Optimization of Biodiesel Production from Castor Oil by Transesterification Process

by

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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

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MAY 2014

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.

_____

(NURUN NAZAHAH BINTI MOHD MAZWIL)

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ABSTRACT

The scarcity of fossil fuels, environmental pollution and food crisis are the world's major issues in current era. Biodiesel is an alternative to fuel, is environmental friendly and biodegradable and can be produced from both edible and non-edible oils. In this work, a non-edible oil, castor oil is used for the production of biodiesel.The biodiesel production is a two-step process, the first step is acid esterification to reduce the free fatty acid (FFA) amount in the oil and the second step is base transesterification to convert the oil completely to biodiesel. Response Surface Methodology is used to optimize the conditions for biodiesel production from castor oil by using a homogeneous catalyst, potassium hydroxide through transesterification process. A quadratic polynomial equation is obtained for biodiesel yield. The studied variables are oil to methanol ratio, catalyst concentration, temperature and time. Results from the optimization are that oil to methanol ratio is 9, 0.5% catalyst, 45^{o}C temperature and 90 minutes. This gives a yield of 83.57% of biodiesel. These parameters is then used for conducting a transesterification process with heterogeneous catalyst, sodium titanate. The results between homogeneous and heterogeneous catalyst is the compared by comparing their density, viscosity and others. The biodiesel's conversion and its composition are also determined by using the Gas Chromatography Flame Ionization Detector (GC FID) unit.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Since the 20th century, fossil fuels such as oil, coal and natural gas supply a major energy demand to the world. However, it has become a problem to the world by the limitation of resources of fossil fuels for the future. The limited number of reserves are already at high production rate and even so, the consumption of these resources are faster than its production. Besides, the consumption of fossil fuels are also contributes to greenhouse gases, global warming, air pollution, incomplete combustion of hydrogen, carbon and other particulate matter. People are opening their minds towards other renewable alternatives for energy conumption which are more sustainable, green and eco-friendly (Ahmad et al., 2014). These alternatives includes the use of oils of plants and animal fats to produce alternative fuel called biodiesel. The biodiesel is biodegradable, non-toxic and it has low emission profiles (Meher et al., 2006).

Biodiesel is produced through the transesterification of triglycerides in the oil and methanol to form methyl esters in the presence of a catalyst. The purpose of this study is to optimize the production of biodiesel from non-edible castor oil with homogeneous and heterogeneous catalyst. The most common practice is to produce the biodiesel from homogeneous catalyst. Unlike the homogeneous catalyst, heterogeneous catalyst can save more cost since it can be recovered and reused after the biodiesel production. It is also efficient in promoting the biodiesel production process in which will ease the separation of biodiesel from its by-products and does not require any expensive separation process or purification (Di Serio et al., 2007).

The comparison of these two catalyst will be based on the optimized parameters in which are determined in this project. The parameters to be

optimized are the feed oil to alcohol ratio, catalyst amount, time taken for production and the heating temperature. Response Surface Methodology (RSM) is used for the parametric optimization. These parameters are then consistently used for the study of biodiesel production process.

1.2 Problem Statement

Most of the research done on biodiesel production optimization is based on changing one separate parameter at a time (COST). However, the biodiesel production reaction is not influenced by only one parameter but is influenced by more than one simultaneously (Kılıç et al., 2013). Thus, in this project, the interactions between parameters are studied.

The conventionally common catalyst used for biodiesel production, homogeneous catalyst needs detailed separation process, washings and cannot be reused after one process of production. Hence, this project also analyze the effect of homogeneous and heterogeneous catalyst on the biodiesel product and to study the potential of replacing the homogeneous catalyst to heterogeneous catalyst for the biodiesel production (Semwal et al., 2011).

1.3 Objective of Study

The objective of this project is to optimize the biodiesel production using castor oil. The experiment is conducted repeatedly by using diiferent parameter values of oil to methanol ratio, catalyst concentration, temperature and time. The

This project will also study the effect of homogeneous and heterogeneous catalyst on the production of biodiesel.

1.4 Scope of Study

To achieve the objective, there are four scopes that have been identified:

i. To optimize the parameters for biodiesel production.
ii. To study the influence of parameters on the biodiesel yield.
iii. To study the difference of homogeneous and heterogeneous process in the biodiesel production.

1.4 Thesis outline

In this project, Chapter 1 Introduction will explain on the reason and purpose of this research, problem statement, objectives and the scope of study.

This following Chapter 2 Literature Review will describe the theory of the biodiesel production based on previous researches. Several research papers and journals have been reviewed in order to understand the process. This chapter will also explain on the elements within this study such as the feed, the catalyst and the processes involved in producing biodiesel.

Chapter 3 Methodology explains in detail the step by step process in biodiesel production. The steps are oil analysis, acid esterification, base transesterification, optimization of parameter values and characterization of biodiesel sample. The chemicals and equipments used in these steps are also clearly stated in this chapter.

Chapter 4 Results and Discussion shows the results obtained from all the experiments from all the steps defined from the methodology. The first step, oil analysis gives the free fatty acid (FFA) content in the raw oil. Acid esterification gives the new result of the FFA content. Base transesterification is repeated for 21 runs to study its optimum yield and the effect of different parameter values on the biodiesel production. The biodiesel sample is characterized and all property data are shown and explained in this chapter.

Chapter 5 Conclusion concludes all the results and gives recommendation for further improvements of the project.

CHAPTER 2

LITERATURE REVIEW

This following chapter will describe the theory of the biodiesel production. Several research papers and journals have been reviewed in order to understand the process. This chapter will also explain on the elements within this study such as the feed, the catalyst and the processes involved in producing biodiesel.

2.1 Biodiesel

Biodiesel are monoalkyl esters of long chain fatty acids derived from renewable feed stock like vegetable oils and animal fats. The production of biodiesel is through transesterification process which the oil reacts with monohydric alcohol with a catalyst (Meher et al., 2006). Biodiesel can be used in pure form or may be blended with petroleum diesel at any concentration in most injection pump diesel engines. Table 1 shows the advantages of biodiesel compared to petroleum-derived diesel.

TABLE 1. Advantages of biodiesel compared to petroleum-derived diesel

Advantages	Disadvantages
• Domestically produced from non-petroluem, renewable resources • Can be used in most diesel engines, especially newer ones • Less air pollutants (other than nitrogen oxides) • Less greenhouse gas emissions (e.g., B20 reduces CO_2 by 15%) • Biodegradable • Non-toxic • Safer to handle	• Use of blends above B5 not yet approved by many auto makers • Lower fuel economy and power (10% lower for B100, 2% for B20) • Currently more expensive • B100 generally not suitable for use in low temperatures • Concerns about B100's impact on engine durability • Slight increase in nitrogen oxide emissions possible in some circumstances

2.2 Transesterification reaction

Transesterification is the displacement of alcohol from an ester by another in a process similar to hydrolysis, except than alcohol is used instead of water (Meher et al., 2006). The transesterification reaction is represented by the general equation as in Figure 1.

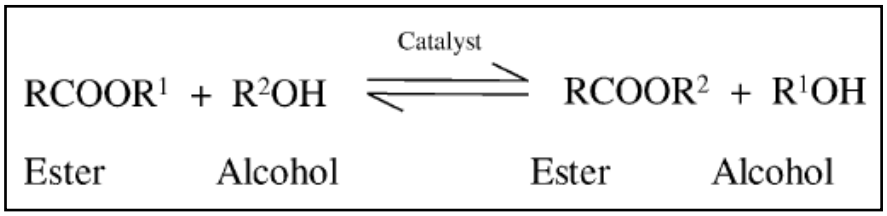

FIGURE 1. General equation of transesterification

In the transesterification reaction, long chain and branched chain fatty acid molecules are converted to monoester. There are three consecutive reversible steps involved;

i. conversion of triglycerides to diglycerides,
ii. diglycerides to mono-glycerides
iii. glycerides are converted into glycerol and one ester molecule in each step

The ester produced in this reaction has similar properties to diesel. The reaction is as shown in Figure 2.

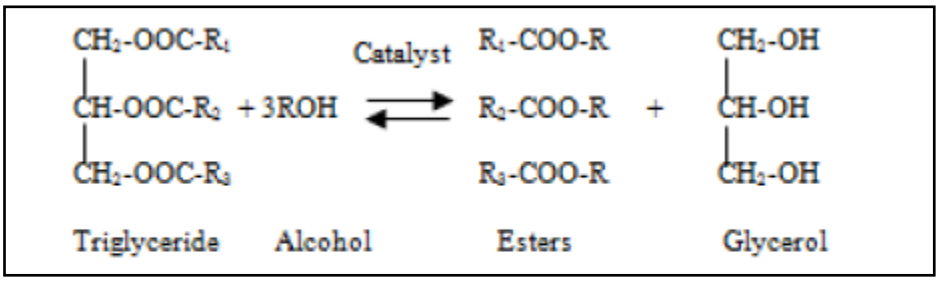

FIGURE 2. Transesterification of triglycerides with alcohol

The most common alcohol used in transesterification are short chain alcohol such as methanol, ethanol and propanol. Methanol is mostly used because it is the cheapest. But the yield of ester is independent of the type of alcohol used (Nur et al., 2012).

The transesterification reaction kinetics either with acid or alkali catalyst is studied by some researchers. Table 2 shows the different kinetic models studied by each paper.

TABLE 2. The different kinetic models for biodiesel production

Model	Reactions	Order	References
Three steps, reversible, alkaline as catalyst	TG + MeOH → DG + ME DG + MeOH → MG + ME MG + MeOH → GL + ME	Second Order	(Darnoko & Cheryan, 2000; Noureddini & Zhu, 1997)
Three steps, irreversible, no catalyst	TG + MeOH → DG + ME DG + MeOH → MG + ME MG + MeOH → GL + ME	First Order	(Diasakou et al., 1998)
One step, reversible, no catalyst	TG + 3MeOH → GL + 3ME	First Order	(Kusdiana & Saka, 2001)
One step, reversible, different base catalyst	TG + 3MeOH → GL + 3ME	First or third order	(Singh & Fernando, 2007)

TG: triglyceride, MeOH: methanol, DG: diglyceride, MG: monoglyceride, GL: glycerol, ME: methyl esters

Table 2 shows thatthe biodiesel production can occur in three step process, in which the triglyceride first react with methanol to produce diglyceride and the process continues by reacting diglyceride to form monoglyceride and lastly glycerol will be formed. In this study, the model used is one step process in which the triglyceride is reacted with methanol to form glycerol immediately with the presence of catalyst.

2.3 Catalyst for biodiesel production

Generally, biodiesel is produced by transesterification process by using homogenous catalyst such as potassium hydroxide and sodium hydroxide. However, using this catalyst produces a huge amount of waste water ro separate and clean the final product (Kim et al., 2004). Heterogeneous catalyst is to replace the conventional homogenous catalyst to ease the process and removal of catalyst after the reaction (Hernández-Hipólito et al., 2014). This is also supported by a study comparing homogeneous and heterogeneous catalysts which concludes that the best option is to use heterogeneous to produce biodiesel (Lam et al., 2010).

The first heterogeneous catalyst used was Na/NaOH/g-Al_2O_3 in the production of biodiesel (Park et al., 2008). Kim et al. (2004) uses the heterogeneous catalyst Na/NaOH/g-Al_2O_3 and found out that the heterogeneous base catalyst showed optimized conditions compared to when using homogeneous catalyst. Among other catalysts used are Magnesium-Lanthanum-mixed oxide (Mg/La) (Babu et al., 2008), calcium oxide (Borges & Díaz, 2012), sodium titanate (Hernández-Hipólito et al., 2014), SO_4^{2-} /Al_2O_3, SO_4^{2-}/SiO_2, H-zeolite, SO_4^{2-}/ZrO_2, WO_3/ZrO_2 and C sheteropoly acid (Park et al., 2008).

Out of other heterogeneous catalysts, sodium titanate is found to have attractive textural properties: high surface area and pore volume. Sodium titanate can also be reused for a few times without significant decrease in the catalytic activity. Because of these advantageous features sodium titanate ($Na_2Ti_3O_7 \cdot nH_2O$) is used as the catalyst in our present work. There are also few other parameters that are found to have significant effects on the biodiesel yield including catalyst loading, reaction time, reaction temperature and methanol to oil molar ratio reaction (Hernández-Hipólito et al., 2014). Figure 3 shows the molecular structure of sodium titanate.

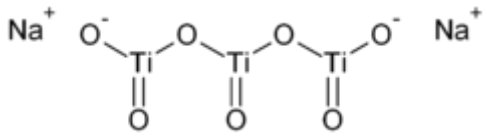

FIGURE 3. Molecular structure of sodium titanate

Sodium titanate is formed from titanium oxide in which the titanium oxide has three crystal arrangement namely (a) Anatase, (b) Rutile and (c) Brookite. Figure 4 illustrates these different crystal arrangements.

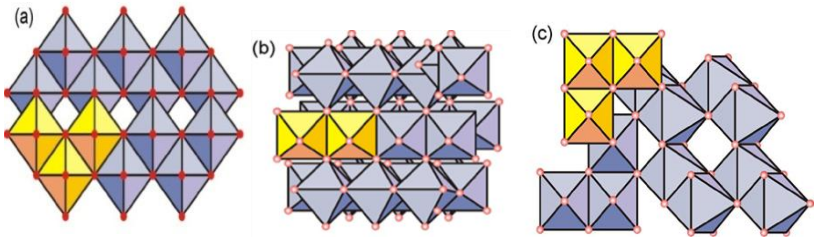

FIGURE 4. Titanium oxide crystal arrangement

Anatase has the highest band gap energy compared to rutile and brookite hence it is widely used for synthesis of the hierarchical structures of titanium oxide (one dimensional, two dimensional and three dimensional). The degradation of any organic matter is much more rapid in anatase phase, the degradation reaction is affected by the crystalline state and the physical properties such as the particle size and higher surface area reaction (Hernández-Hipólito et al., 2014).

2.4 Parameter optimization

Parameter optimization is done to study the parameter values which will give the best production process of biodiesel. The parameters to be investigated are oil to methanol ratio, catalyst concentration, temperature and time. There are also studies on the optimization of biodiesel production but they uses different experimental design methodologies. Cavalcante et al. studied transesterification of castor oilwith ethanol using a central composite rotatable design (Cavalcante et al., 2010). Ramezani et al. used Taguchi method for optimization of castor oil transesterification (Ramezani et al., 2010). Silva et al. studied alkali ethanolysis of castor oil and optimized the process parameters by using factorial design adding central points and axial points as star points (de Lima da Silva et al., 2009). Jeong and Park optimized the biodiesel production from castor oil using response surface methodology (Jeong & Park, 2009). Kılıç et al. study the biodiesel production from castor oil has been optimized by application of the full-factorial design (Kılıç et al., 2013). In this project, the method used is Response Surface Methodology by using the Design Expert 6.0.8 software.

2.4 Vegetable oil as feedstock

Vegetable oil is already tested as fuel for engine by Rudolf Diesel over one hundred years ago (Ma & Hanna, 1999). Even today, vegetable oil are becoming an alternative to normal petroleum-derived diesel because they are renewable, and having a green and environmental friendly effect. Compared to the normal diesel, vegetable oil biodieel have no sulphur content, have excellent lubrication properties and plants producing vegetable oils absorb more carbon dioxide than the amount they use to burn as biodiesel (Ramadhas et al., 2005).

Various vegetable oil can be used for the biodiesel production. In a paper by Nur (2012), the biodiesel production uses Kapok seed oil or its scientific name, Ceiba Pentandara which gives yield to 22 – 25% wt/wt of oil (Nur et al., 2012). Noshadi (2012) uses waste cooking oil as the feedstock to develop an optimal continuous process to produce biodiesel in a distillation column.

This optimization uses response surface methodology and the optimum conditions are determined yielding biodiesel about 93% (Noshadi et al., 2012).

In this project, castor oil is used as the feedstock to produce biodiesel. Castor oil or its scientific name, Ricinus comunis L.(Dias et al., 2013), constitutes mainly of triglycerides consist of three fatty acids and one molecule of glycerol. Table 3 shows the physical and chemical properties of castor oil based on previous study done (Kılıç et al., 2013).

TABLE 3. Physical and chemical properties of castor oil

Property	Value
Density (15°C)	965 kg/m^3
Refractive index (20°C)	1.478
Acid index	0.64 mg KOH/g
Iodine index	86 g I^-/100 g
Saponification index	182 mg KOH/g
Unsaponifiable matter (%)	0.11
Viscosity	6.6 St
Molecular weight	962 kg/mol

CHAPTER 3

METHODOLOGY

The methodology of the experiment basically follows 4 steps which are:

i. Oil analysis
ii. Acid esterification
iii. Base transesterification
iv. Optimization of parameter values
v. Characterization of biodiesel

This chapter will explain in detail on how does the experiment being conducted.

3.1 Chemicals needed

In order to perform the acid esterification and base transesterification to produce biodiesel from castor oil, the following chemicals are needed:

1. Raw Castor Oil
2. Potassium Hydroxide, KOH
3. Sulphuric Acid, H_2SO_4
4. Sodium Titanate
5. Isopropanol
6. Toluene
7. Phenolphthalein

3.2 Oil analysis

The basic oil analysis is to be performed on the crude castor oil and treated castor oil following the American Oil Chemists' Society (AOCS) method (Ahmad et al., 2014). The analysis is to detemine the acid value and the free fatty acid (FFA) content in the oil. It is important to know the FFA content because high FFA can lead to saponification of the oil instead of producing

biodiesel. Therefore, to obtain high yield of biodiesel, it is important to have as low FFA content as possible.

To determine the FFA content, titration is done by using potassium hydroxide as titrate and is titrated to a mixture isoprpylene and toluene with phenolphthalein as the indicator. A blank sample is first titrated and proceed to the second titration with 2 grams of oil added to the conical flask.

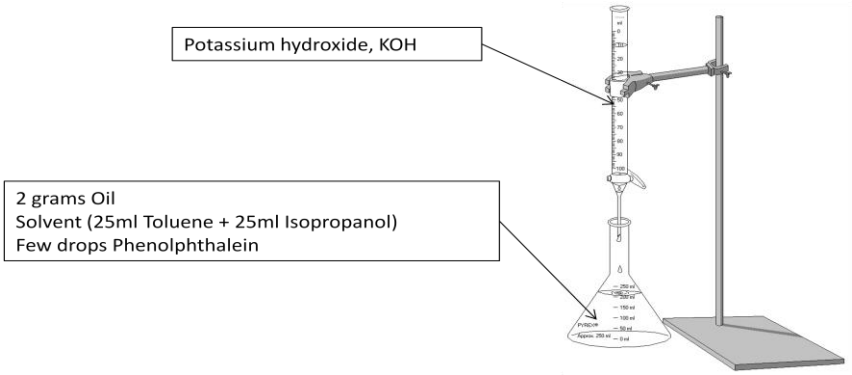

FIGURE 5. Titration to determine FFA content

The acid value is then calculated by using the equation

$$Acid\ value = \frac{(V_a - V_b) \times N \times \rho}{W}$$

whereby

V_a : volume of potassium hydroxide used against sample

V_b : Volume of potassium hydroxide used against blank

N : Normality of oil

ρ : Density of oil

W : Weight of sample used

The FFA value is then calculated from the acid value

$$FFA = \frac{Acid\ value}{2}$$

3.3 Acid esterification

The oil or triglycerides next undergoes acid esterification in which it reacts with lower alcohol such as methanol, CH_3OH with the presence of acid catalyst and to reduce the acid value and free fatty acid amount. In this project, the acid catalyst to be used is sulphuric acid, H_2SO_4 to reduce the acid value and free fatty acid content. The acid value is the number of milligrams of potassium hydroxide needed to neutralize the free acid in 1 gram of sample.

The experiment is conducted by using a triple neck round bottom flask set with a reflux condenser. This is to avoid any methanol loss. Each experimental run requires 50 grams of oil to be heated to the desired temperature. Methanol and sulphuric acid is then added in a pecific amount and stirred for a specific time. The reaction is stopped and the sample is put into the separating funnel to separate the pretreated oil and excess of methanol and catalyst. The treated oil is separated and collected for base transesterification process next. The free fatty acid amount should be less than 2% before proceeding to the base transesterification else the acid esterification should be repeated with different parameter values.

3.4 Base transesterification

Similar to acid transesterification, the oil or triglycerides undergoes esterification but this time with the presence of base catalyst. The base catalyst used is the homogeneous catalyst, potassium hydroxide.

To conduct the base transesterification, 50 grams of pre-treated oil is mixed together with methanol and the catalyst then is heated and stirred for a specific time such as in Figure 6. Then the sample undergo separation in the separating funnel and will be left for 24 hours. Two layers will form such as in Figure 7; the upper layer is methyl ester (biodiesel) while the lower layer is glycerol, methanol and other impurities.

FIGURE 6. Base transesterification process

FIGURE 7. Separation of biodiesel and glycerol after the base transesterification

3.5 Optimization of parameter values

The optimization of base transesterification process is performed and optimized using the Response Surface Methodology (RSM) from the software Design Expert 6.0.8. To investigate the effects of four parameters on the yield, the total number of experiments needed are 21 runs. The process parameters and its ranges of values are shown in Table 4. The low values are denoted as -1 while the high values are denoted as +1. These parameters are randomized to minimize the unexpected viriability on the response.

TABLE 4. The process parameters and its ranges of values

Parameters	**-1**	**0**	**+1**
A: Oil to methanol ratio	3	6	9
B: Catalyst concentration	0.5	1.0	1.5
C: Temperature	45	55	65
D: Time	45	67.50	90

The Response Surface Methodology gives formulation of a second order equation that describes the process. The general equation is as follows:

$$\mathbf{B = b_o + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_4 X_4 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{44} X_4^2 + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{14} X_1 X_4 + b_{23} X_2 X_3 + b_{24} X_2 X_4 + bb_{34} X_3 X_4}$$

where B is the methyl ester yield percentage, b_o is the intercept term, b_1, b_2 b_3 and b_4 are linear coeffiecients, b_{11}, b_{22}, b_{33}, b_{44} are quadratic equations, b_{12}, b_{13}, b_{14}, b_{23}, b_{24}, b_{34} are interactive coefficients and X_1, X_2, X_3, X_4 are the independent variables.

The optimized conditions are then determined and used for further study with heterogeneous catalyst for biodiesel production. Taking these values, the experiment is repeated by using heterogeneous catalyst, sodium titanate. The yield is the compared to determine the effect of catalyst towards the yield of biodiesel.

3.6 Characterization of biodiesel

The castor oil biodiesel is then analyzed by using the equipments as stated in Table 5.

TABLE 5. Equipments used for characterization of biodiesel

No	Parameter	Equipment
1	Conversion	Shimadzu, GC FID 2010
2	Viscosity	Lovis 2000 M, Anton Paar
3	Density	DMA 4500 M, Anton Paar
4	Oxidation stability	873 Biodiesel Ranchimat, Metrohm
5	Flash point	CLA 5, Petrotest
6	Cloud point	CPP 5Gs, ISL by PAC
7	Pour point	CPP 5Gs, ISL by PAC
8	Cold filter plugging point	FPP 5Gs, ISL by PAC

The biodiesel parameter values are then recorded and compared with the standards EN 14214 and ASTM D6751.

3.7 Methodology flow chart

Figure 6 shows the overall methodology flowchart of the project:

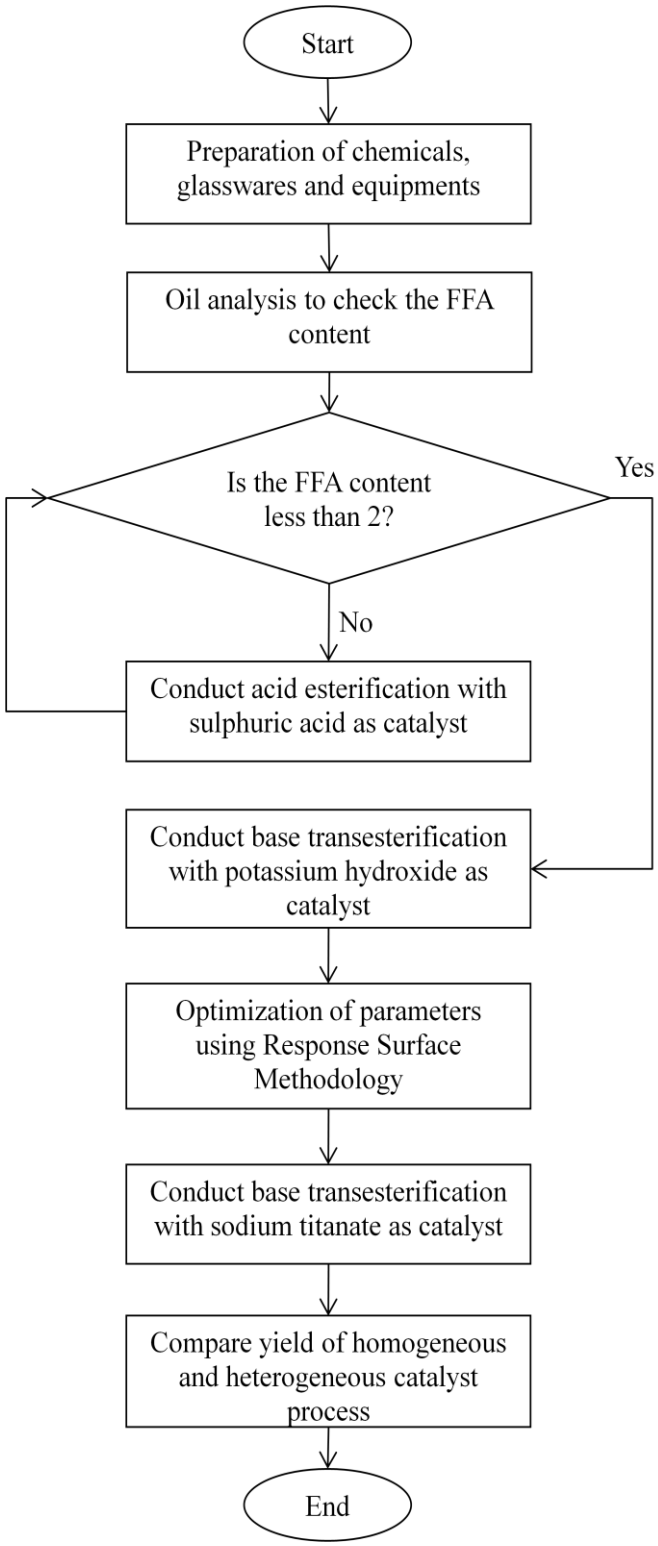

FIGURE 6. Flow diagram of the project methodology

3.8 Key Milestones

Several key milestones for this research project must be achieved in order to meet the objective of this project:

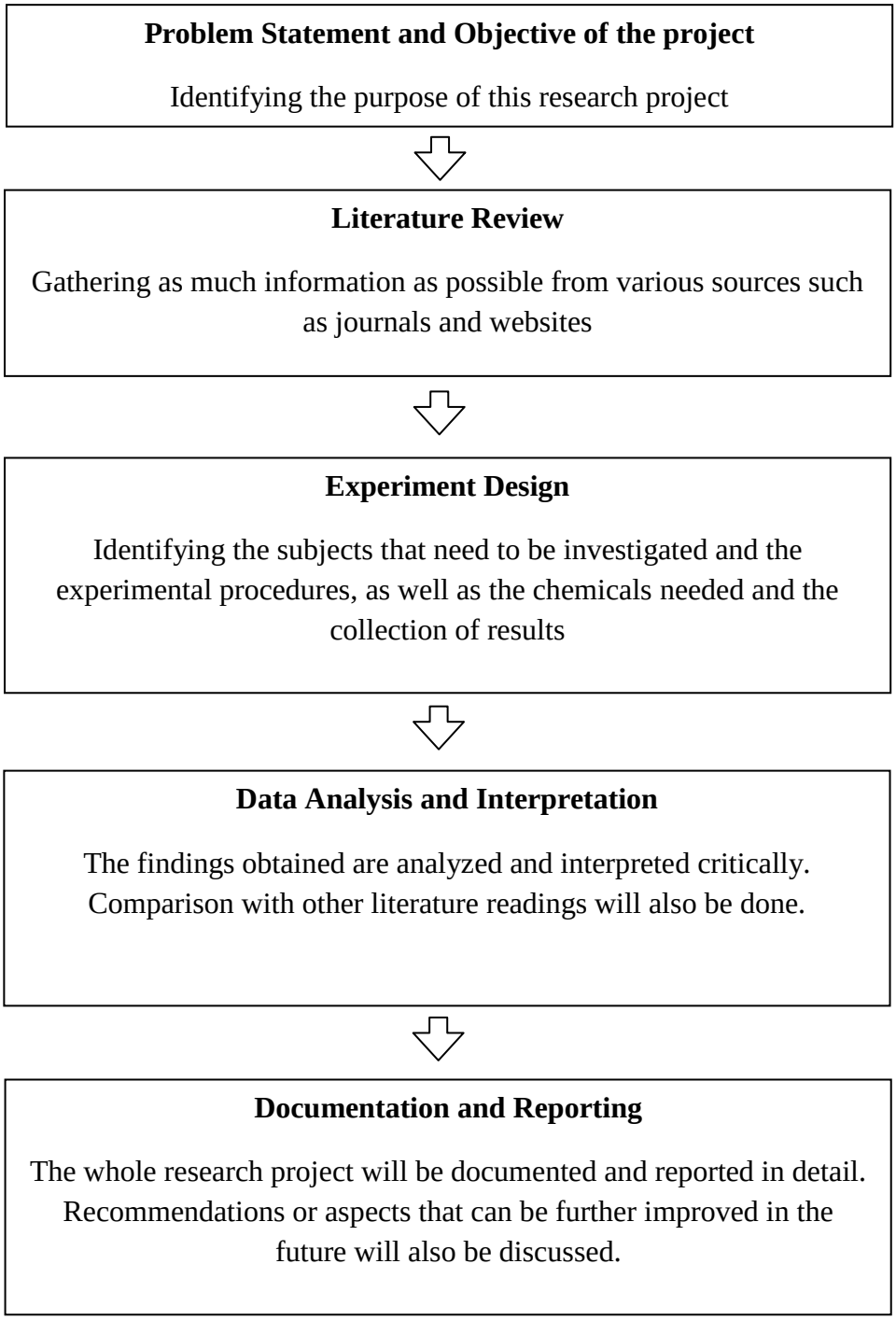

FIGURE 7. Key milestone

3.9 Gantt Chart

TABLE 6. Gannt Chart

NO	WEEK / DETAIL	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work															
	a) Oil analysis															
	b) Acid esterification															
	c) Base transesterification															
	d) Characterization of biodiesel															
2	Submission of Progress Report															
3	Pre-SEDEX															
4	Submission of Draft Final Report															
5	Submission of Dissertation (soft bound)															
6	Submission of Technical Paper															
7	Viva															
8	Submission of Dissertation (hard bound)															

CHAPTER 4

RESULTS AND DISCUSSION

All results, data and observations are recorded and explained in this chapter in detail. This includes the free fatty acid results from the oil analysis and acid esterification and yield values for 21 runs of base transesterification. The yield values are then used to optimize the parameters which are oil to methanol ratio, catalyst concentration, temperature and time using the Response Surface Methodology. The relationship between each parameter is also studied. The biodiesel sample is also characterized to determine its properties to be compared to the current biodiesel standards. The composition of the biodiesel sample is also determined by using the gas chromatography.

4.1 Oil Analysis

The experimental work is currently undergoing using different types of oil to familiarize with the methodology of the experiment. The experiment has been completed from the characterization of oil feed and the acid esterification and is planning to continue with base transesterification.

For the characterization of oil feed, various types of oil were used, namely jathropa oil, waste cooking oil and kapok seed oil. The characterization of feed is done to determine the free fatty acid amount within the oil. The results of the experiment are as in Table 7.

TABLE 7. Free Fatty Acid (FFA) amount in untreated oil

Oil	Acid value (mg KOH/g)	Free fatty acid (FFA) amount
Jathropa oil	61.95	30.97%
Waste cooking oil	44.78	22.39%
Kapok seed oil	17.52	8.76%
Castor oil	3.61	1.81%

The experiment is continued with acid esterification whereby castor oil is taken for is reacted with methanol with the presence of an acid catalyst, sulphuric acid in order to reduce the FFA content. After the esterification process, the oil is extracted and characterized again to determine the new free fatty acid amount. It can be observed that the FFA amount decreases significantly from 8.76% to 1.90% after the acid esterification. After obtaining the desired FFA amount, the base transesterification process can be conducted to produce the biodiesel.

4.2 Acid Esterification

Acid esterification is done for the raw castor oil to reduce the free ftty acid (FFA) content in the castor oil to prevent saponification during the base transesterification process. The parameters used and the results of the acid esterification is as Table 8.

TABLE 8. Results of acid esterification of castor oil

Run	Parameters				Acid value	FFA
	Oil to methanol ratio	Catalyst concentration	Temperature	Time		
1	5	5%	60 °C	120 min	16.10	8.05%
2	10	10%	60 °C	120 min	11.95	5.98%

For the project to be proceeded to the base transesterification, the free fatty acid value should be lower than 2%. However, as shown in Table 8, the value is 8.05% for the first run. The acid esterification is repeated by using higher ratio of oil to methanol and catalyst concentration. The free fatty acid value dropped to 5.98% however it is not low enough to be used for base transesterification.

From Table 8, the FFA content is 8.05% and 5.98% for Run 1 and Run 2 respectively. This is much higher than the FFA amount of raw castor oil which is only 1.81% as stated in Table 6. This shows that the acid content within the oil has not decreased but instead increase significantly. It may be caused by the unseparated acid catalyst within the castor oil which has not washed off. To obtain the correct result without being affected by the acid catalyst, the washed off water should be of the same pH before and after washing the biodiesel. However, to achieve this, a large amount of water is needed and this is not economically preferable. Thus, in this project, acid esterification is not conducted before the base transesterification as the FFA content is raw castor oil is already very low which is 1.81%.

4.3 Base transesterification

The base transesterification is performed by following the design in Table 9. The response factor is the yield percentage based on the mass of biodiesel product.

TABLE 9. The yield percentage of biodiesel for each experimental run

Experimental run	Oil to methanol ratio	Catalyst concentration (wt %)	Time (min)	Temperature (^{o}C)	Yield (%)
1	3.00	0.50	45.00	45.00	73.00
2	6.00	1.00	55.00	67.50	86.16
3	3.00	1.50	65.00	90.00	0.00
4	6.00	0.16	55.00	67.50	90.22
5	6.00	1.00	38.18	67.50	85.64
6	11.05	1.00	55.00	67.50	84.98
7	6.00	1.00	55.00	67.50	85.48
8	9.00	0.50	45.00	90.00	95.66
9	6.00	1.00	71.82	67.50	82.80
10	9.00	1.50	45.00	45.00	79.48
11	3.00	1.50	45.00	90.00	75.40
12	6.00	1.84	55.00	67.50	70.74
13	3.00	0.50	65.00	45.00	0.00
14	9.00	0.50	65.00	90.00	78.00
15	6.00	1.00	55.00	29.66	87.88
16	0.95	1.00	55.00	67.50	90.90
17	9.00	1.50	65.00	45.00	74.14
18	6.00	1.00	55.00	67.50	85.56
19	6.00	1.00	55.00	67.50	68.78
20	6.00	1.00	55.00	105.34	84.26
21	6.00	1.00	55.00	67.50	88.78

4.4 Optimization of parameter values

The optimization of parameter values are conducted by using the Design Expert 6.0.8. All data and graphs are obtained from the software.

Pertubation plot compares all factors that can influence the process. Figure 7 shows the pertubation plot base transesterification for castor oil biodiesel based on studies conducted. The steepest plot shows the most significant and influential factor towards the response which is the yield percentage, which is parameter B, catalyst concentration. The second most influencing factor is parameter A, oil to methanol ratio.

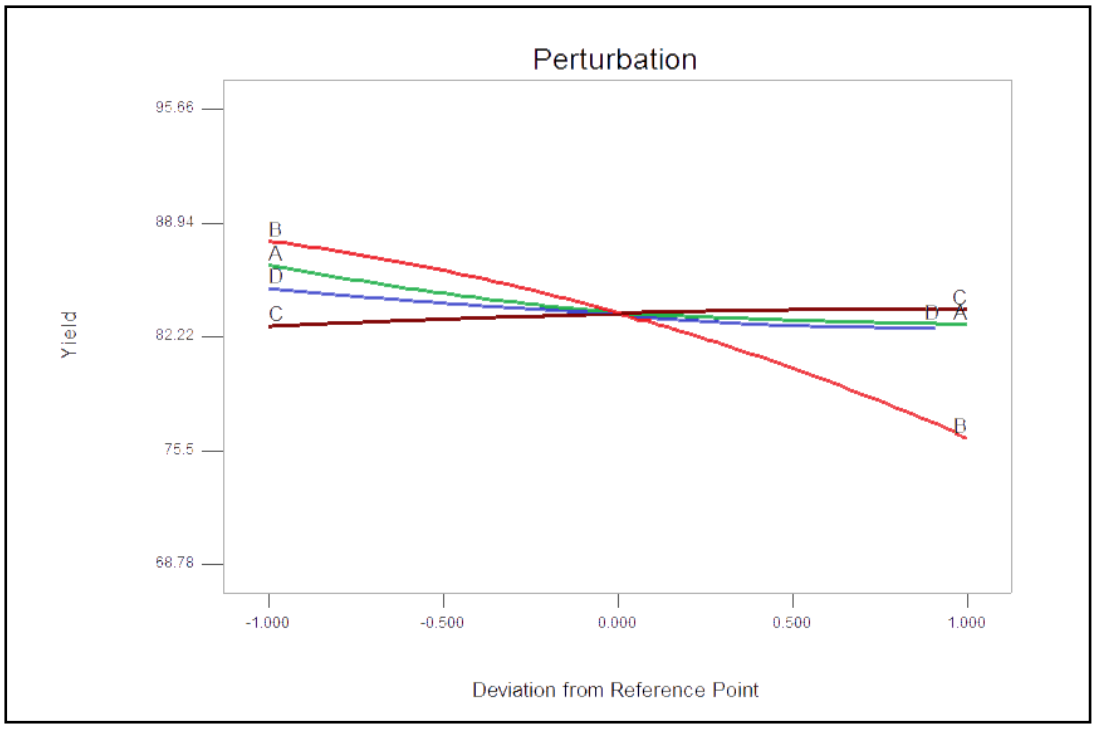

FIGURE 8. Pertubation plot for base transesterification

Figure 8 shows the comparison between actual values of FFA% with the predicted values for acid esterification and Yield with predicted values for base transesterification respectively. From the plot we can see whether the points are linear, which indicates that the predicted values are close to the experimental values.

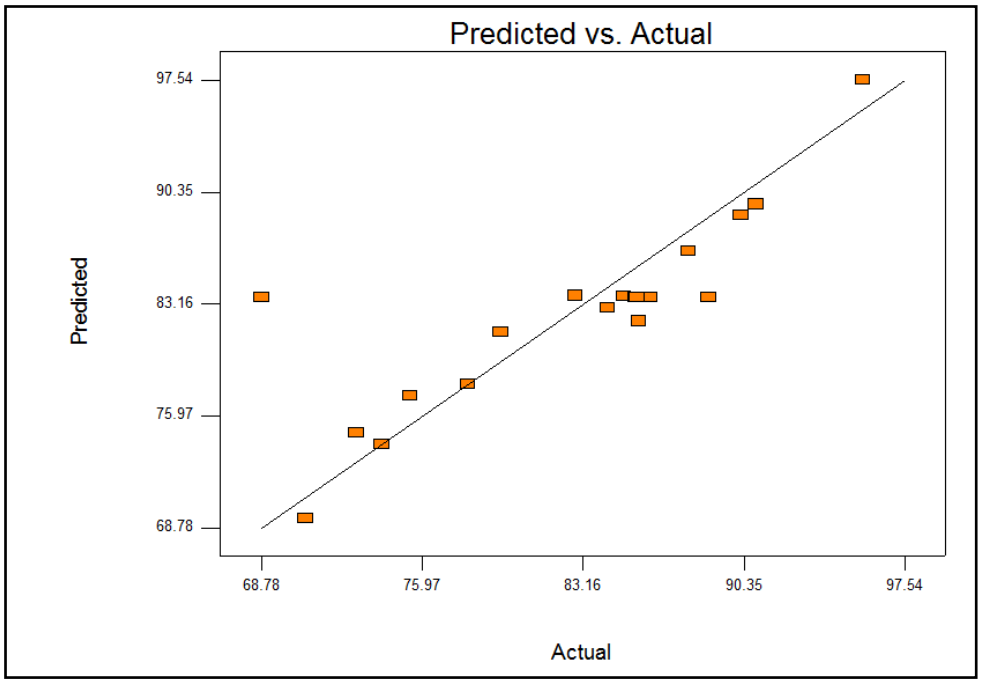

FIGURE 9. Predicted versus experimental values for base transesterification

It can be observed that the actual values lie near the predicted line thus these results are reliable.

To further compare the factors, a three dimensional plot and contour plot can be done. Figure 11 to 16 show the effect of the different parameters on the biodiesel transesterification process.

Figure 11 shows that by increasing the oil to methanol ratio and catalyst concentration, the yield will decrease.

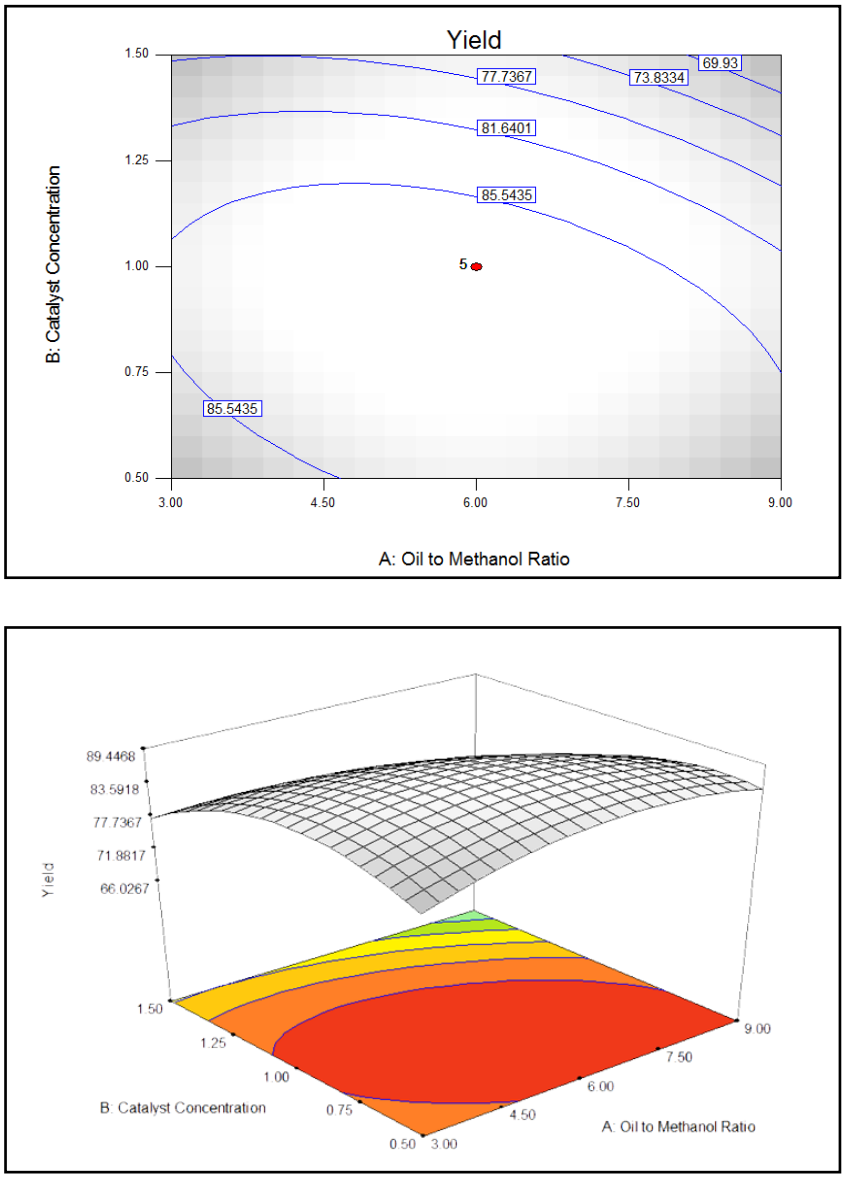

FIGURE 10. Combined effect of oil to methanol ratio and catalyst concentration (a) contour plot and (b) three dimensional plot

Figure 12 shows that by increasing the oil to methanol ratio and temperature, the yield will decrease.

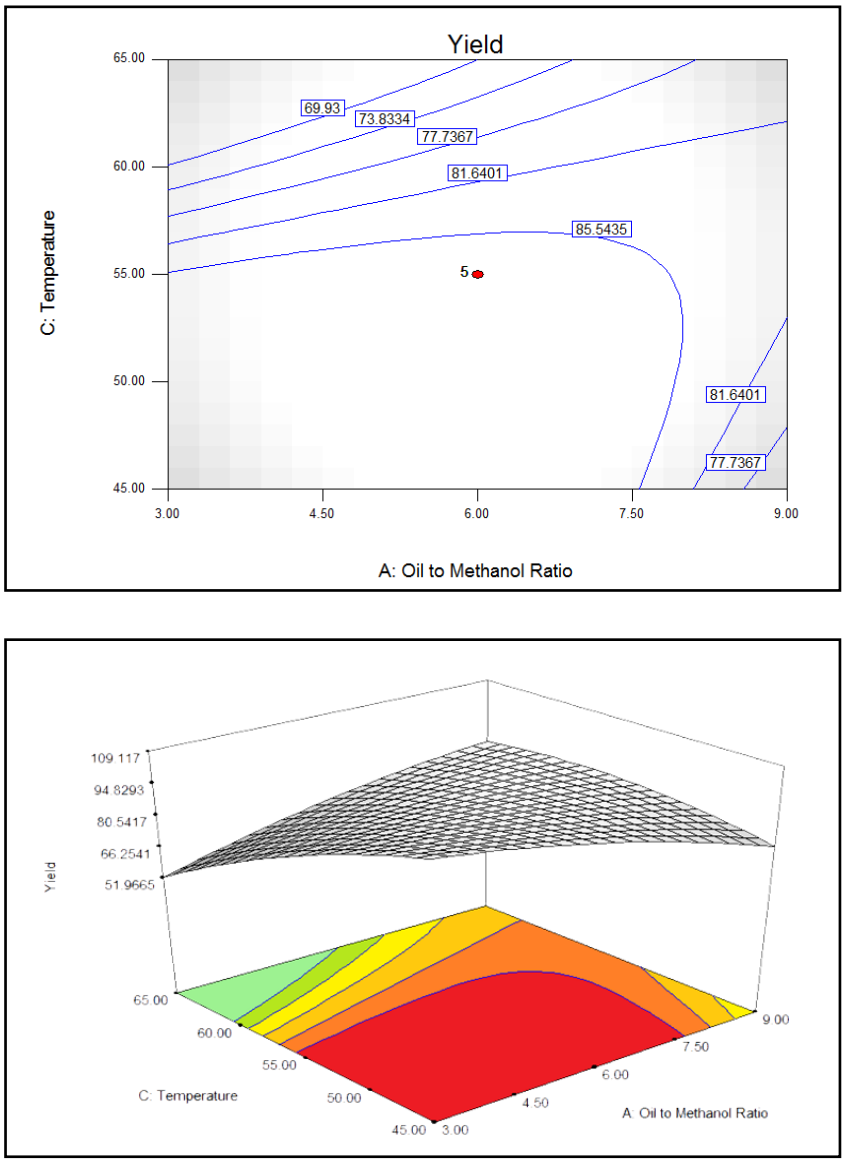

FIGURE 11. Combined effect of oil to methanol ratio and temperature (a) contour plot and (b) three dimensional plot

Figure 13 shows that by increasing the oil to methanol ratio and temperature, the yield will increase at first but will decrease for a higher value of oil to methanol ratio and time.

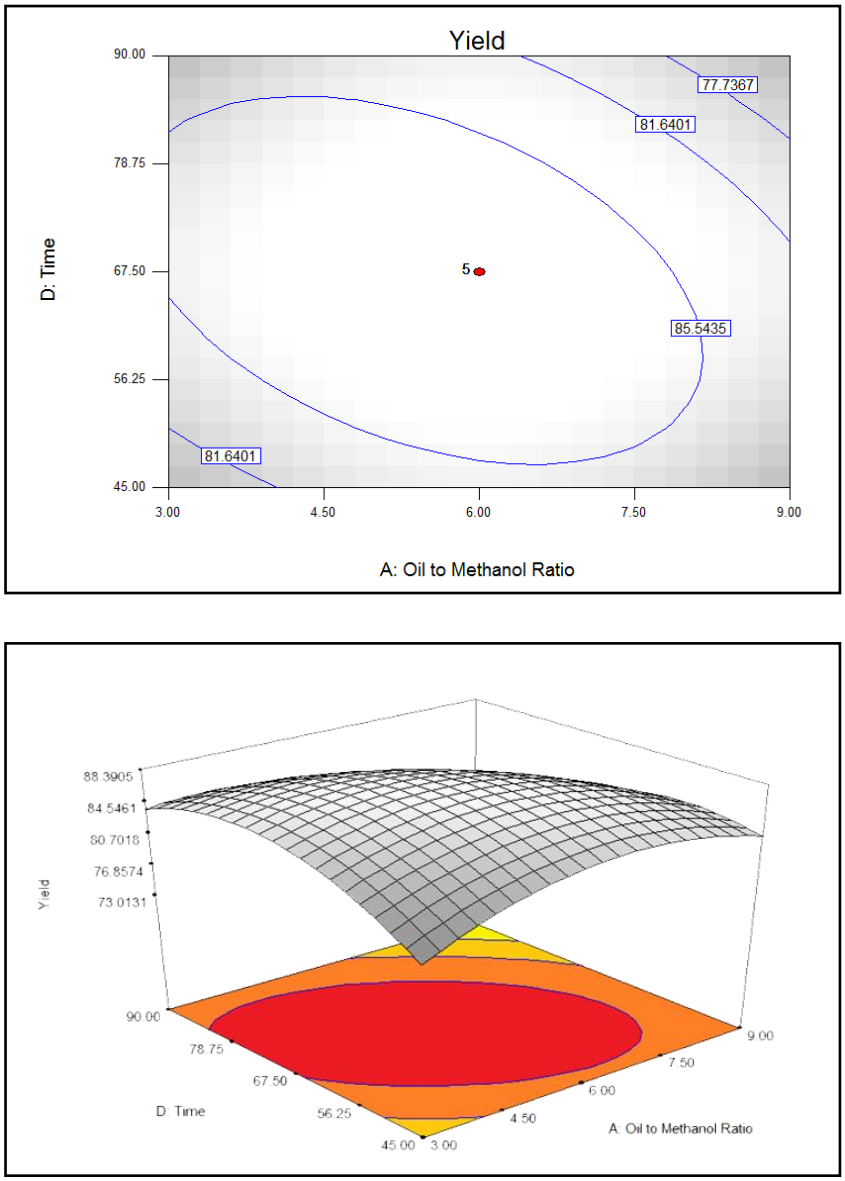

FIGURE 12. Combined effect of oil to methanol ratio and time (a) contour plot and (b) three dimensional plot

Figure 14 shows that by increasing the oil to methanol ratio and temperature, the yield will decrease.

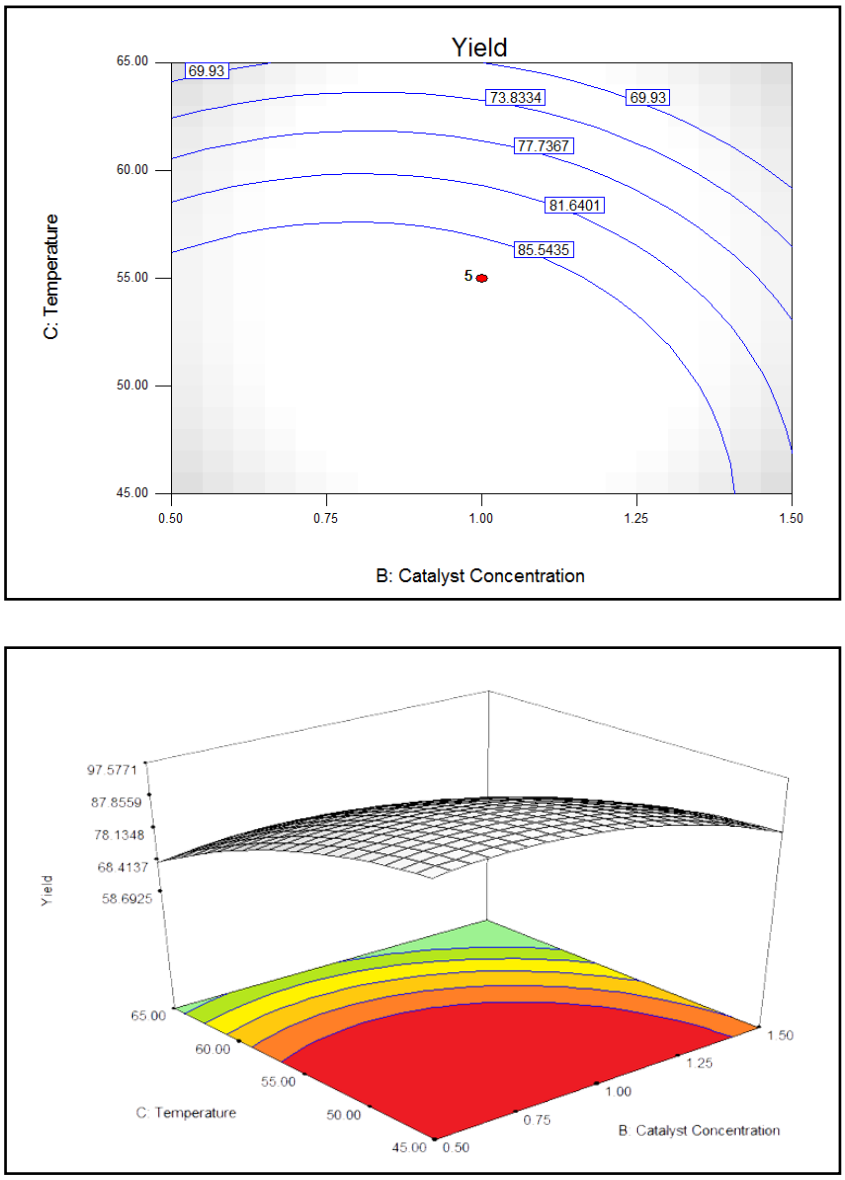

FIGURE 13. Combined effect of catalyst concentration and temperature (a) contour plot and (b) three dimensional plot

Figure 15 shows that at low value of catalyst concentration and time, by increasing the oil to methanol ratio and temperature, the yield will decrease. At high catlyst concentration and time, the yield will decrease.

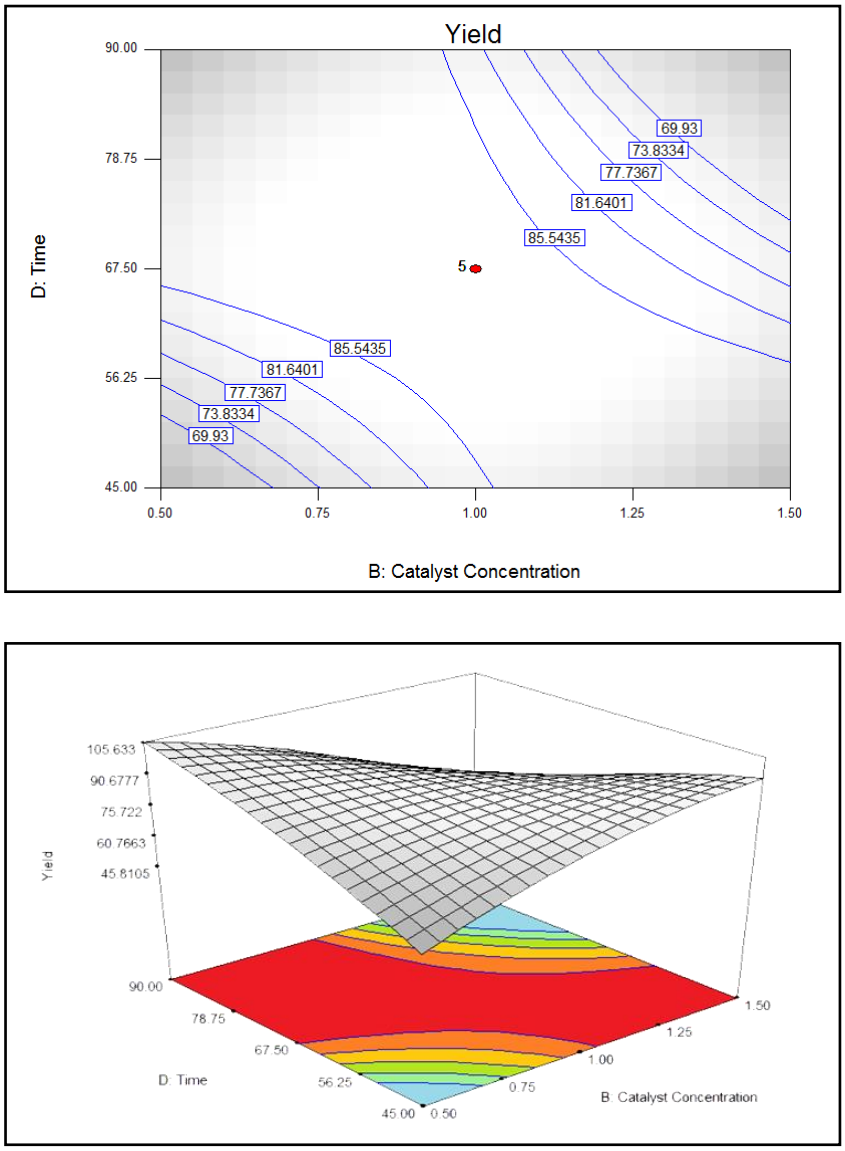

FIGURE 14. Combined effect of catalyst concentration and time (a) contour plot and (b) three dimensional plot

Figure 16 shows that by increasing the oil to methanol ratio and temperature, the yield will decrease.

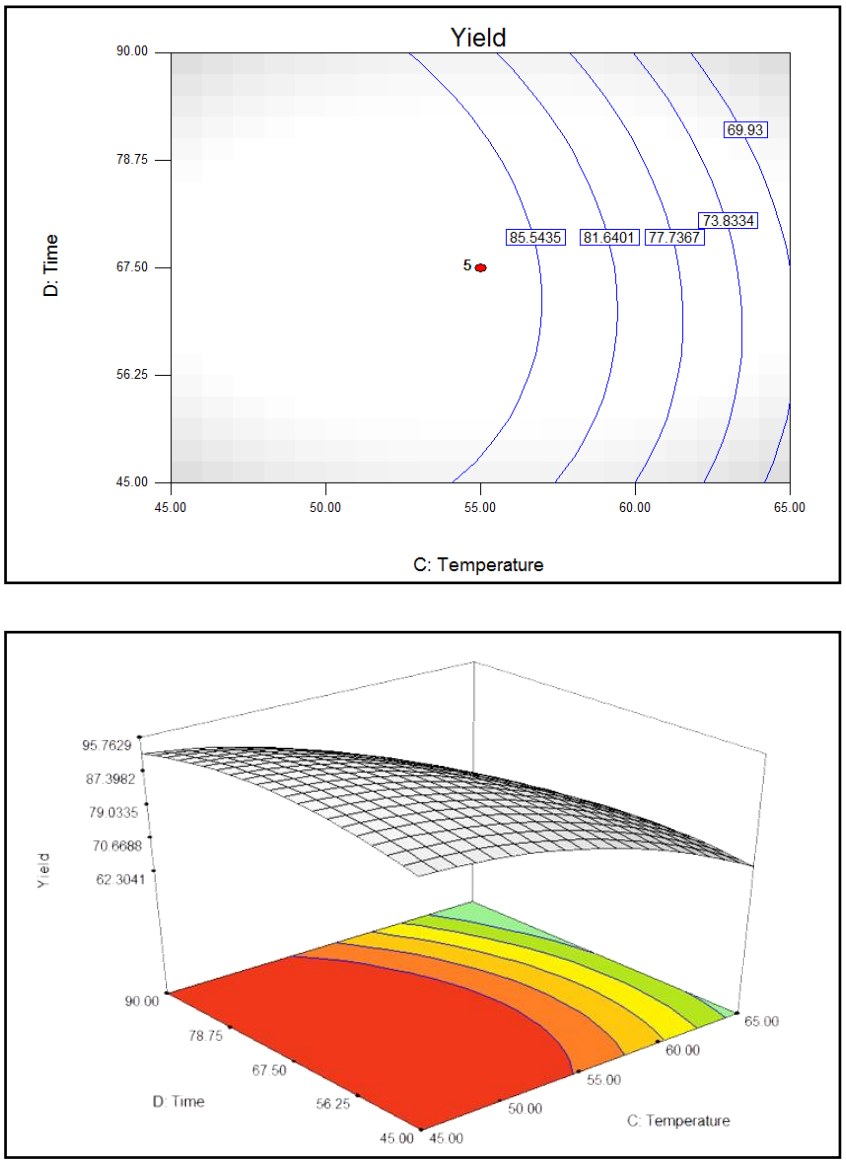

FIGURE 15. Combined effect of temperature and time (a) contour plot and (b) three dimensional plot

Table 10 shows the ANOVA analysis results for the base transesterification process. Factor D (time) has the highest F-value thus representing the most factor as compared to the other three factors.

TABLE 10. ANOVA analysis of base transesterification

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F
Model	10005.94	14	714.71	1.30	0.3945 not significant
A	17.52	1	17.52	0.032	0.8643
B	189.74	1	189.74	0.34	0.5787
C	2272.71	1	2272.71	4.13	0.0885
D	6.55	1	6.55	0.012	0.9167
A^2	245.34	1	245.34	0.45	0.5293
B^2	668.70	1	668.70	1.21	0.3128
C^2	430.46	1	430.46	0.78	0.4107
D^2	331.94	1	331.94	0.60	0.4671
AB	49.92	1	49.92	0.091	0.7735
AC	1965.64	1	1965.64	3.57	0.1078
AD	42.62	1	42.62	0.077	0.7902
BC	12.30	1	12.30	0.022	0.8861
BD	1927.84	1	1927.84	3.50	0.1105
CD	27.08	1	27.08	0.049	0.8319
Residual	3304.80	6	550.80		
Lack of Fit	3046.51	2	1523.25	23.59	0.0061 significant
Pure Error	258.29	4	64.57		

The second order polynomial equations in terms of coded and actual factors are the results of regression analysis. The second order model equation in terms of coded factors are shown below:

$Yield = 83.58 - 1.76\,A - 5.79\,B + 0.49\,C - 1.08\,D + 1.07\,A^2 - 1.56\,B^2 - 0.24\,C^2 + 0.41\,D^2 - 5.72\,AB - 7.18\,AC - 5.43\,AD + 3.08\,BC - 1.27\,BD$

Some studies can be found on the optimization of biodiesel production process from castor oil. These studies utilizes different optimization methodology such as using central composite rotatable design, Taguchi method, using factorial design, adding central points and axial points as star points and response surface methodology (Cavalcante et al., 2010; de Lima da Silva et al., 2009; Jeong & Park, 2009; Kılıç et al., 2013; Ramezani et al., 2010; Rodríguez-Guerrero et al., 2013).

Table 11 summarizes the optimal parameter values obtained for the biodiesel production process. The values are compared to previous studies' findings. The value of yield which is obtained is 83.57%.

TABLE 11. Comparison of process parameter values with previous studies

Parameters	Kılıç et al. [4]	Cavalcante et al. [13]	Ramezani et al. [14]	de Lima de Silva et al. [15]	Jeong & Park [16]	Rodríguez-Guerrero et al. [21]	This project
A: Oil to methanol ratio	7	11	8	16	8.24	40	9
B: Catalyst concentration (%)	1.5	1.75	0.5	1	1.45	0.1	0.5
C: Temperature (^{o}C)	65	-	65	30	35.5	300	45
D: Time (min)	10	90	120	30	45	10	90

The optimal conditions of biodiesel production from this project gives 83.57% yield of biodiesel. Taking the same value of parameters, the experiment is conducted by using heterogeneous catalyst, sodium titanate to produce biodiesel. The new yield obtained is 78% in which is lower than the yield obtained earlier. This shows that using homogeneous catalyst will give a higher yield than the production using heterogeneous catalyst. This is because of the reaction is more active in the same phase catalyst compared to when using a different phase. There is a slower mass transfer due to less contact between the catalyst and the liquid reactants, which further leads to the lower biodiesel yield obtained at the end of the reaction.

4.5 Characterization of biodiesel

Castor oil biodiesel was analyzed by using Gas Chromatography Flame Ionization Detector, Shimadzu, GC FID 2010. The detailed result from the gas chromatography is attached in the appendix. Figure 16 shows the peaks separated by its composition by the gas chromatography. Each peak represents the composition of each components.

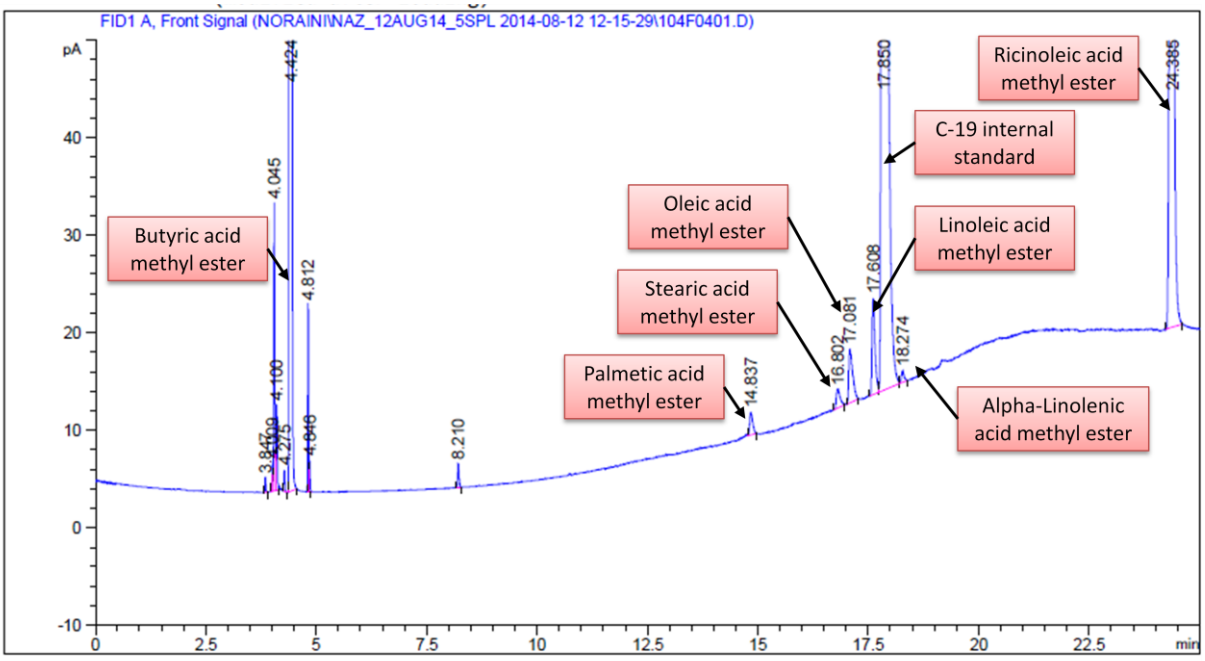

FIGURE 16. The result of Gas Chromatography for biodiesel sample

Table 12 shows the biodiesel composition calculated from the gas chromatography compared to the raw castor oil composition from literature (Gui et al., 2008).

TABLE 12. Composition of raw castor oil and biodiesel sample

No	Raw castor oil		Biodiesel	
	Component	Composition	Component	Composition
1	Butyric acid	-	Butyric acid methyl ester	0.37%
2	Palmitic acid	1.0%	Palmitic acid methyl ester	1.07%
3	Stearic acid	1.0%	Stearic acid methyl ester	1.17%
4	Oleic acid	3.0%	Oleic acid methyl ester	3.37%
5	Linoleic acid	0.3%	Linoleic acid methyl ester	4.48%
6	Alpha-Linolenic	-	Alpha-Linolenic acid methyl ester	0.49%
7	Ricinoleic	89.5%	Ricinoleic acid methyl ester	89.04%
8	Eicosenoic	0.3%	Eicosenoic acid methyl ester	-
9	Dihydroxystearic	0.7%	Dihydroxystearic acid methyl ester	-

Based on Figure 16 and Table 12, it can be seen that the biodiesel produced contains butyric acid methyl ester, palmitic acid methyl ester, stearic acid methyl ester, oleic acid methyl ester, linoleic acid methyl ester, alpha-linolenic acid methyl ester and ricinoleic acid methyl ester. The main composition of the castor oil is ricinoleic acid methyl ester which is comprising of 89.04% of the biodiesel. The other main components of the castor oil biodiesel are palmitic acid methyl ester, stearic acid methyl ester, oleic acid methyl ester and linoleic acid methyl ester. The presence of these five components shows that the castor oil is converted to biodiesel. The other components which is butyric acid methyl ester and alpha-lenolenic methyl ester are present most probably due to the slight impurities in the biodiesel.

The conversion of the biodiesel from oil can also be calculated based on the gas chromatography result using the following equation.

$$Conversion = \frac{Total\ area - Internal\ standard\ area}{Weight\ of\ internal\ standard \times \frac{Weight\ of\ internal\ standard}{Weight\ of\ sample}}$$

The biodiesel production using homogeneous catalyst reaches conversion 74.26% while for the biodiesel production using heterogeneous catalyst, the conversion is only 27.46%.

The biodiesel properties were analyzed and compared with ASTM D6751 and EN 14214 standard methods as shown in Table 13.

TABLE 13. The paramaeter of biodiesel sample and the value of standards EN14214 and ASTM D6751

No	Parameter	Homogeneous	Heterogeneous	EN 14214	ASTM D6751
1	Conversion	74.26%	27.46%	-	-
2	Viscosity	1.03 ns/m^3	22.16 ns/m^3	3.5 -5 ns/m^3	1.9 – 6.0 ns/m^3
3	Density	919.65 kg/m^3	956.41 kg/m^3	860 – 900 kg/m^3	-
4	Oxidation stability	64.86 hr	1.93 hr	6 min	3 min
5	Flash point	123 min	143 min	120 min	93 min
6	Cloud point	4.2 ^oC	4.4 ^oC	-	-
7	Pour point	2.5 ^oC	3.2 ^oC	-	-
8	Cold filter plugging point	3.8 ^oC	4.0 ^oC	-	-
9	Higher heating value	36060 J/g	35783 J/g	-	-

Most of the properties for the castor oil biodiesel produced with homogeneous catalyst is almost similar to the standard while the properties for castor oil biodiesel produced with heterogeneous catalyst deviates slightly from the standards. This shows that the quality of biodiesel produced using homogeneous catalyst is better than that of heterogeneous catalyst.

The density and the viscosity for the biodiesel produced with homogeneous catalyst is to the standard, however, it is observed that the density and the viscosity for the biodiesel produced with heterogeneous catalyst is not within the required limit.

The flash points for both the castor oil biodiesel produced with homogeneous and heterogeneous catalyst are higher than that of diesel, and thus it is safer to be stored. The low temperature properties of biodiesel are represented by cloud point, pour point, and cloud filter plugging point (CFPP). These properties are temperature dependent and the limits are not specified in the standards. Weather condition and the place where the test is conducted should be taken into consideration when determining the properties. The low properties of biodiesel in the present work are considerably good for Malaysian/ Equatorial weather. This shows that the biodiesel will not solidify easily and will flow smoothly in the pipes up till the specified temperature.

One of the most important fuel properties that affects the storage and usage efficiency for the biodiesel is the oxidative stability. Better oxidative stability is indicated by a higher amount of saturated fatty acid acid in the biodiesel. The biodiesel produced in the present work have poor oxidative stability. This may be caused by the excess methanol left in the biodiesel sample. It is recommended that to prevent this, the biodiesel sample should first be heated to evaporate all the methanol in the sample before conducting the characterization of biodiesel. Higher heating value indicates the energy produced during the burning of the biodiesel. This property is related to the stability of biodiesel.

CHAPTER 5

CONCLUSION

The scarcity of fossil fuel nowadays initiates the study to find alternative sources for renewable energy and one of it is from vegetable oil, biodiesel. This project aims to produce biodiesel from castor oil and to explore the heterogeneous catalyst, sodium titanate towards the production of sodium titanate compared to the conventional homogeneous catalyst. The optimal conditions of biodiesel production are firstly determined by using the response surface methodology. The production of biodiesel is done with various parameter values in each run to determine the most significant parameter towards the biodiesel production. The optimal values are; 9 oil to methanol ratio, 0.5% catalyst, temperature of 45 ^{o}C and 45 minutes.

By using the optimized values, the experiment is repeated with heterogeneous catalyst. The yield is compared and it shows that the yield for heterogeneous catalyst is slightly lower than the homogeneous catalyst process. Although the yield is lower for the heterogeneous catalyst process, it has potential for replacing the heterogeneous catalyst due to its reusability and ease of separation. It is recommended that this project is continued to check for the reusability of catalyst in which the same amount of catalyst may be used for many times of production and proves that the yield is higher collectively.

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APPENDICES

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Inj Volume : 1 µl
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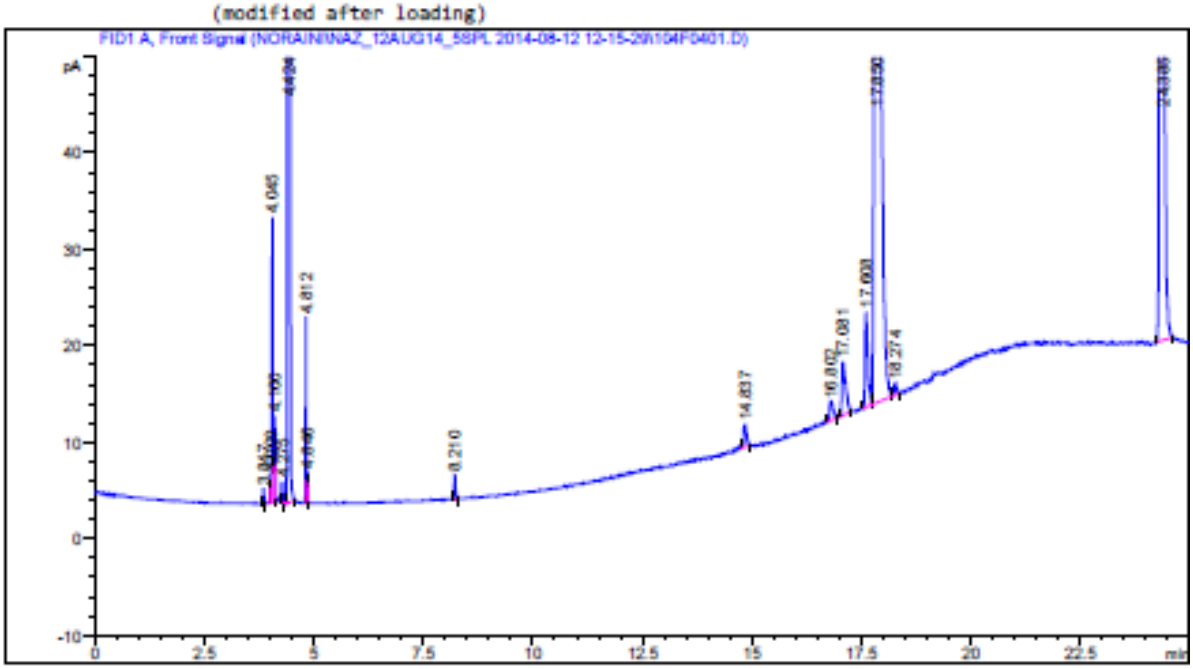

Normalized Percent Report

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Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A, Front Signal

RetTime [min]	Type	Area [pA*s]	Amt/Area	Norm %	Grp	Name
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5.201		-	-	-		
6.886		-	-	-		
8.978		-	-	-		
10.026		-	-	-		
11.036		-	-	-		
11.995		-	-	-		
12.929		-	-	-		

Data File C:\CHEM32\1\DATA\NORAINI\NAZ_12AUG14_5SPL 2014-08-12 12-15-29\104F0401.D
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RetTime [min]	Type	Area [pA*s]	Amt/Area	Norm %	Grp	Name
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13.860		-	-	-		
14.281		-	-	-		
14.837	BB	10.99318	1.67461e-2	0.104328		
15.131		-	-	-		
15.762		-	-	-		
16.113		-	-	-		
16.802	BB	11.95813	2.45558e-2	0.166411		
16.925		-	-	-		
17.081	BB	34.62374	2.45508e-2	0.481730		
17.278		-	-	-		
17.608	BV	45.90763	5.31590e-2	1.383008		
17.850	VB	1357.05774	5.84032e-2	44.915725		
18.274	BB	5.05892	5.85134e-2	0.167755		
18.788		-	-	-		
19.117		-	-	-		
19.700		-	-	-		
19.830		-	-	-		
20.052		-	-	-		
20.286		-	-	-		
20.417		-	-	-		
20.919		-	-	-		
21.051		-	-	-		
21.316		-	-	-		
22.028		-	-	-		
22.127		-	-	-		
23.511		-	-	-		
24.062		-	-	-		
24.385	BB	913.44165	1.01721e-1	52.656964		
Totals :				100.000000		

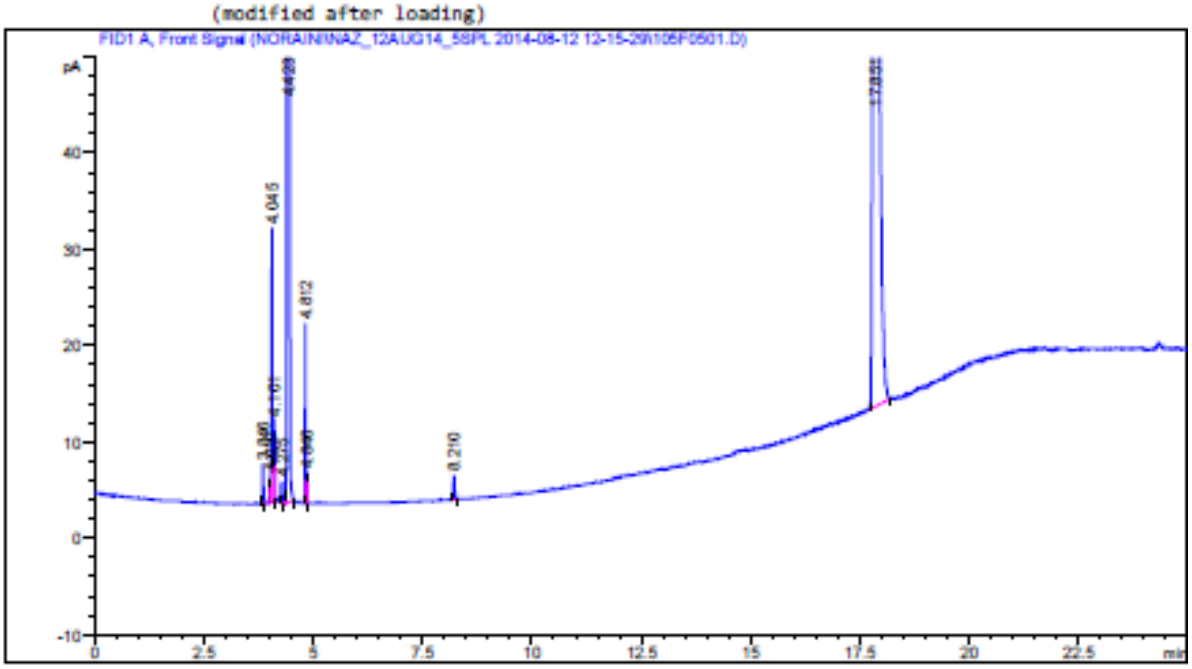
2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Calibrated compound(s) not found

*** End of Report ***

Data File C:\CHEM32\1\DATA\NORAINI\NAZ_12AUG14_5SPL 2014-08-12 12-15-29\105F0501.D
Sample Name: Castor (heterogeneous)

Acq. Operator : SYSTEM Seq. Line : 5
Acq. Instrument : GC7890 Location : Vial 105
Injection Date : 12/8/2014 2:11:45 PM Inj : 1
Inj Volume : 1 µl
Acq. Method : C:\CHEM32\1\DATA\NORAINI\NAZ_12AUG14_5SPL 2014-08-12 12-15-29\FAME MIX.M
Last changed : 12/8/2014 12:15:29 PM by SYSTEM
Analysis Method : C:\CHEM32\1\METHODS\FAME MIX REF MAY 2014.M
Last changed : 13/8/2014 4:52:05 PM by SYSTEM
(modified after loading)



Normalized Percent Report

Sorted By : Signal
Calib. Data Modified : 22/7/2014 11:37:23 AM
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A, Front Signal

RetTime [min]	Type	Area [pA*s]	Amt/Area	Norm %	Grp	Name
4.275	BB	3.77975	5.69157e-2	0.271670		
5.201		-	-	-		
6.886		-	-	-		
8.978		-	-	-		
10.026		-	-	-		
11.036		-	-	-		
11.995		-	-	-		
12.929		-	-	-		

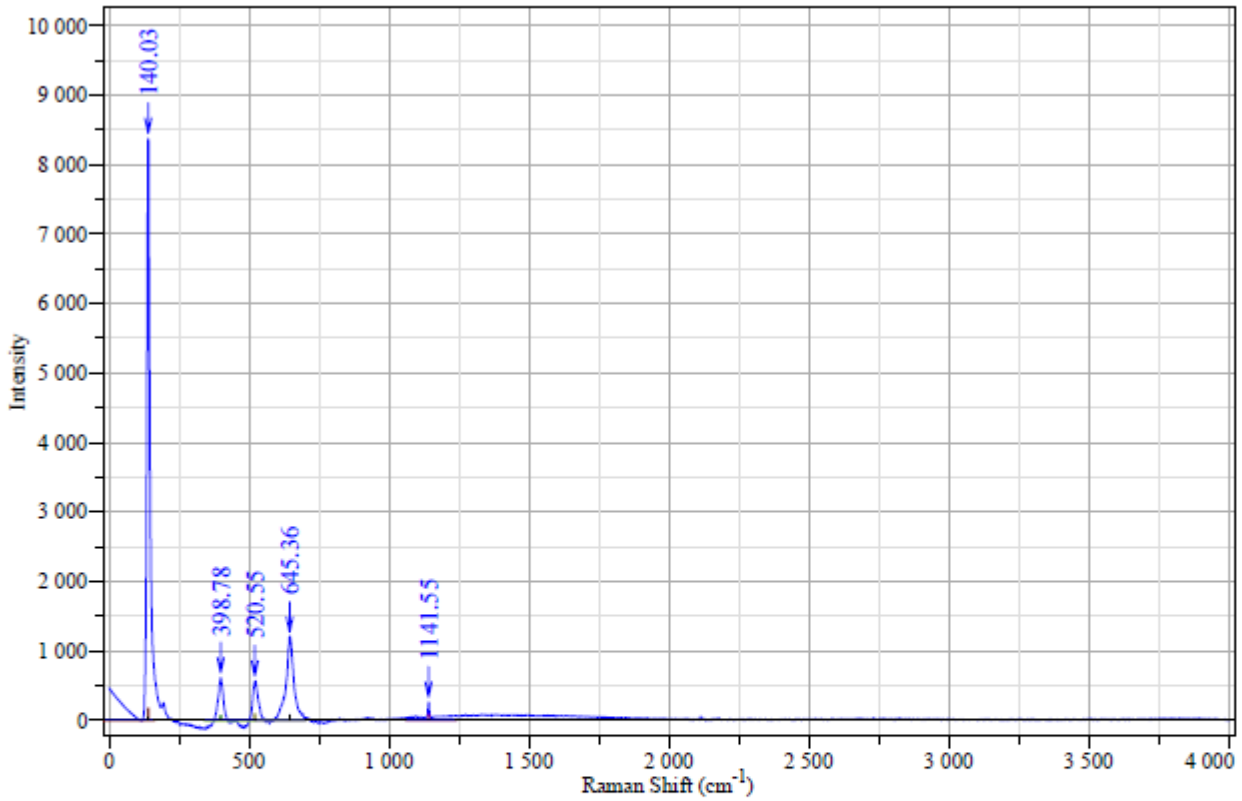
Data File C:\CHEM32\1\DATA\NORAINI\NAZ_12AUG14_5SPL 2014-08-12 12-15-29\105F0501.D
Sample Name: Castor (heterogeneous)

RetTime [min]	Type	Area [pA*s]	Amt/Area	Norm %	Grp	Name
13.331		-	-	-		
13.860		-	-	-		
14.281		-	-	-		
14.804		-	-	-		
15.131		-	-	-		
15.762		-	-	-		
16.113		-	-	-		
16.749		-	-	-		
16.925		-	-	-		
17.046		-	-	-		
17.278		-	-	-		
17.584		-	-	-		
17.851	BB	1352.18311	5.84032e-2	99.728330		
18.254		-	-	-		
18.788		-	-	-		
19.117		-	-	-		
19.700		-	-	-		
19.830		-	-	-		
20.052		-	-	-		
20.286		-	-	-		
20.417		-	-	-		
20.919		-	-	-		
21.051		-	-	-		
21.316		-	-	-		
22.028		-	-	-		
22.127		-	-	-		
23.511		-	-	-		
24.062		-	-	-		
24.191		-	-	-		
Totals :				100.000000		

2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
Warning : Calibrated compound(s) not found

*** End of Report ***



Exposition	1	Slit	100
Accumulation	1	Operator	Hasbullah
Laser	514.53	Sample	NaTiO2
Spectro	Multi	Remark	
Hole	1000	Power	

HORIBA JOBIN YVON