# PREPARATION OF BIODIESEL FROM WASTE COOKING OIL USING SINGLE STEP BATCH CATALYST WITH THE AID OF KOH AS A CATALYST

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**JUDUL:** PREPARATION OF BIODIESEL FROM WASTE COOKING OIL USING SINGLE STEP BATCH CATALYST

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KA 04041

A thesis submitted in fulfilment of the requirements for the award of the degree of Bachelor of Chemical Engineering

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"I declare that this thesis is the result of my own research except as cited references. The thesis has not been accepted for any degree and is concurrently submitted in candidature of any degree"

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# DEDICATION

*Special dedication to my beloved father, mother, brothers and sisters……………….*

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Wassalam

# ABSTRACT

Biodiesel is a cleaner burning diesel replacement fuel processed from natural, renewable derived from biological sources such as used vegetable oils and animal fats. The type of process that used to produce biodiesel is transesterification. Transesterification is the commonly process that used widely compare to other process. This process is important to convert free fatty acid (FFA) in oil to ester. In this research, waste cooking oil is used as raw material with Potassium hydroxide (KOH) as catalyst and methanol as a solvent. The reaction has been done in a water bath. The parameter that been investigated were reaction time and catalyst concentration while reaction temperature was fixed at 65˚C. Finally, the biodiesel sample was tested on several parameters which are the yield, thin layer chromatography (TLC), and moisture content. The analysis has been done using Response Surface Methodology (RSM) to find the optimum condition based on higher yield and TLC. It is to ensure its meet the standard requirement of ASTM D6751 for biodiesel B100. The optimum result for higher yield is 73.38 minute reaction time and using 1.33% catalyst concentration and the optimum result for higher TLC is 90 minute reaction time and 0.5% catalyst concentration.

# ABSTRAK

Biodiesel adalah pengganti minyak pembakar diesel yang bersih yang diproses dari alam semulajadi, ia dapat diperbaharui hasil daripada sumber biologis seperti minyak sayuran dan lemak haiwan. Proses yang digunakan untuk menghasilkan biodiesel adalah *transesterification*. *Transesterification* adalah proses yang selalu digunakan secara meluas berbanding proses yang lain. Proses ini penting untuk merubah asid lemak berlebihan ke minyak atau ester. Dalam eksperimen ini, minyak masak yang telah digunakan dijadikan bahan mentah dan *Potassium hydroxide (KOH)* sebagai mangkin dan methanol untuk pelarut. Tindak balas ini telah dijalankan menggunakan *water bath.* Parameter yang harus dikaji adalah masa tindakbalas dan kepekatan mangkin, manakala suhu tindakbalas ditetapkan pada 65˚C. Akhirnya, sampel biodiesel akan diuji ke atas beberapa parameter iaitu jumlah hasil, kepingan nipis kromatografi (TLC), dan kandungan air. Analisisnya pula menggunakan perisian komputer Response Surface Methhodology (RSM) untuk mencari keadaan optimum berdasarkan jumlah hasil dan TLC yang tinggi. Ini untuk memastikan hasil mencapai norma piawaian ASTM D6751 untuk biodiesel B100. Data optimum untuk mencapai hasil biodiesel tertinggi adalah 73.38 minit masa tindak balas dan 1.33% kepekatan mangkin dan data optimum untuk mencapai TLC tertinggi adalah 90 minit masa tindak balas dan 0.5% kepekatan mangkin.

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# LIST OF ABBREVIATIONS

| | |
|---|---|
| $^0$C | Degree Celcius |
| ANOVA | analysis of variance |
| CCD | Central Composite Design |
| DOE | Design of Experiment |
| FFA | free fatty acid |
| h | hour |
| KOH | Potassium Hydroxide |
| Min | minute |
| ml | mililiter |
| NaOH | Sodium Hydroxide |
| RSM | Response Surface Methodology |
| TLC | Thin Layer Chromatography |

# CHAPTER 1

# INTRODUCTION

## 1.1 Introduction

As world’s petroleum supplies are becoming constrained, attention has been directed to find out alternative sources of fuels for engines. The non-renewable nature and limited resources of petroleum fuels have become a matter of great concern. After the 1973 oil embargo, it had been very important to study the alternative sources of fuel for diesel engine because of the concern over the availability and the price of petroleum based fuels. The present reservation of fuels used in internal combustion (IC) engines including diesel will deplete within 40 years if consumed at an increasing rate estimated to be of the order of 3% per annum. All these aspects have drawn the attention to conserve and stretch the oil reserves by way of alternative fuel research.

Thus, the use of alternatives energy such as hybrid technology and hydrogen cell requires extra modification to the vehicle engine. As a result, higher costs, longer duration of time to develop were needed to upgrade this technology. Thus, research on new energy sources, such as biodiesel has become more importance in recent years.

## 1.2 Background Of Study

Biodiesel is the alternative fuel substance that will replace the petrol one day. It can be commercialize because it has more advantages compare to other source of fuel. Only the little fossil energy is required to move biodiesel. Special pumps or high pressure equipment for fuelling are not needed for this specification. In addition, it can be used in conventional diesel engine, so we do not need any upgrading or new purchasing for our engine.

In petrol, the scientists believe carbon dioxide is one of the main greenhouse gases contributing to global warming. The 100% biodiesel will reduces carbon dioxide emissions by more than 75% over petroleum diesel. If we use a blend of 20% biodiesel, it will reduce carbon dioxide emission by 15%. Biodiesel also produces less particulate matter, carbon monoxide, and sulphur dioxide emission which is all air pollutants under the Clean Air Act.

This renewable fuel can directly replace petroleum product since it can be used in conventional diesel engines. The biodiesel also can reduce the country’s dependence on imported oil. Biodiesel offers safety benefits compare petroleum diesel because it is much less combustible, with a flash point greater than 150°C, compares to 77°C for petroleum diesel.

The energy that release for biodiesel is about same for petroleum diesel (118,000 vs. 130,500BTUs). Therefore, engine torque and horse power remain virtually same. Biodiesel is safer than petroleum diesel to handle or store. Biodiesel does not produce dangerous vapors at normal ambient temperatures or room temperatures, and it can be stored in the same containers and tank as petroleum diesel.

The disadvantages of biodiesel is based on the sources that used to produced the biodiesel, as an example if we produce biodiesel from waste cooking oil, the process will also produce FFA (free fatty acid) as a side product. This biodiesel production to commercialize is still along with the research to cover the disadvantages.

The biodiesel used has grown dramatically during the last few years. The Energy Policy Act was amended by the Energy Conservation Reauthorization Act of 1998 to include biodiesel fuel use as a way for federal, state, and public utility fleets to meet requirements for using alternative fuels. In Malaysia there are plenty company that starting this biodiesel production using Jarak fruit or the scientific name is Jathopra curcas Linn, but during the production it still come along with research with our local university like Universiti Malaysia Pahang (UMP).

## 1.3 Problem Statement

In Malaysia, government have gives subsidized to petroleum oil in transportation sector. It shows that petroleum price is in unstable economy. Thus, using waste cooking oil will make the biodiesel price is compatible than subsidized petroleum diesel. The availability of raw material and cheaper cost are the main criteria for choosing good raw material.

The high contain of fatty acid need to be synthesize using single step of catalyzed process to prevent the high yield of soap. The single step is more economical process compare to other process. The transesterification process is used because its give high yield in reaction, reduce the production cost and low reaction time rate. In the transesterification process, methanol will be use as alcohol solvent because its cheaper compares other alcohol solvent.

## 1.4 Objective

The main purpose of this research is to produce biodiesel from waste cooking oil via single step batch catalyzed process and to find optimum condition using DOE software to get higher yield and thin layer chromatography (TLC).

## 1.5 Scope Of Researchs

This research aim is a study in production of biodiesel using waste cooking oil as the feedstock. To obtain the research objective, three elements have been identified to be studied in this experiment. The three elements are:

i. To study the effect of reaction time and catalyst concentration. In the transesterification process.
ii. To analyze several parameter such as yield, TLC and moisture content.
iii. To study single step catalyzed transesterification process.’

# CHAPTER 2

# LITERATURE REVIEW

### 2.1.1 Biodiesel Properties

Biodiesel is other alternative fuel to replace petrol diesel that is produces by chemically reacting a vegetables oil or animal fat. There are many benefits we get from biodiesel in comparison to petroleum based fuels. It includes, the reduction of CO emission by approximately 50% and carbon dioxide by 78% on a net lifecycle basis. It happens because the carbon in biodiesel emissions is recycled from carbon that was in the atmosphere, rather than the carbon introduced from petroleum that was sequestered in the earth's crust. [Sheehan, 1998]. Biodiesel also contains fewer aromatic hydrocarbons and biodiesel has a higher cetane rating than petrodiesel, which can improve performance and clean up emissions compared to crude petrodiesel.

The most important fact is biodiesel is biodegradable and non-toxic. The U.S. Department of Energy confirms that biodiesel is less toxic than table salt and biodegrades as quickly as sugar. In the United States, biodiesel is the only alternative fuel to have successfully completed the Health Effects Testing requirements (Tier I and Tier II) of the Clean Air Act (1990). The other advantages of biodiesel are, when biodiesel were used in a blend with petroleum diesel, there are fewer formal studies about the effects on pure biodiesel in unmodified engines and vehicles in day to day use. Fuel meeting the standards and engine parts that can withstand the greater solvent properties of biodiesel is expected to run without any additional problems than the use of petroleum diesel.

| Properties | Biodiesel (vegetable oil methyl ester) | | | | | |
|---|---|---|---|---|---|---|
| | Peanut | Soyabean | Palm | Sunflower | Linseed | Tallow |
| Kinematic viscosity at 37.8 1˚C($m^2$/s) | 4.9 | 4.5 | 5.7 | 4.6 | 3.59 | - |
| Cetane number(˚C) | 54 | 45 | 62 | 49 | 52 | - |
| Lower heating value (MJ/l) | 33.6 | 33.5 | 33.5 | 33.5 | 35.3 | - |
| Cloud point(˚C) | 5 | 1 | 13 | 1 | - | 12 |
| Pour point(˚C) | - | -7 | - | - | -15 | 9 |
| Flash point(˚C) | 176 | 178 | 164 | 183 | 172 | 96 |
| Density(g/ml) | 0.883 | 0.885 | 0.88 | 0.86 | 0.874 | - |
| Carbon residue (wt%) | - | 1.74 | - | - | 1.83 | - |

**Table 2.1** Properties of Biodiesel

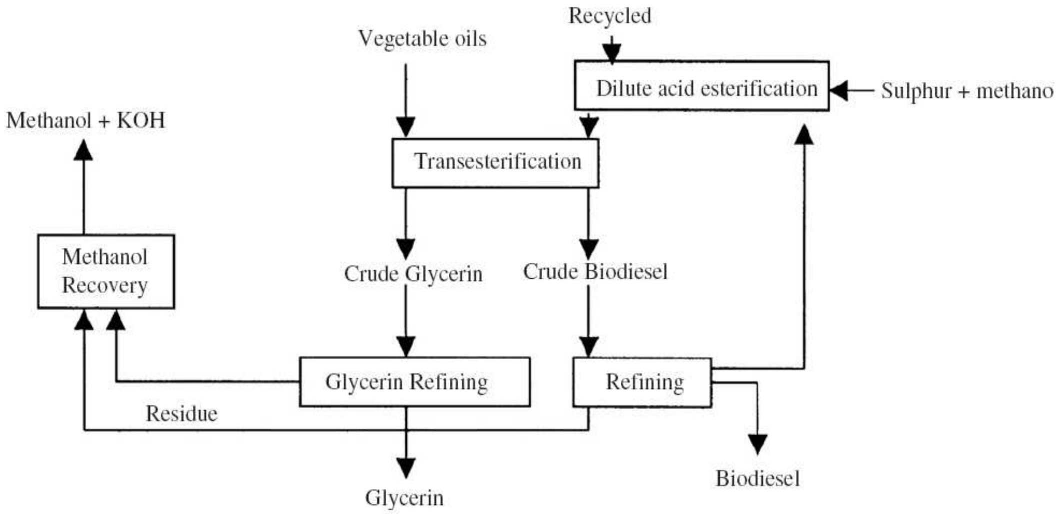


**Figure 2.2** A scheme of the flow chart

The characteristics of biodiesel are close to diesel fuels, and therefore biodiesel becomes a strong candidate to replace the diesel fuels if the need arises. The conversion of triglycerides into methyl or ethyl esters through the transesterification process reduces the molecular weight to one-third that of the triglyceride, reduces the viscosity by a factor of about eight and increases the volatility marginally. Biodiesel has viscosity close to diesel fuels. These esters contain 10 to 11% oxygen by weight, which may encourage more combustion than hydrocarbon-based diesel fuels in an engine. The cetane number of biodiesel is around 50. The use of tertiary fatty amines and amides can be effective in enhancing the ignition quality of the finished diesel fuel without having any negative effect on its cold flow properties. Since the volatility increases marginally, the starting problem persists in cold conditions. Biodiesel has lower volumetric heating values (about 12%) than diesel fuels but has a high cetane number and flash point. The esters have cloud point and pour points that are 15 to 258C higher than those of diesel fuels.

### 2.1.2 Raw Materials

The limitation of available sources for petroleum exploration is a strong motivation to focus on renewable energy. There are few categorize for raw materials to produce biodiesel which is animal fat, vegetable oil and waste cooking oil. Each raw material have advantages and disadvantages.

#### 2.1.2.1 Animal Fat

The fact that is biodiesel can produced from 100 percent animal fats and fulfills the current EN 14214 for fatty acid methyl esters, as long as state-of-the-art process technology is applied.When chemical properties of various feedstock materials for biodiesel production are compared, the main difference between vegetable oils like rapeseed oil and animal fats can be found in the diverse fatty acid composition. While rapeseed oil and soybean oil have a high content of unsaturated fatty acids, mainly oleic acid and linoleic acid, animal fats like tallow or lard have a major content of saturated fatty acids (e.g., palmitic and stearic acid). The increased amount of saturated fatty acids shows a reversed trend for two main fuelproperties. While the oxidationstability of biodiesel derived from animal fats increases, the cold temperature performance decreases with a rising content of saturated fatty acids. Figure 1 shows the dependence of the content of saturated fatty acids in biodiesel – without additives – on its cold filter plugging point (CFPP).

#### 2.1.2.2 Vegetable Oil

Vegetable oil can be divided into two categories, virgin oil and waste vegetable oil. Virgin oil feedstock, rapeseed and soybean oils are most commonly used, soybean oil alone accounting for about ninety percent of all fuel stocks; It also can be obtained from field pennycress and Jatropha other crops such as mustard, flax, sunflower, canola, palm oil, hemp, jatropha, and even algae show promise. The other one is waste vegetable oil (WVO). The advantages of using vegetable oils as fuels are it do not over burden the environment with emissions, vegetable oil’s production requires lesser energy input in production, and simpler processing technology. While the disadvantages of vegetable oil are these are not economically feasible yet and it still need further R&D work for development of on farm processing technology. Many advocates suggest that waste vegetable oil is the best source of oil to produce biodiesel.

#### 2.1.2.3 Waste Cooking Oil

Waste cooking oil is purified from fat of plant or animal origin, which is liquid at room temperature. Proper disposal of used cooking oil is an important waste-management concern. Oil is lighter than water and tends to spread into thin and broad membranes which hinder the oxygenation of water. Because of this a single litre of oil can contaminate as much as 1 million liters of water. Also, oil can congeal on pipes provoking blockages, because of this, cooking oil should never be dumped on the kitchen sing or in the toilet bowl. The proper way to dispose it is to put it in a sealed non-recyclable container and discard it with regular garbage. Better yet, cooking oil can be recycled. It can be used to produce soap and biodiesel.

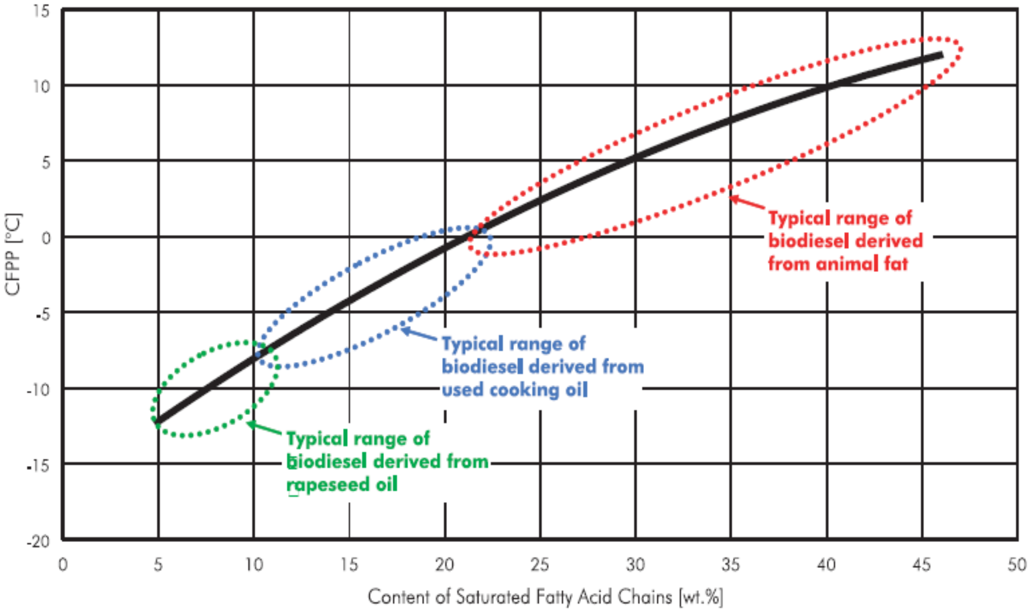


**Figure 2.1** Content of Saturated Fatty Acid Chains

## 2.2 Process

### 2.2.1 Pyrolisis

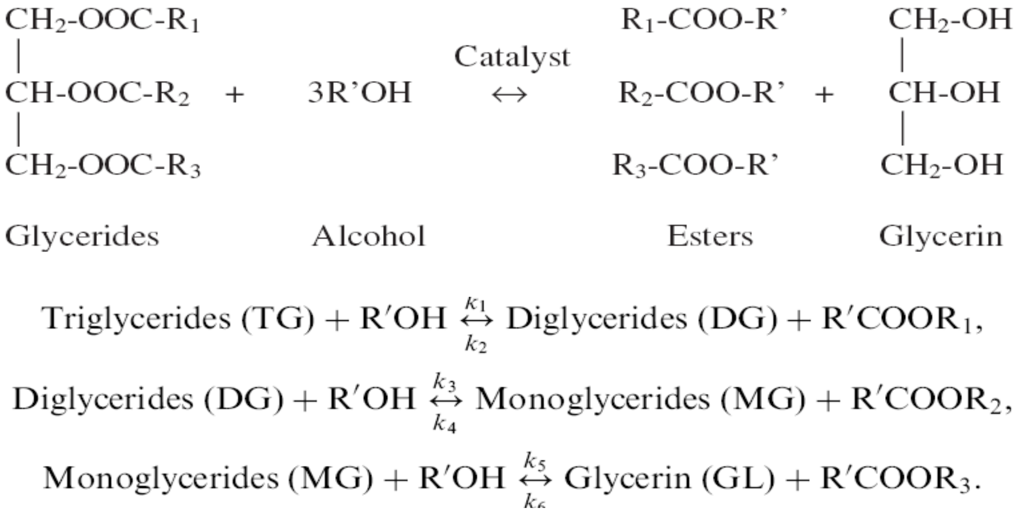
Pyrolysis is the conversion of one substance into another by means of heat or by heat in presence of a catalyst. The paralysed material can be vegetable oils, animal fats, natural fatty acids or methyl esters of fatty acids. The pyrolysis of fats has been investigated for more than 100 years, especially in those areas of the world that lack deposits of petroleum. Many investigators have studied the pyrolysis of triglycerides to obtain products suitable for diesel engine. Thermal decomposition of triglycerides produces alkanes, alkenes, alkadines, aromatics and carboxylic acids [Ma *et al*, 1990]

### 2.2.2 Micro Emulsion

The problem of the high viscosity of vegetable oils can be solving by emulsification with solvents such as methanol, ethanol and 1-butanol. An emulsification is defined as a colloidal equilibrium dispersion of optically isotropic fluid microstructures with dimension generally in the 1–150 nm range, formed spontaneously from two normally immiscible liquids. They can improve spray characteristics by explosive vaporization of the low boiling constituents in the micelles. Short term performance of micro-emulsions of aqueous ethanol in soybean oil was nearly as good as that of no. 2 diesel, in spite of the lower cetane number and energy content [Srivastava *et al*, 2000]

### 2.2.3 Transesterification

Transesterification is the process of exchanging the alkoxy group of an ester compound by another alcohol. The reactions are often catalyzed by an acid or a base. Transesterification is crucial for producing biodiesel from biolipids. Transesterification is the process triglycerides react with an alcohol, generally methanol or ethanol to produce esters and glycerine [Miguel *et al.*, 2001]. A catalyst is added to the reaction to make it possible. The catalyst is usually used to improve the reaction rate and yield. Since the reaction is reversible, excess alcohol is required to shift the equilibrium to the product side. Among the alcohols that can be used in the transesterification process are methanol, ethanol, propanol, butanol and amyl alcohol [Ma *et al*, 1990]. Alkali-catalyzed transesterification is much faster than acid catalyzed transesterification and is most often used commercially [Ma *et al*, 1990].



$$\begin{array}{lcccccc} CH_2\text{-}OOC\text{-}R_1 & & & & R_1\text{-}COO\text{-}R' & & CH_2\text{-}OH \\ | & & & \text{Catalyst} & & & | \\ CH\text{-}OOC\text{-}R_2 & + & 3R'OH & \leftrightarrow & R_2\text{-}COO\text{-}R' & + & CH\text{-}OH \\ | & & & & & & | \\ CH_2\text{-}OOC\text{-}R_3 & & & & R_3\text{-}COO\text{-}R' & & CH_2\text{-}OH \\ \text{Glycerides} & & \text{Alcohol} & & \text{Esters} & & \text{Glycerin} \end{array}$$

$$\text{Triglycerides (TG)} + R'OH \underset{k_2}{\overset{k_1}{\leftrightarrow}} \text{Diglycerides (DG)} + R'COOR_1,$$

$$\text{Diglycerides (DG)} + R'OH \underset{k_4}{\overset{k_3}{\leftrightarrow}} \text{Monoglycerides (MG)} + R'COOR_2,$$

$$\text{Monoglycerides (MG)} + R'OH \underset{k_6}{\overset{k_5}{\leftrightarrow}} \text{Glycerin (GL)} + R'COOR_3.$$

**Figure 2.2** Transesterification Process

R1, R2, R3, R’ represent various alkyl group. The process of transesterification brings about drastic change in viscosity of vegetable oil. The biodiesel thus produced by this process is totally miscible with mineral diesel in any proportion. Biodiesel viscosity comes very close to that of mineral diesel hence no problems in the existing fuel handling system. Flash point of the biodiesel gets lowered after esterification and the cetane number gets improved. Even lower concentrations of biodiesel act as cetane number improver for biodiesel blend. Calorific value of biodiesel is also found to be very close to mineral diesel [Agarwal, 2001].

Diesel engine can perform satisfactory for long run on biodiesel without any hardware modifications. Twenty percent biodiesel is the optimum concentration for biodiesel blend with improved performance. Increase in exhaust temperature however lead to increased NOx emissions from the engine. While short-term tests are almost positive, long-term use of neat vegetable oils or their blend with diesel leads to various engine problems such as, injector coking, ring sticking, injector deposits etc. [Muniyappa *et al*., 1996]. High viscosity, low volatility and a tendency for polymerization in the cylinder are root causes of many problems associated with direct use of these oils as fuels.

The process of transesterification yield vegetable oil ester, which has shown promises as alternative diesel fuel as a result of improved viscosity and volatility. Several researchers investigate the different vegetable oil esters and find esters comparable to mineral diesel [Murayama *et al*,. 2000]. The yield of biodiesel in the process of transesterification is affected by several processparameters/variables.

The most important variables affecting the yield of biodiesel from transesterification are reaction temperature, molar ratio of alcohol and oil, catalys, reaction time, and presence of moisture and free fatty acids (FFA).

## 2.3 Control Process Variables

### 2.3.1 Reaction Temperature

The rate of reaction is strongly influenced by the reaction temperature. However, given enough time, the reaction will proceed to near completion even at room temperature. Generally, the reaction is conducted close to the boiling point of methanol (60–70 °C) at atmospheric pressure. The maximum yield of esters occurs at temperatures ranging from 60 to 80 °C at a molar ratio (alcohol to oil) of 6:1 [Srivastav *et al*., 2000].

Several researchers have studied the effect of temperature on conversion of oils and fats into biodiesel. Freedman et al. studied the transesterification of refined soybean oil with methanol (6:1), 1% NaOH catalyst, at three different temperatures 60, 45 and 32 1C. After 0.1 h, ester yields were 94%, 87% and 64% for 60, 45 and 32 1C, respectively. After 1 h, ester formation was identical for 60 and 45 1C reaction temperature runs and only slightly lower for 32 1C. It shows that temperature clearly influenced the reaction rate and yield of esters and transesterification can proceed satisfactorily at ambient temperatures, if given enough time, in the case of alkaline catalyst

### 2.3.2 Ratio of Alcohol to Vegetable Oil

Another important variable affecting the yield of ester is the molar ratio of alcohol to vegetable oil. The stoichiometry of the transesterification reaction requires 3mole of alcohol per mole of triglyceride to yield 3 moles of fatty esters and 1 mole of glycerol. To shift the transesterification reaction to the right, it is necessary to use either a large excess of alcohol or remove one of the products from the reaction mixture continuously.

The second option is preferred wherever feasible, since in this way, the reaction can be driven towards completion. When 100% excess methanol is used, the reaction rate is at its highest. A molar ratio of 6:1 is normally used in industrial processes to obtain methyl ester yields higher than 98% by weight. Freedman et al. studied the effect of molar ratio (from 1:1 to 6:1) on ester conversion with vegetable oils. Soybean, sunflower, peanut and cottonseed oils behaved similarly and achieved highest conversions (93–98%) at a 6:1 molar ratio. Ratios greater than 6:1 do not increase yield (already 98–99%), however, these interfere with separation of glycerol.

### 2.3.3 Amount of Catalyst

Catalysts are classified as alkali, acid, or enzymes. Alkali-catalyzed transesterification is much faster than acid- catalyzed reaction. However, if a vegetable oil has high free fatty acid and water content, acid catalyzed transesterification reaction is suitable. Partly due to faster esterification and partly because alkaline catalysts are less corrosive to industrial equipment than acidic catalysts, most commercial transesterification reactions are conducted with alkaline catalysts.

Sodium methoxide was found to be more effective than sodium hydroxide. Sodium alkoxides are among the most efficient catalysts used for this purpose, although NaOH, due to its low cost, has attracted its wide use in large scale transesterification. The alkaline catalyst concentrations in the range of 0.5–1% by weight yield 94–99% conversion of vegetable oils into esters. Further increase in catalyst concentration does not increase the conversion and it adds to extra costs because it is necessary to remove the catalyst from the reaction products at the end [Srivastava *et al*,. 2000].

Methanol can quickly react with triglycerides and NaOH is easily dissolved in it. The reaction can be catalyzed by alkalis, acids, or enzymes. The alkalis include NaOH, KOH, carbonates and corresponding sodium and potassium alkoxides such as sodium methoxide, sodium ethoxide, sodium propoxide and sodium butoxide. Sulfuric acid, sulfonic acids and hydrochloric acid are usually used as acid catalysts. Lipases also can be used as biocatalysts.

### 2.3.4 Reaction Time

The conversion rate increases with reaction time. Freedman et al. transesterified peanut, cottonseed, sunflower and soybean oils under the condition of methanol to oil ratio of 6:1, 0.5% sodium methoxide catalyst and 60 °C. An approximate yield of 80% was observed after 1 min for soybean and sunflower oils. After 1 h, the conversions were almost the same for all four oils (93–98%).

### 2.3.5 Reaction Time Moisture and Free Fatty Acid

For an alkali-catalyzed transesterification, the glycerides and alcohol must be substantially anhydrous because water makes the reaction partially change to saponification, which produces soap. The soap lowers the yield of esters and renders the separation of ester and glycerol and water washing difficult. The glycerol is then removed by gravity separation and remaining ester is mixed with hot water for separation of catalyst. Moisture can be removed using silica gel. Ester formation eliminates almost all the problems associated with vegetable oils. Saponification reaction also takes place simultaneously along with transesterification process but soap formation is not a major problem if presence of water is less than 1% [Ma *et al,*. 1999].

Starting materials used for alkali-catalyzed transesterification of triglycerides must meet certain specifications. Freedman *et al.* found that ester yields were significantly reduced if the reactants did not meet these requirements. Sodium hydroxide or sodium methoxide reacted with moisture and carbon dioxide in the air, which diminished their effectiveness. The effects of FFA and water on transesterification of beef tallow with methanol were investigated by Ma and Hanna. The results showed that the water content of beef tallow should be kept below 0.06% w/w and free fatty acid content of beef tallow should be kept below 0.5%, w/w in order to get the best conversion. Water content was a more critical variable in the transesterification process than FFA.

## 2.4 Catalyst

Biodiesel produced by transesterification reactions can be catalyzed with alkali, acid or enzyme. But the former two types have received more attention due to their short reaction time and the low cost compared with the later. Chemical catalyzed processes including alkali and acid ones are more practical, compared with the enzymatic method. Alkali process can achieve high purity and yield of biodiesel product in a short time (30–60 min) [Muniyappa., *et al.*, 1996]

### 2.4 .1 Alkali Catalyst

Several researchers found that alkali-catalyzed transesterificationproceeds much faster than acid-catalyzed transesterification[Dorado *et al*.,2004]. In general, an alkaline catalyst will convert 94–98% yields of vegetable oil into esters when using virgin oil. Sodium hydroxide (NaOH) or potassium hydroxide (KOH) should be used with methanol or ethanol as well as any kind of oils, refine, crude or frying for a basic catalyst. For this process it is better to produce the alcoxy before the reaction to obtain a betterglobal efficiency.

The alcohol–oil molar ratio that should be used varies from N ¼ 1:1–6:1. However, N ¼ 6:1 is the most used ratio giving an important conversion for the alkali catalyst without using a great amount of alcohol.

According to the Zhang literature's, one limitation to the alkalicatalyzed process is its sensitivity to the purity of reactants; especially the alkali-catalyzed system is very sensitive to both water and free fatty acids. The presence of water, under alkaline conditions, may cause ester saponification. Also, free fatty acids can react with an alkali catalyst to produce soaps and water. The resulting soaps can cause the formation of emulsions. These circumstances give rise to a consumption of the catalyst and, in addition, cause difficulties in the recuperation and purification of the biodiesel.

The best results were reached with a concentration of 1.0%. For higher values the yields were lower. This fact, as has it been indicated, seems to be related to the free acidity of the oil. When there is a large free fatty acid content, the addition of more potassium hydroxide, or any other alkaline catalyst.

### 2.4.2 Acid Catalyst

Using acid catalyst is the second conventional way of making the biodiesel production. This process is using the triglycerides with alcohol. The most commonly used is sulphuric acid [Freedman *et al.*, 1984] and some authors prefer sulfonic acid [Stern R *et al.*, 1990]. This type of catalyst gives very high yield in esters but for the reaction it is very slow, where almost more than one day to complete. In the alkali reaction, in excess of alcohol condition, then we get better conversion of triglycerides, but to covering glycerol become more difficult.

This is the reason why optimal relation between alcohol and raw material has should be determined experimentally considering each process as a new problem. 30:1 is the molar ratio for possible operation condition. The type of alcohol, as well as the oils, is the same as the one that can be used in alkali catalyst reaction. The amount of catalyst supposed to be added to the reactor varies from 0.5 to 1 mol%. The typical value is 1%, but Zhang *et al*, 2000 research is using 3.5 mol%. The temperature range varies from 55 to 80 °C. The acid transesterification is a great way to make biodiesel if the sample has relatively high free fatty acid content. In general, a 1 mol% of sulfuric acid is a good amount for a final conversion of 99% in a time around 50 h.

## 2.5 Heterogeneous and Homogeneous Catalyst

Homogeneous alkaline catalysts in the transesterification of such types of fats and oils cannot directly be used due to the presence of large amounts of free fatty acids (FFA>0.5% w/w) and previous purification stages are necessary (Jeromin *et al*, 1987; Frangrui *et al.*, 1990; Ramadhas *et al*. 2002). A useful alternative could be the use of catalysts that have not the drawbacks of the homogeneous alkaline catalysts, as Di Serio *et al.* (2005) have recently shown using homogeneous Lewis acid catalysts.

Moreover, the cost of biodiesel could certainly be lowered by using a heterogeneous catalyst instead of a homogeneous one, resulting in a higher quality esters and glycerol that are more easily separated. Glycerol doesn’t need, in this case, expensive refining operations (Bournay *et al*, 2005).

## 2.6 Response Surface Methodology

Response surface methodology (RSM) explores the relationships between several explanatory variables and one or more response variables. The main idea of RSM is to use a set of designed experiments to obtain an optimal response. Box and Wilson, 1951 acknowledge that this model is only an approximation, but use it because such a model is easy to estimate and apply, even when little is known about the process. It also used to find the responses of two variables of these coefficients by including a large number of trials in each and combinations of them, and using some sort of interpolation to find potentially better intermediate values between them. But since experimental runs often cost a lot of time and money, and also difficult to pinpoint the ideal coefficients, so RSM are better to used to find those values with minimal runs of experiment.

Response surface methodology has been widely used to evaluate and understand this interaction (Ambati and Ayyanna, 2001; Hounjg *et al.*, 1989). The response surface methodology, which includes factorial design and regression analysis, can build models to evaluate the effective factors and study their interaction and select optimum condition limited number of experiments (Chauhan and Gupta, 2004)

# CHAPTER 3

# MATERIALS AND METHOD

## 3.1 Introduction

There are many previous research were been done to produce biodiesel, some of them are production of biodiesel from Jathopra oil, Jojoba oil wax, Madhuca Indica and waste cooking oil. The most common method is by transesterification. In this process, triglycerides the main components will react with an alcohol to produce fatty acid mono-alkyl esters and glycerol. The reaction will refer as methanolysis when methanol is used. Methanol commonly been used widely because have low price compared to other alcohol. Methanolysis reaction requires 3 mol of methanol and 1 mol of triglyceride to poduce 3 mol of fatty acid methyl ester and 1 mol of glycerol in the stoichiometry. There consist three consecutive reversible reactions with intermediate formation of diglycerides and monoglyceides in the reaction. After the reaction, the glycerol is separated by settling or centrifuging and purified using hot water. The product is used by various sectors like pharmaceutical, cosmetic and food industries.

There are three basic routes to produce biodiesel from oils and fats which is base catalyzed transesterification of the oil, direct acid catalyzed transesterification of the oil and conversion of the oil to its fatty acids and then to biodiesel.

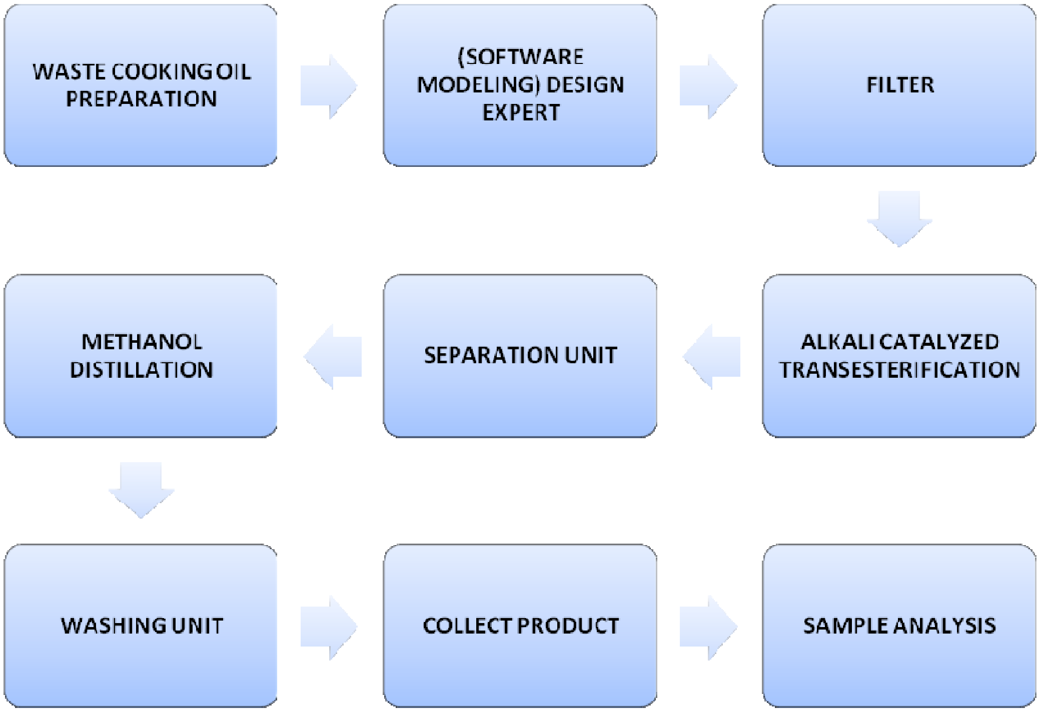


**Figure 3.1** Biodiesel production process

## 3.2 Materials

### 3.2.1 Raw Material

In this experiment, waste cooking oil will be used as the main raw materials for production of biodiesel. Waste cooking oil is collected from food manufacturing industries, catering premises like restaurant, rehabilitations centre, café and food stall. Refined vegetable oil is the best start material in producing biodiesel, but waste cooking oil is still valuable because the price is cheaper compare to vegetable oil.

### 3.2.2 Chemical Material

Potassium hydroxide, KOH will be use as an alkali catalyst whereas methanol will be use as the solvent. All these material will be supplied by Faculty of Chemical Engineering and Natural Resources (FKKSA).

## 3.3 Equipment

The experiment to produce biodiesel will be run in a laboratory scale. The equipment that use in this experiment are:

i. Centrifuges: to filter solid compound in waste cooking oil.

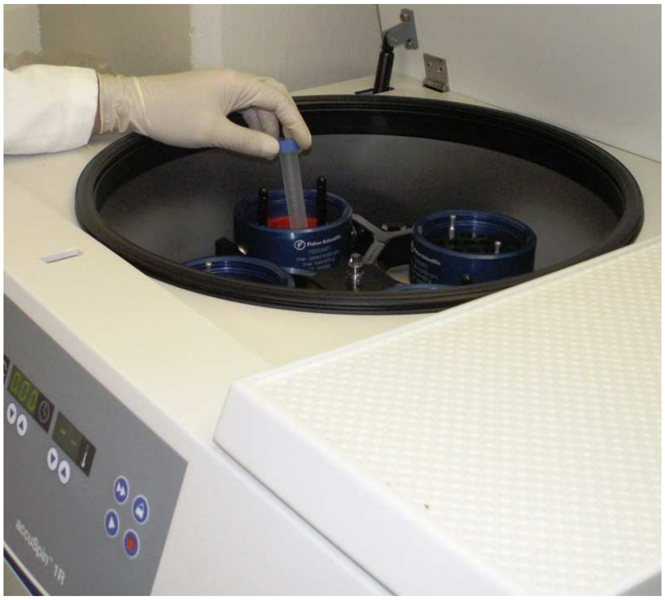


**Figure 3.2** Centrifuge

ii. Hot Plate: to remove water using evaporation process by pre-heating the waste cooking oil.

**Figure 3.3** Hot Plate

iii. Shaking water bath with stirrer: used during transesterification process.

**Figure 3.4** Shaking Water Bath

iv. Rotary evaporator: to recover methanol after transesterification process.

**Figure 3.5** Rotary Evaporator

## 3.4 Experiment Procedure

### 3.4.1 Modeling of Process Using Design Expert

Design Expert will be use to calculate some of the data. It will help in finding the relationship between the investigated parameters which is catalyst concentration and reaction time. Using this software we manage to save the time to run experiment.

### 3.4.2 Pretreatment

The waste cooking oil (WCO) will be filtered using centrifuges to remove impurities solid compounds. Then the filtered waste cooking oil is heated around 80°C to 90°C to remove water by evaporation process. The oil is left to cool before the next step takes place.

### 3.4.3 Transesterification Process

The transesterification process of waste cooking oil will be done in shaking water bath and was connected to mechanical stirrer. In this step, 200 gram of waste cooking oil was poured into a 500 mL beaker and will be heat to 65˚C temperature. In duration for waiting the waste cooking oil to be heated to 65˚C, solvent which is methanol in 38.5 gram and the catalyst, potassium hydroxide (KOH) for a certain weight percentage will be added into the beaker and was stirred for 5 minutes. The reaction will begin right after addition of waste cooking oil with solvent and catalyst. The beaker will placed in shaking water bath depend on certain reaction time. The mixture was stirred at constant rate at 900 rpm during the reaction. During the reaction the oil will separate with glycerine. Placed out the beaker from shaking water bath and leave about 12 hours to let the glycerine settle down.

### 3.4.4 Methanol Recovery and Washing

The upper layer after transesterification process was the FAME, fatty acid methyl esters (crude biodiesel) with lighter colour while the bottom layer was glycerine. FAME and glycerine was separate before methanol recovery process. The FAME was transferred to a rotational evaporator to recover excess methanol under vacuum at 50°C from the transesterification process. FAME at 80°C was washed by ratio 1: 1 with water by slowly pouring hot water to remove the side product which is glycerin, methanol, and catalyst. After this step, the water was drained and these washing steps were repeated once again to get pure product (biodiesel). Finally, heated the biodiesel at 90°C under vacuum with a rotational evaporator to dry up residual water and the biodiesel was filtered using filter paper to remove any remaining solid.

## 3.5 Analysis

In this experiment, the samples were analyze by three parameters which are biodiesel yield, thin layer chromatography (TLC) conversion, and moisture content. Every sample will give different result based on different condition. This sample will be analysing to find the optimum condition of the reaction. Finally, the sample which is produced based on optimum condition will be tested on several parameters to ensure it meet the standard requirement of ASTM D6751 for biodiesel B100 (pure biodiesel).

### 3.5.1 Biodiesel yield

In this experiment we will convert free fatty acids to biodiesel using single steps catalyst, which is alkali, potassium hydroxide (KOH). The biodiesel yields show the percentage of biodiesel conversion from waste cooking oil. The biodiesel yield can be calculated using this equation:

$$\text{Yield}(\%) = \frac{\text{Mf} - \text{Mi}}{\text{Mi}}(100\%)$$

Where:

Yield = conversion of biodiesel

Mi = initial mass

Mf = final mass

### 3.5.2 Thin Layer Chromatography (TLC)

Thin layer chromatography (TLC) is a chromatography technique used to determine the presence of triglycerides and free fatty acids. It involves a plate surface (stationary phase) consisting of a thin layer of adsorbent material, usually silica gel, aluminium oxide, or cellulose immobilised onto a flat, inert carrier sheet. A liquid phase consisting of the solution to be separated is then dissolved in an appropriate solvent and is drawn up the plate via capillary action. The pellet that used is iodine pellet and the solvent is chloroform and hexane.

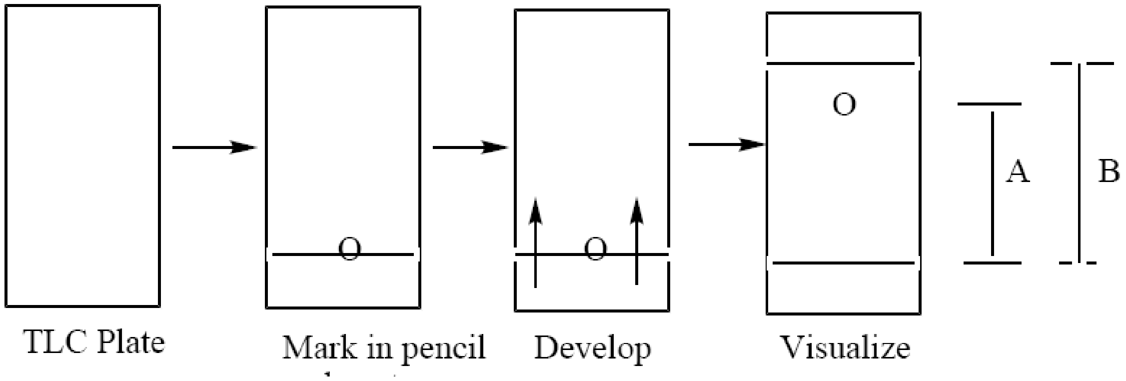


**Figure 3.6** TLC analysis

The procedures in TLC analysis first draw a line lightly using a pencil about 1cm from plate base. The silica adsorbent can not touch with fingers, because it will affect the TLC result. The plates must be hold at the sides. Biodiesel sample was drop for a small spot on a plate at the line, one centimetre from the base. Then dipped the plates into a solvent and placed in a sealed container. The plate for the solvent will moves up by capillary action and meets the sample mixture, which is dissolved and is carried up the plate by the solvent. The thin layer chromatography percentage can be calculated using this equation:

$$\mathbf{Rf = (A / B) \times 100\%}$$

Where:

Rf= TLC conversion

A= distance of the higher spot from base

B= distance of the plate

### 3.5.3 Moisture Content

This step used to determine water and sediment that sensitive to free water. It uses a centrifuge to separate small water droplets and particles to be sure they do not exceed 0.05% (500ppm).

## 3.6 Response Surface Methodology

The program Design Expert (State-Ease Inc, Statistic Made Easy, Minneapolis, MN, USA,Version 6.0.4) was applied for the response surface methodology. The cental composite design (CCD) was chosen in the optimization process due to its ability in providing factorial analysis.

Bases on the best result of one at a time approach, the selected factors were further evaluated for their interactive behaviours and studied using a statistical approach, central composite design (CCD). All the variables were taken at a central coded value considered as zero. The low and the high levels of variables were investigated. The levels of these variables were decided according to the result determined by two factors at a time method and catalyst concentration.

| Factors | Low level | High level |
|---|---|---|
| Reaction time (min) | 30 | 90 |
| Catalyst concentration (%) | 0.5 | 1.5 |

.

# CHAPTER 4

# RESULT

## 4.0 Result

In this experiment, the waste cooking oil is used as a raw material with methanol as a solvent and potassium hydroxide (KOH) as a catalyst. There are several parameters that will affect the yield and the conversion of biodiesel, but the parameters that being monitored in this experiment are catalyst concentration and reaction time. The experiment has been done in the condition 65˚C for reaction temperature, 30minutes to 90minutes for reaction time, 0.5% to 2% catalyst concentration, 1:6 oil to methanol ratio and 900 rpm mixing stirrer.

## 4.1 Optimization of Yield using Response Surface Methodology (RSM)

Optimization of yield percentage was carried out using response surface methodology (RSM). The condition involve are reaction time and catalyst concentration. By using central composite design (CCD), the experiments with different combination of reaction time and catalyst concentration were performed. Experiments arranged by Design Expert Software are listed in Table 4.1.

The results were analysed using analysis of variance (ANOVA) as appropriate to the experimental design used. The regression Final Equation above was obtained from analysis of variance and all term regardless of their significance are included in the following equation:

**Design Summary**

| | |
|---|---|
| **Study Type** | Response Surface |
| **Initial Design** | Central Composite |
| **Design Model** | Quadratic |
| **Experiments** | 13 |
| **Blocks** | No Blocks |

**Reaction temperature = 65˚C**

| **std** | **Run** | **Factor 1 Time,min** | **Factor 2 Cat conc** | **Res 1 Yield (%)** |
|---|---|---|---|---|
| 8 | 1 | 60 | 1.5 | 87.0 |
| 9 | 2 | 60 | 1.0 | 88.6 |
| 12 | 3 | 60 | 1.0 | 86.6 |
| 7 | 4 | 60 | 0.5 | 62.3 |
| 13 | 5 | 60 | 1.0 | 85.8 |
| 6 | 6 | 90 | 1.0 | 88.5 |
| 10 | 7 | 60 | 1.0 | 89.5 |
| 4 | 8 | 90 | 1.5 | 85.8 |
| 1 | 9 | 30 | 0.5 | 59.1 |
| 11 | 10 | 60 | 1.0 | 89.3 |
| 3 | 11 | 30 | 1.5 | 88.0 |
| 2 | 12 | 90 | 0.5 | 64.7 |
| 5 | 13 | 30 | 1.0 | 89.9 |

**Table 4.1:** Yield percentage result

**Final Equation in Terms of Coded Factors:**

$$\text{Yield} = 88.63 + 0.33A + 12.45B - 14.15B^2 - 1.95\,AB$$

Where:

A = reaction time

B = catalyst concentration

| Source | Sum of Squares | DF | Mean Square | F Value | Prob > F | |
|---|---|---|---|---|---|---|
| **Model** | 1592.33 | 4 | 398.08 | 328.62 | < 0.0001 | significant |
| **A** | 0.67 | 1 | 0.67 | 0.55 | 0.4794 | |
| **B** | 930.01 | 1 | 930.01 | 767.74 | < 0.0001 | |
| **$B^2$** | 646.44 | 1 | 646.44 | 533.64 | < 0.0001 | |
| **AB** | 15.21 | 1 | 15.21 | 12.56 | 0.0076 | |
| **Residual** | 9.69 | 8 | 1.21 | | | |
| **Lack of Fit** | 4.23 | 4 | 1.06 | 0.77 | 0.5946 | not significant |
| **Pure Error** | 5.46 | 4 | 1.36 | | | |
| **Cor Total** | 1602.02 | 12 | | | | |

**Table 4.2:** ANOVA

Table 4.2 shows the ANOVA and regression analysis for the production of recombinant biodiesel. Values of P-value (Prob > F) less than 0.05 indicate model terms are significant. The precision of a model can be checked by the determination coefficient ($R^2$) and correlation coefficient (R). The determination coefficient ($R^2$) implies that the sample variation of 98.03% for biodiesel production was attributed to the independent variables tested. A regression model having a $R^2$ value higher than 0.9 is considered to have a very high correlation (Haaland, 1989). The value of R (correlation coefficient ) closer to 1 indicates the better correlation between the experimental and predicted values.

The Model F-value of 328.62 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant.

In this case B, $B^2$, AB is significant model terms, where B is linear effect, $B^2$ is squared effect and AB is interaction effect. Values greater than 0.1000 indicate the model terms are not significant. A is the coded value of reaction time and B is the coded value of catalyst concentration.

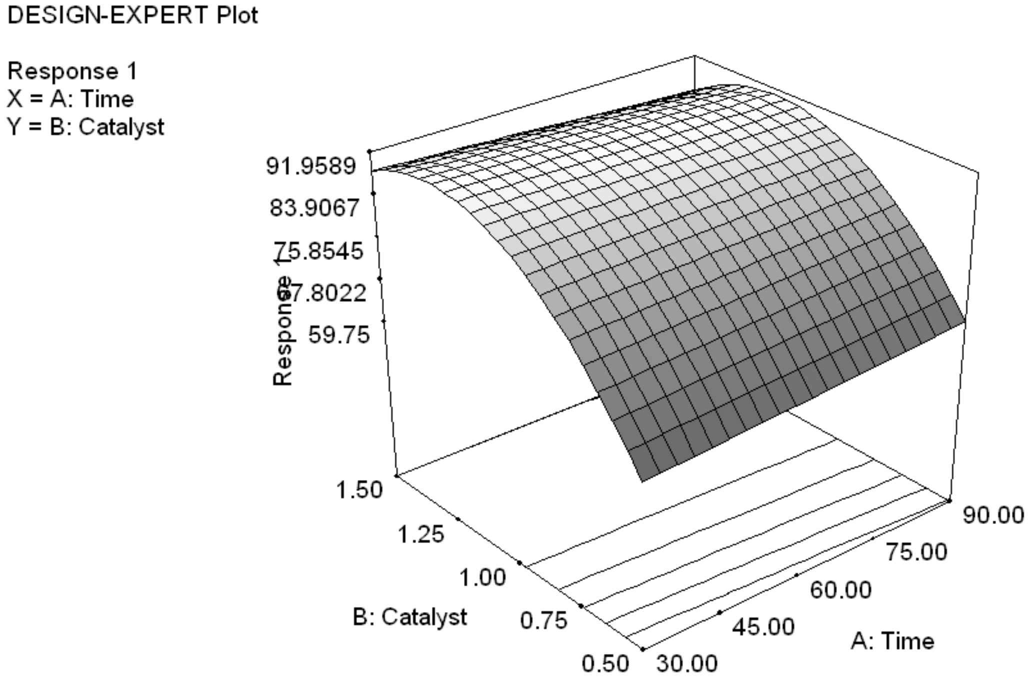


**Figure 4.1:** Interaction graph between yield with catalyst and time

From Figure 4.1 we can indicates that catalyst concentration give a major influence for yield percentage in producing biodiesel. The higher catalyst concentration will give higher value of yield percentage, but based to the graph the optimum catalyst concentration value for get higher yield in the range 1.3 - 1.4% can be seen. While reaction time give a small impact to the yield. The lower reaction time will result smaller yield percentage.

| **Name** | **Goal** | **Lower Limit** | **Upper Limit** |
|---|---|---|---|
| **Time (min)** | **is in range** | **30** | **90** |
| **Catalyst (%)** | **is in range** | **0.5** | **1.5** |
| **Response 1 (%)** | **maximize** | **59.1** | **89.9** |

| **Number** | **Time** | **Catalyst** | **Response 1** | **Desirability** | |
|---|---|---|---|---|---|
| 1 | 73.38 | 1.33 | 90.3185 | 1.000 | Selected |
| 2 | 43.37 | 1.08 | 90.2913 | 1.000 | |
| 3 | 85.29 | 1.30 | 90.3447 | 1.000 | |

**Table 4.3:** Optimum result analysis from RSM

Table 4.4 indicated the optimum condition to get higher value of yield percentage. The suggestion from RSM is condition 1 which the reaction time is 73.38min and catalyst concentration is 1.33%. The predicted yield is about 90.3185% yield. The other value also accepted as an optimum condition because has same desirability. Based on these result we can conclude that catalyst concentration give major influence, while reaction time only give a little impact for yield percentage. A continuation experiment has been done to approve the predicted optimum result from RSM data which got 92.3% of yield percentage.

### 4.2 Optimization of catalyst concentration using Response Surface Methodology (RSM)

Optimization of yield percentage was carried out using response surface methodology (RSM). The condition involve are reaction time and catalyst concentration. By using central composite design (CCD), the experiments with different combination of reaction time and catalyst concentration were performed. Experiments arranged by Design Expert Software are listed in Table 4.1.

The results were analysed using analysis of variance (ANOVA) as appropriate to the experimental design used. The regression Final Equation above was obtained from analysis of variance and all term regardless of their significance are included in the following equation:

**Design summary**

| | |
|---|---|
| **Study Type** | Response Surface |
| **Initial Design** | Central Composite |
| **Design Model** | Quadratic |
| **Experiments** | 13 |
| **Blocks** | No Blocks |

**Reaction temperature = 65˚C**

| std | Run | Factor 1 Time,min | Factor 2 Cat conc | Res 2 TLC |
|---|---|---|---|---|
| 8 | 1 | 60 | 1.5 | 79.0 |
| 9 | 2 | 60 | 1.0 | 80.0 |
| 12 | 3 | 60 | 1.0 | 77.0 |
| 7 | 4 | 60 | 0.5 | 80.0 |
| 13 | 5 | 60 | 1.0 | 79.0 |
| 6 | 6 | 90 | 1.0 | 77.8 |
| 10 | 7 | 60 | 1.0 | 78.2 |
| 4 | 8 | 90 | 1.5 | 76.5 |
| 1 | 9 | 30 | 0.5 | 76.0 |
| 11 | 10 | 60 | 1.0 | 77.0 |
| 3 | 11 | 30 | 1.5 | 77.8 |
| 2 | 12 | 90 | 0.5 | 82.2 |
| 5 | 13 | 30 | 1.0 | 78.9 |

**Table 4.4:** TLC percentage result

**Final Equation in Terms of Coded Factors:**

TLC = 78.11 + 0.63 A – 1.15 B – 1.87 AB

Where:

A = reaction time

B = catalyst concentration

| Source | Sum of Squares | Degree of Freedom | Mean Square | F Value | Prob > F | |
|---|---|---|---|---|---|---|
| **Model** | 24.40 | 3 | 8.13 | 8.86 | 0.0047 | Significant |
| **A** | 2.41 | 1 | 2.41 | 2.62 | 0.1399 | |
| **B** | 7.94 | 1 | 7.94 | 8.64 | 0.0165 | |
| **AB** | 14.06 | 1 | 14.06 | 15.31 | 0.0035 | |
| **Residual** | 8.27 | 9 | 0.92 | | | |
| **Lack of Fit** | 5.35 | 5 | 1.07 | 1.47 | 0.3651 | not significant |
| **Pure Error** | 2.91 | 4 | 0.73 | | | |
| **Correlation Total** | 32.67 | 12 | | | | |

**Table 4.5:** ANOVA

Table 4.6 shows the ANOVA and regression analysis for the production of recombinant biodiesel. The precision of a model can be checked by the determination coefficient ($R^2$) and correlation coefficient (R). The determination coefficient ($R^2$) implies that the sample variation of 74.7% for biodiesel production was attributed to the independent variables tested. A regression model having a $R^2$ value higher than 0.7 is considered to have a accepted correlation (Haaland, 1989). The value of R (correlation coefficient ) closer to 1 indicates the better correlation between the experimental and predicted values.

The Model F-value of 8.86 implies the model is significant. Values of P-value (Prob > F) less than 0.05 indicate model terms are significant. There is only a 0.47% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant.

In this case B and AB are significant model terms, where B is linear effect and AB is interaction effect. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

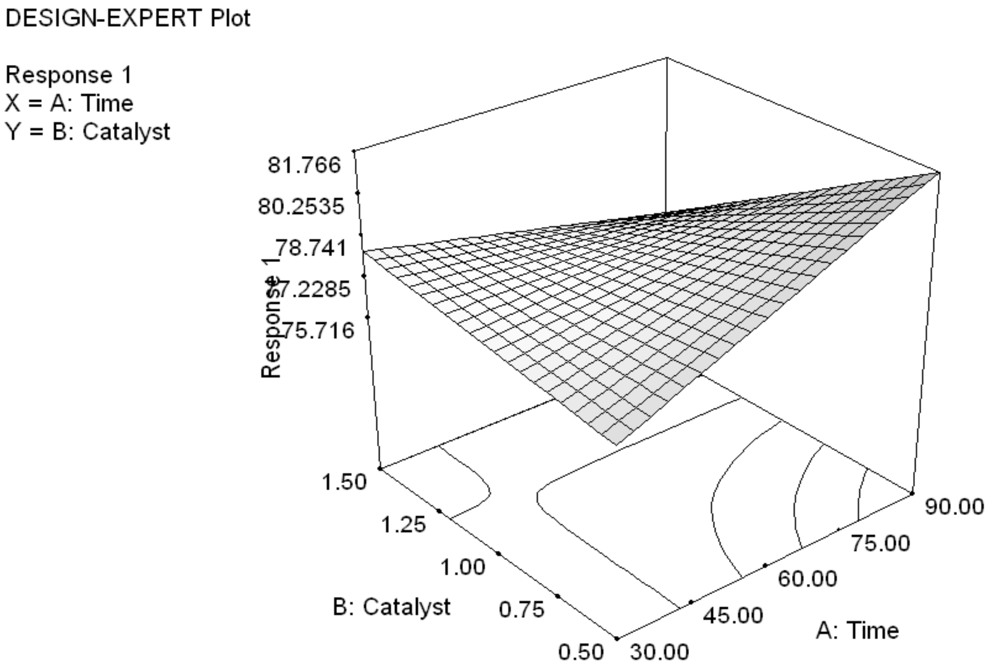


**Figure 4.2** Interaction graph between TLC with catalyst and time

From Figure 4.2 we can indicates that reaction time give a major influence for thin layer chromatography (TLC) percentage in producing biodiesel. The higher reaction time will give higher value of TLC percentage, but based to the graph we could see the optimum reaction time value for get higher TLC in the range 90min. While catalyst concentration give a small impact to the yield.

| Name | Goal | Lower Limit | Upper Limit |
|---|---|---|---|
| Time (min) | is in range | 30 | 90 |
| Catalyst (%) | is in range | 0.5 | 1.5 |
| Response 1 (%) | maximize | 76.0 | 82.2 |

| Number | Time | Catalyst | Response 1 | Desirability | |
|---|---|---|---|---|---|
| 1 | 90.00 | 0.50 | 81.766 | 0.930 | Selected |
| 2 | 30.00 | 1.50 | 78.1994 | 0.355 | |

**Table 4.6:** Optimum result analysis from RSM

Table 4.8 indicated the optimum condition to get higher value of thin layer chromatography (TLC) percentage. The suggestion from RSM is condition 1 which the reaction time is 90.0 min and catalyst concentration is 0.50%. The result that is predicted is 81.766% TLC percentage. The other value also accepted as an optimum condition, but these condition get lower percentage compare to condition 1. Based on these result we can conclude that reaction time give major influence, while catalyst concentration give a little impact for yield percentage. A continuation experiment has been done to approve the predicted optimum result from RSM data which got 80.8% of TLC percentage.

### 4.3 Optimization of moisture content using Response Surface Methodology (RSM)

**Reaction temperature = 65°C**

| std | Run | Factor 1 Time,min | Factor 2 Cat conc | Res 3 Mois cont |
|---|---|---|---|---|
| 8 | 1 | 60 | 1.5 | 0.15 |
| 9 | 2 | 60 | 1.0 | 0.19 |
| 12 | 3 | 60 | 1.0 | 0.18 |
| 7 | 4 | 60 | 0.5 | 0.15 |
| 13 | 5 | 60 | 1.0 | 0.19 |
| 6 | 6 | 90 | 1.0 | 0.19 |
| 10 | 7 | 60 | 1.0 | 0.18 |
| 4 | 8 | 90 | 1.5 | 0.08 |
| 1 | 9 | 30 | 0.5 | 0.13 |
| 11 | 10 | 60 | 1.0 | 0.22 |
| 3 | 11 | 30 | 1.5 | 0.14 |
| 2 | 12 | 90 | 0.5 | 0.20 |
| 5 | 13 | 30 | 1.0 | 0.10 |

**Table 4.7:** Moisture content result

Analysis for moisture content cannot be run because there are several factors that influence the value of moisture content that make the value cannot be use and the result not accurate. The main factor is environment error. The experiment has been run in the clean room about 20°C temperature. These make humidity in the clean room high and affect the value for moisture content.

The other factor is high content of water in waste cooking oil. These make the value of moisture content is high and cannot achieve the standard of biodiesel which should be less than 0.05% content of moisture content. The result show the value of moisture content is higher than 0.05% and cannot be accepted.

Sensitivity of equipment also contributes for the error result which it sensitive to the cold environment. The last factor is the storage of biodiesel sample. Because of the lack of apparatus, the sample cannot be store impermeable condition. The sample has been exposed to cold condition and not 100% air proved. This also contribute to higher value of moisture content.

# CHAPTER 5

# CONCLUSION & RECOMMENDATION

## 5.1 Conclusion

A response surface method (RSM) design has been applied to get optimum condition of the synthesis process of fatty acid methyl ester from waste cooking oil using potassium hydroxide (KOH) as the catalyst. This design procedure has been setting to determine the maximum biodiesel purity (tlc) and yield.

From these equations, it is possible to accurately predict the operating conditions to obtain each condition of biodiesel purity and yield based on reaction time and catalyst concentration without doing the experiment. The study of the factors affecting the biodiesel purity shows that, within catalyst concentration range considered between 0.5 to 4%, the most important factor is the initial catalyst concentration. The ratio of methanol and waste cooking oil molar ratio and the temperature are also significant. All of them have a positive effect on the biodiesel purity and therefore the biodiesel purity increases when the values of these variables increase.

Besides than the factor of catalyst concentration, the other important variable is reaction temperature. Both factors have a negative influence on biodiesel yield and therefore, an increasing in these variables produces a decrease in biodiesel yield. But between these two variables, the major variables that effecting the yield and purity is catalyst concentration.

After completed the experiment, the biodiesel sample must be storage in an airproof and dry place. This is important to prevent it from expose to air and water. If not storage in suitable place, the biodiesel sample may have high moisture content, but for the safety factor the biodiesel is corrosive and need to storage in glass container.

## 5.2 Recommendation

During the experimental work, there a lot action can be done to enhance the quality of biodiesel production. The first recommendation is check for FFA content in the waste cooking oil. This step is to get precise result for each sample. The other suggestion is to store the waste cooking oil at room temperature condition, to preventing the oil from solidifies. Other proposal is used the same equipment in every experiment to minimize the error and getting accurate result. The last suggestion to store the biodiesel sample at air prove place to prevent other element contaminate the sample that will intrude the value such as moisture content.

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## APPENDICE

Profile Experiment Data:

For yield result at 65°C reaction temperature:

| std | Run | Factor 1 Time,min | Factor 2 Cat conc | Res 1 Yield (%) |
|---|---|---|---|---|
| 8 | 1 | 60 | 1.5 | 87.0 |
| 9 | 2 | 60 | 1.0 | 88.6 |
| 12 | 3 | 60 | 1.0 | 86.6 |
| 7 | 4 | 60 | 0.5 | 62.3 |
| 13 | 5 | 60 | 1.0 | 85.8 |
| 6 | 6 | 90 | 1.0 | 88.5 |
| 10 | 7 | 60 | 1.0 | 89.5 |
| 4 | 8 | 90 | 1.5 | 85.8 |
| 1 | 9 | 30 | 0.5 | 59.1 |
| 11 | 10 | 60 | 1.0 | 89.3 |
| 3 | 11 | 30 | 1.5 | 88.0 |
| 2 | 12 | 90 | 0.5 | 64.7 |
| 5 | 13 | 30 | 1.0 | 89.9 |

For TLC result at 65˚C reaction temperature:

| **std** | **Run** | **Factor 1 Time,min** | **Factor 2 Cat conc** | **Res 2 TLC** |
|---|---|---|---|---|
| 8 | 1 | 60 | 1.5 | 79.0 |
| 9 | 2 | 60 | 1.0 | 80.0 |
| 12 | 3 | 60 | 1.0 | 77.0 |
| 7 | 4 | 60 | 0.5 | 80.0 |
| 13 | 5 | 60 | 1.0 | 79.0 |
| 6 | 6 | 90 | 1.0 | 77.8 |
| 10 | 7 | 60 | 1.0 | 78.2 |
| 4 | 8 | 90 | 1.5 | 76.5 |
| 1 | 9 | 30 | 0.5 | 76.0 |
| 11 | 10 | 60 | 1.0 | 77.0 |
| 3 | 11 | 30 | 1.5 | 77.8 |
| 2 | 12 | 90 | 0.5 | 82.2 |
| 5 | 13 | 30 | 1.0 | 78.9 |

For moisture content result at 65˚C reaction temperature:

| **std** | **Run** | **Factor 1 Time,min** | **Factor 2 Cat conc** | **Res 3 Mois cont** |
|---|---|---|---|---|
| 8 | 1 | 60 | 1.5 | 0.15 |
| 9 | 2 | 60 | 1.0 | 0.19 |
| 12 | 3 | 60 | 1.0 | 0.18 |
| 7 | 4 | 60 | 0.5 | 0.15 |
| 13 | 5 | 60 | 1.0 | 0.19 |
| 6 | 6 | 90 | 1.0 | 0.19 |
| 10 | 7 | 60 | 1.0 | 0.18 |
| 4 | 8 | 90 | 1.5 | 0.08 |
| 1 | 9 | 30 | 0.5 | 0.13 |
| 11 | 10 | 60 | 1.0 | 0.22 |
| 3 | 11 | 30 | 1.5 | 0.14 |
| 2 | 12 | 90 | 0.5 | 0.20 |
| 5 | 13 | 30 | 1.0 | 0.10 |