Emulsion Stability and Combustion Characteristics Glycerol -Water Mixture

by

Suliana Binti Abu Bakar

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
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Approved by,

____

(Associate Professor Dr Ye Lwin)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
DEC 2010

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.

SULIANA BINTI ABU BAKAR

ABSTRACT

The **main purpose** of this project is to study the emulsion stability of glycerol-water mixture and its combustion characteristics. Glycerol, as by-product from biodiesel production emerges as significant worth chemical that can be converted into high value products or alternative use.The concerned **problem statement** includes crude glycerol-water emulsion which would most likely separate into 2 or 3 phases after a period of time. Therefore, proper mixing and proportioning of glycerol, water, additive and/or surfactants are very important to create very stable emulsion that is necessary for smooth combustion. The **objectives** for this work are to study the behavior of interaction and mixing between glycerol, water and additives in various proportions to study the effect of alcohol on combustion characteristics and flame stability of glycerol-water mixture. The **methodology** of this research project starts with preparation of glycerol-water at specific volume ratio for emulsion stability test. The emulsion stability needs within 24 hours to be observed. Testing on chemical and physical properties such as viscosity measurement and heating value test has been done for before proceed to combustion characteristics or flame stability test. The final stage of glycerol-water mixture testing is the combustion characteristics or flame stability. Alcohol such as ethanol is added to glycerol-water mixture to induce volatility, thereby increasing the ease of combustion. In this research, for emulsion stability, the best ratio of the glycerol-water mixture was found to be 90 ml of glycerol and 10 ml of deionized water because glycerol is already miscible with water, therefore, no surfactant or additive is needed to stabilize the emulsion. For combustion characteristics, the best ratio of glycerol-water mixture was found to be 90 ml of ethanol and 10ml of glycerol. At the end of this project, the objectives are achieved but further research of this project is still ongoing in improvising the method

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CHAPTER 1

INTRODUCTION

1.1. Background of Study

Glycerol is one of the most valuable chemical substances known to man (Bonnardeaux, 2006). It was identified in 1779, by Swedish chemist Carl W Scheele, who discovered a new transparent, syrupy liquid by heating olive oil with litharge (PbO), used in lead glazes on ceramics). It is completely soluble in water and alcohols, is slightly soluble in many solvents such as ether and dioxane, but is insoluble in hydrocarbons.

Overall, it possesses a unique combination of physical and chemical properties which are utilized in many thousands of commercial products (Boca, 2006)

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Glycerol, as by-product from biodiesel production, emerges as significant worth chemical that can be converted into high value products or other alternative uses. It will become a substitute for many common petrochemicals on the market. Globally, glycerol arising from biodiesel production has increased from 200000 tonnes in 2004 to 800000 tonnes in 2007. Well-regarded experts in the industry consider that biodiesel production could rise to 9 million tonnes by 2010 in Europe alone, translating into 900000 tonnes of glycerol (Mario and Michele, 2010). This gives a clear indication of the developing status of glycerol as a key raw material.

This project is not only to study of the emulsification process of glycerol, water and alcohol, but also to find out the stable emulsion and to study its combustion characteristics whether this emulsified glycerol can give satisfactory combustion efficiency. In addition, alcohols can be added to glycerol to induce volatility, thereby increasing ease of combustion.

1.2. Problem Statement

Crude glycerol-water emulsion would most likely separate into 2 or 3 phases after a period of time. It is a great challenge to create stable emulsion which can stay for some period of time. Proper mixing and proportioning of glycerol, water, and additives and/or surfactants are important to create very stable emulsion that is necessary for smooth combustion.

The idea of emulsified fuel had been developed by many scientists by adding water into the fuel oil. The purposes of the idea are to reduce oil consumption as well as to reduce the pollution cause by burning fuel oil. But the problem is the emulsified fuel is hardly to be in stable form (Johan, 2006). Johan Sjoblom in his book entitled Emulsion and Emulsion Stability, 2nd Edition, 2006 wrote that *"There is still no absolute answer to emulsion stability. There is no one had ever come with the right solution for long emulsion stability."* (Johan, 2006)

The emulsified fuel would most likely separate into two or three phases after a period of time, from a few minutes, a few hours to a few days. Thus it is a great challenge to create emulsified fuel which can stay for long period of time.

1.2.1. Problem Identification

The ratio of glycerol and water, the amount and types of surfactant, the pressure and temperature during mixing and the method to emulsify the mixture are the factors that can affect the stability of emulsion.

1.2.2. Significance of the Project

Proper mixing and proportioning of water, glycerol, additive and surfactant are important to create very stable emulsion that is ready for combustion. The purpose of burning such a mixture of oil and water is to provide alternative usage of glycerol as well as to economize the consumption of oil.

1.3. Objective and Scope of Study

In order to complete this project within the time limit and the scope given, several objectives have been identified.

1. To study the behavior of interaction and mixing between glycerol, water and additive(s) in various proportions.
2. To determine the chemical and physical properties such as viscosity and heating value.
3. To study the effect of on combustion characteristics and flame stability of glycerol-water mixture.

Scope of study for this research work is not just limited to the methods producing stable emulsions, but it covers as well examples of topic shown below:

1. For final year project I, the scopes of the project are more on studying and researching for information on the book, journal and experiment report available in the net and library. This is to ensure that the author has fully understood and have strong knowledge on the fuel emulsification and its combustion characteristics.
2. In final year project II, the project will proceed to the lab analysis. It composed of two stages which are experimenting and data analysing stage.
   - i. The stability of glycerol-water emulsion, the type of glycerol used, the additive and the factor of that can affect the stability of emulsion such as temperature, speed of mixing and time are identified properly.
   - ii. The properties of stable emulsion such as viscosity value and heating value are analyzed using Brookfield viscometer and Bomb Calorimeter.
   - iii. Last but not least is the combustion characteristics test or flame stability of the glycerol and water. Alcohol such as ethanol is used to induce the combustion of glycerol-water mixture.

1.4. The Relevancy of the Project

Glycerol stands to develop into an even more commonly used chemical than it is at present. It will become a substitute for many common petrochemicals on the market to express it in the words of one practitioner. Globally, glycerol arising from biodiesel production has increased from 200 000 tonnes in 2004 to 800 000 tonnes in 2007. Well-regarded experts in the industry consider that biodiesel production could rise to 9 million tonnes by 2010 in Europe alone, translating into 900 000 tonnes of glycerol (Mario and Michele 2010).

This gives a clear indication of the developing status of glycerol as a key raw material. In three to five years it will be seen as an environmentally friendly way of replacing other competing petroleum products and it is one of the next chemical platforms which will become widely available. Experts realistically predict the end of cheap oil by 2040 at the latest since increased consumption of oil will diminish fossil raw materials and build up environmentally pressure.

Something new can be learnt and develop in the future in order to achieve the objective of reduce the oil consumption as well as to save the environment.

Therefore, with this new mixture of glycerol, water and addition of alcohol can improve combustion as well as development of one of the product of bio glycerol for potential replacement of conventional fossil fuels.

1.5. Feasibility of the Project within the Scope and Time Frame

This project involves with 4 main stages shown below:

a. Preparation for sample (1 week)
b. Emulsion stability test (3 weeks)
c. Viscosity measurement and heating value test (1 week)
d. Combustion characteristics or flame stability test (3 weeks)

CHAPTER 2

LITERATURE REVIEW

2.1. Glycerol

Glycerol is an organic compound which has the simplest trihydric alcohol which has IUPAC name of propane-1, 2, 3-triol. It is also commercially known as adourless, glyceritol or glycyl alcohol. Pure glycerol is colourless, adourless, viscous liquid with syrupy and sweet taste (Abdullah, *et. al*., 2009). As shown in Figure 1, glycerol has three hydrophilic hydroxyl groups that are responsible for its solubility in water and its hygroscopic nature. Table 1 lists physical and chemical properties of glycerol which is importantly associates to its application.

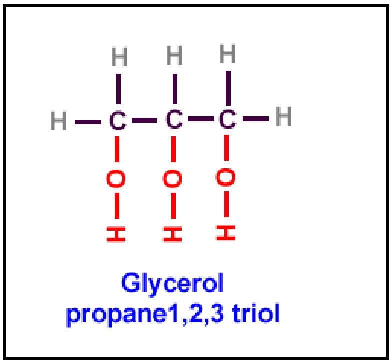

Figure 1: Molecular Structure of Glycerol

Table 1: Physical and Chemical Properties of Glycerol at 20°C

Properties	**Values**
Chemical Formula	$C_3H_5(OH)_3$
Molecular Weight	92.09
Density	1.261gcm^{-3}
Viscosity	1.5 Pa.s
Form and colour	Colourless and liquid
Melting Point	18.2°C
Boiling Point	290°C
Solubility in • Water • Alcohol • Ether	 Soluble Soluble Insoluble

2.2. Glycerol-water mixture

Glycerol has solubility characteristics similar to those of water and the simple aliphatic alcohols such as ethanol and methanol because of its hydroxyl groups. It is completely miscible with water because of similar density (Anonymous, 1990).

When water is added into glycerol, it will form homogenous mixture which means it can form one phase due to higher solubility between glycerol and water. The hygroscopicity that glycerol have make it able attract moisture from the air hold it. This is one of the most valuable properties of glycerol. It is the basis for its use as a humectants and as a conditioning agent in many applications where both the glycerin and the water it holds act as plasticizers.

Glycerol has low volatility. Low volatility is closely allied to hygroscopicity in glycerol's effectiveness as humectants (Anonymous, 1990).

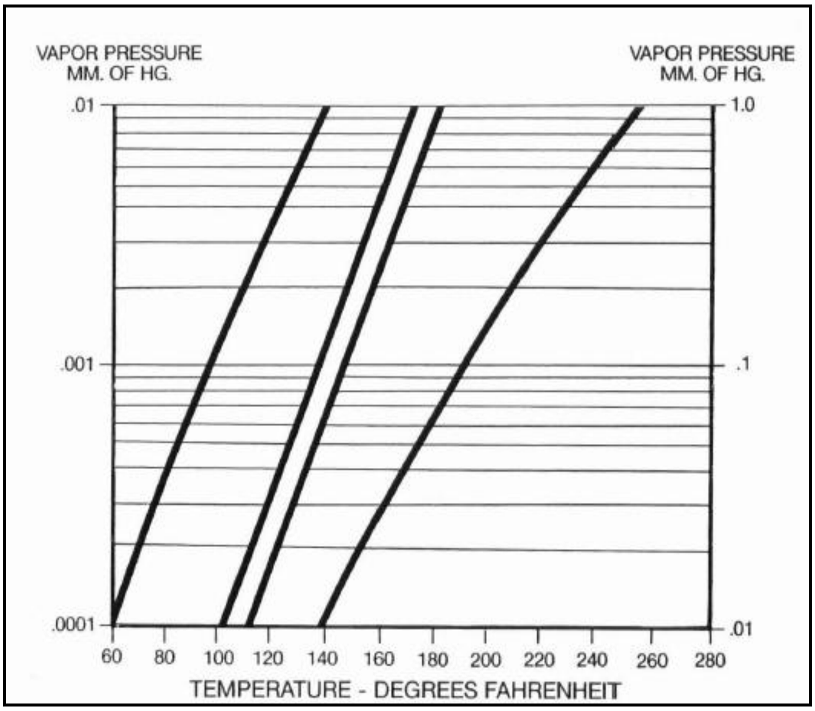

Figure 2: Graph of vapor pressure of 100% glycerol versus temperature

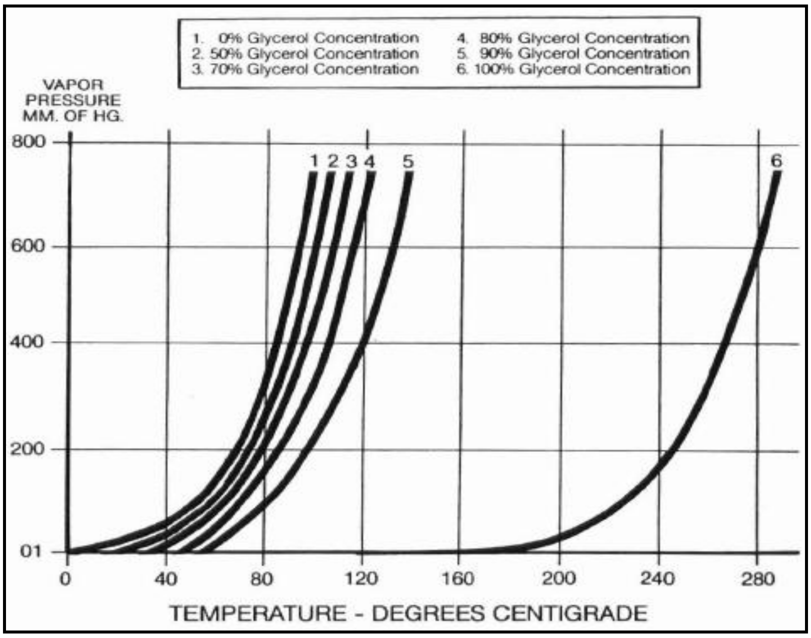

Figure 3: Graph of aqueous solution of glycerol versus temperature

As shown in figure 2 and figure 3, glycerol is practically nonvolatile at normal use temperatures. Moreover, between 0^{o}C and 70^{o}C changes in temperatures have little effect on the relative vapor pressure of glycerol solutions. That is why, a certain amount of alcohol such as methanol or ethanol is added to the glycerol-water mixture to increase the volatility of glycerol and improve the combustion of glycerol.

2.3. Glycerol Economics

Global production of glycerol from biodiesel has climbed from 200000 tonnes in 1995 to 600000 tonnes in 2006. Most of the growth has come within the last three years, due to glycerol arising as a by-product of the production of biodiesel. Figure 2 shows that world production of biodiesel quadrupled between 2000 and 2005 and so correspondingly have global glycerol production (Ott, *et. al.*, 2006).

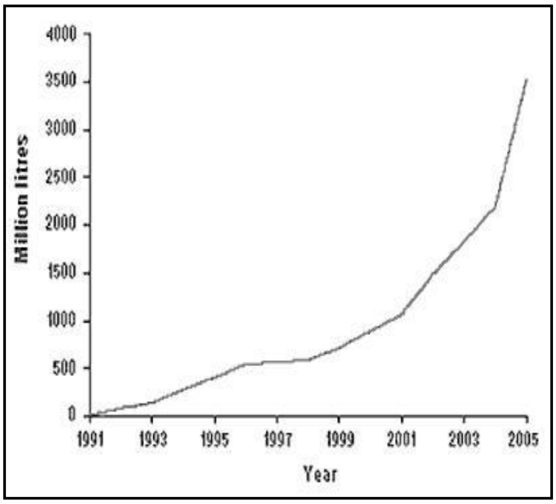

Figure 4: Global glycerol production (1975-2005)

The impact of biodiesel production in the European Union on domestic glycerol supplies has been small in comparison to the effect of imports, which doubled again in 2006. In European, a European Union directive adopted in 2003 aims to promote to the use of transport fuels made from biomass and other renewable fuels, and requires that by the year 2010, some 5.75% of the total fuel consumed in the European Union must come from renewable sources.

Over the past decade environmental legislation has progressively lowered the sulphur content in fossil diesel fuel and biodiesel is widely used in the European Union as fuel additive to achieve this objective.

At the middle of the 1990s production of natural fatty alcohols in Asia has also increased steadily, adding to glycerol surplus. For instance countries such as Malaysia and Indonesia are major producers of glycerol, mostly derive from palm oil and it give big impact to the global market for glycerol to reach 900000 tonnes in 2006, of which 600000 tonnes came from biodiesel production, compared to only 600000 tonnes in 2001. Such a rapid surplus of refined glycerol soon caused prices to plummet to US$0.33perkg or below.

Having become part of the fuel market, glycerol is now an important element of the global market scene (Nilles, 2006). The graph in figure 3 shows the price trend for refined glycerol up to 2003 and reports the prediction by a US researcher that by 2010 the glycerol price will fall to as low as US$0.35perlb.

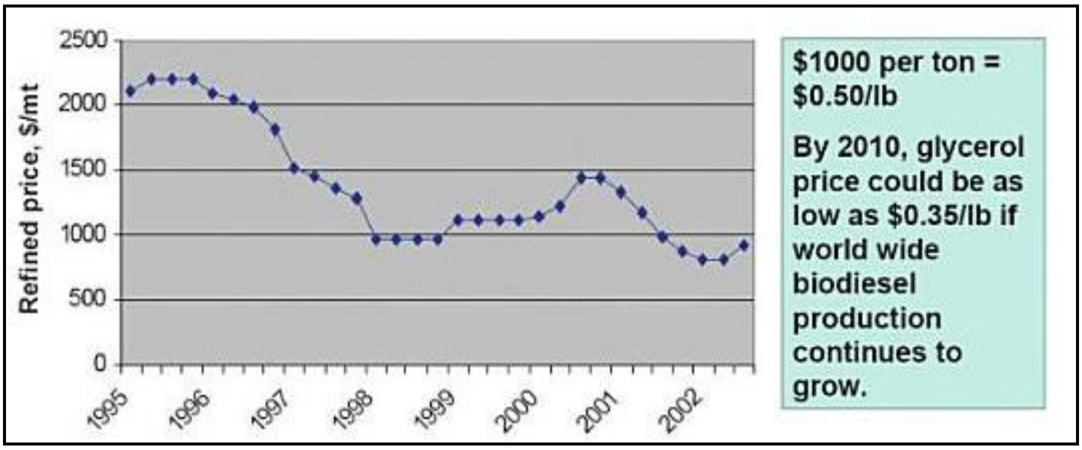

Figure 5: Price trend of industrial glycerol

2.4. Emulsion

Most oils are less dense in water, and if oil and water are mixed then the oil will simply float to the surface (Gary, 2005). An emulsion is mixture of two liquids that would not normally mix. Emulsion is a mixture of two or more immiscible (unblended) liquids. The kind of emulsion fuels, consisting of usual hydrocarbon fuels and water, is divided into two types; water in oil and oil in water emulsions. Water in oil type emulsion fuel has dispersed many fine water particles in the base hydrocarbon fuel. On the other hand, oil in water type emulsion fuel has inversely, dispersed many fine hydrocarbon fuel particles in the water phase. The dispersed oil droplets are stabilised by hydrophilic surface active molecules which form a layer around the oil droplets. By definition, an emulsion contains tiny particles of one liquid suspended in another. Chemically, they are colloids where both phases are liquids. They are typically milky in appearance and the suspended material may be colloidal in nature.

2.4.1. Emulsion Break-down

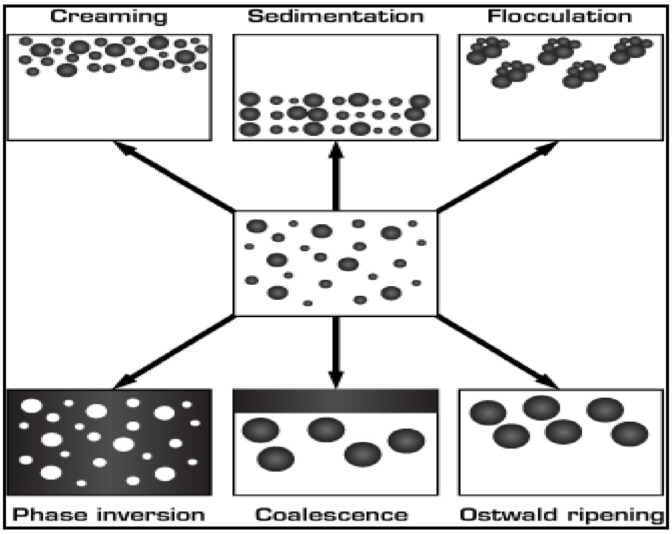

Figure 6: Mechanisms for destabilization of emulsions

Emulsion can break down because of various mechanisms (Croda, 2010). Creaming and sedimentation occurs as a result of the density difference between the phases. Creaming is more common than sedimentation because most emulsions are of the oil-water type and the oils tend to be of lower density than the aqueous phase.

Droplet an also flocculate, which means that they enter the so-called “secondary minimum”, an energetically stable situation where the droplets are close to each other but still retain their integrity (Holmberg, *et. al*. 2003).

Another destabilization mechanism is Ostwald ripening. In this process, small drops which have large area per volume, lose material and finally disappear while bigger drops grow in size. The process is caused by diffusion of molecules of the dispersed phase through the continuous medium (Holmberg *et. al*. 2003).

2.4.2. Emulsion Stability

Stable emulsions can be formed from two immiscible liquids when an emulsifying agent is used. Emulsifying Agent is the substance added to an emulsion to prevent the coalescence of the globules of the dispersed phase (Song, *et. al.*, 2000). They are also known as emulsifier. They act by reducing the interfacial tension between the two phases and forming a stable interfacial film. The result is emulsions do not separate out after a change in conditions like temperature or over time.

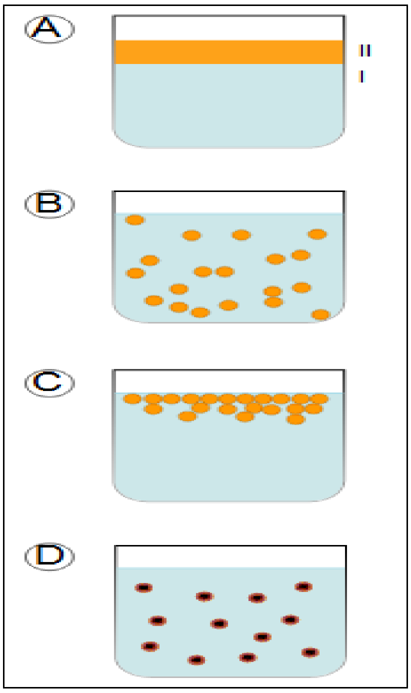

Figure 7: Emulsion from unstable until Stable

A. Two immiscible liquid, not yet emulsified,

B. An emulsion of phase II dispersed in phase I

C. The unstable emulsion progressively separates

D. The surfactant (purple outline) positions itself interfaces between Phase II and Phase I, stabilized the emulsion.

2.5. Factors that Effect of Stability of Emulsion

There are few factors which can affect the stability of oil-water emulsion:

2.5.1. Effect of emulsifier effect

Surfactant dosage is one of the most important factors influencing the stability of emulsions. Several investigations have demonstrated that emulsifier concentration can strongly influence emulsion stability (Ostberg, *et al*, 1995). It was observed that there existed a concentration window, out of which the stability quickly deteriorated.

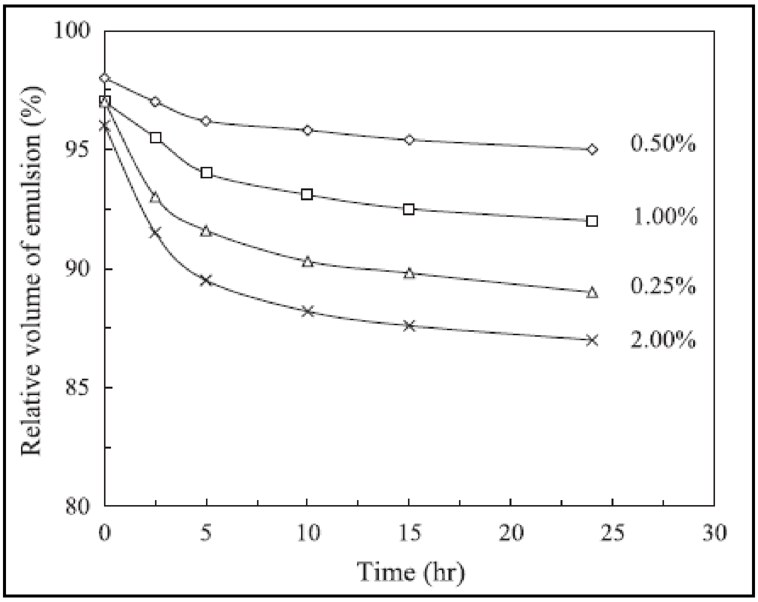

Figure 8: Effect of sorbitan monooleate dosage on emulsification.

Figure 8 shows that the relative volume of emulsion increased as surfactant concentration increased from 0.25% to 0.50%. As the concentration increased further, emulsion stability decreased. This is because at low surfactant concentrations, the emulsion is not stable due to agglomeration of the oil droplets; at high surfactant concentrations, the emulsion destabilization occurs as a result of rapid coalescence. The optimal sorbitan monooleate dosage for the emulsion system was 0.5%. The emulsifier dosage was thus determined to be 0.5% for the following experiments.

2.5.2. Effect of oil/water ratio

Figure 9 shows the effect of volume ratio of oil to water on emulsion stability at the sorbitan monooleate dosage of 0.5%. When the ratio of oil to water was reduced progressively from 9:1 to 5:5, the relative volume of emulsion was increased consistently.

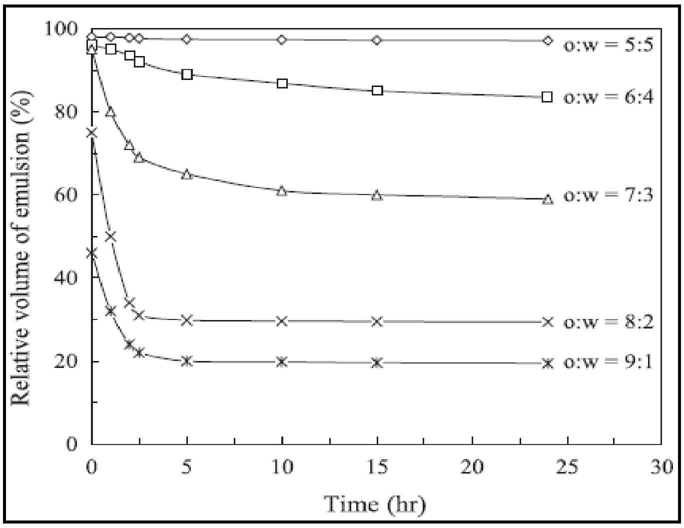

Figure 9: Effect of oil/water ratio on emulsification.

After 24 hours, the relative volume of emulsion was 19.6% at the ratio of 9:1. The relative volume of emulsion was 29%, 60% and 84%, respectively, as the ratio of oil to water was reduced to 8:2, 7:3 and 6:4, respectively. When the ratio was 5:5, the relative volume was 97%. At this ratio, the relative volume was essentially constant over a period of 24 h, indicating that the emulsion was very stable.

2.5.3. Effect of stirring intensity

There are many different ways of preparing emulsions of two liquids that are insoluble or possess only slight mutual solubility. Emulsification is usually achieved by the application of mechanical energy. Initially, the interface between the two phases is deformed to such an extent that large droplets are formed, and these large droplets are subsequently broken up into smaller ones.

During emulsification, the interfacial area between two liquids increases. Liquids tend to minimize this surface area; therefore, mechanical energy is required for emulsification to proceed. It was reported that increasing local dissipation of energy in breaking zone due to the rise of circulation consumption through mixer zone is found to be the most effective to stabilize the emulsion.

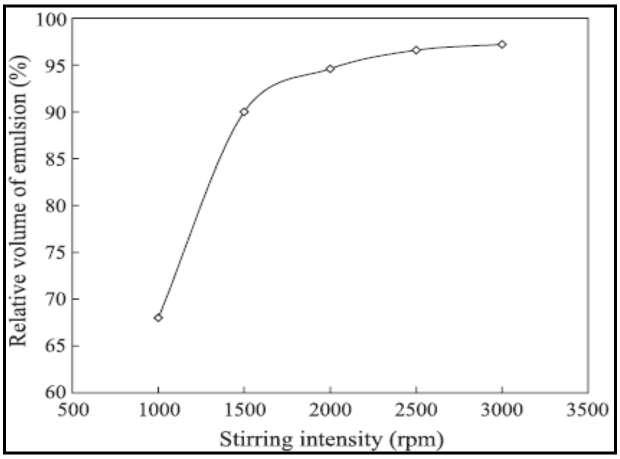

Figure 10: The relationship between stirring intensity and relative emulsion volume after 24 h.

2.5.4. Effect of mixing time

The mixing time is another important factor for emulsification. reported that the radii of the droplets in the emulsion decreased with increasing stirring speed and emulsifying time (Verbich *et al.* 1997). The emulsifier becomes more effective with increased mixing time. However, if the mixing time is too long, the effectiveness of emulsifier will be decreased because the intense stirring will cause the emulsifier to drop out from the oil– water interface.

Figure 11 shows the relative volume of emulsion increased as mixing time increased from 3 to 15 min. As the mixing time increased further, emulsion stability decreased. This experiment also shows that the mean size of the particles decreased very rapidly in the first few seconds and then gradually attained the limiting value after 15 min.

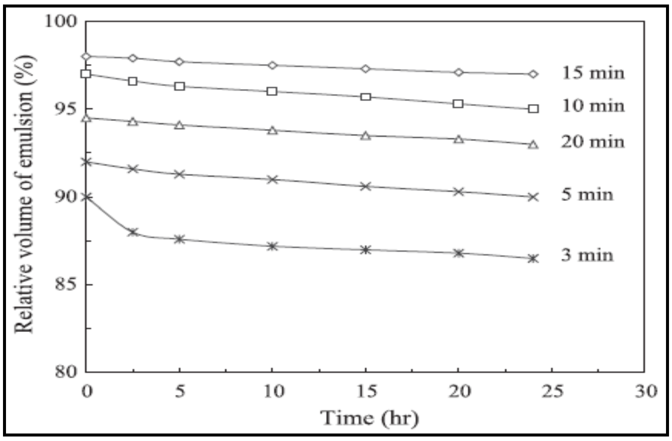

Figure 11: Effect of mixing time on emulsification

To better show the dependence of emulsion stability on mixing time, the relative emulsion volume after 24 h is plotted against mixing time in Figure 12, which clearly indicates that the optimum mixing time is 15 min.

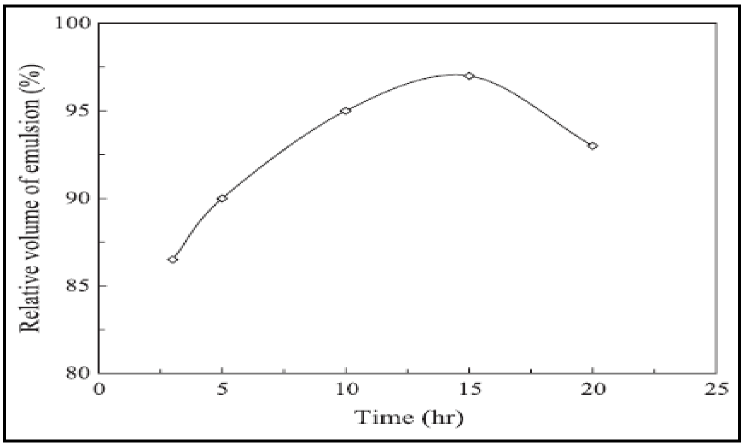

Figure 12: The relationship between mixing time and relative volume of emulsion after 24 h.

From the experiment above, it can be concluded that a stable oil/water emulsion was produced using the diesel as the oil and sorbitan monooleate as the emulsifier. The optimal emulsifier dosage was 0.5% by volume. At lower emulsifier dosages, the emulsion was unstable; the instability at higher emulsifier dosages was a result of rapid coalescence and concentration. The emulsion stability increased with decreasing oil-to-water ratio. The optimum ratio was 1:1 by volume. Higher stirring intensity resulted in more stable emulsion. The optimum stirring intensity was 2500 rpm for the emulsion system investigated. High temperature reduced emulsion stability, and the optimum emulsifying temperaturewas 30 8C. Emulsion stability increased with stirring time up to 15 min, beyond which the stability decreased with further increase in stirring time due to the drop-out of emulsifier from oil–water interface.

2.6. Glycerol Combustion and Emission

Growing capacity in biodiesel production has resulted in higher glycerol by-product. Therefore, there is increasing interest in finding alternative uses for crude glycerol. One option to solve this is by burning the crude glycerol locally for combined process heat and power, replacing fossil fuels and improving the economics of biodiesel production (Bohon, 2010)

However, due to its low energy density, high viscosity, and high auto-ignition temperature, glycerol is difficult to burn. Because of that, several studies had been done to develop a prototype refractory burner to combust glycerol properly and to provide an initial characterization of emissions generated during combustion of crude glycerol in a laboratory-scale (Bohon, 2010)

. Glycerol combustion is examined using 7kW refractory burner (Chen R.H. *et al*., 1990). The burner as shown in figure 13 consists of an air swirl chamber, a ventury restriction, and a refractory-lined combustion chamber. The assembly is designed to be inserted into the first pass of a commercial fire-tube boiler, and provide an insulated environment to promote ignition and flame stability.

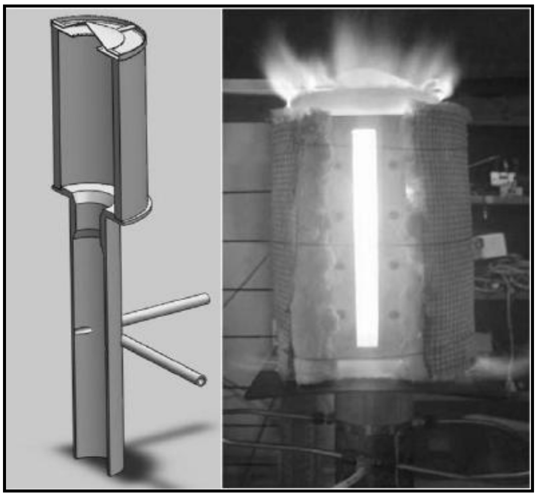

Figure 13: Refractory burner for glycerol combustion

Glycerol grade USP has been tested in this refractory burner. This USP grade glycerol contains 39.1 % of Carbon, 8.7 % of Hydrogen, 52.2% of Oxygen and having higher heating value about 16MJ/kg. Stable flames are possible through a combination of the hot refractory walls and intense gas recirculation. The prototype burner was mounted on a test stand under a large fume hood. U.S. Pharmacopeia (USP) grade glycerol was pumped (28 g/min and 276 kPa) to the spray nozzle and atomized with air (172 kPa).

Gas samples were collected through an uncooled quartz probe at the throat of the exhaust cap, cooled through an ice bath to remove condensing water. Exhaust temperatures were measured using a bare type B thermocouple (Chen R.H. *et al*., 1990). The prototype burner experiments included preliminary measurements of selected volatile carbonyls. A known volume (1.1 L) of burner exhaust gas was drawn through a cartridge containing 2,4-dinitrophenylhydrazine (DNPH) impregnated material purchased (Waters Corp., model 37500, Milford, MA) for the purpose. DNPH reacts with the carbonyls to create DNPH-carbonyl derivatives that were later dissolved in acetonitrile and analyzed by high performance liquid chromatography. In addition to USP grade glycerol and an air blank, methane, propane, and kerosene fuels were also characterized for carbonyl emissions.

The results from this experimental represent that Stable 100% glycerol combustion was achieved for 7kW prototype high swirl burner (using USP grade glycerol). Besides that, the combustor produced gas-phase emissions similar to natural gas and distillate fuel oils indicating low total hydrocarbon emissions and efficient combustion. Interestingly, NO_x emissions from the prototype burner (7–10 ppm, 0% O2) were decreased. These are good finding in order to reduce such harmful emissions (Bohon, 2010)

From the results, it can be conclude that when new value is added in this waste glycerol during biodiesel production, it can optimize the process efficiencies and reduce the impacts of disposal. Although it is not an ideal fuel, waste glycerol might be used in boilers to produce process steam and co-generate electricity with the added advantages of optimizing energy integration, eliminating transportation costs, and displacing the need for fossil fuels.

This work examined efforts to develop a prototype high-swirl refractory burner designed for retrofit applications in package boilers and provide an initial characterization of emissions generated during combustion of crude glycerol fuels.

CHAPTER 3

METHODOLOGY

3.1 Project Methodology

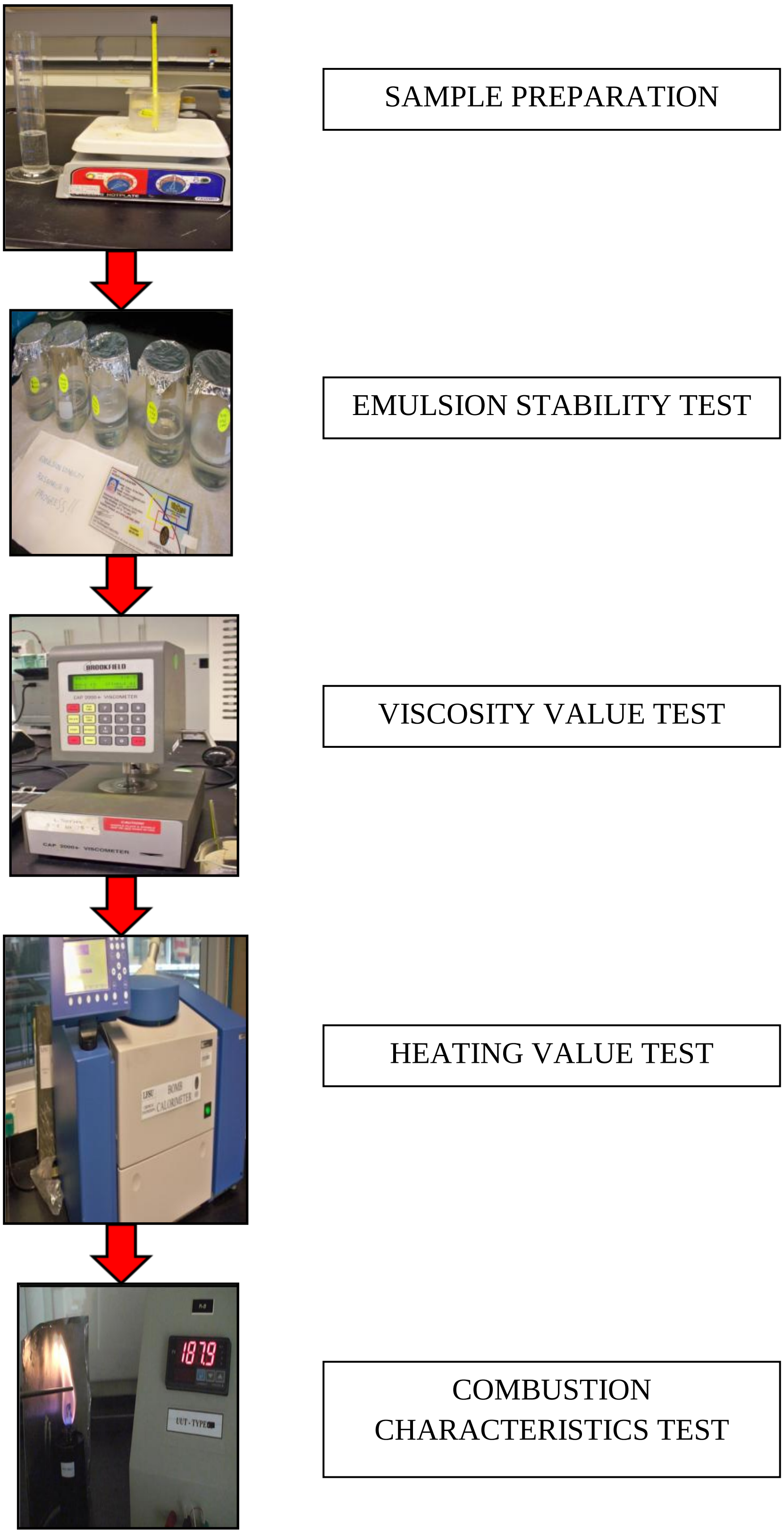

Figure 14: Process Methodology of Emulsion Stability and combustion characteristics test

3.1.1 Emulsion Stability Test

The experiment is performed using equipment such as hot plate, magnetic stirrer, and beaker while the main chemicals used are glycerol, deionized water, and detergent which react as surfactant. 5 mixtures of glycerol and water are prepared as in the table below:

Table 2: Volume ratio of glycerol and water (100ml)

Beaker	Volume Ratio (ml)		Volume of Surfactant (ml)
	Glycerol	Water	
1	90	10	Without surfactant
2	85	15	Without surfactant
3	80	15	5
4	75	20	5
5	70	25	5

1. The solution is mixed using hot plate and magnetic stirrer for 30 minutes at 60-90^{o}C with maximum speed at 700 rpm.
2. The emulsion is put at room temperature for 24 hours.
3. The emulsion is observed everyday to see how fast they separate into two separate layers (demulsification).The steps (2-3) are repeated with other volume ratio of glycerol and deionized water.

3.1.2. Viscosity Test

The sample is tested using Brookfield Viscometer. The Brookfield Viscometer is rotational viscometer, where it measures the torque required to rotate an immersed element (the spindle in the fluid). By utilizing a multiple speed transmission and interchangeable spindles, a variety of viscosity ranges can be measured.

Figure 15: Brookfield Viscometer for Low-Viscosity (LV) type of fluid.

Table 3: Spindle type with the SPDL entry

Spindle type	SPDL Entry
1	1
2	2
3	3

1. Power “ON” is switched on by the left toggle switch UP.
2. “SPDL” is pushed and a number matching with the spindle type is keyed -in.
3. The motor speed is set at 12 rpm. The motor button “ON” is turned on by the right toggle switch UP.
4. “Auto Zero” is pushed to set zero. The motor is turned off by the right toggle switch DOWN.
5. A spindle is installed on the rotor tip. The sample beaker is put below the viscometer.
6. The viscometer is lowered until the notch on the spindle submerges into the sample.
7. The motor speed is set at the lowest by adjusting the knob on the left side.
8. The motor is turned on. The viscometer starts displaying the reading
9. The motor is turned off and change, then the motor is turned on again and the reading is measured.
10. Steps 5-10 are repeated with other type of spindles. Remember to key-in the spindle entry every time we change spindles (Step 2).

3.1.3. Heating Value Test

Heating value for glycerol-water mixture is measured using Bomb Calorimeter equipment. The bomb calorimeter works by conservation of energy. By suspending a combustion reaction in the center of a known mass of water, the majority of the heat must go into the water. Some heat is lost or gained to various side reactions and the non-isothermal nature of the device.

Figure 16: Bomb Calorimeter Equipment

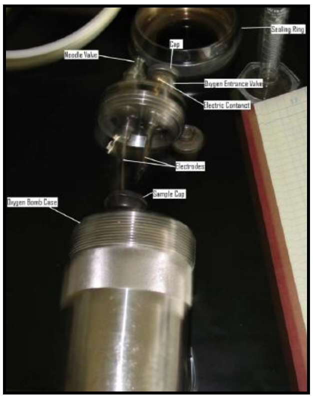

Figure 17: Oxygen bomb case and sample cup

1. The oxygen bomb case is cleaned and dried properly before putting any sample.
2. The wire is cut around 5cm to be tied at the electrode
3. Small thread is tied at the center of the wire.
4. The sample is weighed using weight balance and is put inside the sample cup.
5. The sample cup is inserted into oxygen bomb case.
6. The value of the sample is inserted at the screen monitor of bomb calorimeter which is in stable condition.
7. The oxygen bomb case is inserted into the bomb calorimeter.
8. The button start is clicked and the bomb calorimeter is running automatically.
9. The value is recorded after it displayed the heating value of the sample.

3.1.4. Combustion Characteristics or Flame Stability Test

Combustion characteristics or flame stability test for glycerol-water mixture are tested using thermocouple type J inside the fume hood. Ethanol is added to the mixture to induce the volatility of the glycerol, thereby increasing ease of glycerol-water combustion.

The mixture which is tested for combustion test is as follow:

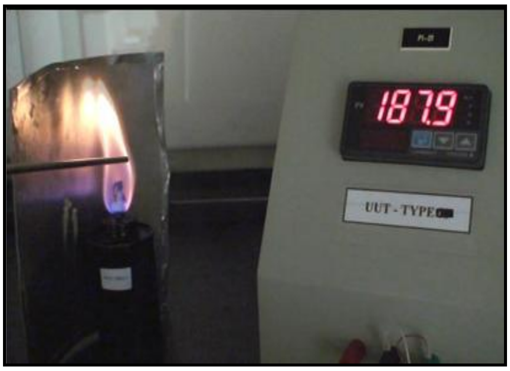

Figure 18: Combustion Characteristics or Flame Stability Test using Type J Thermocouple

Table 4: Volume ratio of glycerol, ethanol and deionized water in 100ml

Sample	Glycerol (ml)	Ethanol (ml)	Deionized water (ml)
1	-	100	-
2	100	-	-
3	10	90	-
4	30	70	-
5	45	45	10
6	35	50	15

1. Sample is put in the oil container and the thermocouple is set up as shown in the figure 15.
2. The thermocouple is switched on. The thermocouple will display the current temperature which is room temperature.
3. Video recorder is switch on to record the burning of the glycerol-water mixture with addition of ethanol.
4. The mixture is burnt in the oil container and the thermocouple is put on the burning flame.
5. Video recording is started when the temperature rise from room temperature and keep increasing. The video is recorded within 1 minute.
6. The flame characteristics as well as the stability are observed and the data is recoded.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Emulsion Stability Test

Table 5: Emulsion observation for 90 ml of glycerol and 10ml of deionized water without adding any surfactant

1. 90ml glycerol, 10ml deionized water

Time	Observation
5 min	Stable
1 hour	Stable
1 day	Stable
2 days	Separate into 2 layers

Table 6: Emulsion observation for 85 ml of glycerol and 15ml of deionized water

2. 85ml glycerol, 15ml deionized water

Time	Observation
5 min	Stable
1 hour	Stable
2 hours	unstable

Table 7: Emulsion observation for 80ml of glycerol, 15ml of deionized water and 5ml of detergent.

3. 80ml glycerol, 15ml deionized water, 5ml detergent

Time	Observation
5 min	Unstable
1 hour	Unstable

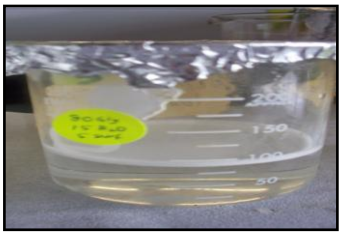

Table 8: Emulsion observation for 75 ml of glycerol, 20ml of water and 5ml of surfactant

4. 75ml glycerol, 20ml deionized water, 5ml detergent

Time	Observation
5 min	Unstable
1 hour	Unstable
2 hours	Unstable

Table 9: Emulsion observation for 70 ml of glycerol, 25ml of water and 5ml of surfactant

5. 70ml glycerol, 25ml deionized water, 5ml detergent

Time	Observation
5 min	Unstable
1 hour	Unstable
2 hours	Unstable

From the result above, it shows that the ratio of 90 ml glycerol and 10 ml water without surfactant and ratio of 85ml glycerol and 10ml water gave more stable emulsion compared to other ratio of emulsion which added with detergent as surfactant. This is because; glycerol is proven to be miscible with water so that, no surfactant is needed to stabilize the mixture. Faster separation is always occur but if the emulsion can still stable for 1 day, is it a good observation.

4.2. Viscosity Test

Temperature : 20^oC

Speed : 300rpm

a) Spindle Type : 1

Table 10: Viscosity for every sample for spindle type 1

Sample	Viscosity
1	0.02
2	0.0169
3	0.0163
4	0.0156
5	0.0138

b) Spindle Type : 2

Table 11: Viscosity for every sample for spindle type 2

Sample	Viscosity
1	0.174
2	0.162
3	0.153
4	0.143
5	0.0497

a) Spindle Type : 3

Table 12: Viscosity for every sample for spindle type 3

Sample	Viscosity
1	1.967
2	1.587
3	1.507
4	1.391
5	1.267

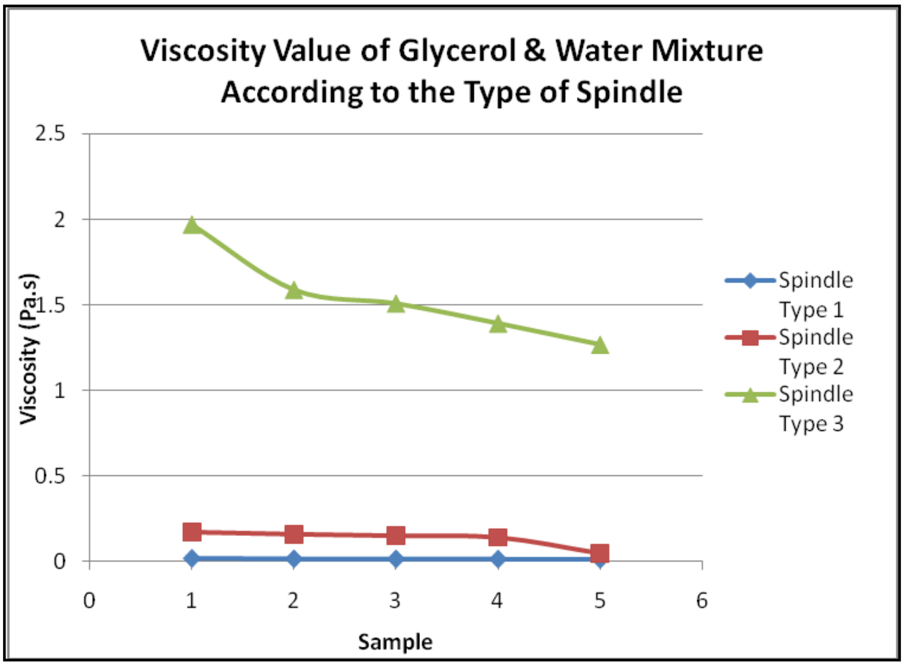

Figure 19: Graph of viscosity value of glycerol-water mixture according to the type of spindle

Based on the result above, the viscosity value of glycerol-water mixture can be measured using LV (Low Viscosity) type viscometer. There are 3 type of spindle. Spindle 1, 2 and 3. Among of these spindles, only spindle type 3 gave the viscosity values same like the theoretical value of glycerol and water viscosity. Theoretically, the viscosity value of glycerol is range from 1.2 to 1.5 Pa.s. From the experimental part, the viscosity value using spindle type 3 ranged from 1.2 to 1.9 Pa.s.

4.3. Heating Value Test

Temperature: 25^{o}C

Equipment: Bomb Calorimeter

Table 13: The heating value of glycerol-water mixture with addition of ethanol

Sample	Mass (g)	Heating Value (J/g)
100 ml Glycerol	0.2028	15961
90ml Ethanol: 10ml Glycerol	0.2533	15641
50 ml Ethanol: 50 ml Glycerol	0.2695	15426
30 ml glycerol : 50 ml Ethanol: 20 ml Deionized Water	0.2590	15323

When determining the experimental value of chemical or physical properties such as viscosity and heating value, it is must be compared with the standard material plot of viscosity and heating value rather than taking the theoretical value of the glycerol and compare with the experimental value.

4.4. Combustion or flame stability test

a) 100ml of ethanol

b) 100ml of glycerol

c) 90ml of ethanol, 10ml of glycerol

d) 70ml of ethanol, 30 ml of glycerol

f) 45ml of ethanol, 45 ml of glycerol,10 ml of DW

e) 50ml of ethanol, 50 ml of glycerol

g) 50ml of ethanol, 35 ml of glycerol,15 ml of DW

Table 14: Flame characteristics for every sample

Sample Observation	a	b	c	d	e	f	g
Length	5.4cm	2.5cm	6.0cm	3.0cm	3.3cm	4.0cm	2.6cm
Colour	Orange, yellow, blue	Orange, blue	Orange, yellow, blue	Orange, blue	Orange, blue	Orange, blue	Orange, blue
Burning Characteristic	Burnt very vigorously Unstable	Weak Unstable	Strong burning stable	Medium burning stable	Medium burning stable	Medium burning stable	Medium burning stable

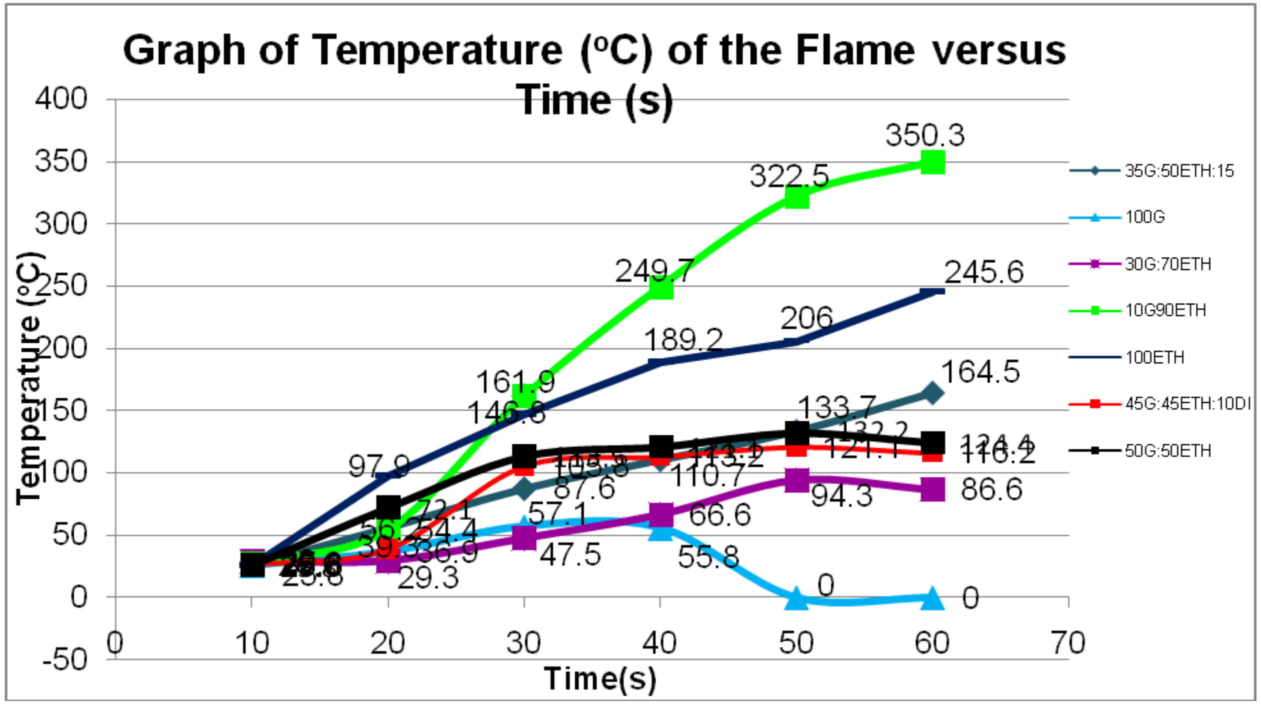

Figure 20: Graph of temperature of the flame versus time

According to the result of combustion characteristics or flame stability test, it can be observed that glycerol cannot burn because of low volatility characteristic. In order to induce the volatility to ease the glycerol combustion, alcohol such as ethanol is added with glycerol to increase the combustion. This is because; ethanol has higher volatility than glycerol. So, good combination of mixture between glycerol, alcohol and also water can improve and increase the flame stability.

In terms of the colour, length, temperature as well as the flame stability, the mixture of 90ml of ethanol and 10ml of glycerol gave the good result. This is because, when burning ethanol only, ethanol burnt very vigorously in the higher temperature and the flame is unstable. While for glycerol burning only, it cannot burnt and extinguish in a few seconds. So, when combining the ethanol and glycerol, it can reach higher temperature up to 350^{o}C (according to the graph) because of the higher volatility of ethanol, it produced stable flame and more blue flame produce compared to the other mixture.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

In conclusion, the factor of temperature, mixing speed, and the type of oil and water that we use can affect the stability of emulsionIt is hard to have emulsion that can be stable for long period of time but, as long as it can be stable within few hours, it is good observation to improvise the method to become more stable using good additive or surfactant.From the emulsion stability test, it can be conclude that the amount of 90 ml glycerol and 10 ml of deionized water can be stable within 2 days compared to other samples.

Ethanol can induce glycerol to burn and glycerol can improve the combustion as well as flame stability.Thus the glycerol-alcohol system for biodiesel production is particularly suitable for combustion.

5.2. Recommendations

For emulsion stability test and combustion characteristics test, few matters which should look into are:

- In order to get homogenous mixture or stable emulsion for long period of time, it is recommended to blend the mixture of glycerol, water and additive rather than using magnetic stirrer. This is because; the speed of blender is higher than the speed of magnetic stirrer. So, the mixture is perfectly mixed and the emulsion which is created can stable for long time.

- It is recommended to use separation funnel for emulsion stability test because it accurately determine whether the mixture is stable or separate into two phases if the mixture is immiscible rather than using beaker and using naked eyes to observe the stability of emulsion because different people will see different observation. Refractive index or turbidity meter can be used how much the percentage of glycerol has been separated from water. These all are recommended and proper methods in determining the stability of glycerol-water emulsion.

- The specific area to combust the mixture is properly needed such as furnace or combustion burner in order to get an accurate result for combustion characteristics or the stability of the flame.

- In order to get better observation of flame stability rather than using manual video camera, it is recommended to use frame grabber. A frame grabber is an electronic device that captures individual and employed as a component of a computer vision system. For this combustion characteristic test, it is very useful since it can capture the flame colors, the height accurately and automatically save in the computer attached with the frame grabber USB.

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APPENDICES

PROJECT KEY MILESTONE

No	Detail	Week														
		1	2	3	4	5	6		7	8	9	10	11	12	13	14
1	**Project Work Continues** **-** Emulsion test (try and error) - Research on other method and oil used	■	■	■				**Mid Semester Break**								
2	**Submission of Progress Report 1**				■											
3	**Project Work Continues** - Emulsion Test using glycerol				■	■	■									
4	**Submission of Progress Report 2**									■						
5	**Seminar**									■						
6	**Project Work Continues** - Viscosity Test - Heating Value Test - Flame Stability Test								■	■	■	■	■	■	■	■
7	**Poster Exhibition**												■			
8	**Submission of Dissertation Final Draft**															■
9	**Oral Presentation**															■
10	**Submission of Dissertation (hard bound)**															■