

**COMPARISON OF BIODIESEL PROPERTIES
PRODUCED FROM
MORINGA OLEIFERA OIL AND PALM OIL.**

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COMPARISON OF BIODIESEL PROPERTIES PRODUCED FROM MORINGA OLEIFERA OIL AND PALM OIL.

AHMAD ADZHIM BIN ABDULL AZIZ

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Gas Technology)

**Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG**

JUNE 2014

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We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering (Gas Technology).

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I hereby declare that the work in this thesis 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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Dedication

Special Dedication for both of my parents:

Abdull Aziz bin Alias

Sharifah Khasidah binti Syed Ali Assegaf

Your continuous support and love is much appreciated

Warmest appreciation goes to:

Dr Eman N. Ali

Your guidance and assistant indeed become a great debt to me

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ABSTRACT

Continuous exploration of fossil fuel had diminished the reserve and turned the valuable non-renewable fuel into scarce. Even worse, the effect of fossil fuel combustion and volatile price had urged the scientific community to find a better substitute to a declining fossil fuel. Years of efforts had shed some light into a better solution of producing fuel from biomass source such as vegetable oils. The *Moringa oleifera* oil was extracted using different solvent in soxhelt extractor. The biodiesel was produced from crude palm oil and extracted *Moringa oleifera* oil through transesterification process using methanol and potassium hydroxide as catalyst. As a conclusion, n-Hexane was the best solvent which yielded the highest oil with an average percentage of 34.3%. The biodiesel properties of *Moringa oleifera* oil and palm oil such as cetane number were 66.7 and 59.5, kinematic viscosity were 4.8mm²/s and 5.03 mm²/s, cloud point were 18°C and 16°C, pour point 12°C and 7°C, flash point 165 and 160, and density 875kg/m³ and 890kg/m³. Biodiesel produced from *Moringa oleifera* had a potential to become a better feedstock as it possess a better properties which compare to biodiesel produce from palm oil.

ABSTRAK

Penerokaan petroleum yang berterusan menyebabkan simpanan bahan bakar tersebut berkurangan. Kesan buruk pembakaran dan harga bahan bakar tersebut yang tidak stabil telah memaksa para saintis untuk mencari penyelesaian terbaik kepada masalah ini. Selepas bertahun penyelidikan, biodiesel yang dihasilkan dari sumber yang boleh diperbaharui ialah jawapan terbaik terhadap kemelut tenaga yang semakin meruncing. Minyak *Moringa oleifera* diekstrak menggunakan tiga pelarut berbeza didalam Pemerah Soxhelt. Biodiesel dihasilkan melalui proses transesterifikasi dengan kehadiran metanol dan mangan. Kesimpulanya, n-Heksana ialah pelarut terbaik kerana menghasilkan minyak yang terbanyak dengan purata 34.3%. Ciri-ciri fizikal biodiesel dari minyak *Moringa oleifera* dan minyak kelapa sawit seperti nombor setana adalah 66.7 dan 59.5, kelikatan kinematic adalah 4.8mm²/s dan 5.03 mm²/s, *cloud point* adalah 18°C dan 16°C, *pour point* adalah 12°C dan 7°C, flash point adalah 165°C and 160°C, dan ketumpatan 875kg/m³ dan 890kg/m³. Biodiesel yang dihasilkan dari *Moringa oleifera* mempunyai potensi untuk menjadi bahan mentah yang lebih baik kerana ia mempunyai ciri-ciri yang lebih baik berbanding dengan biodiesel yang dihasilkan daripada minyak sawit.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
CP	Cloud point
CFPP	Cold filter plugging point
CPO	Crude palm oil
CMOO	Crude <i>Moringa oleifera</i> oil
FA	Fatty Acid
FAME	Fatty acid methyl ester
FP	Flash Point
GHG	Green House Gas
MOME	<i>Moringa oleifera</i> methyl ester
POME	Palm oil methyl ester
PP	Pour point
rpm	round per minute

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1 INTRODUCTION

1.1 Background of study

The world's fossil fuel reserve is diminishing and become scarce (Demirbas, 2007) due to decades of continuous finding pockets of this valuable so called black gold or fossil fuel, pumping them dry and moving on to new finding. As this trend continues and couple with the imbalance of fossil fuel consumption and production, shrink the amount of non-renewable fuel reserve from undiscovered oil well, make it even harder to discover and more expensive to produce (Harper *et al*, 2009). Furthermore, when petroleum fuels are burned, lots of harmful gases such as carbon dioxide will be released into the atmosphere and pose adverse effect to environment and human health. This is because carbon dioxide is a gas that absorb heat. Sulfur dioxide also a side product which will be released from the fossil fuel combustion. When sulfur dioxide react with water in the atmosphere, a sulfuric acid will be formed. This sulfuric acid will lead to acid rain which will alter the natural pH value of soil and give a bad effect to the agriculture sector (Demshemino, 2013). If there is no concrete prevention efforts taken to alleviate above-mentioned threat, it is predicted that greenhouse gas (GHG) emissions from fossil fuels will increase by 39% in 2030 (Mofijur *et al*, 2014a)

This current scenario coupled with negative effect from the intense use of fossil fuel had triggered scientific community to carry out a scientific researches to seek the best alternative fuel source (Lim and Teong, 2010; Jayed *et al.*, 2011 and Atabani *et al.*, 2012) to cope with the problem and meet the ever increase fuel demand by reducing the dependence on petroleum derived fuel and shifting to renewable biofuel that are available, technically feasible, economically viable, and environmentally acceptable (Liaquat *et al*, 2010) which possess a characteristics of lower emissions of carbon monoxide, particulate matter, and total hydrocarbons which would help to reduce the air pollution. After years of scientific research, the effort has paid off as the scientific community successfully developed a new kind of fuel. It is accepted in many countries as an extender or substitute for fossil based diesel. Besides possesses physiochemical properties very similar to those of petroleum-based diesel (Ávila and Sodré, 2012, Amani *et al.*, 2013 and Thomas *et al.*, 2013), this new kind of biofuel having the advantages of being biodegradable, renewable, possesses inherent lubricity, relatively high flash point, 10 times less poisonous compared to the ordinary diesel oil, the waste product is

not black, less sulphur and other aromatic contents, hence the combustion emission produced is safe for environment and perform less accumulation of carbon dioxide gas in atmosphere thus lessen furthermore global heating effect, and it can be used easily because it can be mixed at any proportion with diesel oil, hence enabling us to apply it immediately for diesel engines that are available without much modification (Trakarnpruk and Chuayplod, 2012 ; Abdullah *et al.*, 2009; Christian *et al.*, 2009; Sérgio and Graciela, 2006; Lilian *et al.*, 2008; Haseeb *et al.*, 2011; Cumali *et al.*, 2011).

Biofuels is really an umbrella term and it can mean almost anything, from hydroelectric power, which is generated from waves to wind, solar and other forms of generating energy. However, for the most part the term biofuels is used to refer to that of alternative substitutes for petrol, diesel or aircraft fuel. Biofuel can be explained as transportation liquid or gaseous fuels that predominantly produced from biomass (Demirbas, 2006). Biofuels are a viable alternative to fossil fuels. Many varieties exist and they vary significantly. Some examples are that of biodiesel, which entails growing crops that contain high amounts of natural oil then through a process of hydrogenation or refining a more compatible biodiesel, substitute is created. This creates a biodiesel, which can be mixed with mineral diesel or use on its own then used in any diesel-powered automobile.

Biodiesel is an animal fat or vegetable oil based diesel fuel that burns without the emission of much soot, carbon IV oxide and particulate matter. (Oliveira and Da Silva, 2013). Biodiesel can be produced from numerous primary feed stocks of either vegetable oils or animal fats such as canola (rapeseed) oil, tobacco oil, jatropha oil, cottonseed oil, sunflower oil, soybean oil, palm oil, peanut oil, rubber seed oil, as well as variety of less common oil. Above mentioned primary feed stocks are allowed to react chemically with an alcohol and the most commonly use alcohol is methyl alcohol with the present of catalyst which usually a strong base such as sodium or potassium hydroxide. This chemical reaction will yield a new chemical compound called methyl ester or popularly known by people as biodiesel (Gerpen, 2005).

1.2 Botanical Description of *Moringa oleifera*

Although there is variety of choices of feed stocks for the production biodiesel but only two will be focused in this work which are *Moringa oleifera* seed oil and palm oil. *Moringa oleifera* seeds can be obtained from a tree with a cluster of small oval leaves and delicately perfumed cream-coloured flowers called *Moringa oleifera*. It is also called Miracle tree or Tree of life

(Sue Nelson and Marlene Rau, 2011). *Moringa oleifera* is one of the species in monogeneric family of shrubs and tree, moringaceae (Dalziel, 1995). It is native to the foothills of Himalayas of northern India (Patty Donovan, 2007) and nowadays it is cultivated well beyond its native range throughout South Asia, Arabian Peninsula, tropical Africa, central America, the Caribbean and tropical South America (Roloff et al, 2009).



Figure 1: a) Ripe *Moringa oleifera* pods b) Inside the pod c) *Moringa oleifera* seeds with shell d) *Moringa oleifera* kernels

A highly valued plant and fast-growing *Moringa oleifera* able to tolerate with a wide range of environmental conditions. It can resist a light frost and best grows between 25 to 35°C but able to tolerate up to 48°C in the shade. The drought-tolerant tree also able to adapt in poor soil condition, receive wide range of rainfall amounts of 25cm to 150cm, adaptable best below 600m altitudes and soil pH of 5.0–9.0 (Palada and Changl, 2003). Oil extracted from *Moringa oleifera* can be used as raw material to produce biodiesel just like palm oil. Bark, roots, seeds, flowers and leaves of this tree can be used for various purposes such as treat various ailments, combating malnutrition, culinary, production of cosmetic, soap and many more but besides that it also can be used to produce biodiesel (Ramachandran et al, 1980).

Oil extracted from *Moringa oleifera* seeds concentrate between 35-45 % (w/w) of vegetable oil (Ricardo, 2012) known as “Behen” or “Ben” oil from its high concentration of behenic acid (Abiodun, 2012) possesses significant resistance to oxidative degradation (Stavros Lalas and John Tsaknis, 2002). Fatty acids composition in *Moringa oleifera* resembles olive oil and it is rich in palmetic, stearic, behmic, and oleic acids which is ideal for edible purpose (García-Fayos et al, 2010). Based on comparative analysis using smells and odours electronic detection device or eNose, oil extracted from *Moringa oleifera* also possesses a pleasant peanut-like odour and pale yellow in colour (Abdulkarim et al, 2005)..

1.3 Motivation

Unlike any other primary feed stocks, oil extracted from *Moringa oleifera* seeds have high cetane number which is more than 60 (Trakarnpruk and Chuayplod, 2012). The high cetane number in the oil will ensure a proper solid ignition. Furthermore, a high rating of cetane number would help to reduce a carbon release footprint, improved fuel efficiency and reduce tear and wear for both vehicle starter and batteries (Masina et al, 2012).

As compared to other feed stocks especially palm oil, oil derived *Moringa oleifera* seeds has a better oxidative stability. The price of the *Moringa oleifera* crude oils is comparable to soybean crude oil and palm oil. From economical view, *Moringa oleifera* can be planted with a less water, less nutrient and improper soil condition and this will lead to cheaper cost to cultivate (Masina et al, 2012).

Moreover, the production of biodiesel from *Moringa oleifera* will reduce the demand for the palm oil for biodiesel production thus reduce the price and pressure on food industry to meet the ever skyrocketing demand for palm oil especially during festive seasons. The plantation of these trees can potentially increase green coverage to sequester more CO₂ than other vegetable oil crops. *Moringa oleifera* can withstand drought, it is able to produce seeds in the first year and harvest up to four times in a year (Wahidul and Michele, 2009)

1.4 Problem statement

Nowadays, fatty acid methyl ester (FAME) or popularly known as biodiesel which is derived from various vegetable and animal fats has been hailed as the potential substitute to replace petroleum-based diesel and possible solution for the shortage of transportation fuel and to

reduce the greenhouse effects and pollution caused by conventional diesel. The substitution to tryglyceride-based biodiesel is due to similarity of energy content, viscosity, cetane number and phase changes of biodiesel to those of petroleum-derived diesel fuel (Yee and Lee, 2008). Moreover, the usage of biodiesel from renewable oil sources have the benefits of lower sulfur content, lower aromatic content, higher heat content, biodegradability, renewability, ready availability and liquid nature-portability (Demirbas, 2005).

On the other hand, the production of biodiesel from vegetable oils has triggered a new problem of shortage vegetable oil for human daily consumption as the most abundant vegetable oil in the region is the most common feedstock. For example in Malaysia, palm oil is the feedstock to produce biodiesel and in the same time it also being used to produce foods (Knothe et al, 2005). The increasing usage of palm oil for biodiesel production raised tension for palm oil producer to meet the ever-increase demand and has crop-up a new problem of food production, price and availability (Torrey, 2007). Therefore, the study to seek the best feedstock must be carried out in order to solve fore mentioned problem. Different usage of solvent in oil extraction process might affect the oil recovery or oil yield. Normally, n-Hexane is used in Solvent Extraction which yields about 38.40% but the use of ethanol might increase the oil yield (Sajid Latif and Farooq Anwar, 2008). Therefore, the use of different solvent in the oil extraction is vital in order to improve the oil yield.

1.5 Objectives

The following are the objectives of this research:

- i. To produce an environmental friendly biodiesel from palm oil and extracted *Moringa oleifera* oil.
- ii. To study the effect of different solvent in oil extraction process.
- iii. To compare biodiesel properties produced from *Moringa oleifera* and palm oil.

1.6 Scope of this research

The following are the scope of this research:

- i. The *Moringa oleifera* oil will be extracted through solvent extraction process using Soxhelt Extractor. Ethanol, methanol and n-hexane will be used as solvent. The purpose of using different solvents is to study the amount of yield from different solvent.

- ii. Palm oil and extracted *Moringa oleifera* oil will be converted into biodiesel through transesterification process with the presence of methanol and potassium hydroxide as a catalyst.
- iii. The produced biodiesel will be analysed and to measure properties such as cetane number, kinematic viscosity, cloud point, pour point, density, and flash point.

2 LITERATURE REVIEW

2.1 Overview

Biodiesel is an environmentally attractive alternative fuel to petrodiesel which can be defined as long-chain mono-alkyl esters of vegetable oils or animal fats and is produced through transesterification process with a monohydric alcohol usually methanol. The converted tryglycide is formed depends on the type of alcohol used. If methanol is used, the chemical reaction will yield methyl ester but if ethanol is used, ethyl ester will be produced (Abdulkareem1 *et al*, 2011).

The production of biodiesel from *Moringa oleifera* seeds consists of few methods. The ripe and dried-seeds are removed from long pods, and kernels are obtained from the seeds. Kernels are grounded using laboratory mortar and pestle (Arafat, 2013). The extraction of oil is carried out to proceed the process using different methods such as manual method (batch) which is seeds were defatted by using 95% ethanol in 5 % (w/v) suspension, mixing with a magnetic stirrer for 60 minutes. Supernatant was separated by centrifugation (3000 rpm, 45 min) and the settled powder was dried at room temperature for 24 hours (García-Fayos *et al*, 2010). Besides above method, a much well-known method is using Soxhelt extractor. 10g of grounded *Moringa oleifera* kernel is fed into thimbles of the electro thermal Soxhelt extraction chamber and 170ml hexane is pour into volumetric flask of Soxhelt extractor and the hexane is evaporated into three cycles for 30 minutes each cycle until hexane become colourless (Eman *et al*, 2012). According to Jessica *et al*. (2012), the amount of oil extracted from *Moringa oleifera* will be vary depending on the type of solvent used in the extraction process. For example, if ethanol is used, more oil will be extracted compared to methanol, petroleum and acetone.

Furthermore, the process is continued by transesterification of tryglycide, implementing standard procedure of 6:1 molar ratio of methanol to vegetable oil for one hour at 60°C with 1 wt% Sodium methoxide (NaOCH_3) as catalyst. As the reaction ended, the product is let to cool at room temperature without agitation to separate the product into two phases. After removing the unwanted bottom products which consist of glycerol, excess methanol and catalyst, soaps formed during the reaction, some entrained methyl ester and partial glycerides by decantation, the excess methanol that settled above the methyl ester is also removed at 80°C. Distilled water is used to wash out the remaining catalyst and residual water was removed by treatment with Na_2SO_4 , followed by filtration (Umer *et al*, 2008).

The production of biodiesel from palm oil also undergoes the similar process of transesterification palm oil using methanol with the presence of sodium hydroxide as a catalyst. As the process ended, palm oil will be transformed into ester form and glycerol. The remaining ester is called biodiesel (Khalizani and Khalisanni, 2011; Ghanei *et al.*, 2011; Sylvain *et al.*, 2009; Mário and José, 2011). Products then were used to carry out properties determination process using various machines and standards.

2.2 Physico-chemical Properties of Crude Palm Oil (CPO) and Crude *Moringa oleifera* Oil (CMOO)

Table 2.1: Physico-chemical properties of crude palm oil (CPO) and crude *Moringa oleifera* oil.

Properties	Units	Standards	CPO	CMOO
Dynamic viscosity	mPa s	ASTM D445	36.30	38.90
Kinematic viscosity at 40 °C	mm ² /s	ASTM D445	40.40	43.33
Kinematic viscosity at 100 °C	mm ² /s	ASTM D445	8.43	8.91
Viscosity Index	–	N/A	192.1	193.1
Density	kg/m ³	ASTM D4052	898.4	897.5
Flash point	°C	ASTM D93	165	268.5
Pour point	°C	ASTM D97	9	11
Cloud point	°C	ASTM D2500	8	10
Calorific value	MJ/kg	ASTM D240	39.44	38.05
Acid value	mgKOH/g oil	ASTM D664	3.47	8.62

* Data from Mofiju *et al*, 2014a

2.3 Physico-chemical Properties of Biodiesel from *Moringa oleifera* Seed Oil

Table 2.2 shows properties of *Moringa oleifera* methyl esters with comparison to standards

Property	M. oleifera methyl esters*	ASTM D6751	EN 14214
Cetane number	67.07	47 min	51 min
Kinematic viscosity (mm ² /s; 40° C)	4.83	1.9–6.0	3.5–5.0
Cloud point (°C)	18	-	-
Pour point (°C)	17	-	-
Oxidative stability (h)	3.61	3 min	6 min
Lubricity (HFRR; µm)	135, 138.5	-	-

*Data from Umer Rashid, 2008

Table 2.3: Fatty acid profile of *Moringa oleifera* oil with typical profiles of palm, rapeseed (canola), soybean and sunflower oils shown for comparison purposes. *Moringa oleifera* oil contain high percentage of oleic acid which consider to be a healthy oil.

Fatty acid	Structure* (Carbon number)	Moringa oleifera ^a	Palm ^b	Rapeseed ^b	Soybean ^b	Sunflower
Palmitic acid	C16:0	6.5	44.1	3.6	11	6.4
Stearic acid	C18:0	6.0	4.4	1.5	4	4.5
Oleic acid	C18:1	72.2	39.0	61.6	23.4	24.9
Linoleic acid	C18:2	1.0	10.6	21.7	53.2	63.8
Alpha or gamma-linolenic acid	C18:3	— ^c	0.3	9.6	7.8	— ^c
Arachidic acid	C20:0	4.0	0.2	-	-	-
Eicosenic acid	C20:1	2.0	-	1.4	-	-
Behenic acid	C22:0	7.1	-	-	-	-
	Other	1	1.1% C14:0, traces of others	0.2% C22:1	Traces	Traces

^a Data from Umer Rashid, 2008

^b Data from Gunstone and Harwood, 2007. These values constitute averages of numerous samples.

^c This may indicate traces (<1%) or absence of these fatty acids.

Data from Arjun *et al*, 2008

* Carbon number with ‘zero’ double bonds are saturated fatty acids, with ‘one’ double bonds are monosaturated and with ‘two’ and ‘three’ double bonds are polyunsaturated FA.

2.4 Physico-chemical Properties of Biodiesel from Palm Oil

Table 2.4: shows properties of Palm oil methyl esters with comparison to standards

Property	Palm Methyl Ester	ASTM D6751	EN 14214
Cetane number	65	47 min	51 min
Kinematic viscosity (mm ² /s; 40°C)	4.5	1.9–6.0	3.5–5.0
Cloud point (°C)	16.0	-	-
Pour point (°C)	16.0	-	-
Oxidative stability (h)	16 ^a	3 min	6 min

Data from Jawad Nagi *et al*, 2008

^a Data from Yung Chee Liang *et al*, 2013

2.5 Synthesis Routes

Nowadays, there are numerous synthesis routes in the production of biodiesel which using different alcohols and catalysts. Four of the biodiesel synthesis routes are direct use and blending of raw oils, micro-emulsions, thermal cracking (pyrolysis) and transesterification with process descriptions shown in Table 2.5, 2.6, 2.7 and 2.8 respectively. In this paper, transesterification was chosen to be the method to produce biodiesel because through this process, biodiesel will have a higher cetane number, lower emissions, higher combustion efficiency and renewable.

Table 2.5: Direct use and blending of raw oils process description

Process	<ul style="list-style-type: none">• Direct use and blending
Definition	<ul style="list-style-type: none">• Direct use as diesel fuel or blend with diesel fuel
Advantage	<ul style="list-style-type: none">• Liquid nature-portability• Heat content (~80% of diesel fuel)• Readily available and renewability
Disadvantage	<ul style="list-style-type: none">• Higher viscosity• Lower volatility• Reactivity of unsaturated hydrocarbon chains
Problems of using in engines	<ul style="list-style-type: none">• Coking and trumpet formation• Carbon deposits• Oil ring sticking• Thickening and gelling of the lubricating oil
References	<ul style="list-style-type: none">• Kaya <i>et al</i>(2009)• Issriyakul <i>et al</i> (2008)• Kansedo <i>et al</i> (2009)• Kumar Tiwari <i>et al</i> (2007)• Rao <i>et al</i> (2009).

Table 2.6: Micro-emulsions process description

Process	<ul style="list-style-type: none">• Micro-emulsions
Definition	<ul style="list-style-type: none">• A colloidal equilibrium dispersion of optically isotropic fluid microstructures with dimensions generally in the 1–150 nm range formed spontaneously from two immiscible liquids and one or more ionic or non-ionic amphiphiles
Advantage	<ul style="list-style-type: none">• Better spray patterns during combustion• Lower fuel viscosities
Disadvantage	<ul style="list-style-type: none">• Lower cetane number• Lower energy content

Problems of using in engines	<ul style="list-style-type: none"> • Irregular injector needle sticking • Incomplete combustion • Heavy carbon deposits • Increase lubrication oil viscosity
References	<ul style="list-style-type: none"> • Alonso <i>et al</i> (2008) • Sahoo and Das (2009) • Santos <i>et al</i> (2009) • Saraf and Thomas (2007) • Singh and Singh (2009) • Winayanuwattikun <i>et al</i>(2008)

Table 2.7: Thermal cracking (pyrolysis) process description

Process	<ul style="list-style-type: none"> • Thermal cracking (pyrolysis)
Definition	<ul style="list-style-type: none"> • The conversion of long-chain and saturated substance (biomass basis) to biodiesel by means of heat
Advantages	<ul style="list-style-type: none"> • Chemically similar to petroleum-derived gasoline and diesel fuel
Disadvantage	<ul style="list-style-type: none"> • Energy intensive and hence higher cost
Problems of using in engines	-
References	<ul style="list-style-type: none"> • Alonso <i>et al</i> (2008) • Santos <i>et al</i> (2009) • Saraf and Thomas (2007) • Singh and Singh (2009)

Table 2.8: Transesterification process description

Process	<ul style="list-style-type: none"> • Transesterification
Definition	<ul style="list-style-type: none"> • The reaction of a fat or oil with an alcohol in the presence of catalyst to form esters and glycerol
Advantages	<ul style="list-style-type: none"> • Renewability • Higher cetane number • Lower emissions • Higher combustion efficiency
Disadvantage	<ul style="list-style-type: none"> • Disposal of by-product (glycerol and waste water)
Problems of using in engines	–
References	<ul style="list-style-type: none"> • Schinas <i>et al</i> (2009)

2.6 Basic chemical reactions

Above figure depict the simplified form of chemical reaction which is presented in equation. Vegetable oils or animal fats are esters of saturated and unsaturated monocarboxylic acids with the trihydric alcohol glyceride. These esters are called triglycerides, which can react with alcohol in the presence of a catalyst, a process known as transesterification. R_1 , R_2 and R_3 in

above figure are long-chain hydrocarbons, sometimes called fatty acid chains. Normally, there are five main types of chains in vegetable oils and animal oils: palmitic, stearic, oleic, linoleic, and linolenic. 1 mol of fatty ester is liberated at each step of the conversion process of triglyceride into diglyceride, monoglyceride, and finally to glycerol (Dennis *et al*, 2010).

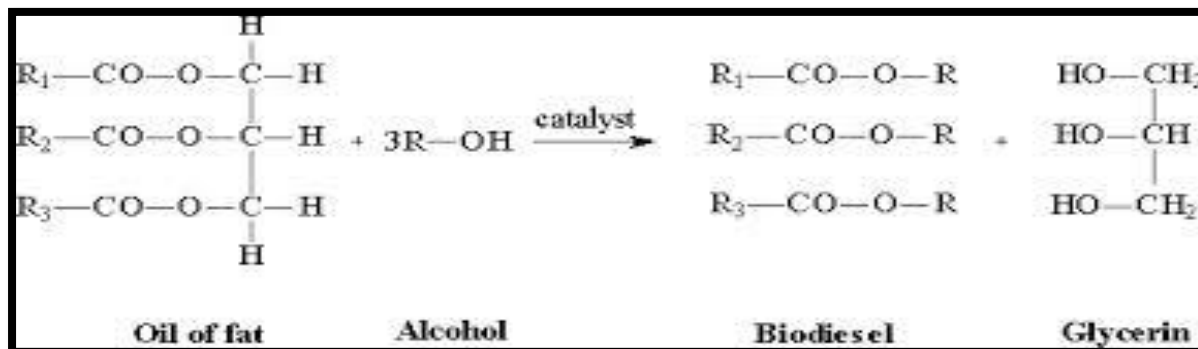


Figure 2.1: Basic chemical reaction

2.7 Catalyst

Normally, there are three type of catalysts being used in transesterification which are alkalis, acids, and enzymes. All three catalysts including their advantages, disadvantages and example of catalyst are shown in Tables 2.9, 2.10, 2.11, 2.12 and 2.13 respectively. In this research, potassium hydroxide was chosen as a catalyst to be used transesterification because it is cheaper and most commercially use in small and large scale biodiesel production compare to acid and enzyme catalyst (Umer and Anwar, 2008; Umer et al, 2008; Demirbas, 2008; Azcan and Danisman, 2007; Dias et al, 2008; Dizge et al, 2009; Dizge and Keskinler, 2008). The cost of transesterification process which use alkali catalyst especially potassium hydroxide is cheap because the process is carried out at low temperature of around 60°C and pressure environment. Moreover, it require no intermediate step and high conversion rate.

Table 2.9: Alkali homogeneous catalyst

Alkali	
Type	<ul style="list-style-type: none"> Homogeneous
Advantages	<ul style="list-style-type: none"> High catalytic activity Low cost Favourable kinetics modest operation conditions
Disadvantages	<ul style="list-style-type: none"> Low FFA requirement Anhydrous conditions Saponification Emulsion formation More wastewater from purification Disposable

Example	<ul style="list-style-type: none"> • NaOH • KOH
References	<ul style="list-style-type: none"> • Dizge <i>et al</i> (2009) • Qian <i>et al</i> (2008) • da Silva <i>et al</i> (2008) • Di Serio <i>et al</i> (2007) • Kawashima <i>et al</i> (2009)

Table 2.10: Alkali heterogeneous catalyst

Alkali	
Type	<ul style="list-style-type: none"> • Heterogeneous
Advantages	<ul style="list-style-type: none"> • Noncorrosive, • Environmentally benign • Recyclable • Fewer disposal problems • Easily separation • Higher selectivity • Longer catalyst lifetimes
Disadvantages	<ul style="list-style-type: none"> • Low FFA requirement • Anhydrous conditions • More wastewater from purification • High molar ratio of alcohol to oil requirement • High reaction temperature and pressure • Diffusion limitations • High cost
Example	<ul style="list-style-type: none"> • CaO • CaTiO₃ • CaZrO₃ • CaO–CeO₂ • CaMnO₃ • Ca₂Fe₂O₅ • KOH/Al₂O₃ • KOH/NaY • Al₂O₃/KI • ETS-10 zeolite • Alumina/silica supported K₂CO₃
References	<ul style="list-style-type: none"> • Liu <i>et al</i> (2008) • Kulkarni <i>et al</i> (2006) • Qian <i>et al</i> (2008) • Di Serio <i>et al</i> (2007) • Kawashima <i>et al</i> (2009)

Table 2.11: Acid homogeneous catalyst

Acid	
Type	<ul style="list-style-type: none"> • Homogeneous
Advantages	<ul style="list-style-type: none"> • Catalyse esterification and transesterification simultaneously • Avoid soap formation
Disadvantages	<ul style="list-style-type: none"> • Equipment corrosion • More waste from neutralization • Difficult to recycle • Higher reaction temperature • Long reaction times • Weak catalytic activity
Example	<ul style="list-style-type: none"> • Concentrated sulphuric acid
References	<ul style="list-style-type: none"> • Dizge <i>et al</i> (2009) • da Silva <i>et al</i> (2008) • Di Serio <i>et al</i> (2007)

Table 2.12: Acid heterogeneous catalyst

Acid	
Type	<ul style="list-style-type: none"> • Heterogeneous
Advantages	<ul style="list-style-type: none"> • Catalyze esterification and transesterification simultaneously, recyclable • Eco-friendly
Disadvantages	<ul style="list-style-type: none"> • Low acid site concentrations • Low microporosity • Diffusion limitations • High cost
Example	<ul style="list-style-type: none"> • ZnO/I₂ • ZrO₂/SO₄²⁻ • TiO₂/SO₄²⁻ • Carbon-based solid acid catalyst • Carbohydrate-derived catalyst • Vanadyl phosphate • Niobic acid • Sulphated zirconia • Amberlyst-15 • Nafion-NR50
References	<ul style="list-style-type: none"> • Lou <i>et al</i> (2008) • Dizge and Keskinler (2008) • Di Serio <i>et al</i> (2007) • Kawashima <i>et al</i> (2009)

Table 2.13: Enzyme catalyst

Enzyme	
Type	<ul style="list-style-type: none"> • Enzymes
Advantages	<ul style="list-style-type: none"> • Avoid soap formation • Non-polluting • Easier purification
Disadvantages	<ul style="list-style-type: none"> • Expensive • Denaturation
Example	<ul style="list-style-type: none"> • <i>Candida antarctica</i> fraction B lipase • <i>Rhizomucor miehe</i> lipase
References	<ul style="list-style-type: none"> • Haas (2005) • Lou <i>et al</i> (2008) • Kawashima <i>et al</i> (2009)

2.7 Alcohol

There are several types of alcohol that can be used in transesterification process such as methanol, ethanol, propanol, iso-propanol, butanol and amyl alcohol (Encinar *et al.*, 2007; Wang *et al.* 2005; Mariod *et al.* 2006; Meneghetti *et al.* 2006; Yao and Hammond 2006; Dantas *et al.* 2007; Issariyakul *et al.* 2007; Kulkarni *et al.* 2007; Alamu *et al.* 2008; Domingos *et al.* 2008; Georgogianni *et al.* 2008; Lima *et al.* 2008; Rodrigues *et al.* 2008; Stavarache *et al.* 2008). Among aforementioned alcohols, methanol is the most extensively used in the production of biodiesel (Chang and Liu, 2009). In this research, methanol also was used to react with both palm oil and *Moringa oleifera* oil to produce biodiesel because of its economical reason as well as its physical and chemical advantages. Moreover, methanol can react with vegetable oils or animal fats in a short period of time and potassium hydroxide is easily dissolved in it.

3 MATERIALS AND METHODS

3.1 Overview

This paper presents extraction of *Moringa oleifera* oil using three different solvent, and transesterification process of *Moringa oleifera* oil and crude palm oil to produce biodiesel. In order to produce biodiesel from *Moringa oleifera* oil, the oil must be extracted using solvent in Soxhelt Extractor. The solvents involved were methanol, ethanol and n-hexane. These solvents were used in the extraction process to identify which solvent will yield the highest oil. On the other hand, biodiesel produced from crude palm oil and *Moringa oleifera* oil were analyzed and the properties were compared to get the best feedstock.

3.2 Introduction

Dry *Moringa oleifera* seeds were obtained from Kota Bharu, Kelantan, Malaysia. The dry seeds were crushed using mortar and pestle and kept at room temperature. Palm oil will be bought from nearest palm oil factory.

3.3 Chemicals

The following chemicals were supplied by Ethanol, n-Hexane, methanol, Fuller's Earth and florosil were supplied by Faculty of Chemical and Natural Resources Engineering of Universiti Malaysia Pahang.

- I. Ethanol
- II. n-Hexane
- III. Methanol
- IV. Fuller's Earth
- V. Magnesium Silicate (florisil)
- VI. Potassium hydroxide

3.4 Apparatus

- i. Beaker (50ml, 100ml, 250ml, 500ml & 1 litre)
- ii. Soxhelt Extractor with condenser

- iii. Rotary Evaporator
- iv. Centrifuge
- v. Oven
- vi. Mortar and pestle
- vii. Digital weighing machine
- viii. Aluminium weighing boat
- ix. Magnetic stirrer
- x. Magnetic stirrer retriever
- xi. Hot plate
- xii. Reflux Condenser
- xiii. Heating mantle
- xiv. Stopper
- xv. Thermometer
- xvi. Separator Funnel (250ml, 500ml & 1 litre)
- xvii. Thimble
- xviii. Spatula
- xix. Reagent Bottle (100ml, 250ml, 500ml & 1 litre)
- xx. Receiving flask (500ml)
- xxi. Rotary clip
- xxii. Rotary flask (500ml)
- xxiii. Round bottom flask (1 litre)
- xxiv. Centrifuge tube

3.5 Overall Methodology Flowchart

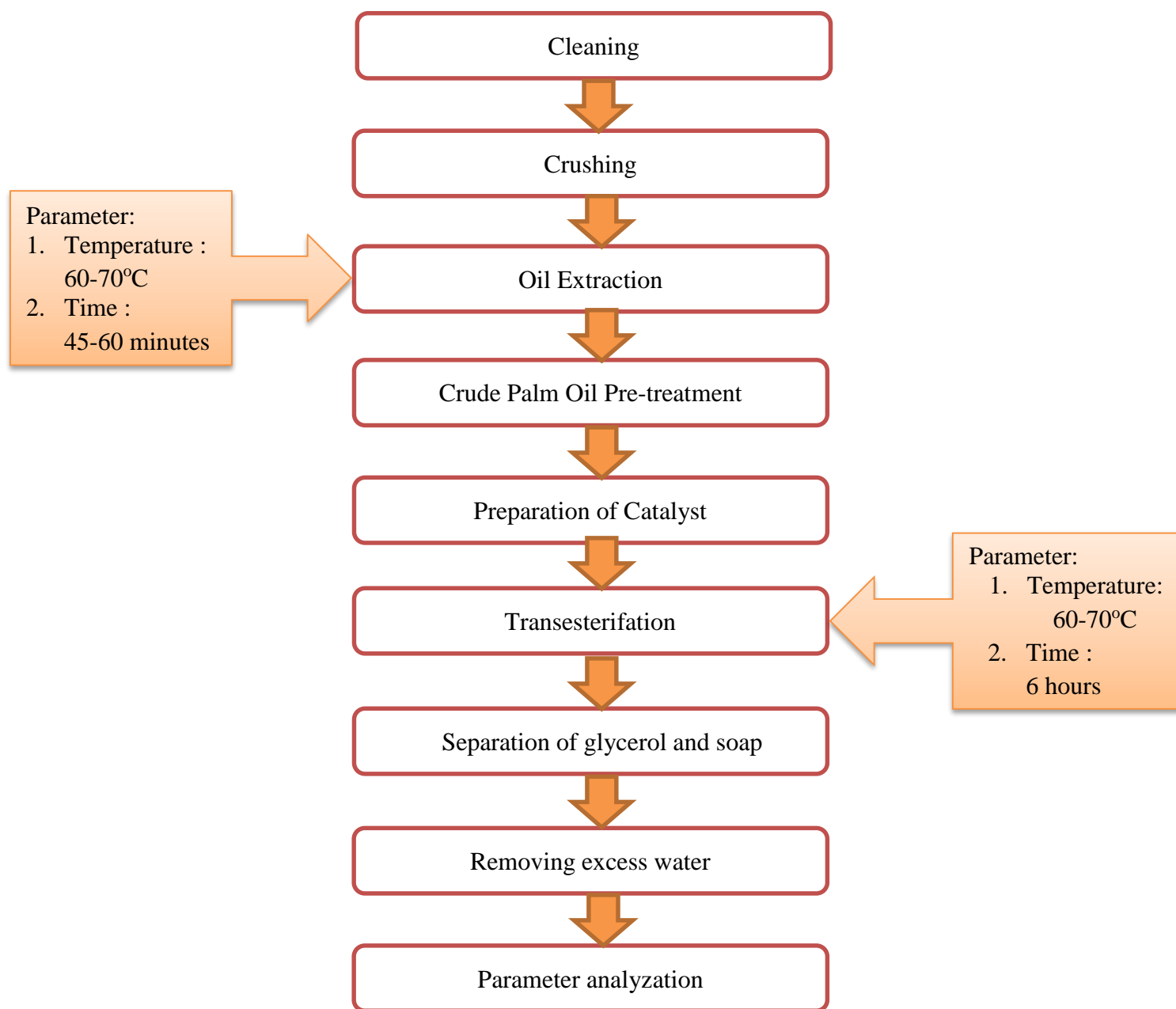


Figure 3.1: Overall Methodology Flow Chart

3.6 Experimental Methodology

Pre-treatment starts with collection of dry *Moringa oleifera* pods and continues with the cleaning. In order to get the ripe seeds, the dry pods were removed together with the three papery wing and light wooden shells. Cleaning process is crucial because a clean seeds yield clean oil without any impurities. Then, seeds were undergone size reduction by crushing it

using mortar and pestle (Ogbunugafor *et al*, 2011). The crushed seeds were weighted using electronic weighting balance.

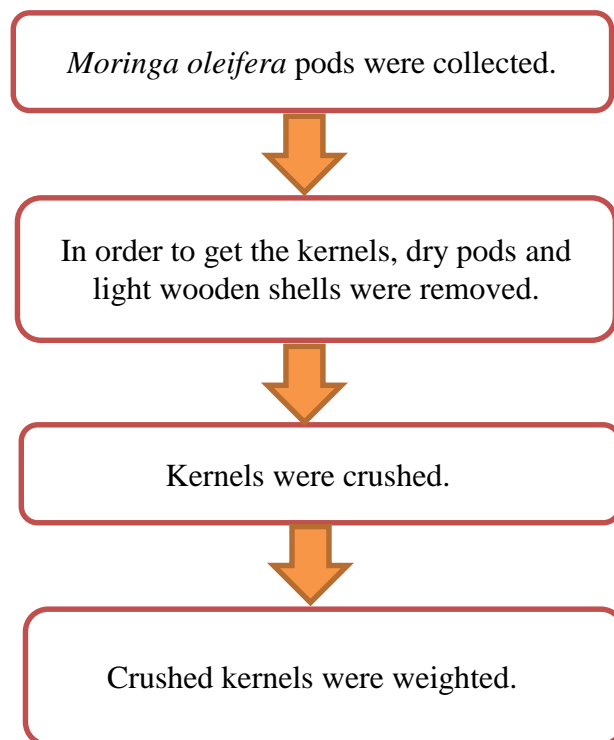


Figure 3.2: Flow diagram of pre-treatment process

3.7 Oil Extraction

30g crushed *Moringa oleifera* kernel was placed into thimble of Soxhelt Extractor fitted with 500mL round-bottom flask and condenser containing 200ml n-hexane for 1 hour (Trakarnpruk and Chuayplod, 2012; Eman *et al*, 2010). A heating mantle was set at 60-70°C and as the heating commenced, the n-hexane was continuously heated and it started to evaporate and condense back into thimble containing crushed kernel. Crude oil was recovered and the solvent was removed by placing the oil into rotary evaporator under vacuum at 70°C. The extracted oil was analysed. Above method was repeated using ethanol and methanol. The cake residue were dried to remove remaining water and solvent using oven for 1 hour at 50°C and the weight of the cake was recorded to calculate the produced oil yield.

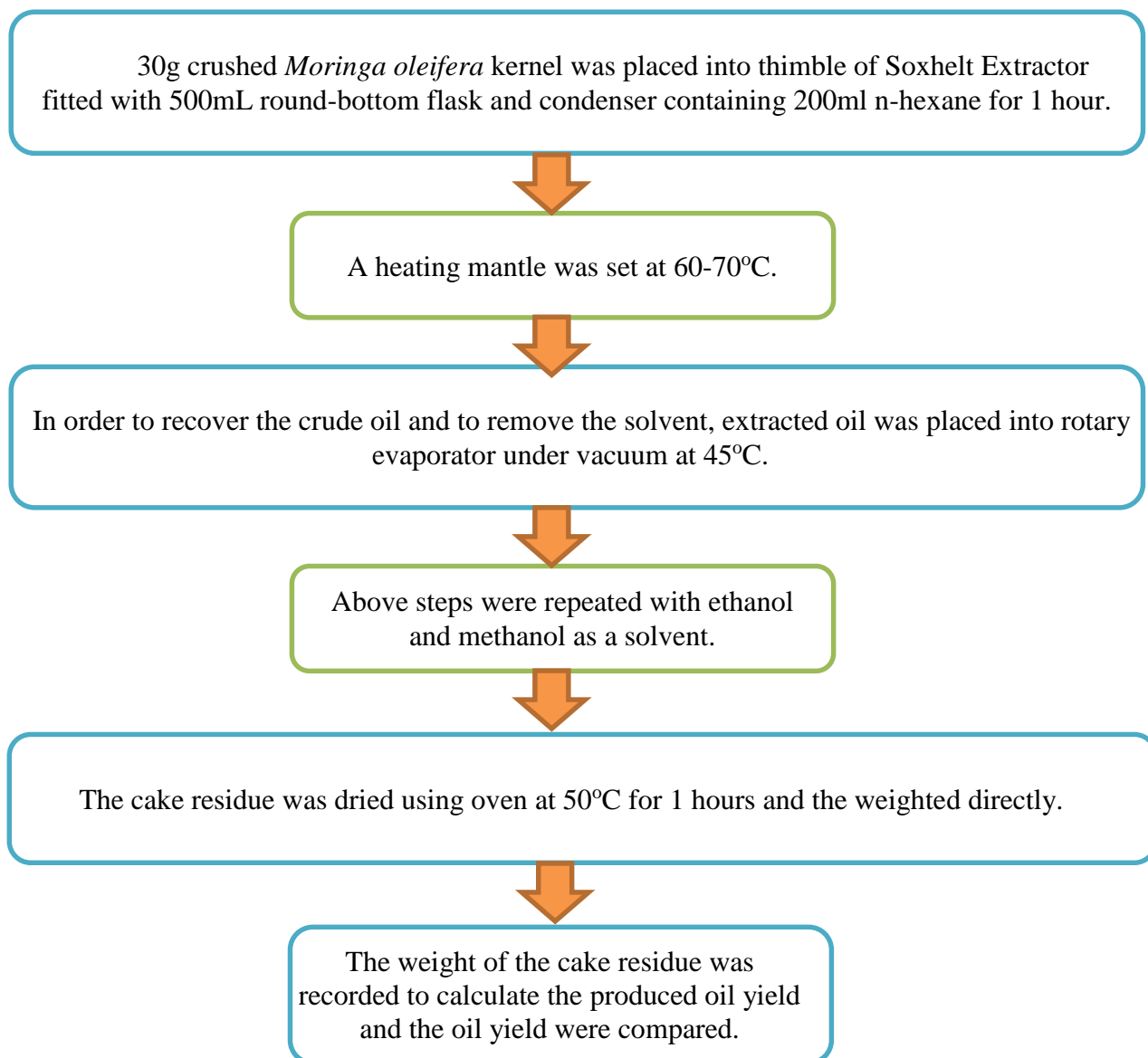


Figure 3.3: Flow diagram of oil extraction

3.7.1 Soxhlet Extractor

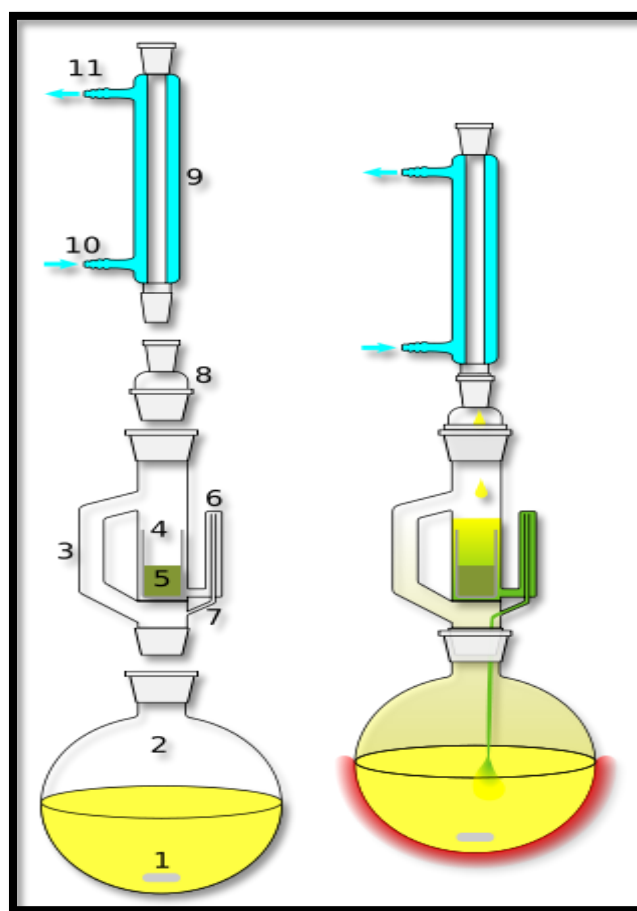


Figure 3.4: A schematic representation of a Soxhlet extractor

1. Stirrer bar
2. Round-bottom flask (the round-bottom flask should not be overfilled and the volume of solvent should be 3 to 4 times the volume of the soxhlet chamber).
3. Distillation path.
4. Thimble
5. Solid
6. Siphon top
7. Siphon exit
8. Expansion adapter
9. Condenser
10. Cooling water in
11. Cooling water out

Extraction Procedure using Soxhelt Extractor

- i. Pour about 300ml solvent (n-hexane or ethanol) into the 500mL round-bottom flask.
- ii. Place grinded and sieved *Moringa oleifera* kernels thimble of Soxhelt Extractor.
- iii. Gently connect the round-bottom flask with the upper part of the Soxhelt Extractor containing *Moringa oleifera* kernels.
- iv. Gently connect condenser with the middle part of the Soxhelt Extractor containing *Moringa oleifera* kernels.
- v. Place the Soxhelt Extractor into the heating mantle.
- vi. Supply the running water to the condenser and make sure water is continuously flow in and out of the condenser.
- vii. Set the heating mantle to 100°C until the solvent start to boil and when the solvent is boiling reduce the temperature to 70°C.
- viii. After 3 cycled of the extraction, the extracted oil is produced.

Precaution during the extraction process

- i. Gently handle the glass part of the Soxhelt Extractor.
- ii. Make sure there is no water leakage at water in or water out section as the leaking water will damage the heating mantle.

3.8 Preparation of Catalyst and neutralization

In this research, it was decided to use molar ratio of vegetable oil to methanol of about 6:1 (Mofijur et al, 2014b). It was reported by Dorado *et al* (2005) and Meher *et al* (2006) that the increment in the concentration of catalyst will give a bad effect to the yield of methyl ester due to formation of soap. This is because as the concentration of catalyst is high, viscosity of reactant will be increased thus lowered the yield. In order to follow the chosen molar ratio, about 0.5gram of potassium hydroxide was diluted in 50ml of methanol. The purpose of dilution of potassium hydroxide with methanol is to produce methoxide which reacts with the base oils. Most of the catalysts (e.g. NaOH, KOH) are in solid form and do not readily dissolve into methanol, it is best to stir methanol in a beaker using magnetic stirrer on a hot plate at 30°C and add the catalyst slowly and carefully. When the catalyst completely dissolves in the methanol, the methoxide is ready to be added to the oil. Once the methoxide is added into the oil, a neutralization reaction will immediately start. Some alkali catalysts will react with rudimental acids during the pretreatment step or will react with the free fatty acids from the oil. Therefore, more catalyst needs to be added to complete the reaction.

3.9 Crude Palm Oil Pre-treatment

This pre-treatment was carried out to get a clean crude palm oil. In order to remove any unwanted solid particle, crude palm oil was centrifuged at 5000rpm for 15 minutes. The pre-treatment continued with bleaching. Fuller Earth was used as bleaching agent and about 6gram of fuller earth were poured into beaker containing biodiesel and stirred for 30 minutes at 100°C. The mixture was left to cool down and centrifuged once again at 5000rpm for 15minute to get a clean and clear palm oil.

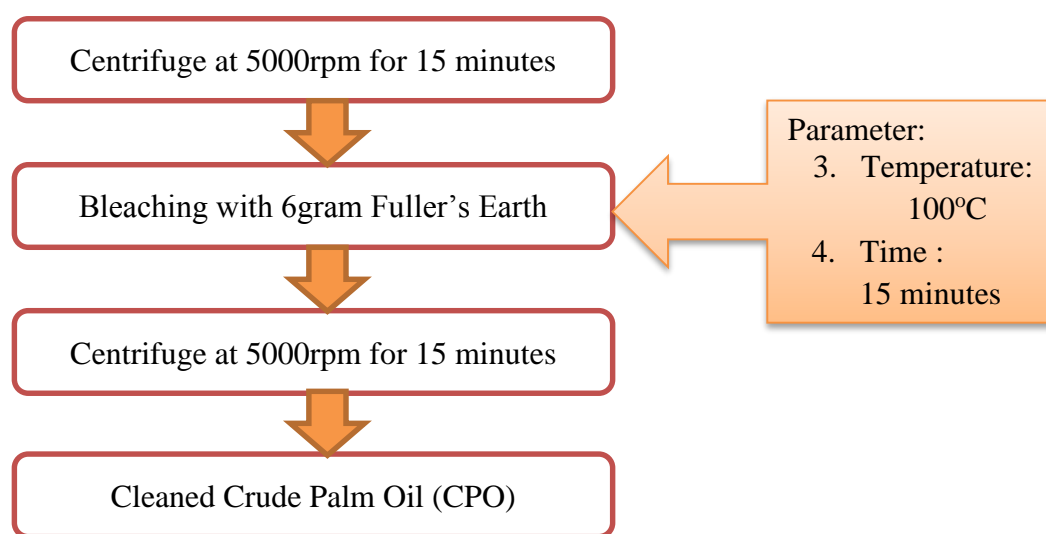


Figure 3.5: Flow diagram of Crude Palm Oil Pre-treatment

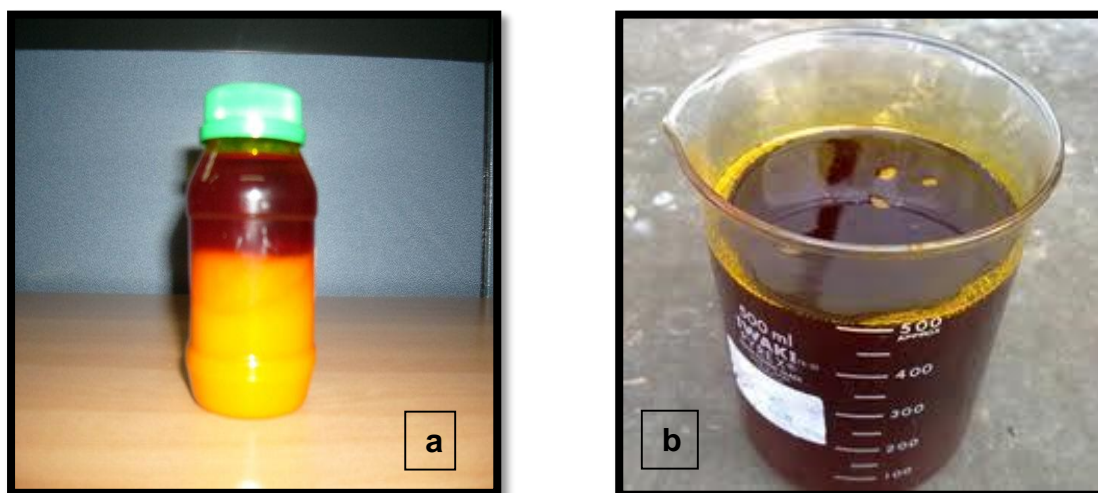


Figure 3.6: a) Crude palm oil before centrifuge b) Crude palm oil after centrifuge

3.10 Transesterification Process

The *Moringa oleifera* oil was converted into Methyl ester using methanol in a 1 litre round-bottom flask with a reflux condenser in a water bath. The mixture was stirred using magnetic

stirrer at 400rpm and heated to 60°C for 1 hour. This method also was repeated using crude palm oil. After 1 hours, the yield product which consists of 2 layers, the upper layer is biodiesel and the bottom layer contain glycerol and unwanted product such soap formed during reaction, excess methanol and partial glycerol (Trakarnpruk and Chuayplod, 2012).

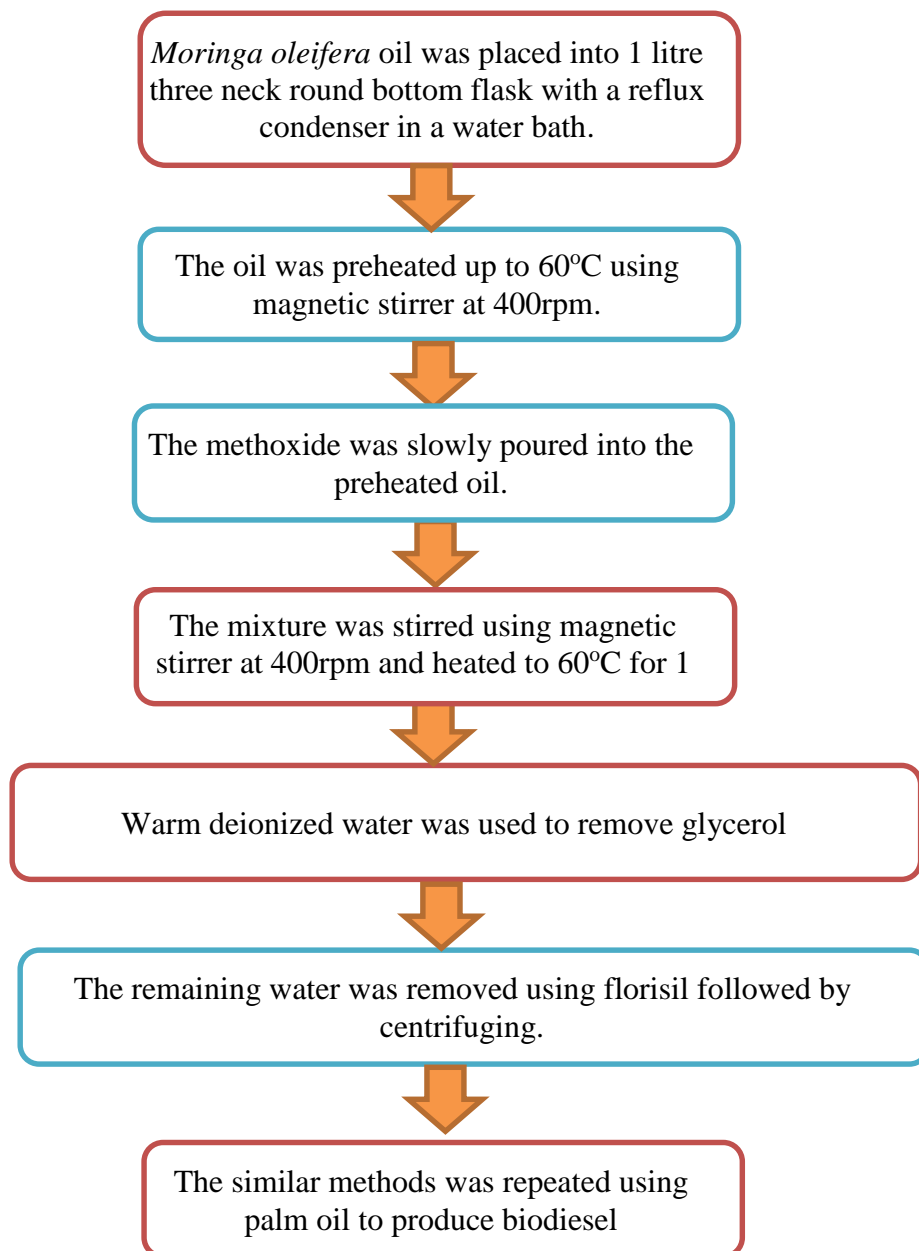


Figure 3.7: Flow diagram of Transesterification process

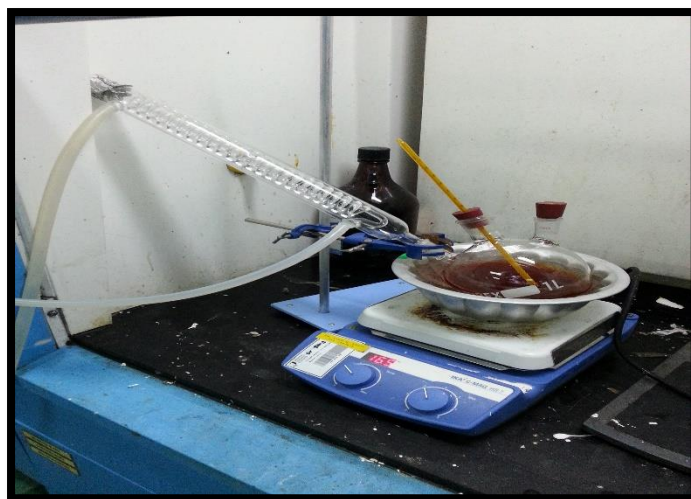


Figure 3.8: Equipment setup

3.11 Settling

After 1 hour of transesterification process, the mixture was poured into separator flask for at least 12 hours (Predojevic, 2008). There were 2 layer after the settling process. The lower layer was glycerol and unwanted product which was removed and the upper part was biodiesel.

3.12 Washing

This method was carried out to remove glycerol using heated deionized water. The deionized was heated up to 55°C and poured into biodiesel. The mixture was settled in a separator funnel for 12 hours. After 12 hours, there were 2 layer of which the lower was water + glycerol and the upper layer was biodiesel. The lower part was removed and biodiesel was continuously washed until the drain water was cleared and the pH was neutral.

3.13 Removing Excess Water

Biodiesel was stirred with 4gram of florosil for 30minutes and centrifuged at 5000rpm for 15minute. This step was carried out to remove excess water from biodiesel.

3.14 Properties Determination

The quality of biodiesel produced depends on their fatty acid ester composition. There are three main structural features that effect the physical fuel properties which are chain length, degree of unsaturation and branching of the chain (Martínez *et al*, 2014). Moreover, numbers of biodiesel fuels properties can be analysed using different machine and standards and these fuels

properties depend on chemical composition which are cetane number, kinematic viscosity, density, cold-flow properties in the form of cloud point (CP), pour point (PP) and flash point (Pinzi *et al*, 2009; Refaat, 2009; Ramos *et al*, 2009).

3.14.1 Cetane Number



Figure 3.9: Shatox SX-100M Portable Octane/Cetane Analyzer

One of the most important fuels properties which also regularly being applied to biodiesel and become a parameter to the evaluation of the self-ignitability and combustion of fuel is cetane number. Cetane number is a dimensionless descriptor for the ignition delay time at which fuel experiences upon injection into the combustion chamber of a diesel engine. The shorter the ignition delay time, the greater the cetane number and vice versa (Knothe, 2014). Cetane number is determined using American Standard Testing Methods D6751 (ASTM D6751)

3.14.2 Kinematic Viscosity

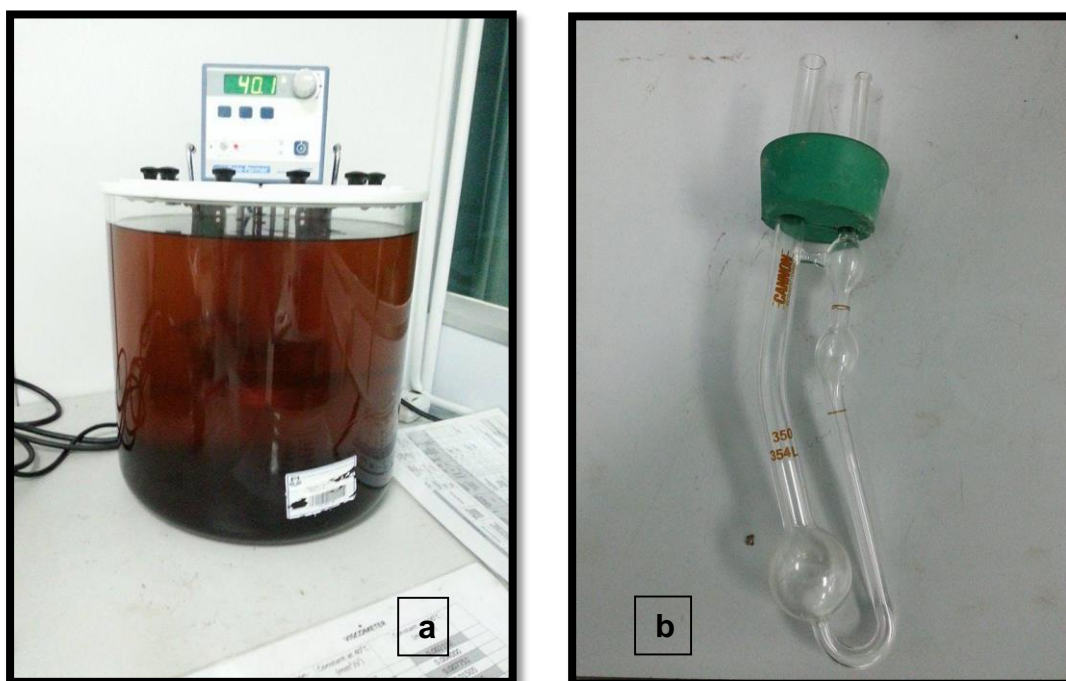


Figure 3.10: a) Viscometer Bath and b) Cannon-Fenske Viscometer (Number 350)

The consumption of biodiesel in diesel engines is limited by some of the physical properties, especially the viscosity (Encinar *et al*, 2005). Kinematic viscosity is key fuel properties for diesel engine and an important fuel parameter required by biodiesel and diesel fuel standard. It was reported by Parente *et al* that viscosity of biodiesel is slightly higher than petro-diesel. Viscosity is the resistance for fluid's motion and indirect measure of the internal friction between molecules. During the combustion process, the diesel or biodiesel is sprayed into compressed air, and atomized into small drops near to the nozzle exit. The liquid fuel, usually, forms a cone-shaped spray at the nozzle exit and its viscosity affects on the atomization quality, size of fuel drop and penetration process in direct-injection diesel engines (Alptekin and Canacki, 2009). Therefore, it influences the quality of combustion (Tate *et al*, 2006). Any biodiesel fuel, including those optimized for fatty acid composition, need to meet the kinematic viscosity specifications (determinations at 40 °C) in biodiesel standards which are 1.9–6.0 mm²/s in the American standard ASTM D6751 and 3.5–5.0 mm²/s in the European standard EN 14214 (Knothe and Steidley, 2007). Implementing ASTM D445 and Cannon–Fenske viscometers was used to obtain the time for both biodiesel to flow under gravity through a calibrated glass capillary viscometer at 40 °C (Luis Felipe *et al*, 2012).

3.14.3 Density



Figure 3.11: a) and b) Chamber used to place the sample c) AccuPyc II 1340 Gas Displacement Density Analyzer (Chamber containing sample will be place inside this machine to be analysed).

Density is another important property of biodiesel that affects production, transportation, and distribution processes as well as all processes that take place in the internal combustion engine (István Barabás, 2013). It is defined as the weight of a unit volume of fluid (Luis Felipe *et al*, 2011). Moreover, density directly influences the fuel injection process in that the amount of fuel injected is measured by volume, thus density

determines the actual injected fuel mass (Alptekin and Canakci, 2008). In addition, density is an important parameter for liquid fuel to correlate the cetane number, heating value and viscosity (Luis Felipe *et al*, 2011). The composition profile, temperature and pressure are the major factors that determine the biodiesel density (Baroutian *et al*, 2008; Baroutian *et al*, 2008; Pratas *et al*, 2011). Biodiesel produce from different sources will yield a different density due to the variation in composition profiles. Just like other liquid fuels, density of biodiesel will decrease with the increase of temperature and increase with the increase of pressure (Xiangzan *et al*, 2013). It is necessary to know density of a fuel as density will assist in designing production and manufacturing facilities for fuels, reactors, tanks, distillation units. In addition, density will help to find the mass and volume flows through such facilities, establish the appropriate size of transfer pumps, transfer and safety valves, and many more (Yaws, 2008; . Density was determined using AccuPyc II 1340 Gas Displacement Density Analyzer by implementing ASTM D4052 standard.

3.14.4 Cloud and pour point

Biodiesel properties such as exhaust gas emissions, internal lubricity, and its renewability nature are comparable with petroleum derived diesel, however poor cold flow properties of biodiesel are one of the obstacles that hinder biodiesel from being commercially consume (Bhale *et al*, 2009). Solidification of the saturated fatty acid exist in biodiesel during winters causes fuel starvation and operability problems as solidified material clogs the fuel lines and filters. Huge amount of saturated fatty acyl esters in biodiesel are responsible for high CP (cloud point), PP (pour point) and CFPP (cold filter plugging point) temperatures. With decreasing temperature more solid is formed and material approaches the pour point. Defining pour points as the lowest temperature at which the fuel becomes semi solid and loses its flow characteristics and become gelled, which makes it no longer pumpable. Pour point is used to characterize the cold flow operability of a fuel because the pour point of a fuel affects the utility of the fuel, especially in cold weather. The pour point of biodiesel is higher than diesel fuels due to having higher amount of saturated fatty acids (Gaurav and Sharma, 2014). Cloud point is the temperature by which liquid fatty material begins to become crystal or wax which contribute to cloudy appearance. It is measured as the temperature at which wax or crystal is first formed first, as the fuel is cooled. This situation lead to

blockages in the pipes and filters of the fuel systems of the vehicles. Moreover, the wax also accumulates on cold surface and forms an emulsion with water. Therefore, cloud point indicates the tendency of the oil to plug filters or small orifices at cold operating temperatures. (Gaurav and Sharma, 2014; Nazmiye and Hakan, 2014; Purnanand et al, 2009). Cloud and pour point determinations were conducted using K46100 Cloud Point & Pour Point Apparatus by implementing ASTM D2500 standard for Cloud Point and ASTM D97 standard for Pour Point.



Figure 3.12: a) K46100 Cloud Point & Pour Point Apparatus b) Test jar with sample and thermometer.

3.14.5 Flash Point

One of the most important physicochemical properties for establishing the potential for fire and explosion of a hazardous material such as a fuel is its flash point (FP). Flash point also one of the biodiesel quality indicator. The flash point is related to the vapor pressure of a flammable liquid and is defined as the lowest temperature at which it can

form a combustible mixture with air. When temperature increases, the vapor pressure increases and the amount of evaporated flammable liquid in equilibrium with the air also increases. As the flash point is reached, a simple ignition source is able to combust the mixture (Liaw and Chiu, 2006). Flash point does not directly affect biofuel combustion, but a higher flash point makes biodiesel safer for storage, handling and transportation (Jorge *et al*, 2011 and Natália *et al*, 2012). Flash point was determined using Pensky-martens flash point – automatic NPM 440 (Normalab, France) by implementing ASTM D93 standard.



Figure 3.13: Pensky-martens flash point – automatic NPM 440

4 RESULT AND CONCLUSION

4.1 Effect of different solvent in oil extraction

Table 4.1: Analysis of percentage of oil yield using three different solvents

Solvents	Amount of solvents (ml)	Weight of seeds (g)	Percentage of oil yield (%)			
			Batch 1	Batch 2	Batch 3	Average
Methanol	200	30	16.2	17.5	15.7	16.5
Ethanol	200	30	18.9	19.8	20.8	19.8
n-Hexane	200	30	33.2	34.5	35.1	34.3

The percentage of oil yield was calculated using below equation:

$$\frac{W_o - W_1}{W_o} \times 100\%$$

Where:

W_o- weight of *Moringa oleifera* seeds

W₁- weight of *Moringa oleifera* cake residue

The percentage of oil yield was studied by carrying out 3 batch of oil extraction using different solvent with the same amount of seeds. After each extraction, the residue cake was dried in oven for 1 hour at 50°C and as the drying process ended, the cake residue was weighted using electronic weighing balance. From **Table 1**, it can be concluded that the highest oil yield was n-Hexane and the percentage was 34.3%.

4.2 Comparison of biodiesel properties produced from *Moringa oleifera* and crude palm oil

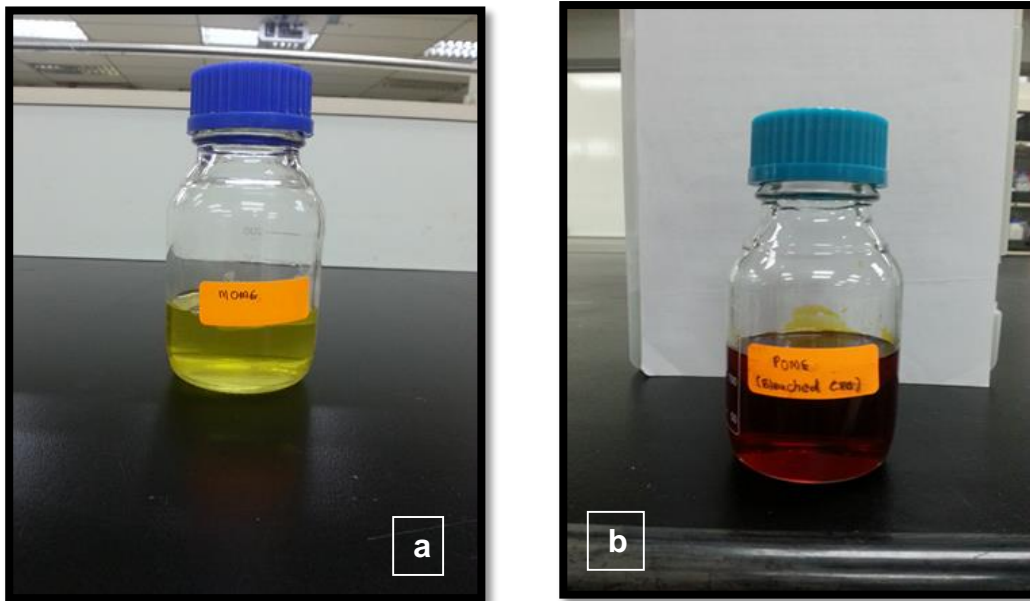


Figure 4.1: a) *Moringa oleifera* Methyl Ester b) Palm oil Methyl Ester

4.2.1 Cetane Number

Cetane number was determined using Ignition Quality Tester. This test was repeated three times using 50ml of biodiesel in order to get the average value and the result were tabulated. Based on the result, biodiesel produced from *Moringa oleifera* oil possessed higher cetane number which was 66.7 compare to biodiesel produced palm oil which was 59.5. This high cetane number will help to ensure low carbon release footprint, improved fuel efficiency and reduce tear and wear for both vehicle starter and batteries (Masina et al, 2012).

4.2.2 Kinematic viscosity

Kinematic viscosity was done because to compare which biodiesel able to flow easily under pressure. According to ASTM D445 standard, viscosity number of No. 350 was used to measure the kinematic viscosity and about 30ml of biodiesel was needed to complete the test. After the average value acquired in Table 3, the kinematic viscosity for biodiesel produced from *Moringa oleifera* oil was 4.8mm²/s and 5.03mm²/s for biodiesel produced from palm oil. Thus it can be conclude that *Moringa oleifera* oil had a lower kinematic viscosity value compare to palm oil biodiesel. It is crucial to keep kinematic viscosity within the range stated

in ASTM and EN standard as low viscosity can cause leakage in the fuel system and high viscosity also bring problems to diesel engine. Although fuel spray penetration into the combustion chamber can be eventually promoted by higher viscosities of fuels, there are more adverse effects that can occur in the engine performance, because a higher viscosity can cause excessive fuel injection pressures for the engine warm-up, increasing the energy demand of pumps and tending to form larger droplets up, leading to a poorer spray. Those phenomena can cause an incomplete combustion, wear of the fuel pump elements, choking of the fuel injectors, and ring carbonization (Baroutian *et al*, 2010; Yuan *et al*, 2005; Yuan *et al*, 2009; Freitas *et al*, 2011; Alptekin and Canakci, 2009; Xue *et al*, 2011; Encinar *et al*, 2005). Moreover, high viscosity also create number of problems in cold weather as viscosity increases as the temperature decreases (Knothe and Steidley, 2007)

4.2.3 Cloud and Pour point

The cold flow properties of biodiesel are characterized by Cloud Point (CP), Cold Filter Plugging Point (CFPP) and Pour Point (PP) (Rajagopala *et al*, 2012). The CP is the temperature at which the fuel shows a haze from the formation of crystals. The CFPP is the temperature at which the crystals formed will cause the plugging of the filters. The PP is the lowest temperature at which the liquid will flow (Soriano *et al*, 2005). About 50ml of biodiesel was pour into test tube until it reached the level indicator on the test tube. The sample was placed inside a refrigerator and observed for every one minutes until solid-crystals appeared. Cloud point was measured at the bottom of test jar while pour point was measured at the centre of biodiesel. After the test, the cloud and pour point for *Moringa oleifera* biodiesel were 18°C and 12°C and palm oil biodiesel were 16°C and 7°C. Although cloud and pour point for *Moringa oleifera* biodiesel is slightly higher compare to palm oil biodiesel, the high content of saturated ester which possess higher melting point than saturated fatty acid in palm oil biodiesel seem to compensate as the cold flow properties for biodiesel are determined based on the amount of higher melting component and not their nature (Imahara *et al*, 2006). If prediction of cold properties can be made feasible, troublesome measurements can be eliminated and the feedstock can be appropriately selected to produce a biodiesel that can meet specification. Since CP is the trigger for negative effect on fuel injection, its prediction is extremely meaningful (Hiroaki *et al*, 2006).

4.2.4 Flash Point

Flash point was crucial to be tested to compare with the classification of the Department of Transportation (DOT) regulations. It is used in shipping and safety regulations to define flammable and combustible material. It was determined that *Moringa oleifera* biodiesel and palm oil biodiesel flash point were 165°C and 160°C which were higher than petroleum diesel (approximately 70° C) and therefore it is much safer to handle and transport. Moreover, The Flash Point is also used to ensure the residual methanol left in the fuel after biodiesel processing will not negatively affect combustion and other fuel system components.

4.2.5 Density

Density is a fuel property which directly affect the engine performance characteristics. Many performance characteristics, such as cetane number and heating value, are related to the density. On the other hand, diesel fuel injection systems measure the fuel by volume. So the changes in the fuel density will influence engine output power due to a different mass of fuel injected. The density and viscosity of the fuels affect the start of injection, the injection pressure, and the fuel spray characteristic, so that they influence the engine performance, combustion and exhaust emissions (Alptekina and Canakci, 2008). Density for *Moringa oleifera* biodiesel and palm biodiesel was 875kg/m³ and 890kg/m³ respectively.

Table 4.2: Biodiesel properties from *Moringa oleifera* seed oil and palm oil.

Properties	<i>Moringa oleifera</i> biodiesel				Palm oil biodiesel			
	1	2	3	Average	1	2	3	Average
Cetane number	66.7	66.8	66.7	66.7	59	59.5	60	59.5
Kinematic viscosity (mm²/s; 40° C)	4.8	4.7	4.8	4.8	5.06	5.0	5.06	5.04
Cloud point (°C)	18	18	18	18	16	16	16	16
Pour point (°C)	12	12	12	12	7	7	7	7
Flash point (°C)	165	164	165	165	159	160	160	160
Density (kg/m³)	875	875	875	875	890	890	891	890

Calculation for kinematic viscosity:

Viscosity constant : 0.4899mm²/s
Efflux time : 10.3 s

$$\begin{aligned}\text{Kinematic Viscosity} &= \text{Efflux time}(s) \times \text{viscosity constant} \left(\frac{\text{mm}^2}{s^2} \right) \\ &= 10.3 \times 0.1098 \\ &= 5.06 \left(\frac{\text{mm}^2}{s} \right)\end{aligned}$$

Table 4.3: The average result of biodiesel properties from *Moringa oleifera* seed oil and palm oil.

Properties	MOME	POME	ASTM D6751	EN 14214
Cetane number	66.7	59.5	47	51
Kinematic viscosity (mm²/s; 40° C)	4.8	5.04	1.9–6.0	3.5–5.0
Cloud point (°C)	18	16	-	-
Pour point (°C)	12	7	-	-
Flash point (°C)	165	160	130	120
Density (kg/m³)	875	890	870-900	860-900

MOME - *Moringa oleifera* Methyl Ester

POME - Palm Oil Methyl Ester

ASTM- American Society for Testing and Materials

EN - European Standard

5 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on result in Table 1, best solvent in *Moringa oleifera* oil extraction was n-hexane which yield about 34.5% of oil. The biodiesel properties of *Moringa oleifera* oil and palm oil such as cetane number were 66.7 and 59.5, kinematic viscosity were 4.8mm²/s and 5.03 mm²/s, cloud point were 18°C and 16°C, pour point 12°C and 7°C, flash point was 162°C and 155°C, and density 875kg/m³ and 890kg/m³. After being compared between biodiesel properties produced from *Moringa oleifera* oil and palm oil, a better biodiesel can be derived from *Moringa oleifera* kernels and possess a high possibility to become a main feedstock in the production of biodiesel with a competitive price. Biodiesel from *Moringa oleifera* also will help to prevent foods crisis, palm oil-based foods price hiking and reduce the tension to palm oil producer. Moreover, this study will help to start commercialise the plantation of *Moringa oleifera* in Malaysia thus generating sustainable income.

5.2 Recommendations

There are few recommendations that must be considered in a future to improve the result and product which are:

- i. The different methods of extraction can be used to maximise the oil produced.
- ii. Further study can be done using different catalyst.
- iii. Crude palm oil must be bleached before the transesterification process to produce a fine quality of biodiesel.
- iv. The extracted oil must be centrifuged before proceed with transesterification process to prevent any impurities from affecting the process.
- v. The raw materials, which can be vegetable oils, animal fats, or recycled greases, used in the production of biodiesel contain triglycerides, free fatty acids, water, and other contaminants in various proportions. Some crude vegetable oils contain phospholipids that need to be removed in a degumming step. Phospholipids can produce lecithin, a commercial emulsifier. Therefore, degumming must be carried out to remove the contaminants.
- vi. Recover the glycerol produced as the refining of glycerol is also important due to its numerous applications in different industrial products such as moisturizers, soaps,

cosmetics, medicines, and other glycerol products (Wen *et al*, 2008 and da Silva *et al*, 2008). It is one of the few products that has a good reactivity on sump oil, and is extremely effective for washing shearing shed floor, so it can be used as a heavy duty detergent and degreaser. Moreover, glycerol can even be fermented to produce ethanol, which means more biofuel can be produced (Whittington, 2006).

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APPENDICES

APPENDIX A.1

Biodiesel properties from *Moringa oleifera* seed oil and palm oil.

Properties	<i>Moringa oleifera</i> biodiesel				Palm oil biodiesel			
	1	2	3	Average	1	2	3	Average
Cetane number	66.7	66.8	66.7	66.7	59	59.5	60	59.5
Kinematic viscosity (mm²/s; 40° C)	4.8	4.7	4.8	4.8	5.06	5.0	5.06	5.04
Cloud point (°C)	18	18	18	18	16	16	16	16
Pour point (°C)	12	12	12	12	7	7	7	7
Flash point (°C)	165	164	165	165	159	160	160	160
Density (kg/m³)	875	875	875	875	890	890	891	890

Calculation for kinematic viscosity:

Viscosity constant : 0.4899mm²/s

$$\begin{aligned}
 \text{Kinematic Viscosity} &= \text{Efflux time}(s) \times \text{viscosity constant} \left(\frac{\text{mm}^2}{s^2} \right) \\
 &= 10.3 \times 0.4899 \\
 &= 5.06 \left(\frac{\text{mm}^2}{s} \right)
 \end{aligned}$$

APPENDIX A.2

MORINGA OLEIFERA



Appendix A.2.1: *Moringa oleifera* tree



Appendix A.2.2: *Moringa oleifera* pods



Appendix A2.3: Collected *Moringa oleifera* pods



Appendix A.2.4: Inside the pod



Appendix A.2.5: *Moringa oleifera* Seeds



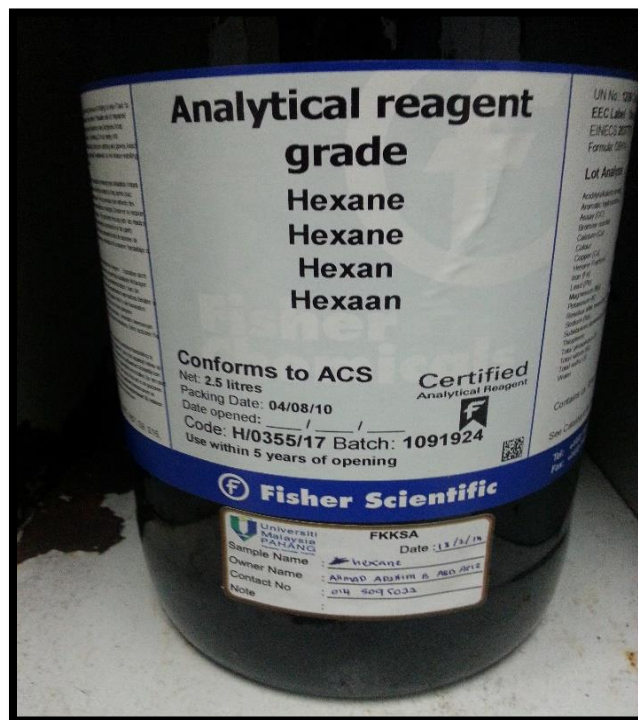
Appendix A.2.6: *Moringa oleifera* kernels



Appendix A.2.7: Removing light wooden shell to get *Moringa oleifera* kernels

APPENDIX A.3

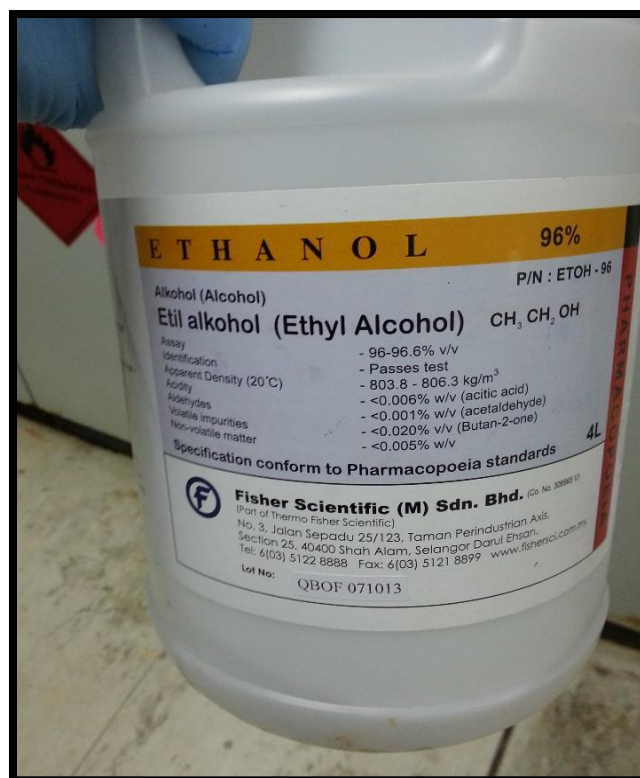
OIL EXTRACTION



Appendix A.3.1: Hexane used as solvent in oil extraction process



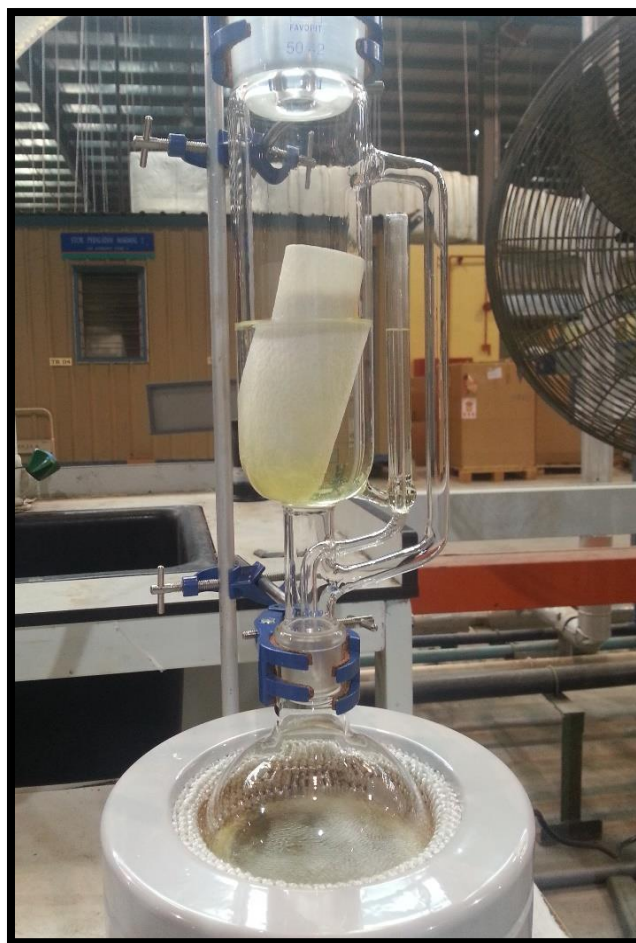
Appendix A.3.2: Methanol used as solvent in oil extraction process



Appendix A.3.3: Ethanol used as solvent in oil extraction process



Appendix A.3.4: Oil extraction process using Soxhelt Extractor



Appendix A.3.5: Solvent was filling the extraction tube



Appendix A.3.6: Separating oil from solvent using rotary evaporator



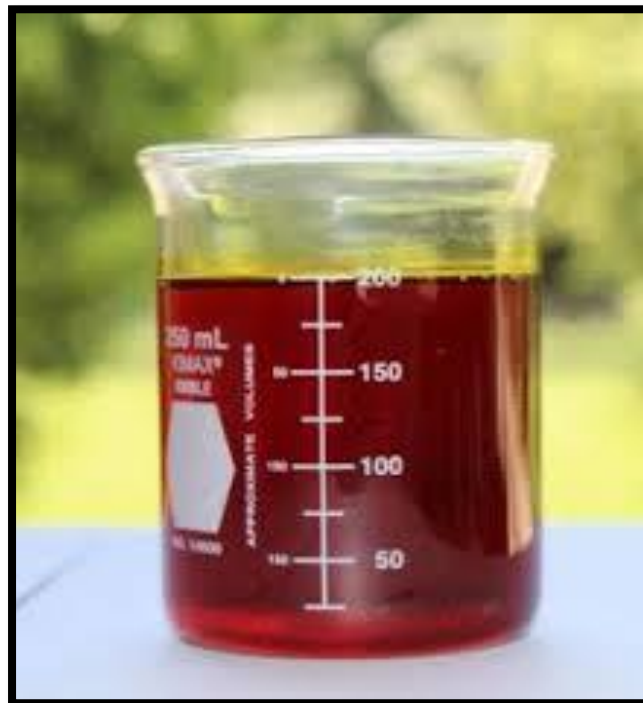
Appendix A.3.7: Recovered *Moringa Oleifera* oil

APPENDIX A.4

CRUDE PALM OIL PRETREATMENT



Appendix A.4.1: Crude palm oil before being centrifuged



Appendix A.4.2: Crude palm oil after being centrifuged



Appendix A.4.3: Crude palm oil after being centrifuged



Appendix A.4.4: Impurities removed from crude palm oil



Appendix A.4.5: Cleaned crude palm oil



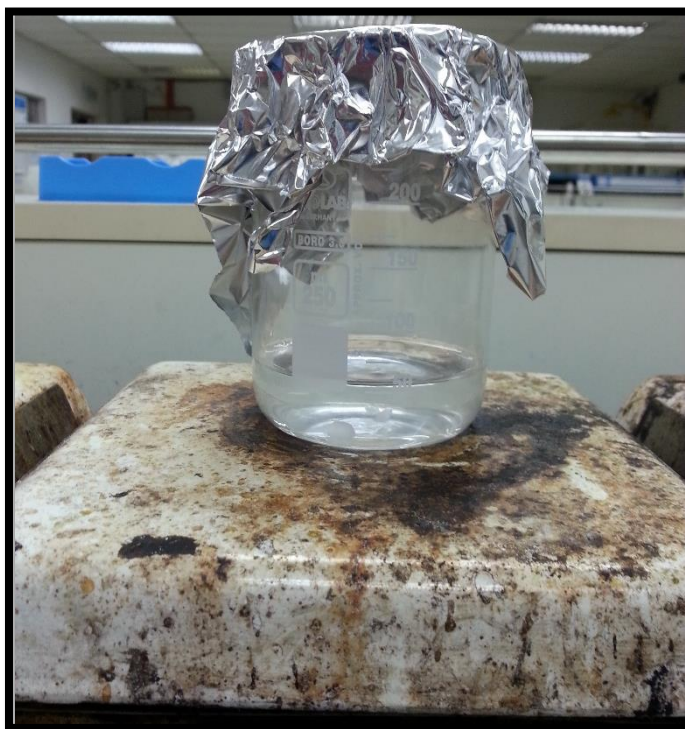
Appendix A.4.6: Fuller's Earth used to bleached crude palm oil



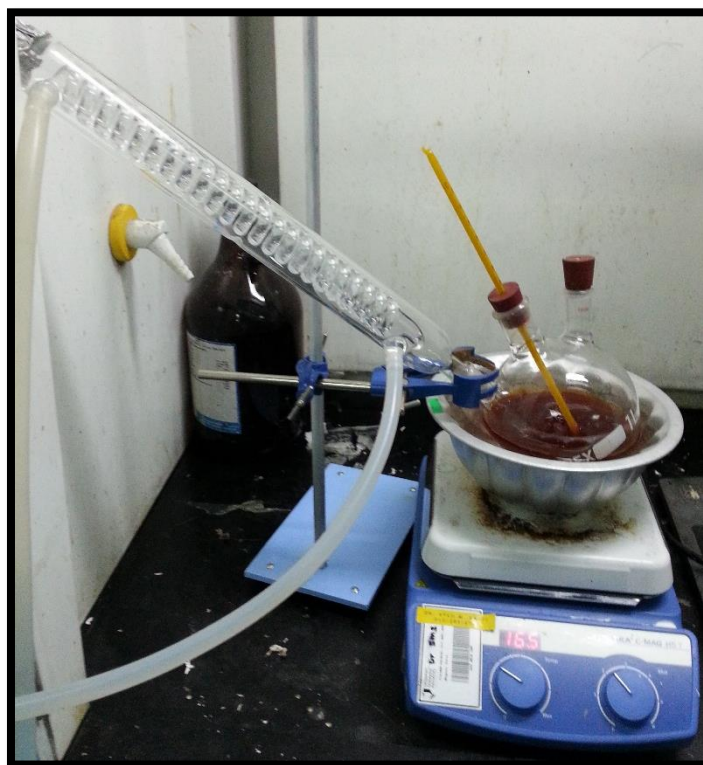
Appendix A.4.7: Bleached crude palm oil

APPENDIX A.5

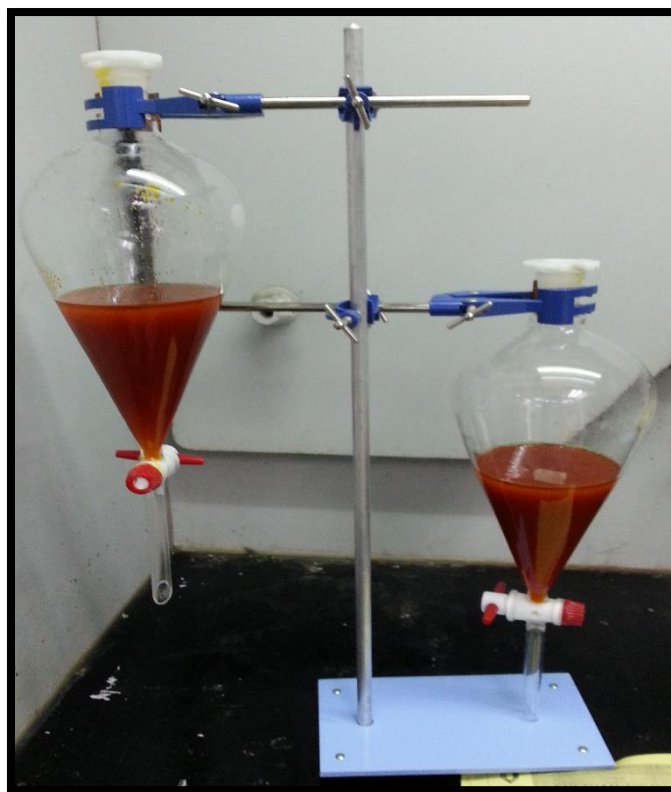
TRANSESTERIFICATION PROCESS



Appendix A.5.1: Preparation of catalyst



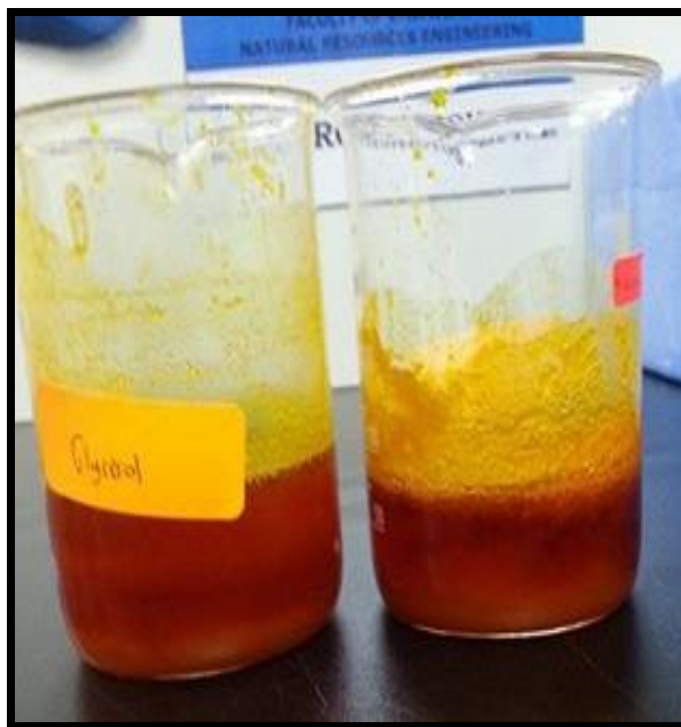
Appendix A.5.2: Experiment setup for transesterification process



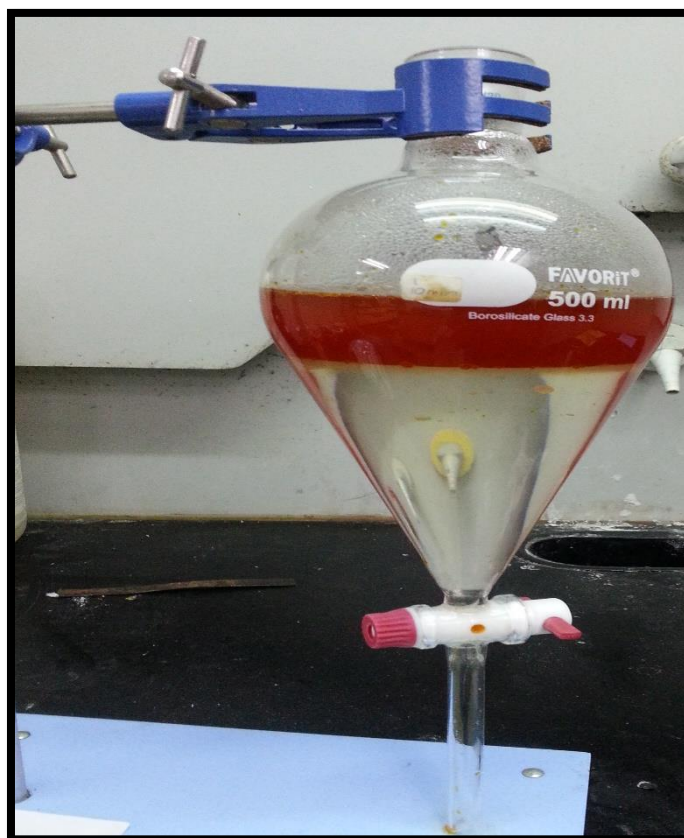
Appendix A.5.3: Settling process (*Palm Oil*)



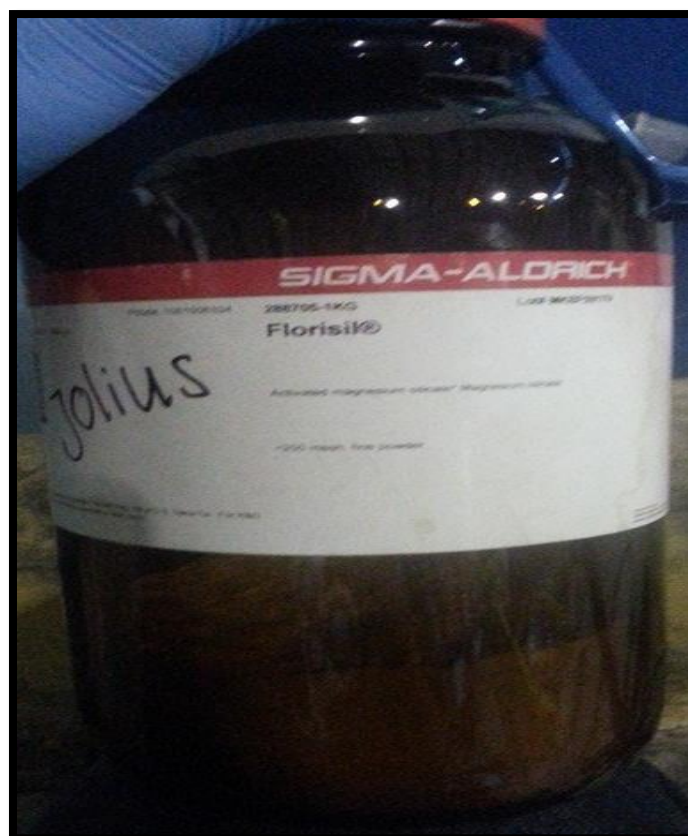
Appendix A.5.4: Settling process (*Moringa oleifera* Oil)



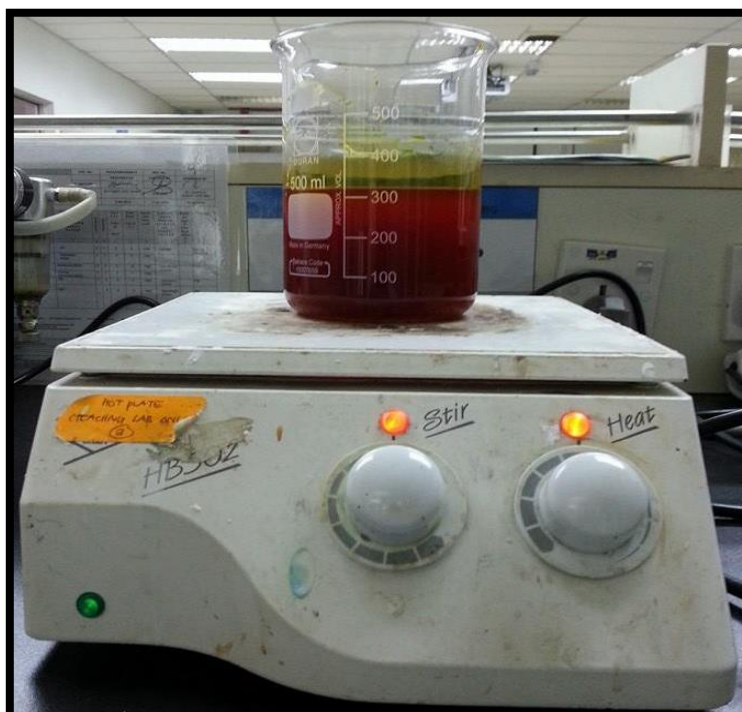
Appendix A.5.5: Glycerol produced from transesterification process



Appendix A.5.6: Washing process



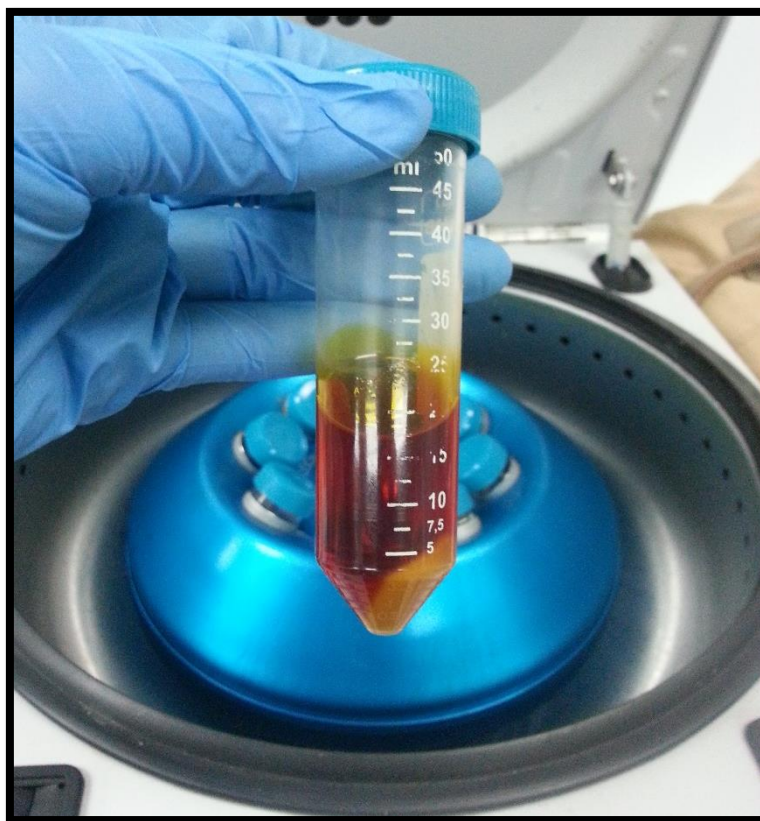
Appendix A.5.7: Florisil used to remove excess water



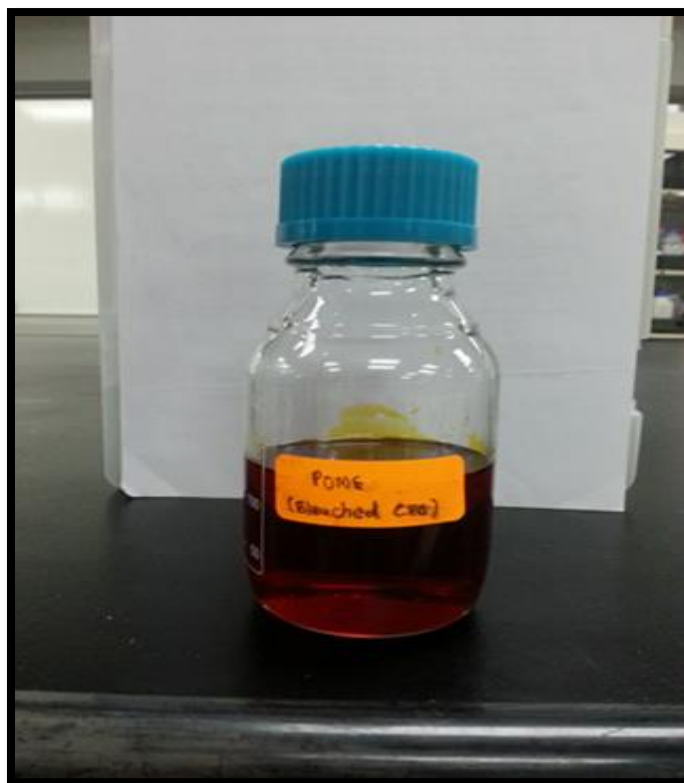
Appendix A.5.8: Stirring biodiesel with florosil to remove excess water



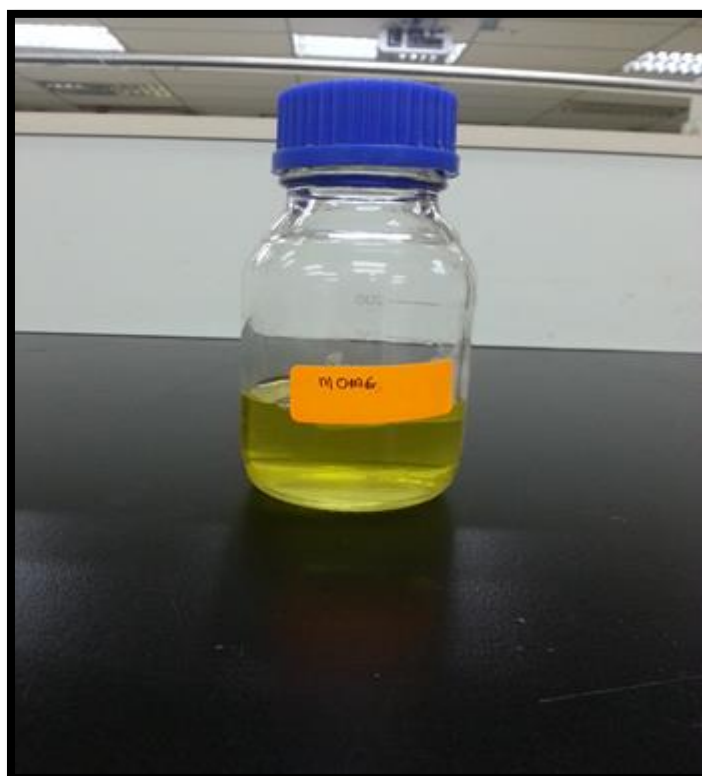
Appendix A.5.9: Centrifuge machine used to separate between florosil and biodiesel



Appendix A.5.10: Biodiesel after being centrifuged



Appendix A.5.11: Palm Oil Biodiesel



Appendix A.5.12: *Moringa oleifera* Biodiesel

APPENDIX A.6

PROPERTIES DETERMINATION- CETANE NUMBER



Appendix A.6.1: Shatox SX-100M Portable Octane/Cetane Analyzer



Appendix A.6.2: Container used to place sample to be analyzed

APPENDIX A.7

PROPERTIES DETERMINATION- KINEMATIC VISCOSITY



Appendix A.7.1: Cannon–Fenske viscometers (number 350)



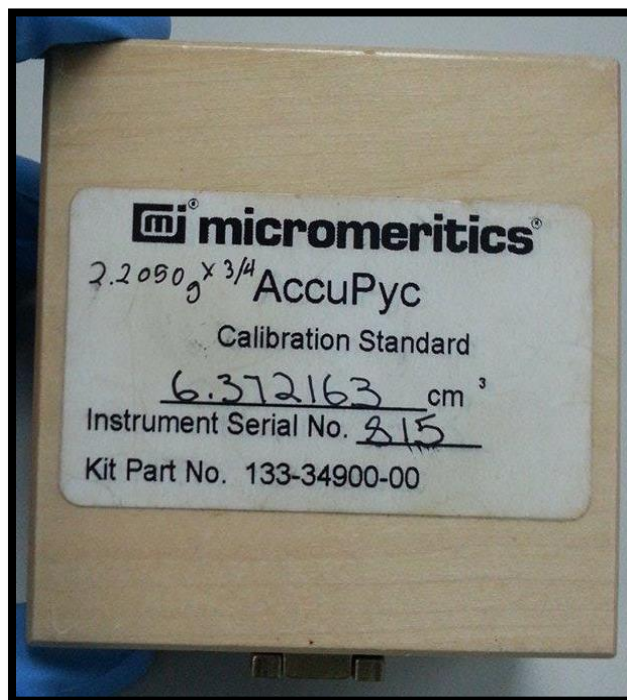
Appendix A.7.2: Viscometer bath



Appendix A.7.3: Hand pump used to pump the biodiesel up to starting point of the viscometer

APPENDIX A.8

PROPERTIES DETERMINATION- DENSITY



Appendix A.8.1: Type of chamber used



Appendix A.8.2: Chamber used to place the sample to be analysed



Appendix A.8.3: Gas Pycnometer

APPENDIX A.9

PROPERTIES DETERMINATION- CLOUD AND POUR POINT



Appendix A.9.1: Test jar containing sample



Appendix A.9.2: Test jar containing sample with thermometer



Appendix A.9.3: K46100 Cloud Point & Pour Point Apparatus



Appendix A.9.4: Test jars with sample inside the K46100 Cloud Point & Pour Point Apparatus

APPENDIX A.10

PROPERTIES DETERMINATION- FLASH POINT



Appendix A.10.1: Pensky-martens flash point – automatic NPM 440 (Normalab, France).