

**IMPROVEMENT OF THE COLD FLOW PROPERTY OF BIODIESEL
FROM PALM OIL**

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ABSTRACT

Biodiesel is a low-emissions diesel substitute fuel which is made from renewable sources that consists of the simple alkyl esters of fatty acids. Research and development in the field of biodiesel showed that one way of reducing the biodiesel production costs to compete economically with petroleum diesel fuels is by use the less expensive feedstock containing fatty acids such as inedible oils. This study subjected to minimize the use of biodiesels synthesized from edible oils like palm oils due to raising food and fuel issue, by blending edible palm oil (PO) biodiesel with non-edible rubber seed oil (RSO) biodiesel. Both of the PO and RSO biodiesel were produced by conventional method. Transesterification is a chemical reaction between alcohol and triglyceride in the presence of a catalyst (KOH/NaOH) at certain temperature to produce methyl ester. The different kinds of biodiesels, palm oils and rubber seed oils, were then blended with the different volume ratios (B100, B80, B60, B40 and B0). The blending biodiesels were characterized for kinematic viscosity, pour point and cloud point. Biodiesel is susceptible to start-up and performance problems, consistent with its chemical composition, when vehicles and fuel systems are subjected to cold temperatures. It was found that blending PO with RSO biodiesels has enhanced the cold flow characteristics of biodiesel by reducing the saturated fatty acids in the biodiesels.

ABSTRAK

Biodiesel adalah pengganti bahan api diesel (rendah pelepasan) yang diperbuat daripada sumber yang boleh diperbaharui yang terdiri daripada alkyl ester asid lemak. Penyelidikan dan pembangunan dalam bidang biodiesel menunjukkan bahawa salah satu cara untuk mengurangkan kos pengeluaran biodiesel untuk bersaing dari segi ekonomi dengan bahan api diesel petroleum adalah dengan menggunakan bahan mentah kurang mahal yang mengandungi asid lemak seperti minyak yang tidak boleh dimakan. Kajian ini tertakluk kepada meminimumkan penggunaan biodiesels disintesis dari minyak kelapa sawit seperti minyak sawit berikutan dengan peningkatan bahan makanan dan isu bahan api, dengan mengadun biodiesel dari minyak kelapa sawit yang boleh dimakan (PO) dengan biodiesel dari minyak bijih getah yang tidak boleh dimakan (RSO). Kedua-duanya, PO dan RSO biodiesel dihasilkan melalui kaedah konvensional. Transesterification adalah tindak balas kimia antara alkohol dan trigliserida dalam kehadiran pemangkin (KOH / NaOH) pada suhu tertentu untuk menghasilkan methyl ester. Biodiesel dari minyak sawit dan minyak biji getah, kemudian dicampur dengan nisbah jumlah yang berbeza (B100, B80, B60, B40 dan B0). Pengadunan biodiesels telah dicirikan untuk kelikatan kinematik, tuangkan titik dan titik awan. Biodiesel mendapat start-up dan masalah prestasi, selaras dengan komposisi kimia, apabila kenderaan dan sistem bahan api tertakluk kepada suhu sejuk. Kajian ini menunjukkan bahawa adunan biodiesel dari minyak kelapa sawit dengan biodiesel dari minyak bijih getah telah meningkatkan ciri-ciri aliran sejuk biodiesel dengan mengurangkan asid lemak tepu biodiesels.

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LIST OF SYMBOLS

°C	Degree Celcius
g	Gram
mL	Mililitre
%	Percentage

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Material
PP	Pour point
CP	Cloud Point
KV	Kinematic Viscosity
TG	Triglycerides
FFA	Free Fatty Acid
CFPP	Cold Filter Plugging Point
FAME	Fatty Acid Methyl Ester

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

In Malaysia, biodiesel production is synonymous to palm oil as all of the established biodiesel production chains in Malaysia are using palm oil as primary feedstock. As a matter of fact, the thriving plantation of palm oil is the main factor which drives Malaysia towards developing biodiesel production and technology. The current approved installed capacity for biodiesel production is about 10.2 million tonnes in Malaysia. Therefore, the availability and accessibility of palm oil supply are crucial in determining the potential growth of biodiesel production in Malaysia. Different from other countries such as U.S. which mainly utilizes soybean oil while Europe utilizes rapeseed oil, biodiesel produced in Malaysia from palm oil offers several distinct advantages. Besides requiring less manual labour for harvesting, oil palm plant is also well-known with high yield of vegetable oil. A hectare of oil palm can produce approximately 5 tonnes of palm oil, compared with other vegetable oils like rapeseed and soybean, which can produce 1 tonne and 375 kg each (Lim & Teong, 2010). However, the major disadvantage of biodiesel is the inverse relationship of oxidation stability of biodiesel with its low temperature properties which includes cloud point and pour point. Higher composition of saturated fatty acids in feedstock will increase the oxidation stability of biodiesel but it will lower its cloud points and pour points. Whereas, higher composition of unsaturated fatty acids will enhance the cloud point and pour point of biodiesel but will have poor oxidation stability. Hence, a balance has to be maintained between the ratio of saturates and unsaturated for the oil to be used as a feedstock for biodiesel production (Sharma et al., 2008).

1.2 PROBLEM STATEMENT

Currently, more than 95% of the world biodiesel is produced from edible oils which are easily available on large scale from the agricultural industry. The use of non-edible plant oils when compared with edible oils is very significant in developing countries because of the tremendous demand for edible oils as food, and they are far too expensive to be used as fuel at present. The production of biodiesel from by blending different non-edible oil seed crops has been extensively investigated over the last few years. Although biodiesel is environmentally compatible, it has some limitations. The major limitation of biodiesel is to crystallize or gel at low temperature. The low temperature flow property of biodiesel is characterized by the CFPP. The CFPP is the temperature at which a fuel causes a filter to plug due to its crystallization. The CFPP of biodiesel is depends on the fatty acid compositions. High CFPP values of biodiesel can be explained by high contents of the saturated fatty acids because the unsaturated fatty compounds have lower melting points than the saturated fatty compounds. Although the transesterification does not alter the fatty acid composition of the feedstock, but this composition plays important roles in influencing certain critical parameters of biodiesel. Blending of biodiesels with different fatty acid compositions is therefore expected to improve the oxidation stability and cold temperature flow properties of biodiesel.

1.3 OBJECTIVES

The proposed research was studied to achieve the following objectives:

- i. To improve the cold flow properties of biodiesels by blending edible palm oils biodiesel and non-edible rubber seed oils biodiesel with different ratios in composition.
- ii. To investigate the effects of the fatty acid compositions in the blended biodiesels on the degree of saturation of fatty acids.

1.4 RESEARCH SCOPES

In order to achieve the objectives stated above, the following scopes of study have been drawn.

- i. Focus on the improvement of CFPP of biodiesel derived from palm oil via blending with non-edible oil biodiesel.
- ii. The goal is to achieve good CFPP of blending biodiesels, as well as to reduce the palm oil content in biodiesel, so that the price of biodiesel was lower.

1.5 RATIONALE AND SIGNIFICANCE

Based on the research scopes mentioned above, the following rationale and significance that we could get have been outlined.

- i. It shall reduce the low cold flow temperature of blending biodiesels
- ii. It shall increased the production of biodiesels from non-edible oils
- iii. Alternative way to produce valuable product from palm oils and rubber seed oils
- iv. New substitute of raw material for biodiesels production
- v. It shall reduce environmental problem as biodiesel is environmentally friendly alternative to conventional diesel fuel.

CHAPTER 2

LITERATURE REVIEW

2.1 BACKGROUND

Biodiesel is popularly known as an alternative diesel fuel especially as diesel substitute in developed countries mainly for transportation and agriculture industries. The American Society for Testing and Material (ASTM) defined biodiesel fuel as monoalkyl esters of long chain fatty acids derived from renewable lipid feedstock such as vegetable oils, animal fats or waste oils. Biodiesel is highly biodegradable, low toxicity, possesses inherent lubricity and a relatively high flash point 154°C and reduces most regulated exhaust emissions in comparison to regular diesel (Smith et al., 2010 & Dunn, 2009). It is a technically competitive and environmentally friendly alternative to conventional fossil-derived diesel fuel for use in compression-ignition engines (Perez et al., 2010). While most of the properties of biodiesel are comparable to petroleum based diesel fuel, improvement of its low temperature flow characteristic still remains one of the major challenges when using biodiesel as an alternative fuel for diesel engines.

2.2 BIODIESEL PRODUCTION

The most common way to produce biodiesel is by transesterification reaction of vegetable oils or animal fats with an alcohol. Dunn (2010) stated in his research, that the chemical structures of these oils are characterized as triacylglycerols (TAGs) consisting of long-chain fatty acid groups attached by an ester linkage to a glycerol (1,2,3-propanetriol) backbone. They are converted to biodiesel by transesterification with a short-chain monohydric alcohol such as methanol or ethanol, normally in the presence of catalyst and

elevated temperature. This process may leave behind very small (trace) concentrations of minor constituents such as saturated monoacylglycerols (MAGs) or free steryl glucosides (FStGs). These materials have high melting points and very low solubilities allowing them to form solid residues when stored during cold weather. Many processes wash the separated biodiesel with water to remove trace concentrations of glycerol, alcohol and catalyst. Transesterification reactions are reversible and a molar excess of alcohol is usually supplied to drive the mechanism towards the desired products. The simplified form of its chemical reaction is as presented below equation:

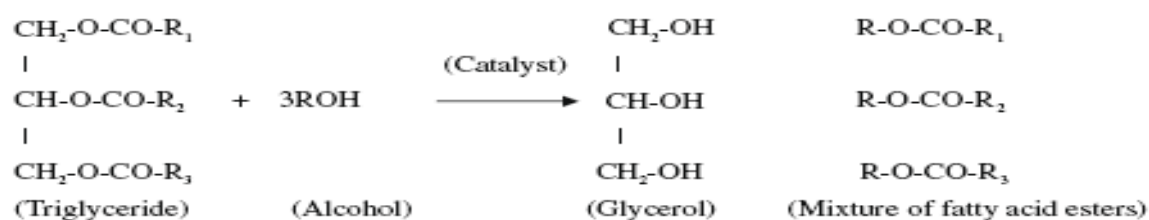


Figure 2.1 Transesterification reactions

The reaction can be alkali-catalyzed, acid-catalyzed and enzyme-catalyzed or carried out under supercritical conditions (Liu et al., 2009). The reaction is fast, inexpensive and high-yielding when using a homogeneous alkali catalyst such as sodium or potassium hydroxide or methoxide. Moisture should be avoided with alkali catalyst because water can hydrolyze biodiesel resulting in the formation of free fatty acids or fatty acid esters of sodium or potassium (soaps). The final composition of biodiesel depends on the fatty acid composition of its feedstock oil. Vegetable oils and fats may contain small amounts of water and free fatty acids (FFA). For an alkali-catalyzed transesterification, the alkali catalyst that is used will react with the FFA to form soap.

Below equation show the saponification reaction of the catalyst (sodium hydroxide) and the FFA, forming soap and water.



Figure 2.2 Saponification reaction of the catalyst (sodium hydroxide) and the FFA

This reaction is undesirable because the soap lowers the yield of the biodiesel and inhibits the separation of the esters from the glycerol. In addition, it binds with the catalyst meaning that more catalyst will be needed and hence the process will involve a higher cost. Water, originated either from the oils and fats or formed during the saponification reaction, retards the transesterification reaction through the hydrolysis reaction. It can hydrolyze the triglycerides to diglycerides and forms more FFA. However, the FFA can react with alcohol to form ester (biodiesel) by an acid-catalyzed esterification reaction. This reaction is very useful for handling oils or fats with high FFA, as shown in the equation below:

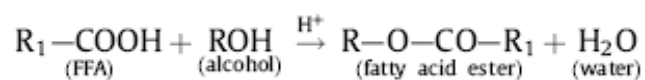


Figure 2.3 Acid-catalyzed esterification reaction

According to Tan et al. (2010), they proposed that supercritical methanol method has received significant attention due to its catalyst-free reaction medium. It was reported that this non-catalytic process can produce biodiesel within a relatively short time compared to catalytic methods.

2.3 RENEWABLE SOURCES FOR RAW MATERIAL IN BIODIESEL PRODUCTION

There are several sources that can be use as raw material for biodiesel production that is non-edible oil, animal fats and vegetable oil. The raw material must contain triacylglycerols (triglycerides) which consist of three long chains fatty acid esterifies to a glycerol back bone. Vegetable oil is divided to two main categories, edible oil and non-edible oil. Edible oil is oil which is use in food industry while non-edible oil is oil which is not use in food industry. Normally, non-edible oil is from vegetable oil which is growth wildly and can survive in bad weather condition (Ismail, 2008). Six oil crops clearly dominate worldwide feedstock sources are soybean, rapeseed, sunflower, palm cottonseed and peanut oil (Perez et al., 2010).

Table 2.1 Feed-stocks categories of biodiesel production

Category	Classification	Feedstocks
1. Oilseeds	edible	C: Soybean, rapeseed/canola, sunflower, palm, coconut, olive A: False fax, safflower, sesame, marula, pumpkin, African peer seed, <i>Sclerocarya birrea</i> , <i>Terminalia catappa</i> L., yellow nut-sedge tuber, rice bran
	inedible	A: Jatropha, karanja, mahua, linseed, ruberseed, cottonseed, neem, camelina, putranjiva, tobacco, polanga, cardoon, deccan hemp, castor, jojoba, moringa, poon, koroch seed, desert date, eruca sativa gar, see mango, pihu, crambe, syringa, milkweed, field pennycress, stillingia, radish Ethiopian mustard, tomato seed, kusum, cuphea, camellia, paradise, cuphea, treminalia, michelia champaca, garcinia indica, <i>Zanthoxylum bungeanum</i>
2. animal fats		C: Beef tallow, pork lard A: Waste salmon, melon bug, sorghum bug, chicken fat
		C: Cooking oil, frying oil A: Vegetable oil soapstocks, acid oils, tall oil, dried distiller's grains (DDG), pomace oil
3. waste materials		
4. algae		<i>Botryococcus braunii</i> , <i>Chlorella</i> sp., <i>Chlorella vulgaris</i> , <i>Cryptocodinium choii</i> , <i>Cylindrotheca</i> sp., <i>Dunaliella primolecta</i> , <i>Dunaliella salina</i> , <i>Isochrysis</i> sp., <i>Haematococcus pluvialis</i> , <i>Monallanthus salina</i> , <i>Muriellopsis</i> sp., <i>Nannochloris</i> sp., <i>Nannochloris oleabundans</i> , <i>Nitzschia</i> sp., <i>Phaeodactylum tricoratum</i> , <i>Porphyridium cruentum</i> <i>Schizochytrium</i> sp., <i>Spirulina</i> , <i>Arthrospira platensis</i> , <i>Tetraselmis sueica</i>

Source: No, S.Y, 2011, *Renewable and Sustainable Energy Reviews 15*.

The different types of edible vegetable oils and biodiesels as substitutes for diesel fuels are considered in the different countries depending on the climate, soil conditions and

availability of the raw material. Generally, the most abundant vegetable oil in a particular region is the most commonly used feedstock for biodiesel production. For example, soybean oil in the USA, rapeseed (canola in Canada) and sunflower oils in Europe, palm oil in South-east Asia (mainly Malaysia, Indonesia and Thailand), coconut oil in the Philippines and cottonseed oil in Greece and Turkey are being produced (No, S.Y, 2011).

Table 2.2 Properties of Vegetable Oils

Table 1
Properties of vegetable oils.

Property	Rubber seed oil	Coconut oil	Palm Kernel oil
Fatty acid composition (%)			
(1) Lauric acid C _{12:0}	—	46.5	0.1
(2) Myristic acid C _{14:0}	—	19.2	1
(3) Palmitic acid C _{16:0}	10.2	9.8	42.8
(4) Stearic acid C _{18:0}	8.7	3	4.5
(5) Oleic acid C _{18:1}	24.6	6.9	40.5
(6) Linolic acid C _{18:2}	39.6	2.2	10.1
(7) Linolenic acid C _{18:3}	16.3	0	0.2
Specific gravity	0.883	0.887	0.866
Viscosity (mm ² /s) at 40 °C	34.69	29.6	38.59
Flash point	237	282	260
Calorific value (MJ/kg)	38.44	37.94	35.78
Acid value (mg KOH/g)	48	2.1	4.5

Source: Department of Mechanical Engineering, NIT Campus, India, 2010.

2.4 THE COLD FLOW PROPERTIES OF BIODIESEL

Biodiesel is susceptible to start-up and performance problems when vehicles and fuel systems are subjected to cold temperatures. In winter, crystallization of high melting saturated fatty acid methyl esters may lead to the plugging of filters and tubes. The cold flow properties of biodiesels are quantified using standard Cloud Point (CP), Pour Point (PP), and Cold Filter Plugging Point (CFPP) tests. These tests establish limits for the use of

fuels under cold weather conditions. Initially, cooling temperatures cause the formation of solid wax crystal nuclei that are submicron in scale and invisible to the human eye. Further decreases in temperature cause the crystal nuclei to grow (Perez et al., 2010). The cloud point (CP) is defined as the temperature at which the smallest observable cluster of wax crystals first appears because the crystals usually form a cloudy or hazy suspension. The pour point (PP) is the lowest temperature at which movement of a test specimen is observed and the cold filter plugging point (CFPP) is then defined as an estimation of the lowest temperature at which a fuel will give trouble-free flow in certain fuel systems (Pope & Hasan, 2009). According to Perez et al., 2010, CFPP can be defined as the lowest temperature at which 40ml of oil safely passes through the filter within 60 seconds. A property of biodiesel that currently limits its use to blends of 20% or less is its relatively poor low-temperature properties. Neat biodiesel can solidify in fuel lines or clog filters when utilised in cold ambient conditions. While the cloud point (CP) of petroleum diesel is reported to be 16°C, biodiesel typically has a CP of 0°C. This limits its application to ambient temperatures above freezing (Smith et. al, 2009). The low temperature flow properties of biodiesel are characterized by the CFPP. The CFPP is the temperature at which a fuel causes a filter to plug due to its crystallization or gelation. The CFPP of biodiesel also depends on the fatty acid compositions (Park et. al, 2008).

2.4.1 Viscosity

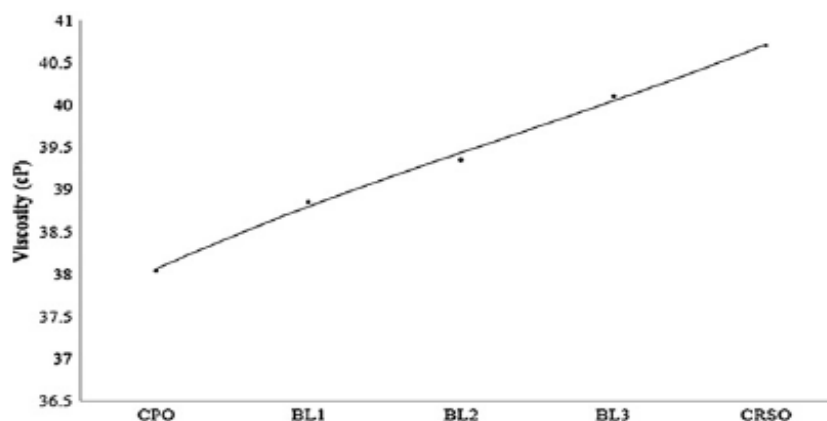


Fig. 4 – Kinematic viscosity analysis for CPO, CRSO and their blends.

Figure 2.4 Kinematic viscosity analysis for CPO, CRSO and their blends

One of the important properties of the oils is the viscosity since biodiesel viscosity needs to meet the criteria of international standards and that for the oils need to be lowered through certain processes. A blend of crude rubber seed oil and crude palm oil had been investigated by Khan, M. & Yusup, S., 2010, as a potential feedstock for biodiesel production. The highest density and viscosity were in rubber seed oil. Viscosity can lead to engine mal-function and high densities are not recommended by the international standards. Transesterification of the oil have shown to rectify these properties to match the acceptable range. Blends of crude palm oil and rubber seed oil were prepared at different volumetric ratios of 30:70, 50:50 and 70:30 (vol%:vol%) respectively. Moreover, the degree of saturation has strong relation with viscosity and melting point where the degree of saturation in oils increases with viscosity. As it can be seen from figure the value of viscosity increased toward crude rubber seed oil. On the other hand, the extra viscosity is an added advantage for agricultural based oils. It provides additional aid for injector lubrication as when compared with that of conventional diesel and prevents leakage or increase wear caused by imprecise fit of fuel injection pumps resulting from low viscous

fuel. Reduction in saturated fatty acids from crude palm oil resulted from blending can lower the pour point of biodiesel produced. The blends were pre-assessed as well for the amount of pollutant present, specifically sulphur, and were found to be in the acceptable range of the international standards.

2.4.2 Pour Point and Cloud Point

For the cloud point test the specimen is cooled at a specified rate and examined periodically. The temperature at which a cloud is first observed at the bottom of the test jar is recorded as the cloud point. After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3oC for flow characteristics. The lowest temperature at which movement of the specimen is observed is recorded as the pour point. According to Udomsap, P. et al, 2008, the usage of B100 from palm stearin at low temperature during the cooler climate in Thailand might pose a cold flow problem. To solve this problem, the biodiesel could be blended with diesel at 40% (B40) for conservative limit or 60% (B60) for practical recommendation. PSME starts to form wax crystal below 19.4oC, and can not flow at all below 18oC, which would definitely clog fuel filters, fuel lines and fuel injector in the engine. As pour point (PP) is equivalent to melting point, the pour point of biodiesel hence depends on the melting point of fatty acid compositions. The melting point depends on the chain length and saturation. Saturated fatty acid compound has much higher melting point than the unsaturated one because the double bonds retard the solidification to lower temperature upon cooling. Within the saturated fatty acid compound, longer chain length requires higher enthalpy for melting.

2.5 IMPROVEMENT OF THE COLD FLOW PROPERTY

Several approaches have been proposed to improve the low temperature properties of biodiesel, including blending with petroleum diesel, the use of additives and the chemical or physical modification of either the oil feedstock or the biodiesel product. Blending with petroleum diesel is only effective at low biodiesel proportions (up to 30 vol %) with cloud

points to around -10°C . Clearly, blends with petroleum diesel do not change the chemical nature and therefore properties of biodiesel and will not facilitate their use at higher concentrations (Smith et. al, 2010).

2.5.1 ADDITIVES TREATMENT

Treatment with chemical additives is the most convenient and economical way of improving the low temperature properties of diesel fuels. The chemical additives are generally referred to as pour point depressants, flow improvers or wax modifiers. Most additives promote the formation of small (10–100 μm) needle shaped crystals. These crystals experience significantly reduced growth and agglomeration rates as temperature decreases below cloud point. However, the rate of nucleation is promoted and causes the formation of a large quantity of the relatively small and more compact crystals. Although most of these crystals will be caught in fuel filters, the cake layer formed on the filter surface is considerably more permeable to fuel flow. Boshui et al., (2010), proposed that amongst the several approaches, treatment with chemical additives seems to be the most convenient and economical, and thereby the most attractive. Most studies reported above found that most additives act as crystal modifiers which reduce the size and shape of wax crystals and create a barrier to crystal agglomeration, however, they do not alter the cloud point (Dunn, 2009; Perez et.al, 2010). As such, more studies are needed to address this concern.

2.5.2 CRYSTALLIZATION OF BIODIESEL

Smith et al. (2010) study that winterization is a method for separating that fraction of oils with a solidification temperature below a specific cut-off. One technique involves refrigeration of the oils for a prescribed period at a specific temperature followed by decanting of the remaining liquid. Another, more energy efficient method is to allow tanks of oil to stand outside in cold-temperatures for extended periods of time. In either case, the fraction that remains molten is separated from the solid producing oil with improved pour

and handling qualities. The nature of biodiesel suggests that crystallization fractionation may be a useful technique for decreasing CP by reducing the saturated alkyl ester content. Crystallization fractionation involves the separation of the components of lipids (such as vegetable oils, fats, fatty acids, fatty acid esters, monodiglycerides and other derivatives) based on differences in crystallization temperatures. The traditional fractionation process includes two stages. The crystallization stage consists of selective nucleation and crystal growth under a strictly controlled cooling rate combined with gentle agitation. Once well-defined crystals with a narrow distribution of specific sizes and characteristics are formed, the resulting slurry is transferred to the second stage for separation into solid and liquid fractions, typically by filtration or centrifugation (Perez et al., 2010). Winterization has also been employed to reduce the pour point of biodiesel by lowering its saturated fatty acid methyl ester components. To achieve significant reductions in pour point, several winterization steps are required to achieve a yield of higher than 25–26% and thereby render this technique viable. The other option is winterizing methyl esters in various solvents. Because of the poor yields of the winterized unblended methyl esters from common oils, researchers have attempted to dilute the esters with various solvents. Methanol, acetone, chloroform, and hexane have been explored as diluting solvents. Methanol offers the advantage that the winterization may be easily integrated into the industrial biodiesel production facilities. Among the different winterization configurations studied, the best results were obtained through solvent winterization with methanol. The biodiesel obtained from this process contains long-chain saturated compounds at a concentration of 0.93 wt. % with a CFPP of -8°C and exhibits a liquid mass percentage of 91.07 wt. % with respect to the initial mass.

2.5.3 BLENDING OF BODIESELS

According to Park et al. (2008), blending of biodiesels with different fatty acid compositions is therefore expected to improve the oxidation stability and cold temperature flow properties of biodiesel. Palm biodiesel with a high content of palmitic acid shows high oxidation stability but has poor low temperature flow properties with $9\text{--}11^{\circ}\text{C}$ of CFPP. Rapeseed biodiesel with a high content of oleic acid shows good low temperature flow

properties with -19 to -8 °C of CFPP. When palm and rapeseed biodiesels are blended, the blended biodiesel will have higher oxidation stability than rapeseed biodiesel and lower CFPP than palm biodiesel. When palm biodiesel was blended with rapeseed biodiesel, the oxidation stability and the CFPP of the blended biodiesels increased with weight percent of palm biodiesel. The high CFPP of palm biodiesel was lowered by supplementing rapeseed biodiesel having remarkably low CFPP. When rapeseed biodiesel became more than 50 wt%, the CFPP of the blended biodiesel dropped to below 0°C. When palm biodiesel was blended with soybean biodiesel of low oxidation stability, the oxidation stability and the CFPP of the blended biodiesels increased with weight percent of palm biodiesel. By adding palm biodiesel having high oxidation stability, low oxidation stability of soybean biodiesel was improved.

CHAPTER 3

METHODOLOGY

3.1 PROCESS FLOW CHART

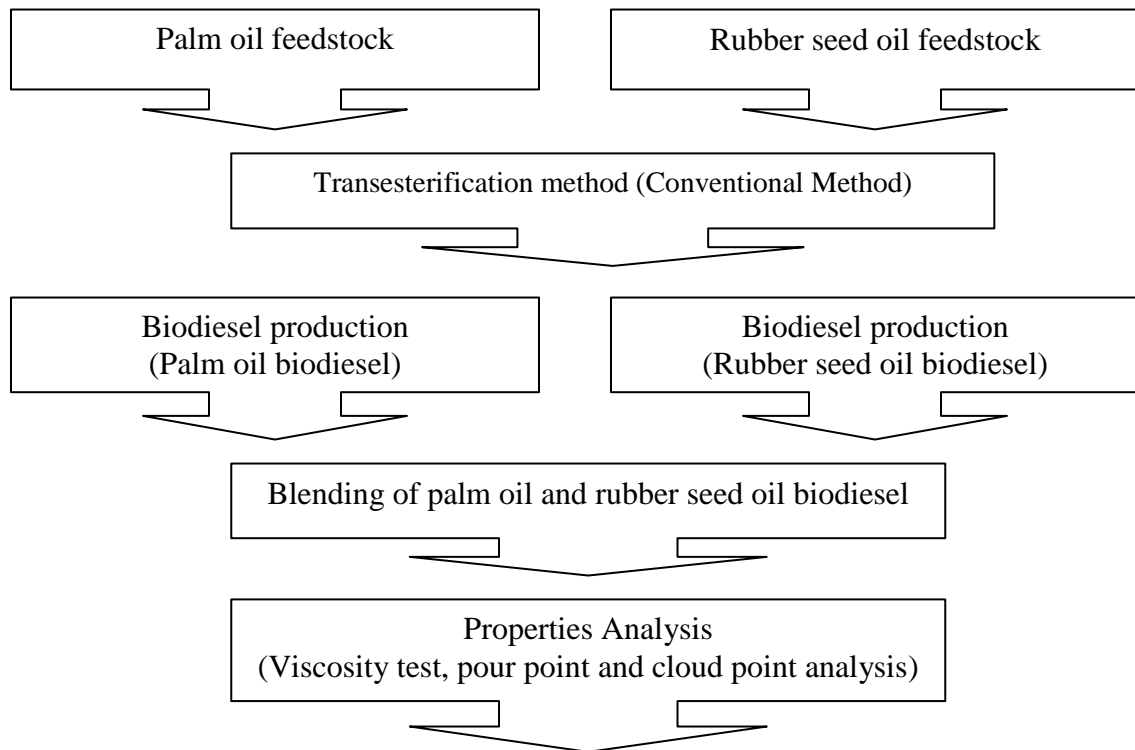


Figure 3.1 Flowchart of Biodiesel Production