SYNTHESIS OF BIODIESEL FROM JATROPHA CURCAS L.: IN-SITU TRANSESTERIFICATION

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by

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CERTIFICATION OF APPROVAL

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Approved by,

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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.

(Ahmad Zharif bin Abdullah)

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 which is more environmental friendly. Biodiesel is non-toxic fuel and biodegradable which is the reason why biodiesel can be a good sources to replace with the petroleum diesel

The experiments were carried out in a batch reactor equipped with a reflux condenser, a magnetic stirrer and a thermometer. Effect such as the particle sizes (0.5mm, 1.0mm, and 2.0mm) and different temperature (30°C and 70°C) were studied.

Basically, the project jobs scope will be carried out are: 1) Sample preparation; 2) Sample characterization; 3) Extraction of oil using Soxhlet Extractor; 4) In-situ Transesterification and 5) Qualitative Analysis. The products then will be analyzed by using Thin Layer Chromatography (TLC). As conclusion, it is believed that this method is feasible and could be commercialized considering the optimum conditions for the variables and parameters used.

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CHAPTER 1

INTRODUCTION

1.1Background study

Biodiesel is an alternative fuel similar to conventional or fossil diesel. Biodiesel can be produced from straight vegetable oil, animal oil/fats, tallow and waste cooking oil. The process used to convert these oils to Biodiesel is called transesterification. Biodiesel has many environmentally beneficial properties. The main benefit of biodiesel is that it can be described as carbon neutral. This means that the fuel produces no net output of carbon in the form of carbon dioxide (CO2). This effect occurs because when the oil crop grows it absorbs the same amount of CO2 as is released when the fuel is combusted. This research and experimental project will be focusing in synthesis of biodiesel from Jatropha oil using the in situ transesterification method. In situ methods transesterification is reaction mixture of Jatropha and alcohol using base-catalyzed or acid-catalyzed to produce biodiesel. The different parameters such as size of particles and temperature different will be manipulated to get the preferred product.

At present, biodiesel is usually produced by reacting methanol and vegetable oil in a batch stirred tank reactor using a liquid alkaline catalyst. The catalyst cannot be recovered from the reactor and is instead neutralized and disposed of as a waste stream. A heterogeneously- catalyzed process offers a number of advantages over current technology, namely the simplification and economization of the catalyst removal step, the removal potential by forming emulsions and the simplification of downstream separation processes. The transesterification reaction can be catalyzed by both acids and bases. The present work studied the transesterification of Jatropha oil with methanol catalyzed by solid base. Effects of various parameters were also studied.

Recently, Jatropha Curcas L. tree has been successfully cultivated in Upper Egypt and its fruit gives non edible oil. Jatropha oil can be utilized to produce biodiesel fuel. This can be done by its esterification with short chain alcohols, preferably at their super critical conditions without the use of catalysts. However, the success of this technology can be achieved if it is applied under its optimum conditions of temperature, pressure as well as alcohol to oil molar ratio.

1.2 Problem Statement

Demand of fuel has increase because of the growth of the human population and the usage of car. The increase in fuel usage has increase the air pollution. Besides that, with the current fuel price fluctuating and the limitation of the fuel resources, many researchers try to find the alternative fuels and one of it is by using biodiesel that is made from stable oil. The stable oil can reduce the air pollution because it has a close loop of CO2 which can reduce CO2 emission and sulfur oxide. Nowadays, many researchers have done the experiment to synthesis of biodiesel from Jatropha Curcas by using the different types of parameters to get the optimum conditions. One of the experiments is done by Surya 2009, where the parameters used were the effect of reaction time, effect of catalyst and their concentration, different speed of stirrer and the ratio of methanol to Jatropha seed. In this experiment, the parameters used are the different which are the sizes of Jatropha seeds and temperature used in order to improve the experiment that had been done by previous researchers.

1.3 Objective

The main objective of the project is to synthesis biodiesel from Jatropha Curcas through in-situ transesterification method by using different parameters to improve the yield or productivity. The project will be conducted by using three different parameters which are size of the Jatropha Curcas particles and different reaction temperature.

1.4 Scope of study

This project will be conducted based on the in situ transesterification methods. The properties and qualities of the biodiesel will be studied through Thin Layer Chromatograph TLC.

1.5 Relevancy of the Project

This project is focused on the production or synthesis of biodiesel from Jatropha Curcas L. which is same as diesel but different in properties. The main benefit of biodiesel is that it can be described as carbon neutral. This means that the fuel produces no net output of carbon in the form of carbon dioxide (CO2). This means that the fuel produces no net output of carbon in the form of CO2. This effect occurs because when the oil crop grows it absorbs the same amount of CO2 as is released when the fuel is combusted. Due to the increase in the price of petroleum crude and products and environmental concerns about air pollution caused by the combustion of fossil fuels, the search for alternative fuels has gained importance. So biodiesel is one of the alternatives to solve the problems.

1.6 Feasibility of the Project

UTP has been provided with the laboratory, the equipment and the chemical for this project, so give the advantages to finish the project. Synthesis of biodiesel from Jatropha also has been done by others but using the different methods. The information about the Jatropha can also easily get from the journal. Due to time constraint the project only conducted to study the synthesis of biodiesel from Jatropha through in-situ transesterification and effects of parameters such as co-solvent used and particle sizes on the synthesis process.

CHAPTER 2

LITRITURE REVIEW

2.1 Jatropha Curcas L.

Figure 2.1: Jatropha Tree

Figure 2.2: Jatropha Seed

According to Adebowale, K.O. and Adedire, C.O., *Jatropha curcas*, a potential anti-feedant candidate, belongs to the family, Euphorbiaccae. The seed which is black and oval in shape is rich in fixed oil (Shukla et al, 1996). The plant is a native of North America but now thrives well in Africa and Asia. It is easy to establish as it grows relatively quickly with high yields (Walls, 1967). Recently there was renewed interest on the utilisation of the seed oils in view of the relatively high oil content.

The role of J. curcas as a substitute for diesel is very remarkable (Sirisomboon et al. 2007). Augustus et al. (2002) reported the energy value of the seed: the gross heat value of the seed (0% moisture content) is 4980.3 cal/g (20.85 MJ/kg), which is higher than the heat value of lignite coal, cattle manure and comparable to the heat value of corn cobs (10% moisture content). The high gross heat value can be attributed to the presence of a high level of oil, polyphenol and hydrocarbon in the seed. The gross heat value of the oil fraction is 9036.1 cal/g (37.83 MJ/kg), which is higher than that of anthracite coal. The gross heat value of the hydrocarbon fraction was 9704.4 cal/g (40.63 MJ/ kg), which is higher than that of anthracite coal and comparable to that of crude oil. These results indicate that J. curcas may serve as an intermediate source of energy.

J. curcas, therefore, has a great potential as biodiesel. The fatty acid methyl ester of the oil of J. curcas was found most suitable for use as biodiesel and it meets most of the specifications of biodiesel standards of USA, Germany and European Standard Organization (Mohibbe Azam et al., 2005). The high viscosity of the J. curcas oil, which is considered as a potential alternative fuel for the compression ignition (C.I.) engine, is decreased by blending with diesel. Pramanik (2003) reported that significant improvement in engine performance is observed, compared to vegetable oil. Acceptable thermal efficiencies of the engine were obtained with blends containing up to 50% volume of Jatropha oil. From the properties and engine test results, it has been established that 40–50% of Jatropha oil can be substituted for diesel without any engine modification and preheating of the blends.

2.2 What is biodiesel and its advantages?

Biodiesel is an alternative fuel similar to conventional or fossil diesel. Biodiesel can be produced from straight vegetable oil, animal oil/fats, tallow and waste cooking oil. The process used to convert these oils to Biodiesel is called transesterification. This process is described in more detail below. The largest possible source of suitable oil comes from oil crops such as rapeseed, palm or soybean.

Most biodiesel produced at present is produced from waste vegetable oil sourced from restaurants, chip shops, industrial food producers such as Birdseye etc. Though oil straight from the agricultural industry represents the greatest potential source it is not being produced commercially simply because the raw oil is too expensive. After the cost of converting it to biodiesel has been added on it is simply too expensive to compete with fossil diesel. Waste vegetable oil can often be sourced for free or sourced already treated for a small price. (The waste oil must be treated before conversion to biodiesel to remove impurities). The result is Biodiesel produced from waste vegetable oil can compete with fossil diesel.

There are a few advantages when using biodiesel oil;

1. Pure Biodiesel is non-toxic, biodegradable and greenhouse gas neutral.
2. Biodiesel is cheap.
3. Both Biodiesel and mineral diesel have similar energy and power content and have proven to be of a similar retail value at the pump.
4. Biodiesel may not require an engine modification.
5. Biodiesel can make your car last longer.

2.3 Transesterification Theory

The transesterification process is the reaction of a triglyceride (fat/oil) with an alcohol to form esters and glycerol. A triglyceride has a glycerin molecule as its base with three long chain fatty acids attached. The characteristics of the fat are determined by the nature of the fatty acids attached to the glycerin. The nature of the fatty acids can in turn affect the characteristics of the biodiesel.

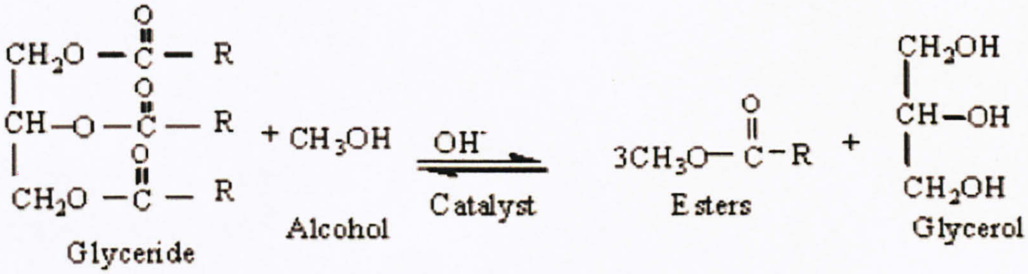

Figure 2.3: Reaction Schemes of Transesterification

The figure above shows the chemical process for methyl ester biodiesel. The reaction between the fat or oil and the alcohol is a reversible reaction and so the alcohol must be added in excess to drive the reaction towards the right and ensure complete conversion. Below is the skeletal formula for transesterification by using methanol as a solvent.

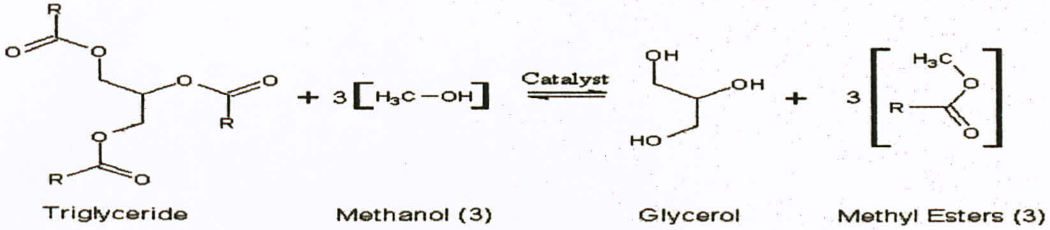

Figure 2.4: Skeletal Formula of Transesterification

During the esterification process, the triglyceride is reacted with alcohol in the presence of a catalyst, usually a strong alkaline like sodium hydroxide. The alcohol reacts with the fatty acids to form the mono-alkyl ester, or biodiesel and crude glycerol. In most production methanol or ethanol is the alcohol used (methanol produces methyl esters, ethanol produces ethyl esters) and is base catalyzed by either potassium or sodium hydroxide. Potassium hydroxide has been found to be more suitable for the ethyl ester biodiesel production, either base can be used for the methyl ester.

According to M. J. Haas et al. 2007, Alkali-catalyzed transesterification of fats and oils in the presence of a simple monohydroxy alcohol, usually methanol, is the predominant technology used for FAME synthesis. For vegetable oils this requires the prior recovery of the lipid by pressing or solvent extraction, and the partial purification of this oil. They have previously described an alternate method of FAME synthesis from soybean oil, termed in situ transesterification, which does not require the extraction and purification of the oil prior to FAME synthesis. Rather, thinly flaked soybeans are themselves the feedstock for this process, and alkali-catalyzed transesterification takes place directly within the flakes during incubation in alkaline alcohol. This process operates at high efficiency, and under optimum reaction conditions achieves maximum theoretical conversion of the acylglycerols of the feedstock into FAME. Furthermore, the resulting ester preparation meets the ASTM specifications for biodiesel.

Harrington and D Arcy-Evans (1985) is the first to explored the feasibility of in-situ method, using homogenized whole sunflower seeds 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 pre-extracted oil.

2.4 Transesterification In-Situ

Harrington and D Arcy-Evans (1985) said that 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 30oC 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 effect.

2.5 Based Catalyst Transesterification

Base on N.C. Om Tapanes et al., 2008, he base-catalyzed transesterification has a long story of development. Biodiesel fuel produced by this method is in the market in some countries such as North America, Brazil and mainly in some European countries like Germany and France. However, although base-catalyzed transesterification represents the best alternative to produce biodiesel, it still has some disadvantages. Low free fatty acid content and anhydrous reagents are required due to the saponification possibility. This soap formation lowers the ester yields and can hinder the stages of separation and purification of ester and glycerol as well as the washing stage. Additional disadvantages are that the catalyst recovery process is slow and expensive, increasing the operating costs. Therefore, the process still needs to be optimized.

2.6 Effect of particle size in oil recovery

According to John W 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 0.86-1.1mm, the total oil recovery was increased from 36% to 82% by mass.

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.

2.7 Reaction temperature

According to Surya 2009, reaction temperature has a negative impact on the yield of biodiesel. As the temperature was increase from 30 to 70°C, the yield of biodiesel was found to decrease inversely with the temperature due to side reaction of triglyceride saponification. On the other hand, Kucek et al., 2007 concluded that the temperature has a negligible effect on the yield of ethyl ester.

Freedman et al. showed that increased reaction yields were obtained when the alcoholysis of soybean oil was carried out at temperatures approaching the alcohol boiling point and this was particularly the case when methanol was used as the transesterification agent. However, other authors have shown that changes in the reaction temperature have little influence on the ethanolysis of TAGs derived from soybeans, sunflower and Raphanus sativus seeds and waste cooking oil.

2.8 Thin Layer Chromatography (TLC) Analysis

Chromatography is used to separate mixtures of substances into their components. All forms of chromatography work on the same principle.

Thin layer chromatography is done exactly as it says - using a thin, uniform layer of silica gel or alumina coated onto a piece of glass, metal or rigid plastic. The silica gel (or the alumina) is the stationary phase. The stationary phase for thin layer chromatography also often contains a substance which fluoresces in UV light.

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.

CHAPTER 3

METHODOLOGY

3.1 Experimental Approach

There are few steps in this experiment before complete it which are:

1. Seed preparation
2. Extraction oil using soxhlet
3. In-Situ Transesterification
4. Qualitative and Quantitative analysis

3.1.1 Jatropha Seeds preparation

Jatropha seeds were dried, dehulled and blended into smaller flakes and filter it to get the various size of Jatropha which are 50µm, 1mm and 2mm size in order to increase the efficiency of the extraction. In order to get the precise sizes of particles, sieve tray was used with the size fractions of 2mm, 1mm and 0.5mm. The Jatropha seeds then were dried using oven at 100°C for 2 hours to eliminate the presence of moisture. Then the seeds were keep in oven at 70°C to prevent presence of moisture in particles.

Figure3.1: Sieve Tray

3.1.2 Extraction of oil using Soxhlet Extractor (chemical extraction)

Chemical extraction is used to determine the oil content of the Jatropha seed. The oil from Jatropha seed was extracted by using the Soxhlet Extractor which operates at 70°C. The Soxhlet Extractor consists of reflux condenser, thimble, Soxhlet container, round bottom flask and heater. 20g of Jatropha seeds were weigh accurately and place in the thimble. Meanwhile 140ml of hexane with 99.9% purity was poured into round bottom flask. The extraction time started when the temperature reach 68°C and stop after 2 hours. Mixing of oil and hexane is then separated by using the rotary evaporator which operates at 70°C and 200rpm. The equipment wet up is shown in figure below:

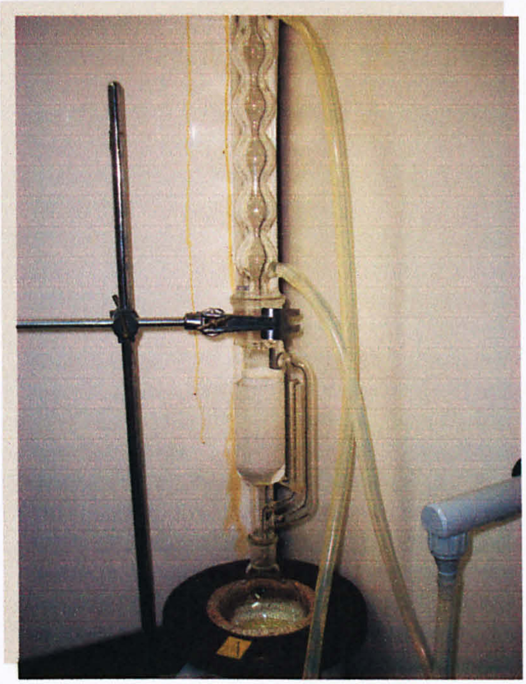

Figure 3.2: Soxhlet Extrator

3.1.3 In-situ Transesterification

Based catalyst which is sodium hydroxide (5ml) was dissolved in the alcohol (140ml ethanol) with concentration of 2.0 wt% of the oil content and the solution was then transferred into two neck round bottom flask and heated at temperature of 30and 70°C. Then 20g seeds were poured into two neck round bottom flask and mix with the heated catalyst and alcohol. Two neck round bottom flask was equip with magnetic bar, a reflux condenser and a thermometer. Two neck round bottom flask was soak into water bath that contain the paraffin oil.

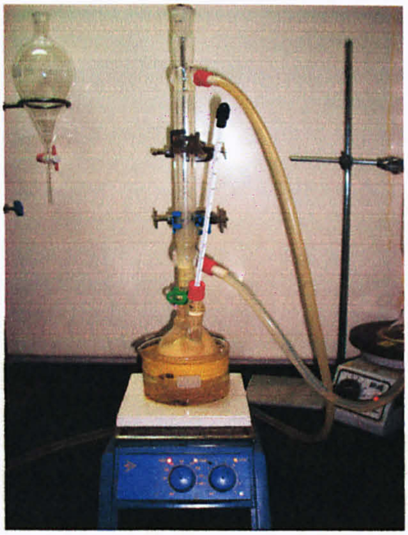

Figure 3.3: In-Situ Transesterification Process

The reaction time was recorded when the temperature the desired value of 30°Cand 70°C and stop after 20, 40, 60, 80, 100 and 120 minutes. Then the mixture was separated by using Buchner funnel to separate the solution from the cake. The filtrate obtained was transferred into separator funnel and added with water (20ml) and three times extraction with hexane (40ml). Separation process would take up 24 hours. After 24 hours separation, there were two layer appeared. The top layer contains ester and a little bit impurities such as triglyceride, water, catalyst and glycerol. The bottom layer contains alcohol, water, catalyst glycerol and a little bit of ester. The two layers then were separated by draining through the bottom of settling funnel. Once the removal of the bottom layer was completed, the top layer was poured into the beaker glass. The top layer was dried over sodium sulphate and the solution was filtered after 10 minutes to separate the solutions from the sodium sulphate precipitate.

Figure 3.4: Buchner Funnel

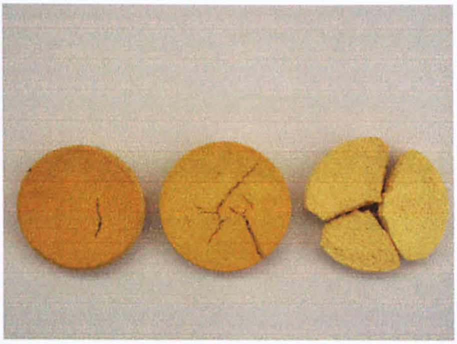

Figure 3.5: Particles Cake after Separation

The solution was then evaporated by using rotary evaporator to separate the alkyl ester from hexane at temperature of 70°C and 200rpm rotary speed. The alkyl ester was placed in a bottle and sealed.

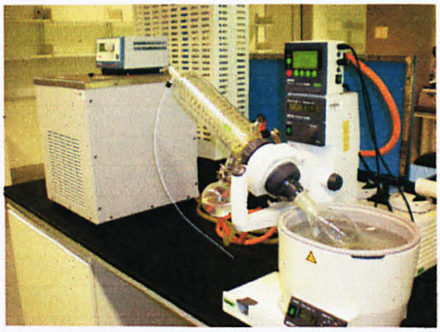

Figure 3.6: Rotary Evaporator

Figure3.7: Oil Sample

3.2 Qualitative Analysis

Thin Layer Chromatography (TLC) method is used to quantify the quality and quantity of the obtained product. A silica plate is used for image development and it soaked inside TLC chamber containing mixture of petroleum ether, diethyl ether and acetic acid glacial as a solvent with a percentage of 80, 20, 1(vol%) respectively based on experimental done by Surya, 2009.the sample was dilute in iso propanol with ratio of 1: 2 and the sample was put on the silica plate with size 10cm x 2 cm. After that the silica plate was moved into iodine chamber for 30 to 45 minutes to observe the spots. The spots will show the alkyl ester, triglyceride, free fatty acid, monodyglyceride and diglyceride. The illustrated can be show in the figure below:

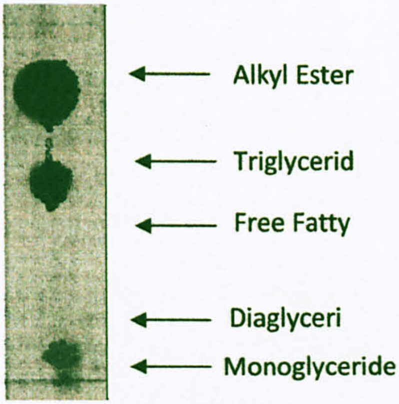

Figure 3.8: TLC results sample, Surya 2009

From the TLC above, calculate the area for the alkyl ester spot for each sample. The biggest area will be assumed as a benchmark to calculate the percentage yield of biodiesel produced by using the equation below:

$$percentage\ yield\ of\ biodiesel = \frac{calculated\ area\ of\ ester}{largest\ area\ of\ ester} x\ 100\%$$

After calculate the percentage yield of biodiesel, plot the graph for each parameter.

3.3 Tools Required

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

3.3.1 Apparatus

1. 250mL reaction flask
2. Hot plate with magnetic stirrer.
3. Heating mantle
4. Thin Layer Chromatography (TLC).
5. Reflux condenser
6. Calcination oven.
7. Vacuum dryer.
8. Soxhlet Extractor
9. Rotary Evaporator

3.3.2 Chemical Reagents

1. Alcohol (ethanol)
2. Sodium Hydroxide
3. Sodium Sulphate
4. n-Hexane
5. Toluene
6. Iso Propanol
7. Petroleum Ether
8. Diethyl Ether
9. Acetic Acid (Glacial)
10. Tetrahydrofuan (THF)

3.4 Project Milestone

Table 3.1: Gantt Chart

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

🟥 Process

🟦 Completed

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Analyzing Experimental Data Results

Data was analyzed by referring the area obtained from TLC. Taking largest area as a bench mark and calculate the percentage yield of biodiesel obtained then plots the graph.

This analysis is to determine the compound present in the produced alkyl (ethyl) ester or biodiesel. Below is the TLC result and analysis as it is concluded that the sample has the highest percentage yield (calculated area) compared to other samples

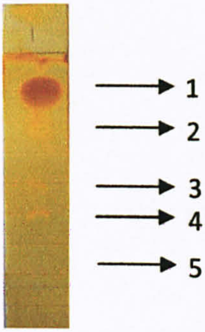

Figure 4.1: Compound Analysis

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 4.1: 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 Jatropha Curcas seed can form ethyl ester qualitatively similar to other typical biodiesel compound.

4.1.1 Effect of particle sizes

Below is the data of the experiment for the amount of oil obtained from the seeds before analyzing it. There are 3 different sizes of particle that being used which are 4 mm, 2 mm and 1mm diameter. Below is the result for the amount of oil obtained from the seeds.

Table 4.2: Data for 4mm of particles sizes

Exp. Number	Time (min)	4mm			
		Weight of Seeds (g)	NaOH(ml)	Oil Weight(g)	Area (cm2)
1	20	20.056	5	0.384	0.812
2	40	20.425	5	0.562	1.000
3	60	20.025	5	0.688	1.062
4	80	20.031	5	0.69	1.125
5	100	20.012	5	0.84	1.125
6	120	20.086	5	0.856	1.250

Table 4.3: Data for 2mm of particles sizes

Exp. Number	Time(min)	2mm			
		Weight of Seeds (g)	NaOH(ml)	Oil Weight(g)	Area (cm2)
7	20	20.01	5	1.28	0.875
8	40	20.015	5	1.923	1.125
9	60	20.032	5	1.988	1.062
10	80	20.019	5	2.787	1.250
11	100	20.003	5	2.879	1.437
12	120	20.011	5	3.165	1.560

Table 4.4: Data for 1mm of particles sizes

Exp. Number	Time(min)	1 mm			
		Weight of Seeds (g)	NaOH(ml)	Oil Weight(g)	Area (cm2)
13	20	20.065	5	2.978	1.000
14	40	20.025	5	3.579	1.187
15	60	20.189	5	3.962	1.375
16	80	20.011	5	4.181	1.187
17	100	20.025	5	4.48	1.500
18	120	20.01	5	5.452	1.560

From table above, the result show increasing the amount of oil produce as the reaction time increase. Besides that, smaller sizes of the particle use greater oil can be produced. Each sample then is tested by using TLC. Below is the data and graph for each sample:

Table 4.5: Data for Effect of Particle Sizes

Reaction time (minutes)	Yield of Ethyl Ester (%)		
	1 mm	2 mm	4 mm
20	64.1	56.1	52.1
40	76.1	72.1	64.1
60	88.1	68.1	68.1
80	76.1	80.1	72.1
100	96.2	92.1	72.1
120	100	96.2	80.1

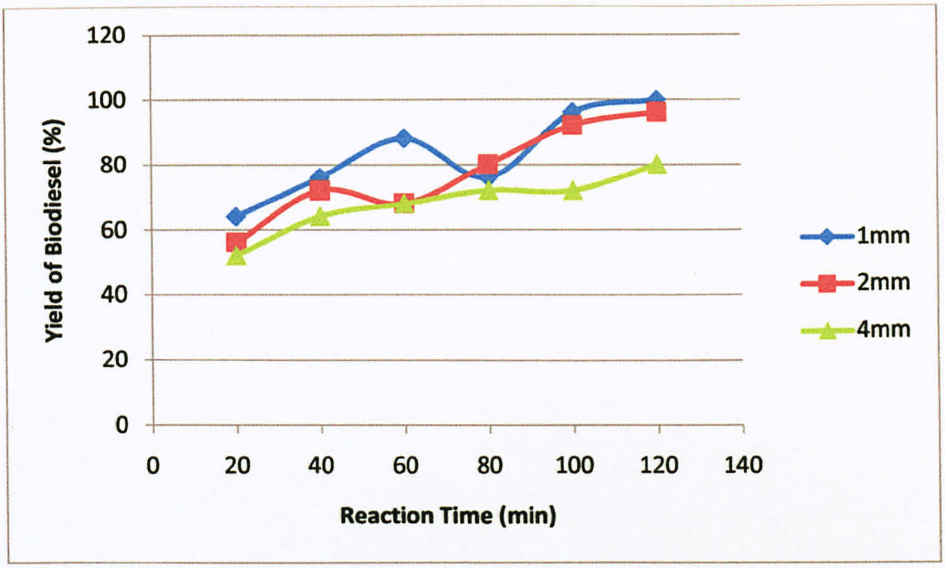

Figure 4.2: Graph Yield of Ethyl Ester vs Reaction Time (min)

Results show that increasing the reaction time will increase the amount of yield of biodiesel. Besides that, smaller particle sizes will also increase the total amount of biodiesel produced. **Figure 4.2** above shows that 1mm particle sizes will give better yield of biodiesel following with 2mm and 4 mm sizes of particles. But there are some samples that shows the negative value which at condition 2mm size and temperature 1 hour and 1mm sizes at temperature 80 minutes. It shows decreasing in the amount of biodiesel produced. The mistakes happened during conducting the experiment. There spills of the solutions during separation process that effect the value of biodiesel itself.

4.1.2 Effect of temperature

Theoretically increasing the temperature will increase the reaction rate to achieve equilibrium. But in this experiment increasing the temperature reaction will increase the possibility for saponification occur. Besides that, this reaction is an exothermic reaction which mean that when the reaction is already in equilibrium there is high possibility for reversible reaction to occur which mean reduce the amount of biodiesel itself. It can be prove by the results below:

Table 4.6: Data for 1mm particle sizes at temperature 70°C

Exp. Number	Time(min)	1 mm,70°C			
		Weight of Seeds (g)	NaOH(ml)	Oil Weight(g)	Area (cm2)
19	20	20.065	5	3.069	1.000
20	40	20.025	5	3.586	1.187
21	60	20.189	5	4.012	1.250
22	80	20.011	5	4.535	1.375
23	100	20.025	5	5.164	1.437
24	120	20.010	5	5.402	1.437

Table 4.7: Comparison data between temperature 30°C and 70°C

Reaction time (minutes)	Biodiesel Yield (wt%)		
	30°C	70°C	
	Biodiesel Yield (wt%)	Biodiesel Yield (wt%)	Oil Weight (g)
20	64.1	64.1	3.069
40	76.1	76.1	3.586
60	88.1	80.1	4.012
80	76.1	88.1	4.535
100	96.2	92.1	5.164
120	100	92.1	5.402

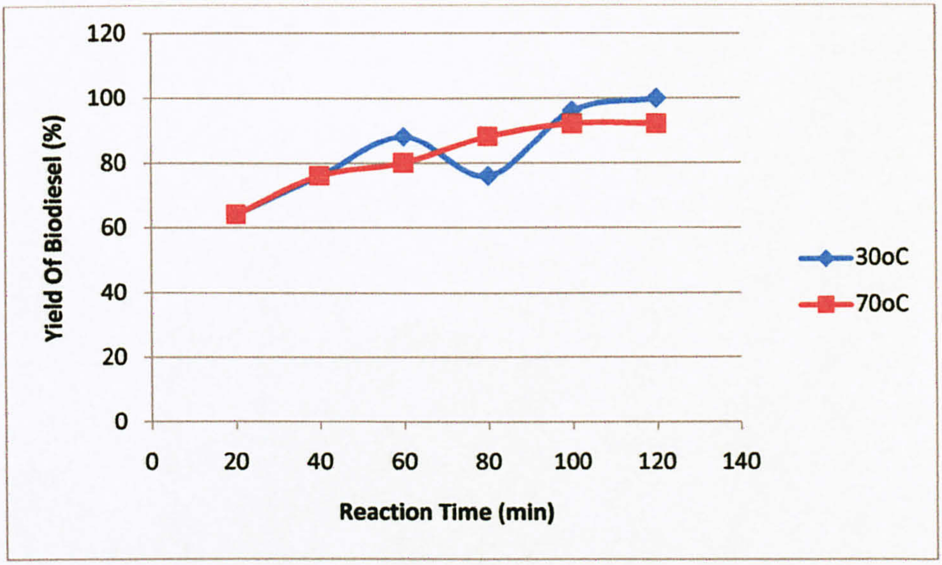

Figure 4.3: Graph Yield of Ethyl Ester vs Reaction Time (min)

From the results above, increasing the temperature from 30°C to 70°C will increase the amount of biodiesel also. But the total amount of biodiesel is decrease due to forming of soap and the reversible reaction occurs. The highest percentage of biodiesel for temperature 70°C is 92.1 which is lower than reaction temperature at 30°C.

4.2 Discussion

4.2.1 Effect of Particle Sizes on Formation of Ethyl Ester

From analysis based on TLC, particle sizes have the largest effect for the formation of biodiesel ($1.56cm^2$). From figure 4.2, the highest formation of ethyl ester (1.56cm2) was when 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 ethyl ester.

4.2.2 Effect of Reaction Temperature on Formation of Ethyl Ester

Figure 4.3 shows that the higher the temperature (70^oC), lower formation of ethyl ester due to formation of soap and reversible reaction to occur. 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. Increasing the temperature will increase the reaction rate thus reduce the time for the reaction to reach equilibrium. The reaction is basically exothermic reaction and when the reaction reach equilibrium, the chemical reaction which is reversible reaction will tend to shift the reaction into the left side. In this experiment, increasing

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Synthesis of biodiesel from Jatropha Curcas using in situ transesterification process was investigated in this work. The yield of biodiesel was studied by manipulating the reaction time and temperature of reaction. The biodiesel was analyzed by using Thin Layer Chromatography (TLC) based on method developed by Hass and Foglia, 2003.

Based on the experiment done in this project, it can be concluding that particle sizes have the largest effect on the production of biodiesel rather than temperature. Smaller particle sizes will produce high amount of yield of biodiesel. Results have shown that 1mm particle sizes five high amount of yield of biodiesel produced. 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.

Temperature has negative impact on the production of biodiesel. Increasing the temperature from 30oC to 70oC will decrease the yield of biodiesel due to formation of soap and reaction shifted to left side. The reaction is basically exothermic reaction and when the reaction reach equilibrium, the chemical reaction which is reversible reaction will tend to shift the reaction into the left side. Therefore it can be conclude that increasing temperature will reduce the yield of biodiesel.

5.2 Recommendation

There are many aspect that need to be improve in this experiment which are:

1. Used ImageJ software to determine the area at TLC plate to get more accurate data.
2. Gas Chromatograph (GC) should be used to get quantitative analysis which the accurate data can be obtained from GC.
3. Increase the reaction time for the effect of temperature different to investigate more on it.
4. The experiment should repeat eda few times to get the average data.
5. Experimentation also can be further proceeding by comparing conventional method and in-situ method to differ the results and analysis between these methods.

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