

**COMPARISON OF BIODIESEL SYNTHESIS
RATES WHEN BEING INTENSIFIED WITH
LIQUID EXTRACTION PROCESS**

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**COMPARISON OF BIODIESEL SYNTHESIS
RATES WHEN BEING INTENSIFIED WITH
LIQUID EXTRACTION PROCESS**

ZULHILMI BIN KHAMIS

Thesis submitted in partial fulfilment of the requirements
For the award of the degree of
Bachelor of Chemical Engineering

**Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG**

JULY 2014

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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Name of main supervisor : Mohd Sabri Bin Mahmud
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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Date :

Dedication

I would like to express my gratitude to Allah for giving such a chance to study in this university, learning new things about the course and of course life that wasn't always easy as always. Nevertheless, life must always to keep move on and see the world in a different perspective. I also would like to thanks to all that help and guide me for finishing the thesis on time although facing many difficulties faced during the process of learning. Thank you to my family who always support me from the start of this study.

ACKNOWLEDGEMENT

I would like to thanks the following people and organisations;

- My supervisors Dr. Sabri bin Mahmud for their guidance through an effective well-arranged weekly meeting.
- Centre of Graduate Studies UMP give opportunity to give a chance to participated in research

ABSTRACT

Biodiesel has become more attractive recently because of its environmental benefits and the fact that it is made from renewable resources. The cost of biodiesel, however, is the main hurdle to commercialization of the product. The used cooking oils are used as raw material, adaption of continuous transesterification process and recovery of high quality glycerol from biodiesel by-product (glycerol) are primary options to be considered to lower the cost of biodiesel. In transesterification, esters and glycerol will be formed by the separation of the vegetable oil. In this study, the characterization of the biodiesel synthesis will be made. Palm oil transesterification reaction with ethanol will be conducted with a set of oil-ethanol ratios under the counter current flow system by using the extractive reactor column so that the data obtained can be used to investigate the behaviour of the reacting fluid that flows counter currently with the reactant solvent (reaction rate study). Intensification of the biodiesel production process may offer significant capital and operating cost benefits due to fewer unit operations. In addition, the continuous removal of co-product glycerol from the reactive phase during transesterification promises higher biodiesel yield relative to conventional batch reactors.

This paper presents the study of comparison synthesis rate of production biodiesel when intensified with liquid extraction process. This study also focuses on rate of reaction in production of biodiesel. Two types of reactor are used in this experiment, batch reactor as an ideal reactor and continuous reactor, Extractive Reactor Column. Both reactors use the same reaction which is transesterification to produce biodiesel. To study the synthesis rate of the biodiesel, the experiment was conducted with different sets of ratios in both reactors, 1:6, 1:12 and 1:24. The experiment is conducted at 45-60°C, which is the optimum temperature to produce biodiesel. For the Extractive Reactor Column, it consists of 9 stages of Continuous Stirred Tank Reactor (CSTR) connected in series. The reactants were pumped into the tank with different flow rates according to the desired ratio. The 0.02 mL sample is taken every 30 minutes, which is the reactant enters every stage of the column and the sample is placed in a 1.5 mL vial tube. The sample is then analysed using Gas Chromatographic- Flame Ionization Detector (GC-FID). From the data that has been analysed, a graph of triglyceride is plotted and a graph of rate of reaction is plotted for every ratio and the results are compared to quantify the goodness of fit.

ABSTRAK

Biodiesel telah menjadi lebih menarik baru-baru ini kerana faedah alam sekitar dan hakikat bahawa ia dibuat daripada sumber yang boleh diperbaharui. Biodiesel kos, bagaimanapun adalah halangan utama kepada pengkomersilan produk. Minyak masak yang digunakan digunakan sebagai bahan mentah, proses adaptasi transesterification berterusan dan pemulihan gliserol yang berkualiti tinggi daripada biodiesel oleh-produk (gliserol) adalah pilihan utama yang perlu mempertimbangkan untuk mengurangkan kos biodiesel. Dalam transesterifikasi, ester dan gliserol akan ditubuhkan oleh pemisahan minyak sayur-sayuran. Dalam kajian ini, pencirian sintesis biodiesel akan dibuat. Palm reaksi transesterification minyak dengan etanol akan dijalankan dengan satu set nisbah minyak etanol di bawah sistem aliran kaunter semasa dengan menggunakan ruangan reaktor ekstraktif supaya data yang diperolehi boleh digunakan untuk menyiasat kelakuan cecair bertindak balas yang mengalir kaunter pada masa ini dengan pelarut bahan tindak balas yang (kajian kadar tindak balas). Penumpuan proses pengeluaran biodiesel boleh menawarkan modal dan operasi penting faedah kos kerana operasi unit yang lebih sedikit. Di samping itu, penyingkiran berterusan gliserol bersama produk daripada fasa reaktif semasa transesterifikasi menjanjikan biodiesel yang lebih tinggi berbanding dengan hasil reaktor kelompok konvensional. Kertas kerja ini membentangkan kajian kadar sintesis perbandingan biodiesel pengeluaran apabila dipergiatkan dengan proses pengekstrakan cecair. Kajian ini juga memberi tumpuan kepada kadar tindak balas dalam pengeluaran biodiesel. Dua jenis reaktor yang digunakan dalam eksperimen ini, reaktor kelompok sebagai reaktor ideal dan reaktor berterusan, Ekstraktif Reaktor Column. Kedua-dua reaktor menggunakan reaksi sama iaitu transesterifikasi untuk menghasilkan biodiesel. Untuk mengkaji kadar sintesis biodiesel itu, kajian ini telah dijalankan dengan set nisbah pada kedua-dua reaktor, 1:06, 1:12 dan 1:24. Uji kaji dijalankan pada 45-60 °C ini adalah suhu optimum untuk menghasilkan biodiesel. Untuk Ekstraktif Reaktor Column, ia terdiri daripada 9 peringkat Berterusan reaktor tangki teraduk (CSTR) yang berkaitan dalam siri. Bahan tindak balas telah mengepam ke dalam tangki dengan kadar aliran yang berbeza mengikut nisbah yang dikehendaki. 0.02 mL sampel diambil setiap 30 minit, yang bahan tindak balas adalah memasuki tiap-tiap peringkat tiang dan sampel diletakkan di dalam 1.5 mL tiub botol. Sampel kemudiannya dianalisis dengan menggunakan Kromatografi Gas-Api Pengionan Pengesan (GC-FID). Dari data yang telah menganalisis, graf trigliserida diplot dan graf kadar tindak balas dalam diplotkan untuk setiap nisbah dan membandingkan kebaikan secara kuantiti yang patut.

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1 INTRODUCTION

1.1 Motivation and statement of problem

In recent times, the world has been confronted with energy crisis due to depletion of resources and increased environmental problem. As result many researcher do a research to overcome or reduce this problem, alternative resources is needed to replace the old resources. The new resource must be environmental friendly and also can be renewed. For developing country, fuels of bio-origin such as alcohol, vegetable oil and biomass are important. Such fuels can be used directly, while other need some sort of modification before use as substitute of conventional fuels.(Barnwal & Sharma, 2005).

The rapid decrease in resources of fossil energy and the accumulation of carbon dioxide and other greenhouse gases in the atmosphere is thought to be at the origin of changes in climate, which are suspected to have dramatic consequences on humans and other living organisms. These changes have led to the development of renewable energy sources, sustainable development and eco-friendly concept.(A. H. Demirbas & Demirbas, 2007)

Renewable energy sources (RESs) are also often called alternative energy sources. It is use indigenous resources have the potential to provide energy services with zero or almost zero emissions of both air pollutants and greenhouse gases. Renewable energy is a promising alternative solution because it is clean and environmentally safe and produce lower or negligible levels of greenhouse gases and other pollutants as compared with the fossil energy sources they replace. Approximately half of the global energy supply will come from renewables in 2040, according to the European Renewable Energy Council (2006).

An alternative fuel to petrol diesel, must be technically feasible, economically competitive, environmentally acceptable and easy available. The current alternative diesel fuel can be termed biodiesel. Biodiesel can offer many benefit, including reduction of greenhouse emission, regional development and social structure, especially to developing country.(A. Demirbas, 2009)

Typical raw material of biodiesel are rapeseed oil, canola oil, soybean oil, sunflower oil and palm oil. Vegetable oil are renewable fuels. They have become more attractive recently because of their environmental benefits and the fact that they are made from renewable resources such as palm oil. Vegetable oil is potentially inexhaustible source of energy, energy content in vegetable oil is near to diesel fuel. Besides, some species of plant yielding non-edible oil such as jatropha, karanja and pongamia may play a significant role in providing resources. Both these plants may be grown on massive scale on agricultural or waste land. Oil from non-edible plant have high free fatty acid (FFA) content. FFAs easily react with alkaline catalyst to form soap that prohibits the separation of biodiesel and glycerol. The soaps of FFAs also cause foaming in aqueous media. As result, the soap cause an increasing in viscosity, formation of gels and foams and make the separation of glycerol and biodiesel is prohibits. In term of price, non-edible oil is much more cheaper and very economical compare to edible oil.(A. Demirbas, 2009)

Intensification of the biodiesel production process may offer significant capital and operating cost benefits due to fewer unit operations. In addition, the continuous removal of co-product glycerol from the reactive phase during transesterification promises higher biodiesel yield relative to conventional batch reactors. The application of a novel liquid-liquid extractive reactor with basket-like mixing impellers. Counter current flow of vegetable oil and aqueous ethanol solution in a multistage, mixer-settler type extractive reaction column (XRC), catalysed by alkali base catalyst. The results revealed that the conversion was insensitive to solvent to feed ratios beyond stoichiometric ratio of ethanol-to-triglycerides, thus making the XRC superior to batch process which uses excess alcohol. Increasing number of stages, stirring speed and raffinate recycle ratio improved conversion over the respective ranges studied.(Chesterfield, Rogers, Al-Zaini, & Adesina, 2013)

1.2 Objectives

The following are the objectives of this research:

- To Comparison of Biodiesel Synthesis Rates When Being Intensified with liquid extraction process.
- To study the effect of ratio in production of biodiesel

1.3 Scope of this research

The following are the scope of this research:

- i) Triglyceride sources are from palm oil.
- ii) The solvent is ethanol which it is also the reactant
- iii) The transesterification reaction is conducted in specific range (basically 0.25-1.5wt %) catalyst concentration and specific range of reaction time (normally 20-60 min) in the batch and continuous reactor column
- iv) Fatty Acid Ethyl Ester (FAEE) concentration would be analysed using gas chromatography (GC).
- v) Using two type of reactor like batch and continuous reactor column

2 LITERATURE REVIEW

2.1 Overview

This paper presents the experimental studies on production of biodiesel from vegetable oil. As the world faces the fossil fuel depletion and environmental degradation, alternative fuels is needed to substitute the fossil fuel. The alternative fuels need to be renewable and also environmental friendly. Most suitable fuel to replace petrodiesel is biodiesel.

2.2 Introduction

Biodiesel is more attractive nowadays because it is more environmentally friendly than petro-fuel and the fact biodiesel is renewable resource. Besides having more efficient burning, the emission of CO₂ from biodiesel combustion is lesser than petro-fuel, biodegradable and non-toxic. Chemically, bio-diesel is referred to a mono-alky-ester (in this proposal Fatty Acid Ethyl Ester (FAEE) is to be considered) which is derived from lipid or triglyceride. Biodiesel is now mainly being produced from soybean, rapeseed, and palm oils. (Ramadhs et al., 2004 ; Sarin and Sharma, 2007). The higher heating values (HHVs) of biodiesels are relatively high. The HHVs of biodiesels (39 to 41 MJ/kg) are slightly lower than those of gasoline (46 MJ/kg), petro-diesel (43 MJ/kg), or petroleum (42 MJ/kg), but higher than coal (32 to 37 MJ/kg).

2.3 Transesterification Methods

Transesterification or also known as alcoholysis is involving reaction between vegetable oils with alcohol to form ester and glycerol. Presence of catalysis is to improve the rate of reaction and yield. As the reaction is reversible, excess alcohol is used to shift the equilibrium to the product. Among alcohol that commercially used in industry is methanol and ethanol, because of its low cost, physical and chemical properties.

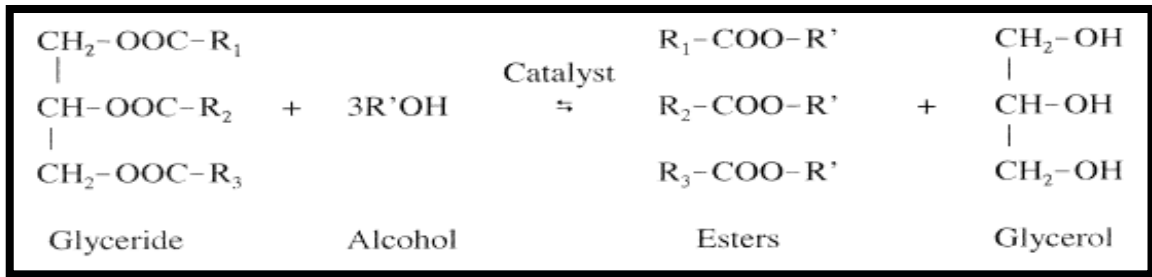


Figure 2-1: Tranesterification Method

The reaction can be catalysed by acid, alkalis and enzymes. Mostly, alkalis-catalyzed transesterification reaction is much faster than acid-catalyzed one and also it is most often in industries nowadays. Alkalis catalyst is much better than acid catalyst because of its low cost and good conversion in short reaction rate. Catalyst such as NaOH and KOH is commercially used. NaOH and KOH are homogeneous. Its easily dissolves in alcohol.

2.4 Problems in Biodiesel Industries

The implementation of using biodiesel in transportation is still lacking due to the economic factors. The price of biodiesel at the global market cannot be at least equivalent to the existing petro-fuels. The followings are the probable causes:-

2.4.1 Expensive feedstock

Production of methyl ester and ethyl ester from edible oil is far more expensive than diesel fuels due to the relatively high cost of vegetable oil. Biodiesel is a technologically feasible alternative to petrodiesel, but nowadays biodiesel costs 1.5 to 3 times more than fossil diesel in developed countries even in small quantities.. The competitiveness of biodiesel to petrodiesel depends on the fuel taxation rates and policies of which most countries do not provide them. In addition, the competitiveness of biodiesel relies on the price of the biomass feedstock and costs associated with the conversion technology. The cost per gallon for the feedstock is shown at table 2.1.

Table 2-1: Price per Gallon Feedstock

Feedstock	Cost (\$/gallon)
Animal fats	1.35
Repeseed oil	1.46
Sunflower oil	2.35
Soybean oil	1.26

Due to the expensive feedstock, alternative method is developed. Non-edible oil is been use instead of edible oil. The non-edible oil such as *J.curcas*, *M.indica*, *Fircus elastic*, *Azardirachta indica*, *Calophyllum inophyllum jatropa*, rubber seed and tall oil microalgae are avaible in developing countries and are very economical comparable to edible oil. *J.curcas* and *P.pinnata* are most suitable for the purpose of producing renewable fuel as biodiesel this is because its have high oil content about 25-30%.

2.4.2 High free fatty acids (FFA) in the non-edible oil.

Since more than 95% of the biodiesel is synthesized from edible oil, there are many claims that a lot of problems may arise. By converting edible oils into biodiesel, food resources are actually being converted into automotive fuels. It is believed that large-scale production of biodiesel from edible oils may bring global imbalance to the food supply and demand market. Recently, environmentalists have started to debate on the negative impact of biodiesel production from edible oil (Butler, 2006). They claimed that the expansion of oil crop plantations for biodiesel production on a large scale may increase deforestation in countries like Malaysia, Indonesia and Brazil. Furthermore, the line between food and fuel economies is blurred as both of the fields are competing for the same oil resources. In other words, biodiesel is competing limited land availability with food industry for plantation of oil crops. Arable land that would otherwise have been used to grow food would instead be used to grow fuel (Anonymous, 2004). In fact, this trend is already being observed in certain part of this world. There has been significant expansion in the plantation of oil crops for biodiesel in the past few years in order to fulfill the continuous increasing demand of biodiesel. Free fatty acid (FFA) has significant effect during the transesterification reaction. High free fatty acids FFA content will

cause soap formation and the separation of products will be exceedingly difficult, and as a result, low yield of biodiesel product would be obtained. It is important to determine the FFA content of the non-edible oil.

2.4.3 Consummation of chemical catalysts by forming a plenty of emulsion phase during the reaction.

The impurities in the non-edible oil such as free fatty acids and saturated fats would react with the conversional catalysts to form an emulsion phase. The appearance of the emulsion phase in industrial processes would consummate the final product. The product would be hard to be refined and thus the cost increases. Emulsion is a mixture of two liquids that would ordinarily not mix together

2.5 Liquid Extraction Process

Liquid-liquid extraction is a process for separating the components (solute) of a liquid (the feed) by contact with a second liquid phase of solvent that is immiscible. The process takes advantage of differences in the chemical properties of the feed components, such as differences in polarity and hydrophobic and hydrophilic character, to separate them. To be more specific, the transfer of components from one phase to the other is driven by a deviation from thermodynamic equilibrium, and the equilibrium state depends on the nature of the interactions between the feed components and the solvent phase. The potential for separating the feed components is determined by differences in these interactions

2.6 Process Intensification in Biodiesel Synthesis

Since triglycerides are esters in which fatty acids are chemically bound with a tertiary alcohol namely glycerol, their solubility is limited to non-polar organic solvent such as the liquids from the group of alkanes and alkenes, and the compounds with the amount of carbon per molecule higher than 4 ($> C_4$) of ketones, alcohols and aldehydes. This phenomenon renders inefficient contact of the reacting molecule in a process as the alcohols used to produce the biodiesel are partially soluble in the vegetable oil the place where the reaction of transesterification occurs.

Several efforts were reported such as Static Mixer, Micro-Chanel Reactor, Oscillatory flow reactor, Cavitation reactor, Membrane reactors, Reactive distillation and Centrifugal contactors. Among those intensifications, the method Centrifugal contactors method is the best for the production of biodiesel. Centrifugal contactor is more commercially use than other method because it is simple and the yield of biodiesel is higher.

The centrifugal contactor is another process intensification technology because it integrates reaction and centrifugal separation into a unit. It consists of a mixing zone and a separating zone.

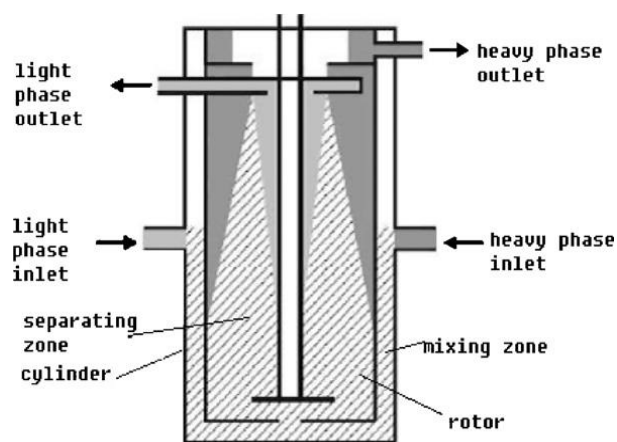


Figure 2-2: Centrifugal Reactor

As the rotor in the contactor is rapidly rotating within a stationary cylinder, it can achieve intense mixing and good mass transfer by high shear stress and quick phase separation by high centrifugal force simultaneously. However, the residence time in a conventional centrifugal contactor is as low as about 1min and cannot allow reaction to reach equilibrium. Comparisons among the intensification method are shown at table 2.

Table 2-2: Comparison of process intensification technologies for continuous biodiesel production with conventional stirred tank reactors.

	Residence time	Operating and capital cost	Temperature control	Current status
Static mixer	~30 min	Low	Good	Lab scale
Micro-channel reactor	28 s–several minutes	Low	Good	Lab scale
Oscillatory flow reactor	30 min	Low	Good	Pilot plant
Cavitation reactor	Microseconds–several seconds	Low	Good	Commercial scale
Spinning tube in tube reactor	<1 min	Low	Good	Commercial scale
Microwave reactor	Several minutes	Low	Good	Lab scale
Membrane reactor	1–3 h	Lower	Easy	Pilot plant
Reactive distillation	Several minutes	Lower	Easy	Pilot plant
Centrifugal contactor	~1min	Lower	Easy	Commercial scale

2.7 Potential of liquid-liquid extraction process in solving the problems in the biodiesel synthesis

The process intensification is important to attain the mass transfer during the reaction for simpler and more economical operations. These technologies either utilize novel reactors or coupled reaction/separation processes. Reaction rate is greatly enhanced and thus residence time may be reduced. Some of the technologies have been applied successfully in commercial production.

Biodiesel known as fatty acid ethyl ester (FAEE) is produced from transesterification between vegetable oil (triglyceride) and ethanol. As the reaction goes, byproduct known as glycerol is needed to be separated from ester. Function centrifugal contactor separator (CCS) is to separate glycerol from ester. After the reaction achieved the steady state, the glycerol with unreacted ethanol flowed out of the heavy phase outlet while the fatty acid ethyl ester with unreacted oil flowed out of the CCS through the light phase exit. Since the homogeneous base-catalyst was used in the reaction, the dissolved catalyst was present in both outlet phases.

3 MATERIALS AND METHODS

3.1 Overview

This paper show how the extractive reactor column in production of biodiesel and how extractive reactor column is contribute to increase the yield of biodiesel and also comparing the yield between batch production and extractive reactor production. In this chapter, it is discussed more specifically about how to run the experiment. The first part is the research design. It is explained about the procedures and materials preparation. As for raw material, it is the first thing essentially to reconsider which is in the experiment that we already used to run-up the experiment. It is divided into three parts of material preparation like raw material, solvent and catalyst. Experiment methodology is conducted to give the pathway how run-up the experiment from initial to the final. Moreover, It is included how you set your experimental data for that experiment. The right choosing of the equipment is also the most important factor and during the whole operation conducted. All the process compulsory to conduct under safety condition and in the authorization personal observation. Lastly, it is also need to follow all rules and procedures of FKKSA's lab that emphasized the 5'S work culture. Every detail steps were discussed in next subchapter.

3.2 Introduction

This paper presents a comparison of yield between batch and extractive reactor column in production of biodiesel. Set of ratio is been made to see the rate of reaction for both batch and extractive reactor column with the same retention time and mixing impeller velocity.

3.3 Chemicals

In this experiment, the main raw material that will be used is palm oil. We can get easily from market, supermarket, hyperstore and etc with the comprehensive prices. Then, ethanol also needed as the solvent and reactant in this process. It is also easily to get from the extraction process of the plant like from corn. Ethanol is hardly to get because it is from the petroleum's cracking complex process

Catalyst is very important during transesterification reaction. Different catalyst gives different effect on product yield but the most important thing is it will enhance the speed rate of reaction and the energy of activation. In this research, the alkaline based catalyst is used. It is potassium hydroxide that prepared and mixed with ethanol. During handling the catalyst, it is important to remember that it needs to be stored in a dry place, wearing mask while weighing the catalyst, bulk storage of chemical solution and etc.

Ethanol is selected as in this experiment, the solvent alcohol will be mixed with alkaline based catalyst to form catalyst solution that placed the medium to chemical reaction activation. This mixture will be placed in the conical flask. During handling ethanol, the most important rule is do not expose openly to atmosphere due to its easily volatilize. Furthermore, please wash thoroughly after handling it, ground and bond containers when transferring material, empty containers retain product residue, keep container tightly closed, avoid contact with heat, sparks and flame, and handle the experiment in a chemical fume hood. After mixing the solvent and catalyst, it will be stirred continuously using magnetic stirrer that will be placed on the hotplate stirrer until well mixed. The time mixing that will be proposed to conduct that part is around 30-60 minutes depending on the amount of catalyst.

3.4 Extractive reactor column

Ethanol and oil will feed to the reactor counter currently. Catalyst, KOH or NaOH will be dissolved first into the ethanol before entering to the reactor. Then, both oil and ethanol are immiscible and thus stirrer is needed. As the reactant react, it will produce glycerol and fatty acid ethyl ester (FAEE). Glycerol will be extracted and will remove at the bottom of the reactor as the glycerol is more dense than ethanol, while fatty acid ethyl ester will be at the top of the reactor as a raffinate.

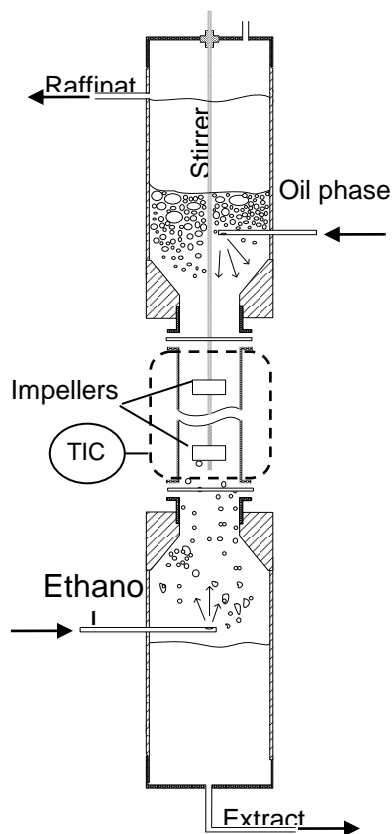


Figure 3-1: Extractive reactor Column

3.5 Gas Chromatography (GC) Analysis

The purity of biodiesel product, denoted by its ester content, is defined as the weight percentage of ethyl ester in the final product after the purification and the removal of glycerine, excess ethanol, alkaline catalyst and soap in the product mixture. In this experiment, the ethyl ester content was analysed using Gas Chromatography (GC). It was employing column HT-S/DB wax, oven ramping, injection 1 micro lid and detector of 280°C. Usually objective of gas chromatography is to separate the various molecular entities of a sample, and this is often accomplished readily with capillary GC. Derivatization, a separate sample treatment which makes substances essentially non-volatile, more volatile, makes possible the determination of many of the impurities by gas chromatography. Finally, a graph of excess ethanol recovery process versus the crude oil conversion will plotted as the final result. Below, showed the gas chromatography (GC) analysis of glycerin in biodiesel, conforming to ASTM D6584



Figure 3-2: Gas Chromatography Flame Ionization Detector

Table 3-1: Condition of Gas Chromatography (GC)

Conditions	
Column:	MET-Biodiesel, 14 m x 0.53 mm I.D., 0.16 μm with integrated 2 m x 0.53 mm I.D. guard (28668-U)
Oven:	50 $^{\circ}\text{C}$ (1 min.), 15 $^{\circ}\text{C}/\text{min.}$ to 180 $^{\circ}\text{C}$, 7 $^{\circ}\text{C}/\text{min.}$ to 230 $^{\circ}\text{C}$, 30 $^{\circ}\text{C}/\text{min.}$ to 380 $^{\circ}\text{C}$ (10 min.)
Det.:	FID, 380 $^{\circ}\text{C}$
Carrier Gas:	helium, 3.0 mL/min.
Injection:	1 μL , cold on-column
Sample:	B100 Biodiesel plus Butanetriol Internal Standard (44896-U) and Tricaprin Internal Standard (44897-U), derivatized with MSTFA (394866) then diluted in heptane

4 RESULT AND DISCUSSION

4.1 Overview

This paper present the all the result obtain during the research and experiment. First step of the research is constructing standard curve, standard curve is vital for every experiment. It is necessary construct it first before the experiment is begin. The main research begin when the experiment of batch reactor begin. Batch reactor is set as a ideal reactor and separator. After that, experiment using continuous reactor is begin. All the sample is analyse using Gas Chromatographic.

4.2 Construction Standard Curve

The standard curve is construct using cooking oil and n-Heptane as a solvent with different set of ratio. Table 4.1 shows the dilution of cooking oil using n-Heptane.

Table 4-1: Standard Curve Preparation

Concentration Ci,ppm	Average area under peak,Ai, pA*s	Ai/Aref	Ci/Cref
16393.44	5224.275	0.970109967	0.819672
17857.14	6610.575	1.22753582	0.892857
19607.84	7340.62	1.36309988	0.980392
21739.13	8075.84	1.499624901	1.0869565
27777.78	12148.26	2.255843751	1.388889

From the data above, a standard curve is drawn. It is necessary to draw the standard curve to interpret the data from the GC analysis data. The graph below shown the standard curve.

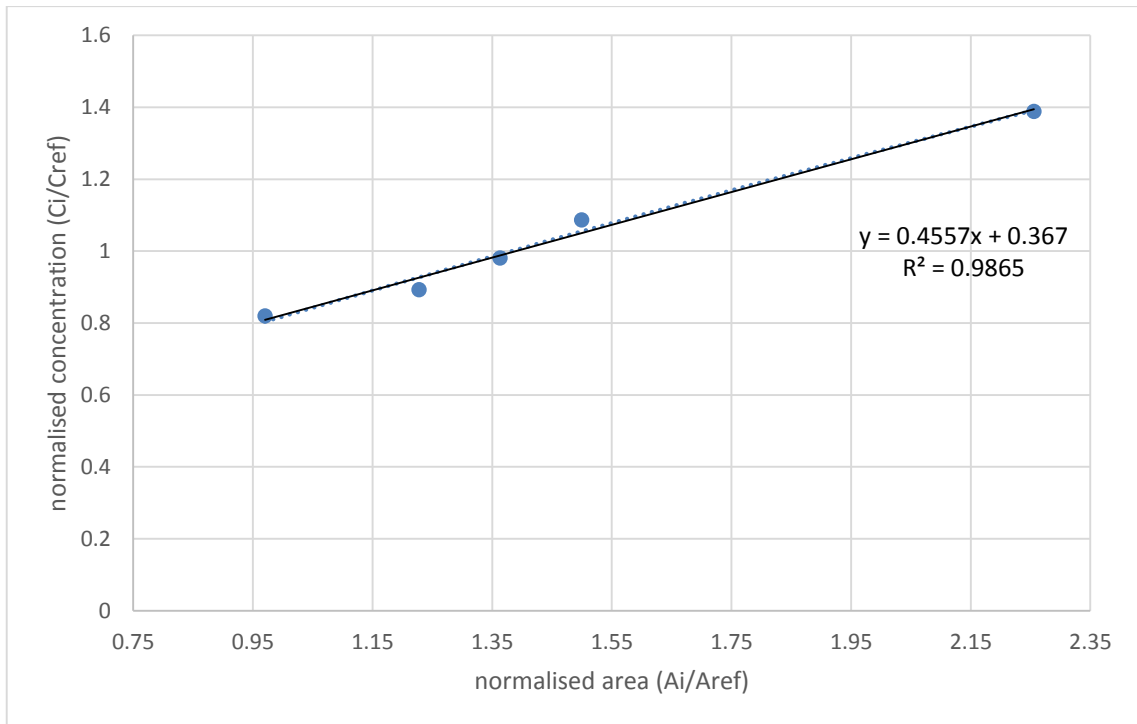


Figure 4-1: Standard Curve

4.3 Batch Reactor Result

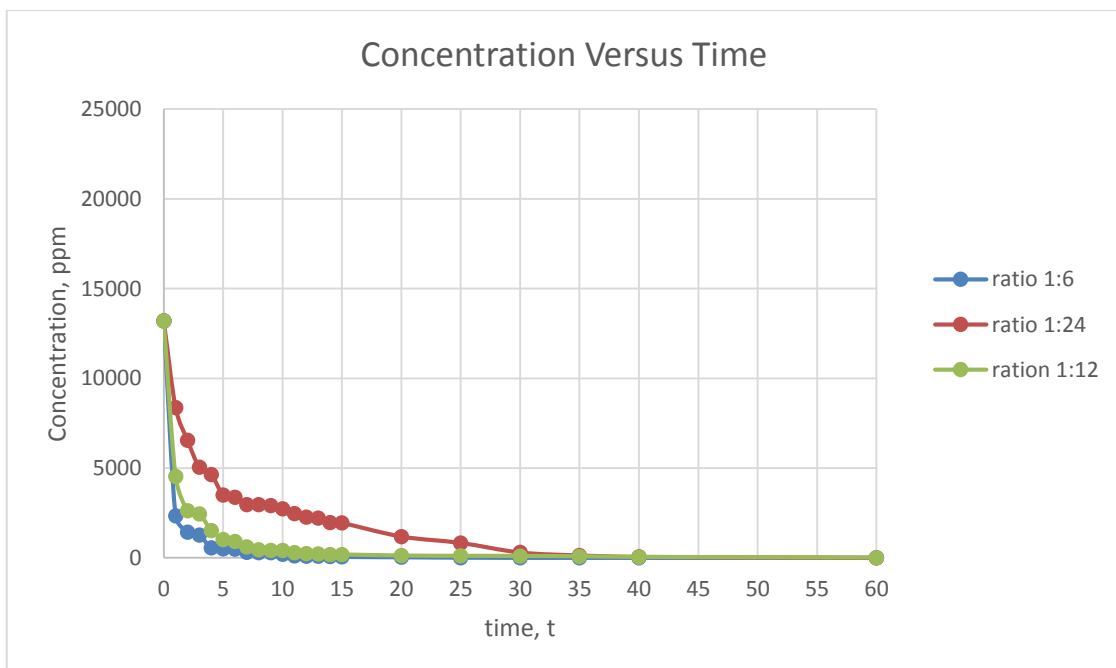


Figure 4-2: Concentration of Triglyceride versus Time

The data above shown that, triglyceride is decreasing when the time is increasing, this shown that the triglyceride is been used in reaction to produce biodiesel and glycerol.

Several ratio is been conducted which is 1:6, 1:12 and 1:24. According to the figure above, ratio 1:6 is rapidly decreasing by time compare to other ratio. In term of conversion, 1:24 is give better conversion than other ratio, which is 98.3% of conversion. For industrial purpose, it is better to use 1:6 compare to the other ratio.

4.4 Extractive Reactor Column Result

The data from this experiment is consider irrelevant cause by the impropriate changes in method of analysis. As result, the data cannot been interpreted in graph to show the difference in synthesis rate of biodiesel. Therefore, there is no discussion in this section which is indicate failure in achieving objective of the research.

Some picture below shown that the experiment have been conducted and there is reaction during the experiment is conducted. The time take for this experiment to be conduct is about 15 hours.

For general idea of this experiment, the reactor are consist of 9 stages of continuous reactor and 2 decanter, top and bottom. The cooking oil is place in Tank 1 and mixture of Ethanol and Potassium Hydroxide is place in Tank 2. Both tank is pump to the reactor with 10 mL/min and 14 mL/min respectively. Bottom decanter is pump with ethoxide for a head-start. Later than, the oil is pump from the top and ethoxide from the bottom. The sample is taken when the reactant is reach the stages.

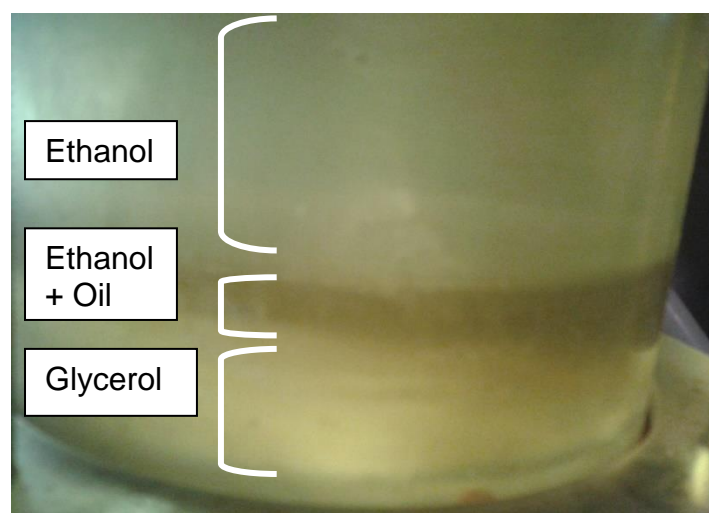


Figure 4-3 Bottom Decanter at 3 Hours

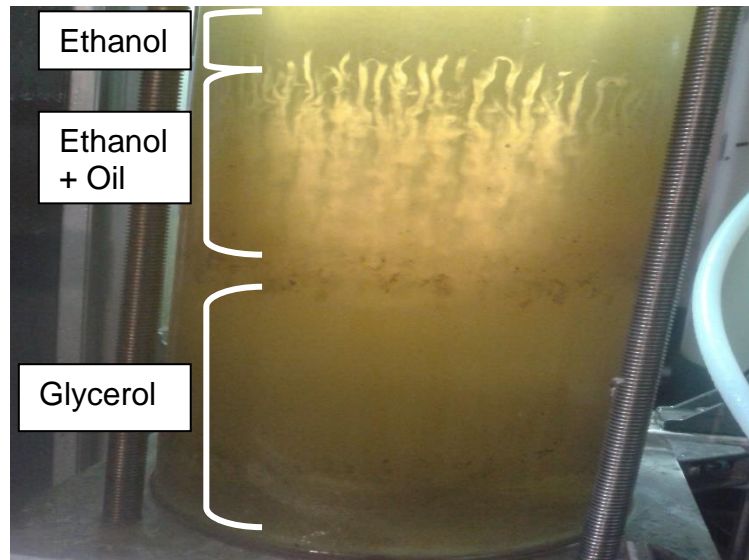


Figure 2-4: Bottom Decanter at 4 hours

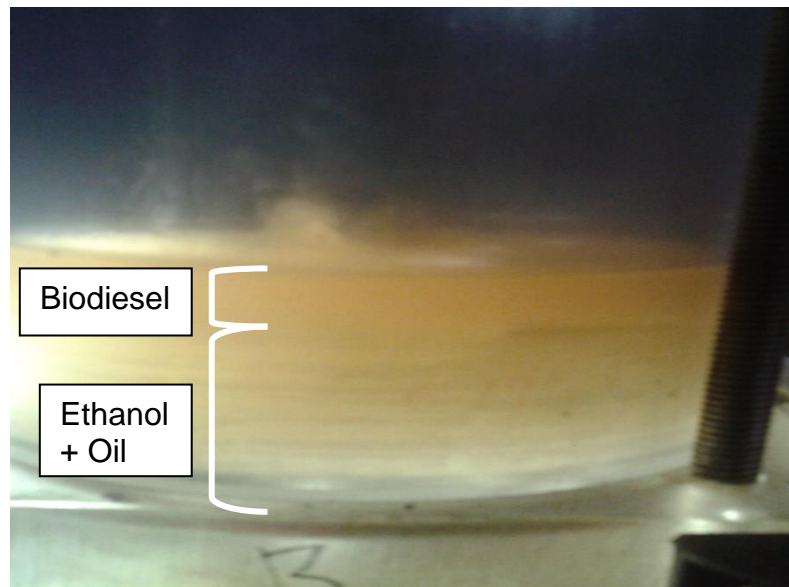


Figure 4-5 Top Decanter at 12 hours

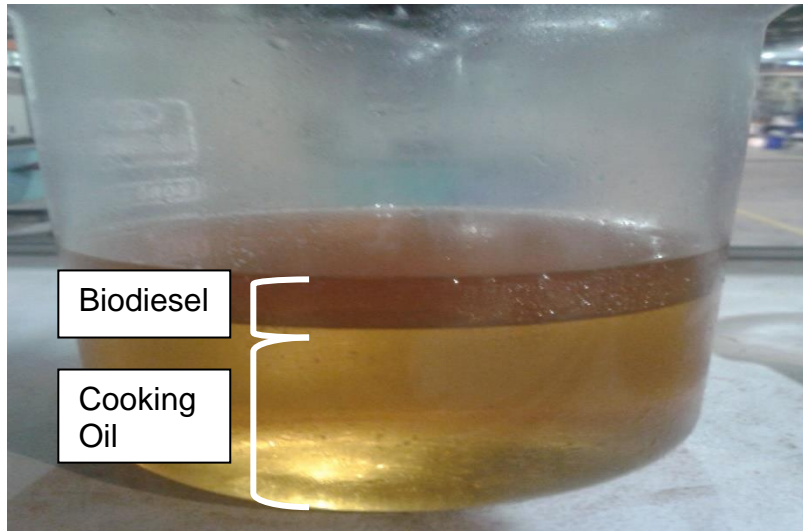


Figure 4-6 Sample from Top Decanter

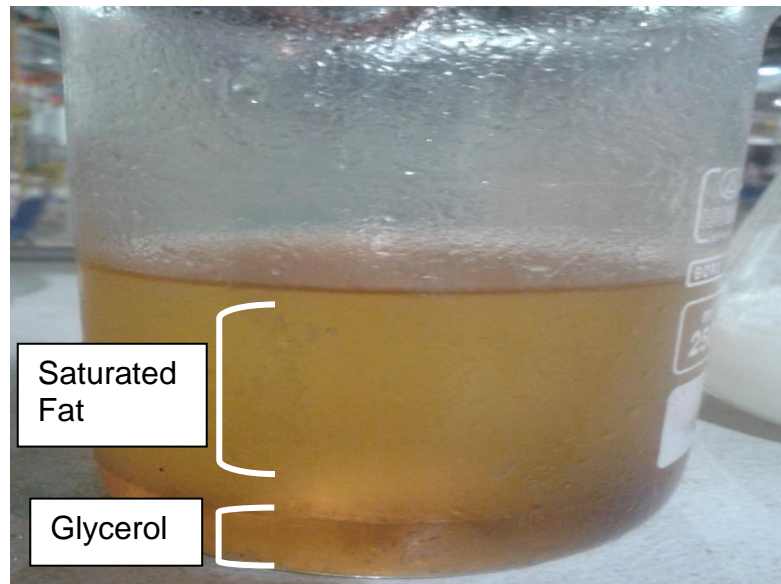


Figure 4-7 Sample from Bottom Decanter

5 CONCLUSION

5.1 Conclusion

Formation of monoglyceride of the biodiesel decreases dramatically in the first period of the reaction but increased slowly in the last 10 minute period of the 30 minutes reactions. The first period indicates that the biodiesel were rapidly form until it reaches the point the reaction becomes constant and steady where reversible reaction occur slowly in the last period of the reaction. Miscible of fatty acid ethyl ester in polar organic rather than glycerol caused the attraction towards ethanol which lead to reversible reaction.

For the Reactive Extraction Column, caused by the unsuitable method in the analysis step, the analysis from Gas Chromatography result for the pilot agitate column obtained were irrelevant and the synthesis rate cannot be determine, this study was consider to be fail in fulfilling the objectives of the study

5.2 Future Work

For future work, it is recommended to put better control of the reactor and for future research, efficiency of the separator is should be determined because this reactor is a intensified reactor and separator.

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APPENDICES

Result from Gas Chromatographic for Batch Reactor

