

**BIODIESEL PRODUCTION FROM *MORINGA*
OLEIFERA SEEDS OIL BY USING MgO AS A
CATALYST**

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BIODIESEL PRODUCTION FROM *MORINGA OLEIFERA* SEEDS OIL USING MgO AS A CATALYST

AQWA BIN MOHD NAWI

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**

JANUARY 2015

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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).

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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.

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Dedication

To my family, friends and supervisor for being supportive.

ACKNOWLEDGEMENT

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

In the first place I would record my gratitude to my family who inspired me, encouraged and fully supported me for every trail that come in my way. In giving me not just financial, but morally and spiritually support.

I gratefully acknowledge and offer my sincerest gratitude to my supervisor, Dr. Eman N Ali, who has supported me throughout my project proposal with her patience and acknowledge while allowing me to work in my own way. Without her encouragement and effort, this report would not have been completed or written. One simply could not wish for a better or friendly supervisor.

Many thanks to my fellow friends, I am much indebted to all of you even though we have different topics but still have time to read and share our ideas together.

ABSTRACT

Biodiesel is a RE (renewable energy) source derived alternative fuel for diesel engine application that is produced through the transesterification of vegetable oil, animal fats or waste vegetable oil with alcohol in the presence of catalyst. *Moringa oleifera* seeds can be used for biodiesel production by transesterification using magnesium oxide (MgO) as a solid base catalyst. Therefore, this study is conducted to investigate the possible production of biodiesel by using *Moringa oleifera* seeds oil through base catalyst method. A sample of 25 mL oil was poured into the 3-neck bottom flask. Carefully, the methanol was poured into the oil with ratios of 8:1, 10:1, and 12:1 methanol to oil for the transesterification process. The MgO was used with different concentration of, 0.5%, 0.7%, 1.0% w/w. As a result, the methyl ester (biodiesel) produced by using *Moringa oleifera* seeds oil exhibits a high yield by using 60-70°C reaction temperature, 1.5 hour reaction periods, and 200 rpm mixing rate. As the result, about 60% yield of biodiesel was produced using 0.5 wt% catalyst concentration and 12:1 methanol to oil ratio. In conclusion, biodiesel production by using *Moringa oleifera* seeds oil appear to be an acceptable alternative fuel as well as renewable source of energy for the future use.

Keywords: Biodiesel, Transesterification, MgO, *Moringa oleifera*, Catalyst.

ABSTRAK

Biodiesel adalah RE (tenaga boleh diperbaharui) sumber yang diperolehi bahan api alternatif untuk aplikasi enjin diesel yang dihasilkan melalui transesterifikasi minyak sayuran, lemak haiwan atau minyak sayuran buangan dengan alkohol dalam kehadiran pemangkin. *Moringa oleifera* benih boleh digunakan untuk pengeluaran biodiesel oleh transesterification menggunakan magnesium oksida (MgO) sebagai asas pemangkin pepejal. Oleh itu, kajian ini dijalankan untuk menyiasat pengeluaran mungkin biodiesel dengan menggunakan *Moringa oleifera* benih minyak melalui kaedah asas pemangkin. Sampel kajian terdiri daripada 25 mL minyak dicurahkan ke dalam 3-leher kelalang bawah. Dengan berhati-hati, metanol yang telah dicurahkan ke dalam minyak dengan nisbah 8: 1, 10: 1, dan 12: 1 metanol untuk minyak untuk proses transesterifikasi itu. The MgO digunakan dengan kepekatan yang berbeza dengan 0.5%, 0.7% dan 1.0% w / w. Akibatnya, ester metil (biodiesel) menghasilkan dengan menggunakan *Moringa oleifera* benih minyak mempamerkan hasil yang tinggi dengan menggunakan keadaan yang 60-70 ° C suhu tindak balas, tempoh tindak balas 1.5 jam dan kadar pencampuran 200 rpm. Oleh itu, kira-kira 60% daripada hasil biodiesel dihasilkan menggunakan 0.5% berat kepekatan pemangkin dan 12: 1 metanol untuk nisbah minyak. Kesimpulannya, pengeluaran biodiesel dengan menggunakan *Moringa oleifera* benih minyak kelihatan yang boleh diterima sebagai bahan api alternatif dan juga sumber tenaga yang boleh diperbaharui untuk kegunaan masa depan.

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LIST OF ABBREVIATIONS

MOME	<i>Moringa oleifera</i> methyl ester.
MgO	Magnesium oxide.
CaO	Calcium oxide.
FFA	Free fatty acid.
IEA	International Energy Agency.

1 INTRODUCTION

1.1 Motivation and problem statement.

Biodiesel is an alternative fuel for diesel engines that is produced by chemically reacting a vegetable oil or animal fat with an alcohol such as methanol (Gerpen, 2005) has recently been considered as the best candidate for a diesel fuel substitution because it can be used in any compression ignition engine without the need for modification. The advantages of biodiesel as diesel fuel are liquid nature portability, ready availability, renewability, higher combustion efficiency, lower sulfur and aromatic content and higher biodegradability (Ma and Hanna, 1999; Demirbas, 2009).

Vegetable oils for biodiesel production vary considerably with location according to climate and feedstock availability. Generally, 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, soybeans and animal fats in the USA (Knothe, 2005). However, biodiesel production from conventional sources (soybeans, rapeseed, palm and many more,) increasingly has placed strain on food production, price and availability. Therefore, the search for additional regional biodiesel feedstock is an important objective. Some recent examples, studies of biodiesel from less common or less unconventional oils include tobacco (Usta, 2005), pongamia (Karmee and Chadha, 2005), jatropha (Foidl et al., 1996) and rubber seed oil (Ramadhas et al., 2005).

The moringaceae is a single-genus family of oilseed trees with 14 unknown species. Of these, *Moringa oleifera*, which range in height from 5 to 10m, is the most widely known and utilized (Sengupta and Gupta, 1970). *Moringa oleifera* seeds contain between 33 and 41% w/w of vegetable oil (Sengupta and Gupta, 1970). Several authors investigate the composition of *Moringa oleifera*, including its fatty acid profile (Anwar and Bhanger, 2003; Anwar et al., 2005; Sengupta and Gupta, 1970) and showed that *Moringa oleifera* oil is high in oleic acid (>70%).

Heterogeneous base catalysts have advantages of being reusable, noncorrosive, show greater tolerance to water and free fatty acids (FFAs) in feedstock, improve biodiesel yield and purity, have a simpler purification process for glycerol and are easy to separate

from the biodiesel product (Hsiao et al., 2011). Calcium oxide (CaO) is one of the most common used base catalysts for the transesterification of vegetable oil. Producing biodiesel using CaO as a solid base catalyst has many advantages, such as higher activity, mild reaction condition, reusable and low cost (Kawashima et al., 2009; Liu et al., 2008; Hsiao et al., 2011). Liu et al. (2008) shows that CaO powder can give about 95% conversion of soybean oil to biodiesel in presence of excess methanol (12:1) at temperature of 60 degree Celsius and reaction time of 3 hours. (Hsiao et al., 2011) achieved 96.6% of conversion of soybean oil to biodiesel using a microwave assisted transesterification with 3% wt of nonpowder CaO catalyst, Methanol to oil ratio of 7:1, reaction temperature of 65°C and residence time of 1 hour. Other than CaO, Magnesium oxide (MgO) also widely used among the other alkaline earth metal. Therefore one of the objective of this research is to study the performance of MgO as a catalyst to replace CaO in transesterification process.

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 and global demand grew by around 1% annually apart from in 2003 when demand increased by 1.8 (Kjarstad 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 and if the production rate is maintained at 0.25 billion barrels per year, the ratio between reserve and production is 12, indicating that within 12 years, Malaysia's oil will be exhausted. While in 2006, Malaysia's oil output decline where production for 2006 average stood at 798,000 bbl/day, down 7% from 2005 (Kjarstad and Johnsson, 2008).

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 country left to ascertain an increasing global oil production in future. Thus, *Moringa oleifera* seeds oil will be 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. *Moringa oleifera* seeds is shown in Figure 1.



Figure 1: Moringa oleifera seeds.

1.2 Objectives

The objectives of this research are:

1. To produce biodiesel from *Moringa oleifera* seeds oil by using MgO as catalyst.
2. To examine the parameters that affect biodiesel yield,(different concentration of catalyst) and (different methanol to oil ratio)

1.3 Scope of this research

In order to achieve the objectives, all parameters must be studied.The main parameters are:

1. The reaction temperature.
2. Agitation speed.
3. Catalyst concentration.
4. Reaction time.

2 LITERATURE REVIEW

2.1 Overview

Biodiesel has become more attractive recently because of its environmental benefits and the fact that it is made from renewable resources. There are four primary ways to make biodiesel which are, direct use and blending, microemulsions, thermal cracking (pyrolysis) and transesterification. The most commonly used method is transesterification of vegetable oils and animal fats. The transesterification reaction is affected by molar ratio of glycerides to alcohol, catalysts, reaction temperature, reaction time and free fatty acids and water content of oils or fats. The processes of transesterification and its downstream operations are addressed.

2.2 Biodiesel production process

In biodiesel production, transsterification is the reaction of fats or oils with alcohols to form biodiesel. There are two method of transesterification generally, the first method employ 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 catalyst appears to be promising because it can simplify the production and the purification processes, decrease the amount of basic waste water, downsize the process equipment, and reduced the environmental impact and process cost (Kawashima et al., 2009). In general, a catalyst is used to initiate the esterification reaction for making biodiesel. The catalyst is essential as alcohol is scarcely soluble in oil or fat. The catalyst enhances the solubility of alcohol and thus increases the reaction rate. The transesterification process for biodiesel production is conventionally achieved using homogeneous acid or base catalysts. Alkali catalytic method is followed when FFA content of the fed stock is less than 1%; while acid catalytic process is followed when FFA content is more than 1%. Most of the current commercial biodiesel production is dominated by base catalyst process. Heterogeneous catalysis is receiving much attention as the solid acid or solid basic catalysts that could be used in a heterogeneously catalyzed process, does not produce soaps through FFA acid neutralization and triglyceride saponification. The rate of transesterification reaction is strongly influenced by the temperature; higher the temperature faster is the reaction rate and shorter the reaction

time. However, the reaction is conducted below the boiling point of the alcohol used, generally 60 °C for methanol and 78 °C for ethanol.

2.2.1 Alkali catalytic

Alkaline catalyzed production process of biodiesel is the process of transesterification of a fat or oil triglyceride with an alcohol to form ester and glycerol, in the presence of an alkali catalyst. The most commonly prepared ester are methyl ester because methanol is the least expensive alcohol. The alkali catalyst such as sodium or potassium hydroxide, or sodium or potassium methoxide are the most common and are preferred due to their high yields. The based-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 based catalysts to form soaps and water. Soaps 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 catalyst (Karmakar et al., 2009)

2.2.2 Acid catalytic

Acid catalyst systems are characterized by slow reaction rate and high ratio of alcohol and triglyceride requirement (20:1 and more). Generally, acid-catalyzed reactions are used to convert FFAs to esters, or soaps to esters as a pretreatment step for high FFA feedstocks. Acid catalyst process requires excess alcohol, hence the transesterification reactor and alkali distillation column of acid-catalyzed process are larger than alkali-catalyzed process for same biodiesel production capacity. A high conversion efficiency with acid-catalyzed transesterification can be achieved by increasing the molar ratio of alcohol to oil, reaction temperature, concentration of acid catalyst and the reaction time (Canakci and Gerpen, 1999).

2.2.3 Bio catalytic

Bio catalyst like immobilized lipase can also be used as transesterification reaction agents. Even though the enzyme reactions are highly specific and chemically clean, the reactions are very slow requiring from 4 to 40 h, or more

at the temperature of 35 to 45 °C. For feedstocks having very high FFA content, enzyme catalysis process is very promising. But this process has not yet been commercialized because of high cost of operation.

2.2.4 Heterogeneous catalytic

As Transesterification reaction using basic catalysts is the most extended process to produce biodiesel. In this process it is necessary to use feedstock (vegetable oils or animal fats) which present low free fatty acids content. When the raw materials contain high percentage of free fatty acids or water, the alkali catalyst reacts with the free fatty acids to forms soaps and the water can hydrolyze the triglycerides into diglycerides and form more free fatty acids (Leung et al., 2010). Metal oxides are the basic heterogeneous catalysts group most studied. There are several metal oxides that have been studied in bibliography: calcium oxide, magnesium oxide, strontium oxide, mixed oxides and hydrotalcites. 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 (Math et al., 2010).

2.3 Properties of biodiesel feedstock

Vegetable oils and animal fats mainly consist of triglycerides and diglycerides with a small fraction of monoglyceride. The chemical structure of these esters is compared with mineral diesel and biodiesel. The vegetable oils consist of long chains with multiple branches resulting in molecules of large size. The molecular weight of vegetable oils ranges from 850 to 995 g/mol, which is much higher than that of diesel which is 168 g/mol on average. The kinematic viscosity and density of vegetable oils are much higher than those of diesel due to their higher molecular weight and complex structure. A comparison of various physical and chemical properties of diesel and of various vegetable oils is shown in table 2.5 (Barnwal and Sharma, 2005). Other than that, the fuel properties are also depending on the amount of each fatty acid present in the feedstock (Karmakar et al., 2009).

2.3.1 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).

Table 1: Fatty acid composition of the *Moringa Oleifera* oil. (Karmakar et al., 2009).

Fatty acid ^a	
Palmitic (16:0)	7.0
Palmitoleic (16:1)	2.0
Stearic (18:0)	4.0
Oleic (18:1)	78.0
Linoleic (18:2)	1.0
Linolenic (18:3)	– ^b
Arachidic (20:0)	4.0
Behenic (22:0)	4.0

^a= experimental results.

^b= this can indicate traces (<1.0%) or absence.

2.3.2 Heat content

The calorific content is the energy content of the oil. The energy content of the biodiesel depends on the energy content of the feedstock oil. 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, greater the potential energy (Aninidita et al., 2012).

2.3.3 Removal of water

Water should be removed from transesterification reaction because at high temperature, water can hydrolyze the triglycerides to diglycerides and form a FFA. This FFA reacts with the alkali catalyst and forms soaps. Even the presence of very little amount (1%) of water will increase soap production and considerably affect transesterification reaction. Heating breaks any emulsions between the water and oil; and then settled water at the bottom can be removed. Centrifuges can also be used to separate water and oil. Heating the oil under pressure and then spraying into a vacuum chamber can bring the water content to a very low level (Gerpen, 2004).

2.3.4 Removal of insoluble impurities

The insoluble impurities may consist of sand, dirt, and seed fragments in case of vegetable oil, and small particles of bones and gums in case of animal fats or used cooking oil. Feedstocks should always be filtered prior to entering the processing system. A 100 μm filter will suffice to remove particles from most feedstocks although all fuel leaving the plant should be filtered to 5 μm (Gerpen, 2004).

2.4 Feedstocks for biodiesel production

Renewable oils are derived from widely available crop seeds depending on the agro climatic region; rapeseed (canola) in northern Europe, soybean in the USA, canola oil in Canada, palm oil, coconut and sunflower in tropical regions; Ireland uses frying oil and animal fats. Among the animal fats, bovine fat, the fish oils, the pig fat, duck and beef tallow, lard are being considered as renewable oil (Aninidita et al., 2012).

Edible oils like soybean, sunflower, rapeseed and palm are used as main biodiesel feedstocks throughout the world. Non-edible oils like jatropha, pongamia, neem, and others have been found to be promising feedstocks in developing countries where edible oils are in short supply. Oil from rapeseed has been the great choice in the early days and is still leading with a share of over 80% as a raw material source with highly suitable properties; sunflower oil takes second place with over 10%, followed by soybean oil. Emerging feedstocks being considered as potential are mustard, hemp, castor oil, peanut

oil, coconut oil, cotton seed oil, corn oil, rice bran oil, coffee ground, mahua oil, neem oil, tobacco oil, sesame oil, pongamia oil, passion seed oil, babassu oil, grape oil, algae oil and waste vegetable oil.

2.4.1 Commonly used edible oil

2.4.1.1 Soybean oil

Soybean oil is used as a major source of edible oil throughout the world. With about 222 million tonnes, soybean is the most important oil bearing plant cultivated world-wide and its production is seeing a further expansion, particularly in the USA, Brazil and Argentina (Bockey, 2006). According to the National Board of Biodiesel (USA), soybean oil is the primary source for biodiesel production in US and an estimated 75 million gallons of biodiesel were to be produced in 2005. Soybeans can be produced without or nearly zero nitrogen. This makes soybeans advantageous for the production of biodiesel as nitrogen fertilizer is one of the most energy costly inputs in crop production. (Pimental and Patzek, 2005) studied the energy estimation for producing soybean biodiesel. They reported that 5546 kg of soybeans were required for producing 1000 kg of oil and biodiesel production using soybean required 27% more fossil energy than the biodiesel fuel produced.

2.4.1.2 Sunflower oil

Sunflower with high oil content is one of the more prominent oilseed crops for biodiesel production. At one point, it was considered to be the second primary source of edible oil next to soybean. Sunflower can grow in a variety of climatic conditions but it is considered to be an inefficient user of nutrients. Average yield is approximately reported to be lower than soybean yields, and necessary inputs are greater (Pimental and Patzek, 2005).

2.4.1.3 Palm oil

Oil is derived from both the flesh and the seed of the palm fruit. The fruit consists of an outer pulp, which is the source of crude palm oil and two or three kernels, which are the source of another oil type – palm kernel oil. Crude palm oil is semisolid at room temperature. Palm kernel oil is rich in lauric and myristic fatty acid with an excellent oxidative stability and sharp melting. Malaysia is the world's largest palm growing country and palm plantation covers two-third of its agricultural lands. Palm oil has been proved to be an efficient biodiesel source. The average yield of approximately 6000 l of palm oil/ha can produce 4800 l of biodiesel. It has been stated that palm oil can have high levels of fatty acids, which require extra methanol transesterification before it can be used as biodiesel, thus increasing the cost of production somewhat (Crabbe et al., 2001). Farmers in Ghana are producing biodiesel from palm kernel oil for powering their farm vehicles and generators.

2.4.1.4 Peanut oil

The peanut or groundnut is native to South America, Mexico and Central America. Rudolf Diesel, the inventor of compression-ignition engines, used first time peanut oil in 1900. The physico-chemical characteristics of peanut oil biodiesel (POB) closely resemble to those of diesel fuel (Mittelbach and Tritthart, 1988). Peanut oil produces approximately 1170 l biodiesel/ha, compared to 475 l for soybean oil (Mittelbach and Tritthart, 1988). But the production of biodiesel from peanut oil is not economically viable as peanut oil is more valuable than soy oil in the world market. Studies are going on at the University of Georgia to develop non-edible peanut varieties which are high in oil, but will not compete with peanuts grown for food or cooking oil purposes.

2.4.2 Commonly used non edible oil

2.4.2.1 *Jatropha*

Recently *Jatropha* is being considered as one of the most promising potential oil source to produce biodiesel in Asia, Europe and Africa. *Jatropha* can grow under a wide variety of climatic conditions like severe heat, low rainfall, high rainfall and frost. *Jatropha* is grown in marginal and waste lands with no possibility of land use competing with food production. *Jatropha* oil content varies depending on the types of species, climatic conditions and mainly on the altitude where it is grown. Various parts of the plant have medicinal values. Apart from supplying oils for diesel replacement, the growing of the tree itself effectively reduces CO₂ concentrations in the atmosphere. In developing countries like India it has been identified as the major source of biodiesel (Aninidita et al., 2012).

2.4.2.2 *Pongamia* oil

It is a hardy tree of 12–15 m height, branches spread into hemispherical crown of dense green leaves and native to the Asian sub-continent. *Pongamia* can survive in adverse conditions like draught, heat, frost, salinity etc. Most of the physical and chemical properties of the directly extracted oil are almost similar to those of the diesel, though “conardson carbon” residue is higher in case of it and due to high viscosity preheating is necessary to start a diesel engine (Shrinivasa, 2001). The toxic substances present in the oil limits its use as cooking oil. The seedcake after oil extraction can be utilized as nematicide.

2.4.2.3 *Neem* oil

Neem oil is light to dark brown in colour and bitter in taste. The *Neem* tree is native to India and Burma and almost the whole tree is usable for various purposes such as medicines, pesticides and organic fertilizer. *Neem* can be grown on very marginal soils that may be very rocky, shallow, dry, or pan-forming. *Neem* tree can tolerate some

extreme conditions like temperature of 45 °C and rainfall less than 35 cm per year. Azadirachtin is the main constituent of *Neem* seed oil which varies from 300 to 2500 ppm depending on the extraction technology and quality of the *Neem* seeds crushed. The oil contains sulfurous compounds which gives it a pungent odor and a less-clean burn than other vegetable oils (Aninidita et al., 2012).

2.4.3 Potential edible oil

2.4.3.1 *Moringa oleifera* oil

The *Moringa* or Ben-oil tree is believed to be native to India, Arabia, and possibly even across Africa and the Caribbean. It thrives in subtropical to tropical dry to moist climates, tolerating rainfall from 25 to 300 plus cm annually with temperatures ranging from 19 to 28 °C. It is a fast growing tree and can tolerate draught, sandy soil, bacteria and fungi. In addition to its nutritional value, *M. oleifera* has got many useful medicinal uses. Ben-oil trees are capable of producing seed pods within the first year after planting. Full fruit bearing can be expected by the second full year. The trees can continue producing pods for several years. Oil qualities are similar to that of olive oil, and the degummed oil has favorable characteristics for use as a replacement for petroleum diesel (Tsaknis et al., 1999). There has been strong international interest in *Moringa* with claims that it can annually produce 1000–2000 l of biodiesel from one hectare. Biodiesel produced from *Moringa* oil exhibit a high cetane number of approximately 67, one of the highest found for a biodiesel fuel, enhanced oxidative stability and high cloud point (Umer et al., 2008).

2.4.3.2 Coconut oil

Coconut is widely harvested in tropical coastal areas. Coconut oil accounts for nearly 20% of the vegetable oil produced in the world. Coconut oil is extracted from the copra, the dried flesh of the nut.

Coconut oil remains solid at relatively high temperature than most of other vegetable oils. In liquid form, Coconut oil burns very neatly in diesel engines. Coconut oil has the potential to yield 50% motor fuel as diesel fuel. The main drawback of using coconut oil in engines is that it starts solidifying at a temperature below 22 °C and by 14 °C it does not flow at all (Aninidita et al., 2012).

2.4.3.3 Corn oil

Corn is a starch crop. It is mainly sown in USA for its starch and protein content. It is not practically viable to grow this crop specifically for biodiesel production as the extraction process cannot produce a grade of oil which is suitable enough for production of biodiesel. But when the crop is turned into ethanol after fermentation, the oil can easily be separated and viably used for biodiesel production. In the USA, interest has grown up to use corn oil as source material for biodiesel production (Aninidita et al., 2010).

2.4.4 Potential non edible oil

2.4.4.1 Algae oil

It was reported that algae (micro and macro) were one of the best sources of biodiesel; microalgae have much more oil than macroalgae and it is much easier and faster to grow (Shay, 1993). The best algae for biodiesel would be microalgae and are the highest yielding feedstock for biodiesel and it can produce up to 250 times the amount of oil per acre as soybeans (Hossain et al., 2008). Microalgae may be the potential and economical source of biodiesel because of its high yield and non-requirement of farmland or fresh water. Oil content in microalgae can exceed 80% by weight of dry biomass (Spolaore et al., 2006). Microalgal oils differ from most vegetable oils in being quite rich in polyunsaturated fatty acids with four or more double bonds (Belarbi et al., 2000). Studies show that biodiesel can be produced from macro algae also even though the lipid content (1.3–

7.8%, dw) of macro algae is less than micro algae (Hossain et al., 2008). Yusuf (2007) reported that microalgae appear to be the only source of renewable biodiesel that is capable of meeting the global demand for transport fuels. Oil productivity of many microalgae greatly exceeds the oil productivity of the best producing oil crops.

2.4.4.2 Castor oil

Castor oil has been identified as a potential raw material for the production of biodiesel in Brazil. Castor oil is comprised almost entirely (ca. 90%) of triglycerides of ricinoleic acid (12-hydroxy-cis-octadec-9enoic acid) in which the presence of a hydroxyl group at C-12 imparts several unique chemical and physical properties and thus, castor oil and its derivatives are completely soluble in alcohols and exhibit viscosities that are up to sevenfold higher than those of other vegetable oils (Kulkarni and Sawant, 2003). Although castor has potential for very high oil yield, the fact that it is considered a weed by many agricultural producers certainly limits its viability as an option to produce significant amounts of biodiesel. In spite of the fact that transesterification reduces the viscosity significantly, the biodiesel capacity of the castor oil is still being researched to find out whether the final viscosity of castor oil biodiesel is within acceptable range for use in the diesel engines.

Table 2: Properties of biodiesel various types of vegetable oils. (Barnwal and Sharma 2005)

Vegetable oil	Kinematic viscosity at 38 °C (mm ² /s)	Cetane no.	Heating value (MJ/kg)	Cloud point (°C)	Pour point (°C)	Flash point (°C)	Density (kg/l)
Corn	34.9	37.6	39.5	−1.1	−40	277	0.9095
Cottonseed	33.5	41.8	39.5	1.7	−15	234	0.9148
Crambe	53.6	44.6	40.5	10.0	−12.2	274	0.9048
Linseed	27.2	34.6	39.3	1.7	−15.0	241	0.9236
Peanut	39.6	41.8	39.8	12.8	−6.7	271	0.9026
Rapeseed	37.0	37.6	39.7	−3.9	−31.7	246	0.9115
Safflower	31.3	41.3	39.5	18.3	−6.7	260	0.9144
Sesame	35.5	40.2	39.3	−3.9	−9.4	260	0.9133
Soya bean	32.6	37.9	39.6	−3.9	−12.2	254	0.9138
Babassu	30.3	38.0	—	20.0	—	150	0.9460
Sunflower	33.9	37.1	39.6	7.2	−15.0	274	0.9161
Palm	39.6	42.0	—	31.0	—	267	0.9180
Diesel	3.06	50.0	43.8	—	−16	76	0.8550

2.5 Properties of *Moringa oleifera* methyl esters

The cetane number of *M. oleifera* methyl esters was determined to be 67.07 using an Ignition Quality Tester™ (IQT™) described previously (Knothe et al., 2003). The cetane numbers of methyl oleate, methyl palmitate and methyl stearate are 59.3, 85.9 and 101, respectively, in the IQT™ (Knothe et al., 2003). Considering that the other saturated fatty acid methyl esters (C20:0 and C22:0) in MOME as well as C22:1 likely have high cetane numbers, the high cetane number of MOME is well-explained. MOME appears to be a biodiesel fuel with one of the highest cetane numbers ever reported for a biodiesel fuel. *M. oleifera*-derived biodiesel easily meets the minimum cetane number requirements in both the ASTM D6751 and EN 14214 biodiesel standards, which are 47 and 51, respectively.

The kinematic viscosity at 40 °C of MOME was determined to be 4.83 mm²/s at 40 °C. The kinematic viscosity values of methyl oleate, methyl palmitate and methyl stearate are 4.51, 4.38 and 5.85 mm²/s, respectively, at 40 °C (Knothe and Steidley, 2005). The contributions of the C20:0, C22:0 and C20:1 esters, with the saturated esters being solids at 40 °C, would lead to high viscosity values. Thus, this result agrees well the viscosity values of the individual fatty ester components. *Moringa oleifera* methyl esters thus meet

the requirements of both the ASTM D6751 and EN 14214 biodiesel standards, which prescribe viscosity ranges of 1.9–6.0 and 3.5–5.0 mm²/s, respectively.

MOME displayed a cloud point 18 °C and a pour point of 17 °C as shown in Table 3. These values are rather high and resemble those for palm oil which also contains even higher amounts of saturated fatty acids. However, the relatively high content of C22:0, which possesses an even higher melting point than C16:0 or C18:0, in *Moringa oleifera* oil likely has the effect of compensating for the higher amounts of saturated fatty acids in palm oil. The reason is that the cold flow properties of biodiesel are determined by the amounts of higher-melting components (usually the saturated esters) and not their nature (Imahara et al., 2006). Thus, decreasing the amounts of higher-melting saturated fatty esters is the only method for improving cold flow properties. The cloud point is the parameter contained in the biodiesel standard ASTM D6751, while the European standard EN 14214 prescribes the cold-filter plugging point (CFPP). The cloud point can be correlated with tests such as the CFPP and is more stringent as it relates to the temperature at which the first solids form in the liquid fuel (Dunn and Bagby, 1995).

The oxidative stability of MOME was determined by the Rancimat method EN 14112, which utilizes 3 g of material per test. The average of three tests was 3.61 h (standard deviation = 0.079 h). Thus, MOME met the oxidative stability requirement in the ASTM D6751 standard, which prescribes a minimum of 3 h but did not meet the minimum prescribed in the EN 14214 standard, which is 6 h. The oxidative stability of MOME is considerably reduced compared to the parent oil as shown in table 3. Possible explanations are that the antioxidants naturally present in *Moringa oleifera* oil are either deactivated through the transesterification process and/or removed by the subsequent purification or separation procedures.

Two tests of MOME using the high-frequency reciprocating rig (HFRR) lubricity tester gave ball wear scars of 135 and 138.5 µm. These values are well below the maximum values prescribed in the petrodiesel standards ASTM D975 and EN 590. Thus, MOME displays excellent lubricity, which is in accordance with the results on lubricity for biodiesel derived from other oils or fats (Knothe and Steidley, 2005).

Table 3: Properties of *Moringa oleifera* methyl esters with comparison to standart (Knothe and steidley, 2005)

Property	<i>M. oleifera</i> 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	Report	— ^b
Pour point (°C)	17	— ^a	— ^b
Oxidative stability (h)	3.61	3 min	6 min
Lubricity (HFRR; µm)	135, 138.5	— ^c	— ^c

^a= Not specified.

^b= Not specified. EN 14214 uses time- and location-dependent values for the cold-filter plugging point (CFPP) instead.

^c= Not specified. Maximum wear scar values of 460 and 520 µm are prescribed in petrodiesel standards EN 580 and ASTM D975

3 MATERIALS AND METHOD

3.1 *Materials and Equipment*

Moringa oleifera seeds were collected from some site in Pengkalan Hulu, Perak. The chemicals are methanol, n-hexane, ferric sulphate, magnesium oxide (MgO), anhydrous sodium sulphate with analytical reagent grade. Methyl esters were produced using Soxhlet extractor fitted with a 1-L round-bottom flask, rotary evaporator, magnetic stirrer, and reflux condenser (Zhang et al., 2009).

3.2 *Pre-treatment*

Pre-treatment starts with collection of dry *Moringa oleifera* pods and continues with the cleaning. 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. After undergo cleaning process, the seeds were undergone size reduction by crushing it using mortar and pestle (Ali et al, 2010).

3.3 *Oil Extraction*

Approximately 20g of *Moringa oleifera* seeds are crushed and placed in a soxhlet extractor fitted with 1-L round-bottom flask and a reflux condenser. After extraction for about 1.5 hours with 250 mL of refluxing n-hexane, removal of the solvent was carried out at 50°C under vacuum using rotary evaporator to get crude *Moringa oleifera* oil which is 35% w/w.

3.4 *Preparation of the Catalyst*

In order to have a 1.0%, 0.7% and 0.5% (%w/w) concentration of magnesium oxide (MgO) for every 25 mL of oil used, 0.612, 0.428, and 0.306 gram of MgO was added in the methanol to produce methoxide solution. The solution was prepared in a 250 mL beaker and stirred at room temperature for about 30 minutes to activate the catalyst.

3.5 Pre-treatment

The 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 5000 rpm for 15 minutes. The clean oil was collected to proceed with transesterification.

The experiments were carried out using the parameters shown in Table 4, and 5 for different catalyst and different methanol: oil ratios, respectively.

Table 4: Different catalyst concentration (at constant methanol:oil ratio of 12:1)

Volume of seed's oil (mL)	Catalyst concentration (%w/w)	Volume of methanol (mL)
25 (61.2 gm)	1 (0.612 gm)	300
25 (61.2 gm)	0.7 (0.428 gm)	300
25 (61.2 gm)	0.5 (0.306 gm)	300

Table 5: Different ratio of methanol: oil

Volume of seed's oil (mL)	Catalyst concentration (%w/w)	Ratio (methanol : oil)
25 (61.2 gm)	0.5 (0.306 gm)	12:1 (300:25)
25 (61.2 gm)	0.5 (0.306 gm)	10:1 (250:25)
25 (61.2 gm)	0.5 (0.306 gm)	8:1 (200:25)

3.6 Transesterification

For the transesterification process, there are two parameters to test the biodiesel production as mentioned at Table 1 and 2. For the first one, pour approximately 25 mL of *Moringa oleifera* oil into a round-bottom flask equip with a reflux condenser after the transesterification and heat to 65°C , just below the boiling point of methanol (Zhang et al., 2009). The methanol was poured carefully into the oil. Catalyst concentration and volume of methanol was set according to Table 1. While, stirring speed, reaction time, and the reaction temperature are fixed at 200 rpm, 90 minutes, and 65°C, respectively. For the second parameters, using the same method as the first one but the best catalyst concentration and the volume of methanol was changed according to Table 2. After the

completion of the reaction, magnesium oxide (MgO) was removed by filtration and the product was left to settle overnight in a separating funnel for separation of biodiesel. The lower glycerol layer was drawn off (Zhang et al., 2009).

3.7 Washing

This method was carried out to remove glycerol using heated deionized water at 55°C and poured into biodiesel. The mixture was settled in a separator funnel for 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.

3.8 Removing Excess Water

Biodiesel was heated up to 80°C for 1 hours or until no more bubble on the surface of the biodiesel. The step was carried out to remove excess water from biodiesel.

3.9 Properties Determination

There are few properties that will be determined using specific methods such as cetane number. Cetane number is determined using American Standard Testing Methods D6890 (ASTM D6890). Implementing ASTM D445 and Cannon–Fenske viscometers is used to obtain the kinematic viscosity of biodiesel from *Moringa oleifera* oil. Oxidative stability measurements were carried out using a Rancimat (Metrohm, Herisau, Switzerland, equipped with software for statistical evaluation) and employing the standard EN14112. Cloud and pour point determinations were conducted with a Phase Technology (Richmond, BC, Canada) cloud, pour and freeze point analyser. Density was determined by ASTM D4052. Flash point was determined using Pensky-martens flash point – automatic NPM 440 (Normalab, France) by implementing ASTM D93 standard.

3.9.1 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. The 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).

3.9.2 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 10ml of biodiesel was needed to complete the test.

3.9.3 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.

3.9.4 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. 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.

3.9.5 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, 2009).

4 RESULT AND DISCUSSION

The result were obtained from the transesterification process with different catalyst concentration (0.5, 0.7, 1.0 %w/w) and different methanol to oil ratio (8:1, 10:1, 12:1) using the best catalyst concentration, while the reaction time, stirring speed and the temperature are fixed at 90 minutes, 200 rpm and 65°C, respectively. The tests and analysis are done to the highest biodiesel yield product shown in Table 6 and 7. The main aim is to find the physical characteristics of the biodiesel product produced from *Moringa oleifera* seeds oil.

Table 6: Yield of biodiesel produced using different catalyst concentration.

Volume oil (ml)	Volume methanol (ml)	Catalyst concentration (% w/w)	Weight catalyst (gm)	Biodiesel produced (ml)	Yield (%)
25	300	0.5	0.306	15	60
25	300	0.7	0.428	11	44
25	300	1.0	0.612	8	32

Table 7: Yield of biodiesel produced using different methanol to oil ratio.

Volume oil (ml)	Methanol to oil ratio	Volume of methanol (ml)	Catalyst concentration (% w/w)	Biodiesel product (ml)	Yield (%)
25	8:1	200	0.5	10	40
25	10:1	250	0.5	13	52
25	12:1	300	0.5	15	60

The comparisons between biodiesel produced using different catalyst concentration and different methanol: oil ratios are drawn in Figure 2, and 3, respectively

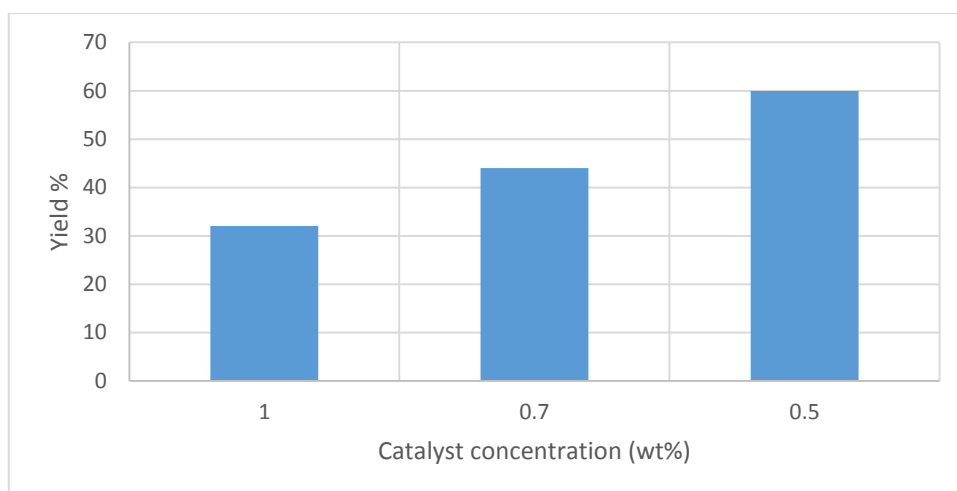


Figure 2: Comparison of yields using different catalyst concentration.

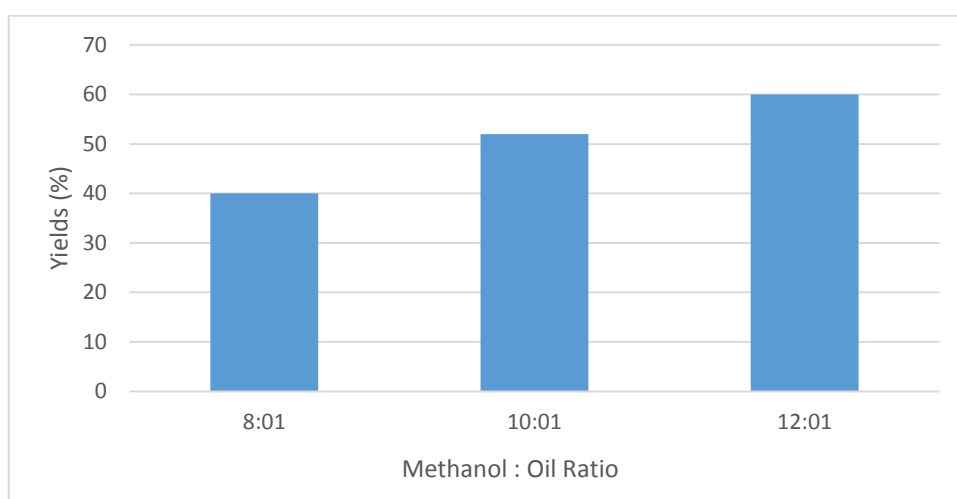


Figure 3: Comparison of yield using different methanol: oil ratios.

Figure 2 above show that the yield of biodiesel produced by using different catalyst concentration. The yield of the catalyst concentration at (0.5, 0.7, and 1.0 %w/w) are about 60%, 44%, and 32%, respectively. In conclusion, the less the catalyst concentration that we used, the more the yield. The best catalyst concentration from this research is at 0.5 %w/w. The oil to methanol ratio was set at 12:1 (25mL: 300mL)

Figure 3 show the yield of biodiesel produced by using different oil to methanol ratio. The yield of the methanol to oil ratio of (8:1, 10:1, 12:1) were (40%, 52%, 60%), respectively. Therefore when ratio of the methanol to oil increase, the yield of the biodiesel also increase. The best methanol to oil ratio from this research is 12:1. The catalyst concentration and the volume of oil was set at 0.5%w/w and 25 mL, respectively.

5 CONCLUSION AND RECOMENDATION

The highest biodiesel yield from the research was about 60% at 0.5 wt% catalyst concentration and for the methanol to oil ratio at 12:1, while the time of reaction, stirring speed, and the temperature were fixed at 90 minutes, 200 rpm and 65°C respectively. The physical characteristic obtained from the biodiesel yield is concluded in Table 8. It can be concluded that the biodiesel can be produced from *Moringa oleifera* seeds oil with 60% yield using MgO as a catalyst which is considered good yield and can be improved by trying MgO concentration less than 0.5 % w/w. it is recommended to use catalyst less than 0.5%w/w, and ratio of methanol to oil ratio higher than 12:1, and different time of reaction for result optimization.

Table 8: Physical properties of *Moringa oleifera* biodiesel (MOME)

Analysis	Transesterification Experiment	ASTM Standard D6751	European Standard 14214
Kinematic viscosity at 40°C (mm ² /s)	4.8	1.9-6.0	3.5-5.0
Cloud point, °C	16	-	-
Pour point, °C	14	-	-
Cetane number	67.07	> 47	> 51
Flash point (oC)	162	> 130	> 120
Density (kg/m ³)	873	870-900	860-900

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APPENDICES

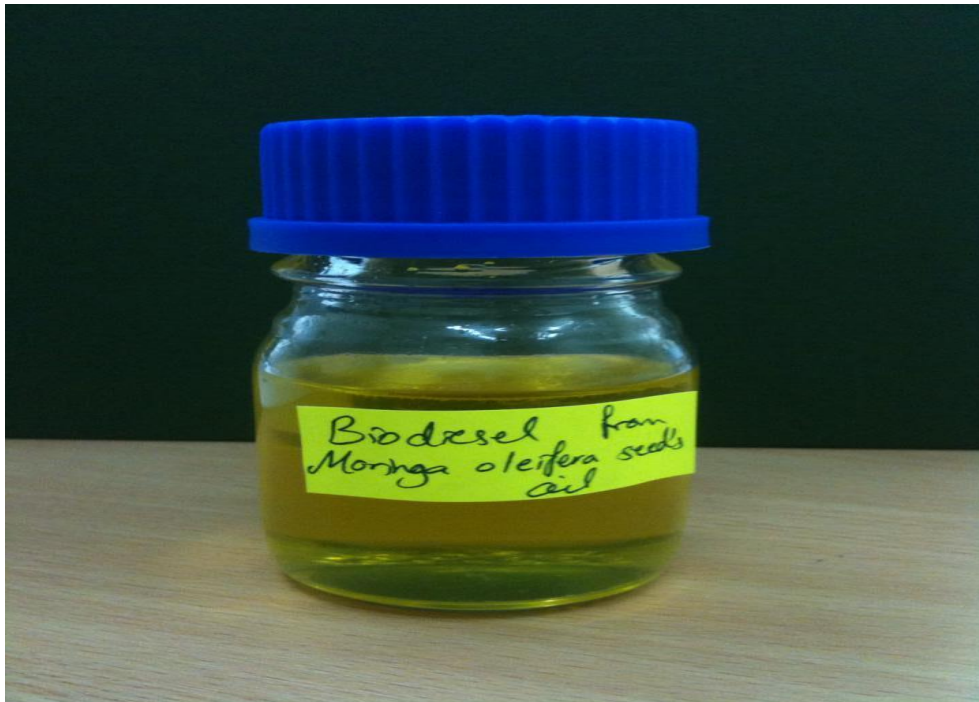
Production process



Appendix 1: Thermal rotary evaporator



Appendix 2: *Moringa oleifera* seeds oil



Appendix 3: Biodiesel from *Moringa oleifera*