

UBC Social, Ecological Economic Development Studies (SEEDS) Student Report

Biodiesel Quality Analysis

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INTRODUCTION

Biodiesel has been considered as a promising alternative to petroleum-based diesel because it is a renewable fuel and greatly reduces particulate emissions. There are many advantages of biodiesel over petroleum-based diesel; such as being biodegradable, having better lubrication properties, safer to transport because of its higher boiling point, and producing a zero net carbon dioxide and sulphur emission¹. Biodiesel is generally recognized as alkyl esters which are produced by reacting triglyceride in lipids, such as vegetable oil, with alcohol and presence of catalyst in a transesterification reaction, where glycerol is produced as a by-product². The biodiesel examined in this project was produced on U.B.C. campus by reacting wasted vegetable oil with methanol and basic catalyst, potassium hydroxide, in a batch transesterification process.

By producing biodiesel from waste vegetable oil, it reduces the harm to environment by significantly decreases on harmful emission and the amount of waste oil discarded to a landfill, furthermore a valuable fuel can be produced from a waste product. At this point, biodiesel production in Canada is in its infancy stage and the use of biodiesel is minimal in the market; nonetheless, it is shown to be a proven technology and marketable fuel in Hawaii and many cities in Europe. Biodiesel will gradually gain popularity as more communities learn about its many advantages and because of the reduction in emission that Canada has targeted in the Kyoto Accord.

To avoid injection clogging and other engine problems, it is important to ensure the high quality of biodiesel with the least contaminates as possible. Biodiesel can be contaminated with moisture, glycerol, mono-, di- and tri- glycerides from incomplete transesterification and insufficient purification³. To ensure the fuel quality, the properties of biodiesel are compared to standards, such as ASTM standard specification D6751. Some of these properties are moisture content, kinematics viscosity, free glycerin and total glycerin content, flash point, cloud point, and cetane number. In this project, some properties of biodiesel, such as glycerol, mono-, di- and tri- glycerides, moisture content, and viscosity, would be studied and correlations between these properties would try to be drawn.

OBJECTIVE

To ensure the quality and performance of biodiesel at production, it is important to measure its properties, including the impurities. The impurities include residual catalyst, un-reacted triglyceride, glycerol, and intermediate, mono- and di- glycerol, produced from the reaction. The properties to be studied in this project are viscosity, free glycerol, free mono-, di- and tri- glycerides, and moisture content. A statistical program, JMP IN, was used to detect if there is any indication of correlation which exists between each of the properties. By indicating any correlation between the properties, one can have a rough estimate of the quality of the sample with the measurements on one of its properties. The objective can be achieved by analyzing the properties of the biodiesel sample obtained from different sections of the production plant. To perform these analyses, a number of techniques were employed: rotary viscometer to determine the viscosity of a sample; a gas chromatograph to determine the composition of sample; and a moisture distillation apparatus to determine the moisture content within a sample.

EXPERIMENTAL APPARATUS

Biodiesel Production Plant

The production plant, Biodiesel Production Lab, located in the annex of the Lower Mall Research Station at U.B.C., is capable of producing 60L biodiesel per batch. It consists of an 80L batch reactor, a condenser, and two 200L barrels (a loading barrel and a washing barrel). The schematic of the plant is shown below.

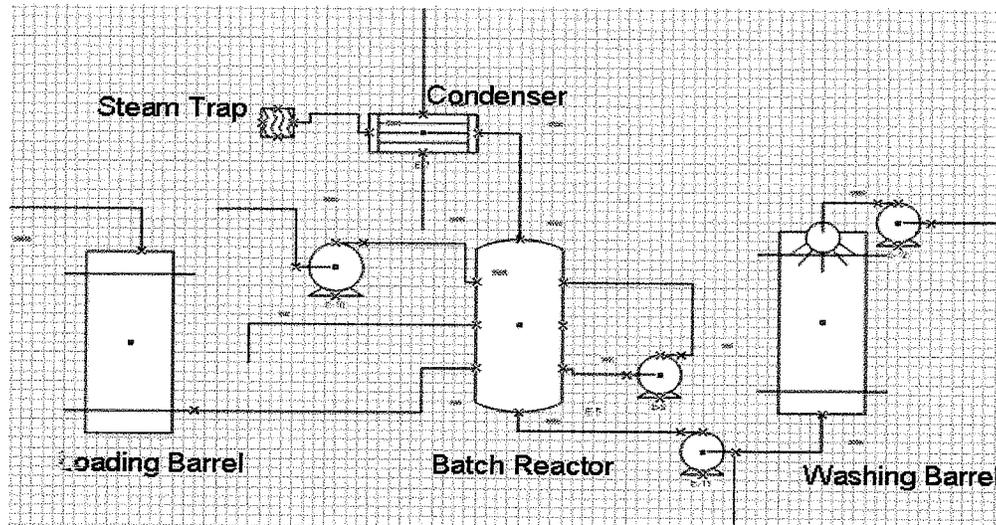


Figure 1: Simplified PFD of Biodiesel Production Plant

Two samples of biodiesel were taken from each batch for quality analysis. A first sample was taken from the reactor after the glycerol was separated from the biodiesel that had reacted for the first time (see Appendix I for detail procedure). A second sample was taken after the glycerol was separate from the biodiesel that had reacted for the second time. Due to problems that were encountered in the Biodiesel Production Lab, only four samples of biodiesel were able to obtain during the period of this project; samples after first reaction and second reaction from February, and the samples after first reaction and second reaction from March. A sample of incompletely reacted biodiesel was obtained. The incompletely reacted batch was caused by high moisture content in methanol that was added to the reaction.

Rotary Viscometer

The viscosity of biodiesel was measured by a Haake Rotovisco rotary viscometer. It is a speed-controlled rotational viscometer driven by a step motor and it consists of a temperature control bath, a computer with software VT500 for data logging and a 60mm x 20.04mm I.D. NV sensor. The NV1 sensor is chosen because it is capable of operating in range from 0.0015Pa.s to 2Pa.s.

Each of the four biodiesel samples was tested for the viscosity with the rotary viscometer at the temperature of 25°C, 30°C and 40°C. The testing at each temperature

setting was carried out three times to verify the reproducibility of the test. By plotting the shear stress of the samples against the shear rate, the viscosity of the samples can be obtained by calculating the slop of the plot.

Moisture Distillation Apparatus

The moisture distillation apparatus consists of a water-cooled condenser, a Bidwell-Sterling 10mL receiver, 1000mL short neck round bottom flask, and a heat plate with stirrer. To reduce formation and condensate on the glass due to heat loss to surrounding, aluminium foil is used to wrap around the flask and the neck of the receiver. The set up of this apparatus is show below.

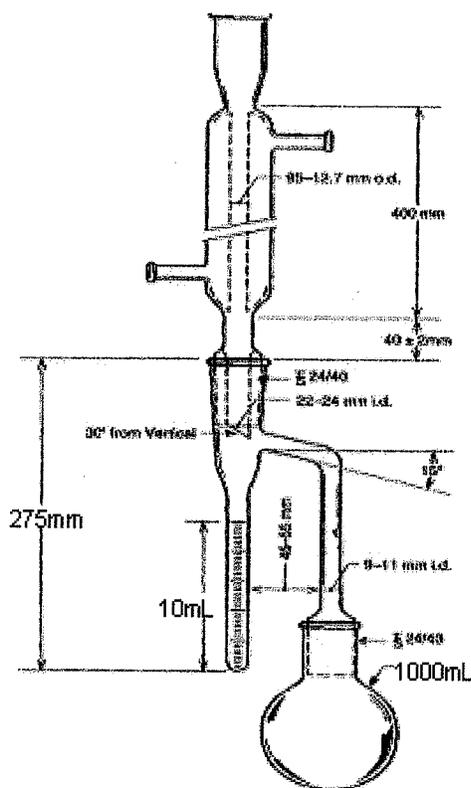


Figure 2: Moisture Distillation Apparatus from “Moisture Distillation Method.” Sampling and Analysis of Commercial Fats and Oils. Reapproved 1997.

For the experiment with the moisture distillation apparatus, 300mL of biodiesel sample was mixed with equal amount of toluene in the flask, the 10mL trap was filled with 5mL of distilled water and rest of the trap was filled with toluene. Due to problems encountered in the lab, the moisture distillation experiments were performed once each on the biodiesel samples obtained in March, where the samples from February were sent to Finnings Oil Lab for testing.

Gas Chromatograph

The GC analysis was performed with a Hewlett-Packard HP 5890 gas chromatograph with a flame ionization detector. There are two columns connected in series that are fitted inside; a 15m x 0.53mm I.D. uncoated, deactivated fused silica guard column connected in series with a 5m x 0.32mm I.D. fused silica capillary column coated with a 0.1 μ m film of DB-5. The two columns are fused together by Agilent with the method of Leak Free Union.

Since biodiesel contains varieties of fatty acids, which some are not favourable for GC analysis, the sample has to be derivatized (see Appendix III for detail) before injecting into the column for analysis. From performing experiments with the standard solution on the GC, multi-peaks results were able to produce from the GC graph, but due to the time limit of this project, more time would be required to identify each of the peaks. The samples were sent to the analytical lab of the U.B.C. School of Occupational & Environmental Hygiene department for testing.

RESULTS AND DISCUSSION

Viscosity

The viscosity testing results for each of the samples is shown on Table 1.

Temperature (°C)	Sample Average Viscosity (mPa.s)							
	1st rxn Feb	St. Dev.	2nd rxn Feb	St. Dev.	1st rxn Mar	St. Dev.	2nd rxn Mar	St. Dev.
25	9.133	0.058	8.167	0.231	7.200	0.173	7.000	0.000
30	8.067	0.058	7.333	0.058	6.567	0.058	6.200	0.000
40	6.500	0.000	6.100	0.000	5.500	0.100	5.000	0.000

Table 1: Biodiesel Viscosity

In Figure 3, the results from Table 1 were plotted against a B100 sample, which is a commercial available pure biodiesel, and a biodiesel sample from the Biodiesel Production Lab which had incompletely reacted. It is shown that the sample which was incompletely reacted has a much higher viscosity than the ones that are properly reacted.

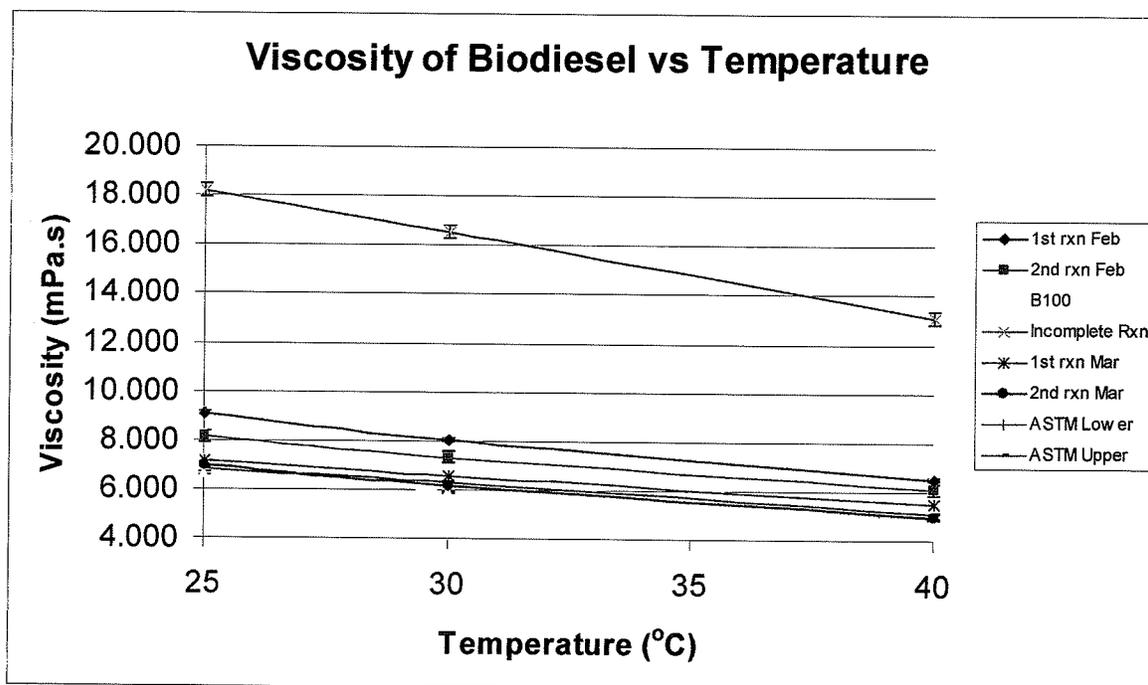


Figure 3: Comparison between Biodiesel Samples

Figure 4 shows the zoomed in version of Figure 3 in the viscosity range between 1mPa.s to 10mPa.s. The viscosity of biodiesel samples from the 2nd reaction decreases for both batches. This is most likely because the amount of residual triglycerides, which has higher viscosity than methyl esters, has been decreased as they react further for the second time to form methyl ester. When compared to the ASTM standard, only B100 sample and the sample from the March 2nd reaction batch are within the standard range.

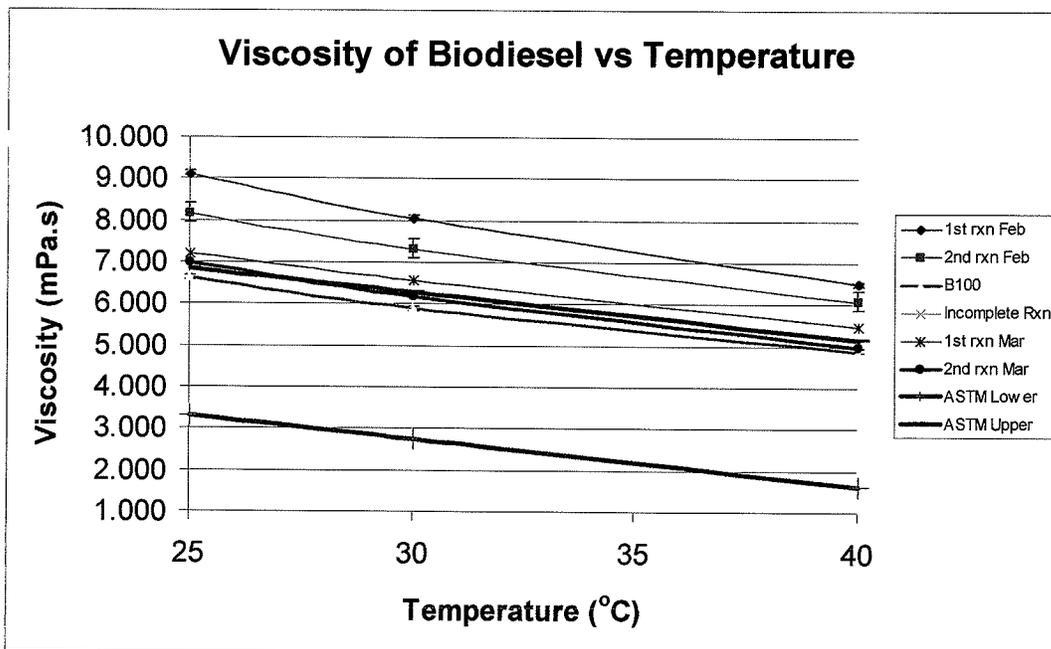


Figure 4: Comparison between Biodiesel Samples (Zoomed-in between 1 to 10mPa.s)

Moisture Content

On Figure 5, it is shown that for both batches the sample after second reaction has lower moisture content than the sample after the first reaction. It is also shown that if a batch has incomplete reaction, it has much higher moisture content. The high moisture content might have affect on the completeness of the transesterification reaction; since there was excessive moisture in the batch, it might have hindered the reaction by consuming the basic catalyst and the methyl esters, which leads to low conversion of the triglycerides. When compared to the ASTM standard, the moisture contents of the biodiesel samples were higher by 5-8 vol%.

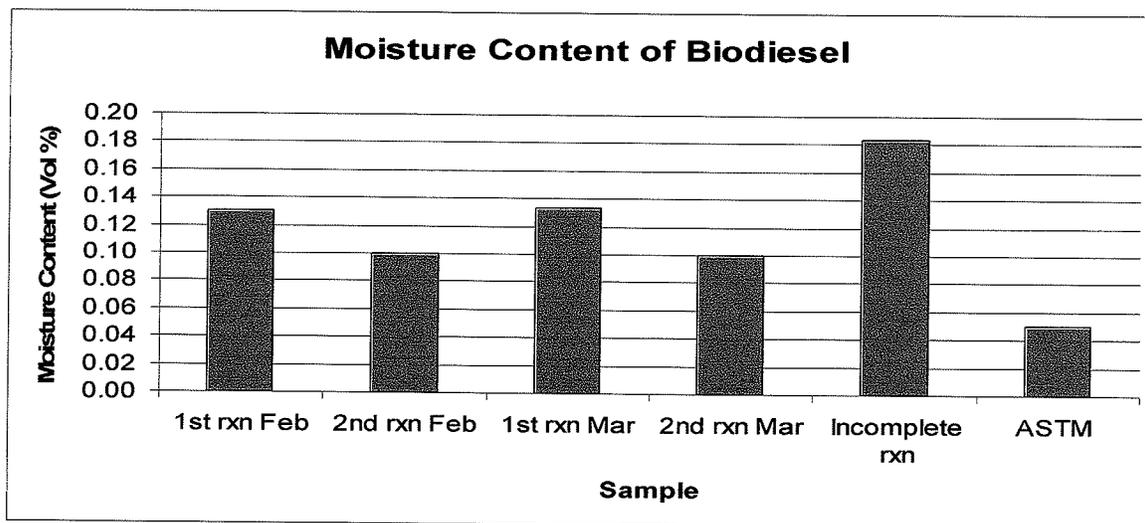


Figure 5: Moisture Content of Biodiesel

Impurities

The initial set-up and procedure of the GC analysis was from a technical paper titled "Simultaneous determination of glycerol, and mono-, di- and triglycerides in vegetable oil methyl esters by capillary gas chromatography". Glycerol, monoolein, diolein, and triolein, each in known amount, were used to prepare standard solutions in pyridine for calibration in GC which represented glycerol, monoglycerides, diglycerides, and triglycerides respectively when analyzing biodiesel samples. Butanetriol and tricaprins standard solutions were also prepared and used as internal standards in calibration testing. Each standard solution was derivatized with 100 μ L of MSTFA at 60°C for 15min and 1 μ L of the solution was injected into the capillary column for quality analysis. Repeatable multi-peaks results were able to obtain with the standard solutions. The peaks obtained were connected together and appeared within a short retention time. Mr. Ben Clifford from the undergraduate laboratory of U.B.C. Chemistry Department had suggested that the peaks might separate better by adjusting the oven temperature and the temperature increment of the temperature program. Due to the time limit of this project, it would require further work in identifying each of the peaks.

The four samples of biodiesel were sent to the analytical lab of the U.B.C. School of Occupational & Environmental Hygiene department for testing. The testing was done by using a GC with ion trap with the following temperature program; Initial oven temperature was at 95°C with 2 minutes isothermal period, then it was raised at the rate of 10°C/min to 250°C, then the temperature was raised again at 5°C/min to 310°C with an isothermal period of 5 minutes. The samples were derivatized with MSTFA at 60°C for 15 minutes. The testing was only able to detect glycerol and monoglycerides contents in the sample, which is shown on Table 2. From the result, it is shown for both batches that the glycerol concentration decreases after the second reaction. For the monoglycerides concentration, the batch of biodiesel from February shows increases in the concentration, where the batch from March shows decreases in the concentration. Since there were few samples for testing, further study would be required to detect if there is any trend in the pattern of changing concentration of glycerol and monoglycerides.

Sample	Glycerol Conc (mg/mL)	Monoglycerides Conc (mg/mL)
1st rxn Feb	10.820	2.250
2nd rxn Feb	6.204	8.140
1st rxn Mar	6.530	7.190
2nd rxn Mar	5.900	5.180

Table 2: Concentration of Glycerol and Monoglycerides in Biodiesel Samples

Data Analysis

With the results obtained, they were analyzed by JMP IN with multivariate method to detect if any of the tested properties might have stronger relationships with one and another. The result from JMP IN is shown Table 3. As the value is closer to 1, it means that the pair of parameter has stronger influence on each other. With the limited amount of data in this project, JMP IN result has shown that viscosity and the glycerol

concentration in the sample both has influence on each other, but this influence reduces as the temperature of the sample increases. To ensure this relationship between the viscosity and the glycerol concentration in biodiesel is valid, further testing and more data would be required.

	Viscosity 25C (mPa.s)	Viscosity 30C (mPa.s)	Viscosity 40C (mPa.s)	Moisture Cont. (vol%)	Glycerol Conc (g/L)	Monoolein Conc (g/L)
Viscosity 25C (mPa.s)	1	*****	*****	0.343	0.8574	-0.5826
Viscosity 30C (mPa.s)	*****	1	*****	0.3826	0.8366	-0.5191
Viscosity 40C (mPa.s)	*****	*****	1	0.3935	0.7605	-0.3798
Moisture Cont. (vol%)	0.343	0.3826	0.3935	1	0.6531	-0.4661
Glycerol Conc (g/L)	0.8574	0.8366	0.7605	0.6531	1	-0.8704
Monoolein Conc (g/L)	-0.5826	-0.5191	-0.3798	-0.4661	-0.8704	1

Table 3: Multivariate Analysis with JMP IN

CONCLUSION

When comparing the biodiesel samples, the sample that was incompletely reacted has a much higher viscosity and moisture content than the others. Since viscosity is much easier to measure than the other properties, one can have a rough estimate of how well the biodiesel has been reacted by taking measurement of the viscosity of the biodiesel. The results of this project have shown that the quality of biodiesel produced at the U.B.C. Biodiesel Production Lab would require improvements in order to meet the ASTM standards. These improvements include lowering in viscosity level, minimizing the moisture content, and decreasing the concentration of contaminants.

It is shown through analysis with JMP IN that, out of the properties that were tested in this project, viscosity and glycerol concentration in biodiesel have strong influence over each other. But none the less, further testing with more biodiesel samples would be prefer to assure this result.

APPENDIX I: Detail Procedure

Biodiesel Production⁴

1. Pour in the collected WVO wasted vegetable oil through wire meshed filter to catch any large particle before pouring into the loading barrel to about 80% capacity
2. Heat up the oil by a coiled steam heater from inside and a belt drum-heater on the outside. They are to melt any solidified grease and improve the pumping by lower the viscosity of the WVO.
3. Transfer the oil from the loading barrel to the reactor, by creating a vacuum in reactor. When all the WVO is transferred to reactor, it would remain in vacuum and heat would be added.
4. Remove the moisture in the oil by allowing the vapour gas pass through the condenser. Since the reactor is under vacuum at approximate -24 psi, the reactor should be heated to 60°C, which is the boiling point for water at such condition.
5. Mix 450g of KOH and 12L of MeOH in a 20L bottle to produce potassium methoxide (KOCH₃). This solution is introduced into the reactor by suction of the vacuum.
6. Start the mixing pump to initiate mixing and reaction of the solution within the reactor. After the reaction is completed, methyl ester (biodiesel) and glycerol would be produced.
7. Let glycerol settle from methyl ester to the bottom of the reactor. The settling time would take in between 1hr – 8hr.
8. Pump the glycerol from the bottom of the reactor into a storage container.
9. Pump rest of the biodiesel into the washing barrel.
10. Wash the biodiesel with 0.01M of sulfuric acid to remove excess catalyst. This process is continued until the pH of the biodiesel is 7. Then allow water to settle and drain.
11. Collect 1L sample (A) of biodiesel for quality analysis.
12. Pump the biodiesel back into the reactor and repeat step 4 – 10
13. Collect 1L sample (B) of biodiesel for quality analysis.

Rotary Viscometer⁵

1. Connect the rotor of the NV1 sensor to computer and turn it on.
2. Type in c:\vt500 and press enter. Then type in vt500 to start the program.
3. Select PROGRAM from the main menu and define substance, the sensor, operating temperature, number of data points, and the shear rate of sensor.
4. Determine the shear rate setting from the following figure where the box corresponds to the acceptable range of shear rate for different viscosity. Since biodiesel has viscosity of approximately 0.005Pa.s, the shear rate should be set at 2000s⁻¹.
5. Turn on the temperature bath of the viscometer and set it to the desired temperature by tuning the digital dial. Turn on the coolant by the green switch at the back of the bath when operating at temperature below 20°C.

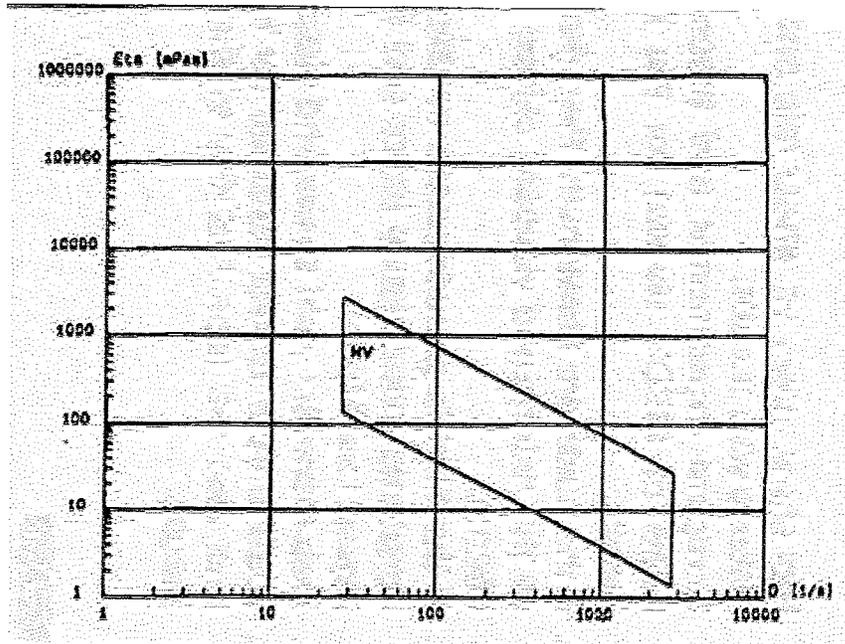


Figure 6: Operating Range for NV Sensor. (Note: the y-axis represents the viscosity in mPa.s and the x-axis represents the rotational speed of the NV sensor in 1/s) From “Experiment A: Rotary Viscometer.” CHBE 362/363 Lab Manual 2003, p. A10.

6. Wait for the temperature to be steady then press space bar to begin the analysis.
7. Press “C” once when the analysis is completed to print out the flow curve.
8. Save the data under another file to a floppy disk by selecting “Save” on the menu.

Moisture Distillation Apparatus⁶

1. Clean apparatus with potassium dichromate-sulfuric acid solution to avoid adherence of water droplet to the wall of apparatus. Let apparatus dry completely before use.
2. Place 300mL of biodiesel and equal amount of toluene in the 1000mL flask.
3. Connect flask to the bottom arm of the moisture trap.
4. Fill the moisture trap with 5mL of distilled water and the remaining volume with toluene until it overflows to the flask.
5. Connect the condenser with tap and run through with cold water.
6. Wrap the flask and the neck of moisture trap with aluminum foil to minimize condensate formation on the wall due to heat loss to the surrounding.
7. Turn on the stirrer and heat the flask with an electric heater to 120°C. Continue heating until water level in the trap remains constant for 30min.
8. Discontinue heating and wash off the moisture droplets adhered to the wall by 5mL of toluene.
9. Place the receiver in 25°C water for 15min or until the toluene layer is clear.
10. Read the water level in the receiver.

Gas Chromatography

1. Install the capillary column with the proper size ferrules (0.8mm and 0.5mm) by following the instruction provided in the HP5890 user's manual.
2. Adjust the carrier gas flow rate, U.H.P. grade helium, such that its linear flow rate is within 30cm/s – 40cm/s. This can be calculated by
$$U = L/t$$
Where U = Linear Flow Rate
L = Length of column
t = Retention Time of Simple Hydrocarbon Structure, ie MeOH
3. Condition the column by baking it at its highest operation temperature 350°C for 15 minutes.
4. Turn off the carrier gas and adjust the oven to 80°C.
5. Use a bubble flow meter to measure and adjust the flow rate of hydrogen to approximate 0.9mL/s at the exhaust.
6. Turn off the hydrogen flow.
7. Use a bubble flow meter to measure and adjust the flow rate of air to approximate 1.15mL/s. The air to hydrogen flow ratio shall be kept at about 1:0.8 so that the flame in the flame ionized detector (FID) would remain sensitive enough for testing.
8. Turn on the carrier gas and air to their desire flow rates and wait for a few minutes so that the flow would be steady.
9. Press and hold on to the ignition button and gently blow on the exhaust to ignite the flame until a “pop” is produce from the exhaust.
10. Hold a piece of cold metal or glasses above the exhaust. Observe if steady amount of steam condensate is form on the surface, which indicate that the flame is successfully lit. If no steam condensate is observed, check the flow rate of each gas and repeat step 9.
11. Check the signal of FID by pressing “SIG 1” on the GC control panel. The signal would overshoot to high value then slowly decrease to a steady signal. It should take about 45 minutes to an hour until the signal is steady.
12. Check again if the flame is still burning. If the flame is extinguished, repeat step 9.
13. Check if the FID signal is not too high or low. The signal should be steady in the range generally between 40mA – 300mA. If the signal is too low the FID would be too sensitive, and if the signal is too high then the FID would not be sensitive enough. To raise or lower the signal level, the amount of hydrogen flow rate should be increased or decreased respectively. Once the flow rate of hydrogen is changed, it would require sometime for the signal to reach a new steady level.
14. Inject 1µL of methanol to test out the signal output from the integrator. Tune the attain value of the integrator by pressing “ATTN” and desired value. The attain value would reduce the high of the signal peak by 2^x, therefore higher the attain value, the higher signal can be printed out.
15. Set the injector temperature, detector temperature, and oven temperature program to desire value. The injector should be at 375°C; it should be 25°C to 50°C above the highest operating temperature. The detector should be at the highest operating temperature which is 350°C. The oven temperature program is as following; oven is initially at 80°C with isothermal period of 1 minute, with purge turn on after 1 minute, then the temperature is raised at 10°C/min to 220°C, then 15°C/min to 350°C

- with isothermal period of 5 minutes.
16. Prepare 5 stock solutions in different concentration with glycerol (0.5mg/mL), monoolein (4.0mg/mL), diolein (5.0mg/mL), triolein (5.0mg/mL), 1,2,4-butanetriol (3.0mg/mL), and tricaprin (8.0mg/mL) in pyridine. Glycerol, monoolein, diolein, and triolein represent the retention time which glycerol, monoglycerides, diglycerides, and triglycerides would appear respectively. Triolein and butanetriol are used as internal standard for calibrating in concentration.
 17. Prepare standard solutions, at different concentration of each component, with the stock solutions.
 18. Derivatize the standard solutions by injecting 100 μ L MSTFA and heat up the solutions to 60°C for 15 minutes. Then dilute the solutions with n-heptane to 9mL.
 19. Run the temperature program and inject 1 μ L of the derivatized solution to the injector for calibration curve. By running the standard solutions with different concentrations of each component, one can observe the changes in peaks' high and identify the component accordingly for each peak.
- (**Further work is required on the following procedures**)
20. Identify the retention time correspond to each component in the standard solutions.
 21. Prepare sample by mixing 110mg of biodiesel sample with 70 μ L butanetriol, 100 μ L tricaprin, 100 μ L MSTFA, and pyridine. Heat up the solution to 60°C for 15min and dilute with n-heptane to 9mL.
 22. Run the temperature program and inject 1 μ L of the derivatized solution to the injector.
 23. Identify the class of compounds by matching the retention time with the calibration curve, and concentration can be calculated by calculating the peak area of the compound with respect to the peak area of the internal standard.

APPENDIX II: Transesterification

The process of transesterification is employed to convert the triglyceride in vegetable oil into biodiesel, which is generally classified as fatty acid alkyl esters. This process can be applied to most types of oil including pure or waste vegetable oil. It is performed in the presence of alcohol, commonly methanol since it is least expensive alcohol, and with either acidic or basic catalyst.

The base-catalyzed process is usually chosen over the acid-catalyzed process because this process proceeds faster than the acidic process and the catalysts are less corrosive⁷. In the basic process, catalysts such as alkaline metal alkoxides or alkaline metal hydroxides can be used. During transesterification, the alkoxide, produced from the mixing of alcohol and basic catalyst, attacks the triglyceride molecule from vegetable oil. The triglyceride molecule is broken down to glycerine molecules and forms methyl ester with glycerol as byproduct; for each molecule of triglyceride reacted, there are three molecules of methyl ester and one glycerol formed.

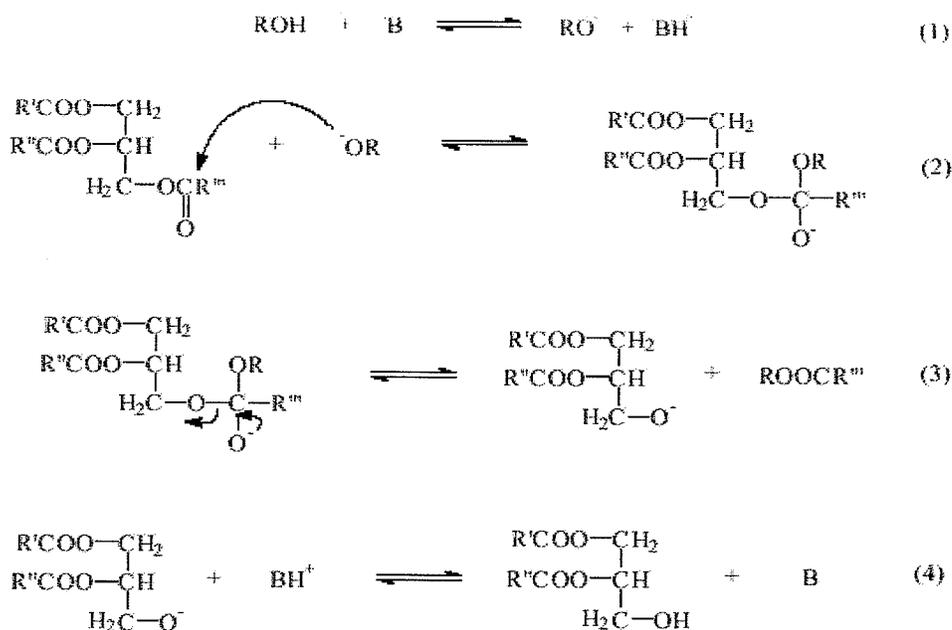


Figure 7: Mechanism of Transesterification, from "Transesterification of Vegetable Oils: a Review." J. Braz. Chem Soc., Vol. 9, No. 1, p. 201.

APPENDIX III: Derivatization⁸

Derivatization is a process which chemically modifies a compound in way such that it has properties that are suitable for GC analysis. This chemical process improves the detectability of the compound in GC by increases volatility and thermal stability. With derivatization, GC can detect compounds which are otherwise non-detectable with GC.

The type of derivatization applied for this project is silylation. It increases the volatility and thermal stability of the alkyl esters in biodiesel by replacing the active hydrogens with trimethylsilyl group (TMS). MSTFA (*N*-Methyltrimethylsilyltrifluoroacetamide) is used as the derivatizing reagent and pyridine is used as the solvent as it drives the silylation reaction forward. The silylation should be performed at temperature that does not excess 60°C for 15 minutes. This derivatizing reagent is very sensitive to moisture, where small trace of moisture would cause incomplete derivatization. When performing silylation with MSTFA, one should always make sure that the sample should be prepared as recent as possible, and any possible source of moisture (ie, trace of moisture in a syringe after washing) should be avoided. One way to reduce the chance of moisture trace in syringe or other glass equipment is to rinse it with acetone and dry it by blowing compress air to it.

APPENDIX IV: List of Contacts

Name	Department	Phone#	e-mail
Ms. Lufiani Madilao (Lina) Facility Manager, Analytical Core Laboratory	Wine Research Center/ Biotechnology Lab Rm 315, 2205 East Mall, UBC	(604)827- 5578	madilao@interchange.ubc.ca
Mr. Ben Clifford Undergraduate Lab Technician	Rm C326C Chemistry Building, UBC	(604)822- 6384	ben@chem.ubc.ca
Mr. Timothy Ma Research Technician	School of Occupational & Environmental Hygiene 3rd Floor, 2206 East Mall, UBC	(604)822- 9877	tima@interchange.ubc.ca
Mr. Holger Hermann GC Technician	N/A	(604)418- 6147	holger@hermann.ca

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