Synthesis of Biodiesel from Ceiba Pentandra using Microwave Assisted Technique

By

Tan Meng Kiat

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
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Universiti Teknologi PETRONAS
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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

TAN MENG KIAT

ABSTRACT

The rapid growth of industrial and human population had encouraged the exploration of renewable fuel such as biodiesel which is produced from plant oil and animal fat. Ceiba Pentandra which is one of the non-edible feedstocks, is much explored in Malaysia for biodiesel production. Biodiesel is usually produced by transesterification process using conventional heating which requires large amount of heat energy and long reaction time. This project studies on Kapok oil methyl ester (KOME) production using microwave assisted technique. Studies have been conducted to investigate the optimum operating conditions for the microwave assisted transesterification of Ceiba Pentandra seed oil including the temperature, catalyst concentration, methanol to oil molar ratio and irradiation time. The optimum operating conditions which result in highest KOME yield (95.6%) are found to be at 55 ℃, 2 wt% KOH catalyst, 10:1 methanol to oil molar ratio and 3.5 minutes of irradiation time. The microwave synthesis reactor which is able to deliver energy directly to the reactant at molecular level has drastically reduced the reaction time and total energy consumption in the transesterification process. The properties of the biodiesel produced under the optimum conditions are characterized and it shows that the KOME meets the international standard of biodiesel (ASTM 6751 & EN 14214).

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CHAPTER 1
INTRODUCTION

This chapter briefs about the background study followed by the problem statement and objectives of the project. Also, the scope of study is included.

1.1 Background Study

The fast growing population, rapid modernization and industrialization have relentlessly increased the demand of energy in developing countries like Malaysia. The demand of energy is mostly met from non-renewable resources such as petrochemicals, natural gas and coal. These sources are non-renewable and have serious implications on environment. Hence, development of alternative fuel technology is essential.

Biodiesel, a renewable biofuel has been considered as an alternative diesel fuel. Knothe (2007) defined it as the mono alkyl ester of long chain fatty acid derived from the vegetable oil and animal fat. Unlike fossil fuels, available sources for biodiesel production is unlimited and varies, since it is produced from renewable sources, hence, able to cope with energy demand consumption that is increasing rapidly. Biodiesel is known to have low emission of pollutant. This makes it to be a more attractive choice. It is biodegradable, non-toxic and has low emission profiles as well as environmental friendly. A life cycle analysis of biodiesel showed that overall carbon dioxide emissions were reduced by 78% compared with petroleum-based diesel fuel (Gerpen, 2005).

It can be easily produced through transesterification of oil or esterification of fats using basic or acidic catalysts with heating functions, respectively. Direct utilization of vegetable oil is not recommended due to viscosity problem (Ramadhas et al., 2003). Thus, conversion of different types of edible and non edible oils is explored. Utilizing edible oil as biodiesel feedstock will cause fuel versus food controversy. Therefore, more sources of non edible oil are being explored and studied as potential feedstock for biodiesel.

Among the non edible feedstocks used for researches in biodiesel production are Moringa Oleifera seed oil, tobacco seed oil, Jatropha Curcas seed oil and caster seed oil. New sources from non edible feedstock for biodiesel production have been explored and it is found that Ceiba Pentandra seed oil has potential to be another biodiesel feedstock. Ceiba Pentandra seed oil which is locally known as *Kapok* or *Kekabu* is a native in America and West Africa (Salimon and Khairul Asmak, 2005).

Figure 1.1: Ceiba Pentandra fruit pods with imbedded seeds

Ceiba Pentandra belongs to the Malvaceae family (Abdullah et al., 2010). It is grown naturally in humid and sub humid tropical region. Ceiba Pentandra is generally drought-resistant tree and pods from these trees are leathery, ellipsoid and pendulous capsule (Sivakumar et al., 2013). Ceiba Pentandra seeds occupy about 25-28% (w/w) of each fruit (Walia et al., 2009). The oil seed yield is producing on average 1280kg/ha. The seeds are low feeding value due to its higher fiber content. Tye et al. (2012) reported that kapok fiber contains 34-64% of cellulose and high potential to produce cellulosic ethanol. Traditionally, kapok fibers are utilized as stuffing material for beds and pillows. Besides, Salimon and Khairul Asmak (2005) reported kapok seeds contain relatively high crude oil which is about 22-25% by weight but up till now it is still underutillized.

Many studies had been done on producing biodiesel from kapok seed oil using the conventional heating method. Sivakumar et al. (2013) presented the work on biodiesel production from Ceiba Pentandra seed oil. It was able to achieve 99.5% conversion under optimized conditions using conventional heating method. The main problems faced by researchers using conventional heating are large amount of heat energy and long reaction time are needed to achieve high conversion of oil. It will impact the production cost thus the commercialization potential.

Therefore, this project comes out with a well-established technique to deliver energy directly to the reactant at shorter reaction time which is microwave assisted technique. It offers higher biodiesel yield and selectivity within minutes instead of hours. None of the reported literature on biodiesel production emphasized on utilization of from Ceiba Pentandra seed oil using microwave technique for biodiesel production. Therefore, the present project attempts to explore the potential of utilizing indigenously available Ceiba Pentandra seed oil as the biodiesel feedstock using microwave technology.

1.2 Problem Statement

Several problem statements of this research are:

(i) What are the optimized conditions for the production of Ceiba Pentandra biodiesel using microwave assisted technique?

(ii) Will microwave assisted technology enhance the oil and fatty acid alkyl ester yield derived from Ceiba Pentandra seed?

(iii) Will energy consumption be reduced with the application of microwave assisted transesterification as opposed to conventional transesterification?

1.3 Objectives

This study embarks on the following objectives:

(i) To investigate the optimum operating condition for the microwave assisted transesterification of Ceiba Pentandra seed oil including the effects of solvent, catalyst concentration, microwave irradiation time and temperature.

(ii) To characterize the oil and the biodiesel product following international standard (ASTM 6751 & EN 14214).

1.4 Scope of Study

This project focus on the application of microwave assisted technique to produce biodiesel from Ceiba Pentandra seed oil. Optimization of the four parameters affecting transesterification process including effect of solvent, catalyst concentration, microwave irradiation time and temperature will be studied. The response surface methodology (RSM) which is a collection of mathematical and statistical techniques for empirical model building will be used for the optimization purpose. Comparison will be made between results of experiments using conventional heating and microwave assisted technique. Study will also be made to see if microwave heating is able to reduce the overall energy consumption for biodiesel production. The biodiesel product produced from optimized condition with highest yield is to be characterized and compared with the international standard for biodiesel. The European biodiesel standard, EN 14214 and American standard, ASTM 6751 are used as reference. The properties of biodiesel to be characterized include:

- Density
- Acid value
- Water content
- Flash point
- Cloud point
- Pour point
- Oxidative stability
- Kinematic viscosity
- Cetane number
- Higher heating value
- Cold filter plugging point

CHAPTER 2

LITERATURE REVIEW

2.1 Kapok Seed

Kapok seed is one of the non-edible oil sources. Scientifically, it is known as Ceiba Pentandra. It is native in tropical America and West Africa after which the seeds were introduced to Southeast Asia via India. In Malaysia, the distribution of kapok tree is commonly found in northern part of Peninsular Malaysia.

In 1964 up to 1966, several researches had been done about the Ceiba Pentandra seed oil content as well as the fatty acid composition inside the oil. Ferreira-Dias et al. (2003) reported that Ceiba Pentandra seed oil contain cyclopropenoid fatty acids (CPFA) which can give various types of physiological disorders in farm. Therefore, the seed of Ceiba Pentandra is not suitable for consumption. Instead of abandoning, the seed oil is being fully utilized in producing biodiesel. Several physiochemical properties of Ceiba Pentandra seed oil had been analyzed by Salimon and Khairul Asmak (2005) and it is shown in *Table 2.1.*

Table 2.1: Physiochemical properties of Ceiba Pentandra seed oil

Properties	Average value
Water content (%)	0.04
Refractive index (at 28 ℃)	1.46
Free Fatty Acid, FFA (%)	15.3
Saponification value (mg/g)	208
Iodine value	104

Fatty acid composition is important in order to identify the percentage of saturated and unsaturated fatty acid in the oil. Gas Chromatography Mass Spectroscopy (GC-MS) is used to analyze the percentage of fatty acid of the oil. The free fatty acid in the oil is converted to fatty acid methyl esters (FAME) when Ceiba Pentandra seed oil is converted to biodiesel through transesterification. The fatty acid composition of Ceiba Pentandra seed oil shown in *Table 2.2* was presented in different research work.

Table 2.2 : Fatty acid composition of Ceiba Pentandra

Ceiba Pentandra seed oil	Fatty Acid Composition (wt%)		
	Berry, K. (1979)	Enser, M. (1991)	Yusup et al. (2011)
Caproic acid (C16)	-	-	9.42
Myristic acid (C14)	0.25	-	-
Palmitic acid (C16)	24.31	20.90	23.17
Palmitoleic acid (C16:1)	0.40	-	-
Stearic acid (C18)	2.65	13.70	4.73
Oleic acid (C18:1)	21.88	20.80	22.88
Linoleic acid (C18:2)	38.92	42.60	30.00
Linolenic acid (C18:3)	1.00	1.9	-
Arachidic acid (C20)	-	-	1.18

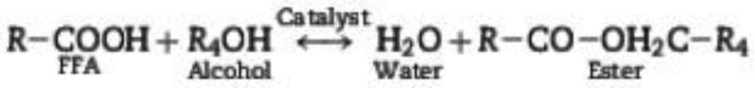
The value of fatty acid was determined in weight percentage. It shows that the Ceiba Pentandra seed oil contained high linoleic acid which is unsaturated fatty acid and it was strongly proven by all the analysis. It can be concluded that, Ceiba Pentandra seed oil contain high unsaturated fatty acid. The oil properties which contained unsaturated fatty acid can be excellent oil for engine performance during cold weather after conversion to biodiesel (Yusup and Khan, 2010).

Determintaion of free fatty acid (FFA) content in the oil is one of the important parts before proceeding to transesterification process. For feedstock which contains less than 2 to 3% of FFA content, pre-treatment process is not required and one-step direct transesterification reaction is sufficient to produce high quality of biodiesel

(Thamsiriroj and Murphy, 2010). Higher content of FFA will reduce the overal conversion of FFA to FAME through base catalyzed transesterification. Therefore, two-step acid catalyzed esterification and base-catalyzed transesterification process have been introduced to reduce the FFA content in seed oil to less than 1% and maximize the conversion of FFA to FAME (Ye et al., 2010).

2.2 Esterification Reaction

Generally, the esterification process is done using acid as the catalyst. Sulphuric acid (H_2SO_4) is commonly used for the reaction. Esterification is relatively slow even though it can give higher biodiesel yield compared to transesterification reaction and it requires the reactor to be in acidic environment (Dube et al., 2007). Esterification reaction as shown in *Figure 2.1* is one of the most important alcoholisms techniques for its ability to neutralize the high FFA content in the biodiesel feedstock.



$$\underset{\text{FFA}}{R-COOH} + \underset{\text{Alcohol}}{R_4OH} \overset{\text{Catalyst}}{\longleftrightarrow} \underset{\text{Water}}{H_2O} + \underset{\text{Ester}}{R-CO-OH_2C-R_4}$$

Figure 2.1: General equation for esterification process

If the pre-treatment process is not done to reduce FFA content, saponification will occur where the FFA will be converted into soaps when base catalyst is intorduced in the transesterification reaction and it will further inhibit the separation of the alkyl esters and glycerol. Saponification reaction of oil and fats which take part with the base catalyst in the transesterification process is shown in *Figure 2.2*.

$$RCOOR' + H2O \rightleftharpoons RCOOH + R'OH$$

$$RCOOH + ROH \rightleftharpoons RCOOR + H2O$$

Figure 2.2: Saponification reaction of fatty acid alkyl ester

These soap and emulsion formations make separation of product and by-product of biodiesel become longer and harder (Serioet et al., 2008). Therefore, acid catalyzed esterification reaction is essestial to reduce the FFA content in the seed oil below 1% to avoid shortcoming of the subsequent reaction, which is based-catalyzed transesterification (Gerpen, 2005).

2.3 Transesterification Reaction

The common method used in biodiesel production is through transesterification process. Also known as alcoholysis, transesterification is the well-established chemical reaction of vegetable oils and animal fats with an alcohol to form fatty acid alkyl esters and glycerol (Leung et al., 2010). Vegetable oils and animal fats cannot be used directly in diesel engines because of the nature of its high viscosity and low volatility. Hence, the main objective of transesterification is to overcome these problems (Yan et al., 2009). Stoichiometrically, 3 moles of alcohol is required for 1 mole of oil for complete transesterification process. However, since the reaction is reversible, excess alcohol is practically used to shift the equilibrium to the product side and thus raising the product yield (Srivastava and Prasad, 2000).

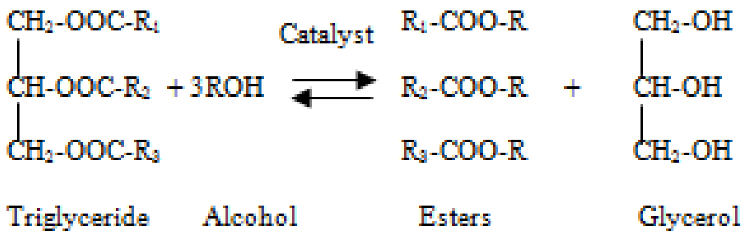


Figure 2.3: General equation for transesterification of triglyceride

During transesterification, the long chain and branched fatty acid molecules are converted to mono-ester. The reaction consists of three consecutive reversible steps; firstly conversion of triglycerides to diglycerides, followed by diglycerides to mono-glycerides. The glycerides are converted into glycerol and one ester molecule in each step. The ester produced has the similar properties as the diesel. The reaction is as shown in *Figure 2.4*.

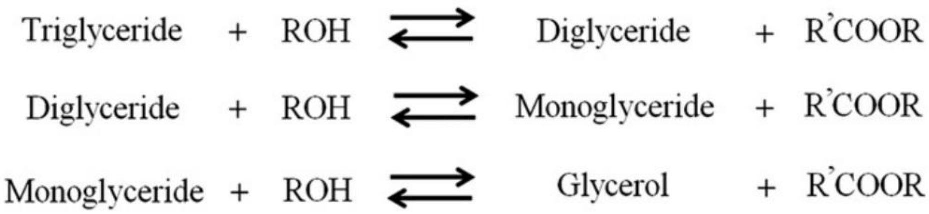


Figure 2.4: The three reversible and consecutive reactions in transesterification

Methanol and ethanol are the most common alcohols used in transesterification. However, methanol is more commonly used due to its physical and chemical advantages (polar and shortest chain alcohol) and its low cost (Sinha et al., 2008).

2.4 Biodiesel Production using Microwave Assisted Technique

There are several methods to convert vegetable oils and animal fats to biodiesel such as microemulsion, pyrolysis and transesterification. Among these methods, transesterification is the best and easiest step in producing a cleaner and environmental friendly fuel (Meher et al., 2006). Traditionally, transesterification process for biodiesel production is carried out by conventional heating technique which takes hours instead of minutes to achieve 99% of biodiesel yield (Yaakob et al., 2008). On the other hand, microwave assisted technique is an alternative heat source to carry out the transesterification process in shorter reaction time and simultaneously, improve the yields (Leadbeater and Stencel, 2006).

A fast and easy method for preparation of biodiesel from triolein under microwave irradiation was reported by Leadbeater and Stencel (2006). The highest yield of biodiesel (98%) was obtained using 5% KOH or NaOH, a 6:1 M ratio of methanol to oil, and a reaction temperature of 323 K for 1 min under 25 W microwave exit power on batch scales up to 3 kg of oil each time. Lertsathapornsuk et al. (2003) reported that the microwave irradiation reduced reaction time in 10 seconds to complete the transesterification process from used cooking oil. Also, Kumar et al. (2011) investigated the microwave assisted alkali-catalyzed transesterification of Pongamia pinnata seed oil. It was found that the biodiesel was succesfully produced by microwave assisted reaction within 5-10 minutes compared to conventional heating which required about 3 hours for the reaction.

Besides, the production of biodiesel in the presence of homogeneous catalysts (methyl *t*-butyl ether) uner microwave irradiation was carried out by J. Hernando et al. (2007). The transesterification was done using either commercial rapeseed or soybean oil. A small scale batch microwave reactor and a microwave flow system were used to perform the experiment. A very high yield of 97% was obtained in 1 minute for both system. Microwave assisted technique of oil from conventional feedstocks (soybeans and rice bran) and an alternative feedstock (Chinese tallow tree (CTT) seeds) for biodiesel production was carried out by Kanitkar (2009). The study concluded that microwave assisted transesterification required as little as 5 minutes to obtain 95% conversion of biodiesel as compared to conventional method.

Reefat et al. (2008) obtained the optimum parametric conditions for biodiesel production from used cooking oil using the conventional heating technique and it was compared with the microwave irradiation method. Under the same optimal reaction conditions, microwave irradiation was able to make the production of biodiesel very fast (2 minutes) compared with the conventional technique (60 minutes) and also increased the yield to 100%. Conversion of jatropha oil to biodiesel using microwave technique was conducted by El Sherbiny et al. (2010). The study showed that the application of radio frequency microwave energy offers a fast, easy route to produce biodiesel with the advanteges of enhancing the raction rate (2 minutes instead of 150 minutes) and improved the separation process.

Many researches had proven that microwave assisted technique enhance the production of biodiesel in terms of reaction time and yield. Thus, the current work is focuesd on exploratory of utilizing microwave technology with Ceiba Pentandra as the feedstock for biodiesel production.

CHAPTER 3

METHODOLOGY

3.1 Experimental Procedure / Approach

The flow chart in *Figure 3.1* shows the general experimental procedure.

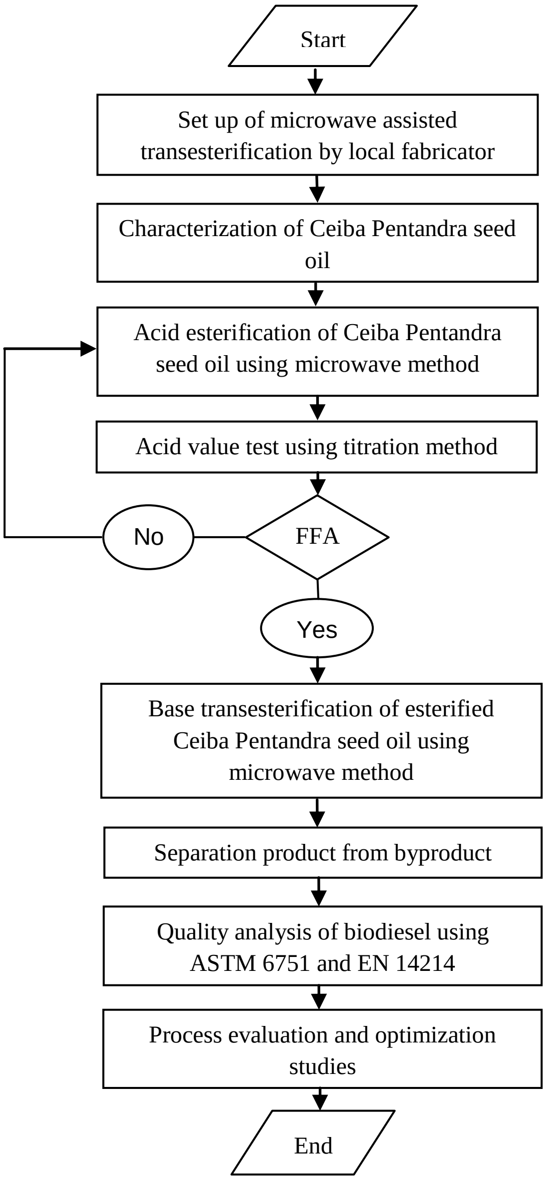


Figure 3.1: Flow chart of research activities

Process variables such as oil to methanol molar ratio, catalyst concentration, temperature and microwave irradiation time will be optimized for obtaining good quality biodiesel with optimum yield and less energy consumption. *Figure 3.2* below shows the block diagram for the proposed experimental process.

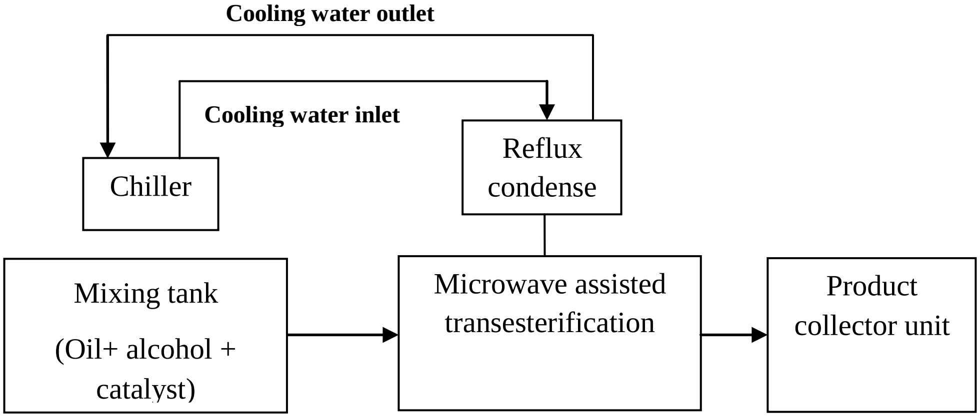


Figure 3.2: Block diagram of the proposed experimental process

Both esterification and transesterification will be conducted using microwave assisted system. Catalyst will be dissolved in solvent. The mixtures of catalyst, solvent and oil will be mixed prior to microwave unit as shown in *Figure 3.3*.

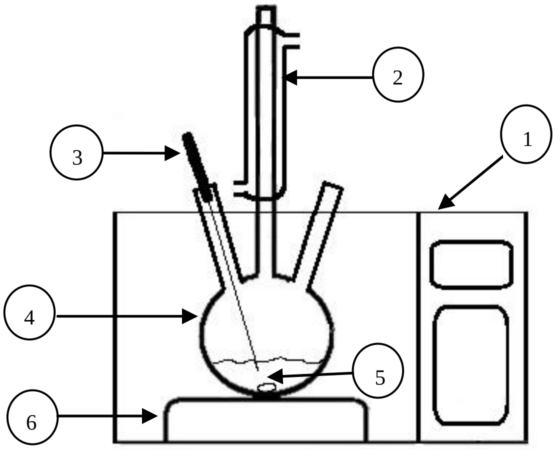


Figure 3.3: Schematic diagram of microwave system for batch experiments

1. Microwave oven, 2. Condenser, 3. Thermocouple, 4. Three-neck round bottom flask, 5. Magnetic bar, 6. Base

3.1.1 Acidity Test

Acidity test is done to check the free fatty acid content (FFA) in the crude kapok seed oil as well as the esterified seed oil. The manual titration is an adaptation of AOCS Method Cd 3d-63.

Reagents:

Titrant : KOH (85% Assay); 0.66g KOH/500mL Isopropanol = 0.02M

Solvent : Isopropanol:Toluene ratio (1:1)

Indicator : 1.0 g Phenolphtalein/100mL Isopropanol = 1%

Procedure:

1. KOH titrant is filled into a burette and the initial volume is noted.
2. 25mL of solvent is poured into a conical flask with 2 – 3 drops of indicator.
3. Titrant is added into the solvent drop-wise with regular stirring until faint pink colour remains.
4. The amount of titrant used is recorded.
5. 2 g of sample (exact weight is noted) is added into the flask and mixed well.
6. KOH titrant is again added drop-wise with regular stirring until faint pink colour remains.
7. The volume of titrant used is recorded.

The equation below is used to determine the acid value of kapok seed oil:

$$Acid\ value = \frac{(A - B) \times M \times MW_{KOH}}{W}$$

Where, A = Volume of titrant used for sample

B = Volume of titrant used for blank

M = Molarity of titrant

MW = Molecular weight for KOH

W = Exact weight of sample

3.1.2 Esterification

Esterification which uses acid as catalyst is aimed to reduce the free fatty acid (FFA) content in the oil to less than 1%. It is vital to prevent the occurrence of saponification in transesterification later on. Hence, separation of biodiesel from by-products will be easier and it also increases the yield of biodiesel. The operating conditions of acid esterification are at 65 ℃, 6:1 molar ratio of methanol to oil, 1.5 wt% catalyst and 5 minutes reaction time.

Procedure:

1. 250g of kapok seed oil is weighed.
2. Measuring cylinder is used to measure methanol of 6:1 molar ratio to oil.
3. 1.5 wt% of sulphuric acid is added to mix with the sample and methanol.
4. The mixture is then poured into the three-neck round bottom flask and heated to 65 ℃ in the microwave system.
5. When the temperature reaches 65 ℃, the heating is continued for 5 minutes.
6. Upon completion of reaction, the content is poured into a separating funnel and left to settle into two layers for 24 hours.
7. The upper layer of by-product is removed.
8. Warm de-ionized water is used to wash the esterified oil for several times until the pH value of washing water close to 7.0.

3.1.3 Transesterification

Transesterification process converts long chain of fatty acid in the seed oil to mono-ester. The process reduces the viscosity of the oil and fats, so that it can be used as proper alternative for diesel fuel. The experiments are designed using the response surface methodology (RSM) to investigate the optimized conditions for production of Ceiba Pentandra biodiesel using microwave assisted technique. The main idea of RSM is to use a sequence of designed experiments to obtain an optimal response. It is able to explore the relationships between several explanatory variables and one or more response variables.

Table 3.1 shows the designed experiments which consist of 30 runs where the variables being studied are methanol-to-oil ratio, amount of catalyst, reaction temperature and reaction time.

Table 3.1: Designed experiments for transesterification

Run	Temperature (℃)	Time (min)	Catalyst amount (wt%)	Methanol:Oil molar ratio
1	40.00	2.00	1.00	6.00
2	70.00	2.00	1.00	6.00
3	40.00	5.00	1.00	6.00
4	70.00	5.00	1.00	6.00
5	40.00	2.00	3.00	6.00
6	70.00	2.00	3.00	6.00
7	40.00	5.00	3.00	6.00
8	70.00	5.00	3.00	6.00
9	40.00	2.00	1.00	14.00
10	70.00	2.00	1.00	14.00
11	40.00	5.00	1.00	14.00
12	70.00	5.00	1.00	14.00
13	40.00	2.00	3.00	14.00
14	70.00	2.00	3.00	14.00
15	40.00	5.00	3.00	14.00
16	70.00	5.00	3.00	14.00
17	25.00	3.50	2.00	10.00
18	85.00	3.50	2.00	10.00
19	55.00	0.50	2.00	10.00
20	55.00	6.50	2.00	10.00
21	55.00	3.50	0.00	10.00
22	55.00	3.50	4.00	10.00
23	55.00	3.50	2.00	2.00
24	55.00	3.50	2.00	18.00
25	55.00	3.50	2.00	10.00
26	55.00	3.50	2.00	10.00
27	55.00	3.50	2.00	10.00
28	55.00	3.50	2.00	10.00
29	55.00	3.50	2.00	10.00
30	55.00	3.50	2.00	10.00

Procedure:

1. 30g of kapok seed oil is weighed.
2. Measuring cylinder is used to measure the ratio of methanol required.
3. Potassium hydroxide (KOH) is added and mixed with the sample and methanol.
4. The mixture is poured into the three-neck round bottom flask and heated to the desired temperature in the microwave system.
5. The reaction is started once the desired temperature is reached.
6. Upon completion of reaction, the content is poured into a separating funnel and left to settle into two layers for 24 hours.
7. The lower layer of by product is removed.
8. Warm de-ionized water is used to wash the esterified oil for several times until the pH value of washing water close to 7.0.
9. Rotary evaporator is used to remove excess moisture and methanol from the biodiesel.
10. 20 wt% of sodium thiosulphate is used to remove moisture from the oil.
11. The sodium thiosulphate is filtered off from the oil after around 1 hour.

3.1.4 Characterization of Biodiesel

The biodiesel produced from each run of experiment will be analyzed for the percentage of fatty acid methyl ester (FAME) using the gas chromatography. Upon obtaining the response of each run, the results are used in RSM to obtain the optimized conditions for highest yield of biodiesel production.

The quality attributes and fuel properties of the biodiesel produced under the optimum conditions will be evaluated by determining its physical and chemical parameters. The properties to be tested include density, acid value, water content, flash point, cloud point, pour point, oxidative stability, kinematic viscosity, cetane number, higher heating value, and cold filter plugging point following recommended American Standards for Testing and Materials (ASTM 6571) and European Standard (EN14124).

3.2 Key Milestones

The key milestones in this research include:

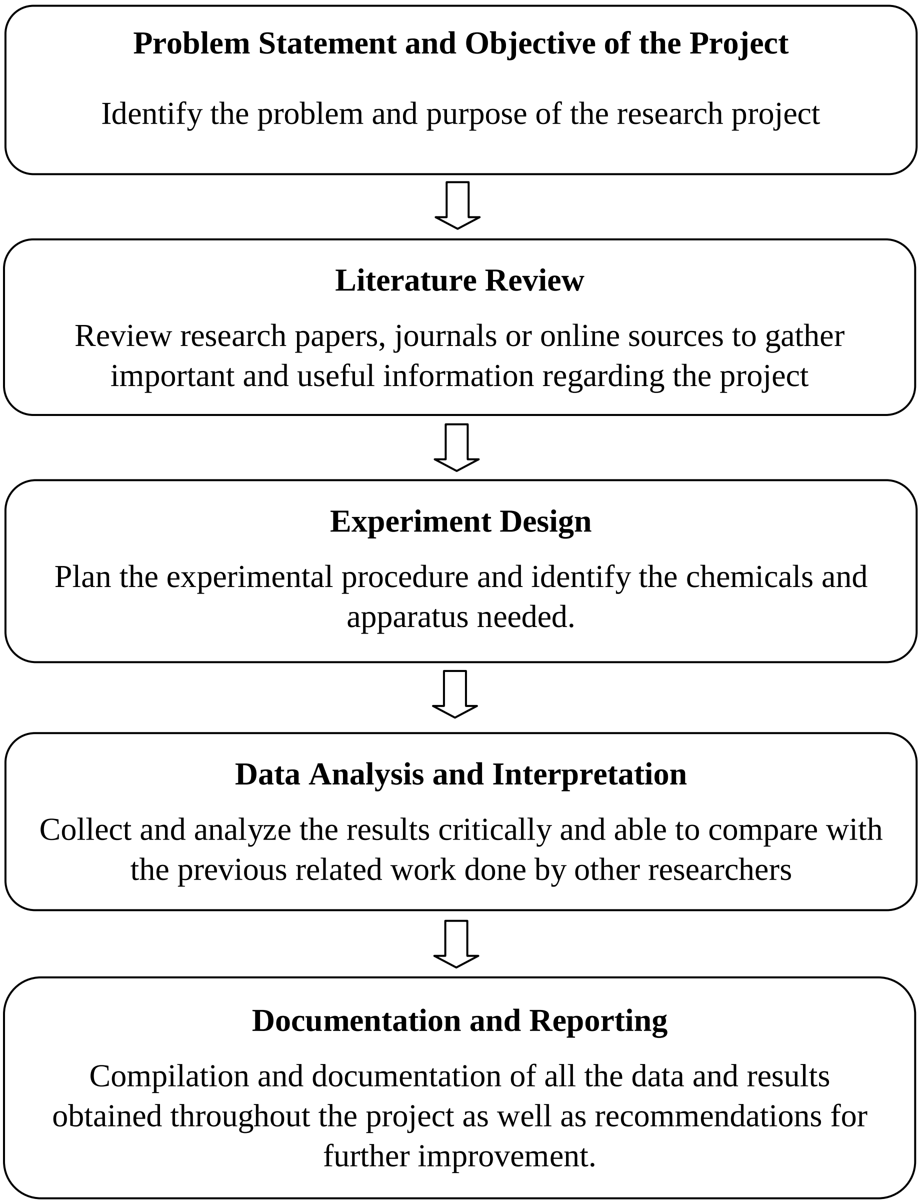


Figure 3.4: Key milestones

3.3 Gantt Chart

NO	DETAIL WEEK	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project Work Continues	■	■	■	■	■	■	■									
2	Submission of Progress Report									●							
3	Project Work Continues									■	■	■	■	■	■	■	
4	Pre-EDX												●				
5	Submission of Draft Report													●			
6	Submission of Dissertation (soft bound)														●		
7	Submission of Technical Paper														●		
8	Oral Presentation															●	
9	Submission of Project Dissertation (hard bound)																●

Table 3.2: Gantt Chart

3.4 Materials and Chemicals

The Kapok seed oil is purchased from East Jawa province, Indonesia. The acid value of the crude oil is tested prior to esterification process. Solvent used in the experiment is analytical grade of methanol. 95% concentration of sulphuric acid (H_2SO_4) is used in esterification process and potassium hydroxide (KOH) is used to produce potassium methoxide for transesterification process. Nonadecanoic acid methyl ester is used as standard solution for Gas Chromatography analysis. All chemicals are purchased from Merck except the H_2SO_4 which is purchased from Sigma Aldrich.

3.5 Equipment and Instrument

The apparatus that are used for the project can be divided into two categories which are the reaction equipments and the analytical instruments.

The modified microwave system is used for both esterification and transesterification processes. The components of the microwave synthesis reactor are shown in *Figure 3.5*.

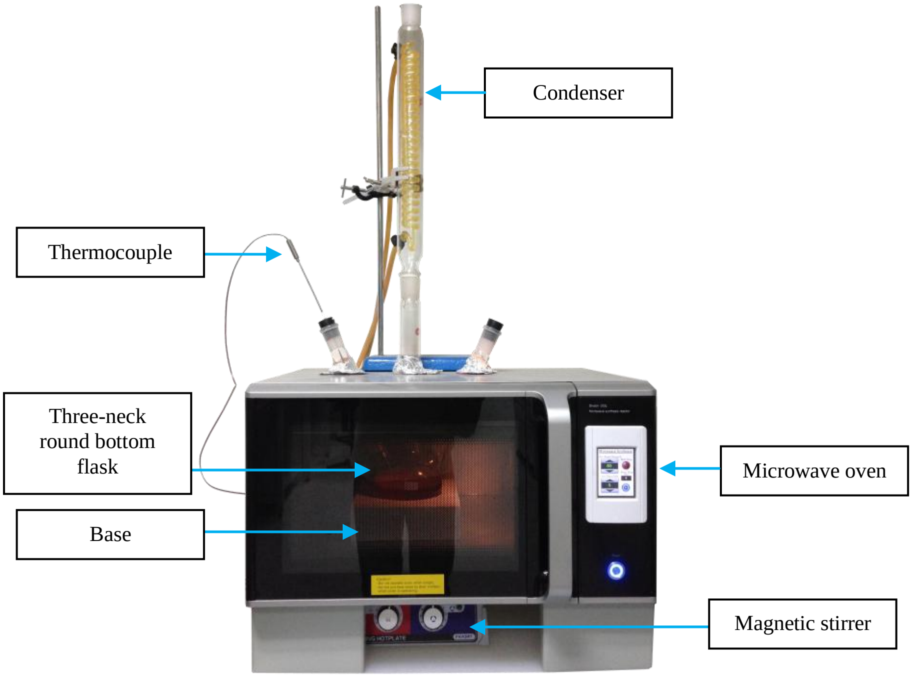


Figure 3.5: Microwave synthesis reactor

After transesterification process, the Ceiba Pentandra biodiesel is washed with warm deionized water. The water content is then removed with rotary evaporator as shown in *Figure 3.6*. The rotary evaporator is operated at 80 ℃, 300 mbar and rotated at 45 rpm for 15 minutes. Under pressure lower than atmospheric pressure, water can be evaporated at a temperature lower than its boiling point. The evaporated water is condensed and collected in a round bottom flask.

Figure 3.6: Rotary evaporator

After the biodiesel is produced, all the samples are tested in Gas Chromatography as shown in *Figure 3.7*.

Figure 3.7: Gas chromatography

The Gas Chromatography (GC) used is model 7890A from Agilent-Technologies. The GC system is equipped with a variable split flow injector, a temperature programmable oven, a flame ionization detector and capillary column coated with polyethylene glycol (30 x 0.25 mm; film thickness 0.25 µm). Helium is used as the carrier gas at 1.2 ml min^{-1}. Column temperature is adjusted from 60 ℃ hold for 2 minutes, programmed at 10 ℃ min^{-1} up to 200 ℃, programmed at 5 ℃ min^{-1} up to 240 ℃, and final temperature is hold for 7 minutes. The injector temperature and detector temperature are set at 250 ℃. Volume of sample injected is 1 µL.

Other analytical instruments used include those for biodiesel characterization purpose. The instruments are listed as shown in *Table 3.3* below.

Table 3.3: Analytical instruments for characterization of biodiesel

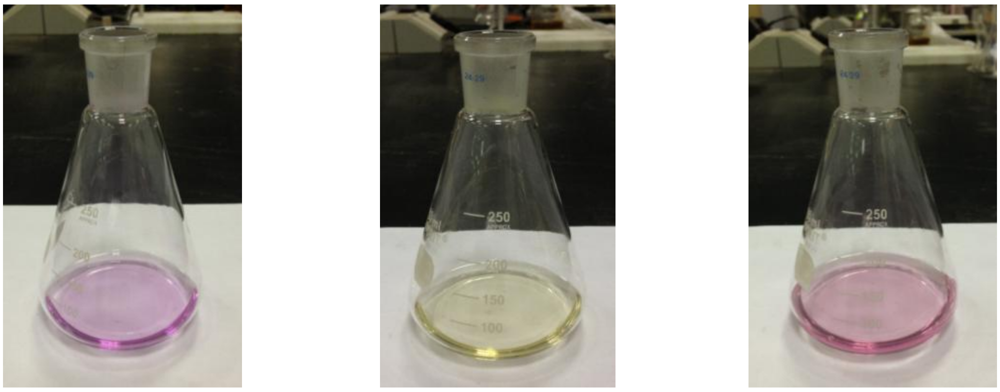
Properties	Instruments
Cloud point and pour point	ISL Cloud and Pour Point Analyser (CPP) 5Gs
Cold filter plugging point	ISL Cold Filter Plugging Point Analyser (CFPP) 5Gs
Flash point	Flash Point Tester CLA 5
Water content	Metrohm 870 Karl Fischer Titrino Plus
Density	DMA 4500M Density Meter
Kinematic viscosity	Brookfiled DV-II + PRO Digital Viscometer
Oxidative stability	Metrohm 873 Rancimat
Cetane number	Shatox Octane Meter
Higher heating value	Bomb calorimeter

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Acidity Test of Crude Kapok Seed Oil

The acid value of the crude kapok seed oil is tested with titration method before proceeding with the pretreatment process. Three repetitions are performed and the average value of the free fatty acid value is obtained. *Figure 4.1* below shows the titration before pretreatment process.



Titration for blank

Oil sample is added

Titration for sample

Figure 4.1: Acidity test before pretreatment

Table 4.1: FFA value before esterification

No. of Titration	Acid Value (mg KOH/g oil)	Free Fatty Acid (FFA) (%)
1	11.21	5.60
2	11.17	5.58
3	11.18	5.59
	Average FFA	**5.59**

The average acid value of the raw kapok seed oil is determined to be 11.19 mg KOH/g oil which corresponds to 5.59% of free fatty acid (FFA) content in the oil. Higher FFA content (> 1%) can lead to soap formation during transesterification reaction. These soap and emulsion formations make separation of product and by-product of biodiesel become longer and harder (Serioet et al., 2008). Therefore, the FFA content of KSO has to be reduced below 1% using acid catalyzed esterification as the pretreatment of the raw material.

4.2 Acid Esterification

The acid esterification reaction of kapok seed oil is carried out at 65 ℃ for 5 minutes using 6 to 1 methanol to oil ratio and 1.5 wt% of sulphuric acid as catalyst. After the reaction, the oil is allowed to settle into separated layers of product and by-product in a separating funnel for 24 hours as shown in *Figure 4.2*.

Figure 4.2: Separation of product and by-product layers after esterification

The upper layer of by-product which is the glycerol and some unreacted methanol is removed. Three batches of pretreated oil are mixed and it is tested with titration method for the FFA value. Two tests are conducted and the average reading is taken as shown in *Table 4.2*.

Table 4.2: FFA value after esterification

No. of Titration	Acid Value (mg KOH/g oil)	Free Fatty Acid (FFA) (%)
1	1.15	0.57
2	1.17	0.58
	Average FFA	**0.575**

Figure 4.3: Esterified kapok seed oil

From the data in *Table 4.2*, it can be seen that acid catalyzed esterification has effectively reduced the FFA percentage to below 1% which is within the specified range suitable for an efficient transesterification. The pretreatment process has achieved up to 89.71% of FFA value reduction.

4.3 Transesterification

30 transesterification processes are conducted based on the RSM design experiment. Each run has different condition on either temperature, irradiation time, catalyst concentration or ratio of methanol to oil. Similar to the esterification process, the oil is left in the separating funnel for phase separation of both the product and by-product layers as shown in *Figure 4.4*.

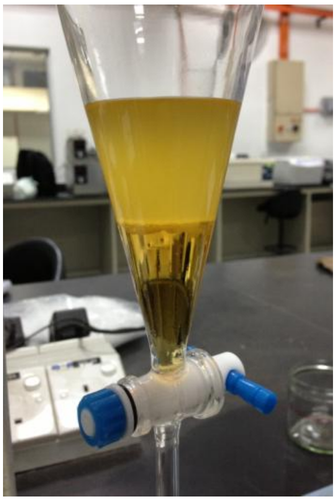
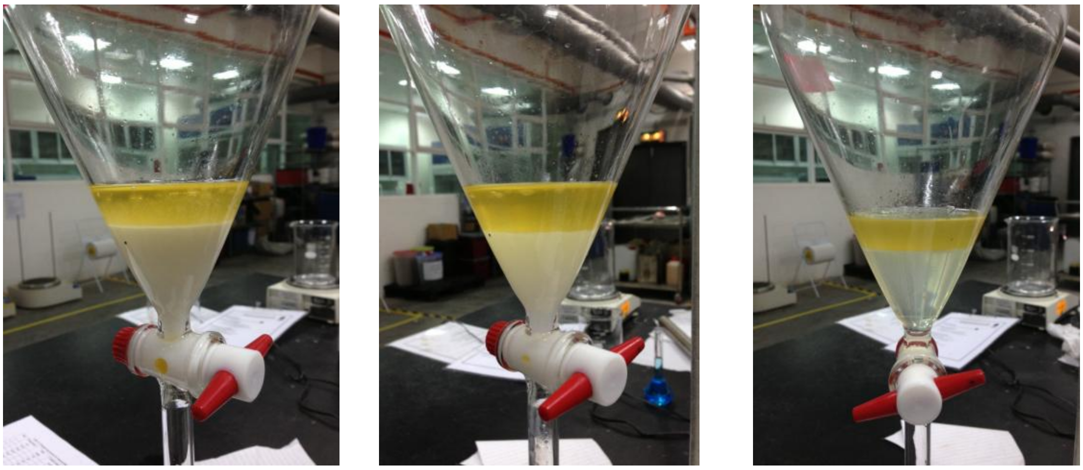


Figure 4.4: Seperation of product and by-product layers after transesterification

The by-product at the bottom layer is removed and the kapok oil methyl ester (KOME) produced is washed with warm distilled water as shown in *Figure 4.5* below.



Initial washing:
water turns very milky

Water gets less milky

Final washing:
water is not milky

Figure 4.5: Washing of biodiesel after transesterification

Initially, the warm distilled water poured into the separating funnel turns very milky. This is due to the saponification reaction where soap is formed due to the reaction between the water with the base catalyst remaining in the oil. Vigorous movement or shaking of the separating funnel is avoided to prevent further formation of soap which will cause the separation of product and by-product layers to be very difficult. The oil and milky water layer are allowed to separate and the water is removed. Again, water is poured into the separating funnel to wash the oil. The cycle repeats until the water does not turn milky which proves that the base catalyst remained in the oil has been entirely washed off. After washing, the water content in all the KOME samples are removed using rotary evaporator.

4.3.1 Acidity Test of KOME

The free fatty acid (FFA) percentage of all the biodiesel produced is also tested to study the effect of different parameters in reducing the acidity of biodiesel product. Two tests are performed for each sample and the average value of acid value is obtained. FFA percentage is obtained by dividing the acid value by 2. The results are shown in *Table 4.3*.

Table 4.3: Acidity test of kapok seed biodiesel

Sample	Acid Value (mg KOH/g oil)			FFA (%)
	1	2	Average	
1	0.24	0.22	0.230	0.115
2	0.20	0.17	0.185	0.093
3	0.18	0.22	0.200	0.100
4	0.17	0.19	0.180	0.090
5	0.28	0.25	0.265	0.133
6	0.22	0.20	0.210	0.105
7	0.28	0.25	0.265	0.133
8	0.22	0.19	0.205	0.103
9	0.17	0.14	0.155	0.078
10	0.19	0.16	0.175	0.088
11	0.19	0.17	0.180	0.090
12	0.17	0.15	0.160	0.080
13	0.28	0.31	0.295	0.148

14	0.28	0.33	0.305	0.153
15	0.38	0.33	0.355	0.178
16	0.33	0.36	0.345	0.173
17	0.22	0.28	0.250	0.125
18	0.22	0.17	0.195	0.098
19	0.28	0.33	0.305	0.153
20	0.38	0.33	0.355	0.178
21	0.83	0.84	0.835	0.418
22	0.28	0.27	0.275	0.138
23	0.28	0.33	0.305	0.153
24	0.17	0.11	0.140	0.070
25	0.28	0.22	0.250	0.125
26	0.33	0.28	0.305	0.153
27	0.39	0.33	0.360	0.180
28	0.28	0.34	0.310	0.155
29	0.22	0.22	0.220	0.110
30	0.28	0.28	0.280	0.140

From the data obtained in *Table 4.3*, it can be seen that the FFA content of all the samples are below 0.2% except sample 21 which has a FFA% of 0.418%. This might due to the absence of base catalyst for transesterification process in run 21 thus causing it to have a slightly higher acid value compared to other samples. However, all the samples have achieved very low FFA content. It is lower than the limit set by the European biodiesel standard, EN 14214 and American standard, ASTM 6751, where standard biodiesel should have acid value lower than 0.5 mg KOH/ g oil. Also, the results show that different parameters do not reduce the acidity of biodiesel in a specific trend, despite the FFA value of all samples are below 1%.

4.3.2 Fatty Acid Methyl Ester (FAME) yield

Each sample is then analyzed for the FAME yield using Gas Chromatography. The results are shown in *Table 4.4*.

Table 4.4: Results for FAME conversion

Sample	KOME yield (%)	Sample	KOME yield (%)
1	74.14	16	75.32
2	77.57	17	82.69
3	72.52	18	85.94
4	76.34	19	91.65
5	79.75	20	93.89
6	80.25	21	15.06
7	75.00	22	95.00
8	80.96	23	89.65
9	76.03	24	92.87
10	77.56	25	95.50
11	73.87	26	95.90
12	77.27	27	94.96
13	79.70	28	95.98
14	77.52	29	96.02
15	75.10	30	95.43

The FAME conversion percentage is calculated from the chromatograms obtained from Gas Chromatography as attached in Appendix A. The calculations are done with reference to the internal standard used, methyl nonadecanoate. The equation is as shown below:

$$C = \frac{(\Sigma A) - A_{IS}}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m} \times 100$$

Where, $\sum A$ = Total peak area

A_{IS} = Peak area of internal standard

C_{IS} = Concentration of internal standard (1mg/mL)

V_{IS} = Volume of internal standard solution (1mL)

m = Mass of sample

From the results, it can be seen that Sample 25 to Sample 30 have the highest yield of FAME. All the six runs are performed under similar conditions of parameters. Hence, it can be concluded, experimentally that the highest yield of KOME can be achieved at 55 ℃, 2 wt% catalyst, 10:1 methanol to oil ratio and 3.5 minutes of reaction time. The KOME yield obtained at these conditions is around 95.6% after taking the average value of all the 6 runs.

Yusup et al. (2010b) analyzed the same design of experiment for transesterification of rubber seed oil (RSO) and discovered that the optimum conditions for rubber seed oil methyl ester (RSOME) conversion (98%) were at temperature of 55 ℃, 2 wt% of catalyst, 8:1 methanol to oil ratio and 5 hours of reaction time. Another research was done by Norazahar et al., (2012) on KOME production from kapok seed oil using conventional heating. The highest yield of KOME (98%) was obtained at 55 ℃, 8:1 of methanol-to-oil molar ratio, 2 wt% of KOH and 3 hours reaction time.

The result of current project is comparable with the project done on KOME production using conventional heating. The significant achievement is the drastic reduction of reaction time from 3 hours to 3.5 minutes although the yield obtained using microwave system (95.6%) is slightly less than the 98% yield obtained using conventional heating.

4.4 Energy Consumption of Microwave Synthesis Reactor

The irradiation time for production of KOME using microwave synthesis reactor takes only 3.5 minutes. Whereas, conventional heating method requires 3 hours to achieve 98% of KOME yield (Norazahar et al., 2012). The energy consumption of both methods is calculated to compare the efficiency in biodiesel production.

The power output of the microwave synthesis reactor which is modified from domestic microwave oven is 1200 Watts. Also, the microwave is equipped with a mechanical stirrer which consumes energy as well. The maximum electricity consumption by the stirrer is 90 Watts.

Total energy consumption (microwave heating)

$$= 1290W \times 3.5\min \times \frac{1 hour}{60\min} \times \frac{1kW}{1000W}$$

= 0.075 kWh

As for conventional heating method, Favorit stirring hot plate model HS0707V2 which has 1007 Watts power consumption is used.

Total energy consumption (conventional heating) $= 1007W \times 3hours \times \frac{1kW}{1000W}$

= 3.021 kWh

Table 4.5: Comparison of energy consumption between microwave heating and conventional heating

	Microwave heating	**Conventional heating**
Energy dissipated (W)	1290	1007
Time of reaction (min)	3.5	180
Total energy required (kWh)	0.075	3.021

For 100 g of Kapok seed oil (KSO) to undergo transesterification, microwave synthesis reactor requires 0.075 kWh to yield 95.6% of KOME whereas conventional heating requires 3.021 kWh to yield 98% of KOME. The comparable amount of KOME yield has a very big difference in energy consumption. Conventional heating consumes almost 40 times more of the energy required by microwave system to produce comparable yield of KOME.

4.5 Fatty Acid Profile of KOME

Table 4.6: Fatty acid methyl esters profile of KOME

KOME	**Percentage (wt%)**	
	Current work	**Norazahar et al., 2012**
Caproic acid (C6)	-	9.42
Palmitic acid (C16)	20.44	23.17
Stearic acid (C18)	12.47	4.73
Oleic acid (C18:1)	19.32	22.88
Linoleic acid (C18:2)	39.14	30.00
Linolenic acid (C18:3)	1.61	-
Arachidic acid (C20)	-	1.18

Base on the FAME profile in *Table 4.6*, it shows that KSO contains high unsaturated fatty acid which is linoleic acid. The oil properties which contain unsaturated fatty acid can be an excellent oil for engine performance during cold weather after conversion to biodiesel. In this study, only the major groups of methyl esters are considered into the yield calculation. Hilditch et. al. also reported that KSO consists mainly of oleic acid and linoleic acid forming together about 70 wt% of the total fatty acids.

4.6 Characterization of KOME

The fuel properties of KOME produced under the optimized condition are analyzed and compared with the American biodiesel standard, ASTM 6751 and the European standard, EN 14214.

Table 4.7: Properties of KOME in comparison to ASTM 6751 and EN 14214 standards

Properties	KOME	ASTM 6751	EN 14214
Density @ 25 ℃ (kgm^{-3})	874	-	-
Cloud point (℃)	2	5	[a]
Pour point (℃)	0	-15	[b]
Cold filter plugging point (℃)	3	-	[b]
Flash point (℃)	149	93 min	120 min
Kinematic viscosity @ 40 ℃ (mm^{2}s^{-1})	1.9	1.9 – 6.0	3.5 – 5.0
Oxidative stability (hr)	3.69	3 min	6 min
Moisture content (%)	0.03	< 0.05	< 0.03
Acid value (mg KOH g^{-1})	0.3	< 0.5	< 0.5
Cetane number	57.08	47 min	51 min

[a] Not specified

[b] Not specified. EN 14214 use time and location dependant values for cold filter plugging point instead

It is important to determine the quality of the biodiesel produced by comparing with the international standards for biodiesel, ASTM 6751 and EN 14214.

The flow of biodiesel consists of cloud point (CP), pour point (PP) and cold filter plugging point (CFPP). Cloud point is the temperature at which dissolved solids are no longer completely soluble, where wax in biodiesel starts to form cloudy appearance. Pour point is the lowest temperature at which biodiesel becomes semi solid and losses its flow characteristics. Cold filter plugging point gives a estimate for the lowest temperature that a fuel will give trouble free flow in certain fuel systems. This is important as in cold temperature countries, a high cold filter plugging point will clog up vehicle engines easily. These properties are temperature dependent and EN 14214 does not specify the limit. Generally, these flow properties are associated with the weather condition and the place where the test is conducted. The cloud point and pour point of KOME produced are 2 ℃ and 0 ℃ respectively. The pour point meets the standard specify by ASTM 6751, which is 5 ℃. However, the cloud point does not meet the standard and this might due to the presence of water in the biodiesel, thus causing the value obtained for cloud point to be 0 ℃, which is the freezing temperature of water.

Besides, the flash point of KOME, 149 ℃ meets both the standard. Biodiesel requires high flash point, as biodiesel having low flash point will cause premature ignition, hence producing sound in the engine. Biodiesel with low flash point is also less efficient to be used as fuel.

Kinematic viscosity is a measurement of fluid’s thickness or resistance to flow over time. In determining the biodiesel quality, kinematic viscosity is one of the important elements which need to be monitored. Both low and high viscosities affect on an engine performance. Higher viscosity of the oil causes operational problems such as formation of large droplets on injection which results in poor combustion, engine deposits as well as increase in exhaust smoke or gas emission. The kinematic viscosity measure meets both the standards.

The oxidative stability of biodiesel is analyzed by biodiesel rancimat method EN 14112. The oxidation of biodiesel contributes to major problem during storage. The presence of unsaturated bonds influences this property largely. Oxidation occurs due to presence of air, eminent temperature and presence of metals content which promote the oxidation of the unsaturated bonds. The induction time of KOME is determined to be 3.69 hours which only satisfy the ASTM 6751 standard.

Low moisture content in biodiesel is very important to make it an efficient fuel. This is because water lowers the heat of combustion of the fuel, where it leads to increase in smoke emission, and eventually less power to the engine. The moisture content of KOME is 0.03 % which is lower than both the specification of ASTM 6751 and EN 14214, <0.05% and <0.03% respectively.

The acid value of biodiesel has been successfully reduced to 0.3 mg KOH g^{-1} oil. Both esterification and transesterification are effective in reducing the acid value of biodiesel product. *Figure 4.6* below shows the acid value profile throughout the experiment.

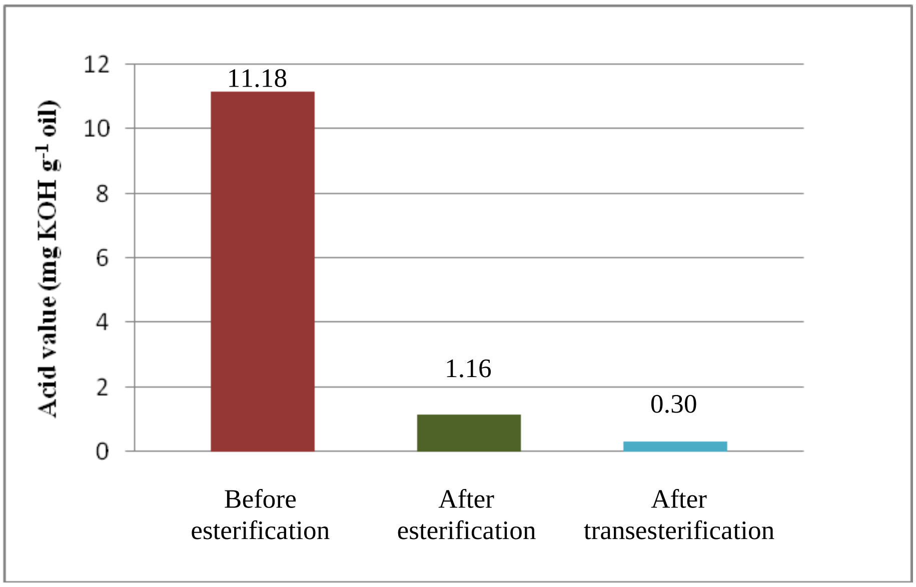


Figure 4.6: Acid value profile

The cetane number expresses the ignition property of biodiesel similar to cetane number in fossil based diesel. It is related to the ignition delay time a fuel experiences upon injection into the combustion chamber. The cetane number decreases with decreasing chain length and increasing degree of un-saturation and branching. Higher cetane number gives shorter ignition delay and it gives high tendency of the fuel to ignite. In this study, the cetane number of KOME is 57.08. The minimum cetane number specified by ASTM 6751 and EN 14214 are 47 and 51 respectively.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Biodiesel has been successfully produced from Ceiba Pentandra using microwave assisted technique. 30 runs are performed base on the Design of Experiment varying different parameters namely the temperature, irradiation time, methanol to oil ratio and the concentration of catalyst.

Esterification, the pre-treatment process followed by transesterification has successfully reduced the free fatty acid (FFA) of the Kapok oil methyl ester (KOME) to 0.15 %. The optimum operating conditions for transesterification process to synthesize biodiesel are at 55 ℃, 2 wt% KOH catalyst, 10:1 methanol to oil molar ratio and 3.5 minutes of irradiation time. The highest KOME yield at this condition is 95.6 %.

It is also proven that biodiesel synthesis using microwave assisted technique is able to reduce the reaction time drastically compared to conventional heating. Reducing the reaction time can eventually reduce the energy consumption. Conventional heating consumes almost 40 times of energy required by microwave system to produce comparable yield of KOME.

Besides that, quality of Ceiba Pentandra biodiesel is ensured by performing various characterization analyses. It has been identified as the potential sources of biodiesel, since its properties are comparable and satisfy the standard for biodiesel (ASTM 6751 and EN 14214). Further research and development are required to improve its fuel properties.

5.2 Recommendation

The esterification and transesterification of kapok seed oil should be scaled down to less than 30g of raw material. This is one of the limitations of laboratory scale equipment where the 3-neck round bottom flask has only a maximum allowable volume of 500mL. Scaling down the experiment work is to avoid the excessive evaporation of methanol solvent into the atmosphere and causing inaccuracy in results.

Also, the acidity of the kapok seed oil or biodiesel should be tested at regular basis, once in a week for example. The reason is that the acidity of the oil will tend to increase as time goes by. Thus, more accurate results and analysis can be achieved by monitoring the oil more frequently.

It is also recommended that more properties analysis should be done on the Ceiba Pentandra biodiesel and compare to the international standard for biodiesel. This will provide further solid verification on the suitability of kapok seed oil to be used as biodiesel feedstock.

5.3 Future Work

Further detailed studies should be done on the four parameters – temperature, catalyst concentration, solvent to oil molar ratio and reaction time. The relationship between each parameter can be studied. The kinetic data of all the parameters in affecting the FAME yield is also very crucial. This is because kinetic study is an important tool to study the reaction rate of both esterification and transesterifiaction reactions. This kinetics will be very suitable and useful for reactor design or commercializing it into larger scale.

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APPENDIX A

YIELD BY MASS OF TRANSESTERIFICATION PROCESS

Sample	Weight of oil (g)	Weight of biodiesel product (g)	Weight of by-product (g)	Yield (%)
1	30.35	28.80	4.34	94.89
2	30.46	27.23	5.20	89.40
3	30.26	27.79	4.47	91.84
4	30.75	27.05	5.45	87.97
5	30.21	25.44	5.36	84.21
6	30.44	19.38	13.51	63.67
7	30.01	25.49	5.56	84.94
8	30.05	17.16	15.58	57.10
9	30.04	27.60	9.43	91.88
10	31.10	29.62	7.88	95.24
11	30.28	28.53	11.67	94.22
12	30.04	27.55	6.37	91.71
13	30.08	23.80	11.10	79.12
14	30.01	20.36	16.48	67.84
15	30.17	23.12	13.72	76.63
16	30.12	16.57	17.96	55.01
17	31.15	25.46	9.99	81.73
18	30.22	23.81	10.12	78.79
19	30.01	26.11	8.75	87.00
20	30.26	25.20	8.90	83.28
21	30.10	30.02	4.64	99.73
22	30.58	22.94	9.45	75.02
23	30.09	16.69	12.79	55.47
24	31.07	25.18	16.25	81.04
25	30.34	25.61	8.88	84.41
26	30.02	22.34	9.51	74.42
27	30.28	22.00	8.26	72.66
28	30.10	22.76	10.98	75.62
29	30.34	25.32	9.57	83.45
30	30.11	24.35	8.83	80.87

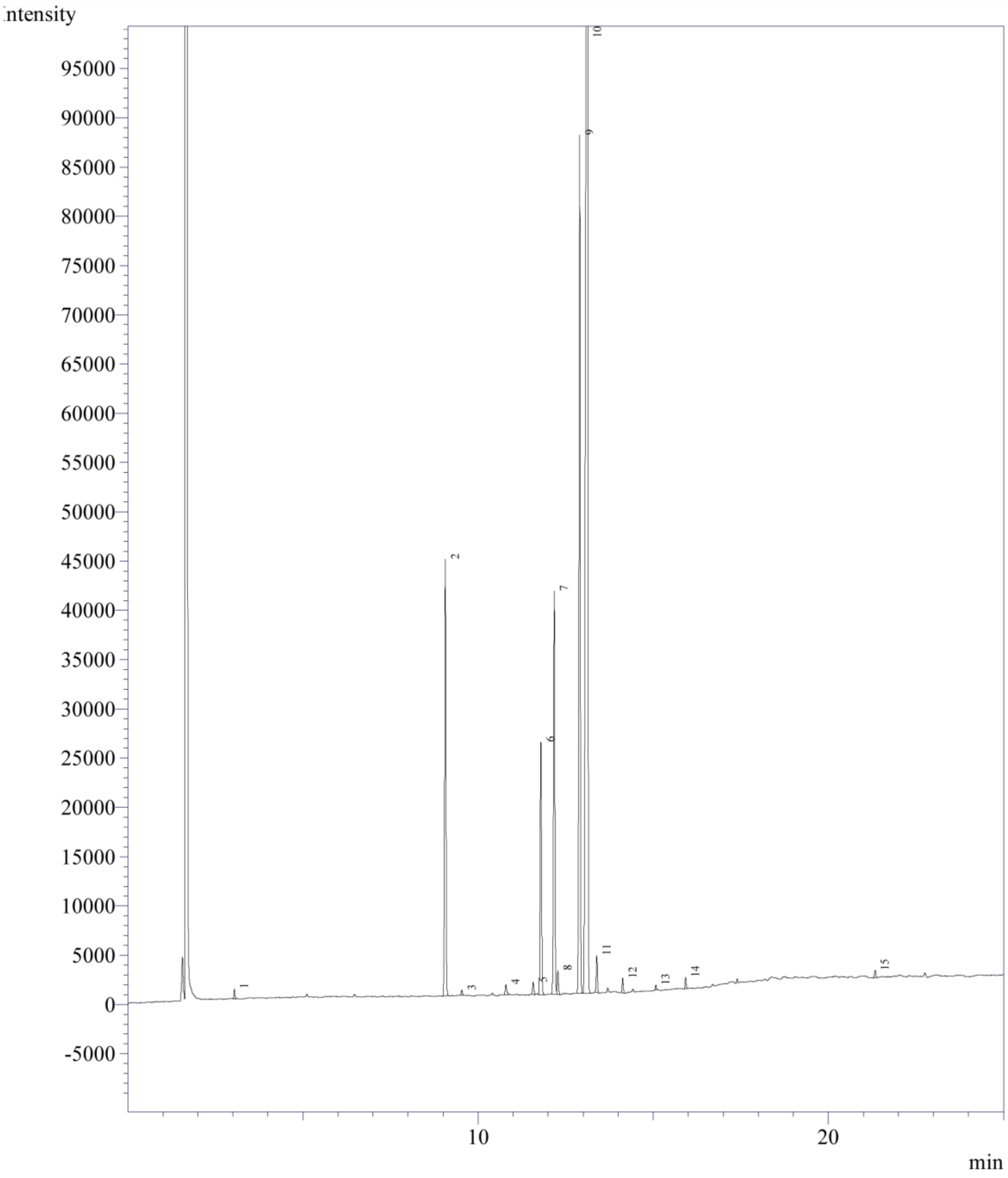
APPENDIX B

CHROMATOGRAM OF GAS CHROMATOGRAPHY

Sample Information

Analysis Date & Time : 2/27/2003 1:56:49 AM

Sample Name : sample 18
Data Name : C:\GCsolution\Data\FYP\2013\20130718 tan 04.gcd
Method Name : C:\GCsolution\Data\mfab\bpx70\FAME-bpx70 2011.gcm



Peak#	Ret.Time	Area	Area%	Height	Height%	Conc.	Unit	Cmpd Name
1	3.042	1965	0.1613	1033	0.2650	0.0000		M Caprate
2	9.064	116389	9.5557	44094	11.3158	0.0000		M Palmitate
3	9.536	1373	0.1128	541	0.1389	0.0000		M Palmitoleate
4	10.794	3097	0.2543	1035	0.2656	0.0000		
5	11.573	3278	0.2691	1255	0.3221	0.0000		
6	11.792	71471	5.8679	25613	6.5731	0.0000		M Stearate
7	12.175	111092	9.1208	40805	10.4717	0.0000		M Oleate
8	12.278	6208	0.5097	2430	0.6236	0.0000		
9	12.902	228432	18.7547	86960	22.3165	0.0000		M Linoleate
10	13.120	656846	53.9281	178171	45.7237	0.0000		
11	13.390	8828	0.7248	3780	0.9700	0.0000		M Linolenate
12	14.130	3403	0.2794	1501	0.3852	0.0000		M Arachidate
13	15.077	1107	0.0909	518	0.1330	0.0000		
14	15.923	2521	0.2070	1191	0.3055	0.0000		M Behenate
15	21.338	1993	0.1636	741	0.1903	0.0000		
Total		1218003		389668		0.0000		