TEAM RESEARCH PROJECT REPORT

Project # 2: Making Biodiesel for Research and Education

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ABSTRACT

Continued use and need of oil has depleted natural resources, also resulting in air pollution from exhaust emission of vehicles. Biodiesel provides a cleaner, renewable fuel source that can be made from waste cooking oil. Biodiesel production industry has been expanding since 2004, in 2008 700 million gallons of biodiesel was produced. One issue with the expanding need for biodiesel is its sustainability, crop derived biodiesel, such as soybean, uses land space and water resources. This problem can be amplified with the continued need for expansion of biodiesel production. Waste cooking oil is a great renewable resource that can produce biodiesel without the problem of soybean. One issue with using waste cooking oil is the high content of free fatty acids, which is harmful to the conventional alkaline catalytic transesterification. Free fatty acids can be removed through an acid catalytic esterification through the conversion of free fatty acids into esters. In this report, several experiments with different parameters were conducted for the purpose of converting free fatty acid content below 1%, which is the highest free fatty acid level acceptable for the alkaline based catalytic transesterification. The results show that the best configuration of experimental parameters are 50:1 molar ratio of methanol to free fatty acid, and 15% of sulfuric acid to free fatty acid, with a temperature of 56°C. Under these circumstances the free fatty acid level was reduce from 5.2% to 0.47% after a 5 hour 47 minute reaction time. Also performed in this experiment was the citric acid washing for crude biodiesel, lab scale biodiesel production, and GC analysis of biodiesel samples.

KEY WORDS: Biodiesel, Free fatty acids, Titration, Transesterification, Molarity, Soap, and Concentration.

1. INTRODUCTION

In 2007, America was using 20.6 million barrels of oil per day; this number has been estimated to be on a 2% increase every year to sustain our ways of life. The United States is also estimated to produce one-fourth of the world's carbon emissions. With the increased need of oil, need to import foreign oil, and amount of pollution emitted from the burning of oil, the United States has realized the many societal and economic problems associated with the continued dependence on oil. Many alternative energy sources to petroleum derived diesel have become of interest to help lower the dependence of oil, biodiesel has become of special interest due to the fact that many automobiles and machines do not have to be replaced at expensive cost to use biodiesel as a replacement of oil. The burning of biodiesel also reduces the amount of particulate matter, CO_2, and sulfur oxides emitted into the air, which can be a clean burning alternative for oil. There are many sources that can produce biodiesel that are renewable such as waste cooking oil, soybeans, corn, and algae. All of which can provide an energy supply so less importing is necessary.

Waste cooking oil is one of the most inexpensive and feasible substances to produce biodiesel from. Unlike many biodiesel feedstock developments, waste cooking oil does not consume water in the production of waste cooking oil. Using waste cooking oil also provides a solution to disposing of waste cooking oil, much of which ends in landfills and some in possible water sources which can be very harmful to the environment. Rather than throwing away waste cooking oil, it can be used as a recyclable energy source that is relative cheap to buy from many restaurants instead of current disposal methods.

2. LITERATURE REVIEW

Air pollution has become an increasing problem in the United States since the Industrial Revolution; today mortalities can be associated with "levels of air pollution that contain particulate matter with levels of fine, inhalable, and sulfate particles than with the levels of total particulate pollution, aerosol acidity, sulfur dioxide, or nitrogen dioxide" (Dockery et al. 1993). Sydbom (2000) elucidates "of the motor vehicle generated air pollutants, diesel exhaust particles account for a highly significant percentage of the particles emitted in many towns and cities." Sydbom (2000) goes on to explain that diesel engines produce a high amount of "nitrogen oxides, which are particularly prone to cause irritation of the upper respiratory tract." With air pollution problems as well as problems with limited natural resources, biodiesel has become an increasingly popular alternative fuel source. Biodiesel can be blended with diesel to lower toxic emissions, and can work in many diesel engine automobiles as well as energy fuels for power plants. This allows many cars to stay on roadways without having expensive engine replacements or the building of new cars altogether. Biodiesel is also a renewable resource produced from plant oils and animal fat, which can lower the United States' dependence on foreign oil, which can stimulate the economy (Agnew et al. 2009). Both plant oils and animal fat are biodegradable and contain no sulfur or nitrogen which in turn, when burned is much cleaner than regular diesel.

Biodiesel can have many different sources such as soybeans, corn, algae, and waste cooking oil. Waste cooking oil can be one of the best producers of biodiesel since it has a continuous resource from many restaurants and fast-food chains. Soybean fields and corn fields require a large amount of water for growing, taking land resources and using possible water sources could be detrimental. Using waste cooking oil eliminates the need for extra land and water, making the most attractive choice for biodiesel production. Other products can also be made from the byproducts of the biodiesel.

Biodiesel can be made by method of transesterification, "which converts triglycerides into esters" (Agnew et al. 2009). The waste cooking oil, or triglyceride, is mixed with alcohol and driven by a catalyst; in the transesterification process the "alcohol molecule replaces the glycerin molecule" producing glycerol and an alky ester, or biodiesel (Agnew et al. 2009). The

glycerol is denser than the biodiesel and settles to the bottom, the glycerol can then by extracted and the biodiesel can be washed for further purification. According to Gerpen (2005), this process was developed in the 1940s for soap and explosives production; this process was not discovered as an alternative fuel source until the late 1970s and early 1980s. Soap forms when glycerol is mixed with NaOH, which then can be used as a soap product. Although soap production from glycerol is a good product of glycerol, soap can also be produced during the transesterification process which is not wanted and must be avoided.

Waste cooking oil can have a large amount of free fatty acids; the longer the cooking oil is used the amount of free fatty acids in the oil increase due to high cooking temperatures and hydrolysis. According to Canakci (2001), "as free fatty acids levels increase this becomes undesirable because of the loss of feedstock as well as the deleterious effect of soap on glycerin separation." The free fatty acids content can be reduced in the waste cooking oil by reacting the free fatty acids with an alcohol, which is known as esterification. The acceptable free fatty acid content in the waste cooking oil must be 1% or lower. If there are too much free fatty acids in the oil, they can react with the NaOH to form soap which makes the separation of the glycerol very difficult. To prevent soap formation, the waste cooking oil must first undergo an acid pre-treatment to reduce the free fatty acid content. The pre-treatment step improves the yield of the biodiesel by esterifying the free fatty acids with methanol by acid catalysis (Wang et al. 2006).

Although there are many benefits to using waste cooking oil as a source of biodiesel, there are also some problems besides have high free fatty acid content. The washing process of biodiesel can use a large amount of water for purification processes. Recent research has found that the use of citric acid can reduce the amount of water needed for the washing process by reducing the number of times needed to wash the biodiesel once it is made (Dmytryshyn et al. 2004). This process greatly improves the efficiency of biodiesel washing and makes the use of waste cooking oil a great choice as a biodiesel resource.

3. GOALS AND OBJECTIVES

The overall goals for this research project is to determine a more sustainable way to make biodiesel that is viable and can sustain our needs and when mixed with regular diesel, lower harmful emission such as particulate matter and sulfur oxides. To obtain these goals we must determine whether or not citric acid can lower water usage in the washing stages of biodiesel. Also, we must determine if there is a set of parameters that can be used to guarantee that free fatty acid concentration in waste cooking oil can be reduced to 1% or under consistently.

4. RESEARCH STUDY DETAILS

4.1 Acid Pre-Treatment

Waste cooking oil has a high free fatty acid concentration and must be pre-treated to lower the amount of free fatty acid in the waste cooking oil. To do this, 500mL of waste cooking oil was obtain, the initial free fatty acid concentration was calculated by performing a titration using ethyl alcohol as a solvent and NaOH to neutralize the free fatty acids. From this original free fatty acid calculation, the pre-treatment material amounts could be determined.

4.1.1 Original Titration Procedure

1. 1mL sample of original waste cooking oil was placed into a 250mL Erlenmeyer flask.
2. 10mL of ethyl alcohol was placed into the flask; the flask was shaken to dissolve the waste oil.
3. 3 drops of bromophenol indicator was also added to the flask.
4. 0.5% concentration of NaOH was added to the flask in 30µL increments and shaken lightly into the solution. This was repeated until the color of the solution changed to a light pink color (See Photo 1 & 2).
5. The amount of NaOH was recorded; from this amount the initial free fatty acid concentration could be determined.

4.1.2 Acid Pre-Treatment Procedure

1. After determining the mass of the free fatty acids from the initial percent weight in a 500mL sample of waste cooking oil, the amount of H_2SO_4 can be determined by taking 10 to 15% of the free fatty acid weight and suing that amount of H_2SO_4.
2. Using a 9:1 molar ratio the amount of methanol could be determined for the pre-treatment of the waste cooking oil.
3. The waste cooking oil is placed into the reactor, then place into a hot water bath on a hot plate and heated to the desired temperature for carrying out the reaction.
4. The H_2SO_4 is added to the methanol very slowly under a hood, and then added to the cooking oil in the reactor.
5. 1.5mL samples are taken from the reactor at designated times and placed in individual test vials. After the sample is taken, the test vial is then immediately placed in the freezer to cool.
6. After all desired samples are taken and cooled; titrations are performed on each sample using the procedure described in section 4.1.1 (See Photo 3).
7. From the recorded amount of NaOH, the conversion percent of free fatty acid is calculated and graphed. (See Table 1 for Experiment 1 amount calculations) (See Graph 1 for free fatty acid conversion rates)

4.2 Lab Scale Production

Different feedstock oils and alcohols were used for biodiesel production in the lab and the GCMS analysis was performed for all the samples to examine the profile of the biodiesel products. Citric acid washing was also conducted on some of the resulting biodiesels. Specifically, two approaches were tested for lab-scale biodiesel production: the empirical approach is convenient but not very accurate; therefore the titration approach is adopted as the final approach for all the lab scale biodiesel productions.

4.2.1 Empirical Approach

1. 40mL of waste cooking oil is placed into a 100mL Erlenmeyer flask.
2. The empirical ratios of 0.3g/100mL oil and 0.4g/100mL oil are adapted and the amount of NaOH is weighed out using weighing paper.
3. Using the 4:1 ratio, 10mL of methanol is measure using a graduated cylinder.
4. The pre-weighed amount of NaOH is added and dissolved into the 10mL of methanol.
5. The mixture is then placed into the Erlenmeyer flask containing the waste cooking oil.
6. The flask is cover and shaken vigorously for 1 minute.
7. The flask is placed into a microwave for 30 seconds on high power, cooled for 15 seconds, and then placed back into the microwave for another 30 seconds on high power.
8. The flask is then allowed to cool, a separation between the biodiesel and glycerol will begin to form.
9. The two layers are allowed to settle out, revealing a distinct lower dark layer of glycerol and a lighter yellow layer of biodiesel. At this point the biodiesel is ready for the washing phase (See Photo 4).

4.2.2 Titration Approach

1. Place 1mL of waste cooking oil into a 100mL Erlenmeyer flask.
2. Add 10mL of ethanol to the flask and 3 drops phenolphtalein, and shake until dissolved.
3. Titrate with 0.05% NaOH solution until color changes to pink.
4. The amount of NaOH consumed in titration is supposed to neutralize the free fatty acids in the waste cooking oil.
5. Calculate the additional NaOH as the catalyst by rule of 3.1g/L of waste cooking oil.
6. Calculate the amount of methanol according to a 6:1 molar ratio of waste cooking oil, which is ¼ in volume of waste cooking oil. Pre-mix the methanol with the NaOH, making sure NaOH is completely dissolved in the mixture.

7. Heat the waste cooking oil to 60°C, use stir rod to continually mix the waste cooking oil in the beaker. When temperature is stable, add mixture of NaOH and methanol to the waste cooking oil.
8. Heat the mixture at a consistent 60°C for roughly 2hrs. Remove from heat and remove stir bar.
9. Let the mixture set for at least 24hr so glycerol can settle out to the bottom of the beaker.

4.3 Biodiesel Washing

After crude biodiesel has been produced, it must go through a washing phase remove the NaOH and drop the pH, it is also used to remove the residual glycerol, NaOH, and soap. The glycerol is soluble in water, where the biodiesel is not which results in two distinct layers forming. The washing phase can require a large amount of water to be used, so two different washing techniques are used to compare if using citric acid could be a better washing technique that cleans the biodiesel and uses less water compared to the water washing technique. Citric acid neutralizes the NaOH as well as the soap, making it a good substance for biodiesel washing.

4.3.1 Water Washing Procedure

1. Place the 40mL crude biodiesel into large funnel with an on/off spout.
2. Using a graduated cylinder, measure 40mL of room temperature filtered water.
3. Very slowly, pour the water in the funnel. Do not let the water directly hit the crude biodiesel, but allow the water to run down the side of the funnel into the crude biodiesel.
4. Let the funnel sit untouched for 30 minutes to allow the water to settle out of the biodiesel.
5. Turn the spout on and drain the water from the funnel into a 100mL beaker.
6. Observe the clarity of the water, if cloudy repeat the procedure until water becomes clear.

4.3.2 Citric Acid Washing Procedure

1. Using a graduated cylinder, measure 40mL of water and place into a 100mL beaker.
2. Weigh the wanted amount of citric acid onto weigh paper (either 1, 2, 3, or 4% citric acid).
3. Place the citric acid into the water and stir until all citric acid is dissolved in the water.
4. Repeat steps 3-6 in section 4.3.1
5. Compare the water washing and citric acid washing clarity of the water after every wash to help determine the best washing method (See Picture 5).

4.3.3 "3/27" Methanol Test

1. Mix 3mL biodiesel with 27mL methanol.
2. Shake vigorously for 10 seconds under 20°C conditions.
3. Let mixture settle for 10 minutes.
4. If the biodiesel is sufficiently washed, there should only be one phase of mixture; otherwise there would be 2 distinct layers, a clear mixture and a murky bottom layer which is the residual impurities.

4.4 Preparation of GCMS Sample

1. Take 0.1µL of washed biodiesel and dilute with 10mL DCM (10,000ppm).
2. Take 1mL of diluted solution and dilute with 10mL of DCM (1,000ppm).
3. Take 1mL of diluted solution and dilute with 10mL of DCM (100ppm).
4. Inject into GCMS for analysis.

5. ANALYSIS: RESEARCH RESULTS

Using the acid pre-treatment procedures describe above, four acid pre-treatment were performed using different parameters to determine what could be a set of configuration that could result in the largest conversion of free fatty acids in the given amount of time. The first acid pre-treatment and an initial free fatty acid percent weight of 4.76%, using this information 10% of H_2SO_4 and a 9:1 molar ratio of methanol to free fatty acid was used in the reactor. The reactor was kept at an average temperature of 58.1°C for a total of 167 minutes. After the allotted time, the ending percent weight conversion of free fatty acids was 47.5%, lowering the end free fatty acid percent weight to 2.51% (**Table 1**). Experiment 2 had an initial free fatty acid percent weight of 4.57%, using this information 15% of H_2SO_4 and a 9:1 molar ratio of methanol to free fatty acid was used in the reactor. The reactor was kept at an average temperature of 55.9°C for a total of 197 minutes. The ending percent weight conversion of free fatty acids was 57.5%, lowering the end free fatty acid percent weight to 1.94% (**Table 1**). Experiment 3 had an initial free fatty acid percent weight of 5.56%, using this information 15% of H_2SO_4 and a 9:1 molar ratio of methanol to free fatty acid was used in the reactor. The reactor was kept at an average temperature of 55.8°C for a total of 407 minutes. The ending percent weight conversion of free fatty acids was 64.74%, lowering the end free fatty acid percent weight to 1.96% (**Table 1**). Experiment 4 had an initial free fatty acid percent weight of 5.03%, using this information 15% of H_2SO_4 and a 50:1 molar ratio of methanol to free fatty acid was used in the reactor. The reactor was kept at an average temperature of 55.9°C for a total of 347 minutes. The ending percent weight conversion of free fatty acids was 90.55%, lowering the end free fatty acid percent weight to 0.475% (**Table 1**). **Figure 1** shows the free fatty acid conversion for Experiment 1, 2, 3, and 4 with a logarithmic line representing the rate of the free fatty acid

conversion. All of the regression coefficient R^2 values are above 0.9, meaning the results are acceptable.

Using the biodiesel washing procedure describes above, two different experiments were performed to test which washing treatment is most effective. The first experiment was the washing of the biodiesel using water and 1% citric acid. The biodiesel was only washed one time to observe the difference between the collected water through the funnel. **Picture 5** illustrates the clarity of the water versus the citric acid. From the picture, the citric acid wash resulted in much clearer wash water. The next part of the experiment used different concentration of citric acid (2, 3, and 4%) as well and pure water to wash the biodiesel (**Picture 6**). There was only a very slight difference between the individual concentrations of citric acid (**Table 3**).

Table 4 shows the amounts of the materials used in the empirical approach for making biodiesel. Due to the reaction between the NaOH and the free fatty acids there is an observable layer of soap that forms on top of the biodiesel. Batch 1 contained 0.12g NaOH, while Batch 2 contained 0.16g NaOH. Batch 2 contained more soap that was produced.

Table 5 show the yield of five different feedstocks, the virgin cooking oil had the highest yield whereas the virgin cooking oil using ethanol as a catalyst had the lowest yield. Each feedstock oil had a distinct color variation which was dependent on the time oil was used for and type of cooking oil it was. The zoo oil had the darkest coloration, and the virgin cooking oil had the lightest coloration (**Picture 7 and 8**).

6. CONCLUSION

The results discussed in the acid pre-treatment section provides the chance to find the influence of different parameters on the free fatty acid conversion rate to determine an optimal configuration for free fatty acid conversion of waste cooking oil. In Experiment 1 and 2, the initial free fatty acid concentration was very close in value percent. The concentration of sulfuric acid affected the rate of conversion, which outweighed the influence of slight temperature change on the reaction rate. This can be seen in **Figure 1**. Since sulfuric acid determined the conversion rate, temperature can be reduced properly to conserve energy during the pre-treatment process. This can be helpful in large scale production to conserve energy and lower large scale production costs. Experiment 2 and 3 had the same parameters but different initial free fatty acid concentration, the results illustrated that the higher the initial free fatty acid concentration the better conversion rate happened. This may be attributed to the relatively higher concentration of the reactants of the free fatty acids and the methanol. Experiment 4 resulted in the highest conversion of free fatty acids, resulting in a 98% conversion. This is due to the excess of methanol pushed the reaction forward converting a

higher percent of free fatty acids. From these tests, Experiment 4's parameters had the best set of condition to result in the highest conversion of free fatty acids.

Citric acid can provide an alternative for using pure water, which can lower the amount of water used for washing biodiesel. From these washing experiments, the citric acid provides much clearer water result in the first few round of washing compared to using water only. The optimal percent of citric acid used for washing cannot be determined through observational tests only, there was little to no difference between the different percentages. Citric acid can reduce the impurities such as soap and NaOH in the biodiesel product (Picture 5).

In lab scale production, the virgin cooking oil had the lightest coloration and the highest yield of free fatty acid conversion. This is because the oil had the lowest amount of free fatty acid, and it was unused oil. The zoo waste cooking oil had the darkest coloration because it has been stored for the longest amount of time, and there was a high amount sulfuric acid in the oil which can result in a dark coloration. Using ethanol as a catalyst resulted in the lowest amount of free fatty acid conversion; this may be due to ethanol and KOH loss during the operation.

Figures 2-6 shows the GCMS results of five different biodiesel samples. Different Feedstock oils will produce biodiesel with different ester profiles; this is due the difference in the composition of the parent oils. From the biodiesel made from waste cooking oil and methanol, the dominant esters are C16s and C18s; but the relative concentrations vary among different sources of the waste cooking oil. For the canola oil with methanol the dominant components are also C16s and C18s, with a small amount of C20s. The canola oil with ethanol had a much larger range of esters and the dominant components were C17 and C18. This shows that using different alcohols has a direct influence over the profile of the final biodiesel product.

7. RECOMMENDATIONS

A recommendation suggested of future work of the acid pre-treatment is to perform more parallel experiments to better understand the influence of the concentration of sulfuric acid, different reaction temperatures, as well as different methanol concentration amounts. All of these factors can interact together to form the most cost efficient way to perform the pre-treatment in a large scale scenario.

Another recommendation suggested for future work is to determine a quantitative way to test the different concentration of citric acid to determine the best amount to use to wash biodiesel. Only performing an observable test was not enough for the varying concentrations, it was only effective for showing that citric acid produced a better rinse than water alone.

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9. APPENDIX I: NOMENCLATURE USED

mL = milliliters

µL = micro-liters

C = carbon atom

CO_2 = carbon dioxide

DCM = methylene chloride

EE = ethyl ester

FFA= free fatty acid

GCMS= gas chromatography/mass spectrometric

H_2SO_4= sulfuric acid

L = liters

ME = methyl ester

NaOH = sodium hydroxide

R^2 = regression coefficient

WCO = waste cooking oil

10. APPENDIX II: RESEARCH SCHEDULE

Week 1	Lab skill: Titration for acid value; measurement of soap value in crude biodiesel; calculation of iodine value Biodiesel basics: what is biodiesel; the benefits of biodiesel; overview of biodiesel production; basic chemistry
Week 2	Acid Pre-treatment of waste cooking oil with high FFA concentration Introduction of FFA and its negatives effects; how to remove FFA
Week 3	Acid Pre-treatment of waste cooking oil with high FFA concentration Introduction of FFA and its negatives effects; how to remove FFA
Week 4	Acid pre-treatment of waste cooking oil with high free fatty acid concentration Biodiesel washing test
Week 5	Acid pretreatment of waste cooking oil with high free fatty acid concentration
Week 6	GC processing, lab wrap up

11. APPENDIX III: GRAPHS

Figure 1: FFA Conversion for Experiment 1, 2, 3, & 4 under varying experimental parameters

Figure 2: GCMS analysis of biodiesel made from waste cooking oil from restaurants from the Cincinnati zoo with methanol

Figure 3: GCMS analysis of biodiesel made from waste cooking oil from Market Point at the University of Cincinnati with methanol.

Figure 4: GCMS analysis of biodiesel made from waste cooking oil from Center Court at the University of Cincinnati with methanol

Figure 5: GCMS analysis of biodiesel made from canola oil with methanol

Figure 6: GCMS analysis of biodiesel made from canola oil with ethanol

12. APPENDIX IV: TABLES

Table 1: Acid Pretreatment Experiment materials, amounts, initial and ending free fatty acid percentage

Experiment #	1	2	3	4
WCO (ml)	500	500	500	400
Initial FFA (% wt)	4.756	4.567	5.557	5.026
H_2SO_4 (ml)	1.0, 10%	2.0, 15%	2.1, 15%	1.5, 15%
Methanol (ml), ratio	22, 9:1	30, 9:1	35, 9:1	134, 50:1
Ave. Temp.	58.1	55.90	55.76	55.91
End FFA	2.513	1.941	1.955	0.475
Conversion	47.50	57.50	64.74	90.55

Table 2: Amount of waste cooking oil, water, and citric acid used for washing experiments

	Funnel 1	Funnel 2
Washing method	Tap water	Acidic water (1% citric acid)
Water volume (ml)	40	40

Table 3: Percent of citric acid used for washing biodiesel, and amount of water used for washing biodiesel

Funnel #	1	2	3	4
Method	Tap water	2% citric acid	3% citric acid	4% citric acid
Water usage	300%	300%	300%	300%

Table 4: Alkaline pre-treatment illustrating the amount of cooking oil, the amount of NaOH, and the amount of methanol used

	Batch 1	Batch 2
Waste Cooking Oil (ml)	40	40
NaOH (g)	0.12	0.16
Methanol (ml)	10	10

Table 5: Lab scale biodiesel production featuring oil from five different sources

Case #	1	2	3	4	5
Feedstock oil	Restaurant Waste Cooking Oil (Market Pointe)	Restaurant Waste Cooking Oil (Center Court)	Restaurant Waste Cooking Oil (Zoo)	Virgin Canola Oil (Kroger)	Virgin Canola Oil (Kroger)
Alcohol	Methanol	Methanol	Methanol	Methanol	Ethanol
Catalyst	NaOH	NaOH	NaOH	NaOH	KOH
Initial FFA (wt)	0.88%	1.02%	0.48% (after pretreatment)	0.24%	0.24%
Yield	94.2	92.5%	85%	98%	73%

Table 6: GCMS analysis of five biodiesel samples

	WCO ME (Market Pointe)	WCO ME (Center Court)	WCO ME (Zoo)	Canola ME (Kroger)	Canola EE (Kroger)
C15:0	-	-	-	-	2.97%
C16:0	15.69%	19.24%	14.76%	10.24%	5.60%
C17:0	-	-	-	-	0.25%
C17:1	-	-	-	-	35.47%
C17:2	-	-	-	-	7.09%
C18:0	5.14%	6.59%	8.29%	3.16%	1.76%
C18:1	22.06%	40.29%	29.42%	57.00%	31.55%
C18:2	57.11%	33.88%	47.22%	29.07%	16.44%
C20:0	-	-	0.31%	0.54%	0.51%
C20:1	-	-	0.25%	1.40%	1.33%

13. APPENDIX V: PHOTOS

Photo 1: Original free fatty acid titration right before indicator turns color

Photo 2: Original free fatty acid titration immediately after solution changed color

Photo 3: Acid pre-treatment titration to determine free fatty acid conversion

Photo 4: Small batch of biodiesel with two distinct layers of dark glycerol and light colored biodiesel

Picture 5: Clarity of water after washing crude biodiesel, the left beaker is the water containing 1% citric acid and the right beaker contains the tap water

Picture 6: Washing of Biodiesel with water and citric acid

Picture 7: Right container feature biodiesel make from waste cooking oil from restaurant of Cincinnati Zoo (after acid pre-treatment), left container features biodiesel made from waste cooking oil from Market Point Cafeteria a the University of Cincinnati

Picture 8: Pictured on right is biodiesel made from canola oil and ethanol, pictured left is biodiesel made from canola oil and methanol