Equation 6 satisfies the representation of mpy as mils per year, W as weigh loss in grams, D as the density of the metal in g/cm^3, T as the exposure time in hours and A as the exposure area as cm^2.

3.2.3 CPBD blends properties analysis

The physical properties of CPBD-commercial diesel blends were analysed by using SVM 3000 automatic from Anton Paar, UK which is available in UM fuel technology lab. The CPBD– diesel blend was previously investigated to ensure its compatibility for biodiesel in accordance to ASTM D6751 (Yunus Khan et al., 2015). However, this test is repeated with CPBD-commercial diesel which has 5% palm biodiesel to ensure the properties are within the range as specific by the standard. Each blend (30ml) is injected to the equipment and validation were conducted to determine the density °C, kinematic viscosity and dynamic viscosity. Each testing was conducted thrice to ensure mean value calculated. Toluene reagent was used in between each testing and the tube was rinsed with distilled water as a cleaning agent so that the residue of oil in the system does not influence the results. Figure 10 shows both test equipment and cleaning agent.

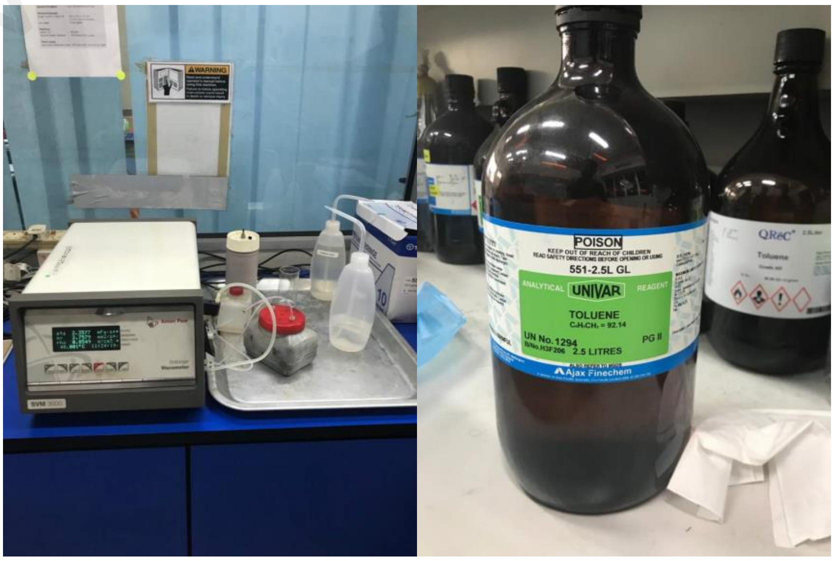


Figure 10: SVM 3000 Viscometer and Toluene Reagent

3.2.4 Microstructure analysis using SEM and EDS

Surface analysis was conducted at Central Advance Research Enabler (CAREF) facility using Phenom Pro X table top SEM with integrated EDS capabilities. The post immersion samples were subjected to SEM to identify the surface condition and to determine evidence of passivation. The images were observed using back scattered electron at the vacuum modes at up to 15kV.

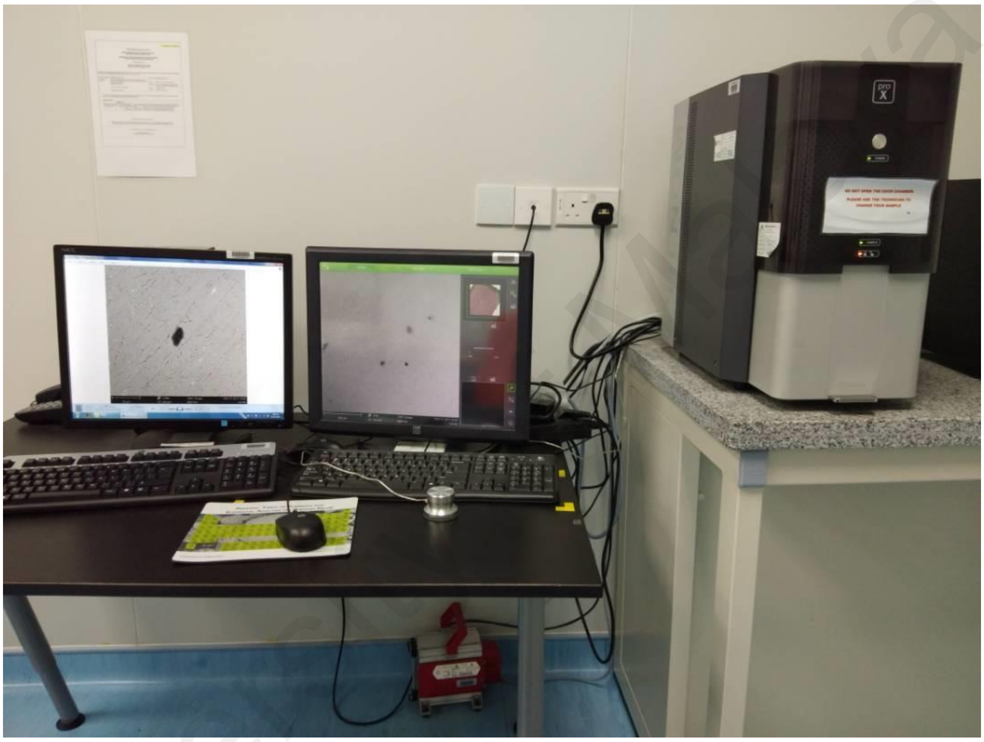


Figure 11: Phenom Pro X SEM equipment

The EDS analysis was also conducted using the same equipment as shown in Figure 11 whereby it uses silicon drift as the detector.

CHAPTER 4: RESULTS AND DISCUSSIONS

This chapter gives comprehensive views on data and findings based on the investigation which was conducted in parallel to the chapter 3 methodology. Results were divided into three parts which are corrosion rate analysis using weight loss method, biodiesel blends properties and lastly using the SEM and EDS.

4.1 Corrosion rate

The corrosion reaction of metals is one of the most important prerequisite of biodiesel and it always influences the percentage of biodiesel blending when it is used as engine's combustion fuel. The corrosion rate of different grades of aluminium alloy with respect to the different blend of CPBD is shown in Figure 12.

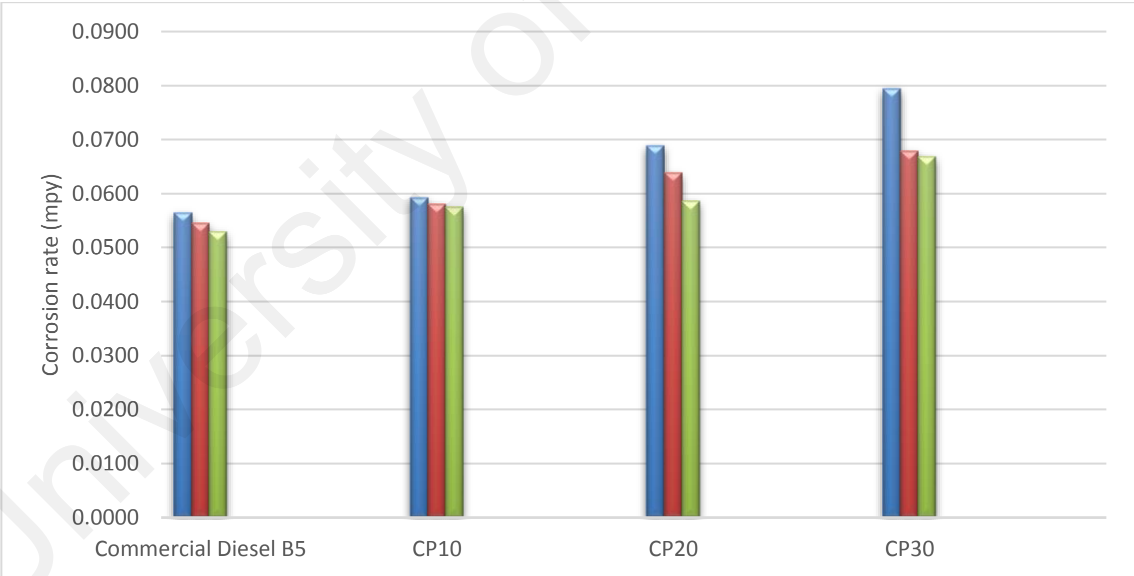


Figure 12: Corrosion rates for different percentage of CPBD blends

The corrosion of AA3003 is more severe than both AA5052 and AA6061 in all 4 biodiesel conditions. Corrosion rate of AA3003 is at 0.0564 mpy in commercial diesel compared to 0.0794 mpy in CP30. This change contributes to almost 30% of increase in the corrosion rates.

This was justified with the investigation by Fazal et al. (2010) which states the presences of free fatty acids, water content, impurities and oxygen moieties in biodiesel is significantly higher compared to diesel. However, AA3003 corrosion rate does not shown any significant increase for CP10 compared to the one in commercial diesel. This is may be due to the only small increase in the CPBD concentration. Nevertheless, the corrosion rate of aluminium is higher in biodiesel compared to diesel as reported by (Fazal et al., 2010).

AA5052 and AA6061 exhibits corrosion rates at similar level in equivalent to the increase of CPBD blends. Comparison of the both grades of aluminium alloys reveals that there was 20% of increase in the corrosion rates for CP30 with respect to the samples immersed in commercial diesel. From the Figure 12, it is clear that the corrosion rates increase with the higher concentration of CPBD blends. Kaul et al. (2007) have reported that the higher concentration of unsaturated acid components further increases the corrosiveness. Fazal et al. (2010) have also observed same results using palm biodiesel whereby the unsaturated fatty acids increase the corrosion reaction.

Norouzi et al. (2012) found that the composition of free fatty acid particularly unsaturated acids of oleic acid (C18:1) and linoleic acids (C18:2) may influence corrosion effect of biodiesel. Based on the feedstock characterisation study by Yunus Khan et al. (2015) , CPBD exhibits both oleic acid at 20.1% and linoleic acid at 38.1% respectively. This contributes up to almost half of the fatty acid composition which proves to be the reason behind the increase of corrosion rate when the CPBD concentration raised up to 10%, 20% and 30%.

4.2 Surface morphology

The SEM micrographic of aluminium alloys after immersion in B5 commercial diesel are shown in Figure 13. Figure 13 (a) represent for AA3003, Figure 13 (b) for AA5052 and Figure 13 (c) for AA6061. At range of 1700- 3000X magnification, the grinding lines are clearly visible in all samples. It was observed that the aluminium alloy surface does not undergone any significant microstructure variations.

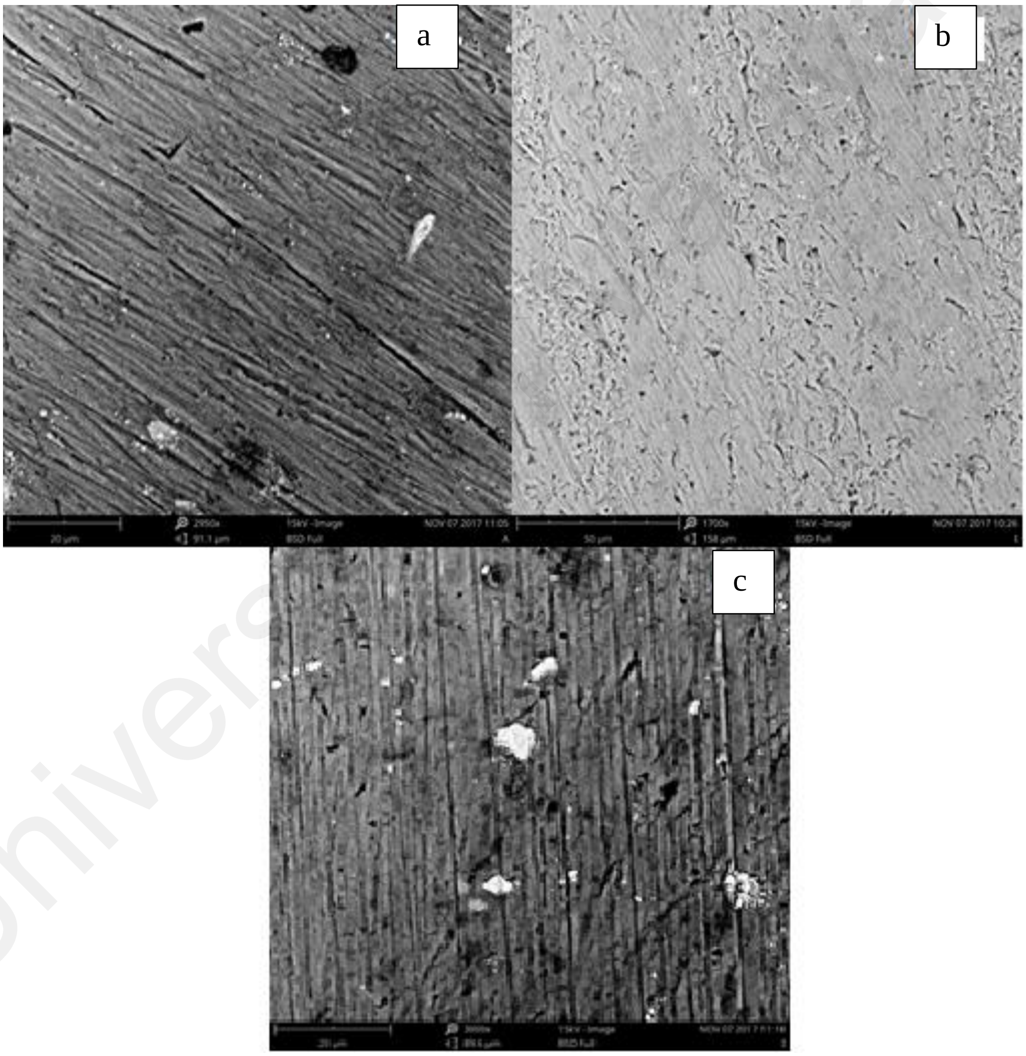


Figure 13: SEM images of AA after immersion in B5 for 1200 hours at room temperature

Despite samples were immersed in commercial diesel, the oxide layer was also detected from EDS analysis. This was mainly due to the 5% palm oil biodiesel blend on our Malaysian commercial diesel. It was reported in numerous pages that there was no carbon and oxygen on the surface after immersed in petrol-diesel. Adding to that, Hu et al. (2012) also reported that there was no presence of both carbon-oxygen element from the samples immersed in diesel.

Figure 14 (a, b) shows the microstructure image of AA3003 samples immersed on CP10 blend. EDS analysis confirmed that passivation occurs on the samples whereby an oxide film is detected at Figure 14 (a).

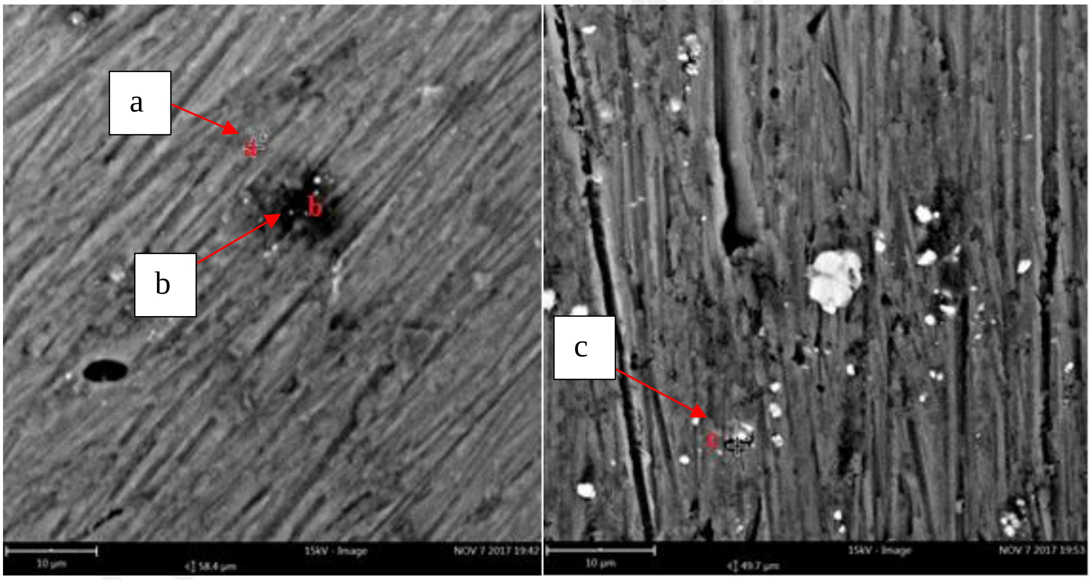


Figure 14: SEM image for AA after immersion on CP10 for 1200 hours at room temperature

Table 4.1: EDS results for AA immersed in CP10

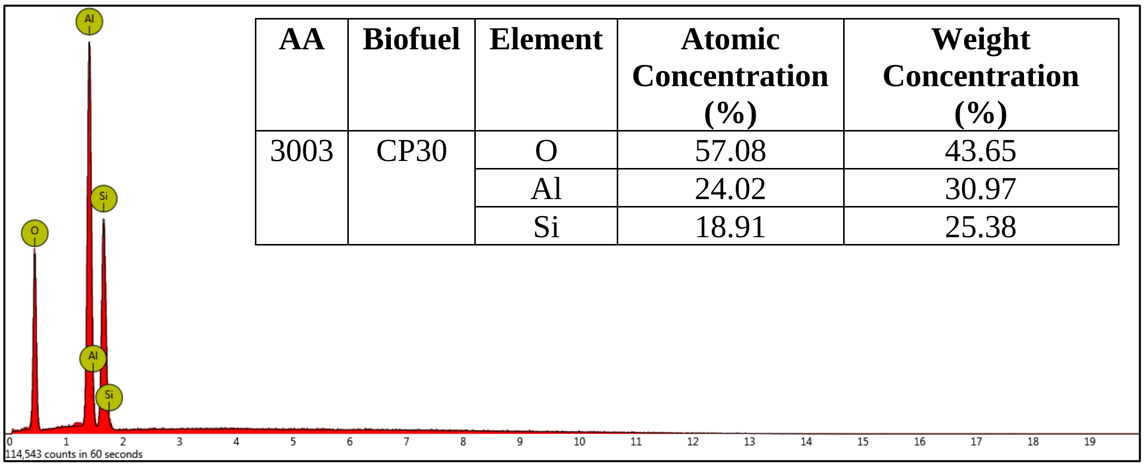
AA	Location	Element	Atomic Concentration (%)	Weight Concentration (%)
3003	a	Al	80.89	87.71
		O	19.11	12.29
	b	Al	37.56	53.64
		O	31.56	26.73
		C	30.88	19.63

Table 4.1: continued,

AA	Location	Element	Atomic Concentration (%)	Weight Concentration (%)
5052	c	Al	70.69	77.18
		O	20.81	13.47
		Si	6.49	7.37
		Mg	2.01	1.98

Despite the fact that carbon element was not presence on the AA samples immersed in B5 commercial diesel but it appears on the AA3003 immersed in CP10. The results from EDS analysis reveals that there is presence of carbon at Figure 14 (b). The works from Hu et al. (2012) noted that the reaction of biodiesel's fatty acids influence in the production of salts that appears on the surface of metals leading to an increase of carbon and oxygen. The carbon content at the corroded surface signifies the formation of organic deposit since there is no carbon in the AA5052 composition as reviewed earlier in literature. Despite cleaning the immersed samples with diluted nitric acid and acetone, deposits appear to have adhesive characteristic. Figure 14 (c) represents sample of AA5052 whereby the EDS analysis as shown in Table 4.1 detects the present of oxide layer. Thus, we can conclude that the passivation has occurred on as early as in CP10 blends including in AA6061 sample.

Generally, the biodiesel contains approximate at range of 10-12% of oxygen while no oxygen in presence in diesel (Fazal, Haseeb, & Masjuki, 2011) . The CP30 blend exhibits even higher oxide layer concentration on AA3003 as shown in Figure 15.



AA	Biofuel	Element	Atomic Concentration (%)	Weight Concentration (%)
3003	CP30	O	57.08	43.65
		Al	24.02	30.97
		Si	18.91	25.38

Figure 15: EDS spectrum of AA3003 immersed in CP30

Higher concentration oxygen may be due to the oxygenated compound which are adherent to the aluminium surface despite cleaning. The oxygen compound was at 57.08% the surface spot of AA3003 exposed in CP30 as referred in Figure 15. Thus, the presence of higher oxygen in CP30 biodiesel may influence the interaction with metal. Besides, the nature of biodiesel's ester molecules which are more hygroscopic and polar could increase the chemical reaction with metal leading to surge in corrosion (Sgroi, Bollito, Saracco, & Specchia, 2005; Tsuchiya et al., 2006). Therefore, the correlation of increased corrosion in higher concentration of biodiesel are well observed.

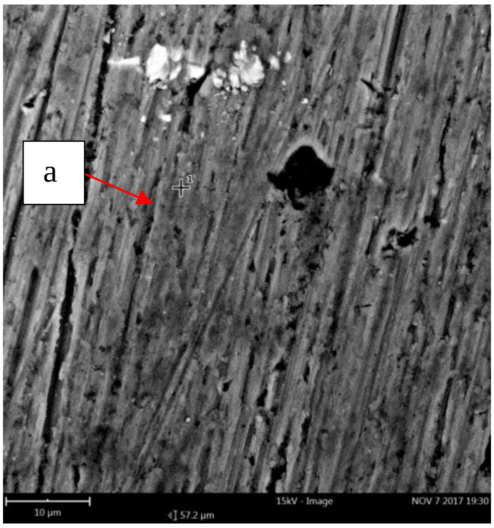
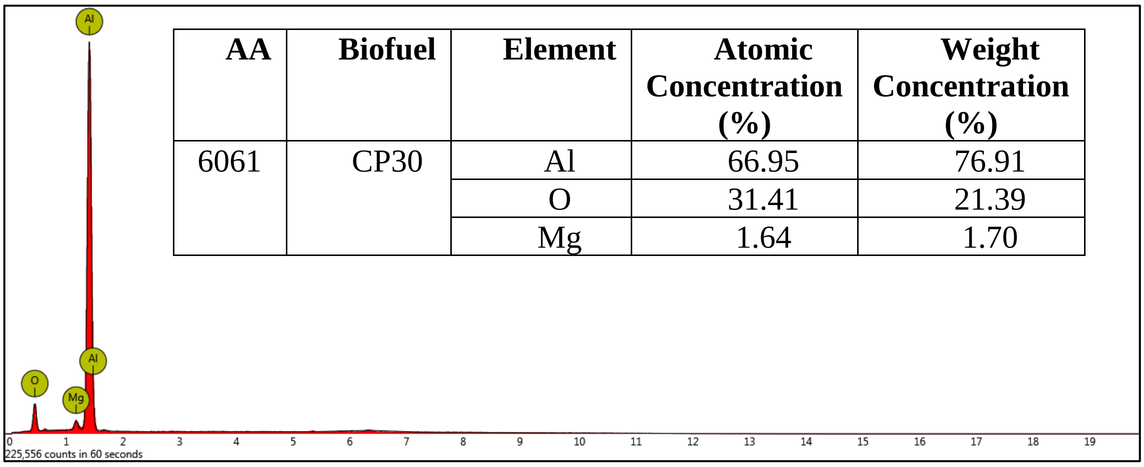


Figure 16: SEM image for AA6061 after immersion on CP30 for 1200 hours at room temperature



AA	Biofuel	Element	Atomic Concentration (%)	Weight Concentration (%)
6061	CP30	Al	66.95	76.91
		O	31.41	21.39
		Mg	1.64	1.70

Figure 17: EDS spectrum of AA6061 immersed in CP30

The oxygen element detected on EDS analysis appears to be scattered around the AA surfaces. Fluctuation weight concentration of oxygen on AA surface throughout different immersion parameter may be due to uneven metal degradation. The EDS spectrum shown in Figure 17 also results in oxygen presence.

4.3 CPBD blends properties

The characterisation of the CPBD and its blends with B5 commercial diesel was performed. This investigation is essential to determine which biodiesel blends satisfies the both ASTM D6751 and EN14214 limits. It was observed that the density increases with respect to the increase in the concentration of CPBD. However, the commercial diesel B5 density and CP10 density almost similar with value of 851.0 kg/m^3 and 851.1 kg/m^3 respectively. The converging trend in shown in Figure 18 whereby the 10% increase of CPBD concentration only contributes 1.8-2.8 % of differences.

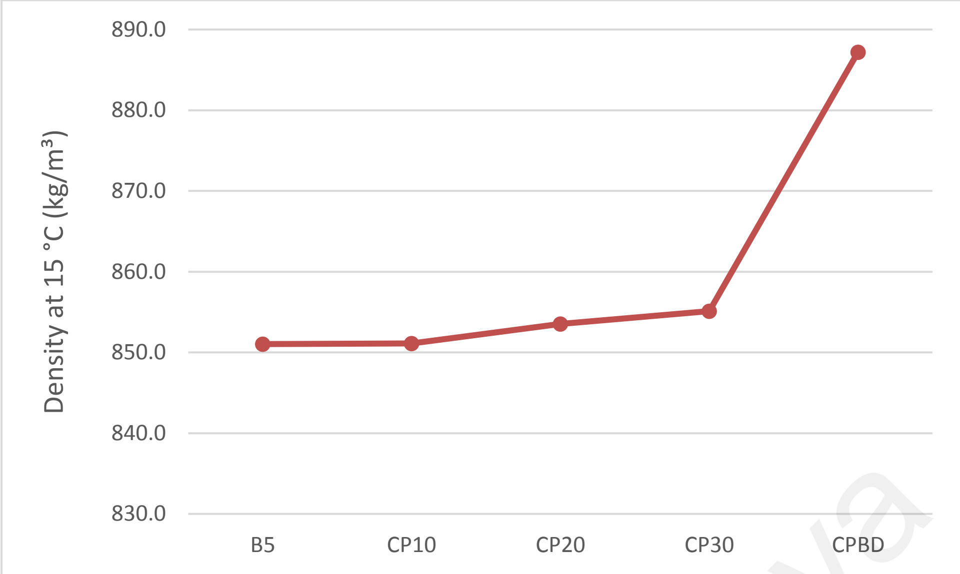


Figure 18: Density of CPBD blends at 15°C

The density value of CP10, CP20 and CP30 falls slightly behind the EN14214 of 860-900 kg/m³ but still does not exceed the upper limit (Silitonga, Ong, et al., 2013). Nevertheless, the value is still within the acceptable in ASTM D6751 limit of 880 kg/m³. Hence, the density value satisfies for all three CPBD blends.

Kinematic viscosity is an important characteristic of fuel's physical properties. This is mainly due to an increase of kinematic viscosity may lead into excessive fuel injection pressure which eventually lead to engine problem (Demirbas, 2006). Both dynamic and kinematic viscosity have exhibits converging value in relation to the increase of CPBD concentration as shown in Figure 19.

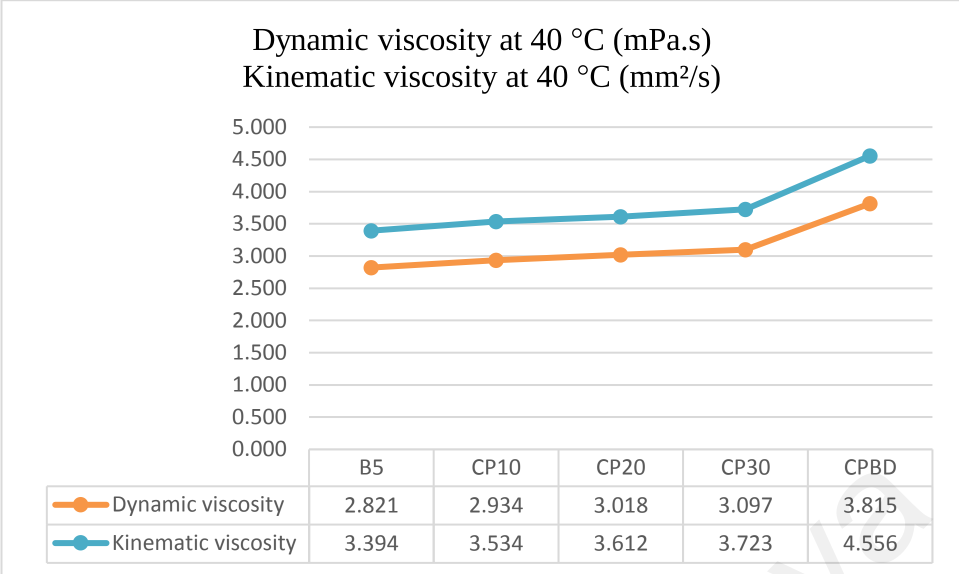


Figure 19: Dynamic and kinematic viscosity of B5 & CPBD blends

Despite the increase of viscosities of CPBD blends compared to commercial diesel B5, it is still within the ASTM D6751 limit of 1.9 to 6 mm^2/s. It was reported that higher viscosity blends are more prone to oxidation (Yaakob, Narayanan, Padikkaparambil, Unni K, & Akbar P, 2014) . The rapid oxidation on the higher viscosity blend are mainly due to the isomerisation of double bond typically cis to trans (Yaakob et al., 2014). Besides, Yaakob et al. (2014) have mentioned that the addition of higher molecular weight products influences the tendency to rapid oxidation. Controlling the viscosity level at lower limit are essential since degradation are highly anticipated during the storage.

Studies reveals that biodiesel samples of palm oil methyl ester (POME), coconut oil methyl ester (COME), jatropha oil methyl ester (JME), 20% blends of JME with diesel and 20% blends of PME have an increased viscosity at 40°C as the storage time increases (Shahabuddin, Kalam, Masjuki, Bhuiya, & Mofijur, 2012).

Despite that, the diesel-biodiesel blends fuel had exhibits minimal change in viscosity level. The investigation by Shahabuddin et al. (2012) justifies the oxidation compatibility of all CPBD-commercial diesel blends.

CHAPTER 5: CONCLUSIONS

This study is based on the different blends of ceiba pentandra biodiesel and its corrosion behaviour towards different grades of aluminium alloy. The corrosiveness of ceiba pentandra biodiesel at 30% blends with B5 commercial diesel are higher than the standalone commercial diesel. The increase of ceiba pentandra biodiesel's concentration has strong influence on the corrosion reaction of aluminium alloy. The corrosion rate was calculated based on static immersion weight loss method and it was observed that the corrosion tendency was converging towards (B5>CP10>CP20>CP30).

Both SEM and EDS analysis reveals presence of oxygen and carbon element on the surface of aluminium for the samples immersed in CP10 whereas only oxygen at B5 commercial diesel. The presence of oxygen reveals that passivation has been occurred even in commercial diesel stage for all grade of aluminium alloy. However, the corrosion rate was kept in minimal since the oxide layer deemed to be a protective layer. In contrast to that, the unsaturated free fatty acid from biodiesel has penetrated into the oxide surface and interaction between metals was observed.

Nevertheless, the CP10 blend does not exhibit any significant changes on the corrosion effect compared to the samples immersed in B5 commercial diesel. Hence, CP10 blend are considered to be ideal for any further works since it has the lowest corrosion level without any significant changes on its microstructure despite immersion for 1200 hours. Further investigations are required to broaden up the research area where additional parameter such as influence of temperature, tribological effects and many more may be analysed to study its compatibility.

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