CHARACTERIZATION OF THE ETHANOL-TRIGLYCERIDE TRANSESTERIFICATION REACTION UNDER COUNTER CURRENT FLOW SYSTEM

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CHARACTERIZATION OF THE ETHANOL-TRIGLYCERIDE TRANSESTERIFICATION REACTION UNDER COUNTER CURRENT FLOW SYSTEM

CHONG EE RONG

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Pure)

Faculty of Chemical & Natural Resources Engineering
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JAN 2014

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 (Pure).

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Date : 28 JAN 2014

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.

Signature :
Name : CHONG EE RONG
ID Number : KA10134
Date : JAN 2014

Dedication

For my mum Ng Choy Kuan. Without her I would not be here. Actually mum doesn’t read, so if someone doesn’t tell her about this, she will never know.

ACKNOWLEDGEMENT

I cannot express enough thanks to my supervisor for his continued support and encouragement: Dr. Mohd Sabri bin Mahmud who without his help and guidance this research would not have been completed. Your encouragement when the times got rough are much appreciated and duly noted. Thanks for your guidance through an effective well-arranged weekly meeting.

Besides, I offer my sincere appreciation for the learning opportunities provided by Centre of Graduate Studies UMP for providing this program for undergraduate student to learn and gain more knowledge.

Finally, I wish to thank my parents for their tremendous contributions and support both morally and financially towards the completion of this research. I also show my gratitude to my friends and all who contributed in one way or the other in the course of the research.

ABSTRACT

Biodiesel is a mono-alkyl ester of vegetable oil, animal fat and recycled cooking oil. It has been recognized worldwide as an alternative source to replace the known petroleum reserves that are limited and eventually run out as to many aspects to be concerned like environmental and sustainability issues. It is commonly prepared by transesterification of triglycerides or the esterification of free fatty acid with methanol or ethanol by stirring and accelerated by the presence of base or acidic catalyst. In this study, palm oil transesterification reaction with ethanol will be conducted with a set of cooking oil-absolute 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. Different composition of the cooking oil and absolute ethanol were mixed and observed using viscometer under different RPM. Torque, viscosity, shear strength and shear stress were observed, recorded and plotted in figure 3, figure 4, figure 5 and figure 6. The torque was decreasing when the ratio of the cooking was decreasing. For RPM 50, torque of ratio (cooking oil:absolute ethanol) 8:1 was 8.3% decreasing to 0.2% when the ratio was (cooking oil:absolute ethanol) 1:8. The viscosity was decreasing when the ratio of cooking oil decreasing as well. For RPM 50, viscosity of ratio (cooking oil:absolute ethanol) 8:1 was 50.4 mPaS decreasing to 1.8 mPas when the ratio was (cooking oil:absolute ethanol) 1:8. The shear strength was decreasing as well when the ratio of cooking was decreasing. For RPM 50, shear strength of ratio (cooking oil:absolute ethanol) 8:1 was 0.85 N/m^2 decreasing to 0.02 N/m^2 when the ratio was (cooking oil:absolute ethanol) 1:8. However, the shear rate of each ratio of reactant mixture had no changes when the ratio was changing. Unfortunately, the extractive reactor column had some problems when the experiment was carried out. No data can be obtained from the experiment. From the observation, few phenomena can be seen through the photos. The white colour fatty acid that was formed after 1 day. Besides, figure 9 shown that the corroded flow meter by absolute ethanol which contained KOH. In figure 10, the broken flow meter which affect the research hardly to continue.

TABLE OF CONTENTS

SUPERVISOR'S DECLARATION IV
STUDENT'S DECLARATION V
Dedication VI
ACKNOWLEDGEMENT VII
ABSTRACT VIII
TABLE OF CONTENTS IX
LIST OF FIGURES XI
LIST OF TABLES XII
1 INTRODUCTION 1
  1.1 Background of study 1
  1.2 Problem statement 2
  1.3 Objectives 2
  1.4 Scope of this research 2
  1.5 Main contribution of this work 3
2 LITERATURE REVIEW 4
  2.1 Overview 4
  2.2 Introduction 4
  2.3 Fundamental of biodiesel production - Transesterification 7
  2.4 Basic industrial process of biodiesel production 9
  2.5 Biodiesel 11
  2.6 Torque 12
  2.7 Viscosity 12
  2.8 Shear Stress 13
  2.9 Shear rate 13
  2.10 Dimensionless numbers 14
    2.10.1 Weber number 14
    2.10.2 Froude Number 14
    2.10.3 Weeping point 15
  2.11 Rheometer 15
3 MATERIALS AND METHODS 16
  3.1 Overview 16
  3.2 Introduction 16
  3.3 Chemicals 17
  3.4 Sample preparation 17
    3.4.1 Characterization of cooking oil and absolute ethanol mixture (Torque, viscosity, shear strength and shear rate) 17
    3.4.2 Extractive Reactor Column 17
  3.5 Characterization of ethanol-triglyceride 19
  3.6 Extractive Reactor Column 20
    3.6.1 Preliminary run with water 20
    3.6.2 Washing with absolute ethanol 20
    3.6.3 Investigation of the reacting liquid 20
  3.7 Chromatographic analysis of tripalmitolein by using agilent Gas Chromatography (GC) 22

4 RESUTLS AND DISCUSSIONS 23
4.1 Overview 23
4.2 Introduction 23
4.3 Characterization of ethanol-trigylceride 23
4.4 Extractive Reactor Column 29
4.5 Fatty acid which formed after 1 night left over 30
5 CONCLUSION AND RECOMMENDATION 32
5.1 Conclusion 32
5.2 Recommendations 32

LIST OF FIGURES

Figure 1: The fundamental equation to produce biodiesel production by using transesterification. 7

Figure 2: The basic flow diagram of conventional biodiesel production. 9

Figure 3: Dissolving KOH pallets using magnetic stirrer 18

Figure 4: Absolute ethanol which contained dissolved KOH pellets 18

Figure 5: Brookfield Viscometer 19

Figure 6: Extractive Reactor Column 21

Figure 7: Top decanter which was filled with reactant 21

Figure 8: The flow rate of cooking oil (FI 4) and absolute ethanol(FI 5) set at ratio 6:1 which is “50” and “300 22

Figure 9: Graph of RPM vs ratio for torque 26

Figure 10: Graph of RPM vs ratio for viscosity 27

Figure 11: Graph of RPM vs ratio for shear strength 27

Figure 12: Graph of RPM vs ratio for shear rate 28

Figure 13: Flow meter that was broken caused by the over pressure created by the pump due to the fatty acid stuck in it. 29

Figure 14: Flow meter that was corroded by the absolute ethanol which contained KOH 30

Figure 15: Fatty Acid which produced after 1 night left over in top decanter 30

Figure 16: Fatty Acid which produced after 1 night left over in bottom decanter 31

Figure 17: The fatty acid slowly melted from time to time when the heater is turn on. 31

LIST OF TABLES

Table 1: Variables of studies 3
Table 2: Global renewable energy scenario by 2040 6
Table 3: Cooking Oil : Absolute ethanol (8:1) 23
Table 4: Cooking oil : Absolute ethanol (7:2) 24
Table 5: Cooking oil : Absolute ethanol (6:3) 24
Table 6: Cooking oil : Absolute ethanol (5:4) 24
Table 7: Cooking oil : Absolute ethanol (4:5) 25
Table 8: Cooking oil : Absolute ethanol (3:6) 25
Table 9: Cooking oil : Absolute ethanol (2:7) 25
Table 10: Cooking oil : Absolute ethanol (1:8) 26

1 INTRODUCTION

1.1 Background of study

Biodiesel is a mixture of fatty acid methyl or ethyl esters, mainly obtained through the transesterification of fats and oils (Cadavid, Godoy-Silva, Narvaez, Camargo, & Fonteix, 2013). Biodiesel has potential as an alternative source for petroleum diesel because of the increasing worldwide concern for environmental sustainability and depleting petroleum resources. Methanol has been commonly used alcohol as acyl acceptor for biodiesel production. However, by using ethanol as the acyl acceptor for biodiesel production is getting increasing attention mainly due to advantages of ethanol of being less toxic than methanol. Besides, ethanol can be obtained from sugar- and starch-based feedstock and lignocellulose biomass, therefore it achieves total independence from petroleum-based alcohols. Another attractive characteristics of biodiesel produced from ethanol are that FAEEs have higher heat content and cetane number compared to methyl esters (FAMEs) due to the extra carbon contained in ethanol (Li, Xu, Du, Li, & Liu, 2013). Therefore using ethanol as the acyl acceptor for biodiesel (fatty acid ethyl esters, FAEEs) production is drawing increasing interest in recent years.

Most commercial biodiesel productions use batch stirred tank reactors. However, these processes have disadvantages in the reaction step, associated with mass transfer and chemical equilibrium limitations, as well as problems in the separation stages, especially when poor quality oils are used as feedstock. Chemical equilibrium, mass transfer limitations and the maximum total glycerol content of the final product, which can be as low as 0.24% w/w, in addition to other quality requirements set forth in biodiesel standards, necessitate the search for process strategies to increase oil conversion and yield to FAEE (Cadavid et al., 2013). Thus, this research is aims to investigate the behaviour of the reactant liquid that flows counter currently with the reactant solvent.

1.2 Problem statement

When the 2 reactants cooking oil and absolute ethanol flowing counter currently through the extractive reactor column, the cooking oil from the top decanter might flows too fast to the bottom decanter which causes the reaction time to be less. This phenomenon will definitely be a significant impact the reaction rate limitation of the process. Therefore, the 9 stages in the column consists of stirrer which will keep on stirring to retain the cooking oil from keep flowing down too fast so that the cooking oil got enough time to react with the absolute ethanol.

1.3 Objectives

The objectives of this study is to investigate the behaviour of the reacting liquid that flows counter currently with the reactant solvent that is reaction rate study. Besides, this study is to obtain important dimensionless numbers for the agitated column such as Weber number, Weeping point and Froude number.

1.4 Scope of this research

1. Flow rate
2. Ratio between oil and ethanol
3. Characterization of reactant mixture
    - I. Torque
    - II. Viscosity
    - III. Shear stress
    - IV. Shear rate

Constant Variables	Manipulated Variables	Responding Variables
Stirring speed	Flow rate : min, average, max	Concentration of Tripalmitolein
Temperature	Ratio between oil and ethanol	Outlet flow rate
Size of hole of each plate		Liquid hold up for total volume
Number of stages : 9		

Table 1: Variables of studies

1.5 Main contribution of this work

The following are the contributions:

- Universiti Malaysia Pahang Undergraduate Research Program Unit
- Universiti Malaysia Pahang Chemical Laboratory

2 LITERATURE REVIEW

2.1 *Overview*

This paper presents the experimental studies of liquid-liquid stirred extractive reactor column agitated by an impeller operating at number stages of nine. Four reactions can potentially be used in mass biodiesel production i.e. pyrolysis by thermal cracking, dilution with hydrocarbons (blending), emulsification and transesterification. However, due to the degree of similarity of the product properties to the existing petro-diesel and low energy consumption, only transesterification is currently practised in industries (Chongkhong et al. 2007; Demirbas and Karslioglu 2007; Leung et al. 2010; Ma and Hanna 1999). In this study, biodiesel is manufactured by transesterification of fats and oils. The triglycerides are being extracted from palm oil in the presence of alkaline catalyst at atmospheric pressure and at temperature of approximately 60 to 70ºC with an excess ethanol. In the end of process, the product is left to settle down. There will be 2 layers seen after the product is settled down. For this study, the most important thing is that the flow of the reactant in the column is by using counter current flow.

2.2 *Introduction*

For the last century, petroleum derived fuels have been the main source of the world's energy. However, it is forecasted that fossil fuel will be depleted in the close future. In addition to that, environmental concerns have trigged the inspection of alternative energy sources. Future estimations indicate the economics and energy needs will rise the emphasis on the production of synthetic fuels derived from non-petroleum sources, including biomass and waste products among others (Ghassan, Al-Widyan, & Al-Shyouck, 2002).

The higher public awareness in recent years of the impacts of fossil fuel emissions on the environment and their potential health hazards triggered the government to impose restrictions on fossil combustion emissions. One way to solve the problem mentioned above is to look for alternative and renewable energy sources (Demirbas, 2008).

Renewable energy sources (RESs) are also often called alternative energy sources. RESs that use native resources have the possible to deliver energy services with zero or almost zero discharges of both air pollutants and greenhouse gases. Renewable energy technologies produce marketable energy by converting natural materials into useful forms of energy (Demirbas, 2008). RESs are resulting from those natural, mechanical, thermal, and development processes that repeat themselves within our lifetime and may be relied upon to produce expectable quantities of energy when essential. Renewable energy scenarios depend on environmental protection, which is a vigorous characteristic of sustainable development. World biomass production is estimated at 146 billion metric tons a year, comprised mostly of wild plant growth. Worldwide biomass ranks fourth as an energy source, providing approximately 14% of the world’s energy needs. Biomass now represents only 3% of primary energy consumption in commercialised countries. However, much of the rustic population in developing countries, which represents about 50% of the world’s population, relies on biomass, mostly in the form of wood, for fuel (Demirbas, 2008).

Renewable energy is an auspicious alternative solution because it is clean and environmentally safe. RESs also produce lower or negligible levels of greenhouse gases and other pollutants as compared with the fossil energy sources they change. Table 2 shows the global renewable energy scenario by 2040. About half of the global energy supply will come from renewables in 2040, according to the European Renewable Energy Council (2006). The major growths in renewable energy production will be witnessed in photovoltaic (from 0.2 to 784 Mtoe) and wind energy (from 4.7 to 688 Mtoe) between 2001 and 2040 (Demirbas, 2008).

	2001	2010	2020	2030	2040

Total consumption (million ton oil equivalent)	10,038	10,549	11,425	12,352	13,310
Biomass	1,080	1,313	1,791	2,483	3,271
Large hydro	22.7	266	309	341	358
Geothermal	43.2	86	186	333	493
Small hydro	9.5	19	49	106	189
Wind	4.7	44	266	542	688
Solar thermal	4.1	15	66	244	480
Photovoltaic	0.2	2	24	221	784
Solar thermal electricity	0.1	0.4	3	16	68
Marine (tidal/wave/ocean)	0.05	0.1	0.4	3	20
Total renewable energy sources	1,365.5	1,745.5	2694.4	4,289	6,351
Renewable energy source contribution (%)	13.6	16.6	23.6	34.7	47.7

Table 2: Global renewable energy scenario by 2040

When biomass is used directly in an energy claim without chemical processing, it is combusted. Conversion may be stimulated by thermochemical, biological, or chemical processes. These may be characterized as follows: direct combustion, pyrolysis, gasification, liquefaction, supercritical fluid extraction, anaerobic digestion, fermentation, acid hydrolysis, enzyme hydrolysis, and esterification. Biomass can be changed into biofuels such as bioethanol and biodiesel and thermochemical conversion products such as syn-oil, bio-syngas, and biochemical. Bioethanol is a fuel derived from renewable sources of feedstock, typically plants such as wheat, sugar beet, corn, straw, and wood. Bioethanol is a petrol additive/substitute (Demirbas, 2008). Biodiesel is made from biochemical or thermo-chemical process of biomass. A number of edges have been implemented to encourage biodiesel use in numerous countries since the early 1990s, such as biofuel-friendly engines, government support and policies, market opportunities and industrial-scale production incentives(Demirbas, 2008). In the current and foreseeable climate, biodiesel will become cheaper as markets expand to cover rural, regional and urban communities on the basis that global technology using non-edible, waste cooking oil and etc. This is very relevant due to high cost of the biodiesel production that employs virgin oil as the feedstock relative to the petro-diesel. Hence, using other source of triglycerides such as non-edible and waste oils would reduce the cost by up to 60 % and indirectly, this will help prevent inflation in the price of the edible oils thus minimising the food-energy tension. Additionally, rather than using methanol as the alcohol, bioethanol is a better alternative since it is relatively more environmentally–friendly and better solvent as automobile fuel.

2.3 Fundamental of biodiesel production - Transesterification

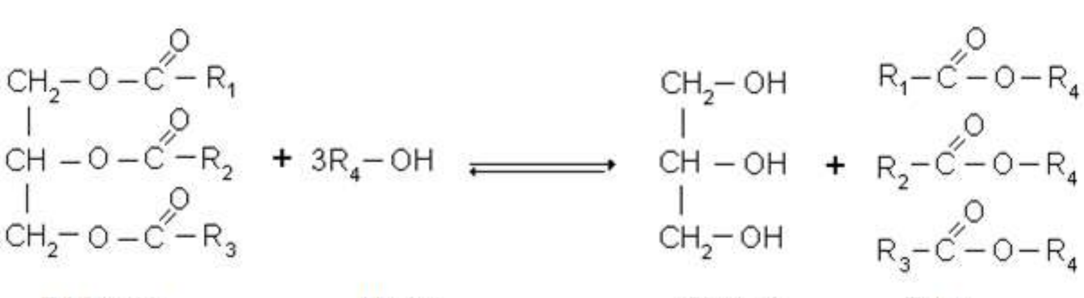

Figure 1: The fundamental equation to produce biodiesel production by using transesterification.

Of the several methods available for producing biodiesel, transesterification of natural oils and fats is currently the method of choice. The purpose of the process is to lower the viscosity of the oil or fat. Transesterification is basically a sequential reaction. Triglycerides are first reduced to diglycerides, which are subsequently reduced to monoglycerides, which are finally reduced to fatty acid esters. The order of the reaction changes with the reaction conditions. The main factors affecting transesterification are the molar ratio of glycerides to alcohol, catalysts, reaction temperature and time, and free fatty acid and water content in oils and fats. Transesterification is extremely important for biodiesel.

The production processes for biodiesel are well known. There are four basic routes to biodiesel production from oils and fats:

- Base-catalyzed transesterification
- Direct acid-catalyzed transesterification
- Conversion of the oil into its fatty acids and then into biodiesel
- Non-catalytic transesterification of oils and fats

Biodiesel produced by transesterification reactions can be alkali catalyzed, acid catalyzed, or enzyme catalyzed, but the first two types have received more attention because of the short reaction times and low cost compared with the third one (Wang et al., 2007). Most of the biodiesel produced today is made with the base catalyzed reaction for several reasons:

- It involves low temperature and pressure.
- It yields high conversion (98%) with minimal side reactions and reaction time.
- It allows a direct conversion into biodiesel with no intermediate compounds.
- It requires simple construction materials.

2.4 Basic industrial process of biodiesel production

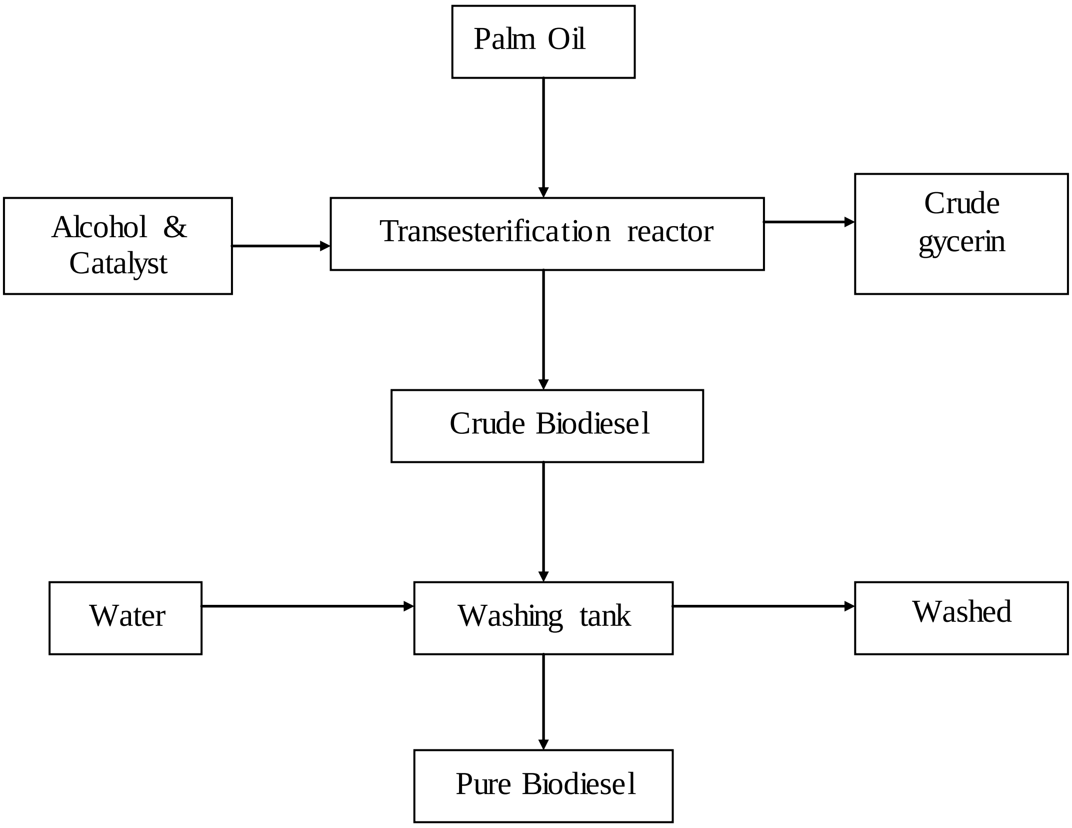

Figure 2: The basic flow diagram of conventional biodiesel production.

The basic catalyst is normally sodium hydroxide (caustic soda) or potassium hydroxide (caustic potash). It is dissolved in alcohol using a standard agitator or mixer. The methyl alcohol and catalyst mix is then charged into a closed reactor and the oil or fat is added. The reaction mix is kept just above the boiling point of the alcohol (around 344 K) to speed up the reaction, and the transesterification reaction takes place. Suggested reaction time varies from 1 to 8 h, and optimal reaction time is about 2 h (Van Gerpen et al., 2004). Excess alcohol is normally used to ensure total conversion of the fat or oil into its esters. After the reaction is complete, two major products form: glycerine and biodiesel. Each has a significant amount of the excess methanol that was used in the reaction. The reacted mixture is sometimes nullified at this step if needed. The glycerine phase is much denser than the biodiesel phase and the two can be gravity separated with glycerine simply drawn off the bottom of the settling vessel. In some cases, a sieve is used to separate the two materials faster. The biodiesel product is sometimes purified by washing gently with warm water to remove residual catalyst or soaps, dried, and sent to storage (Ma and Hanna, 1999; Demirbas, 2002).

For an alkalicatalyzed transesterification, the triglycerides and alcohol must be substantially anhydrous (Wright et al., 1944) 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 the water washing difficult. Low free fatty acid content in triglycerides is required for alkali-catalyzed transesterification.
If more water and free fatty acids are in the triglycerides, acid-catalyzed transesterification can be used (Keim, 1945). When an alkali catalyst is present, the free fatty acid will react with alkali catalyst to form soap. It is common for oils and fats to contain trace amounts of water. When water is present in the reaction, it generally manifests itself through excessive soap production. The soaps of saturated fatty acids tend to solidify at ambient temperatures, so a reaction mixture with excessive soap may gel and form a semisolid mass that is very difficult to recover. When water is present, particularly at high temperatures, it can hydrolyze the triglycerides to diglycerides and form a free fatty acid. If an oil or fat containing a free fatty acid such as oleic acid is used to produce biodiesel, the alkali catalyst typically used to embolden the reaction will react with this acid to form soap. This reaction is disagreeable because it binds the catalyst into a form that does not contribute to accelerating the reaction. Extreme soap in the products can prevent later processing of the biodiesel, including glycerol separation and water washing. Water in the oil or fat can also be a problem (Van Gerpen et al., 2004). In some systems the biodiesel is distilled in an extra step to remove small amounts of color bodies to produce a colorless biodiesel. Once the glycerine and biodiesel phases have been separated, the excess alcohol in each phase is removed using a flash evaporation process or by distillation. The glycerine byproduct contains unused catalyst and soaps that are neutralized with sulfuric acid and sent to storage as crude glycerine. In most cases the salt is left in the glycerine. Water and alcohol are removed to produce 90% pure glycerine that is ready to be sold as crude glycerine.

2.5 Biodiesel

Vegetable oil (m) ethyl esters, commonly stated to as “biodiesel”, are conspicuous applicants as alternative diesel fuels. The name biodiesel has been given to trans-esterified vegetable oil to describe its use as a diesel fuel (Demirbas, 2008). There has been new attention in the use of vegetable oils for making biodiesel due to its less polluting and renewable nature as opposed to conservative diesel, which is a fossil fuel that can be exhausted. Vegetable oils can be converted into their (m) ethyl esters via a transesterification process in the presence of a catalyst. Methyl, ethyl, 2-propyl, and butyl esters have been prepared from vegetable oils through transesterification using potassium and/or sodium alkoxides as catalysts. The purpose of the trans- esterification process is to lower the viscosity of the oil. Ideally, transesterification is potentially a less expensive way of transforming the large, branched molecular structure of bio-oils into smaller, straight-chain molecules of the type required in regular diesel combustion engines. Biodiesel esters are characterized by their physical and fuel properties including density, viscosity, iodine value, acid value, cloud point, pure point, gross heat of combustion, and volatility. Biodiesel fuels produce slightly lower power and torque and consume more fuel than No. 2 diesel (D2) fuel. Biodiesel is better than diesel fuel in terms of sulfur content, flash point, aromatic content, and biodegradability (Demirbas, 2008). The cost of biodiesels varies depending on the base stock, geographic area, variability in crop production from season to season, the price of crude petroleum, and other factors. Most of the biodiesel currently made uses soybean oil, methanol, and an alkaline catalyst. The high value of soybean oil as a food product makes production of a cost effective fuel very interesting. However, there are large amounts of low cost oils and fats such as restaurant waste and animal fats that could be changed into biodiesel. The problem with processing these low cost oils and fats is that they often contain large amounts of free fatty acids (FFA) that cannot be converted into biodiesel using an alkaline catalyst (Demirbas, 2008). Biodiesel is an environmentally friendly alternative liquid fuel that can be used in any diesel engine without modification. There has been renewed interest in the use of vegetable oils for making biodiesel due to its less polluting and renewable nature compared with conventional petroleum diesel fuel. If the biodiesel valorized efficiently at energy purpose, so would be benefit for the environment and the local population, job creation, provision of modern energy carriers to rural communities.

2.6 Torque

Torque, moment or moment of force, is the tendency of a force to rotate an object about an axis, fulcrum, or pivot. Just as a force is a push or a pull, a torque can be thought of as a twist to an object. Mathematically, torque is defined as the cross product of the lever-arm distance and force, which tends to produce rotation. Loosely speaking, torque is a measure of the turning force on an object such as a bolt or a flywheel. For example, pushing or pulling the handle of a wrench connected to a nut or bolt produces a torque (turning force) that loosens or tightens the nut or bolt (Marshall, 2014).

2.7 Viscosity

The physical property of substances that characterized their resistance to flow is the viscosity (Ramírez Verduzco, 2013). The viscosity of a fluid is measure of its resistance to gradual deformation by shear stress or tensile stress. For liquids, is corresponds to the informal notion of “thickness”. Viscosity is due to friction between neighboring parcels of the fluid that are moving at different velocities. When fluid is forced through a tube, the fluid generally moves faster near the axis and very little near the walls, therefore some stress (such as pressure difference between the two ends of the tube) is needed to overcome the friction between layers and keep the fluid moving. For the same velocity pattern, the stress is proportional to the fluid viscosity (Ramírez Verduzco, 2013).

2.8 Shear Stress

A shear stress, denoted as τ, is defined as the component of stress coplanar with a material cross section. Shear stress arises from the force vector component parallel to the cross section. Normal stress, on the other hand, arises from the force vector component perpendicular to the material cross section on which it acts. Any real fluids (liquids and gases included) moving along solid boundary will incur a shear stress on that boundary. The no-slip condition dictates that the speed of the fluid at the boundary (relative to the boundary) is zero, but at some height from the boundary the flow speed must equal that of the fluid. The region between these two points is aptly named the boundary layer. For all Newtonian fluids in laminar flow the shear stress is proportional to the strain rate in the fluid where the viscosity is the constant of proportionality. However for Non Newtonian fluids, this is no longer the case as for these fluids the viscosity is not constant. The shear stress is imparted onto the boundary as a result of this loss of velocity. (Qiu, Duan, Luo, Kaloni, & Liu, 2013).

2.9 Shear rate

Shear rate is the speed of deformation in the shear mode which is typical of fluids and can be represented as layers sliding one onto another. For Newtonian fluids, shear rate is directly proportional to flow rate, and a simple relationship is used to calculate its maximum value at the wall of the channel. When the fluid is non-Newtonian, this relationship gives only a first approximation, which is called the apparent value of wall shear rate. The true wall shear rate can be estimated by applying the Rabinowitsch correction, and is higher than the apparent value (Kumar & Guria, 2013).

2.10 Dimensionless numbers

2.10.1 Weber number

The Weber number is a dimensionless number in fluid mechanics that is often useful in analysing fluid flows where there is an interface between two different fluids, especially for multiphase flows with strongly curved surfaces. It can be thought of as a measure of the relative importance of the fluid's inertia compared to its surface tension. The quantity is useful in analyzing thin film flows and the formation of droplets and bubbles (Nikolopoulos, Strotos, Nikas, & Bergeles, 2012).

2.10.2 Froude Number

The Froude number is a dimensionless number defined as the ratio of a characteristic velocity to a gravitational wave velocity. It may equivalently be defined as the ratio of a body's inertia to gravitational forces. In fluid mechanics, the Froude number is used to determine the resistance of a partially submerged object moving through water, and permits the comparison of objects of different sizes (Prešeren, Steinman, Širok, & Bajcar, 2013).

2.10.3 **Weeping point**

Liquid will start to flow through a hole when the diameter of the hole forms an open area that sufficiently overcomes the liquid's surface tension or interfacial tension with another liquid. The following variables were studied in order to get the weeping point of two immiscible liquid or gas-liquid systems (Zanelli and Del Bianco 1973):-

1. Diameter of perforated plate
2. Height of liquid level on top of bottom of the perforated plate
3. Plate pressure drop

Since the weeping point and rate is highly subjective, it is hard to predict exactly a particular contactor weeping characteristic based on any rigorous material balance calculation unless a pilot scale test would usually be employed at least (Thorat et al. 2001). This theory applies to physical solvent extraction. In a system that involves reactions and dealing with impure feedstocks, this empirical behaviour would be more significant (Prešeren et al., 2013).

2.11 Rheometer

A rheometer is a laboratory device used to measure the way in which a liquid, suspension or slurry flows in response to applied forces. It is used for those fluids which cannot be defined by a single value of viscosity and therefore require more parameters to be set and measured than is the case for a viscometer. It measures the rheology of the fluid. There are two distinctively different types of rheometers. Rheometers that control the applied shear stress or shear strain are called rotational or shear rheometers, whereas rheometers that apply extensional stress or extensional strain are extensional rheometers. Rotational or shear type rheometers are usually designed as either a native strain-controlled instrument (control and apply a user-defined shear strain which can then measure the resulting shear stress) or a native stress-controlled instrument (control and apply a user-defined shear stress and measure the resulting shear strain).

3 MATERIALS AND METHODS

3.1 Overview

This paper presents a study on the characterization of the ethanol-triglyceride transesterification reaction under counter current flow system. In this chapter, it is discussed more specifically about how to run the experiment. The first part is the experiment of the characterization of the ethanol-triglyceride. It is explained about the procedures and materials preparation. As for raw material, it is the first thing that needed to be taken in matter the most as it is the core of the whole studies. It is divided into three parts of material preparation like raw material, solvent and catalyst. Experiment methodology is conducted to show the whole process from initial to the final. Moreover, it is included how the experiment is set. The right choosing of the equipment is also the most important factor and during the whole operation conducted. All the steps and works are compulsory to be conducted 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 the every single step of the experiment from initial to final that will be conducted in future. Besides, the analysis and characterization also is specified and introduced in this chapter.

3.3 Chemicals

Chemical	Supplier	Grade and Purity	Application
Absolute Ethanol	Sigma-Aldrich	Absolute (100%)	Solvent
Cooking Oil	Gambang Store	Cooking oil	Raw material
Potassium Hydroxide Pellets	Sigma-Aldrich	99.95%	Catalyst

3.4 Sample preparation

3.4.1 **Characterization of cooking oil and absolute ethanol mixture (Torque, viscosity, shear strength and shear rate)**

The cooking oil was pipetted 8mL using the pipette and released into the viscometer cylinder. The absolute ethanol was pipetted 1mL as well using the pipette and released into the viscometer cylinder. Both cooking oil and absolute ethanol was mixed together in the viscometer cylinder. The sample was prepared again later with different ratios (8:1, 7:2, 6:3, 5:4, 4:5, 3:6, 2:7, 1:8).

3.4.2 **Extractive Reactor Column**

First of all, catalyst needed to be dissolved so that it can be in liquid phase. The KOH was dissolved in some amount of ethanol. The KOH was measured to be 10% wt to oil. 445g of KOH was calculated for 5L of oil in this reaction. So, 445g of KOH was dissolved in 2750mL of absolute ethanol by using the magnetic stirrer. Meanwhile, 5L of oil was poured into tank 2 and 30L of absolute ethanol was poured into tank 1.

Figure 3: Dissolving KOH pallets using magnetic stirrer

Figure 4: Absolute ethanol which contained dissolved KOH pellets

3.5 Characterization of ethanol-triglyceride

The sample that was ready inside the viscometer cylinder was stirred by the viscometer spindle. Before the experiment started, few things have to be concerned. The viscometer is in appropriate position by adjusting the bubble into the circle on the top of the viscometer. Second is correct spindle used. Third is zero calibration every time the viscometer is turn on. After zero calibration of the viscometer, first ratio of cooking oil to absolute ethanol (8:1) was run. Started with RPM 50, when the reading on the screen of the viscometer was stable, torque, viscosity, shear strength and shear rate value was recorded. After that, RPM 100, RPM 150, RPM 200 and RPM 250 was run. When the first ratio of cooking oil and absolute ethanol was done, ratio 7:2, 6:3, 5:4, 4:5, 3:6, 2:7, 1:8 was run consecutive. All the reading was recorded as well. The reading was then plotted on to the graphs for each parameter versus RPM of all ratios.

Figure 5: Brookfield Viscometer

3.6 Extractive Reactor Column

3.6.1 **Preliminary run with water**

The extractive reactor column was run with water at first to make sure everything was smooth. The water was filled in tank 1, therefore the water was started filling from bottom decanter. After the preliminary run, the water was released to make sure the tank was clean and clear. This was because when water mix with the cooking oil, it will produces fatty acid which would stuck inside the pipe and pumps.

3.6.2 **Washing with absolute ethanol**

After the preliminary run with water, the extractive reactor column was run again with absolute ethanol. This was to make sure the left over water which stuck in the pipes and pumps was totally removed.

3.6.3 **Investigation of the reacting liquid**

Tank 1 was filled with absolute ethanol which contained KOH. Tank 2 was filled with cooking oil. The main switch was turned on after that. Heater 1, pump 1, pump 2 and stirrer are turned on as well. Heater 1 was set at temperature 40°C. Stirrer was set at 200 RPM. The flow rate of absolute ethanol and cooking oil was set at ratio 6:1 as well which is “300” for absolute ethanol and “50” for cooking oil. The absolute ethanol was filled from the bottom decanter while the cooking oil was filled from top decanter which both reactant will mix with each other and react later. Heater 2 was turned on when the reactant achieves the level at heater 2. Heater 2 was set at temperature 40°C as well. The stirrer was kept on running to retain the oil in the absolute ethanol so that it got longer time to react. While the filling process was going on, temperature and torque was observed. The temperatures of heaters were increased 5°C every half an hour and stop at temperature 50°C. RPM of the stirrer was increased to 230 after half an hour as well. The filling and flowing of reactants was kept on running until the top decanter was overflowed. Samples were then taken from each stages of the column. The overflowed product was then flow into tank 4.

Figure 6: Extractive Reactor Column

Figure 7: Top decanter which was filled with reactant

Figure 8: The flow rate of cooking oil (FI 4) and absolute ethanol(FI 5) set at ratio 6:1 which is "50" and "300

3.7 Chromatographic analysis of tripalmitolein by using agilent Gas Chromatography (GC)

Each sample will be obtained by using syringe from each stages of the column. After that, the sample is put into the n-heptane solution. The mixture is then sent to agilent GC for analysis. This analysis is used to obtain the calibration curve of the concentration of tripalmitolein which is the dominant content of triglyceride in palm oil goes along the time during experiment in the reactant mixture.

4 RESUTLS AND DISCUSSIONS

4.1 Overview

This chapter mainly summarizes all tabulated data from the study findings. Later in this chapter, detailed justifications are available on the conducted experiments.

4.2 Introduction

The first parts of this chapter will presents the results obtained from the experiment of characterization of the ethanol-triglyceride. Data of four parameters (torque, viscosity, shear stress and shear rate) will be presented and graphs are shown for each parameter versus RPM. The later part will be presenting the results obtained from the experiment of extractor reactive column.

4.3 Characterization of ethanol-trigylceride

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	8.3	16.4	24.3	32.1	39.9
Viscosity	50.4	49.3	48.6	48.1	47.8
Shear Strength	0.85	1.67	2.48	3.27	4.06
Shear Rate	17	34	51	68	85

Table 3: Cooking Oil : Absolute ethanol (8:1)

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	4.5	8.9	13	16.9	20.0
Viscosity	27.0	26.7	26.2	25.3	23.6
Shear Strength	0.45	0.91	1.34	1.71	2.00
Shear Rate	17	34	51	68	85

Table 4: Cooking oil : Absolute ethanol (7:2)

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	3.8	7.9	11.8	15.8	19.7
Viscosity	24.6	24.3	24.0	23.7	23.8
Shear Strength	0.39	0.82	1.21	1.62	2.01
Shear Rate	17	34	51	68	85

Table 5: Cooking oil : Absolute ethanol (6:3)

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	2.3	4.9	7.3	9.7	12.2
Viscosity	13.8	15.0	14.6	14.5	14.8
Shear Strength	0.23	0.51	0.74	1.00	1.25
Shear Rate	17	34	51	68	85

Table 6: Cooking oil : Absolute ethanol (5:4)

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	1.3	3.3	4.6	6.3	7.9
Viscosity	9.0	9.9	9.4	9.45	9.48
Shear Strength	0.13	0.34	0.47	0.64	0.81
Shear Rate	17	34	51	68	85

Table 7: Cooking oil : Absolute ethanol (4:5)

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	1.1	2.9	4.1	5.7	7.4
Viscosity	6.6	8.7	8.2	8.7	8.76
Shear Strength	0.11	0.3	0.41	0.59	0.74
Shear Rate	17	34	51	68	85

Table 8: Cooking oil : Absolute ethanol (3:6)

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	0.4	1.2	1.5	2.3	3.0
Viscosity	2.4	3.6	3.0	3.45	4.2
Shear Strength	0.04	0.13	0.15	0.23	0.35
Shear Rate	17	34	51	68	85

Table 9: Cooking oil : Absolute ethanol (2:7)

	RPM 50	RPM 100	RPM 150	RPM 200	RPM 250
Torque	0.2	1.3	1.7	2.1	2.6
Viscosity	1.8	3.6	3.4	3.55	3.0
Shear Strength	0.02	0.12	0.17	0.22	0.27
Shear Rate	17	34	51	68	85

Table 10: Cooking oil : Absolute ethanol (1:8)

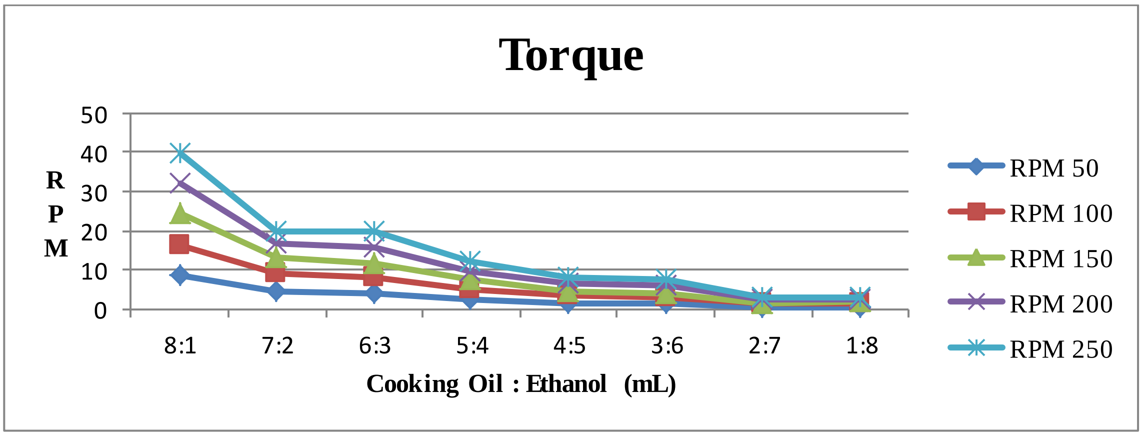

Figure 9: Graph of RPM vs ratio for torque

From the graph shown above, the torque of each ratio of reactant mixture was more when the 50 RPM is increased every time. For ratio 8:1, the torque was 8.3% when RPM was 50, 16.4% when RPM was 100, 24.3% when RPM was 150, 32.1% when RPM was 200 and 39.9% when RPM is 250. However, the torque was decreasing when the ratio of the cooking oil was decreasing. For RPM 50, torque of ratio 8:1 was 8.3%, torque of ratio 7:2 was 4.5%, torque for ratio 6:3 was 3.8%, torque for ratio 5:4 was 2.3%, torque for ratio 4:5 was 1.3%, torque for ratio 3:6 was 1.1% torque for ratio 2:7 was 0.4% and torque for ratio 1:8 was 0.2%.

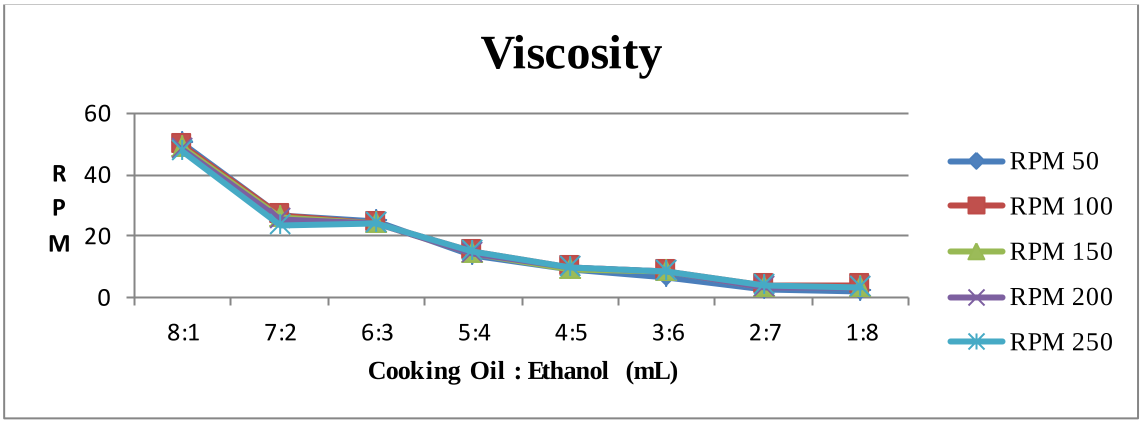

Figure 10: Graph of RPM vs ratio for viscosity

From figure 10, the viscosity of each ratio of reactant mixture was slightly decreasing every time 50 RPM was increased. For ratio 8:1, the viscosity was 50.4 mPaS when RPM was 50, 49.3 mPaS when RPM was 100, 48.6 mPaS when RPM was 150, 48.1 mPaS when RPM was 200 and 47.8 mPas when RPM was 250. While the ratio of cooking oil was decreasing, the viscosity was decreasing as well. For RPM 50 at ratio 8:1, the viscosity was 50.4 mPaS, at ratio 7:2, the viscosity was 27 mPaS, at ratio 6:3, the viscosity was 24.6 mPaS, at ratio 5:4, the viscosity was 13.8 mPas, at ratio 4:5, the viscosity was 6.6 mPaS, at ratio 2:7, the viscosity was 2.4 mPaS and at ratio 1:8, the viscosity was 1.8 mPaS.

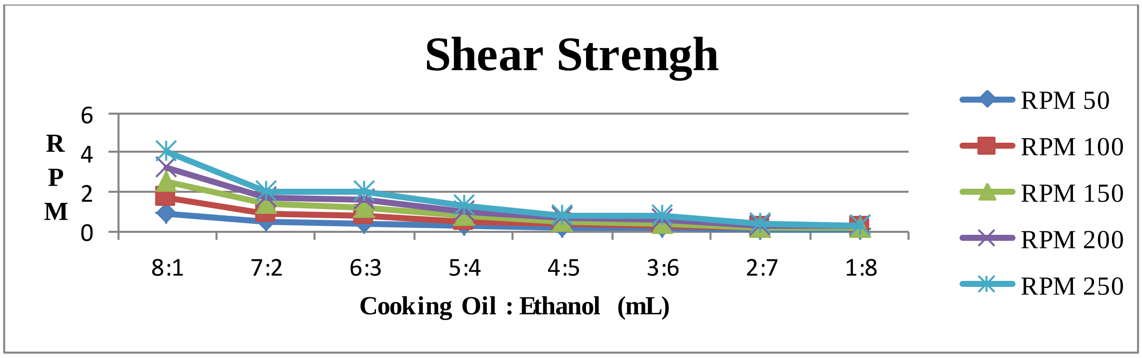

Figure 11: Graph of RPM vs ratio for shear strength

From figure 11, the trend of the graph was more or less the same as the graph of torque. The shear strength was more for each ratio of reactant mixture every time 50 RPM was increased. For ratio 8:1, the shear strength was 0.85 N/m² when RPM was 50, 1.67 N/m² when RPM was 100, 2.48 N/m² when RPM was 150, 3.27 N/m² when RPM was 200 and 4.06 N/m² when RPM was 250. However, the shear strength was decreasing as well when the ratio of cooking oil was decreasing. At RPM 50 at ratio 8:1, the shear strength was 0.85 N/m², 0.45 N/m² at ratio 7:2, 0.29 N/m² at ratio 6:3, 0.23 N/m² at ratio 5:4, 0.13 N/m² at ratio 4:5, 0.11 N/m² at ratio 3:6, 0.04 N/m² at ratio 2:7 and 0.02 N/m² at ratio 1:8.

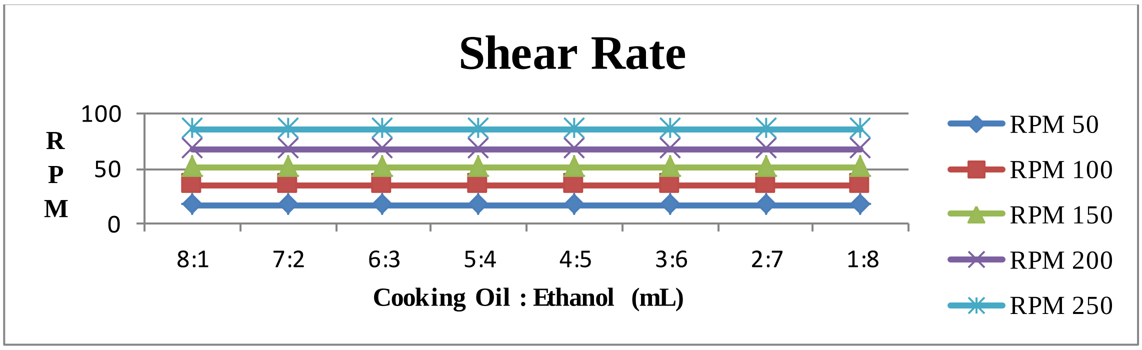

Figure 12: Graph of RPM vs ratio for shear rate

From figure 8, the shear rate of each ratio of reactant mixture had no changes when the ratio was changing. The shear rate only will be more when the RPM was increased for each ratio. For ratio 8:1, the shear rate was 17 1/sec at RPM 50, 34 1/sec at RPM 100, 51 1/sec at RPM 150, 68 1/sec at RPM 200, 85 1/sec at RPM 250.

4.4 Extractive Reactor Column

For the experiment using extractive reactor column, there were problem when conducting the experiment. Due to the low quality cooking oil used, it contained a lot of fatty acid contents. The fatty acid caused the flow meter on the extractive reactor column stuck. Due to this consequence, when the pumps were turn on, the forces caused the flow meter to break. The reactants flow inside extractive reactor column kept spilling out of the column. Unfortunately, this experiment cannot be carried on. Furthermore, the corrosive absolute ethanol which contained the KOH did corrode the flow meter too which the flow meter made of plastic.

Figure 13: Flow meter that was broken caused by the over pressure created by the pump due to the fatty acid stuck in it.

Figure 14: Flow meter that was corroded by the absolute ethanol which contained KOH

4.5 Fatty acid which formed after 1 night left over

Figure 15: Fatty Acid which produced after 1 night left over in top decanter

Figure 16: Fatty Acid which produced after 1 night left over in bottom decanter

From figure 15 and figure 16, this phenomenon happened after 1 night left over. The white colour fatty acid formed in the top decanter and bottom decanter. The fatty acid formed caused the stirrer cannot be functioning because the fatty acid stuck and blocked the stirrer from stirring. To solve this problem, heater 2 needed to be turned on for 2 hours to melt the fatty acid.

Figure 17: The fatty acid slowly melted from time to time when the heater is turn on.

5 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In the previous chapter, clearly discussed the results obtained from this study. Throughout this study, only the characterization of the ethanol-triglyceride can be achieved. For the experiment of the extractive reactor column, although it cannot be carried on after the flow meter had broken, fortunately the major problem was known. It can be a lesson for next research.

From the characterization of the ethanol-triglyceride experiment, the viscosity of each ratio of reactant mixture was slightly decreasing every time 50 RPM was increased. Viscosity is one of the most important transport properties for fluids involved in the continuos counter current flow system. Most of the lipid feedstocks for biodiesel fuel production are very viscous at the ambient temperature and pressure. The viscosity decreases with the stirring of the feedstock.

For the extractive reactor column study, the most important factors in the transesterification process are the quality of triglyceride feedstocks. The differences in quality characteristics between various lipid feedstocks may causes the flow and quality of the studies. Thus, the choosing of the raw materials is very important.

5.2 Recommendations

From this study, raw material or feedstock choosing is very important. Avoid using of low quality cooking oil. The fatty acid content which low quality cooking oil contained seriously was a major problem for this study. Besides, the reactor made should have be confirmed can retains the corrossiveness of the chemicals.

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