SYNTHESIS OF BIODIESEL
FROM PENAGA OIL

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UNIVERSITI TEKNOLOGI PETRONAS
JULY 2009

Synthesis of Biodiesel from Panaga Oil

by

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Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

JULY 2009

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CERTIFICATION OF APPROVAL

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Chemical Engineering Programme
Universiti Teknologi PETRONAS
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BACHELOR OF ENGINEERING (Hons)
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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2009

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.

(MUHAMMAD IBRAHIM SA'DAN)

ACKNOWLEDGEMENTS

Firstly, I would like to thank to God because by His willing, I have completed my Final Year Project (FYP).

I would personally give my big appreciation to my supervisor, AP Dr Suzana Yusup for her countless effort and guidance that motivated me to work on this project. There are no equivalent words that can describe her determination and contribution to me.

I would like to thank Mr. Surya Abadi Ginting and Mr. M Hafeez for their help, effort and guidance throughout my project.

I also would like to thank all UTP technologists especially, Mr. Jailani, Mr. Fazli and Ms Azimah for helping and providing me with the equipments and chemical needed to complete the project.

Last but not least, I would like to express my gratitude to other lecturers, all my fellow colleagues and family for their support, assistance and ideas in the completion of this project.

ABSTRACT

Biodiesel refers to a non-petroleum-based diesel fuel consisting of long chain alkyl esters, made by transesterification of vegetable oil or animal fats, which can be used in unmodified diesel-engine vehicles. Basically, biodiesel is originated because of depleting in earth crude oil sources and environmental concern. So, biodiesel can be considered as renewable replacement for petroleum-based diesel engine fuel. In the study, the main objective is to synthesis biodiesel where the biodiesel will be extracted from Penaga oil through in-situ method; reaction mixture of penaga oil and alcohol using acid-catalyst to produce biodiesel. Some parameters such as reaction temperature, catalyst concentration and particle sizes are manipulated to observe the effects and get the desired product with optimize condition. Literature review section discussed about Penaga plant background, biodiesel production through conventional method and in-situ method of transesterification, effects of each parameter; reaction temperature, catalyst concentration and particle size respectively. Design of experiment being used is Taguchi Method; L9 Array as it is provides uniformly distributed coverage of the test domain, concise test set with fewer test cases is created, all pair-wise combinations of test set created, simpler to generate and less error prone than test sets created manually and reduces testing cycle time. Basically, the project jobs scope will be carried out are: 1) Penaga Seed Preparation; 2) Oil extraction; 3) Acid Value; 4) In-situ Transesterification and 5) TLC Qualitative Analysis. As conclusion, it is believed that this method is feasible and Penaga could be commercialized as biodiesel feedstock considering the optimum conditions for the variables and parameters used.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 Background Study

The major part of all energy consumed worldwide comes from fossil sources (petroleum, coal and natural gas). However, these sources are limited and depleted by the near future. Furthermore, the fuel crisis of the 1970s and early 1980s focused attention on the desirability to develop alternative fuels and decrease the dependency on fossil fuel reserves. Alternative and renewable fuels have some characteristic that give them environmental advantage over petroleum fuels. Hence, research for alternative sources of renewable energy such as biodiesel is essential and significance.

Biodiesel is the most common biofuel in Europe. It is produced from oils or fats using transesterification and is a liquid similar in composition to fossil/mineral diesel. Its chemical name is fatty acid methyl (or ethyl) ester (FAME). Oils are mixed with sodium hydroxide and methanol (or ethanol) and the chemical reaction produces biodiesel (FAME) and glycerol. One part glycerol is produced for every 10 parts biodiesel. Feedstocks for biodiesel include animal fats, vegetable oils, soy, rapeseed, jatropha, mahua, mustard, flax, sunflower, palm oil, hemp, field pennycress, and algae.

Biodiesel can be used in any diesel engine when mixed with mineral diesel. Pure biodiesel (B100) is by far the lowest emission diesel fuel. Although liquefied petroleum gas and hydrogen have cleaner combustion, they are used to fuel much less efficient petrol engines and are not as widely available. The majority of vehicle manufacturers limit their recommendations to 15% biodiesel blended with mineral diesel. In some countries manufacturers cover their diesel engines under warranty for B100 use.

However, the current cost of producing biodiesel using transesterification method far exceeds the retail price of diesel in most countries due to the high cost of producing the vegetable oil feed stocks. Hence it is unlikely to become more cost-effective unless a new technique such as in-situ transesterification is developed. Overall, this project was conducted to determine the feasibility of in-situ transeterification process which combine both extraction and reaction steps into a single step.

Besides that, according to Datuk Seri Najib Tun Razak (News Straits Time, 2006), Malaysia is hoped to be able to position itself as the world's foremost biodiesel producer. The Ninth Malaysia Plan which was launched on 1st April 2006 has been identified agriculture as a key anchor activity to boost nation's economy and also declared a keen intent to identify and promote new growth, in this case Penaga plant or seed. It also emphasized on the need to focus on research and development. Obviously, in-situ transesterification and Penaga plant appeared to position neatly in all these ambitions.

1.2 Problem Statement

Due to the depleting sources of petroleum that and environmental concerns, a search for sustainable alternative fuel has gained significant attention. The need for energy is increasing from day to day due to rapid development of the industrialization and population. Over the Eight Malaysia Plan (8MP) period (2001-2005), the energy consumption was projected to grow at an average of 7.8% annually and more than double over a 10-years period. Since energy demand is increasing, many ways are being researched and developed to fulfill this need. Basically, the energy which is used nowadays is based on petroleum product, coal, hydro and nuclear. Petroleum diesel continues to be a worldwide major fuel.

However, petroleum-based fuels give a lot of drawbacks. The major disadvantage is lead to the atmospheric pollution since the combustion of the petroleum diesel emits greenhouse gases (GHG). As petroleum-based fuel is a non-renewable energy, the fossil fuel reserves are decreasing day by day. Due to the environmental effect created by petroleum-based fuel and decreasing of the fossil fuel reserves, it is urged to find and moved on to the alternative source of energy. Alternative fuels such as ethanol, natural gas, propane, methanol and biodiesel have been suggested. Of these, biodiesel holds the most promise, being both renewable and energy efficient.

Commercialization of biodiesel has occurred in numerous countries around the world with varying economic and legislative conditions. Biodiesel is an environmental-friendly fuel which can support the national energy demand and supply. Although biodiesel has been widely used in U.S and other European countries, its implementation in Malaysia is still under development. Currently, Malaysia uses palm oil as the raw material of biodiesel production. Problems occurred when the cost of biodiesel is primarily limited by the cost of feedstock. Quoting from EIA report "Biodiesel Performance, Costs and Use", "Unless palm oil prices decline dramatically, it does not appear that biodiesel can be produced in large quantities at a cost that is competitive with petroleum diesel." Therefore, it is crucial to assure that there is a possibility on utilizing other material as potential feedstock, in order to reduce the dependency towards the palm oil as source of biodiesel. So, in this project Penaga seed have been identified as one of possible source to produce biodiesel.

Furthermore, the current cost of transesterification method is far exceeds the selling price of diesel in most countries because of high cost in producing the vegetable oil feed stocks. Therefore, it is unlikely to develop into more cost-effective unless a new technique such as in-situ transesterification is developed. Basically, the aim of this project is to determine the feasibility of in-situ transeterification process which combine both extraction and reaction steps into a single step.

1.3 Objectives & Scope of Study

Objectives of the project are:

1) To synthesis biodiesel from Penaga seed through in-situ transesterification method.
2) To study the effect of reaction temperature, catalyst concentration and seed particle sizes.
3) To analyze qualitatively the produced biodiesel using Thin Layer Chromatograph (TLC).

In order to achieve the objectives, this project will be done according to the time frame and planned scheduled. Basically, the literature review had done provide deep insight into the biodiesel related issues. Besides, the experiments are planned based on the information obtained from the literature review and later the results obtained will be compared to the reference material. In the experiment, parameters such as reaction temperature, catalyst concentration and seed particle sizes have been manipulated to get the expected results. Qualitative results are determined and discussed to conclude the project.

1.4 Relevancy of The Project

As mentioned in the problem statement and objective, this project is mainly to produce or synthesis biodiesel from one of the plant seed called Penaga which has the same function as diesel (produced from crude oil); to generate engine power. Furthermore, biodiesel has better lubricating properties than today's lower viscosity diesel fuels and biodiesel addition reduces engine wear and thereby increasing the life of the fuel injection equipment that relies on the fuel for its lubrication, such as high pressure injection pumps and pump injectors; also called unit injectors and fuel injectors. So, by successfully producing the biodiesel from this plant seed, we can have another source to generate power and improved the usage of diesel which proved can give a lot of benefit to humanity.

1.5 Feasibility of The Project

It is expected the project can be conducted with no difficulty. Since Panaga seed can be acquired from UTP's woods itself, compared to other seed such as Jatropha or rubber seed which have to be ordered first and bought from outside of the UTP. Furthermore, the laboratory equipments and chemicals are supplied by the UTP laboratory itself which allowed the project to be conducted inside UTP; can give advantages to the time constraint in order to complete the project. But, due to the time constraint, the project only conducted to study the synthesis of biodiesel from Panaga oil through in-situ transesterification and effects of parameters such as reaction temperature, catalyst concentration used and particle sizes on the synthesis process.

CHAPTER 2
LITERATURE REVIEW

2.1 Raw Material

Figure 2.1.1: Penaga Tree

Figure 2.1.2: Penaga Seed

Penaga or Mesua Ferrea seed is the only raw material that will be used in the study which can be obtained in the UTP's woods itself. Mesua ferrea (Ceylon ironwood, Indian rose chestnut, Cobra's saffron or locally, Penaga Lilin, Na (Sinhalese) or Nahar/Nahor is a species in the family Clusiaceae. The plant is named after the heaviness of its timber and cultivated in tropical climates for its form, foliage, and fragrant flowers. It is native to tropical Sri Lanka but also cultivated in Assam, southern Nepal, Indochina, and the Malay Peninsula (Bhide, M.B., et al, 1977).

It is a tall tree reach up to 100ft tall, often buttressed at the base with a trunk up to 2 meters in diameter. It is comment in wet zone at Sri Lanka up to 1500 meters. It has simple, narrow, oblong, dark green leaves 7-15 cm long, with a whitish underside; the emerging young leaves are red to yellowish pink and drooping. The flowers are 4-7.5 cm diameter, with four white petals and a center of numerous yellow stamens (Duke J.A., et al, 1983).

Based on Bhide, M.B, et al, 1977, The National Ironwood Forest is a 96 ha (238 acre) forest in Sri Lanka where Mesua ferrea trees dominate the vegetation. It is said that during King Dappula IV's period (8th century AD) this forest was created and the remaining trees are the shoots of it. Hence it is considered the oldest man made forest in Sri Lanka. According to botanists this is the only ironwood forest in the dry zone with wet zone vegetation.

2.2 Transesterification

Based on Gerpen J. V., et al., 2005, biodiesel production is the act of producing the biofuel or biodiesel through either transesterification or alcoholysis. The process involves reacting vegetable oils or animal fats catalytically with a short-chain aliphatic alcohols (typically methanol or ethanol).

A reaction scheme for transesterification is as follows (Brunelle D. J., et al., 1991):

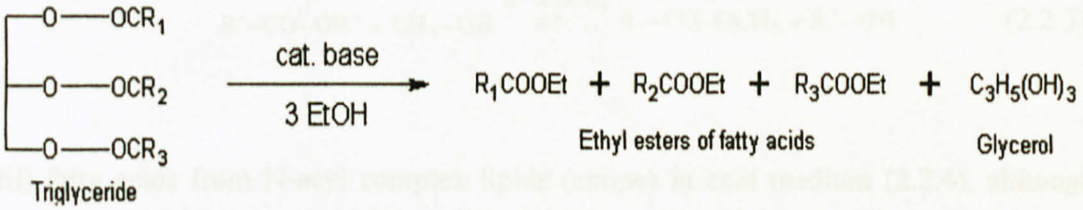


Figure 2.2.1: Reaction Scheme of Transesterification

R$_1$, R$_2$, and R$_3$ in this diagram represent long carbon chains (alkyl group) that are too lengthy to include in the diagram.

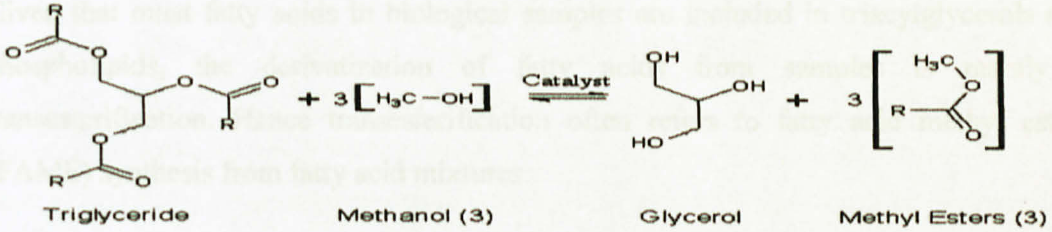


Figure 2.2.2: Skeletal Formula of Transesterification

According to Carrapiso A. I. and Garcia C., et al., 2000, before performing the fatty acid alkyl ester synthesis, certain knowledge about reaction principles is required so that accurate and reliable results will be obtained by using brief procedures for each sample type. Derivative reactions are usually catalyzed by acidic or basic media (Table 2.2), but they can also occur in the presence of diazomethane or other catalysts. The reactions usually occur with the following: (i) free fatty acids in media with acid pH (2.2.1) or with diazomethane (2.2.1). The reaction is called esterification or methylation.

$$R'-CO-CH + CH_3-OH \overset{H^+}{\rightleftharpoons} R'-CO-OCH_3 + HOH \qquad (2.2.1)$$

$$R'-CO-OH + CH_2N_2 \longrightarrow R'-CO-OCH_3 + N_2 \qquad (2.2.2)$$

(ii) fatty acids included in complex lipids by *O*-acyl bonds (ester) (e.g., triacylglycerols, phospholipids) in acid or basic media. The reaction is called transesterification or transmethylation (also known as alcoholysis or methanolysis) (2.2.3).

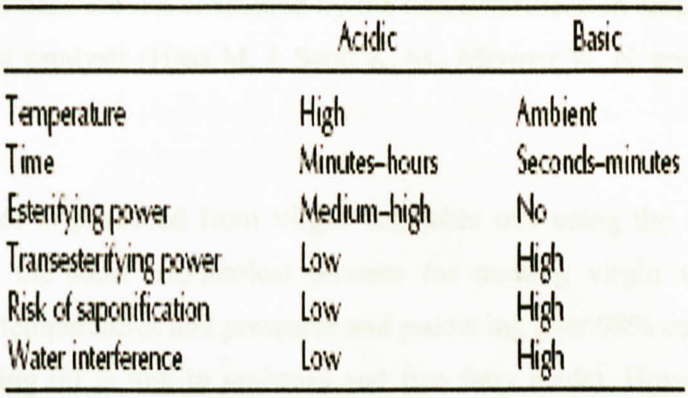
$$R'-CO-OR'' + CH_3-OH \overset{H^+ \text{or } OCH_3^-}{\rightleftharpoons} R'-CO-OCH_3 + R''-OH \qquad (2.2.3)$$

(iii) fatty acids from N-acyl complex lipids (amine) in acid medium (2.2.4), although special considerations have to be taken into account.

$$R'-CO-NHR'' + CH_3-OH \overset{H^+}{\rightleftharpoons} R'-CO-OCH_3 + R''-NH_2 \qquad (2.2.4)$$

Given that most fatty acids in biological samples are included in triacylglycerols and phospholipids, the derivatization of fatty acids from samples is mainly a transesterification. Hence transesterification often refers to fatty acid methyl esters (FAME) synthesis from fatty acid mixtures.

Table 2.2: Characteristic of Catalytic Medium in Transesterification Reactions (Carrapiso A. I. and Garcia C., et al., 2000)



	Acidic	Basic
Temperature	High	Ambient
Time	Minutes-hours	Seconds-minutes
Esterifying power	Medium-high	No
Transesterifying power	Low	High
Risk of saponification	Low	High
Water interference	Low	High

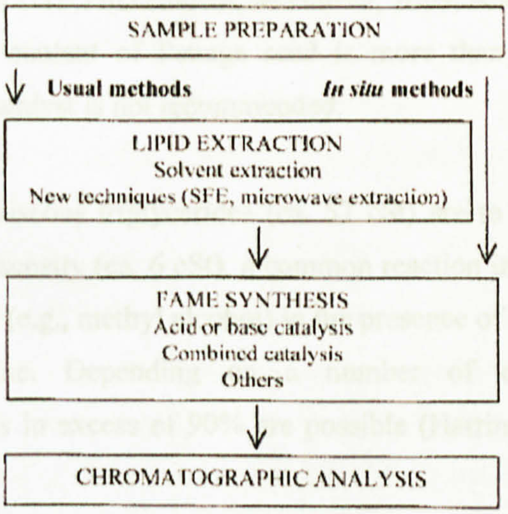


Figure 2.2.3: The Flow of Usual Transesterification and In-situ Transesterification (Carrapiso A. I. and Garcia C., et al., 2000)

Based on Ma F. and Hanna M. A., et al., 1999, animal and plant fats and oils are typically made of triglycerides which are esters of free fatty acids with the trihydric alcohol, glycerol. In the transesterification process, the alcohol is deprotonated with a base to make it a stronger nucleophile which commonly used is ethanol or methanol. As can be seen, the reaction has no other inputs than the triglyceride and the alcohol.

Normally, this reaction will precede either exceedingly slowly or not at all. Heat, as well as an acid or base are used to help the reaction proceed more quickly. It is important to note that the acid or base are not consumed by the transesterification reaction, thus they are not reactants but catalysts (Haas M. J, Scott K. M., Marmer W. N. and Foglia T. A., et al., 2004).

Almost all biodiesel is produced from virgin vegetable oils using the base-catalyzed technique as it is the most economical process for treating virgin vegetable oils, requiring only low temperatures and pressures and producing over 98% conversion yield (provided the starting oil is low in moisture and free fatty acids). However, biodiesel produced from other sources or by other methods may require acid catalysis which is much slower (Dubé M. A., et al., 2007) (Carrapiso A. I. and Garcia C., et al., 2000). In this study case, based on UTP PhD student, M Hafeez, 2009, acid catalyst has to be used because of acid value content of Penaga seed is more than 7.5% FFA, thus, acid pretreatment and base catalyst is not recommended.

In the case where the viscous triglycerides (ca. 57 cSt) are to be converted to simple esters of much lower viscosity (ca. 6 cSt), a common reaction involves treatment with a simple aliphatic alcohol (e.g., methyl alcohol) in the presence of a catalyst which may be either acid or alkaline. Depending on a number of experimental variables, transesterification yields in excess of 90% are possible (Harrington K. J. and D'Arcy-Evans C., et al., 1985).

2.3 In-situ Transesterification

The in-situ transesterification process is a process that integrates the oil extraction and transesterification raction into a single step process which can reduce the biodiesel production cost because oil extraction and refining processes are eliminated. Harrington K. J. and D'Arcy-Evans C., et al., 1985 is the first to explored the feasibility of in-situ method, using homogenized whole sunflower seed as substrate. They had conclude that the technique of transesterification in-situ of sunflower seed oil provides a yield of fatty acid esters qualitatively similar to, but quantitatively greater than, the yield obtained from treatment of the preextracted oil. The concept of transesterification in-situ was considered for the following reasons:

i) By subjecting the whole seed to the esterification process, the lipid content of the hull itself could contribute to the overall yield of esters from the seed.
ii) Lipid losses due to imperfect hull-kernel separation could be avoided.
iii) The esterified lipids, having viscosity and solubility parameters different from those of the intact triglycerides, could prove easier to recover from the solid residue and perhaps even allow the use of a solvent less dangerous and less expensive than hexane.
iv) Some improvement in meal carbohydrate digestibility could result from the action of either the acid or alkaline transesterification catalyst, although the overall nutritional value could be lessened due to the presence of hull material, which itself is of low nutritional value

Based on Harrington K. J. and D'Arcy-Evans C., et al., 1985, the technique of transesterification in-situ of sunflower seed oil provides a yield of fatty acid esters qualitatively similar to, but quantitatively greater than, the yield obtained from treatment of the preextracted oil. It appears likely that the increase in yield is predominantly due to the enhanced accessibility of the oil in the seed, which in turn is a consequence of the acidic reaction medium. It appears less likely that yield increases are due to the avoidance of workup losses or the possible greater ease of extraction of the esterified product over the intact triglycerides.

In addition, Siler-Marinkovic and Tomasevic, 1998, demonstrated that yield of methyl esters obtained from acid-catalyzed in-situ transesterification of sunflower seed is higher than conventional method of transesterification. For their experiments it is concluded that the reaction gave high yield when the reaction was carried out at room temperature 30°C with molar ratio alcohol to oil of 300:1 in 4 hours reaction time.

Georgogianni et al., 2007 reported a comparison of biodiesel yield which is produced by conventional method and in-situ transesterification methods, both using mechanical stirrer of 600 rpm and low frequency ultrasonicator at 24 kHz effect. A higher yield of biodiesel of methanolysis sunflower oil using conventional method was reported for both effects when the concentration of catalyst, sodium hydroxide 2.0 wt% was used. Meanwhile, for in-situ transesterification process, the yield was almost similar for both effects when methanolysis of sunflower oil was done. But, when ethanolysis was done, a higher yield was recorded on ultrasonication effect compared to mechanical stirrer effcect.

2.4 Effect of Catalyst Concentration

In Transesterification process, catalyst concentration in term of weight percentage (wt%) is one of the limiting conditions of the reaction to the amount of yield conversion. According to Xie W and Huang X, 2006, catalyst concentration also is the main parameter affecting the yield of alkyl ester throughout the reaction.

Ma F, Clements L and D Hanna M A, 1998, demonstrated that catalyst is an important factor in the transesterification of beef tallow. Alkali catalysts are the most commonly used, especially NaOH and NaMeO. Both were used in their experiment. **Figure 2.4** shows the results of the transesterification of beef tallow with NaOH and NaMeO. When the amount of the catalyst was at 0.1% of beef tallow, the yields were lowest for both catalysts. At 0.3%, however the yield reached the maximum value for NaOH, decreased slightly at 0.5%, and remained relatively constant at higher NaOH contents. The 0.3% level of NaOH was significantly better than the other levels. The transesterification with NaMeO followed a similar trend. The 0.5% level of NaMeO gave the maximum yield. However, the 0.5% level of NaMeO was not significantly different from 0.3% and 0.7%. Comparing the two catalysts, NaOH was significantly better than NaMeO which was different from previous reports (Freedman et. Al,1984 and Hartman, 1956).

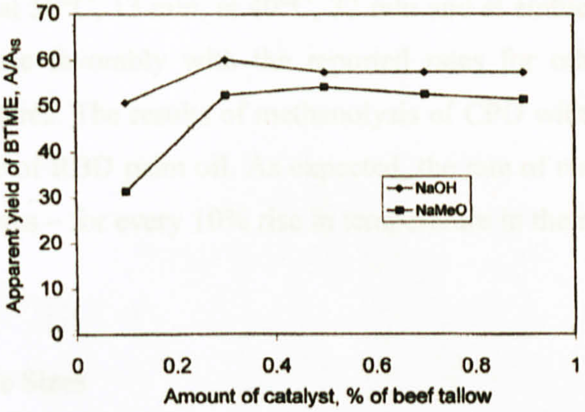


Figure 2.4 The Effect of Catalysts on Transesterification of Beef Tallow

2.5 Effect of Reaction Temperature

Hanh H D, Dong N T, Okitsu K, Maeda Y, and Nishimura R, 2007 demonstrated the test temperature dependence of the transesterification of triolein with methanol and a base catalyst (NaOH and KOH) was investigated using a molar ratio of methanol to triolein of 6:1, a catalyst concentration of 1% and a temperature range of 3–50 °C under a low-frequency ultrasonic irradiation condition (40 kHz). It was found that the methyl ester concentration at an irradiation time of 5 min increased with increasing temperature, where it tended to level off at temperatures higher than 20°C. Furthermore, apparent activation energy was estimated from the relationship between the rate and the reciprocal of temperature.

Choo Y M, 2004, stated that Feuge and Gros, 1949 concluded that transesterification can occur at a moderate temperature of 50°C in their work on the effect of temperature (at 70°C, 50°C and 30°C) on the ethanolysis of peanut oil (6:1 molar ratio) with 0.2% NaOH. Pryde, 1981 worked on methanolysis of soyabean oil (6:1 molar ratio, 1% sodium hydroxide) at 60°C, 45°C and 32°C. It was concluded that the transesterification proceeded satisfactorily at 32°C over several hours. Therefore, it was useful to investigate how the methanolysis of RBD palm oil (15.6:1 molar ratio, 0.75% sodium hydroxide) is influenced by temperature, *i.e.* at 60°C, 50°C, 40°C and 28°C (ambient). The results of these experiments showed that at 60°C the time for completion of the reaction was 7 min, at 50°C, 13 min, at 40°C, 32 min and at ambient temperature, 2 hr. These results compare favorably with the reported rates for other vegetable oils at comparable temperatures. The results of methanolysis of CPO with 3.9% FFA are quite consistent with those of RBD palm oil. As expected, the rate of reaction increased with increasing temperatures – for every 10% rise in temperature in the range 40°C-60°C, the rate almost doubled.

2.6 Effect of Particle Sizes

Based on Ted A. Russin, Yves Arcand and Joyce I. Boye, et al, 1997 a study was undertaken to determine if the yield and purity of soy protein isolates could be improved by changing the particle size of the starting raw material. Soy protein isolates were extracted from hexane defatted soy flour ground to three different average particle sizes (89.5 ± 1.1, 184.2 ± 1.6 and 223.4 ± 6.4 μm). By decreasing the average particle diameter of the starting raw material (soy flour) from 223.4 to 89.5 μm the total solids recovery increased from 23 to 32% ($P = 0.00008$), while the protein recovery increased from 40 to 52% ($P = 0.00004$). Final protein content (i.e., purity) of the soy protein isolates was not significantly impacted by average particle size. The results clearly demonstrated that protein recovery can be increased by >30% by decreasing the average particle size of the starting raw material (i.e., defatted soy flour), without having any detrimental impact on the purity of the final soy protein isolate.

Meanwhile, according to Goodrum John W. and Kilgo Mary B., et al, 1987, by reducing the particle size of the peanuts has also been shown to have a positive effect on the extraction. Smaller particles have a larger amount of surface area as well as an increased number of ruptured cells resulting in a high oil concentration at the particle surface. Little diffusion into the particles takes place; therefore, the amount of oil available for extraction is proportional to the surface area. By decreasing the particle size range from 3.35-4.75 mm to a range of 0.86-1.19 mm, the total oil recovery was increased from 36% to 82% by mass.

CHAPTER 3
METHODOLOGY

3.1 Research Methodology

Robertson's Ideal Research Model has been chosen as research methodology to complete this project. First, the problem is defined to determine the main objective of the project. Next, literatures are reviewed in order to get the ideas or theory related to the project. Then, possible hypothesis are formulated to initiate the project and based on the hypothesis, one researched design is chosen. After chose the design, data started to be collected and results are analyzed. In this project, all data and results being collected and analyzed based on laboratory results. Finally, the conclusion of the project is determined based on the laboratory analyzed results.

3.2 Design of Experiment (DOE)

To precede the experiment, **Taguchi Method L9 Array** has been chosen to complete the designated synthesis process of biodiesel (transestrification). Taguchi Method is a system of cost-driven quality engineering that emphasizes the effective application of engineering strategies rather than advanced statistical techniques. L9 Array is chosen because there are three parameters and three levels used throughout the experiment as shown in **Table 3.2.1.**

The method allows a project to rapidly and accurately acquire technical information to design and produce low-cost, highly reliable products and processes. Its most advanced applications allow engineers to develop flexible technology for the design and production of families of high quality products, greatly reducing research, development, and delivery time. Most typical applications of Taguchi Method thus far have centered around two main areas:

1) Improving an existing product
2) Improving a process for a specific product

Thus the advantages of Taguchi Method can be simplified as below:

1) Provides uniformly distributed coverage of the test domain.
2) Concise test set with fewer test cases is created.
3) All pair-wise combinations of test set created.
4) Simpler to generate and less error prone than test sets created manually.
5) Reduces testing cycle time.

By using this method, number of samples to be taken has been reduced to nine samples based on the manipulated variables; where initially there are suppose to be around twenty seven samples. Below is the number of experiments with different parameters that will be conducted based on Taguchi Method L9 Array:

Table 3.2.1: Experiment Parameters

		Level		
	Factor	**1**	**2**	**3**
A	Reaction Temperature (°C)	30	50	70
B	Catalyst concentration (%)	1.0	1.5	2.0
C	Seed Particle Size (mm)	0.5	1.0	2.0

Table 3.2.2: Experiment Trial Based On L9 Method

Trial	Control Factors		
	A	B	C
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

3.3 Project Activities

This section focused on the experimental methodology, materials and equipments used during the experiments done. Basically, the main jobs scope will be carried out are:

1) Penaga Seed Preparation
2) Oil Extraction
3) Acid Value
4) In-situ Transesterification
5) TLC Qualitative Analysis

3.3.1 Penaga Seed Preparation

Proceeding to in-situ transesterification, Penaga seed obtained from UTP's woods needs to be dehulled and ground to increase the process effectiveness as shown in the results. Penaga seeds were ground into smaller particle sizes and separated from 0.5mm to 2.0mm mesh size using sieve trays. Then, the seed was placed in the oven to reduce or remove any moisture content and the oven was set at 80°C for 2 hours. The moisture content was measured using Mettler Toledo moisture analyzer. To start analyzing, 10g of seed is placed in the sample pan and ramp time is being set to 3 minutes, followed by setting of the temperature to 100°C.

Figure 3.3.1.1 Sieve Trays

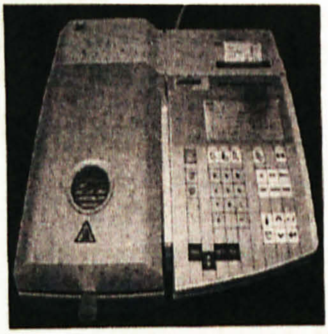


Figure 3.3.1.2 Moisture Analyzer

Figure 3.3.1.3 Ground Penaga Seed

3.3.2 Oil Extraction

In this project oil is extracted chemically from Penaga seed using soxhlet extractor and hexane is used as solvent since it can extract up to 99% of the total available oil. Extracted oil is used to determine the Penaga seed oil content. A soxhlet extractor unit consists of reflux condenser, a soxhlet container, a round bottom flask and a heater. 20g of Penaga seed is weighed and placed in the thimble. 140 ml of hexane with 99.9% purity was put into the round bottom flask. The extraction was operated on hexane boiling point, 68 °C and stopped after 2 hours.

Figure 3.3.2 Soxhlet Extractor

3.3.3 Acid Value

Based on American Oil Chemists' Society (AOCS) Method for Biodiesel Feedstock Quality, acid value is defined as the number of milligrams of potassium hydroxide necessary to neutralize the free acids in 1 gram of sample. With samples that contain virtually no free acids other than fatty acids, the acid value may be directly converted by means of a suitable factor to percent free fatty acids. The formula is as shown below:

Acid value, mg KOH/g of sample =

$$\frac{(A-B) \times N \times 56.1}{W} \qquad (3.3.3.1)$$

Where

A= volume, mL of standard alkali used in the titration

B= volume, mL of standard alkali used in titrating the blank

N= normality of standard alkali

W= mass, grams of sample

To determine the acid value, 10g of Penaga oils were dissolved in a mixture of toluene and isopropyl alcohol (containing a small amount of water) which resulting a single-phase solution. The solution was titrated at ambient temperature, 25°C with 0.1 N standard base; potassium hydroxide to the end point which indicated by the color change.

3.3.4 In-situ Transesterification

For in-situ transesterification, the synthesizing biodiesel was carried out in a batch process. Sulphuric acid used as catalyst was dissolved in the 140 ml alcohol; ethanol with various concentrations from 0.5% to 1.5% of the oil content. The mixture was then transferred into a two neck round bottom flask. Later, a weighed of 20g Penaga seeds were poured into the two neck round bottom flask and heated at the desired temperature of 30, 50 or 70°C. The flask was fully equipped with a magnetic stirrer, a thermometer and a reflux condenser. Ratio of ethanol to seed (volume: weight) used is 140:20.

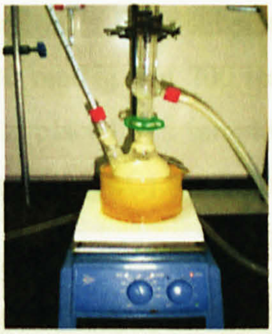
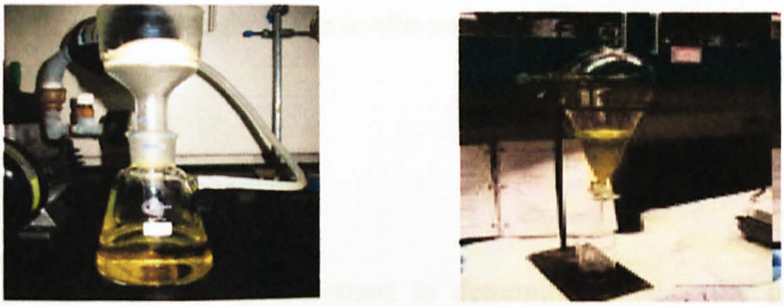


Figure 3.3.4.1 In-situ Transesterification Process

The reaction was stopped after 1 hour to minimize reverse reaction occurrence since tranesterification is a reversible reaction. Then, the mixture was vacuum-filtered using a Buchner funnel to separate the solution from the cake or solid particle. After that, the filtrate was transferred to a separator funnel and water was added. Further washing and extraction with hexane was done for three times.



(a) (b)

Figure 3.3.4.2 (a) Filtration (b) Settling

The separation process would take up to one day. After separation was finished, there were two layers appeared in the separator funnel which top layer mainly contained alkyl ester and bottom layer contained impurities such as alcohol, water, catalyst and glycerol. But there are possibilities that a little bit of ester might be escaped into the bottom layer and vice versa. Then, the solution was separated by draining bottom layer and the top layer was poured into a beaker.

Finished with separation, the top layer solution was then evaporated using rotary evaporator to separate alkyl ester from solvent; hexane. The rotary evaporator was operated at 70°C, 200 mmHg and 200 rpm. Next, the finalized sample of alkyl ester or biodiesel was placed in a sample bottle and further analysis using Thin Layer Chromatography (TLC) was done to qualitatively analyze the biodiesel.

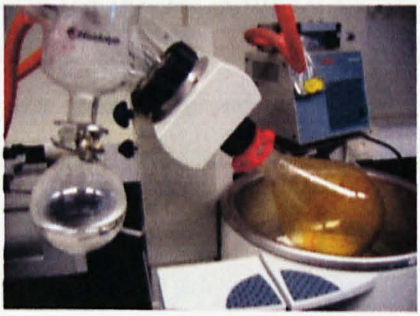


Figure 3.3.4.3 Rotary Evaporator

The parameters used throughout the in-situ transesterification process are as shown in **Table 3.2.1**.

3.3.5 TLC Qualitative Analysis

Analyzing biodiesel is very important to determine whether the product is acceptable as fuel to be used in diesel engines based on American Standard for Testing Method (ASTM) D 6751. The acceptance is depends on the impurities levels present in the produced biodiesel. There are two types of analytical methods can be used to analyze the biodiesel which namely as TLC (qualitative) and Gas Chromatography (GC) (quantitative). Since ASTM D 6584-00 is not available in UTP laboratory, only qualitative analysis was done using TLC.

TLC is a very common method for trying to determine how many different compounds are present in a sample. This test provides qualitative information about how many different compounds are present in a mixture. It is also to determine if two different samples contain different materials. For this analysis technique, a very small quantity of the biodiesel sample was placed on the special TLC silica plate with size of 2cm x 10cm. The plate was put in a container with a solvent or solvent mixture. In this experiment, solvents used were hexane, diethyl ether and acetic acid. The solvent runs up the plate and will separate the different kinds of molecules based on polarity differences and size differences. Once the eluent reached the desired distance which is 7cm, represent as retention factor, the plate was transferred into iodine chamber. 30 to 45 minutes later, a few spots were observed which represented the alkyl ester, triglyceride, free fatty acid, diglyceride and monoglyceride.

3.4 Tools Required

There are various tools required in order to complete the synthesis process especially the laboratory apparatus and chemical reagents such as:

3.4.1 Apparatus

1. 250 mL reaction flasks
2. Soxhlet Extractor unit
3. Hot plate with magnetic stirrer
4. Two neck round bottom flask
5. Thermometer
6. Buchner funnel
7. Separator Funnel
8. Rotary Evaporator
9. Thin Layer Chromatography (TLC) unit.
10. Reflux condenser
11. Calcination oven

3.4.2 Chemical Reagents

a) Alcohol (ethanol)
b) Acid catalysts (sulphuric acid, H_2SO_4)
c) Potassium Hydroxide
d) Sodium Sulphate
e) n-Hexane
f) Toluene
g) Iso propyl alcohol
h) Petroleum Ether
i) Diethyl Ether
j) Acetic Acid Glacial

3.5 Project Process Flow

Table 3.5: Gantt Chart

No.	Detail/ Week	1	2	3	4	5	6	7	8	9		10	11	12	13	14
1	Lab Work Continue	🟩	🟩	🟩							MID SEMESTER BREAK					
2	Submission of Progress Report 1				☆											
3	Lab Work Continue				🟩	🟩	🟩	🟩								
4	Submission of Progress Report 2								☆							
5	Seminar (compulsory)									🟩		🟩	🟩			
5	Lab work continue								🟩	🟩		🟩	🟩			
6	Poster Exhibition											☆				
7	Submission of Dissertation (softbound)													☆		
8	Oral Presentation														☆	
9	Submission of Dissertation (hardbound)															☆

☆ Suggested milestone

🟩 Completed Process

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Penaga Oil Properties

4.1.1 Moisture Content

The moisture content was measured using Mettler Toledo moisture analyzer. To start analyzing, conditions and parameters are set as below:

Weight of seed (g) = 10
Ramp Time (minutes) = 3
Temperature (^{o}C) = 100

4.1.2 Acid Value

Acid value, mg KOH/g of sample =

$$\frac{(A-B) \times N \times 56.1}{W} \tag{4.1.2}$$

Where

A= volume, mL of standard alkali used in the titration
B= volume, mL of standard alkali used in titrating the blank
N= normality of standard alkali
W= mass, grams of sample

To express in term of free fatty acids as percent lauric, divide by 1.99 **(AOCS Method)**

Table 4.1.1: Volume of Standard Alkali Used for Titration

No	A, mL	B, mL
1	45	17
2	40	13
3	43	17

Sample calculation (reading no. 1),

A= 45 mL

B= 17 mL

N= 0.1 (0.1 N KOH is used)

W= 10 g of sample

Acid value,

$$= \frac{(45-17) \times 0.1 \times 56.1}{10}$$

= 15.71 mg KOH/ g of sample

Average acid value

$$= \frac{15.71 + 15.15 + 14.59}{3}$$

= 15.15 mg KOH/ g of sample

In term of free fatty acids as percent lauric

= 15.15/1.99

= 7.61 % lauric (% FFA)

Based on calculation above, it is determined that extracted Penaga oil is containing acid value of 15.15 mg KOH/ g of sample or 7.61 % lauric which is more than 7.5% FFA (free fatty acid) and can be considered as high acid value. So, acid pretreatment method and base catalyst are not recommended as cited in research by UTP PhD student, M Hafeez, 2009 and transesterification can be proceed with acid catalyst, and in this project sulphuric acid being used as the catalyst. Further information on Penaga seed/oil properties as shown below:

Table 4.1.2: Penaga Seed/Oil Properties

Properties	**Values**
Acid Value (mg KOH/g)	15.15
Acid Value (%FFA)	7.61
Moisture Content (%)	0.08
Specific Gravity at 30°C*	0.87
Kinematic Viscosity*	46
Heat Value (J/g)*	37562
Saponification Value Acid Value (mg KOH/g)*	202

* taken from properties of Penaga oil researched by M Hafeez, 2009

Since Penaga seed has not yet being used as feedstock for current biodiesel production, above properties cannot be used for comparison from other previous reports or experiments to determine the product quality. Therefore, further experimentation had to be done to determine the quality of biodiesel produce from Penaga seed or oil.

4.2 Compound Analysis

This analysis is to determine the compound present in the produced alkyl (ethyl) ester or biodiesel. Below is the TLC result and analysis for sample 8 (trial 2) as it is concluded that the sample has the highest percentage yield (calculated area) compared to other samples (Temperature = 70°C, Catalyst concentration = 1.0%, Particle size = 0.5mm):

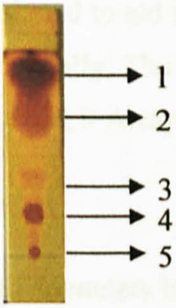


Figure 4.2.1 Compound Analysis for Sample 8 (trial 2)

The compound present can be determined as each spotted area signifies the type of component present in the ethyl ester produced. For typical biodiesel, there are usually five compounds present; alkyl ester, triglyceride, free fatty acid, diglyceride and monoglyceride. But, by using TLC only the qualitative compound can be verified. Any possible functional group cannot be determined from TLC analysis. The compound present can be concluded as shown below:

Table 4.2 List of Compound Present in Ethyl Ester

No	Compound
1	Ethyl Ester
2	Triglyceride
3	Free Fatty Acid
4	Diglyceride
5	Monoglyceride

From analysis above, it is shown that Penaga seed can form ethyl ester (bodiesel) qualitatively similar to other typical biodiesel compound.

4.3 Ethyl Ester Formation Analysis

For TLC analytical method, the exact value of ethyl esters formed cannot be determined, but using the spot area formed on TLC, the yield percentage can be calculated and it will be represent by ethyl ester spot area (cm^2) calculated. The area of ethyl ester formed of each sample can be calculated manually (as shown in appendix) or using Image J software. Image J software is a tool designed to aid in calculation and calculated area is more accurate compared to calculate manually. The parameters going to be used in the experiment are determined using Taguchi L9 Array technique and as shown in **Table 4.3.1.**

Table 4.3.1 Experiment Parameters Based On L9 Method

Sample	Control Factors		
	Temperature (°C)	Catalyst Concentration (%)	Particle Size (mm)
1	30	1.0	0.5
2	30	1.5	1.0
3	30	2.0	2.0
4	50	1.0	1.0
5	50	1.5	2.0
6	50	2.0	0.5
7	70	1.0	2.0
8	70	1.5	0.5
9	70	2.0	1.0

After calculated manually, ethyl ester spot areas for every sample were calculated and the results were tabulated as shown in **Table 4.3.2:**

Table 4.3.2: Ethyl Ester Spot Area of Each Sample

Sample	**Area Calculated (cm^2)**		
	Trial 1	**Trial 2**	**Mean**
1	0.680	0.625	0.653
2	0.750	0.875	0.813
3	0.940	1.000	0.970
4	0.750	0.875	0.813
5	0.980	1.125	1.053
6	1.270	1.250	1.260
7	1.020	1.188	1.104
8	1.360	1.500	1.430
9	1.290	1.375	1.333

Next, to determine which parameters affect the most, SN Ratio Analysis is done. SN Ratio is an average performance for each sample. Shown below is the sample calculation and tabulation of the SN ratio for sample 1:

$$S_{m1} = \frac{(0.68 + 0.625)^2}{2} = 0.8515$$

$$S_{T1} = 0.68^2 + 0.65^2 = 0.8530$$

$$S_{e1} = S_{T1} - S_{m1} = 0.8515 - 0.8530 = 0.00015$$

$$V_{e1} = S_{e1} / (2-1) = 0.00015$$

$$SN_1 = 10 \log \frac{(1/2)\,(0.8515-0.00015)}{0.00015} = \mathbf{24.49}$$

Below is tabulated data SN ratio for all samples:

Table 4.3.3 SN Ratio for Samples

Sample	Control Factors			SN
	Temperature (°C)	**Catalyst Concentration (%)**	**Particle Size (mm)**	
1	30	1	0.5	24.49
2	30	1.5	1	19.24
3	30	2	2	27.18
4	50	1	1	19.24
5	50	1.5	2	20.21
6	50	2	0.5	39.00
7	70	1	2	19.36
8	70	1.5	0.5	23.18
9	70	2	1	26.91

Shown below is the response calculation. This table was created by calculating an average SN value for each factor. An average SN value and response are the average performance for each level of parameters. A sample calculation is shown for Factor A (temperature) for level 1, 2 and 3 respectively:

$$SN_{B1} = \frac{(24.49 + 19.24 + 27.18)}{3} = \mathbf{23.64}$$

$$SN_{B2} = \frac{(19.24 + 20.21 + 39.00)}{3} = \mathbf{26.15}$$

$$SN_{B3} = \frac{(19.36 + 23.18 + 26.91)}{3} = \mathbf{23.15}$$

The effect of this factor is then calculated by determining the range:

$$\triangle = \max - \min = 26.15 - 23.15 = \mathbf{3.00}$$

Shown below is the mean SN ratio and response table for each sample. This table was created by calculating an average SN value for each factor as shown in sample calculation above:

Table 4.3.4 Average SN Ratio of Each Level

Parameter	SN Ratio		
	level 1	level 2	level 3
Temperature (°C)	23.64	26.15	23.15
Catalyst concentration (%)	21.03	20.88	31.03
Particle size (mm)	28.89	21.80	22.25

Table 4.3.5 Response Table

Level	Temperature (°C)	Catalyst Concentration (%)	Particle Size (mm)
1	23.64	21.03	28.89
2	26.15	20.88	21.80
3	23.15	31.03	22.25
△	3.00	10.15	7.09
Rank	**3**	**1**	**2**

Below are the plotted graphs of SN ratio for each level for every parameter involved from **Table 4.3.4**:

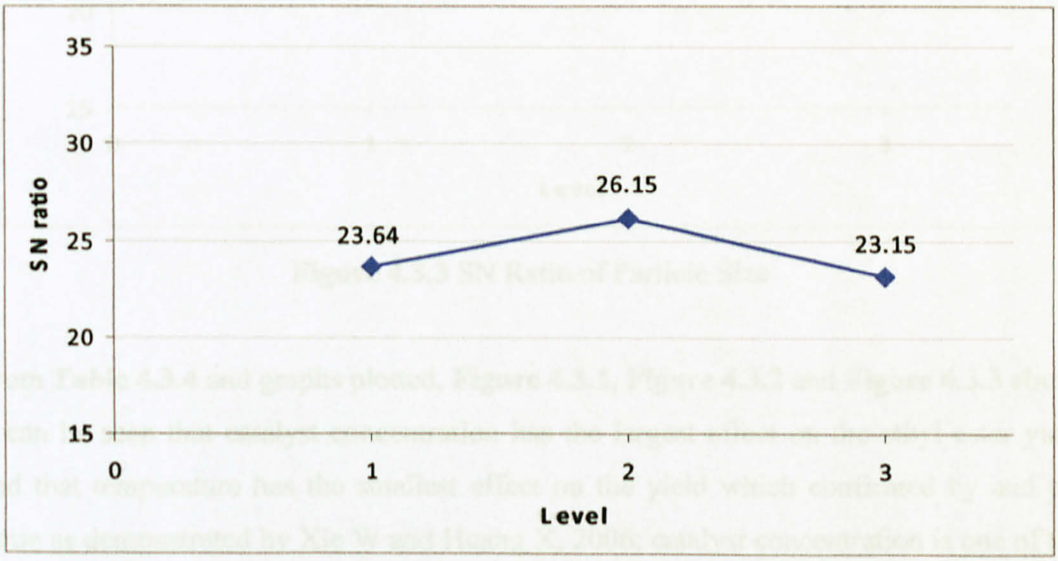


Figure 4.3.1 SN Ratio of Reaction Temperature

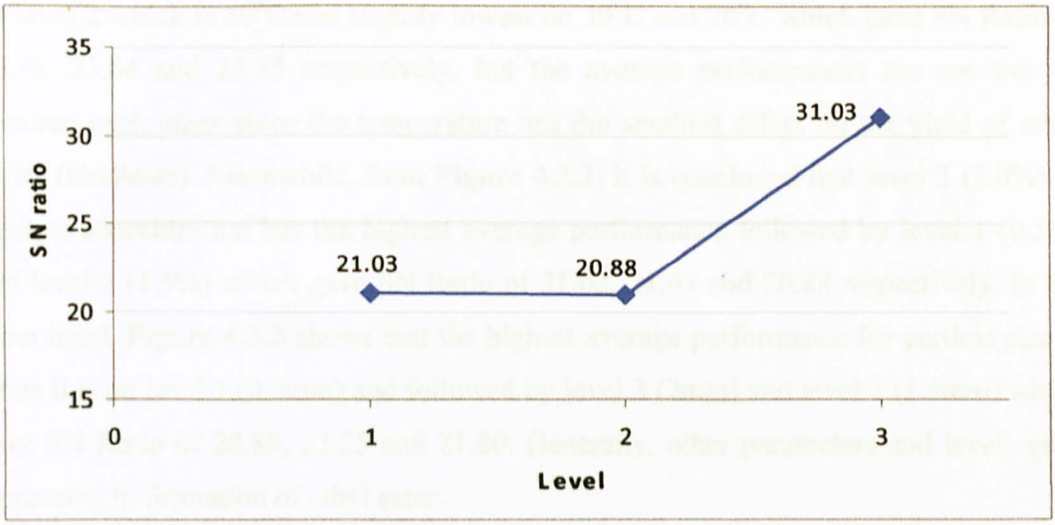


Figure 4.3.2 SN Ratio of Catalyst Concentration

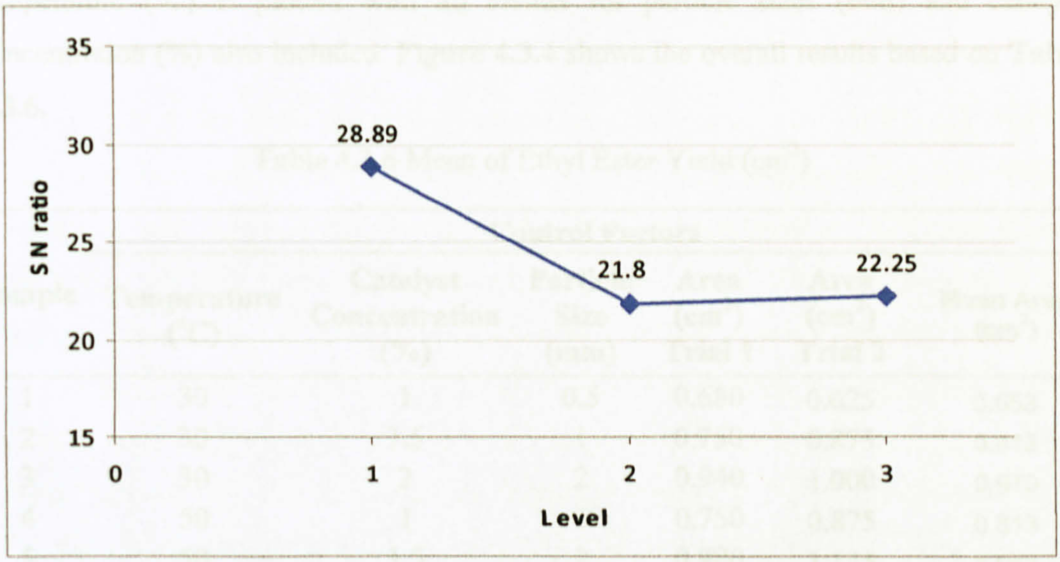


Figure 4.3.3 SN Ratio of Particle Size

From **Table 4.3.4** and graphs plotted, **Figure 4.3.1**, **Figure 4.3.2** and **Figure 4.3.3** above it can be seen that catalyst concentration has the largest effect on the ethyl ester yield and that temperature has the smallest effect on the yield which confirmed by and the same as demonstrated by Xie W and Huang X, 2006; catalyst concentration is one of the main parameter affecting the yield of alkyl ester throughout the reaction. Based on **Figure 4.3.1**, it shows that the highest average performance for temperature is when it is on level 2 which is 50°C and slightly lowest on 30°C and 70°C which gave SN Ratio of 26.15, 23.64 and 23.15 respectively, but the average performances are not too far between each other since the temperature has the smallest effect on the yield of ethyl esters (biodiesel). Meanwhile, from **Figure 4.3.2**, it is concluded that level 3 (2.0%) of catalyst concentration has the highest average performance followed by level 1 (0.5%) and level 2 (1.5%) which gave SN Ratio of 31.03, 21.03 and 20.88 respectively. In the other hand, **Figure 4.3.3** shows that the highest average performance for particle size is when it is on level 1 (0.5mm) and followed by level 3 (2mm) and level 2 (1.5mm) which gave SN Ratio of 28.89, 22.25 and 21.80. Generally, other parameters and levels gave increasing in formation of ethyl ester.

To further analyze the ethyl ester yield for each sample, graph of mean yield (cm^2) vs. temperature (°C) is plotted with all results for particle sizes (mm) and catalyst concentration (%) also included. **Figure 4.3.4** shows the overall results based on **Table 4.3.6.**

Table 4.3.6 Mean of Ethyl Ester Yield (cm^2)

	Control Factors					
Sample	**Temperature (°C)**	**Catalyst Concentration (%)**	**Particle Size (mm)**	**Area (cm^2) Trial 1**	**Area (cm^2) Trial 2**	**Mean Area (cm^2)**
1	30	1	0.5	0.680	0.625	0.653
2	30	1.5	1	0.750	0.875	0.813
3	30	2	2	0.940	1.000	0.970
4	50	1	1	0.750	0.875	0.813
5	50	1.5	2	0.980	1.125	1.053
6	50	2	0.5	1.270	1.250	1.260
7	70	1	2	1.020	1.188	1.104
8	70	1.5	0.5	1.360	1.500	1.430
9	70	2	1	1.290	1.375	1.333

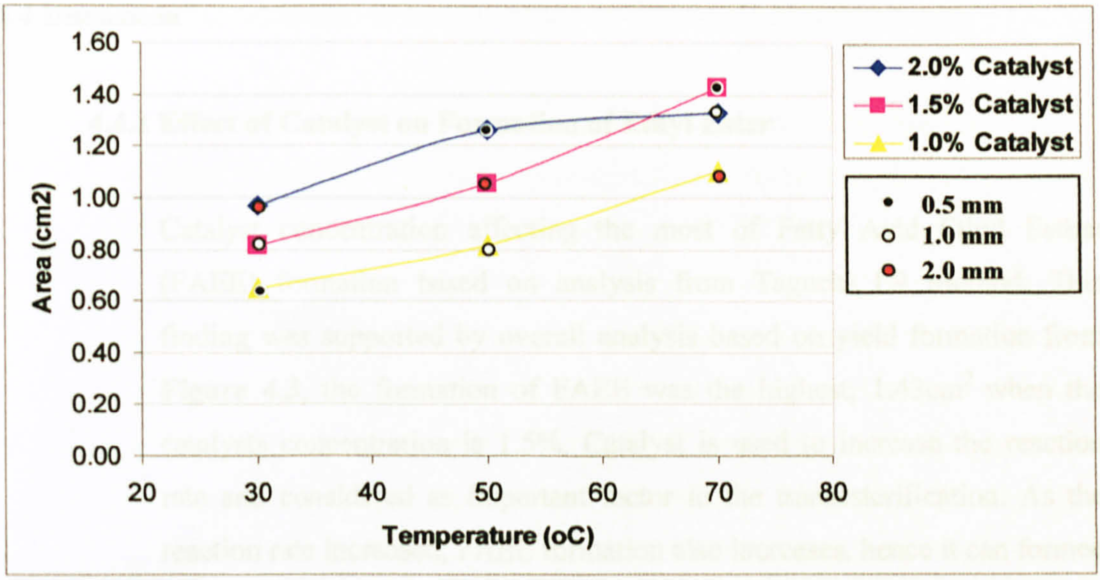


Figure 4.3.4 Fatty Acid Ethyl Ester Profile (FAEE) vs. Temperature, Catalyst Concentration and Particle Sizes

Figure 4.3.4 shows when the amount of the catalyst was at 1.0% of Penaga seeds at 30°C and 0.5mm particle size, the yields were lowest for all samples which is 0.653cm^2. Meanwhile, with 1.5% of catalyst concentration, at temperature of 70°C and 0.5mm particle size, the yield reached the maximum value of 1.43cm^2. The 1.5% level of H_2SO_4 catalyst at temperature of 70°C and particle size of 0.5mm was significantly better than the other levels. The in-situ transesterification with 2.0% catalyst at temperature of 30°C and 50°C gave high yields for 2.0mm and 0.5mm particle sizes which are 0.97cm^2 and 1.26cm^2 respectively. But the yield was decreased slightly at 2.0% with temperature of 70°C and 1.0mm particle size which gave 1.333cm^2 of yield. Therefore, Based on graph plotted above the highest yield in term of area (cm^2) was given by **sample 8** which the conditions and parameters as stated below:

Temperature = 70°C
Catalyst concentration = 1.5%
Particle sizes = 0.5mm
Yield (area) = 1.43cm^2

4.4 Discussion

4.4.1 Effect of Catalyst on Formation of Ethyl Ester

Catalyst concentration affecting the most of Fatty Acid Ethyl Esther (FAEE) formation based on analysis from Taguchi L9 Method. This finding was supported by overall analysis based on yield formation from **Figure 4.3**, the formation of FAEE was the highest, 1.43cm^2 when the catalysts concentration is 1.5%. Catalyst is used to increase the reaction rate and considered as important factor in the transesterification. As the reaction rate increased, FAEE formation also increases, hence it can formed FAEE almost as much as when the experiment conducted in higher temperature or smaller particle sizes. From the results, the yield slightly decreased with catalyst strength of 2.0% at 70°C because at this concentration the reaction equilibrium has been shifted due to reaction temperature.

4.4.2 Effect of Particle Sizes on Formation of Ethyl Ester

From analysis using Taguchi Method L9 Array, particle sizes have second choices that affecting the FAEE formation. From results, **Figure 4.3**, the highest formation of FAEE (1.43cm^2) was when the seed particle size is 0.5mm. Smaller particles have a larger amount of surface area as well as an increased number of ruptured cells resulting in a high oil concentration at the particle surface. Little diffusion into the particles takes place; therefore, the amount of oil available for extraction is proportional to the surface area. Hence, by reducing the particle sizes will increase the formation of FAEE.

4.4.3 Effect of Reaction Temperature on Formation of Ethyl Ester

Figure 4.3 shows that at the highest temperature, 70°C the formation of Ethyl Ester (cm^2) was the highest, $1.43cm^2$. It is known that activation energy related to rate constant, $k=A\exp-(Ea/RT)$ and rate constant related to temperature, where rate constant is proportional to the temperature; high temperature gives high rate constant. Thus, increased in reaction rate constant affecting the process thus caused more FAEE formed and gives the highest possible result to the FAEE production. Temperature also affects the reaction equilibrium. Temperature can shift the equilibrium either by increasing or decreasing it. It this experiment it shown that temperature lower the equilibrium at higher temperature thus the yield decreased at 70°C with 2% of catalyst concentration and 1.0mm particle size. Based on Taguchi L9 method, it is determined that temperature of 50°C is the best temperature. Overall, temperature has the lowest effect on FAEE formation among the parameters.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

Penaga seed can be considered as one of feedstock to produce biodiesel based on the results obtained. Although the quantitatives analysis is not done, but based on qualitative analysis Penaga seed or oil can produce alkyl ester (ethyl ester or biodiesel) but in slower reaction as the catalyst recommended to be used is acid-catalyst because of containing high acid value number. According to Ma F, Clements L D and Hanna M A, 1998, higher acid value number will gives smaller amount of alkyl ester formation. From results obtained, it is concluded that Penaga seed produced qualitatively similar to other typical biodiesel feedstocks.

Based on the results obtained with Taguchi Method L9 Array, catalyst concentration in weight percentage (wt %) parameter has the most effect in alkyl ester formation from Penaga oil. Followed by seed particle sizes in millimeter (mm) and reaction temperature (°C) has the smallest effect comparing to these three parameters. General overall, high catalyst concentration will gives higher performance, followed by particle sizes and reaction temperature respectively. But, the in-situ transesterification has to be stopped before reversible reaction start to react. Consequently, from the experimentation and results obtained the best conditions and parameter to be used for ethanolysis in-situ transesterification of Penaga oil which gave the highest yield in the experiment, 1.43cm^2 are:

a) Temperature = 70°C
b) Catalyst concentration = 1.5%
c) Particle sizes = 0.5mm

Finally to conclude the project, it is believed that in-situ transesterification method to synthesis biodiesel from Penaga seed or oil is feasible and could be commercialized to current marketing by considering the optimum conditions for the variables and parameters used.

5.2 Recommendation

1. Quantitative analysis can be made using gas chromatograph (GC) to determine the exact yield of biodiesel and functional groups that might be present in the biodiesel.

2. Further investigation also can be made to analyze in-situ transesterification of Penaga with addition of some other parameters such as:
    a) Ratio of alcohol to seed (volume to weight)
    b) Effect of reaction time
    c) Type of stirrer
    d) Stirrer sizes
    e) Stirrer speed

3) To get more accurate analysis results number of trials could be added as in this project only two trials have been done. Level of parameters also could be added to further investigate of each parameter as in this project the parameters level used were three levels.

4) Experimentation also can be further proceeding by comparing conventional method and in-situ method to differ the results and analysis between these methods.

5) Further comparison between normal method (by experimenting all twenty seven samples) and Taguchi Method also can be done to analyze advantages and disadvantages of each method.

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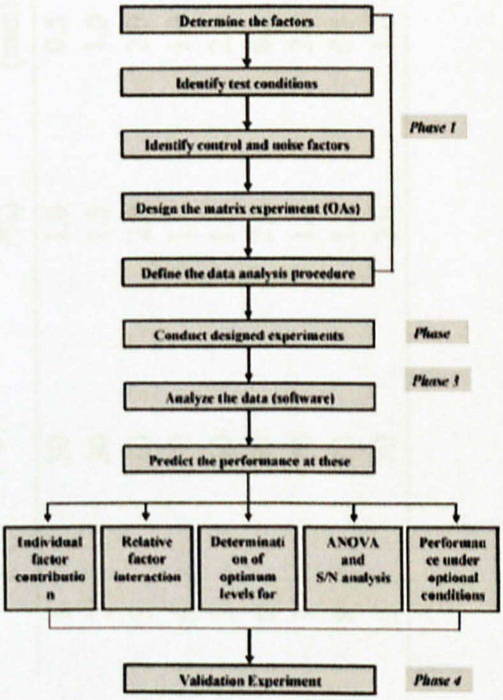
APPENDICES

1) Thesaurus

Noun	**1.**	**Mesua ferrea** - handsome East Indian evergreen tree often planted as an ornamental for its fragrant white flowers that yield a perfume; source of very heavy hardwood used for railroad ties ironwood, ironwood tree, rose chestnut genus Mesua, Mesua - genus of tropical Asiatic trees having large solitary flowers tree - a tall perennial woody plant having a main trunk and branches forming a distinct elevated crown; includes both gymnosperms and angiosperms 

Based on WordNet 3.0, Farlex clipart collection. © 2003-2008 Princeton University, arlex Inc.

2) A pictorial depiction of these and additional possible steps, depending on the complexity of the Taguchi Method analysis:



3) Formulas and calculated data to determine SN ratio:

Sample	Control Factors			Area(cm2) Trial 1	Area(cm2) Trial 2	Sm	St	Se	Ve	SN
	Temperature (°C)	Catalyst Concentration (%)	Particle Size (mm)							
1	30	1.0	0.5	0.680	0.625	0.851513	0.853025	0.001512	0.001512	24.49
2	30	1.5	1.0	0.750	0.875	1.320313	1.328125	0.007813	0.007813	19.24
3	30	2.0	2.0	0.940	1.000	1.881800	1.883600	0.001800	0.001800	27.18
4	50	1.0	1.0	0.750	0.875	1.320313	1.328125	0.007813	0.007813	19.24
5	50	1.5	2.0	0.980	1.125	2.215513	2.226025	0.010512	0.010512	20.21
6	50	2.0	0.5	1.270	1.250	3.175200	3.175400	0.000200	0.000200	39.00
7	70	1.0	2.0	1.020	1.188	2.436528	2.450556	0.014028	0.014028	19.36
8	70	1.5	0.5	1.360	1.500	4.089800	4.099600	0.009800	0.009800	23.18
9	70	2.0	1.0	1.290	1.375	3.551113	3.554725	0.003613	0.003613	26.91

Sm = ((Trial 1 + Trial 2)^2)/2

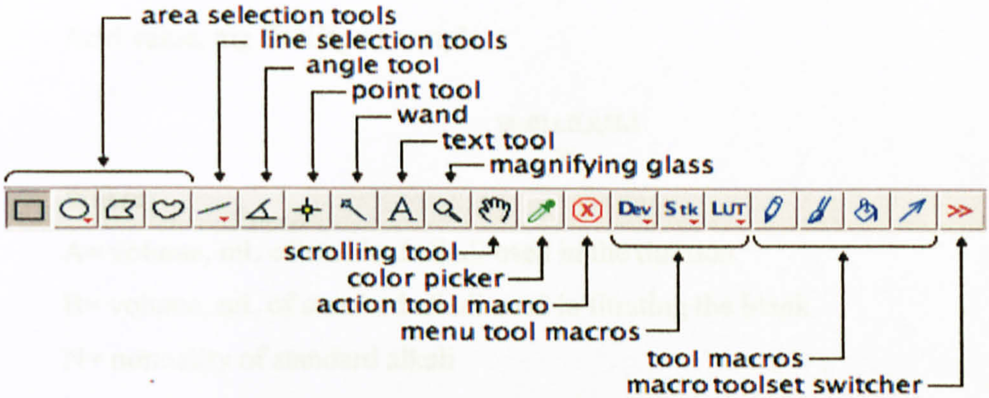
St = (Trial 1)^2 + (Trial 2)^2

Se = St1 – Sm1

Ve = Se1/(N-1)

SN = 10 log ((1/N)(Sm-Ve)/Ve))

4) ImageJ software application



5) Area Calculation (manually)

Sample calculation for sample 8 (trial 2),

Area of 1 small box = 0.5cm x 0.5cm

= 0.25cm^2

Therefore,

Area of Ethyl Esther = $0.25\text{cm}^2 \times 6$

= 1.5cm^2

6) Acid Value Calculation

Acid value, mg KOH/g of sample =

$$\frac{(A-B) \times N \times 56.1}{W}$$

Where

A= volume, mL of standard alkali used in the titration

B= volume, mL of standard alkali used in titrating the blank

N= normality of standard alkali

W= mass, grams of sample

To express in term of free fatty acids as percent lauric, divide by 1.99, from Table 4.1.1 (pg. 26),

Table 4.1.1: Volume of Standard Alkali Used for Titration

No	A, mL	B, mL
1	45	17
2	40	13
3	43	17

Calculation (reading no. 2 and 3),

Reading 2,

A= 40 mL

B= 13 mL

N= 0.1 (0.1 N KOH is used)

W= 10 g of sample

Acid value,

$$= \frac{(40-13) \times 0.1 \times 56.1}{10}$$

= 15.15 mg KOH/ g of sample

Reading 3,

A= 43 mL

B= 17 mL

N= 0.1 (0.1 N KOH is used)

W= 10 g of sample

Acid value,

$$= \frac{(43-17) \times 0.1 \times 56.1}{10}$$

= 14.59 mg KOH/ g of sample