BIODIESEL PRODUCTION FROM *MORINGA OLEIFERA* SEEDS USING HETEROGENEOUS ACID AND ALKALI CATALYST

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AQILAH BINTI YAHYA

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
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JULY 2013

SUPERVISOR'S DECLARATION

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).

Signature :
Name of main supervisor : DR. EMAN N. ALI
Position : SENIOR LECTURER
Date : JULY 2013

STUDENT'S DECLARATION

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.

Signature :
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ID Number : KC09021
Date : JULY 2013

This thesis is dedicated to my parents and my siblings.

For their endless love, support and encouragement

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First and foremost, I would like to say Alhamdulillah for the strength that keeps me standing and for the hope that keeps me believing that this report would be done without any difficulties.

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ABSTRACT

Due to the increasing energy demand and pollution problems caused by the use of fossil fuels, it has become necessary to develop alternative fuels as well as renewable sources of energy. The use of biodiesel as a substitute for conventional diesel has been of great interest. This is because biodiesel is biodegradable, non-toxic, renewable, and has low emission of carbon oxide, sulphur dioxide, particulates and hydrocarbons as compared to conventional diesel. Therefore, this study is conducted to investigate the possible production of biodiesel by using *Moringa oleifera* seeds oil through heterogeneous (acid and alkali) catalyst process. *Moringa oleifera* seeds oil can be used for biodiesel production by transesterification using calcium oxide (CaO) as an alkaline catalyst followed by esterification by using ferric sulphate catalyst. A sample of 50 mL oil was poured into the 3-neck round-bottom glass flask. Carefully, the methanol was poured into the oil with ratio 6:1, 12:1 and 18:1 for both alkali-catalyzed transesterification and acid-catalyzed esterification process. The catalyst concentrations, time reaction, agitation speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively. As a result, the methyl esters (biodiesel) produced from *Moringa oleifera* seeds oil exhibits a high yield which is 48 mL (96%) and 45 mL (90%) for both alkali-catalyzed transesterification and acid-catalyzed esterification process by using methanol to oil ratio of 18:1, and catalyst concentrations, time reaction, agitation speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively. As a conclusion, biodiesel can be produced from *Moringa oleifera* seeds oil as an alternative fuels as well as renewable sources of energy for future use.

ABSTRAK

Disebabkan oleh permintaan tenaga yang semakin meningkat dan masalah pencemaran yang disebabkan oleh penggunaan bahan api fosil, telah menjadi keperlu untuk membangunkan bahan api alternatif serta sumber-sumber tenaga yang boleh diperbaharui. Penggunaan biodiesel sebagai pengganti diesel konvensional kini menjadi kepentingan yang besar. Ini kerana biodiesel adalah mesra alam, tidak toksik, boleh diperbaharui, dan mempunyai kadar karbon oksida yang rendah, sulfur dioksida, habuk dan hidrokarbon berbanding diesel konvensional. Oleh itu, kajian ini dijalankan untuk menyiasat pengeluaran biodiesel dengan menggunakan biji *Moringa oleifera* melalui asid heterogen dan pemangkin proses alkali. Biji *Moringa oleifera* boleh digunakan untuk pengeluaran biodiesel dengan menggunakan sulfat ferik pemangkin esterification diikuti dengan transesterification menggunakan kalsium oksida (CaO) sebagai pemangkin alkali. Satu sampel 50 mL minyak telah dicurahkan ke dalam 3-leher bulat-kelalang kaca. Dengan berhati-hati, tuangkan methanol ke dalam minyak mengikut nisbah 6:1, 12:1 dan 18:1. Kepekatan pemangkin, reaksi masa, kelajuan pergolakan dan suhu telah ditetapkan pada 1wt%, 90 minit, 200 rpm dan 70^{o}C. Hasilnya, methyl ester (biodiesel) dihasilkan dengan menggunakan biji *Moringa oleifera* menunjukkan hasil yang tinggi dengan menggunakan methanol kepada nisbah minyak 1:18, manakala kepekatan pemangkin, masa tindak balas, kelajuan pergolakan serta suhu ditetapkan pada 1wt%, 90 minit, 200 rpm dan 70^{o}C. Kesimpulannya, biodiesel boleh dihasilkan daripada biji *Moringa oleifera* sebagai bahan api alternatif begitu juga untuk sumber-sumber tenaga yang diperbaharui untuk kegunaan masa depan.

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LIST OF ABBREVIATIONS

bl	barrel
mbls/d	million barrels per day
rpm	round per minute
wt.%	weight percent

Greek

v_l	kinematic viscosity
ρ	density

LIST OF ABBREVIATIONS

ASPO	Association for the Study of Peak Oil
ASTM	American Society for Testing and Materials
CERA	Cambridge Energy Research Associates
CFR	Code of Federal Regulations
EN	European Standard
EWG	Energy Watch Group
FFA	Free fatty acid
IEA	International Energy Agency
IHS	International Handling Service
MOME	*Moringa oleifera* Methyl Ester
MW	Megawatt
ODAC	Oil Depletion Analysis Centre
O&GJ	Oil and Gas Journal
OPEC	Organization of the Petroleum Exporting Countries

1 INTRODUCTION

1.1 *Motivation of Study*

With the vast world population and energy demand problems that keep increasing each year, it has become necessary to develop alternative fuels as well as renewable sources of energy. Kjärstad and Johnsson (2008) concluded that global supply of oil probably will continue to be tight, both in the medium term as well as in the long term mainly as a consequence of above-ground factors such as investment constraints, geological tensions, limited access to reserves and mature super-giant fields. In the Middle East, oil demand grew by 3.0% per annum between 2000 and 2007, while corresponding growth averaged 2.7% per annum in Africa and Asia-Pacific, rising global demand by 6.3 million barrels per day (mbls/d). In total, these three regions accounted for more than 70% of total worldwide increase in oil demand between 2000 and 2007 (Kjärstad and Johnsson, 2008). In Malaysia, it can be seen that the energy demand increases rapidly as the energy demand increase almost 20% within the last 3 years (from 1999 to 2002) and the energy demand is further expected to increase to 18,000 megawatt (MW) by the year 2010 (Mohamed and Lee, 2005).

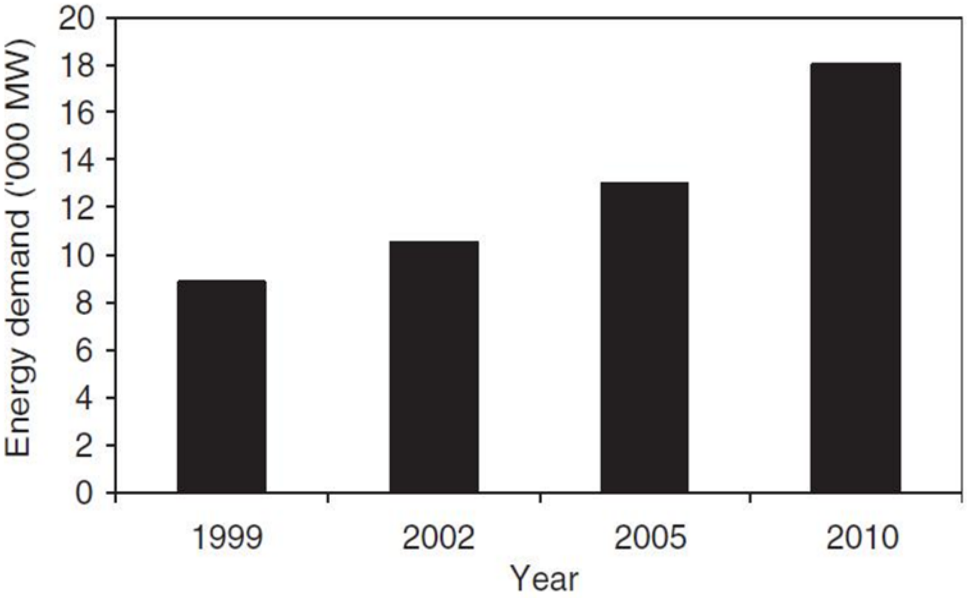

Figure 1.1: Energy demand in Malaysia. Sources: Thaddeus (2002) and UK Trade & Investment (2003).

Nevertheless, global liquids fuel demand is expected to increase by 1.3-1.4% in average per annum up to 2030 reaching between 116 and 118 (mbls/d) in 2030 (International Energy Agency, 2006). Association for the Study of Peak Oil and Gas (ASPO, 2007), Oil Depletion Analysis Centre (ODAC, 2006), and Energy Watch Group (EWG, 2007) claimed that oil production may have already peaked or will soon peak due to limited resources.

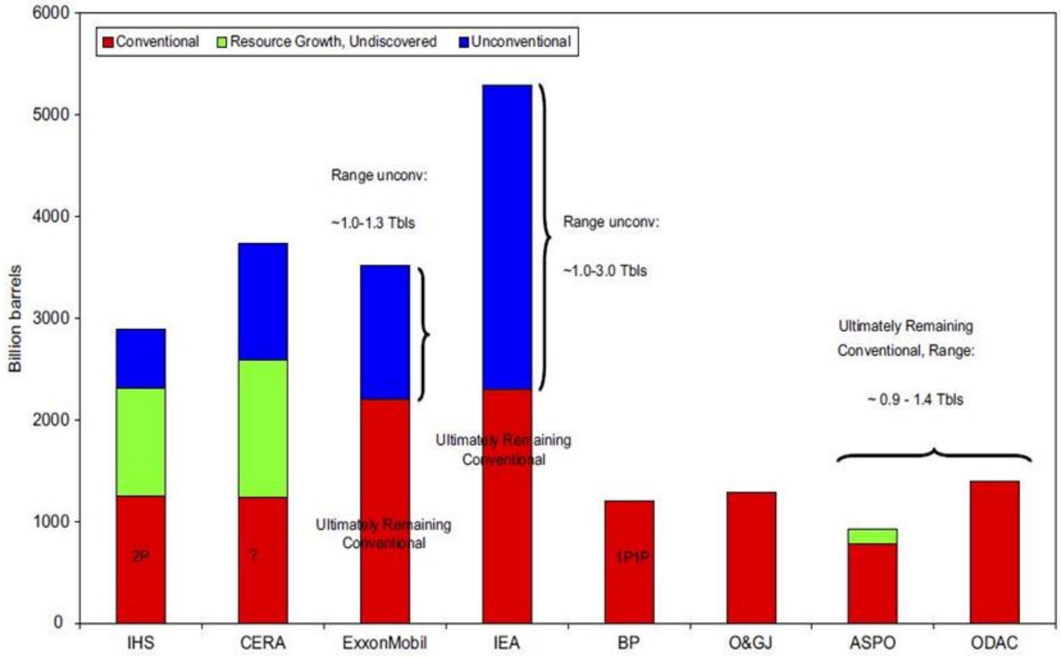

Figure 1.2: Various estimates of proven reserves and remaining oil resources by the end of 2005 (International Energy Agency end of 2003, Association for the Study of Peak Oil end of 2003). Sources: Oil Depletion Analysis Centre 2006. *International Handling Service (IHS), Cambridge Energy Research Associates (CERA), International Energy Agency (IEA), Oil and Gas Journal (O&GJ), Association for the Study of Peak Oil (ASPO) and Oil Depletion Analysis Centre (ODAC).

Global oil reserves have received much attention in the recent years with the concept of peak oil being widely discussed in media. In order to determine when oil production will peak in a specific oil field, basin, country or globally, one will needs as well as to know how production will evolve over time, thus it has become necessary to develop alternative fuels as well as renewable sources of energy. Indeed, new discoveries have fallen sharply since the 1960s and discoveries in the last decade (1993-2002) have only replaced half the oil production, the declining trend seems to continue with discoveries in 2004 and 2005 being noted as the lowest since the World War II, (International Energy Agency, 2006). Kjärstad and Johnsson (2008) stated that, the best examples of countries having large fluctuations in annual oil production with several intermediately peaks are found among the Organization of the Petroleum Exporting Countries (OPEC) members, e.g. Iran, Iraq, Nigeria and Saudi Arabia. In Malaysia, oil reserves have declined in recent years and the oil production fell to 693,000 billion barrels per day (bbls/d) in 2008, a 13% decrease from 2006 (Tick et al., 2009).

Hence, to overcome the challenges of fossil fuel resources depletion and the global warming threats and climate changes, the world must look for alternative, renewable sources of energy. One of the distinction renewable resources of energy is biodiesel. Nowadays, biodiesel has receiving its own attention as alternative source because it is non-toxic, biodegradable and renewable fuel that can help our world decrease air pollution and global warming. Biodiesel has low emission of carbon monoxide (CO), sulphur dioxide (SO_2), particulates and hydrocarbons as compared to conventional diesel (Kafuku et al., 2010).

According to Kafuku and Mbarawa (2009), biodiesel is the mono alkyl ester of long chain fatty acids derived from a renewable lipid feedstock, such as vegetable oil or animal fat after the process of transesterification with the aim of reducing the viscosity of that lipid feedstock. Transesterification is a chemical process usually used to produce biodiesel in which triglycerides are allowed to react with an alcohol (mostly methanol) under acidic or basic catalytic conditions. It is a technically competitive and environmentally friendly alternative to conventional petrodiesel fuel for use in compression-ignition (diesel) engines (Rashid et al., 2007). Moreover, biodiesel possesses inherent lubricity, a relatively high flash point, and reduces most regulated exhaust emissions in comparison to petrodiesel. The use of biodiesel reduces the dependence on imported fossil fuels, which continue to decrease in availability and affordability (Rashid et al., 2007).

Vegetable oils for biodiesel production vary considerably with location according to climate and feedstock availability. Normally, the most abundant vegetable oil in a particular region is the most common feedstock. Thus, rapeseed and sunflower oils are predominantly used in Europe; palm oil predominates in tropical countries, and soybean oil and animal fats in the USA (Knothe et al., 2005). In this research, the examination of the *Moringa oleifera* becomes fundamental as a potential biodiesel production. The Moringaceae is a single-genus family of oilseed trees with 14 known species (Rashid et al., 2007). *Moringa oleifera* is one of tree species which ranges in height from 5 to 10 meter, is the most widely known and utilized (Morton 1991). *Moringa oleifera*, indigenous to sub-Himalayan regions of northwest India, Africa, Arabia, Southeast Asia, the Pacific and Caribbean Islands and South America, is now distributed in the Philippines, Cambodia and Central and North America (Morton, 1991). It thrives best in a tropical insular climate and is plentiful near the sandy beds of rivers and streams (Council of Scientific and Industrial Research, 1962). The fast growing, drought-tolerant *Moringa oleifera* can tolerate poor soil, a wide rainfall range (25 to 300+ cm per year), and soil pH from 5.0 to 9.0 (Palada and Changl, 2003). When fully mature, dried seeds are round or triangular shaped, and the kernel is surrounded by a lightly wooded shell with three papery wings (Council of Scientific and Industrial Research, 1962).

Moringa oleifera seeds in Figure 1.3 contain between 33 and 41% w/w of vegetable oil (Sengupta and Gupta, 1970). Many authors investigated the composition of *Moringa oleifera*, including its fatty acid profile (Anwar and Bhanger 2003; Anwar et al., 2005; Sengupta and Gupta 1970; Somali and Bajneid, 1984) and showed that *Moringa oleifera* oil is high in oleic acid (more than 70%). *Moringa oleifera* is commercially known as “ben oil” or “behen oil”, due to its content of behenic (docosanoic) acid, possesses significant resistance to oxidative degradation (Lalas and Tsaknis, 2002), and has been extensively used in the enfleurage process (Council of Scientific and Industrial Research, 1962). *Moringa oleifera* oil (among others), has a good potential for biodiesel production (Azam et al., 2005).

Figure 1.3: *Moringa oleifera* seeds.

1.2 Problem Statement

There is an increasing concern that global oil production is close to peak and that peak will be followed by a rapid decline in production of the conventional diesel. Over the last few years the oil price has risen to new record levels, between 2000 and 2003 the oil price remained roughly constant around US$25/barrel (bl) and global demand grew by around 1% annually apart from in 2003 when demand increased by 1.8% (Kjärstad and Johnsson, 2008). In 2003, Malaysia contains proven oil reserves of 3.0 billion barrels, while the production has been relatively stable at around 700,000 barrels per day (International Energy Agency, IEA 2003) and if the production rate is maintained at 1.250,000 (mbls/d), the ratio between reserve and production is 12, indicating that within 12 years, Malaysia’s oil will be exhausted (Tick et al., 2009). While in 2006, Malaysia’s oil output declined with average production for 2006 stood at 798,000 (bbl/d), down 7% from 2005 (Tick et al., 2009).

Therefore, the key factor for preserving the reserves oil globally is to develop alternative fuels as well as renewable sources of energy such as biodiesel. It is often being claimed by the “Peak Oil” community that most countries have passed their peak production and consequently that there are fewer and fewer countries left to ascertain an increasing global oil production in future. Thus, *Moringa oleifera* seeds will be the target in this study as alternative and renewable sources of energy to replace the conventional diesel as well as to overcome the challenges of fossil fuel resources depletion.

1.3 Objectives

The following are the objectives of this research:

i. To produce biodiesel from *Moringa oleifera* seeds by using heterogeneous method (acid and alkali catalyst).

i. To identify optimum conditions for biodiesel production from *Moringa oleifera* seeds to get optimum yield.

1.4 Scope of This Research

This research is an experimental study in production of biodiesel using *Moringa oleifera* oil as the feedstock. In order to achieve these research objectives, the methanol to oil ratio of 6:1, 12:1 and 18:1 were used. While the catalyst concentrations, time reaction, stirring speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively.

1.5 Organisation of This Thesis

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides a description of the applications and general study of biodiesel production in the world. A general description on the production of biodiesel by using *Moringa oleifera* seeds oil, as well as the characteristics of *Moringa oleifera* tree itself. This chapter also provides a brief discussion of the advanced experimental techniques available for producing biodiesel from *Moringa oleifera* seeds oil.

Chapter 3 discuss the steps of samples preparation, the parameters that were used to determine the best yield, optimum ratio of methanol:oil and physical characteristics of the biodiesel produced.

Chapter 4 presents all the results obtained from the three sets of experiments for alkali-catalyzed transesterification and acid-catalyzed esterification process with different methanol to oil ratios (6:1, 12:1, 18:1), while the catalyst concentrations, time reaction, stirring speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively. The tests and analysis is done to sample of highest biodiesel yield product. The main aim is to find the physical properties of the biodiesel produced from *Moringa oleifera* seeds oil.

2 LITERATURE REVIEW

2.1 Biodiesel As Renewable Sources of Energy

Biodiesel is defined as the fatty acid alkyl esters of vegetable oil, animal fats or waste oils. It is a technically competitive and environmentally friendly alternative to conventional petrodiesel fuel for use in compression-ignition (diesel) engines (Rashid et al., 2007). Biodiesel is biodegradable, renewable, non-toxic, possesses inherent lubricity, a relatively high flash point, and reduces most regulated exhaust emissions in comparison to petrodiesel. The advantage of biodiesel is that it reduces the dependences on imported fossil fuels, which continue to decrease in availability and affordability. Since the demand for global petroleum has been increasing, it has become our obligations to develop alternative fuels as well as renewable sources of energy.

2.2 Transesterification Process

In biodiesel production, tranesterification is the reaction of fats or oils with alcohols to form biodiesel. There are two methods of transesterification generally, the first method employs a catalyst, second method is non-catalyst option such as supercritical process, and co-solvent systems (Karmakar et al., 2009). Application of transesterification by using heterogeneous catalysts appears promising because they can simplify the production and purification processes, decrease the amount of basic waste water, down size the process equipment, and reduce the environmental impact and process cost (Kawashima et al., 2009). Methanol and ethanol are the two main light alcohols used for tranesterification process. Calcium oxide (CaO) has attracted much attention for tranesterification reaction since it has high basic strength and less environmental impact due to its low solubility in methanol and can be synthesized from cheap sources (Zabeti et al., 2009). Hence, catalyst is essential as alcohol to scarcely soluble in oil or fat. It is improved the solubility of alcohol and therefore increases the reaction rate. Tranesterification can be affected by many factors, such as methanol-to-oil molar ratio, catalyst amount, reaction temperature, and reaction time (Zhang et al., 2009).

The transesterification reaction is represented by the general equation as in the equation (1). Transesterification is one of the reversible reactions and proceeds essentially by mixing the reactants. However, the presence of a catalyst (a strong acid or base) will accelerate the conversion.

$$\text{Triglycerides + Methanol} \quad \overset{\text{Catalyst}}{\rightleftarrows} \quad \text{Glycerol + Methyl Ester} \qquad \text{------- (1)}$$

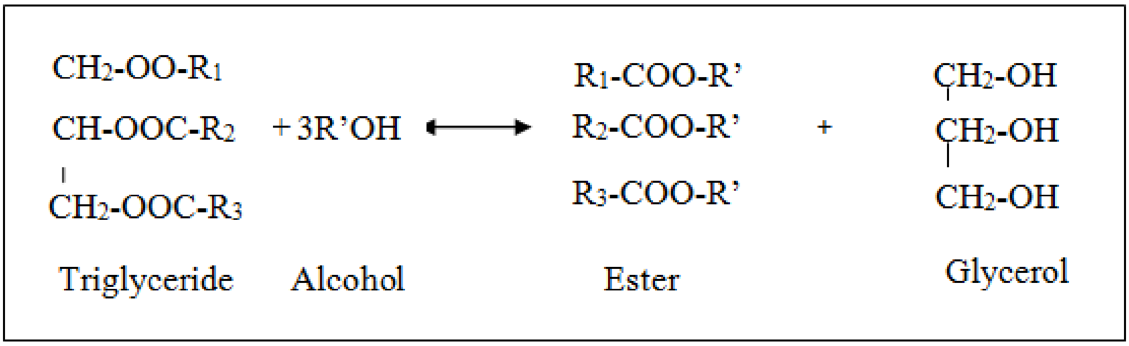

Figure 2.1: Transesterification of triglycerides with alcohols

Transesterification of triglycerides with methanol and aid of catalyst produce methyl ester and glycerol. The glycerol layer settles down at the bottom of the reaction vessel. The step wise reactions are reversible and a little excess alcohol is used to shift the equilibrium towards the formation of ester. In presence of excess alcohol, the forward reaction is first order reaction and the reverse reaction is found to be second order reaction. It was observed that transesterification is faster when catalyzed by alkali (Freedman et al., 1986).

2.3 Heterogeneous Acid Esterification

Conventional homogeneous acids like solid acid catalysts have many significant advantages such as less corrosion, less toxicity and less environmental problems (Lou et al., 2008). However, the use of these solid acids usually requires high reaction temperature, long reaction time and relatively high pressure (Zhang et al., 2009). Recently, a lot of work has been carried out in relation to solid acid as catalysts for esterification reaction (Zhang et al., 2009). Ferric sulphate was used as a solid acid catalyst to catalyze the esterification of free fatty acid in waste cooking oil and showed a high catalytic activity (Patil et al., 2010). This is because; ferric sulphate has a lower price and could be easily recovered due to its very low solubility in oil (Zhang et al., 2009).

2.4 Alkali Catalyst

Alkaline catalyzed production process of biodiesel is the process of transesterification of a fat or oil triglyceride with an alcohol to form esters and glycerol, in the presence of an alkali as a catalyst. The most commonly prepared esters are methyl esters, because methanol is easily available. Alkali catalysts, such as sodium or potassium hydroxide, and sodium or potassium methoxide are the most common and are preferred due to their high yields. The base-catalyzed process is relatively fast but is affected by water content and free fatty acids of oils or fats. Free fatty acids can react with base catalysts to form soaps and water. Soap not only lowers the yield of alkyl esters but also increases the difficulty in the separation of biodiesel and glycerol and also in the water washing because of the formation of emulsion. It was found that methoxide catalysts give higher yields than hydroxide catalysts, and potassium-based catalyst give better biodiesel yield than sodium-based catalysts (Karmakar et al., 2009).

2.5 Properties of Biodiesel Feedstock

Fats and oils are primarily water-insoluble, hydrophobic substances in the plant and animal kingdom that are made up of one mole of glycerol and 3 mole of fatty acid and are commonly known as triglycerides (Sonntag, 1979). The fuel properties of biodiesel dependents on the amount of each fatty acid present in the feedstock (Karmakar et al., 2009).

2.5.1 Free fatty acid content

Free fatty acid (FFA) content is the amount of fatty acid (wt%) in oil which is not connected to triglyceride molecule. Heating of oil can cause breakage of long carbon chain and formation of FFAs (Karmakar et al., 2009). During transesterification process, free fatty acids react with alkali, and form soaps and water both of which must be removed during ester purification process because free fatty acid attracts water in their hygroscopic nature (Karmakar et al., 2009). Fatty acid composition of *Moringa oleifera* is tabulated in Table 2.1 (Karmakar et al., 2009).

Table 2.1: Fatty acid composition of the *Moringa oleifera* oil.

Fatty acid[a]	
Palmatic (16:0)	7
Palmitoleic (16:1)	2
Stearic (18:0)	4
Oleic (18:1)	78
Linoleic (18:2)	1
Linolenic (18:3)	–[b]
Arachidic (20:0)	4
Behenic (22:0)	4

*a = experimental results
*b = this can indicate traces (less than1.0%) or absence

2.5.2 Heat content

The calorific content is the energy content of the oil and the energy of biodiesel depends on the feedstock oil (Karmakar et al., 2009). Fuels with more unsaturation generally have lower energy (on a weight basis) while fuels with greater saturation have higher energy content. Denser fuels provide greater energy per gallon and since fuel is sold volumetrically, the higher the density, the greater the potential of energy (Karmakar et al., 2009).

2.6 Properties of Moringa oleifera Methyl Esters

The properties of the *Moringa oleifera* methyl esters are summarised below. This research was done to confirm the results acording to Kafuku and Mbarawa, (2009).

2.6.1 Cetane Number

The properties of the *Moringa oleifera* methyl esters (MOME) are summarised in Table 2.2. MOME showed a high *cetane* number of 62.12. The *cetane* number was determined using a Waukesha Code of Federal Regulations (CFR) F-5 engine as specified by ASTM 613 (American Society for Testing and Materials) (Kafuku and Mbarawa, 2009).

2.6.2 *Viscosity*

The viscosity of MOME in the Table 2.2 is 4.91 mm^2/s thus; it meets the requirement of the biodiesel ASTM D6751 and EN 21414 standards which prescribe that the viscosity ranges should lie between 1.9-6.0 and 3.5-5.0 mm^2/s, respectively (Kafuku and Mbarawa, 2009).

2.6.3 *Cloud and Pour Point*

One of the major problems associated with the use of biodiesel is its poor temperature flow property, measured in terms of cloud point, and pour point temperature. MOME has high values of cloud and pour points of 10 ^oC and 3 ^oC, repectively (Kafuku and Mbarawa, 2009).

Table 2.2: Properties of *Moringa oleifera* methyl esters.

Property	Unit	Value	ASTM D6751	EN 14214
Cetane number		62.12	>47	>51
Kinematic viscosity at 40°C	mm^2/s	4.91	1.9-6.0	3.5-5.0
Cloud point	°C	10	Not specified	Not specified
Pour point	°C	3	Not specified	Not specified
Acid value	mg KOH/g	0.012	<0.80	<0.50
Density at 15°C	kg/m^3	877.5	870-900	860-900
Flashpoint	°C	206	130 min	120 min

*ASTM D6751 (American Society for Testing and Materials).
*EN 14214 (European Norm).
Sources: Kafuku and Mbarawa 2009.

2.6.4 *Density*

The density of biodiesel usually varies between 860 and 900 kg/m^3 according to EN 14214 standard. In many studies, it was observed that the density of biodiesel has not changed a lot, because the densities of methanol and oil are close to the density of the biodiesel produced (Alaptekin and Canaki, 2008). Table 2.2 showed that the density of MEMO is 877.5 kg/m^3 (Kafuku and Mbarawa, 2009).

2.6.5 Flash Point

The flash point temperature of MOME is 206 ^{o}C as stated in the Table 2.2. This value is higher than the minimum requirements for biodiesel standards. The high flash point temperature of the MOME is beneficial safety feature, as this fuel can safely be stored at room temperature (Kafuku and Mbarawa, 2009).

3 MATERIALS AND METHODS

3.1 Introduction

This chapter presents a study about preparing biodiesel from *Moringa oleifera* seeds oil using heterogeneous acid and alkali catalyst. This research intends to achieve two main goals. Firstly, to prepare feedstock of *Moringa oleifera* oil from the seeds. Secondly, to produce biodiesel by transesterification process using calcium oxide (CaO) as an alkaline catalyst followed by esterification process using ferric sulphate-catalyzed.

a) Material

- *Moringa oleifera* seeds.
- Alcohol selection.
- Catalyst selection.

b) Equipments and Glassware

- Hot plate with stirrer.
- 250 mL separating funnel.
- 250 mL 3 necked round-bottom glass flask equipped with a reflux condenser and a thermometer.
- Rotary evaporator.
- Electro thermal soxhlet (ROSS, UK).

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 *Moringa oleifera* seeds. It was collected from Pengkalan Hulu, Perak.

3.2.2 Alcohol selection

Price is the main factor in determining which alcohol will be used as a solvent in the production process. High quality methanol is cheaper than ethanol, therefore it was used on nearly all biodiesel operations. In this experiment, methanol used was purchased from ChemPur Chemicals Sdn Bhd.

3.2.3 Catalyst selection

A catalyst was required to facilitate the reaction between the oil and the alcohol. In conducting this experiment, calcium oxide (CaO) purchased from ChemPur Chemicals Sdn Bhd was chosen because it has high basic strength and less environmental impact due to its low solubility in methanol. CaO is the most widely used as a solid basic catalyst as it presents many advantages such as long catalyst life, high activity and requires only moderate reaction conditions.

3.2.4 Drying agent

Anhydrous sodium sulphate (Na_2SO_4) purchased from Fisher Scientific Chemicals Sdn Bhd was used as a drying agent to remove excess water from the raw *Moringa oleifera* oil because the presence of water will causes saponification of the product.

3.3 Equipments

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

a) Soxhlte extractor: To extract oil from seeds.
b) Three-neck round-bottom glass flask with reflux condenser: To heat and stir the mixtures of methoxide and oil.
c) Hot plate with stirrer: To warm up the water for the reflux condenser.
d) Separating funnel: To separate two layer of glycerine and biodiesel
e) Rotary evaporator: To recover excess methanol and hexane.

3.4 Research Method

3.4.1 Collecting sample

The *Moringa oleifera* seeds used in this study was collected from Pengkalan Hulu, Perak.

3.4.2 Oil extraction experiment

Oil extraction from *Moringa oleifera* seeds was done using hexane as a solvent to the seed powder, to separate oil from seeds using electro thermal soxhlet. The extraction procedure was as follow: weighing 30 gm of *Moringa oleifera* seed powder and setting it in the thimbles of the electro thermal soxhlet extraction chamber. Adding 200 mL of hexane in the heating chamber; evaporating of hexane within three cycles each for 30 minutes to ensure the extraction of oil from seeds (until the hexane become colourless) (Eman et al., 2010). After extraction for 1.5 hour, the solvent was removed at 60 ^{o}C under vacuum using rotary evaporator so that the crude *Moringa oleifera* oil can be collected.

3.4.3 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. 6:1 (300 mL : 50 mL)
ii. 12:1 (600 mL : 50 mL)
iii. 18:1 (900 mL : 50 mL)

In order to have a 1.0 % concentration of calcium oxide (CaO) for every 50 mL of oil used, 0.5 gram of CaO was added in the methanol to produce methoxide solutions. The solutions was prepared in a 250 ml beaker and stirred without heat for about 30 minutes to activate the catalyst.

3.4.4 Experiment of biodiesel production

In this research, three experiments were conducted according to the methanol to oil ratios as shown in the Table 3.1.

Table 3.1: Sets of samples and trial

Ratios (methanol : oil)	Temperature (^oC)	Time (min)
6:1	70	90
12:1	70	90
18:1	70	90

1. ***Alkali-catalyzed transesterification of Moringa oleifera biodiesel***

   A sample of 50 mL oil was poured into a 3-neck round-bottom glass flask equipped with a reflux condenser. The methanol was poured carefully into the oil according to the ratio selected in Table 3.1. While, stirring speed, reaction time, catalyst concentration (calcium oxide) and reaction temperature are fixed at 200 rpm, 90 minutes, 1 wt% and 70 ^oC, respectively. After the reaction completed, calcium oxide (CaO) was removed by filtration and the product was allowed to settle overnight in a separating funnel for separation of biodiesel. The lower glycerol layer in the separating funnel was drawn off. After that, the biodiesel layer was washed four times using hot water (80 ^oC) and dried with enough anhydrous sodium sulphate (Zhang et al., 2009).

2. ***Acid-catalyzed esterification of Moringa oleifera biodiesel***

   The sample from the transesterification process was poured into the 3-neck round-bottom glass flask equipped with a reflux condenser. Again, the methanol was poured carefully into the oil according to the ratio selected in Table 3.1. While, stirring speed, reaction time, catalyst concentration (ferric sulphate) and reaction temperature are fixed at 200 rpm, 90 minutes, 1 wt% and 70 ^oC respectively. After the reaction completed, ferric sulphate was removed by filtration and the product was allowed to settle overnight in a separating funnel for separation of biodiesel. The lower glycerol layer in the separating funnel was drawn off. After that, the biodiesel layer was washed four times using hot water (80 ^oC) and dried with enough anhydrous sodium sulphate (Zhang et al., 2009).

3.4.5 *Product analysis*

After calculating the percentage yield for each sample, the biodiesel of *Moringa oleifera* oil properties were determined. The following tests were being held as follow:

1. Density at 15 ^{o}C (kg/m^{3})
2. Kinematic viscosity at 40 ^{o}C (mm^{2}/s)
3. Cetane number
4. Cloud point (^{o}C)
5. Pour point (^{o}C)

3.4.5.1 *Density*

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 average value.

3.4.5.2 *Kinematic viscosity*

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, USA.

1. A sample of 30 mL biodiesel was pumped into the capillary tubes until it reached the ‘start mark’.
2. The time taken for the oil to flow through the capillary tube can be converted directly to a kinematic viscosity using a sample calculation calibration constant provided for each tube.
3. The experiment was repeated three times to get accurate calibration.

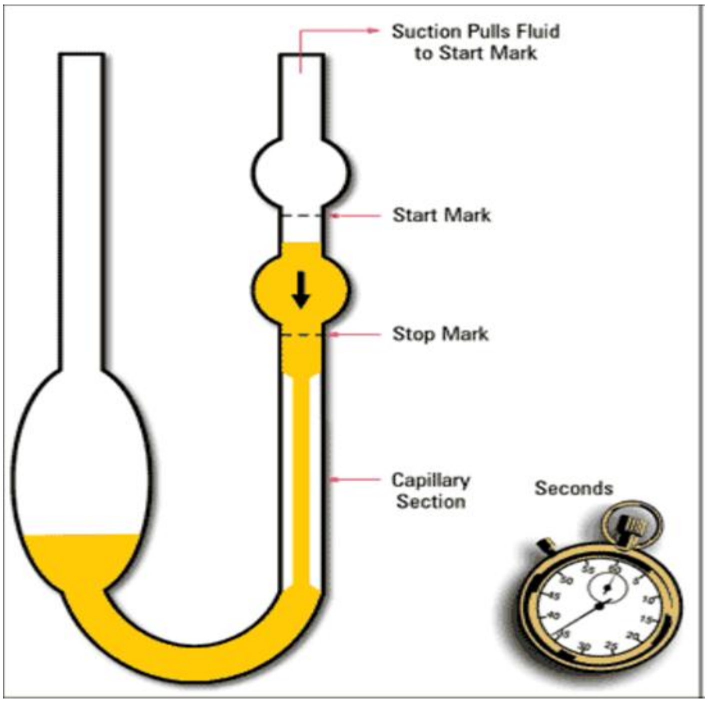

Figure 3.1: Example of capillary tube.

3.4.5.3 Cetane Number

Below are the steps in order to determine *cetane* number of *Moringa oleifera* biodiesel:

1. A sample of 40 mL was needed.
2. The sample was poured slowly into the *cetane* number tester until it indicated label ‘S’ which means *cetane* number was determined based on summer season.
3. Once the indicator displayed ‘S’, the *cetane* number was recorded.
4. The experiment was repeated three times to get the average value.

Figure 3.2: Example of *cetane* number tester

3.4.5.4 Cloud and Pour Point

The sample of 40 mL biodiesel was poured into the test tube until it reached the level indicated on the test tube. Then, the sample was placed was 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 of the *Moringa oleifera* methyl esters.

After solid-crystals appeared, the sample was placed in a refrigerator. Once the temperature decreased and the sample still flowing inside the test tube, that temperature was considered as the pour point of the *Moringa oleifera* methyl esters.

3.5 Summary

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

4 RESULTS AND DISCUSSION

4.1 Introduction

This chapter presents all the results obtained from the three sets of experiments for alkali-catalyzed transesterification and acid-catalyzed esterification process with different methanol to oil ratios (6:1, 12:1, 18:1), while the catalyst concentrations, time reaction, stirring speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively. The best biodiesel yield was produced by using method described in previous chapter. The tests and analysis are done to the highest biodiesel yield product. The main aim is to find the physical properties of the biodiesel produced from *Moringa oleifera* seeds oil.

4.2 Biodiesel Yields

In this study, the parameter that was taken into consideration was methanol to oil ratio. While other parameters were fixed. Table 4.1 and 4.2, shows the percentage yields for all three samples, for alkali-catalyzed transesterification and acid-catalyzed esterification process, respectively.

Table 4.1: Biodiesel yield for alkali-catalyzed transesterification process

Ratio (methanol : oil)	Temperature (^{o}C)	Time (min)	Total Product (mL)	Yield (%)
6:1	70	90	43	86
12:1	70	90	46	92
18:1	70	90	48	96

Table 4.2: Biodiesel yield for acid-catalyzed esterification process

Ratio (methanol : oil)	Temperature (^{o}C)	Time (min)	Total Product (mL)	Yield (%)
6:1	70	90	41	82
12:1	70	90	43	86
18:1	70	90	45	90

Due to the limited amount of *Moringa oleifera* feedstock oil, this research manage to produce only three sample for each alkali-catalyzed transesterification and acid-catalyzed esterification process of biodiesel, with different methanol to oil ratio which are 6:1, 12:1 and 18:1. Further studies with different parameters such as reaction temperature, reaction time and catalyst concentration cannot be manipulated to find the optimum yield of biodiesel because of the limited amount of *Moringa oleifera* feedstock oil.

4.3 Yield Comparisons

The comparisons between alkali-catalyzed transesterification and acid-catalyzed esterification process yields can be seen in Figure 4.1 and 4.2 below.

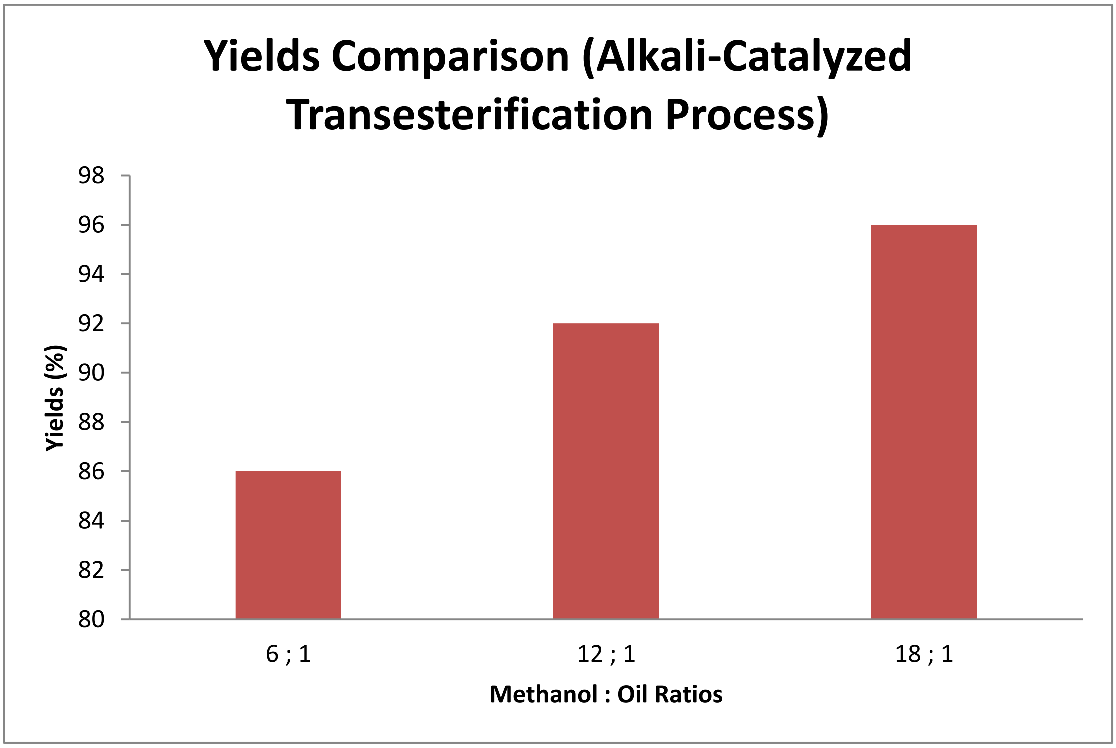

Figure 4.1: Comparisons of yields for different methanol to oil ratios alkali-catalyzed transesterificatin process

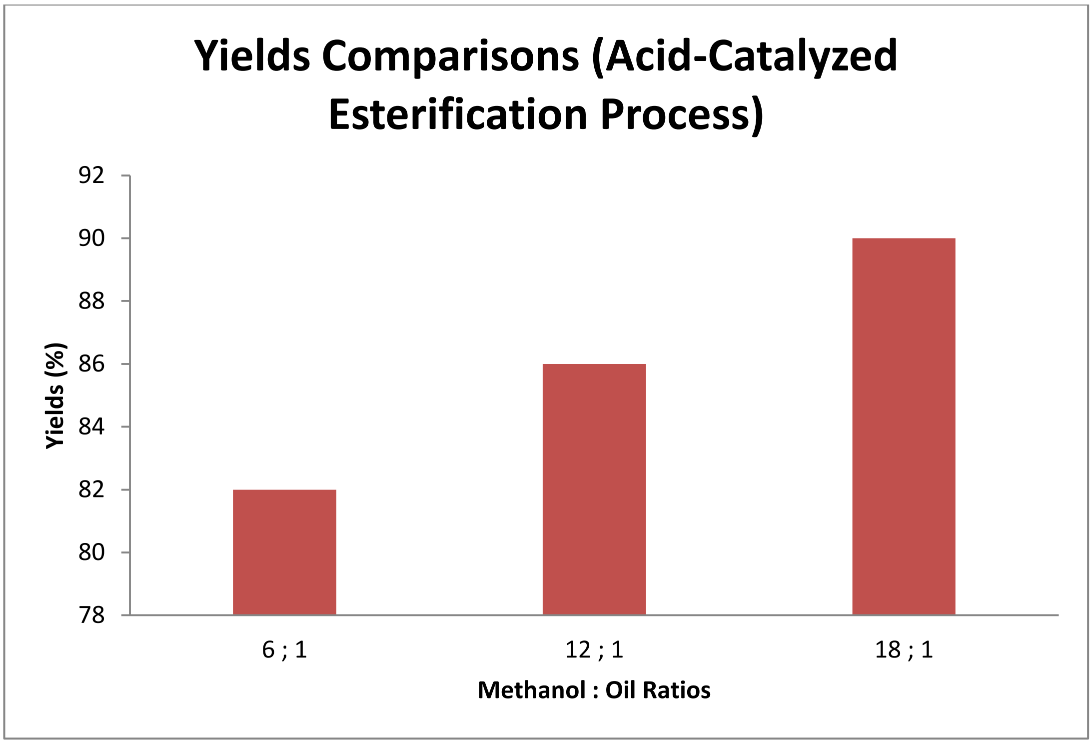

Figure 4.2: Comparisons of yields for different methanol to oil ratios acid-catalyzed esterification process

It can be observed that as the methanol to oil ratios increasing, the biodiesel yield is increasing too for both alkali-catalyzed transesterification and acid-catalyzed esterification experiment. When the lowest amount of methanol was used, the reaction did not proceed to completion and produce only 43 mL and 41 mL of biodiesel product, respectively. It can be concluded that, the higher the amount of methanol, the higher the biodiesel yields. Thus, the methanol to oil ratios that yielded the highest biodiesel for both alkali-catalyzed transesterification and acid-catalyzed esterification process is:

Ratio : 18:1
Temperature : 70 ^{o}C
Time : 90 minutes

The yield of 96% and 90% was achieved by using 18:1 methanol to oil ratio for both alkali-catalyzed transesterification and acid-catalyzed esterification experiment, while the catalyst concentrations, time reaction, stirring speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively.

4.4 Biodiesel Properties Test

4.4.1 Optimum Biodiesel Yield Preparation

According to result obtained in Table 4.1 and 4.2, it was decided to choose methanol to oil ratio at 18:1, while the catalyst concentrations, time reaction, stirring speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively.

4.4.2 Physical Properties Test and Analysis

From the experiment, the final yield for optimum biodiesel of 96% and 90% was chosen. Table 4.3 shows the physical properties tested on the final biodiesel sample according to the ASTM and European Standards methods.

Table 4.3: Physical properties analysis

Analysis	Alkali-Catalyzed Transesterification Experiment	Acid-Catalyzed Esterification Experiment	ASTM D6751 Standard	European 14214 Standard
Cetane number	62.46	62.6	>47	>51
Kinematic viscosity at 40 ^{o}C (mm^2/s)	4.97	4.90	1.9-6.0	3.5-5.0
Density at 15 ^{o}C (kg/m^3)	879.3	878.6	870-900	860-900
Cloud point, ^{o}C	11.3	10.3	-	-
Pour point, ^{o}C	6.7	6.2	-	-

From the Table 4.3, it is shown that the values from the experimental data for both alkali-catalyzed transesterification and acid-catalyzed esterification experiment are within the biodiesel standards which are the American Society for Testing and Materials (ASTM D6751) and European 14214 Standards. The tests were carried out for biodiesel as follow:

4.4.2.1 Density (ρ), (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.

4.4.2.2 Kinematic Viscosity, mm^2/s

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

4.4.2.3 Cetane Number

It is a dimensionless measure of the ignition performance of a diesel fuel. According to Department of Environment and Heritage, reported that *cetane* number is a measure of the readiness of fuel to auto-ignite when injected into a diesel engine. It relates to the delay between the moments of fuel in injected into the cylinder till the ignition occurs. According to (Rashid et al., 2007). *Moringa oleifera* methyl esters appear to be a biodiesel fuel with one of the highest *cetane* numbers ever reported for a biodiesel fuel. This is because of a high presence of saturated fatty acids methyl esters in MOME which is C18:1. *Moringa oleifera* biodiesel easily meets the minimum *cetane* number requirements in both the ASTM D6751 and EN 14214 biodiesel standard, which are >47 and >51, respectively.

4.4.2.4 Cloud Point and Pour Point, °C

The cloud point is the temperature of the fuel at which, small solid crystals can be observed as the fuel cooled. While pour point is the lowest temperature at which the fuel will still pour from a container. *Moringa oleifera* biodiesel has high cloud point and pour point because of the higher amounts of saturated fatty acids (Rashid et al., 2007).

4.5 Summary

This paper presents all the results obtained from the three sets of experiments with different methanol to oil ratios (6:1, 12:1, 18:1), for alkali-catalyzed transesterification and acid-catalyzed esterification experiment, while the catalyst concentrations, time reaction, stirring speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively. The tests and analysis is done to the sample of highest biodiesel yield product. The properties of MOME were measured and found to be within the ASTM D6751 and EN 14214 Standard.

5 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This research showed the possibility of producing biodiesel from *Moringa oleifera* seeds oil with specification of density, kinematic viscosity and *cetane* number, within the ASTM D6751 and EN 14214 Standard.

The optimum biodiesel yield from the research was 96% and 90% at 18:1 for alkali-catalyzed transesterification and acid-catalyzed esterification experiment, while the catalyst concentrations, time reaction, stirring speed and the temperatures were fixed at 1wt%, 90 minutes, 200 rpm and 70 ^oC, respectively. The physical properties obtained from the final optimum biodiesel yield are concluded in Table 5.1.

Table 5.1: Physical properties of *Moringa oleifera* biodiesel

Analysis	Alkali-Catalyzed Transesterification Experiment	Acid-Catalyzed Esterification Experiment
Density, kg/m^3	879.3	878.6
Kinematic viscosity (mm^2/s)	4.97	4.90
Cetane number	62.46	62.6
Cloud point, ^oC	11.3	10.3
Pour point, ^oC	6.7	6.2

The physical properties of the produced biodiesel from *Moringa oleifera* for both alkali-catalyzed transesterification and acid-catalyzed esterification experiment were shown in Table 4.3 and compared with ASTM D6751 and European Standards 14214, which shows that the properties are accepted within the ASTM D6751 and European Standard 14214.

As a conclusion, *Moringa oleifera* seeds oil produces biodiesel from both alkali-catalyzed transesterification and acid-catalyzed esterification process can be used as an alternative for biodiesel as renewable sources for the future usage according to the results obtained in this research study. Therefore, it can be produced by homogeneous or heterogeneous method.

5.2 Recommendations

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

i. Carry out the study by using *Moringa oleifera* oil instead of using seeds and then turn it into oil to produce biodiesel to save more time.
ii. The fatty acids of the biodiesel produced must be determined to support the results for this research.
iii. Further study can be done if the supply of raw materials is sufficient and easy to get.
iv. The research can be done with different parameters to do optimization of yield.

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APPENDICES

APPENDIX A.1
PHYSICAL PROPERTIES ANALYSIS FOR ALKALI-CATALYZED TRANSESTERIFICATION PROCESS

Appendix A.1.1: Density of *Moringa oleifera* biodiesel, ρ

Readings	**Unit (kg/m^3)**
Reading 1	878.9
Reading 2	879.4
Reading 3	880.2
AVERAGE	**879.3**

Appendix A.1.2: *Cetane* number of *Moringa oleifera* biodiesel

Readings	**Unit (kg/m^3)**
Reading 1	61.78
Reading 2	62.82
Reading 3	62.88
AVERAGE	**62.46**

Appendix A.1.3: Cloud point of *Moringa oleifera* biodiesel, ^{o}C

Readings	**Unit (^{o}C)**
Reading 1	10
Reading 2	12
Reading 3	12
AVERAGE	**11.3**

Appendix A.1.4: Pour point of *Moringa oleifera* biodiesel, ^{o}C

Readings	**Unit (^{o}C)**
Reading 1	6
Reading 2	7
Reading 3	7.1
AVERAGE	**6.7**

Appendix A.1.5: Kinematic viscosity of *Moringa oleifera* biodiesel,ν

Readings	Time (s)	Viscosity mm^2/s
Reading 1	44.7	4.91
Reading 2	44.8	4.92
Reading 3	45.0	4.94
AVERAGE	**44.83**	**4.97**

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)

= 44.83 x 0.1098

= **4.97 mm^2/s**

PHYSICAL PROPERTIES ANALYSIS FOR ACID-CATALYZED ESTERIFICATION PROCESS

Appendix A.1.6: Density of *Moringa oleifera* biodiesel, ρ

Readings	Unit (kg/m^3)
Reading 1	878.0
Reading 2	878.9
Reading 3	879.0
AVERAGE	**878.6**

Appendix A.1.7: *Cetane* number of *Moringa oleifera* biodiesel

Readings	Unit (kg/m^3)
Reading 1	62.1
Reading 2	62.8
Reading 3	62.8
AVERAGE	**62.6**

Appendix A.1.8: Cloud point of *Moringa oleifera* biodiesel, ^{o}C

Readings	Unit (^{o}C)
Reading 1	10
Reading 2	10
Reading 3	11
AVERAGE	**10.3**

Appendix A.1.9: Pour point of *Moringa oleifera* biodiesel, ^{o}C

Readings	Unit (^{o}C)
Reading 1	6
Reading 2	6
Reading 3	6.5
AVERAGE	**6.2**

Appendix A.1.10: Kinematic viscosity of *Moringa oleifera* biodiesel,v

Readings	Time (s)	Viscosity mm^2/s
Reading 1	44.4	4.87
Reading 2	44.7	4.91
Reading 3	44.7	4.91
AVERAGE	**44.6**	**4.90**

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)

= 44.6 x 0.1098

= **4.90 mm^2/s**

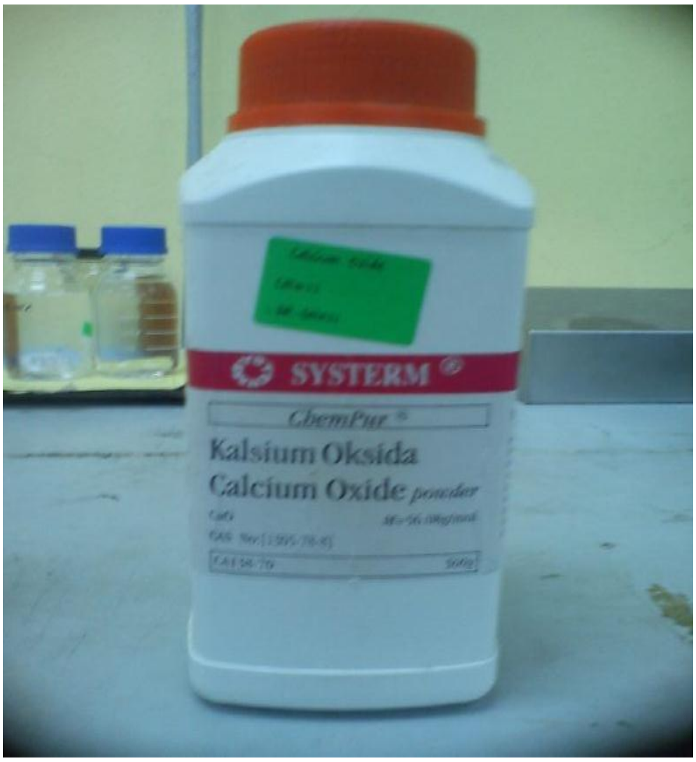
APPENDIX A.2
PRODUCTION PROCESSES

Appendix A.2.1: *Moringa oleifera* seeds after grinding and sieving

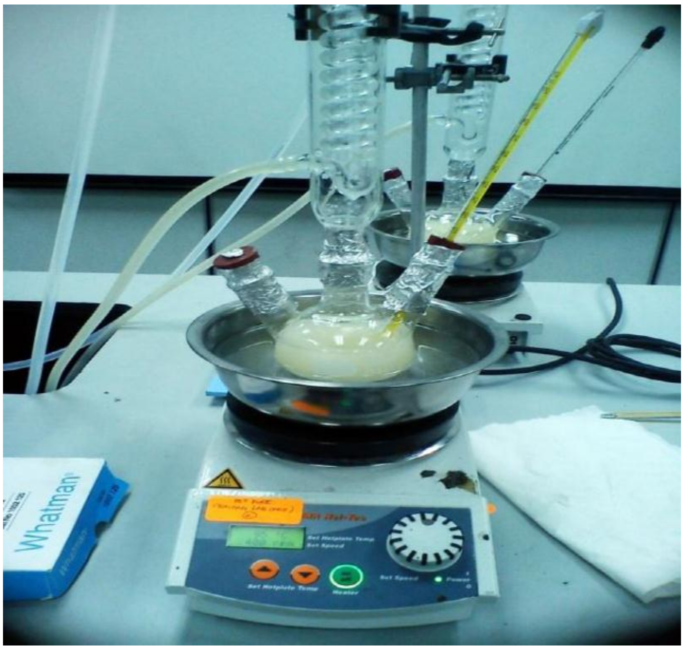
Appendix A.2.2: Thermal oil rotary evaporator

Appendix A.2.3: *Moringa oleifera* feedstock oil

Appendix A.2.4: Methanol, CH_3OH (99.5%)



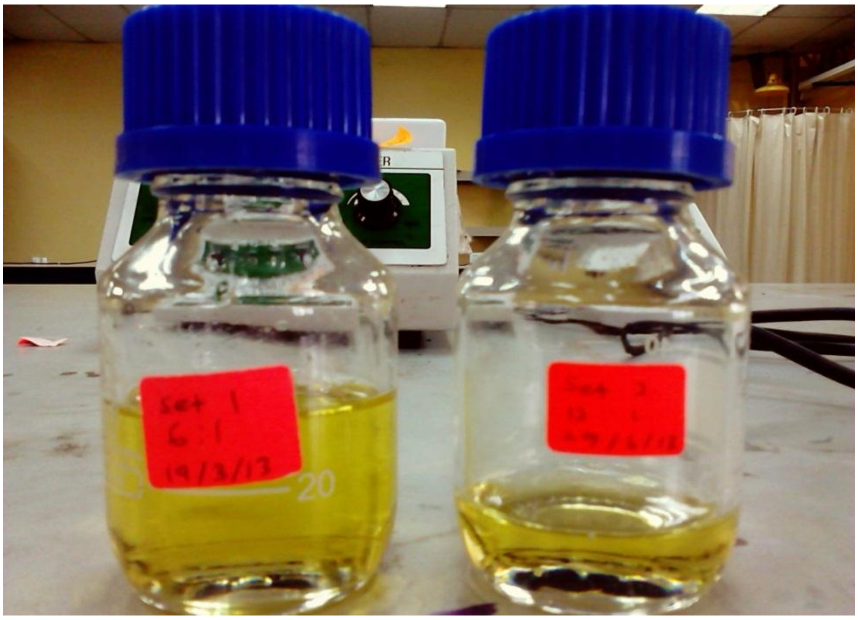
Appendix A.2.5: Calcium oxide (alkali catalyst)



Appendix A.2.6: Mixing of *Moringa oleifera* oil and methoxide

Appendix A.2.7: 24 hours settling in a separating funnel

Appendix A.2.8: Biodiesel washing



Appendix A.2.9: Biodiesel from *Moringa oleifera*

APPENDIX A.3
TECHNICAL PAPER

BIODIESEL PRODUCTION FROM *MORINGA OLEIFERA* SEEDS USING HETEROGENEOUS ACID AND ALKALI CATALYST

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ABSTRACT

Due to the increasing energy demand and pollution problems caused by the use of fossil fuels, it has become necessary to develop alternative fuels as well as renewable sources of energy. The use of biodiesel as a substitute for conventional diesel has been of great interest. This is because biodiesel is biodegradable, non-toxic, renewable, and has low emission of carbon oxide, sulphur dioxide, particulates and hydrocarbons as compared to conventional diesel. Therefore, this study is conducted to investigate the possible production of biodiesel by using *Moringa oleifera* seeds oil through heterogeneous (acid and alkali) catalyst process. *Moringa oleifera* seeds oil can be used for biodiesel production by transesterification using calcium oxide (CaO) as an alkaline catalyst followed by esterification by using ferric sulphate catalyst. A sample of 50 mL oil was poured into the 3-neck round-bottom glass flask. Carefully, the methanol was poured into the oil with ratio 6:1, 12:1 and 18:1 for both alkali-catalyzed transesterification and acid-catalyzed esterification process. The catalyst concentrations, time reaction, agitation speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively. As a result, the methyl esters (biodiesel) produced from *Moringa oleifera* seeds oil exhibits a high yield which is 48 mL (96%) and 45 mL (90%) for both alkali-catalyzed transesterification and acid-catalyzed esterification process by using methanol to oil ratio of 18:1, and catalyst concentrations, time reaction, agitation speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 ^{o}C, respectively. As a conclusion, biodiesel can be produced from *Moringa oleifera* seeds oil as an alternative fuels as well as renewable sources of energy for future use.

Keywords: Biodiesel; Heterogeneous Method; Alkali Catalyst; *Moringa oleifera*

INTRODUCTION

Biodiesel is the mono alkyl ester of long chain fatty acids derived from a renewable lipid feedstock, such as vegetable oil or animal fat by transesterification process to reduce the viscosity of that lipid feedstock. Transesterification is a chemical process usually used to produce biodiesel in which triglycerides are allowed to react with an alcohol (mostly methanol) under acidic or basic catalytic conditions. It is a technically competitive and environmentally friendly alternative to conventional petrodiesel fuel for use in compression-ignition (diesel) engines (Rashid et al., 2007). Moreover, biodiesel possesses inherent lubricity, a relatively high flash point, and reduces most regulated exhaust emissions in comparison to petrodiesel. The use of biodiesel reduces the dependence on imported fossil fuels, which continue to decrease in availability and affordability (Rashid et al., 2007).

The examination of the *Moringa oleifera* becomes fundamental as a potential biodiesel production. The Moringaceae is a single-genus family of oilseed trees with 14 known species (Rashid et al., 2007). *Moringa oleifera* is one of these species which ranges in height from 5 to 10 meter, is the most widely known and utilized (Morton 1991). *Moringa oleifera*, indigenous to sub-Himalayan regions of northwest India, Africa, Arabia, Southeast Asia, the Pacific and Caribbean Islands and South America, is now distributed in the Philippines, Cambodia and Central and North America (Morton, 1991). It thrives best in a tropical insular climate and is plentiful near the sandy beds of rivers and streams (Council of Scientific and Industrial Research, 1962).

MATERIALS AND METHODS

Oil preparation from *Moringa oleifera* seeds

Oil extraction from *Moringa oleifera* seeds was done using hexane as a solvent to the seed powder, to separate oil from seeds using electro thermal soxhlet. The extraction procedure was as follow: weighing 30 gm of *Moringa oleifera* seed powder and setting it in the thimbles of the electro thermal soxhlet extraction chamber. Adding 200 mL of hexane in the heating chamber; evaporating of hexane within three cycles each for 30 minutes to ensure the extraction of oil from seeds (until the hexane become colourless) (Eman et al., 2010). After extraction for 1.5 hour, the solvent was removed at 60 ^{o}C under vacuum using rotary evaporator so that the crude *Moringa oleifera* oil can be collected.

Table 1: Sets of samples and trial

Ratios (methanol : oil)	Temperature (^{o}C)	Time (min)
6:1	70	90
12:1	70	90
18:1	70	90

Alkali-catalyzed transesterification of *Moringa oleifera* biodiesel

A sample of 50 mL oil was poured into a 3-neck round-bottom glass flask equipped with a reflux condenser. The methanol was poured carefully into the oil according to the ratio selected in Table 1. While, stirring speed, reaction time, catalyst concentration (calcium oxide) and reaction temperature are fixed at 200 rpm, 90 minutes, 1 wt% and 70 ^{o}C. After the reaction completed, calcium oxide (CaO) was removed by filtration and the product was allowed to settle overnight in a separating funnel for separation of biodiesel. The lower glycerol layer in the separating funnel was drawn off. After that, the biodiesel layer was washed four times using hot water (80 ^{o}C) and dried with enough anhydrous sodium sulphate (Zhang et al., 2009).

Acid-catalyzed esterification of *Moringa oleifera* biodiesel

The same alkali-catalyzed transesterification experiment was repeated using ferric sulphate as an acid catalyst.

RESULTS AND DISCUSSION

The results obtained from the three sets of experiments for alkali-catalyzed transesterification and acid-catalyzed esterification process with different methanol to oil ratios (6:1, 12:1, 18:1), while the catalyst concentrations, time reaction, stirring speed and the temperatures are fixed at 1wt%, 90 minutes, 200 rpm and 70 °C respectively. The tests and analysis are done to the highest biodiesel yield product shown in Table 2 and 3. The main aim is to find the physical characteristics of the biodiesel produced from *Moringa oleifera* seeds oil.

Due to the limited amount of *Moringa oleifera* feedstock oil, this research manage to produce only three sample for each alkali-catalyzed transesterification and acid-catalyzed esterification process of biodiesel, with different methanol to oil ratio which are 6:1, 12:1 and 18:1. Further studies with different parameters such as reaction temperature, reaction time and catalyst concentration cannot be manipulated to vary the optimum yield of biodiesel because of the limited amount of *Moringa oleifera* feedstock oil.

Table 2: Biodiesel yield for alkali-catalyzed transesterification process

Ratio (methanol : oil)	Temperature (°C)	Time (min)	Total Product (mL)	Yield (%)
6:1	70	90	43	86
12:1	70	90	46	92
18:1	70	90	48	96

Table 3: Biodiesel yield for acid-catalyzed esterification process

Ratio (methanol : oil)	Temperature (°C)	Time (min)	Total Product (mL)	Yield (%)
6:1	70	90	41	82
12:1	70	90	43	86
18:1	70	90	45	90

The comparisons between alkali-catalyzed transesterification and acid-catalyzed esterification process yields can be seen in Figure 1 and 2.

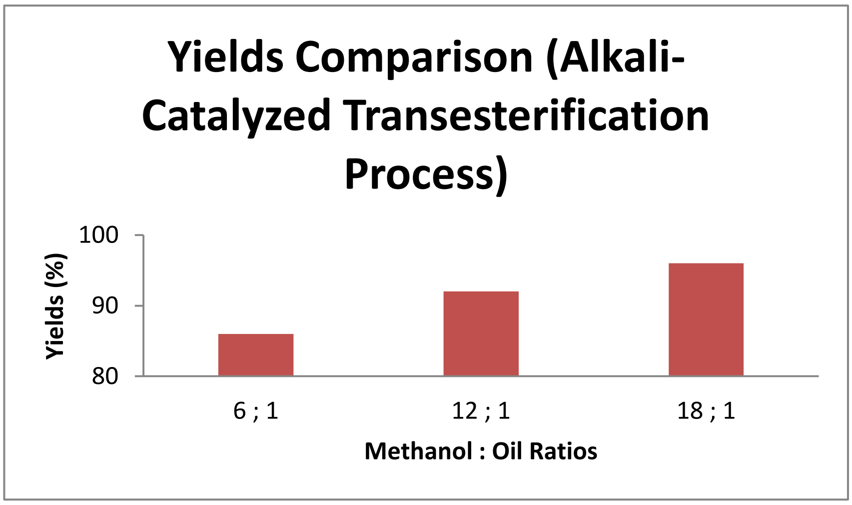

Figure 1: Comparisons of yields for different methanol to oil ratios alkali-catalyzed transesterificatin process

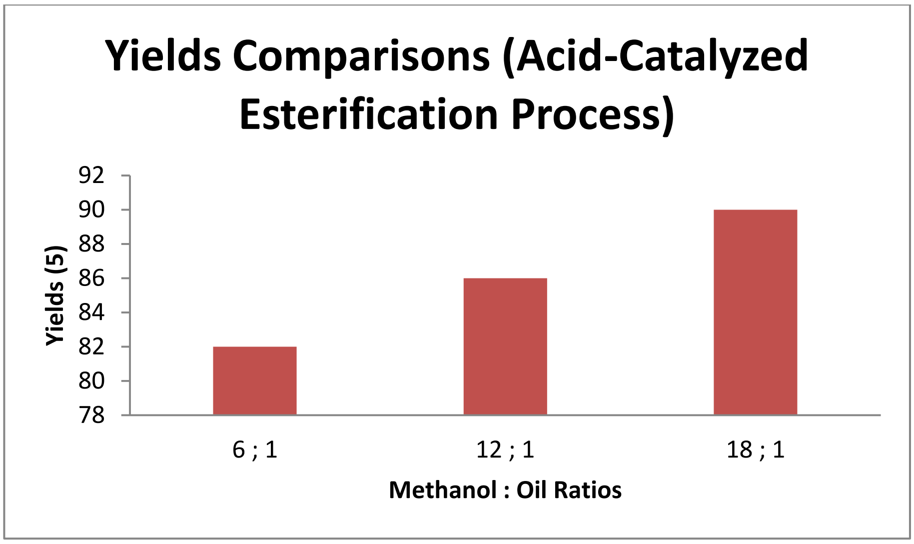

Figure 2: Comparisons of yields for different methanol to oil ratios acid-catalyzed esterification process

It can be observed that as the methanol to oil ratios increasing, the biodiesel yield is increasing too for both alkali-catalyzed transesterification and acid-catalyzed esterification experiment. When the lowest amount of methanol was used, the reaction did not proceed to completion and produce only 43 mL and 41 mL of biodiesel product respectively. It can be concluded that, the higher the amount of methanol, the higher the biodiesel yields. Thus, the methanol to oil ratios that yielded the highest biodiesel for both alkali-catalyzed transesterification and acid-catalyzed esterification process is:

Ratio : 18:1

Temperature : 70 °C

Time : 90 minutes

CONCLUSION

The highest biodiesel yield from the research was 96% and 90% at 18:1 for alkali-catalyzed transesterification and acid-catalyzed esterification experiment, while the catalyst concentrations, time reaction, stirring speed and the temperatures were fixed at 1wt%, 90 minutes, 200 rpm and 70 °C, respectively. The physical characteristics obtained from the final biodiesel yield are concluded in Table 4

Table 4: Physical properties of *Moringa oleifera* biodiesel

Analysis	**Alkali-Catalyzed Transesterification Experiment**	**Acid-Catalyzed Esterification Experiment**	**ASTM D6751 Standard**	**European 14214 Standard**
Cetane number	62.46	62.6	>47	>51
Kinematic viscosity at 40 °C (mm^2/s)	4.97	4.90	1.9-6.0	3.5-5.0
Density at 15 °C (kg/m^3)	879.3	878.6	870-900	860-900
Cloud point, °C	11.3	10.3	-	-
Pour point, °C	6.7	6.2	-	-

The physical properties of the produced biodiesel from *Moringa oleifera* for both alkali-catalyzed transesterification and acid-catalyzed esterification experiment were shown in Table 4. The table shows that the properties are accepted within the ASTM D6751 and European Standard 14214 which is 4.97 and 4.90 mm^2/s for kinematic viscosity, 879.3 and 878.6 kg/m^3 for density, 11.3 and 10.3 °C for cloud point and 6.7 and 6.2 °C for pour point, respectively.

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