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JUDUL : CHARACTERISTICS OF BIODIESEL PRODUCED FROM PALM OIL VIA BASE CATALYZED TRANSESTERIFICATION

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CHARACTERISTICS OF BIODIESEL PRODUCED FROM PALM OIL VIA BASE CATALYZED TRANSESTERIFICATION

CADENCE ISIS TAY

Thesis submitted in partial fulfillment of the requirements
for the award of the degree of
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JUNE 2012

SUPERVISOR’S DECLARATION

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis entitled "Characteristics of Biodiesel Produced from Palm Oil via Base Catalyzed Transesterification" is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Special Dedication to my supervisor, my family members,
my friends, my fellow colleagues and all faculty members
for all your support.

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ABSTRACT

The depletion of petroleum has prompted the global oil industry to look at an alternative source for fuel from renewable energy source, one of which is biodiesel. Biodiesel is a notable alternative to the widely used petroleum-derived diesel fuel since it can be generated by domestic natural resources such as palm oil, soybeans, rapeseeds, coconuts and even recycled cooking oil, and thus reduces dependence on diminishing petroleum fuel. Interest in biodiesel has been expanding recently due to government incentives and high petroleum prices. The majority of biodiesel today is produced via base-catalyzed transesterification with methanol. In order to find the optimal values of biodiesel yield, it is suggested to find the optimum reaction temperature, reaction time and the methoxide:oil ratio. In this study, the parameters were: reaction temperature of 40, 50, and 60 (°C); reaction time of 40, 50 and 60 (minutes); and methanol to oil ratio of 4:1, 6:1, and 8:1. For every experiment done, raw CPO was treated with magnesium sulphate to remove excess moisture then heated at 50 °C and mixed with methoxide (potassium hydroxide and methanol) according to the considered parameters. The agitation speed was set at 250 rpm. After the transesterification process was completed, the hot mixture was left to settle for at least 12 hours in a separator funnel. As soon as the separation was done, two layers were formed, the lower layer of glycerine and the upper layer of palm oil methyl ester. The product of palm oil methyl ester was washed with 70 °C hot tap water for few times. The final product of biodiesel was transferred to a heated oven at 50 °C to remove excess water. The optimum biodiesel yield from the research was 88 % at methoxide:oil ratio of 6:1, time of 60 minutes and temperature of 60 °C. These parameters were chosen as optimum because it is cost effective regarding time and chemical consumption. According to the result, around 500 mL of biodiesel was produced and homogenised to be used in the physical properties tests. The tests showed the properties of biodiesel produced with density of 876.0 kg/m^3, kinematic viscosity of 4.76 mm^2/s, *cetane* number of 62.8, flash point of 170 °C, cloud point at 13°C, pour point at 17 °C and saponification value 206.95 mg/L. The physical properties biodiesel produced showed that the properties are accepted within ASTM D6751 and European Standards.

Keywords: Biodiesel, palm oil, base catalyzed, transesterification

ABSTRAK

Penyusutan bahan api petroleum telah mendorong industri minyak global untuk mencari sumber-sumber alternatif bahan api daripada sumber tenaga yang boleh diperbaharui yang mana salah satunya adalah biodiesel. Biodiesel merupakan bahan api alternatif yang penting kepada petroleum diesel dan digunakan secara meluas kerana ia boleh dihasilkan daripada sumber-sumber semulajadi seperti minyak sawit, kacang soya, kelapa dan juga minyak masak kitar semula. Secara langsung, ia mengurangkan kadar penggunaan bahan api petroleum daripada sumber asing. Peningkatan minat terhadap penyelidikan biodiesel telah berkembang selaras dengan insentif kerajaan dan harga petroleum yang semakin melambung tinggi. Majoriti biodiesel kini dihasilkan melalui proses *base catalyzed transesterification* dengan metanol. Untuk memperoleh nilai optima biodiesel yang terhasil, adalah penting untuk mengkaji nilai optima suhu tindakbalas, masa tindakbalas dan nisbah methanol kepada minyak. Dalam kajian ini, parameter yang ditentukan adalah suhu tindakbalas pada 40 , 50, dan 60 (°C); masa tindakbalas pada 40, 60 dan 80 (minit); dan nisbah methoxide:minyak sawit iaitu 4:1, 6:1 dan 8:1. Untuk setiap eksperimen yang dilakukan, minyak mentah sawit dirawat dengan magnesium sulfat untuk mengeluarkan lebihan lembapan air. Kemudian, minyak mentah sawit itu dipanaskan pada suhu 50°C and dicampurkan bersama *methoxide* (kalium hidroksida dan metanol) mengikut parameter yang dikaji. Kelajuan pergolakan ditetapkan pada 250 rpm. Selepas proses *transesterification* lengkap, campuran panas itu dibiarkan tidak kurang dari 12 jam di dalam corong pemisah. Dua lapisan terbentuk iaitu lapisan bawah dikenali sebagai gliserin dan lapisan atas sebagai minyak sawit metil ester. Lapisan minyak sawit metil ester akan dicuci menggunakan air paip bersuhu 70 °C selama beberapa kali. Hasil POME iaitu biodiesel akan dipanaskan di dalam ketuhar pada suhu 50 °C untuk mengeluarkan lebihan air. Kadar optima biodiesel daripada penyelidikan ini menghasilkan 88 % dengan keputusan suhu tindakbalas optima adalah 60 °C, masa tindakbalas optima adalah 60 minit dan nisbah *methoxide*:minyak adalah 6:1. Berdasarkan keputusan itu, 500 mL biodiesel telah dihasilkan untuk digunapakai dalam analisis ciri-ciri fizikal biodiesel tersebut. Analisis yang dilakukan ke atas biodiesel ini memperoleh nilai ketumpatan 876.0 kg/m^3, kelikatan kinematik 4.76 mm^2/s, nilai *cetane* 62.8, *flash point* 170 °C , *cloud point* 13 °C dan nilai saponifikasi 206.95 mg/L. Ciri-ciri fizikal biodiesel yang dianalisa ini telah membuktikan bahawa nilai-nilai itu diterima dan masih lagi merangkumi ASTM D6751 dan European Standards.

Kata kunci: Biodiesel, minyak sawit, base catalyzed, transesterification

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LIST OF ABBREVIATIONS

FFB	Fresh Fruit Bunches
POME	Palm Oil Methyl Ester
PO	Palm Oil
CPO	Crude Palm Oil
KOH	Potassium Hydroxide
MetOH	Methoxide
$MgSO_4$	Magnesium Sulphate
PORIM	Palm Oil Research Institute of Malaysia
O&GJ	Oil and Gas Journal
FAME	Fatty Acid Methyl Ester
USDA	U.S. Department of Agricultural
ASTM	American Society of Testing and Materials
SVO	Straight Vegetable Oil
EPAct	Energy Policy Act

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CHAPTER 1

INTRODUCTION

1.0 BACKGROUND OF STUDY

The demand of energy has increased rapidly with growing of world population. The reserves of fossil fuel are being depleted, while the environmental problems caused by their use became serious issue. Thus, the renewable energy has been promptly developed (Manh et al., 2011; S. Mori, 2009; and Werther, 2009). Among the various alternative fuels being developed, the biodiesel has received special attention because it is easy to produce from available and renewable sources (vegetable oils and animal fats), safe to handle and use, eco-friendly, and miscible with petroleum diesel in all proportional for use in existing diesel engines without modification (Tyagi et al., 2010).

Considering the problems faced by agriculture in which the products market ability is limited and much of the land is not utilized as in the case in many countries nowadays, agriculture should be directed to the invention of new alternative products. Biodiesel industry would have a significant beneficial impact on agriculture and rural communities. It would provide an outlet for excess vegetable oil crops and land currently being used to produce surplus crops could be switched to oilseeds to provide additional feed stock for biodiesel (Gerpen et al., 2007).

This research presents the experimental expectation to visually determine the physical properties of biodiesel produced from palm oil. This biodiesel is defined as the alkyl ester of palm oil also known as palm oil diesel.

Palm oil is an extremely versatile commodity which traditionally has been used both as food stuff and as a raw material in non-food items such as cosmetics, soaps, shampoos and washing detergent. Only recently, with rising mineral oil prices and challenges from climate change, there have been calls for palm oil to be used as a renewable energy source. Like any other vegetable oils, palm oil can be used as a fuel in vehicles or for electricity or heat generation (Reinhardt et al., 2007). Due to increasing global needs to reduce dependence on fossil fuel, palm oil biomass offers great potential as a cost-effective feed stock for biodiesel. In this capacity, it is capable of reducing carbon dioxide emission by more than 80%. Therefore, biodiesel is seen as an approach to reduce the impact of the greenhouse effect by 41-57% and as a way diversifying energy supplies to support national energy security plans (Balat & Balat, 2010; Rao et al., 2011).

Biodiesel is produced by the chemical transformation of vegetable oils or animal oils into the corresponding long-chain fatty acid alkyl esters. This transformation is carried out by the transesterification of large, branched triglycerides in oil with a lower alcohol in the presence of an acidic or a basic catalyst (Tyagi et al., 2010). One benefit of this chemical conversion is that these esters have similar properties as the mineral diesel fuel and even better in terms of its *cetane* number (Balat & Balat, 2010; Knothe, 2010). The *cetane* number is related to the time that passes between injection of the fuel into the cylinder and onset of ignition (Predojevic, 2008; Nagi et al., 2008).

Malaysia is the second palm oil producer, therefore Malaysia in the past have focused on palm oil as raw stock used for biodiesel production (Mekhilef et al., 2010) and due to the higher prices of fuel and increasing demand for alternative renewable energy in Western World. In 1983, the Palm Oil Research Institute of Malaysia (PORIM) successfully converted crude palm oil to palm oil methyl ester (POME) through transesterification (Musa, 2010).

1.1 PROBLEM STATEMENT

Depletion of petroleum is an urgent problem need to be solved and it led to much attention on producing biodiesel. Some countries including Malaysia decided to look for bio-based alternative energies which lead to the rising in demand for bio-based feed stocks such as palm oil and rapeseed oil. Therefore, this study is important to check the properties of bio-based fuel to be an alternative for the fossil fuel compared to the standard properties that can be accepted worldwide.

1.2 OBJECTIVES

a) To produce biodiesel from palm oil
b) To analyse the physical properties of biodiesel produced from palm oil (PO) and compare to standard properties.

1.3 SCOPE OF STUDY

The scopes of this study are finding the alternative to fossil fuel from natural resources and the use of vegetable oil which is palm oil for biodiesel production.

1.4 RATIONAL AND SIGNIFICANCE

According to Oil and Gas journal (O&GJ), the estimated worldwide reserves of fuels at the beginning of 2004 were 1.27 trillion barrels of oil and 6,100 trillion cubic feet of natural gas (Briggs and Vasudevan, 2008), which is proven recoverable reserves. At today’s consumption level of about 85 million barrels per day of oil and 260 billion cubic feet per day of natural gas, the reserves represent 40 years of oil and 64 years of natural gas. The growing economic risk of relying primarily on fossil fuels with sources, clean and renewable bio fuels has been known as the answer to the issue of diminishing fossil fuels (Nagi et al., 2008).

Nowadays, the world-wide palm oil market is increasing and this is mainly because of the growing production of biodiesel. The rising prices are bad news to biodiesel producers who rely on palm oil for their feed stock, unless the biodiesel producer happens to be the producer of the palm oil itself. This is one of major reasons why investment in palm oil-based biodiesel refineries is being led by palm oil producers. Based on few criteria, palm oil is the most potential vegetable oil which can be used as raw material to manufacture biodiesel. On the other hand, the usage of palm oil consider to be the most wanted palm oil products for its cheap price, consume low energy and readiness for downstream processing (Khalid and Khalid, 2011).

CHAPTER 2

LITERATURE REVIEW

2.1 BIODIESEL

2.1.1 Introduction to Biodiesel

In the last few years, against the background of increasing concerns regarding the energy supply security as well as environmental concern has increased the interest for renewable energy sources (Borges & Diaz, 2012). This have resulted in some countries to look for bio-based alternative energies which lead to the increase in demand for bio-based feed stocks such as palm and rapeseed oil (for biodiesel) and sugar cane and corn (for ethanol). Due to the increased importance of biodiesel in Malaysia and elsewhere, the impact of this new demand has added a new dimension in the fats and oils market particularly palm oil (Applanaidu et al., 2010).

Biodiesel is a clean burning alternative fuel that comes from 100 % renewable resources such as natural vegetable oils and fats. Biodiesel is made through a chemical process which converts oils and fats of natural origin into fatty acid methyl esters (FAME) that can be used in any diesel engine without modifications. Chemically, it is defined as mono alkyl esters of long chain fatty acids derived from renewable lipid sources (Sagiroglu et al., 2011).

As stated by (Yee et al., 2009), biodiesel also known as biofuel which is biodegradable non-toxic, and typically produces about 60 % less net carbon dioxide emissions than petroleum-based diesel, making it relatively safe and easy to process. Many people confident that biodiesel is the fuel of the future, considering it as energy that could be locally-produced, used and controlled. Biodiesel is gradually gaining acceptance in the market as an environmentally friendly fuel and the demand is expected to increase sharply as an alternative renewable energy source in the near future (Mahlia et al., 2010).

In first half of year 2009 alone, Malaysia exports almost 150,000 tonnes of palm oil biodiesel to other countries as shown in Figure 2.1 (Mekhilef et al., 2010).

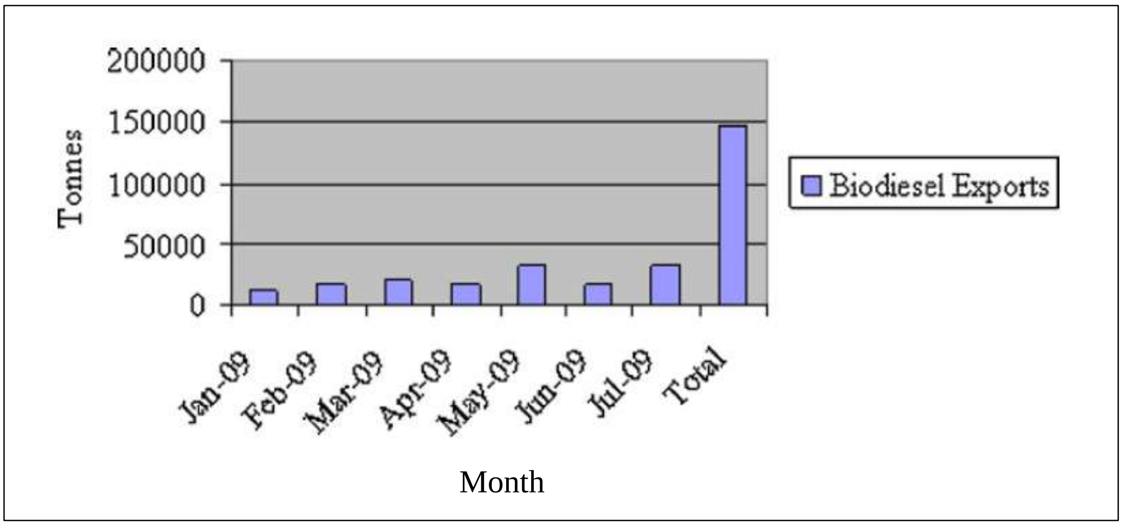

Figure 2.1: Malaysian biodiesel exports (January – July, 2009)

Malaysia exports palm oil biodiesel to several countries like European Union (EU), United States (US), Singapore, Taiwan and other countries as shown in Figure 2.2. European Union has increasing demand for biodiesel; therefore it is important for Malaysia to capture the EU market to become top palm oil biodiesel exporter (Mekhilef et al., 2010).

The development of the oil palm sector in Malaysia is best described as having been most colourful. The growth of the palm oil industry in Malaysia has been phenomenal over the past 30 years. From merely 400 ha planted in 1920, the hectarage increased progressively to 54,000 ha by 1960 (Abdullah et al., 2009). Figure 2.3 shows the cultivated area for oil palm in Malaysia which is divided into three categories of Peninsular Malaysia, Sabah and Sarawak within the next four decades. As noted, the total land fertilized for oil palm plantation shows dramatic rise within these years. In Sabah and Sarawak, the drastic increase in the 1990s can be attributed to the government policy in the intensification of palm oil industries in East Malaysia (Butler, 2005).

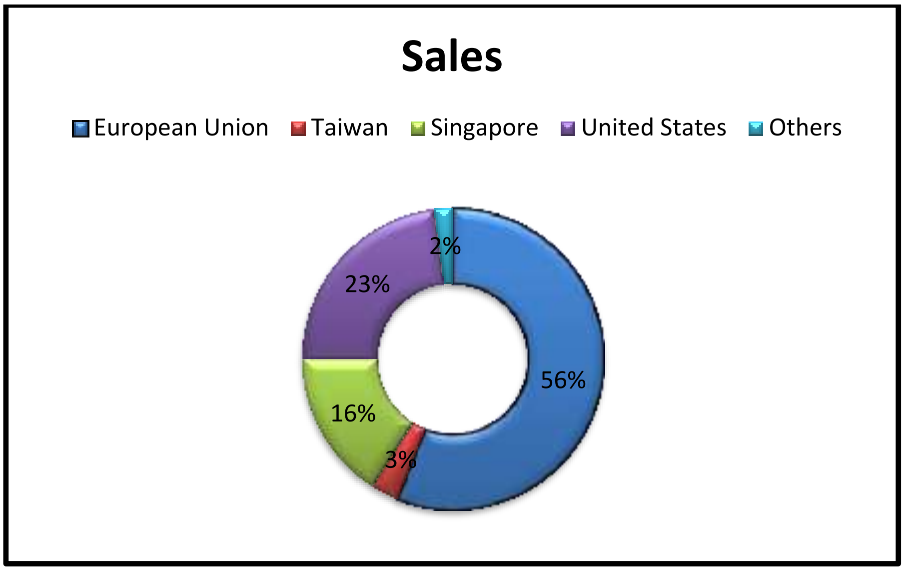

Figure 2.2: Malaysian biodiesel export destinations

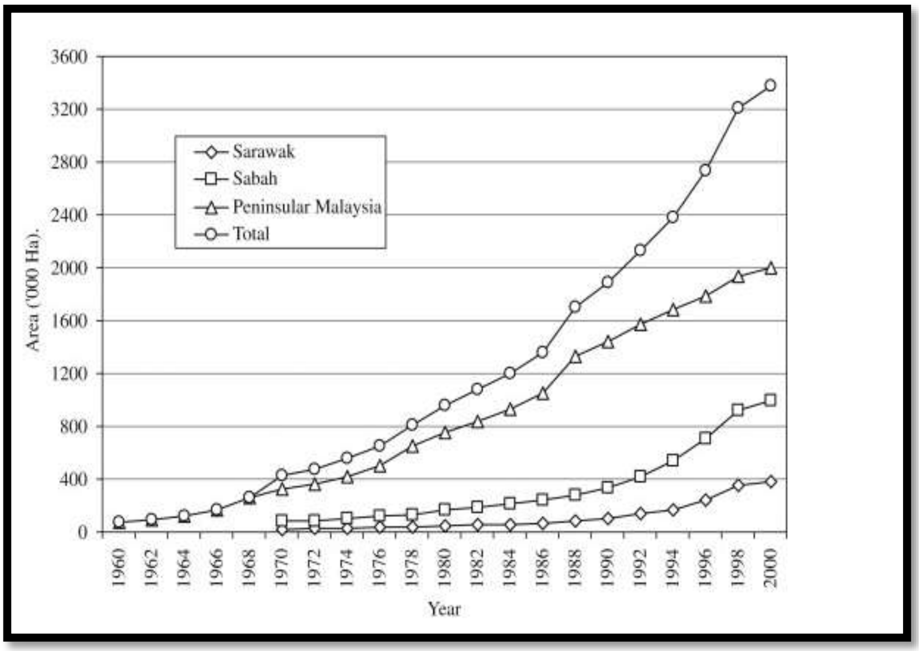

Figure 2.3: Cultivated area for palm oil in Malaysia (1960-2000)

However, Malaysia is currently in danger of being marginalised as our natural competitive advantages diminish. Nations such Indonesia with the resources, capabilities and ambition to grow their domestic palm oil industry are beginning to catch up and threaten our position as a palm oil market leader. From Figure 2.4, it is seen that Indonesia has surpasses Malaysia in production of palm oil and now is the world leader (Butler , 2008).

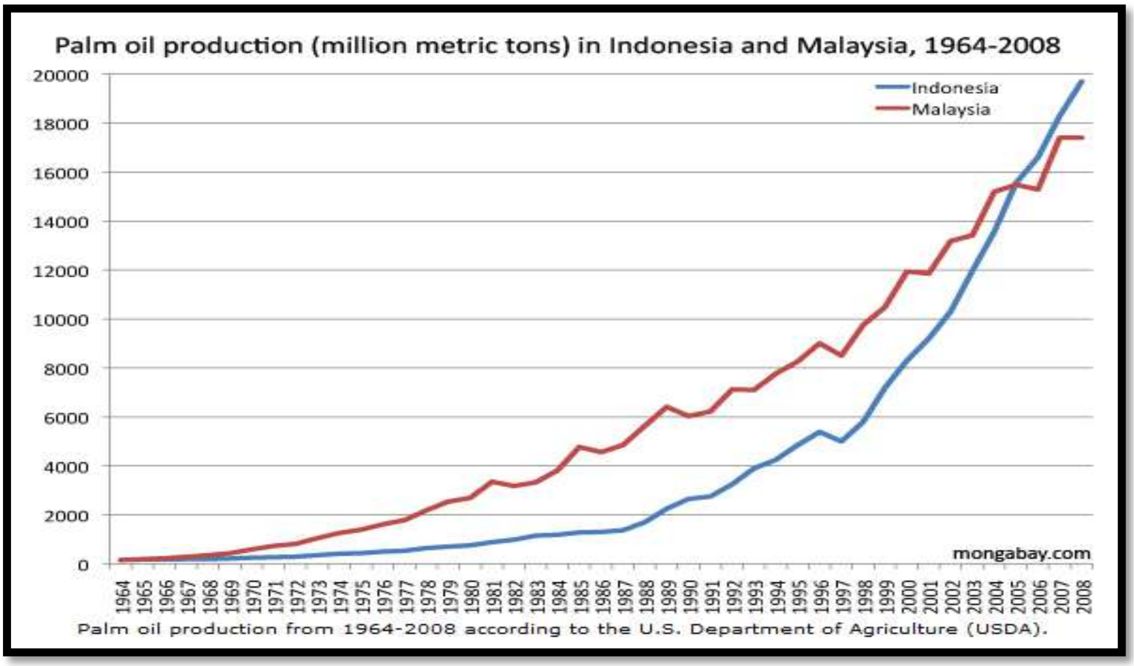

Figure 2.4: Palm oil production from 1964-2008 according to U.S. Department of Agriculture (USDA)

2.1.2 Early History of Biodiesel

The process to produce fuel from fats is not a new process. The history started in the 1880s, when Rudolph Diesel designed a single 10 feet iron cylinder with a flywheel at its base in Augsburg, Germany on August 10, 1893. Later, he designed the compression engine for the World Exhibition in Paris, France. Diesel demonstrated his engine using a common substance, peanut oil. He believed that this form of fuel can be one of the sources needed (Sharma & Singh, 2008). During that era, steam engines were run by coal and processed oils. However, Diesel intended to show that there was a far better method than using biomass fuel. Away in 1912, Diesel has long predicted that the use of vegetable oils for engine fuels would one day become as important as petroleum and the coal-tar products of the present time (Lin et al., 2011).

There were few individuals in the engine industry believed on Diesel's prediction. Henry Ford had created a factory producing mass bio fuels, he believed that this power was perfect in every way and wanted to create all of his automobiles that accepted this fuel. However, in the 1920s, diesel engine manufacturers decided to modify their engines using lower viscosity fossil fuel known as petro diesel.

2.1.3 Biodiesel as an Alternative Source for Fossil Fuel

The term "biodiesel" has its specific and technical definition approved by the American Society of Testing and Materials (ASTM). According to its general definition stated that, "Biodiesel is a domestic, renewable fuel for diesel engines derived from natural oils like soybean oil, and meet the specifications of ASTM D 6751" (Balat and Balat, 2010). Biodiesel can be used in any concentration with petroleum-based diesel fuel in any types of diesel engine. Biodiesel is not the same thing as raw vegetable oil, also known as Straight Vegetable Oil (SVO). To produce biodiesel, vegetable oils or animal fats with short chain primary alcohols such as methanol or ethanol are used as feed stock. It is produced by a chemical process called transesterification by removing glycerine from the oil. Triglycerides in oils or fats can have chemical reaction with the alcohols under suitable conditions (Permsuwan et al., 2011).

Based on its technical definition, biodiesel is defined as the mono-alkyl ester of natural fatty acids produced via transesterification of fats and oils. It is made mainly from renewable biological resources (vegetable or animal fats and oils) and is therefore an ecologically friendly alternative to petroleum-based diesel. Biodiesel fuel is adapted to the diesel engine and may be used in standard diesel engines (Blagoev et al., 2008). In this process, biodiesel is called methyl ester if the alcohol used is methanol and called as ethyl ester if ethanol is used. Usually, methanol is preferable because it is much cheaper than ethanol (Knother, 2010). Meanwhile commonly used catalysts are potassium hydroxide (KOH) or sodium hydroxide (NaOH).

Like petroleum diesel, biodiesel operates in compression-ignition engines. Biodiesel is most often blended with petroleum diesel in ratios of 2% (B2), 5% (B5), and 20% (B20) or as pure biodiesel (B100) (Briggs & Vasudevan, 2008). This blending process of biodiesel and petroleum diesel gives more benefits because it runs in any conventional, unmodified diesel engines. (Gerpen, V.J et al., 2007) reported that B2 is of interest because of its lubricity benefits. Meanwhile, B20 qualifies for fleet compliance under the Energy Policy Act (EPAct) of 1992 helps reduce engine emissions in addition to improved fuel lubricity. B100 provides provides complete replacement of petroleum diesel if the biodiesel could be supplied in sufficient amounts with an affordable cost.

Biodiesel operated on diesel engines produce lower emissions of carbon monoxide, unburned hydrocarbons, and particulate matter and air toxics than operated on petroleum-based diesel fuel. Table 2.1 gives a comparison of palm biodiesel and petroleum diesel based on their characteristics.

Table 2.1: Physical-chemical properties of palm biodiesel and petroleum diesel (Nagi et al., 2008)

Characteristics	**Palm biodiesel (palm methyl ester)**	**Petroleum diesel**
Type of source	Renewable	Fossil
Calorific value (MJ/kg)	41.3	46.8
Gross heat of combustion (KJ/kg)	40.135	45.8
Cetane level	65	53
Flash point (°C)	174.0	98.0
Pour point (°C)	16.0	15.0
Cloud point (°C)	16.0	18.0
Density at 40°C (kg/L)	0.855	0.823
Viscosity at 40°C (cST)	4.5	4.0
Sulphur content (wt. %)	0.04	0.10
Carbon residue (wt. %)	0.02	0.14

2.2 PRODUCTION OF BIODIESEL FROM CRUDE PALM OIL

2.2.1 Raw Stock for Biodiesel

According to (Singh & Singh, 2009), there are several sources which are used as feed stock for biodiesel production such as: soybean, sunflower, palm, canola, cotton seed, Jathropa, rapeseed and soybean oil. However, compared with other vegetable oil, palm oil has far better advantage and potential as feed stock for biodiesel production. Palm oil is a perennial crop, unlike soybean and rapeseed. Perennial crop means the production of oil is continuous and uninterrupted, though annual production has its seasonal peak and down cycle. Palm plantation has the highest oil yield in terms of oil production per hectare of plantation. Palm oil yield from palm plantation is a factor of ten times higher than oil yield from soybean, sunflower or rapeseeds. According to Figure 2.5 below, palm oil has more yield per hectare than any other crops, which makes it the best source to produce biodiesel (Mahlia et al., 2010).

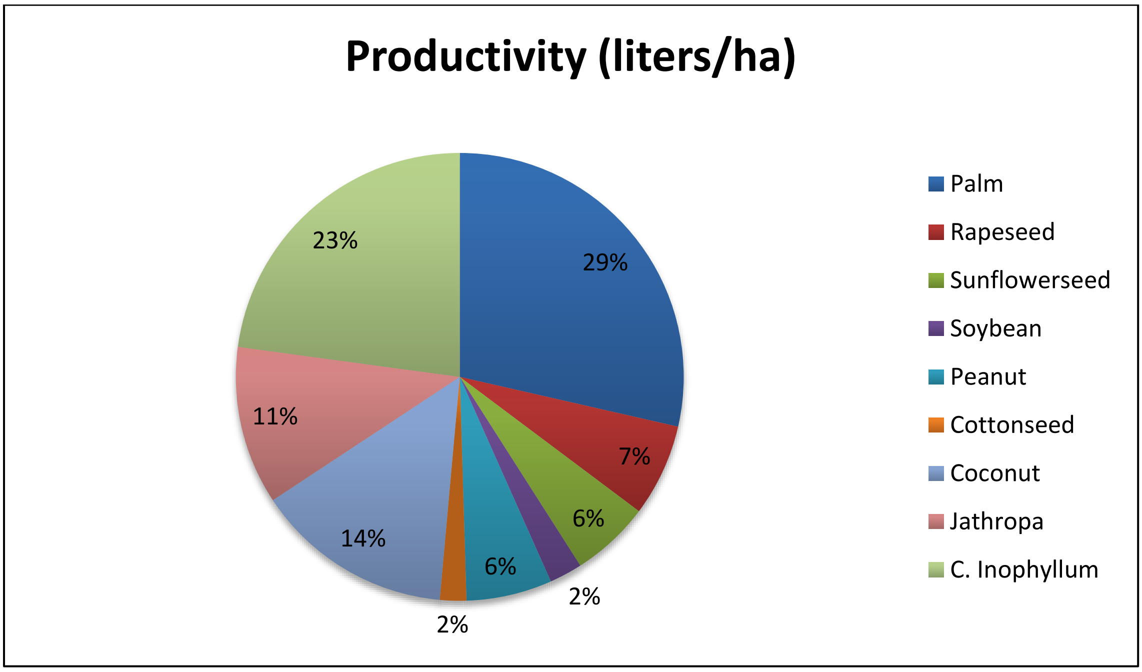

Figure 2.5: Production oil yield for various source biodiesel feed stocks

2.2.2 Palm Oil Processing

Carlsson (2009) reported that palm oil promises the most potential biodiesel feed stock compared to other oil seeds. Palm oil gives higher production yield, low fertilizer, water and pesticides for the plantation. Also, it takes less sunlight in terms of energy balance to produce a unit of oil as it produces more oil per hectare. Palm oil is known for its nutrient fact that makes it suitable as vegetable oil used for daily cooking.

In the process, the Fresh Fruit Bunches (FFB) will undergo some processes in order to separate the palm kernels and palm oil. FFB will be graded according to its ripeness (Mohd et al., 2011). Then, FFB will be transported by cages into the sterilizer to be exposed to steam. The main objective of the sterilisation is to inactive the oil splitting enzyme and to loosen the fruits in the bunch which will make it easier to be removed by shaking or tumbling in the threshing machine (Khalid & Sukaribin, 2009). Later, the fruitlets will be transferred into the digester by using conveyer. Empty fruit bunches will be transported to the empty bunch hopper and used as fertilisers.

In the digester, the palm oil will be released from the fruit by pounding at high temperature which helps to reduce the viscosity of the oil, destroy the *exocarp* and completes the disruption of the oil cells (Amelia et al., 2008). Thus, some palm oil is released and collected in the crude oil tank together with the pressed oil. Also, there is a pressing station to squeeze out the oil from the fibre and remove the nuts.

Nuts will undergo a nut polishing drum and a ripple mill to remove the shells from the kernels (Jekayinfa & Bamgboye, 2006). Kernels will be processed to palm kernel oil, meanwhile the crude palm oil is pumped to a vertical clarification tank for oil separation.

Figure 2.6 shows the simplified step by step process of palm oil transformed into crude palm oil which is then processed into other products including biodiesel (Mekhilef et al., 2010).

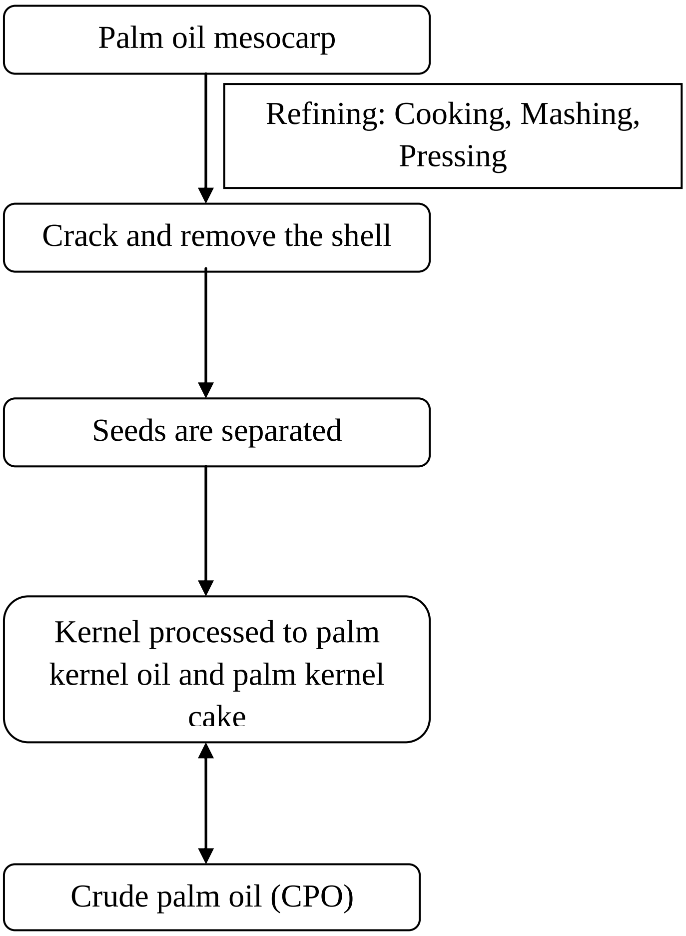

Figure 2.6: Palm plant (crude palm oil process)

The PO is being harvested from palm trees and transported to palm oil refinery to be refined, the palm oil is produced and this output can be converted into methyl ester and directly used as biodiesel. Another method is to blend the refined oil with petroleum diesel to make diesel fuel which is shown in Figure 2.7, this blending process is called Envo Diesel.

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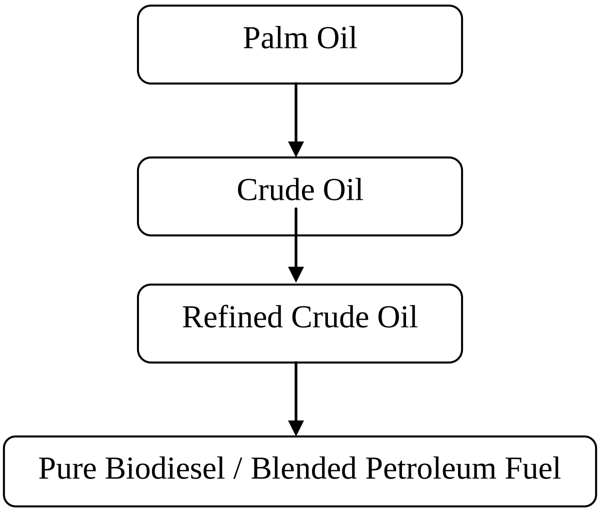


Figure 2.7: Palm oil to biodiesel process

2.2.3 Biodiesel by Transesterification

Yap et al., (2011) reported that transesterification or alcoholysis is the displacement of alcohol from an ester in a process similar to hydrolysis, except that alcohol is used instead of water. The reaction is one of the reversible reactions and proceeds essentially by mixing the reactants and represented by the following equation:

$$\text{Oil or Fat + Methanol} \longrightarrow \text{Methyl esters + Glycerol}$$

Many studies have shown that transesterification with methanol is more practical than with ethanol. Another advantage of using methanol is the separation of glycerine, in which can be obtained through simple decantation and can be used for different purposes (Nagi et al., 2008).

Meanwhile, Sarma & Chouhan (2011) reported that there are many types of catalysts that can be used for transesterification depending on the amount of free fatty acids in the oil. The alkalis include sodium hydroxide, potassium hydroxide, carbonates or corresponding sodium and potassium alkoxides such as sodium methoxide, sodium ethoxide, sodium propoxide and sodium butoxide. Sulphuric acid, sulfonic acids and hydrochloric acid are usually used as acidic catalysts, while enzymes as catalysts are limited in biodiesel production because of the fact that enzymes are more expensive and can lose some initial activity, and regeneration of enzymes are limited (Sagiroglu et al., 2011). Nowadays, the reaction under the conventional heating in presence of alkaline catalysts is the most common way to prepare biodiesel; all mentioned studies have been established on accelerating the chemical reaction and finally obtaining the higher reaction yields within a short time (Khoshandam et al., 2011). The production of biodiesel by transesterification of palm oil is as follows:

1. Mixing of alcohol and catalyst
2. Reaction of alcohol/catalyst with palm oil
3. Separation of glycerine and biodiesel
4. Removal of alcohol
5. Methyl ester washing
6. Biodiesel drying

Currently, most biodiesel is produced by using homogenous base catalyst, such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) (Felizardo et al., 2006 and Dalai & Kulkarni, 2006). These catalysts are commonly used for the ability to catalyze reaction at low reaction temperature and atmospheric pressure in addition; high conversion can be achieved in a minimal time (Lam et al., 2009 and Lotero et al., 2005).

In the alkali catalytic methanol transesterification method, the catalyst (KOH or NaOH) is dissolved in methanol by vigorous stirring in a small reactor. However, methanol is preferable because of its low cost and its physical and chemical advantages (Demirbas, 2005). The oil is transferred into the biodiesel reactor, and then, the catalyst/alcohol mixture is pumped into the oil (Demirbas, 2008b). Colucci et al. (2005) and Veljkovic et al. (2012) reported that methanol and ethanol gave higher biodiesel yields (99%) than 1-butanol (92%) or 2-propanol (29%).

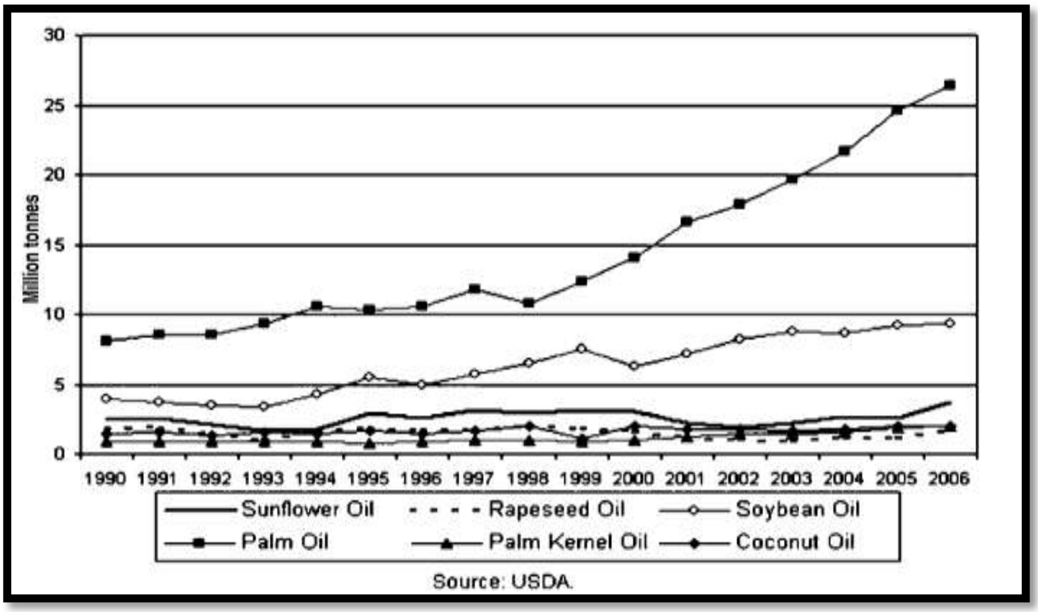
After the mixing reaction between methoxide and oil and settling for 24 hours, two layers are formed. Crude glycerine, the heavier liquid, will collect at the bottom after several hours of settling, preferably 24 hours (Demirbas, 2009). The glycerol phase which is separated after settling as bottom layer needs to be removed, and the methyl ester biodiesel phase (top layer) is evaporated with a thermostatic bath or rotary evaporator at 65 °C to remove the excess methanol (Miao et al., 2009). The products are washed several times with warm water to remove spent catalyst and by products of saponified reaction (Yusup & Khan, 2010). Ferella et al. (2010) reported that the water is dried off in an evaporator and a vacuum dryer, finally, the biodiesel is stored to be distributed and sold.

2.2.4 Palm Oil versus Other Feed Stock

As mentioned earlier, there are many possible feed stock can be considered as potential biodiesel raw stock for future prospects. One of them is *Jatropha*, which was identified as high potential raw stock having suitable criteria as palm oil replacement for biodiesel raw stock. Globally, *Jathropa Curcas* has created an interest for researchers because it is non-edible oil, does not create a food versus fuel conflict and can be used to produce biodiesel with same or better performance with blending biodiesel when testing in diesel engines (Silitonga et al., 2011).

The reason why many countries nowadays looking for other potential feed stock is that due to the competition on the use of palm oil as either food or fuel. World biodiesel producers are now focusing on using primary raw materials of non-food based in order to avoid competition of raw stock with food industry. Though, almost all biodiesel projects in Malaysia have been focusing on palm oil as raw material (Khalid & Khalid, 2011).

Mekhilef et al., (2010) reported that European United (EU) might ban biofuel derived from crops grown on some sensitive ecosystems. In the meantime Malaysia as the second largest palm oil producer after Indonesia, rely on these crops for biodiesel production. Countries like Brazil and US which produce biodiesel from ethanol are focusing on EU market, giving the heavy competition to palm oil biodiesel production. Figure 2.8 shows that palm oil is the top vegetable oil produced worldwide, followed by soybean oil and sunflower oil (Janaun & Ellis, 2009).



Source: USDA.

Figure 2.8: Global vegetable oil production

2.2.5 Advantages and Disadvantages of Biodiesel

The main target of biodiesel production is not to replace conventional diesels totally. However, its ultimate goal is to widen its usefulness, efficiency and value. As one of the various alternative fuels, biodiesel has a mark in the development and improvement of a balanced energy policy. Its contribution to sustainability and cleanliness of diesel engines has been one of the focuses of biodiesel production. Below are few advantages of palm oil as biodiesel:

i. Availability of palm oil feedstock

Fossil fuels are expensive and the growing global demand for fossil fuels has led to an increase in the price of petroleum products. This makes palm oil bio fuel a desirable product because it is cheaper than fossil fuels. Also it is shown in Table 2.2 that palm trees yield more oil per hectare as compared with other plant sources (Pahl, 2005):

Table 2.2: Yield of feed stocks for biodiesel production

Source	**Yield (liters/hectare)**	**Comparison of yields**
Palm	5950	1.00
Coconut	2689	0.45
Jathropa	1818	0.31
Rapeseed	1190	0.20
Soybean	446	0.07

ii. Renewable Resource

Biodiesel is derived from vegetable oils and these sources could always be re-planted or grown to ensure its sustainability. Moreover, unlike diesel, biodiesel feed stock is renewable and the use of environmental resources can potentially be sustainable (Sukkasi et al., 2010). A renewable fuel such as biodiesel, along the lesser exhaust emissions is the need of the present scenario worldwide (Demirbas, 2008b).

iii. Lower Emissions

It contributes to the reduction of the unburned hydrocarbons, carbon monoxide, sulphates, aromatic hydrocarbons emissions and emits less CO_2 than conventional fuels due to the low or zero content of pollutants such as sulphur (Bozbas, 2005).

iv. Biodegradability

Palm oil bio fuels are biodegradable, non-toxic and degrade about four times faster than petroleum diesel. Its oxygen content improves the biodegradation process, leading to an increased level of quick biodegradation (Yusuf et al., 2011). Other advantage of biodiesel as diesel fuel is high flash point which makes it safer to handle, store and transport (Shahid & Jamal, 2007).

v. Good lubricity

Biodiesel acts as a good lubricant to diesel engines (Shahid & Jamal, 2007). This could therefore prolong the shelf-life of the engines and it can be used to replace sulphur, a lubricating agent, that when burned, produces sulphur oxide, which is the primary component in acid rain (Nagi et al., 2008).

Meanwhile, palm oil also has its own disadvantages stated as below:

a) Weather

Palm oil is saturated oil. It has a better ignition quality but hardens at 54 °F making it impractical to use in cold weather. The challenge is that the use of biodiesel has faced during the last years is the pour point, which determines the feasibility for being used in cold climates, which limits its use only in tropical climates. However, according to Malaysian Palm Oil Board (MPOB) a technical procedure has been developed that has turned palm oil-based biodiesel into a more versatile product, achieving a pour point of -10°C during spring and fall seasons, 0°C in summer and -20°C in winter (Lozada et al., 2009).

b) Greenhouse Emissions

Important disadvantage of biodiesel is the increased emission of NO_X (Salic & Zelic, 2011). The emissions of NO_X may increase, especially at high engine loadings (Cheung et al., 2007).

c) Production cost

Taking into account the higher production costs of biodiesel compared to petroleum diesel, this increase in fuel consumption compounds the overall increased cost of application of biodiesel as an alternative to petroleum diesel (Yusuf et al., 2011).

Nevertheless, the advantages of biodiesel outmoded the disadvantages generally on the environmental aspects, making it very popular alternative to petroleum derived-diesel oil.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this chapter, the detailed description of the materials, equipments and methods was discussed.

a) Material
  - Raw Material.
  - Alcohol Selection.
  - Catalyst Selection.

b) Equipment
  - Hot plate with stirrer.
  - 1000 mL 2-necked round-bottom glass flask equipped with a reflux condenser and a thermometer.
  - 1000 mL separating funnel.
  - Rotary evaporator.

c) Research Method
  - Collecting Sample.
  - Performing Experiment.
  - Product Analysis.

3.2 MATERIAL

3.2.1 Raw Material

The raw material used in this research was crude palm oil (CPO).

3.2.2 Alcohol Selection

Price is the main factor in determining which alcohol as a solvent used in the production process. Ethanol (C_2H_5OH) and methanol (CH_3OH) are the two most common options. High quality methanol is cheaper than ethanol, and therefore, is used on nearly all biodiesel operations including in this research.

3.2.3 Catalyst Selection

A catalyst is required to facilitate the reaction between the oil and the alcohol. The most common catalysts used in small-scale biodiesel production are sodium hydroxide (NaOH) and potassium hydroxide (KOH). In conducting this experiment, potassium hydroxide was chosen because of easy-handling and dissolves faster in methanol.

3.2.4 Drying Agent

Magnesium sulphate ($MgSO_4$) is used as a drying agent to remove excess water from the raw crude palm oil because the presence of water will cause saponification in the product.

3.3 EQUIPMENT

To achieve the production of biodiesel through base-catalysed production, few types of equipment are required in this experiment:

a. **Hot plate with stirrer**: To warm up the oil and heat water for washing

b. **Two-necked round-bottom glass flask with reflux condenser**: To heat and stir the mixtures of methoxide and oil

c. **Separating funnel**: To separate two layers of glycerine and biodiesel

d. **Rotary evaporator**: To recover excess methanol

3.4 RESEARCH METHOD

3.4.1 Collecting Sample

The crude palm oil used in this study was collected from Kilang Sawit Lepar Hilir 3 and Kilang Sawit Dominion Square at Jalan Gambang-Kuantan, Pahang Darul Makmur.

3.4.2 Preparation of Samples

In the laboratory, three sets of different ratios of methanol to oil were prepared. The ratios of oil to methanol were:-

i. 4:1 (200 mL : 50 mL)
ii. 6:1 (300 mL : 50 mL)
iii. 8:1 (400 mL : 50 mL)

In order to have a 1.0 % concentration of KOH for every 50 mL of oil used, 0.5 gram of potassium hydroxide (KOH) pellets was added in the methanol to produce methoxide solutions. These solutions were prepared in a volumetric flask. The flask was tipped back and forth until KOH was dissolved.

3.4.3 Experiment

Before the experiment started, a certain amount of magnesium sulphate is added in the raw crude palm oil to remove excess water. The raw product is left for a day and the excess water is filtered from the CPO. Nine experiments for each set of parameters with a total of 27 experiments are done according to the table below:

Table 3.1: Sets of samples and trials

Ratios (methoxide:oil)	**Temperature (°C)**	**Time (min)**
4:1	40	40
		60
		80
	50	40
		60
		80
	60	40
		60
		80
6:1	40	40
		60
		80
	50	40
		60
		80
	60	40
		60
		80
8:1	40	40
		60
		80
	60	40
		60
		80
	60	40
		60
		80

1) Reaction of methoxide with palm oil

A sample of 50 mL oil in the beaker was pre-heated up to 60 °C on the hotplate. A higher reaction temperature can decrease the viscosities of oils and result in an increased reaction rate, and a shortened reaction time. Depending on the oil used, the optimal temperature ranged from 50 °C to 60 °C (Leung et al., 2010). Then, the oil was poured into the 2-necked round-bottom glass flask. Carefully, the methoxide was poured into the oil according to the ratio selected in Table 3.1. Stirring speed was set up at 250 rpm. It increased the intact area between oils and potassium hydroxide methanol solution. Without the mixing, the reaction occurred only at the interface of the two layers and considered too slow to be feasible (Rashid & Anwar, 2008).

2) Separation of glycerine and biodiesel

As soon as the transesterification process was completed, the hot mixture from 2-necked round-bottom glass flask was poured into the separator funnel for settling. The products which were palm oil methyl ester (POME) and glycerine were settled for at least 12 hours (Predojevic, 2008; Issariyakul et al., 2007).

3) Removal of methanol

As soon as the separation of POME and glycerine was done, the POME was heated in a flask placed in a rotary evaporator. The temperature and the speed were set up at 70 °C and 60 rpm. Methanol was evaporated and condensed in the centre tube of a condenser and collected.

4) Methyl ester washing

The methyl ester produced from the reaction was washed with 70 °C hot tap water. The water was poured in carefully and gently to avoid emulsifying.

5) Biodiesel drying

In order to remove impurities and excess water, the product was transferred into an open container and heated in the oven for 15 minutes at 50 °C.

3.4.4 Product Analysis

After calculating the percentage yield for each sample, the biodiesel was prepared with a volume of 500 mL at the optimum parameters to run the biodiesel tests. The following testings were:

1) Density at 15 °C (kg/m^3)
2) Kinematic viscosity at 40 °C (mm^2/s)
3) Cetane number
4) Flash point (°C)
5) Cloud point (°C)
6) Pour point (°C)
7) Saponification value (mg/L)

3.5 SUMMARY

This chapter discussed the steps of preparing the samples, the parameters that we used to determine the best biodiesel yield, optimum ratio of methanol:oil and physical characteristics of the biodiesel produced.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter presents all the results obtained with best biodiesel yield, optimum reaction temperature, reaction time, ratio of methanol:oil and physical characteristics of biodiesel produced by using the method described in Chapter 3. The tests and analysis is done to the highest biodiesel yield product. The main aim is to find the physical characteristics of the biodiesel produced from palm oil.

4.2 BIODIESEL YIELDS

In this study, the parameters that are taken into consideration are reaction time, reaction temperature and methanol:oil ratio. The agitation speed was set at 250 rpm. Table 4.1 below shows the percentage yields for all samples at different parameters.

Table 4.1: Biodiesel yield

Ratio (methoxide:oil)	**Temperature (°C)**	**Time (min)**	**Total Product (mL)**	**Yield (%)**
4:1	40	40	5	10
		60	21	42
		80	24	48
	50	40	20	40
		60	28	56
		80	38	76
	60	40	32	64
		60	35	70
		80	39	78
6:1	40	40	9	18
		60	23	45
		80	30	60
	50	40	24	48
		60	35	70
		80	40	80
	60	40	35	60
		60	44	88
		80	43	86
8:1	40	40	11	22
		60	28	56
		80	29	58
	50	40	28	55
		60	37	74
		80	40	82
	60	40	29	58
		60	43	86
		80	41	88

4.2.1 Yield Comparisons

The comparisons between the yields can be seen in the graphs below. The graphs are characterized based on the ratios and temperatures.

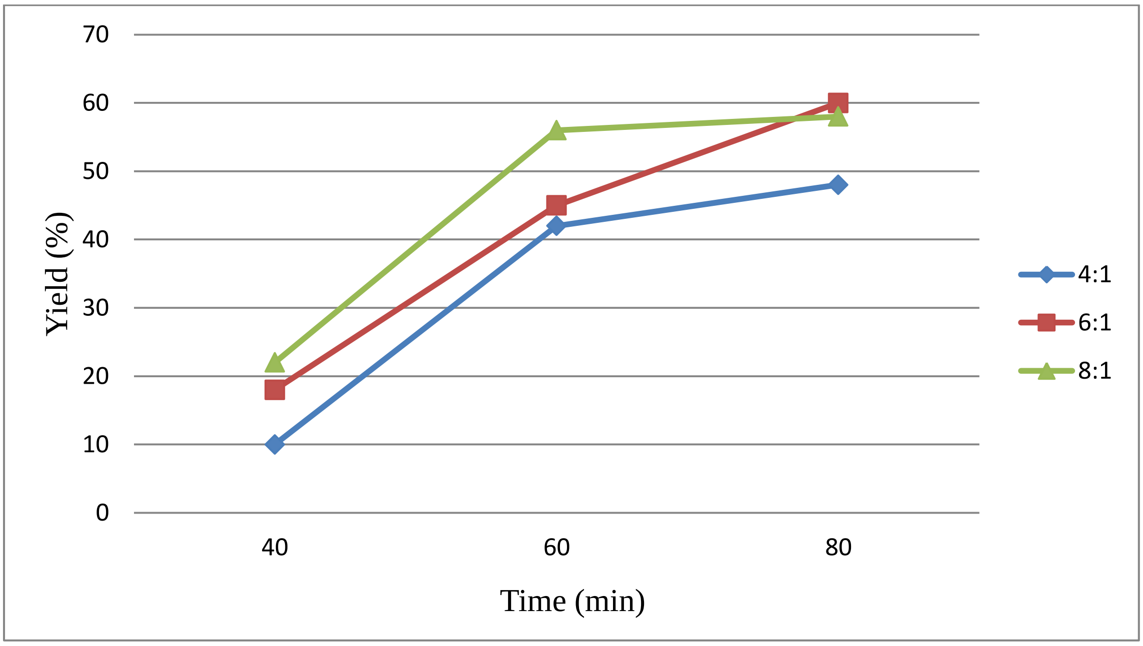


Figure 4.1: Comparisons of yields at 40 °C for different ratios and time

Figure 4.1 shows that as the time and methoxide ratios are increasing, the biodiesel yield is increasing too. Same pattern with Figure 4.2 which shows the increasing of biodiesel yields as the time and methoxide ratios is increasing.

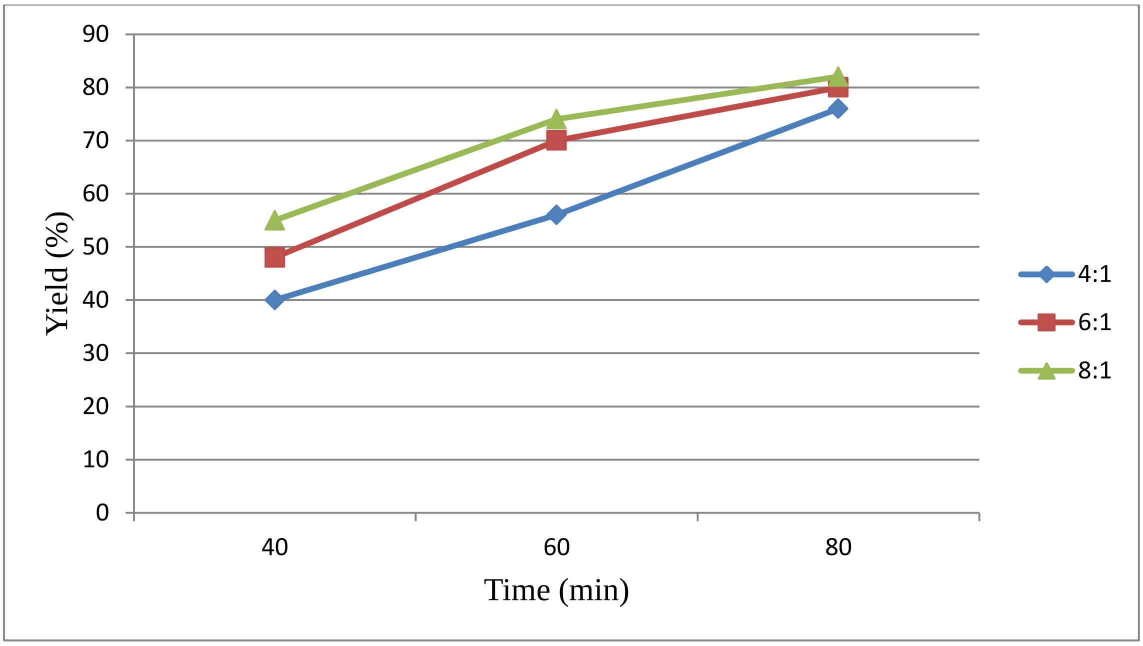


Figure 4.2: Comparisons of yields at 50 °C for different ratios and time

Meanwhile, Figure 4.3 shows different pattern from Figure 4.1 and Figure 4.2. There are two points of optimum yields, which are at 60 minutes and 80 minutes for the ratios 6:1 and 8:1.

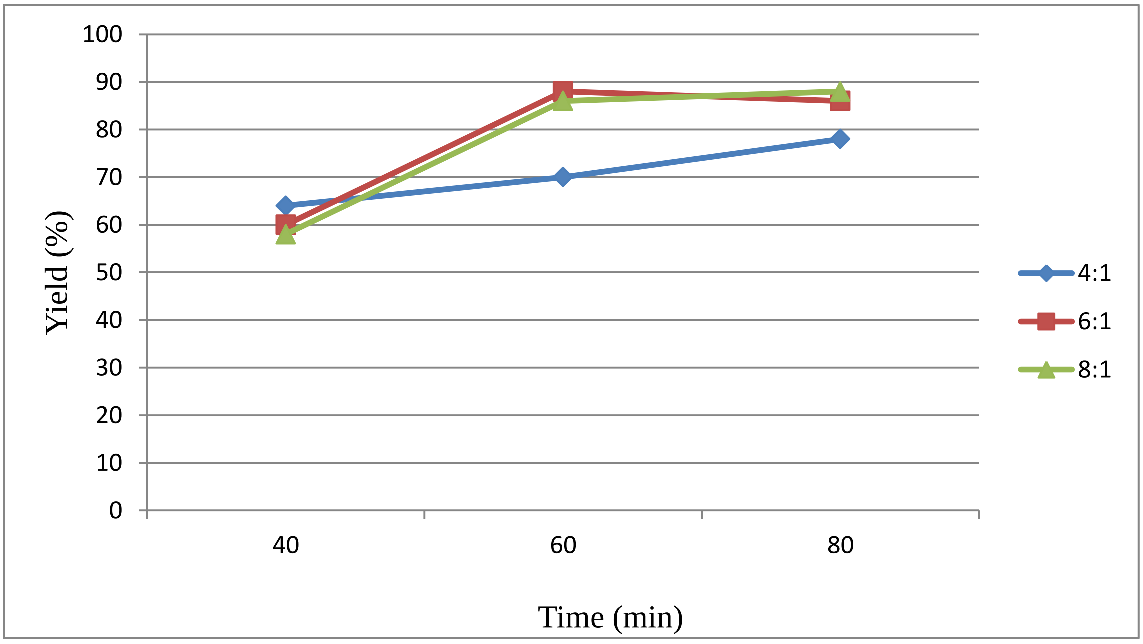


Figure 4.3: Comparisons of yields at 60 °C at different ratios and time

From Table 4.1 and Figure 4.1 until Figure 4.3 above, it can be concluded the parameters that yielded the optimum biodiesel are:

i. Ratio : 6:1
Temperature : 60 °C
Time : 60 minutes
ii. Ratio : 8:1
Temperature : 60 °C
Time : 80 minutes

The yield of 88% was achieved by using both parameters above but for feasibility reasons, the first parameters were chosen as optimum because it is cost effective regarding time and chemical consumption.

4.3 OPTIMUM BIODIESEL YIELD TEST

4.3.1 Optimum Biodiesel Yield Preparation

According to the results obtained in Table 4.1, it was decided to choose at methoxide:oil ratio of 6:1, time of 60 minutes, temperature of 60 °C and around 500 mL of biodiesel was prepared and homogenised to be used in the physical properties tests.

4.3.2 Physical Characteristics Tests and Analysis

From the experiment, the final yield for optimum biodiesel of 88% was chosen. Table 4.2 shows the physical properties tested on the final biodiesel sample. The ASTM and European Standards are based on (Man et.al, 2012).

Table 4.2: Physical properties analysis

Analysis	**Experiment**	**ASTM D6751 Standard**	**European Standard**
Density (kg/m^3) at 15 °C	876.0	870-900	860-900
Kinematic viscosity (mm^2/s)	4.76	1.9-6.0	3.5-5.0
Cetane number	62.8	< 47	< 51
Flash point, °C	170	130	120
Cloud point, °C	13	-	-
Pour point, °C	17	-	-
Saponification value (mg/L)	206.95	-	-

From Table 4.2, it is shown that the values from the experimental data are within the biodiesel standards which are the American Society for Testing and Materials (ASTM D6751) and European Standards. The tests were carried out for biodiesel as follow:

4.3.2.1 Density (ρ) Test, kg/m^3

In order to determine the density of the biodiesel produced, the equipment used was the Density Tester at 15 °C. The chemical needed was 99 % purity ethanol and the biodiesel sample used was 20 mL.

Steps to determine the density of the optimum biodiesel were as follow:-

1. The inner tube of the density tester was cleaned by suction of ethanol into the tube. This step had to be repeated few times to remove excess impurities and other mixing solutions.

2. The biodiesel sample was pumped into the density tester until it fully occupied the inner tube. Ensure that there were no bubbles inside the tube to avoid inaccurate reading.

3. Step no. 2 was repeated for three times to get the average value.

4.3.2.2 Kinematic Viscosity

Kinematic viscosity measures the ease with which a fluid will flow under force. Kinematic viscosity is traditionally measured by noting the time it takes oil to travel through the orifice of a capillary under the force of gravity.

In order to determine the kinematic viscosity of the produced biodiesel, various glass capillary tubes needed to be prepared. In this research, viscosity number of No. 200 was selected depending on the ASTM Standard. The kinematic viscosity equipment used was Cole-Parmer, made from United States of America. A sample of 30 mL biodiesel was pumped into the capillary tubes until it reached the ‘start mark’. The time taken for the oil to flow through the capillary tube can be converted directly to a kinematic viscosity using a simple calibration constant provided for each tube. The experiment was repeated three times to get accurate calibration.

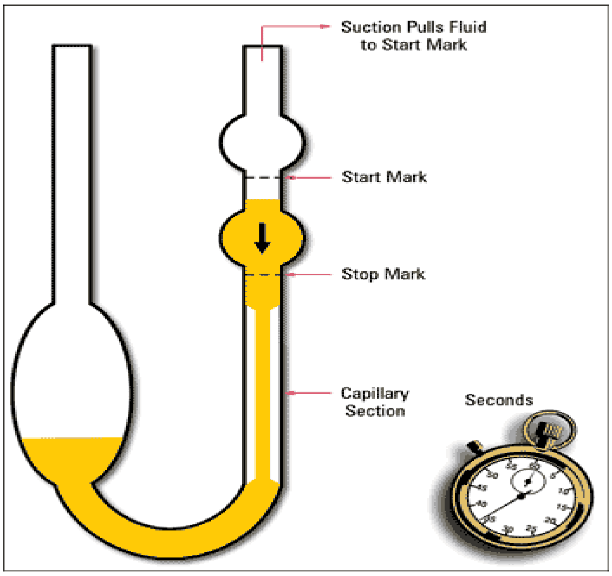


Figure 4.4: Example of capillary tube

4.3.2.3 *Cetane* Number

It is a dimensionless measure of the ignition performance of a diesel fuel. In order to determine *cetane* number of palm biodiesel, a sample of 50 mL was needed. The sample was poured slowly into the *cetane* tester until it indicated label ‘S’ which means cetane number was determined based on summer season. Once the indicator displayed ‘S’, the cetane number was recorded. The experiment was repeated three times to get the average value.

Figure 4.5: Example of *cetane* number tester

According to Department of Environment and Heritage, reported that *cetane* number is a measure of the readiness of a fuel to auto-ignite when injected into a diesel engine. It relates to the delay between the moments a fuel is injected into the cylinder till the ignition occurs. According to (Karmakar et al., 2010), in case of palm biodiesel, the *cetane* number is very high because of the presence of saturated fatty acids such as palmitic (C16:0) an (Karmakar, Karmakar, & Mukherjee, 2010)d stearic (C18:0) acid.

4.3.1.4 Flash Point

Biodiesel in general has a very high flash point of 150 °C or more. The reason why palm biodiesel has high flash point is due to the longer methyl ester chains.

Figure 4.6: Pensky-Marten Closed Cup Tester

In flash point analysis, Pensky-Martens Closed Cup Flash Tester (Company: DKSH Technology Sdn. Bhd, Selangor Darul Ehsan, Malaysia) was used and a 50 mL sample of optimum yield biodiesel was prepared and poured into the small aluminium-based tank. Then the sample was placed onto the flash point tester with a thermometer attached on top of it. The lid was closed tightly, the equipment was turned on and the temperature was observed until it reached more than 100 °C. Then slowly, the oil was ignited until small spark was observed. Once the spark was noticeable, the flash point was recorded.

4.3.2.5 Cloud Point and Pour Point

The cloud point is the temperature of the fuel at which, small solid crystals can be observed as the fuel cooled. Palm oil biodiesel is typically associated with high cloud and pour point limiting its usage just in warmer climate (Janaun & Ellis, 2009).

The sample of 50 mL biodiesel was poured into the test tube until it reached the level indicated on the test tube. Then, the sample was placed inside a refrigerator. The sample had to be observed regularly for every one minute until solid-crystals appeared. The temperature at which first observation of solid-crystals appeared was the cloud point at 13 °C.

After solid-crystals appeared, the sample was placed in a room temperature. Once the temperature increased and the sample started to melt, the melting temperature was considered as the pour point at 17°C.

4.3.2.6 Saponification Value

Saponification value is the measurement of potassium hydroxide in mg required to saponify one gram of fat. The equipment used was Saponification Number Tester (Brand: Metrohm, Made from: Herisau, Switzerland). A sample of 2.0 g of biodiesel was delivered into the 50 mL conical flask. An amount of 25 mL of 0.5 mol/L potassium hydroxide ethanol was added into the flask and a cooling pipe was fixed to the flask. The mixture was heated slowly by controlling the heater for 30 minutes. After the mixture was heated, the flask was cooled and the mixture was titrated with 0.5 mol/L hydrochloric acid until the liquid was solidified. The saponification takes place at 206.95 mg/L.

The above procedures were automatically performed by the saponification number tester itself including the calibrations and the amount of titration. However, in this test, the electrode was rinsed regularly to avoid drying and inaccurate reading.

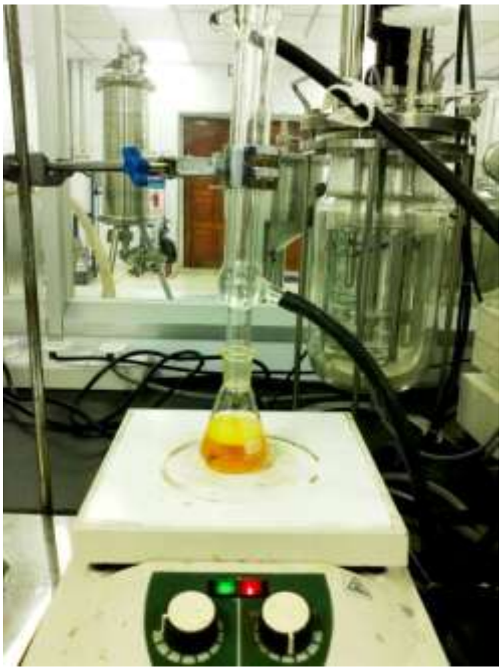


Figure 4.7: Heating of the sample and KOH/EtOH

Figure 4.8: Saponification tester

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSIONS

This research showed the possibility of producing biodiesel from palm oil with specification of density, kinematic viscosity, *cetane* number, flash point, cloud point, pour point and saponification value.

The optimum biodiesel yield from the research was 88 % at methoxide:oil ratio of 6:1, time of 60 minutes and temperature of 60 °C. The physical characteristics obtained from the final optimum biodiesel yield are concluded in Table 5:1.

Table 5.1: Physical properties of palm biodiesel

Analysis	**Experiment**
Ratio (MetOH:Oil)	6:1
Temperature, °C	60
Time, min	60
Density, kg/m^3	876.0
Kinematic viscosity, mm^2/s	4.76
Cetane number	62.8
Flash point, °C	170
Cloud point, °C	13
Pour point, °C	17
Saponification value, mg/L	206.95

The physical properties of the produced biodiesel from CPO were shown in Table 4.4 and compared to ASTM D6751 and European Standards, which shows that the properties are accepted within the standards.

5.2 RECOMMENDATIONS

There are few recommendations needed to improve this research in order to get better results and outcomes:-

i. The by-product glycerol can be used for further studies such as in cosmetics industries;

ii. Further study can be done by using mixer with higher temperature than 80 °C to decide if it gives better yield;

iii. The fatty acids and heat content of the biodiesel produced must be determined to support the results for this research;

iv. Carry out the study by using other raw materials such as *Moringa oleifera* and Castor oil.

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APPENDICES

APPENDIX A.1
PHYSICAL PROPERTIES ANALYSIS

Appendix A.1.1: Density of palm biodiesel, ρ

Readings	**(Unit) kg/m^3**
Reading 1	876.0
Reading 2	875.0
Reading 3	876.0
AVERAGE	**876.0**

Appendix A.1.2: *Cetane* number of palm biodiesel

Readings	**(Unit) °C**
Reading 1	62.8
Reading 2	62.0
Reading 3	63.5
AVERAGE	**62.8**

Appendix A.1.3: Cloud point of palm biodiesel

Readings	**(Unit) °C**
Reading 1	13
Reading 2	13
Reading 3	13
AVERAGE	**13**

Appendix A.1.4: Pour point of palm biodiesel

Readings	**(Unit) °C**
Reading 1	17
Reading 2	16
Reading 3	17
AVERAGE	**17**

Appendix A.1.5: Saponification number of palm biodiesel

Readings	**(Unit) mg**

Reading 1	200.98
Reading 2	200.10
Reading 3	219.76
AVERAGE	**206.95**

Appendix A.1.6: Kinematic viscosity of palm biodiesel, *v*

Readings	**(Unit) s**
Reading 1	44
Reading 2	43
Reading 3	43
AVERAGE	**43.33**

Calculation for kinematic viscosity:

Given:

Viscometer No : 200

Viscosity constant : 0.1098 mm^2/s^2

= Efflux time (s) *x* viscosity constant (mm^2/s^2)

= 43.33 *x* 0.1098

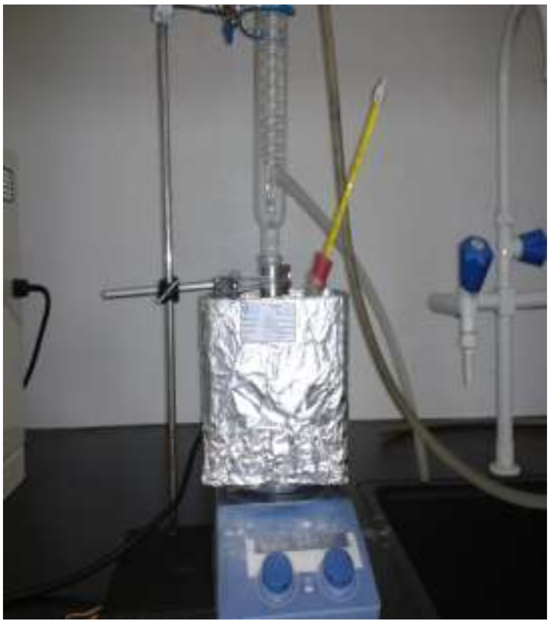
= 4.76 mm^2/s

APPENDIX A.2

PRODUCTION PROCESSES

Appendix A.2.1: Addition of drying agent to remove excess water and impurities

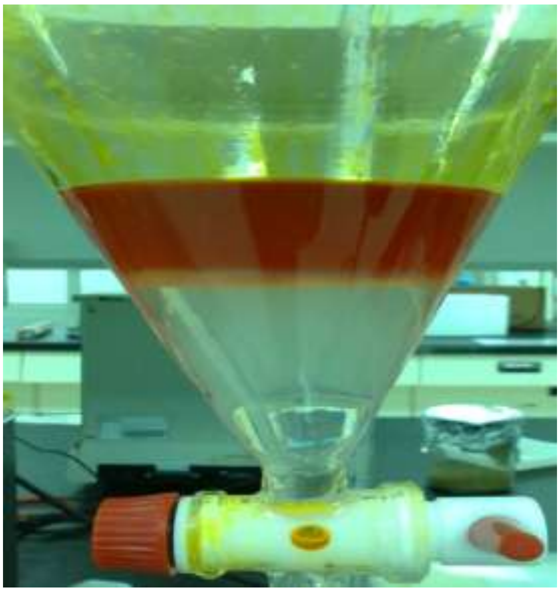
Appendix A.2.2: Mixing of alcohol and catalyst to produce methoxide



Appendix A.2.3: Mixing of methoxide and palm oil using reflux condenser

Appendix A.2.4: POME-Glycerin 24 hours settling

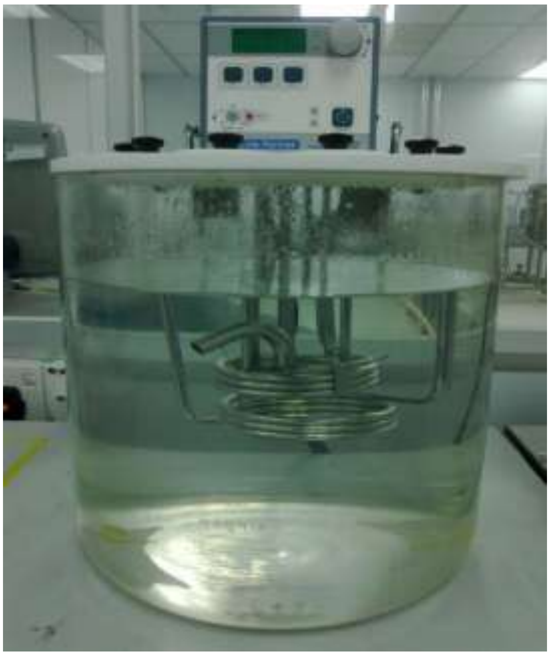
Appendix A.2.5: Water bath rotary evaporator



Appendix A.2.6: Biodiesel washing

Appendix A.2.7: Biodiesel drying to remove excess water

APPENDIX A.3
ANALYSIS EQUIPMENTS



Appendix A.3.1: Kinematic viscosity tester

Appendix A.3.2: Flash point tester

Appendix A.3.3: Heating of palm BD for saponification

Appendix A.3.4: Saponification tester