BIODIESEL PRODUCTION SYSTEM
AS A SUSTAINABLE SOURCE OF FUEL FOR FARM MACHINERY

Presented to
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Bioresource Engineering Capstone Design Project

BREE 495
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Executive Summary

The consumption of diesel at the Macdonald campus is mostly used by farm machinery, and biodiesel is an ideal candidate to replace it in a more sustainable farm operation. This design project looks into the system required to process a part of the stream of waste vegetable oil produced by the residences of the downtown campus. Throughout the project, the oil has been characterized to determine its quality, and different methods have been tested to select the ideal process for this type of oil. A system was then designed in order to provide the highest quality fuel, with a prototype to test and demonstrate the feasibility of the project. It was found that the oil is difficult to process, and would require further testing in order to improve the quality of the biodiesel up to standards.
Acknowledgements

We would like to express our special thanks to Dr. Grant Clark for his valuable guidance throughout both parts of this project, as well as Dr. Mark Lefsrud for his constructive advice and for allowing us to use his lab and resources. Thank you to Mr. Matt Bohan for sharing with us the information he has collected through his biodiesel projects, Mr. Angelo Tambasco for explanations about the past SVO project, and Chef Olivier de Volpi for giving us access to the oil from the cafeterias of the residences. Thanks to Mr. Marc Samoisette for showing us the farm tractors and thoroughly answering our survey, and Mr. Marc Legault for helping in the assembly of the prototype. Finally, we want to thank the Bioresource Engineering design project classmates for giving us useful feedback in our presentations and poster.

Introduction

McGill background

A couple of projects have been initiated at McGill to make biodiesel from oil and use it on campus. One was done by the downtown facilities, where unprocessed Straight Vegetable Oil was mixed with diesel and put straight in the tank of a vehicle. The high viscosity of the SVO caused the particle filter to clog, which resulted in an overall loss of power in the engine. Eventually, the car broke down on the highway and the project was terminated for safety issues. The second project was led by a group of students who received a Sustainability Project Fund grant within the McGill University (contact: Mr. Matt Bohan). It was aimed to find the most efficient chemical reaction to produce biodiesel from waste cooking oil, and was conducted in a lab for about one academic year. The issue originated from the fact that the free fatty acid content of waste oil is too high which causes the formation of soap in the end product. This project was also terminated and a sustainable process to remove the free fatty acid content from the waste oil has yet to be found.

Feedstock

The McGill food and dining services produce a lot of fried foods for the students on a yearly basis, and the three main residences were visited to find their production rate of waste cooking oil. After assessment, 600 liters each month would be readily available as feedstock (Table 1). However, only 160 L/month would be needed to suffice the needs of the Macdonald campus.

<table>
<thead>
<tr>
<th>Table 1: Rate of production of waste vegetable oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>McGill Residences</td>
</tr>
<tr>
<td>Bishop Mountain Hall</td>
</tr>
<tr>
<td>Royal Victoria College</td>
</tr>
<tr>
<td>New Residence Hall</td>
</tr>
</tbody>
</table>
Project objective

The main goal of this project is to design a complete biodiesel production system within McGill Macdonald campus boundaries that would rely on waste vegetable oil. This idea was retrieved from previous analyses regarding potentials alternatives fuels which would drive Macdonald campus utilities and farm tractors. To fulfill this task our team will use scientific knowledge associated with oil conversion to biodiesel as well as empirical and mathematical analyses to design a processing system. This is done in the optic of re-using waste oil, in other words energy, to transform it into a more useful energy that will enter in a sustainable concept.

Figure 1. Waste vegetable oil collected at the Royal Victoria College
Analysis & Specification

Waste Vegetable Oil Characterization

Many different researchers have shown that the characterization of oil is critical in choosing the most appropriate process for producing biodiesel. To have a decent idea of the oil's condition, it helps to know what it has been used for, and more importantly in the case of frying, how long it has been subjected to elevated temperatures. Virgin vegetable oil is composed of triglycerides, which means 3 long chain fatty acids attached to a glycerin backbone (Talebian et al., 2012). During frying, some of these long chain fatty acids are broken apart from the glycerin backbone and results in the oil consisting of free fatty acids (FFA) and resulting in monoglycerides, diglycerides and triglycerides (Mangesh et al., 2006). So it has to be considered that WVO will likely have a high FFA content which can potentially have a negative interaction in the chemical process toward biodiesel production. Hence it will have to be taken into account in the process and this interaction will be discussed later on in subsequent sections.

Oil Supply and Collection

In order to reduce the cost of the process, WVO from the downtown McGill residences was obtained free of charge. This oil has been subjected to elevated temperatures for 2 weeks before being disposed of and has also been left to settle to minimize the quantity of solids and water present. It was stored in big vessels held in the McGill residences dining services storage facilities. According to Olivier De Volpi, the manager in charge of food services, there is approximately 600L/month of WVO from all the McGill downtown residences available.

Chemical Processes to Produce Biodiesel

To design a fully functional and reliable biodiesel production system it is necessary to test the conversion process of vegetable oil to methyl ester in order to successfully obtain a viable product. There are many different methods to produce biodiesel (pyrolysis, microemulsification and transesterification) but for this design project, transesterification will be assessed because it is currently by far the most common method used in the industry and only transesterification result in the product of alkyl esters from oil or fat, commonly known as biodiesel. Furthermore, even within transesterification there are several ways to manage the process depending on the type of catalyst or alcohol. Hence what follows will briefly describe the two different transesterification chemical processes that will be applied to testing; alkali catalysed WVO process and alkali-acid catalysed two-step WVO process (Knothe, 2005).
The production of biodiesel from vegetable oil (VO) or WVO essentially involves trying to separate the triglycerides of the oil into long chain fatty acids and glycerin and connecting the long chain fatty acids to an alcohol molecule. This leaves the glycerin to be removed resulting in the production of 3 moles of alkyl ester (methyl ester is the primary by-product) for every mol of glycerol. Figure 2 shows the involved steps of the reaction (Mangesh et al., 2006).

The most popular process for producing biodiesel is the base catalysed transesterification which uses an alkaline catalyst (KOH OR NaOH) and an alcohol (methanol or ethanol) to separate the fatty acid chains from their glycerin backbone to connect them to the methanol molecules. An acid catalyst can also be used but according to literature alkali catalysts make the process quicker than the acid catalysed process and have a higher methyl ester yield. In terms of alcohol type, it was shown to not have a specific impact but really depends on the type of oil or fat that is chosen. Moreover, since methanol is usually cheaper than ethanol, methanol is more frequently used as an alcohol. Since there several different transesterification processes exist to produce biodiesel from WVO, further experiments will be constrained by two main methods consisting of a base-catalysed and acid-catalysed transesterification, as well as a two step acid-base transesterification process (Knothe, 2005).

**Alkali (Base) Catalysed Transesterification**

The alkali catalysed transesterification of WVO commonly involves the use of methanol as an alcohol and KOH as a catalyst. Generally, researchers recommend a methanol to oil molar ratio of 6:1 which translates to roughly 20% methanol to oil depending on the oil feedstock and 1% w/w KOH at reaction temperatures of 65°C and reaction time of about 0.5 – 1 hour for optimum yields (Mangesh et al., 2006). If the concentration of FFAs is higher than 1%, which is usually the case for WVO, then the methyl ester yield can be reduced since FFA’s react with the catalyst which produces soap and emulsification. Hence, the amount of catalyst must be accounted for accordingly by adding more catalyst to the chemical process. Therefore, the most typical pre-treatment step is a titration to determine the extra amount of KOH needed to neutralize the FFAs. Thus Alkali Catalysed transesterification is the method of choice for WVO with low levels of FFA (Mangesh et al., 2006).

**Acid Catalysed Transesterification**

This process is similar to the alkali catalyst treatment except it uses sulfuric acid (H₂SO₄) as the catalyst instead of KOH because the acid catalysts are less sensitive to higher concentrations of FFA’s and is usually a preferred treatment for FFA concentrations above 2% (Mangesh et al., 2006). The reaction is much longer however and the methanol required is also substantially.
greater. Optimum results are achieved with a molar ratio of 20:1 and 1% \( \text{H}_2\text{SO}_4 \), temperature of 65°C and reaction time from 48 to 96 hours. This method is recommended for FFA content above 2%. (Mangesh et al., 2006)

**Acid and alkali catalyzed two-step transesterification**

This process combines the two previous treatments in an attempt to avoid disadvantages the alkali treatment of saponification and long reaction time for acid catalysed method. The first step involves the acid catalysed treatment that esterifies the FFAs, reducing their level below 1% (Koh, 2011). Different procedures are followed by researchers, but generally it is recommended to use a molar ratio of methanol to oil of 10-20:1, 1% \( \text{H}_2\text{SO}_4 \), temperature of 55-65°C and reaction time of about an hour. The second step is to carry out the alkali-catalysed transesterification method as described above (Mangesh et al, 2006).

**Biodiesel Production Steps**

The following section will discuss all steps required for biodiesel production. This will help to subsequently implement our design criteria for designing a small-scale biodiesel production unit and identify constraints specific to the system.

So for the sake of this project, and to simplify experimentation biodiesel production methods, alkali catalysed transesterification processes will be attempted. This was decided due to the fact that acid catalysed method needs a lot of methanol which would be a limiting factor in terms of feasibility in comparison to the base catalysed method. Also, the base catalysed process, as stated, is faster regarding processing time and generally has better yields. Additionally, it will be assumed, at first, that FFA content doesn’t hinder the process.

**Step by step manipulation**

At first when the oil is gathered, it needs to be filtered to remove possible suspended solids from prior foods frying deposits. Even if the majority of impurities might have settle from previous storage, it is important to make sure to get rid of the major contents. In addition, since water particles might also be present, water evaporation through heating can be performed as well as settlement of the solution if two different phases showed. Keep in mind that water increases the hydrolysis of triglycerides (Mangesh et al, 2006).

Secondly, the oil is now ready to be titrated in order to determine the amount of FFA present which will indicate the amount of lye needed in the transesterification reaction. FFAs react with the catalyst; hence extra amounts need to be accounted for to enable triglycerides reaction with methanol and the catalysis. Titration steps are shown in the Appendix 1. Usually it is relevant to make three titrations and averaging their results for precision.

Hence from the lab test experiment performed on the WVO sample from McGill residences, the following FFA content and extra lye addition are shown in the table below.
**Table 2: Titration of WVO**

<table>
<thead>
<tr>
<th><a href="M">KOH</a></th>
<th>KOH added (ml)</th>
<th>KOH:FFA (mol)</th>
<th>% FFAs</th>
<th>Extra KOH addition (g per L of oil)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01488</td>
<td>5.346</td>
<td>$7.95 \times 10^{-5}$ mol</td>
<td>2.25</td>
<td>4.54</td>
</tr>
</tbody>
</table>

*Description of calculation steps are in the Appendix 1.*

Thus from the nominal amount of lye (KOH in this experiment) addition of 4.54 g/l is needed while the percentage of FFAs in the oil range around 2.25%.

Now that relevant parameters are presents, the transesterification reaction can begin. According to literature a 6:1 methanol to oil molar ratio will be used (Knothe, 2005). There are numerous other numbers of molar ratios, but this one will be attempted since it is most commonly found. Further analysis with other molar ratio could be tried in the future to correlate the best suited amount according to results. In terms of KOH a 1% catalyst weight to oil weight will be added in the solution (Knothe, 2005). The oil must be preheated to 60°C and the reaction performed during 1h (Knothe, 2005). Settling will take place during 24h (Knothe, 2005). Notice that it is important to adequately agitate the solution so that the reaction efficiently takes place. Also from the current amount of lye added, the extra amount of KOH calculated above need to be added. Once the transesterification is done and the glycerol is settled, separation of the two phases can be followed up by emptying the given vessel of its glycerol content.

The methyl ester solution would then be washed and dried to get rid of impurities; explanation of these steps will be discussed in the optimization section. The glycerol can be taken aside, there is no other usage in the function of biodiesel production (Knothe, 2005).

*The flowchart in Figure 3 describes general steps of the production of biodiesel. A point form transesterification reaction process is in the Appendix 1.*

![Flowchart](image-url)
System Concept

Many small-scale biodiesel systems already exist on the market. However, a small scale biodiesel system needs to be appropriate for the location. Some large scale system use technologies such as cavitation to enhance the mixing of the biodiesel (Piedmont Biofuels, 2013). However, this technology would be too expensive for the small batches of biodiesel produced at the Macdonald Campus. An example of a functioning small scale biodiesel system is the one built by Keith Addison (2013). A building manual is available for constructing the reactor where the system uses a drill to mix the biodiesel. The drill was installed in a way that it was sealed to the closed tank to prevent any splashing. This method would be very good for a very small batch but it would not give the required mixing for a Macdonald Campus small scale biodiesel production.

Other mixing systems such as mixing vanes using a small motor can be used in a biodiesel system (Gire et al., 2007). However, the simplest and most efficient design for mixing the biodiesel at Macdonald Campus would be a pump recirculation system. Another system designed by Edibon (2012) has a fueling unit which makes it very practical to directly fuel a vehicle. Ion exchange resin is used in the system to help purify the biodiesel. Also, the pressure is controlled and Venturi pump recirculation is used.

To keep the Macdonald Campus biodiesel production system simple and cheap, the pressure in the system would be atmospheric therefore there would be no variation of pressure in the system. Another small-scale system was designed by Gire et al. (2007). They use pump recirculation for mixing and centrifugation to separate the end product of making biodiesel. Different methods exist for the separation such as gravity, centrifugation and water washing column (Gire et al., 2007). The gravity method can take up to 24 hours, which is acceptable for occasional batch system, as it is the case on Macdonald Campus. Therefore considering the idea of gravity settling and a pump recirculation system, the throughput of the system would be as follows and is visually presented in the following Figure 4.
Throughput of the System

1. Pump pumps oil from oil storage tank to the reaction tank after being filtered.
2. Heater heats up oil in reaction tank.
3. Catalyst mixture of acid and methanol is pumped into the reaction tank from catalyst tank.
4. Pump circulates the mixture to speed up the esterification process through agitation.
5. After settling, catalyst mixture of KOH and methanol is pumped into reaction tank from catalyst tank.
6. Pump circulates the mixture to speed up the transesterification process through agitation.
7. Let the mixture settle.
8. Glycerine is drained from valve 7.
9. Biodiesel is ready for washing and post treatments.
10. When washed biodiesel is pumped into Biodiesel storage tank.

*Note that this throughput is for the process with the pre-treatment step. For the process with only transesterification, steps 3 and 4 can be removed. Valves need to be opened and closed between steps.*
Design Considerations

Safety Considerations

Chemicals and Mixtures
Many different safety concerns may arise when producing biodiesel. The chemicals needed for making biodiesel are dangerous including methanol, potassium or sodium hydroxide and sulfuric acid. It is recommended that material safety data sheets (MSDS) should be available at all times for all chemicals and mixtures involved in the process. A safety station should be available in which all MSDS can be readily available in the event of an accident (Steiman, et al., 2008) and chemicals should also be stored in a fireproof cabinet.

Whenever handling the chemicals and mixtures, safety goggles, lab coats, and gloves need to be worn at all times. All the human hazards related to the chemicals and mixtures are all presented in their respective MSDS. Running water, phone access and fire extinguishers should all be available in the facility (Steiman, et al., 2008).

Fumes and Fire Hazards
The flash point of methanol is only 11˚C and therefore it is constantly evaporating. Fumes from methanol are toxic and flammable. These considerations make it mandatory that the processor is designed as a closed system and the room and reactor need to both be properly vented to the outside (Steiman, et al., 2008). The systems proximity to ignition sources should be considered as well as there may be methanol fumes present (Steiman, et al., 2008). The tank should be ideally constructed out of metal, however many small scale systems use plastic tanks.

Equipment Considerations
At different stages of the process, the system will need to be able to support different compositions of fluid. After determining a transesterification process, with the potential optimization involving an acid pre-treatment step, the system should be designed to be able to resist:

- Temperatures up to 60˚C
- Alkali conditions
- Acidic conditions
- Fire
- Pressure build up
- Escape of fumes into facility

Location of Processing Facility
There are several potential locations for the biodiesel processing facility at Macdonald Campus. Possible locations include the Macdonald Campus Machine Shop, the Mac market and possibly the farm. At the moment there is a small room

Figure 5. Available area on campus
(Figure 5) available at the machine shop but proper ventilation would need to be installed. The dimensions of the room are 11.5 feet by 23 feet.

**Chosen components**

**Materials**

**Tanks**
In order to process a volume of 160 litres of WVO every week, fairly large tanks will need to be used. 160 litres converts to about 42.3 gallons and therefore tanks of 60 gallons will be used so that the tanks will not be completely full and the inlet at the top will not be submerged. Conical bottom tanks are important as there are several draining stages during the process of fluid separation and conical bottoms will facilitate the separation of glycerin from the biodiesel. Two options exist that are both corrosion resistant and can withstand the design temperatures. One is a stainless steel grade 316 tank (Mixer Direct, 2013) and the other is a medium density polyethylene (MDPE) tank which both have the desired conical bottom shape for drainage (The Tank Depot, 2013).

**Piping**
The piping will serve the purpose of transferring the different fluids and will also need to withstand the design conditions just as the tank. Several different options were considered for the pipes including stainless steel pipes, schedule 40 pipes, and flexible clear PVC tubing and all options respect the design conditions. The flexible clear PVC tubing was chosen as its transparency allows the operator to visually inspect the systems operation and transfers at all times and stages of the process (Harrison Hose & Tubing Inc, 2013). Another benefit is that it allows for flexibility in the systems design and possible orientations. In the event of expansion or change of location, the flexible tubing could easily be disconnected and reconnected as needed. The same type of tubing can be used for ventilation of the tanks and air entry to prevent pressure buildup and to vent the methanol fumes properly.

**Fittings**
The fittings include all ball valves, tees, nipples, couplings, hose connectors and reducing couplings. The network of pipes created from these fittings allow for the fluids to be transferred, drained and mixed using different paths as needed. Keeping the design considerations in mind, options available were to use stainless steel – grade 316 or schedule 40 PVC pipe fittings. The stainless steel fittings were chosen due to their strength and high resistance to corrosion and elevated temperatures (Stainless Steel Fittings, 2013). Their ability to resist fire is their advantage over schedule 40 PVC pipe fittings.

**Heating**
Heating the WVO prior and during the reaction speeds up the reaction significantly and as it has been seen; a temperature of 60°C is desired. Two main heating mechanisms are available including in tank heating and an external tank heater. The in-tank heating system is the most
commonly used for small scale systems with the use of an immersion heater. An inline oil heater could be used; however its operation requires constant pumping of oil through the heater for heating thus using more energy (Inline Heaters, 2013). A screw plug immersion heater allows for heating a variety of fluids and is inserted into the tank. It can operate without a pump and heats the fluids at any point in the reaction as needed. Placement in the tank also makes the system more compact and so this option was chosen. A thermostat will also be needed to regulate and control the temperature, which often comes with the heater itself as part of a kit (The Biodiesel Store, 2012). The heater sizing calculations are available in the Appendix 2.

**Pump**

A pump was needed for circulation of the fluids, performing both transferring and mixing duties. All pumps are designed for specific fluids and purposes and for this biodiesel processor, the pump will be required to transfer and mix viscous oil and mixtures, as well as respecting the design considerations. There are two main categories of pumps including centrifugal pumps and positive displacement pumps. Centrifugal pumps are commonly used in small scale biodiesel processors, however they are not designed or recommended to be used for high viscosity liquids whereas positive displacement pumps are designed for higher viscosities and are available in non corrosive materials and would therefore be the ideal pump (Pump Scout, 2012).

**Stirring**

As it was shown above, stirring or agitation during the reaction process is important in speeding up the reaction and proper separation. Mechanical stirring can be used such as a paint stirrer by using an electric motor but this option complicates the system as it requires an extra electrical component, increases the size of the system and also complicates the ventilation requirements making it hard to perfect a seal on the top of the reactor. As a pump already needs to be used for the system, it can also perform the mixing duties by circulating the mixture from the bottom of the reactor to the top of the reactor through the opening and closing of valves (Addison, 2013).

**Storage Tank and Filter**

At least two storage tanks will be needed at all times. One will be needed for containment of the WVO after arriving on campus. One will be needed for containment of the biodiesel after completing the process. Both tanks can be either plastic or steel 55 gallon drums as they are often readily available. Since the WVO has been settled for a while at the downtown residences, filtering requirements are minimal or not required (Addison, 2013). A filter of 75 microns is chosen as it will filter the WVO to a satisfactory state (Utah Biodiesel Supply, 2013). The outlet hole from which the WVO will be pumped into the reactor is 4 inches from the bottom to allow for materials that have settled to the bottom to stay there (Addison, 2013).
**Final Design and Economics**

There are two different designs that have been developed based on cost and differing in the materials of choice. Both designs can accommodate a simple transesterification process but are also able to produce biodiesel using the two step method with an acid pre-treatment if that method is needed. The first design (Figure 6) is considered as the ideal design and the second is considered as the likely design.

The first design consists of a stainless steel tank (Mixer Direct, 2010) as the reactor and will also include all stainless steel fittings and plumbing (Stainless Steel Fittings, 2013). A plastic MDPE tank will be used as the catalyst tank for containment of the alcohol and catalyst (The Tank Depot, 2013). This tank does not need to be fireproof since it will be empty most of the time as its main purpose is for mixing the alcohol and catalyst. The operator would be supervising the mixing process as well. Clear tubing will be used as the advantages of flexibility and transparency are desired (Harrison Hose & Tubing Inc, 2013). A positive displacement diaphragm pump (Rae Pro Stores, 2013) would be recommended as the fluid will be viscous at the start of the process and a steel 55 gallon storage drums will be used as well (Uline, 2013) with a 75 micron drum filter (Utah Biodiesel Supply, 2013). This is the optimal design as it respects all the design considerations and is also fire proof.
The cost of the final design needs to be considered as well, as the budget will be somewhat limited. This first ideal design is expensive as stainless steel is incredibly costly but is required for sufficient resistance to corrosion as tank and fittings will be continuously exposed to acidic and alkali conditions. A positive displacement pump that is designed for viscous fluids and corrosive fluids is also expensive. These are the main components having the most influence on the cost of the system as a whole. A summary of the parts is presented in Figure 7 with their respective prices.

<table>
<thead>
<tr>
<th>Components</th>
<th>Part</th>
<th>Qty.</th>
<th>Size</th>
<th>Material</th>
<th>Unit Cost</th>
<th>Total Cost ($)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plumbing</td>
<td>Hex Nipple</td>
<td>20</td>
<td>1&quot; NPT-male</td>
<td>Stainless Steel-316</td>
<td>7.62</td>
<td>152.40</td>
</tr>
<tr>
<td></td>
<td>Male Hose Connector</td>
<td>8</td>
<td>1&quot; NPT-male</td>
<td>Stainless Steel-316</td>
<td>12.65</td>
<td>101.20</td>
</tr>
<tr>
<td></td>
<td>Hex Coupling</td>
<td>3</td>
<td>1&quot; NTP - Female</td>
<td>Stainless Steel-316</td>
<td>41.97</td>
<td>125.91</td>
</tr>
<tr>
<td></td>
<td>Female Tee</td>
<td>5</td>
<td>1&quot; NTP-Female</td>
<td>Stainless Steel-316</td>
<td>40.78</td>
<td>203.90</td>
</tr>
<tr>
<td></td>
<td>Female Valves</td>
<td>12</td>
<td>1&quot;-female</td>
<td>Stainless Steel-316</td>
<td>18.09</td>
<td>217.08</td>
</tr>
<tr>
<td></td>
<td>Reducing Coupling</td>
<td>2</td>
<td>2&quot;-1&quot;Female</td>
<td>Stainless Steel-316</td>
<td>36.14</td>
<td>72.28</td>
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<tr>
<td></td>
<td>Tubing</td>
<td>10</td>
<td>1&quot;in-1 1/8&quot;out</td>
<td>PVC Tubing</td>
<td>0.80</td>
<td>8.02</td>
</tr>
<tr>
<td></td>
<td>Tube Clamp</td>
<td>8</td>
<td>-</td>
<td>Stainless Steel</td>
<td>1.05</td>
<td>8.40</td>
</tr>
</tbody>
</table>

| Pump       | Double Diaphrahm | 1    | 1" NPT in-out | Polypropelene     | 1,099.00  | 1,099.00       |

| Tanks      | Conical Bottom Reactor | 1    | 60 gal       | Stainless Steel-316 | 3,098.00  | 3,098.00       |
|           | Conical Tank + stand   | 1    | 60 gal       | MDPE                 | 211.65    | 211.65         |
|           | Storage tanks          | 2    | 55 gal       | Steel                | 112.00    | 224.00         |
|           | Filter                  | 1    | 75 micron    | Nylon Mesh Screen    | 17.00     | 17.00          |
| Heating    | Heater                 | 1    | 3000 Watts, 23W/sq.in | Brass Plug | 749.00    | 749.00         |
| TOTAL      |                        |      |             |                       |           | 6,287.84       |

*Note that prices are rough estimates and references are cited in text

The second and more likely design will be mainly composed of plastics parts. The MDPE tanks would be used in combination with the schedule 40 PVC fittings (1PVC, 2013). These components would also respect the design considerations with the exception of being fireproof. A clear water centrifugal pump could also be chosen as a compromise due to cost. Although this pump is not specifically designed for biodiesel processors and for handling the design conditions, it is commonly used by many home brewers and has been said to work fine (Utah Biodiesel Supply, 2013) and can be easily replaced at a low cost. The 55 gallon storage tank could be plastic as well reducing the cost slightly (Uline, 2013). All other parts would be identical and the system would work in the same manner as the first. A summary of the parts for this system is presented in Figure 8 with their respective prices.
Consideration of the economic factors would realistically have an influence on the final design choice. The stainless steel design is ideal as mentioned above, however its price would likely make the system unfeasible which is why the plastic version was developed like many home brewers processors. The prices listed are assuming the components were bought at their full regular prices and if some of the components could be collected from second hand sources, the design would likely be more feasible. The plastic design does however respect all design considerations with the exception of being fireproof, but under proper operations, there should be very minimal fire risks as the main risks stem from improper ventilation with an open system (Addison, 2013).

<table>
<thead>
<tr>
<th>Components</th>
<th>Part</th>
<th>Qty.</th>
<th>Size</th>
<th>Material</th>
<th>Unit Cost</th>
<th>Total Cost ($)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plumbing</td>
<td>Hex Nipple</td>
<td>20</td>
<td>1” NTP-male</td>
<td>Schedule 40 PVC</td>
<td>0.88</td>
<td>17.60</td>
</tr>
<tr>
<td></td>
<td>Male Hose Connector</td>
<td>8</td>
<td>1” NTP-male</td>
<td>Schedule 40 PVC</td>
<td>0.93</td>
<td>7.44</td>
</tr>
<tr>
<td></td>
<td>Hex Coupling</td>
<td>3</td>
<td>1” NTP - Female</td>
<td>Schedule 40 PVC</td>
<td>0.59</td>
<td>1.77</td>
</tr>
<tr>
<td></td>
<td>Female Tee</td>
<td>5</td>
<td>1” NTP-Female</td>
<td>Schedule 40 PVC</td>
<td>1.92</td>
<td>9.60</td>
</tr>
<tr>
<td></td>
<td>Female Valves</td>
<td>12</td>
<td>1”-female</td>
<td>Schedule 40 PVC</td>
<td>8.00</td>
<td>96.00</td>
</tr>
<tr>
<td></td>
<td>Reducing Coupling</td>
<td>2</td>
<td>2”-1”Female</td>
<td>Schedule 40 PVC</td>
<td>1.51</td>
<td>3.02</td>
</tr>
<tr>
<td></td>
<td>Tube Clamp</td>
<td>8</td>
<td></td>
<td>Stainless Steel</td>
<td>1.05</td>
<td>8.40</td>
</tr>
<tr>
<td></td>
<td>Tubing</td>
<td>10</td>
<td>1”in-1 1/8”out</td>
<td>PVC Tubing</td>
<td>0.80</td>
<td>8.02</td>
</tr>
<tr>
<td>Pump</td>
<td>Centrifugal Pump</td>
<td>1</td>
<td>1” NPT in-out</td>
<td>Cast Iron</td>
<td>79.99</td>
<td>79.99</td>
</tr>
<tr>
<td>Tanks</td>
<td>Conical Tank + stand</td>
<td>2</td>
<td>60 gal</td>
<td>MDPE</td>
<td>211.65</td>
<td>423.30</td>
</tr>
<tr>
<td></td>
<td>Storage tanks</td>
<td>2</td>
<td>55 gal</td>
<td>Plastic</td>
<td>89.00</td>
<td>178.00</td>
</tr>
<tr>
<td></td>
<td>Filter</td>
<td>1</td>
<td>75 micron</td>
<td>Nylon Mesh Screen</td>
<td>17.00</td>
<td>17.00</td>
</tr>
<tr>
<td>Heating</td>
<td>Heater</td>
<td>1</td>
<td>3000 Watts, 23W/sq.in</td>
<td>Brass Plug</td>
<td>749.00</td>
<td>749.00</td>
</tr>
<tr>
<td>TOTAL</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1599.14</td>
</tr>
</tbody>
</table>

Figure 8. Characteristics of different parts of the second design with their respective prices
Prototyping

This prototype was made according to the designed system, using scaled down components readily available. For example, the processor and storage pails were recycled from plastic 5 gallon buckets, made out of high-density polyethylene. They offer heat resistance up to 110°C (Five Gallon Ideas, 2013), which is beyond the maximal temperatures of 60°C required by the transesterification process.

The heating element is a water heater replacement element, meant to output 3000W at a power density of 80W/sq.in. This value is clearly too high for vegetable oil, which will inevitably lead to oil being burnt on the element. In order to fix this problem, applying a tension of 120V instead of the rated 240V will reduce the power and power density by four according to Joule’s and Ohm’s law (Figure 9). This results in an element that is much more appropriate for vegetable oil, with 750W of power and 20W/sq.in. A regular extension cord was screwed with two ring terminals on the element, in order to connect it with a wall socket.

The Little Giant Pump Company pump (Model 2-MD) is a standard centrifugal pump with excellent chemical resistance, which can handle fluids with a specific gravity up to 1.1, and with a temperature up to 65°C (Little Giant Pump Co., 2013). It can pump fluids at a rate of 1750L/hr, which means that it can theoretically turnover the contents of the container every 35 seconds. The Bioresource Engineering Shop had one available, and was deemed to suffice the need for the prototype. The turnover time would be reduced when using the oil due to its higher viscosity, but it would then decrease as the oil gets transformed into lower-viscosity biodiesel.

Clear braided PVC hose was chosen in order to have a clear sight of the activity that would happen in the piping. It is readily available from hardware stores and affordable, yet does resist to temperature of 80°C (New Age Industries, 2013), which should be sufficient for our non-food use. This pipe is also flexible and can easily be cut, which makes it even better suited for the prototype. In order to direct and stop the flow of fluid, PVC straight ball valves were used, and polyethylene fittings and tees joined everything together. There was even a special fitting cut for the bottom of the biodiesel processor, in order to allow a maximal amount of biodiesel to drain out of it when emptied.

Once all of the components were selected and found, the system was assembled together using hose clamps, which allowed for later modifications of the system, if required. Considering so many parts were recycled or found, the only parts purchased were the tubing, fittings, valves and heating element, cumulating to a total of $80. The only issue was to assemble them into a working system, and then getting the system to work. There was also
high-temperature silicon and screws used to secure the flange-mounted heating element at the bottom of the processor. The special fitting used to drain the processor was also glued using the silicon, as well as the electric terminals to prevent any risks of electric shocks.

The prototype was then tested with water, to make sure all of the fittings were watertight, and the pump was functioning correctly (Figure 10). Once done, then a test batch was done using the prototype and WVO. The prototype assembly and test with water were quite accurate, as very little adjustments were made afterwards. There was one issue that arose with the tube going from the pump to the reactor, where the tube was not attached at first, which has led to a spill of oil as the vibration from the pump pushed the tube out of the reactor. There also has been another spill event after adding an elastic band that was not sufficiently strong to hold the tube. The tube was finally held in place using duct tape, which solidly held it in place until the end of the experiment.

The experiment was done using a 1% H₂SO₄ acid pretreatment for 1 hour at 35°C (see p. 27 for more details), with a transesterification using a methanol ratio of 7:1 and KOH catalyst. Running the prototype was quite difficult at the beginning for someone who wasn’t used to doing the whole process, yet once done thoroughly it becomes much easier. There could be improvements made to the prototype to make the process even easier, as it is quite consuming when done at a small scale. First, there would need to be volume indicators in the processor, which would make the measurements of the quantity of oil easier, instead of using a 1-L graduated cylinder.

It also seemed that the element was running hot when turned on, at least on the part that was exposed to air. If left uncooled, the temperature would rise indefinitely, so it was corrected by shooting the flow of pumped oil directly on the exposed part. This could be a fire hazard, and
means that the tank angle was steep, reducing the play between the elevation of the tip of the element and the top of the bucket. The test run of 13L was not sufficient to cover the top of the element during the experiment, but it would have been better if started with 15L of oil. In the full-scale model, the element is placed horizontally at the lowest point possible, which is more flexible; yet still sets a minimum level of oil required to run. The element of the prototype was powerful enough to raise the temperature of the oil from room temperature to 60°C in roughly 30 minutes, which is twice as fast as the full scale model would run.

Once the running temperature was reached, the smell of fumes became quite powerful. Simply putting the lid back on the bucket was sufficient, which showed the importance of closed containers and well ventilated areas. Other improvements would be to have two switches to independently run the pump and element, making it simpler to operate. The tubing attachment would have been better if high temperature duct tape had been used, since the glue for standard duct tape seems to lose strength and grip when the processor is running at 60°C.

The quality of the biodiesel produced was not evaluated, as the point of this system was not to validate an experiment that already did not function on a small-scale, but rather make sure that the large-scale system would be functional and well adapted to the production of biodiesel.
Testing

Spreadsheet

In order to learn how to make biodiesel, a spreadsheet was made to easily calculate all the required chemicals for a batch of a certain size (Figure 12), by accounting for the purity of the chemicals, as well as the quality of the oil. All that needs to be done is to fill up the white cells with the required data, the quantity of oil to be processed and the results from the titration of the oil, and the final recipe will be displayed in the green cells. The spreadsheet file is also saved as a model, which forces the user to save data for every batch made, and can be used to archive the results. It also eliminates all possible human errors made in calculations, while making it much easier for anyone to process biodiesel with the written instructions.

Fuel quality standards

In order to assess a good fuel quality, rigorous testing needs to be done. If the biodiesel doesn’t meet standard there are almost no possibilities that it can be used in modern engine. Neither industries nor manufactures are willing to take the chance of using biodiesel that could reduce their motor capacity and lifespan. Hence, taking this into account, steps to overcome potential standard accomplishment is huge and is almost insurmountable within small-scale “home-made” design facilities. This doesn’t mean the biodiesel cannot be use nor is bad, it simply underlines the fact that criteria to meet are high and from a student project design perspective this achievement can be difficult.

Nevertheless the following section will overview the methanol test and wash test that were done to observe if the biodiesel from laboratory transesterification reaction was done with diligence and if it is suitable for potential future usage. Furthermore, a brief discussion will be intended to reveal other test factors recommended by the American Society of Testing Materials (ASTM) to ensure a product quality for biodiesel usage in engine.

Methanol Test

This quick test was done with the initial laboratory batch to see if the completeness of the reaction had occurred. For this test, biodiesel needed to dissolve in methanol. The proportion is not really important but it usually account for a 10:1 methanol to oil ratio by volume. To have an effective reaction the biodiesel should be soluble in the methanol and should form a
clear bright phase. If there are un-dissolved materials at the bottom of the sample or phase separation, it means that the reaction did not completely proceed. This method doesn’t cover all the aspect of quality but it gives a hint on the quality. After doing the test, it was found that the reaction didn’t occur completely since some droplet of glycerol and FFA was found at the bottom of the sample. (Burton R, 2008)

**Wash Test**
Wash test is another all-around test. After the transesterification reaction was done, dissolution of 150ml of biodiesel in 150ml of water was performed in a tightly seal jar. It was shaken for 10 sec and settled for an hour. To have a quality fuel, the biodiesel should separate with an amber color at the top and milky water at the bottom. A thin layer of white material composition should separate oil and water. On the other hand, if it turns somehow into what looks like mayonnaise and slowly or doesn’t separate with a creamy white layer in between the two phases, the fuel process needs improvement.

From the photographs it clearly shows the difference. The top picture represents the two phases after the wash test from WVO transesterification. It can be observed that there a thick creamy layer in between and the separation is not obvious which conclude that the reaction was not completed. As a comparison, the middle jar from the picture below stipulates that one of our tests with straight vegetable was conduct with better results. Separation is clearer. (Addison, 2013)

Mains causes of these results are usually due to excess of catalyst or a poor conversion of oil to biodiesel where tri-di and monoglycerides act as an emulsifier. Hence more accurate manipulations, a better titration, a longer processing time and a better temperature control are all solutions that might have an effect to positively enable a quality biodiesel. (Addison, 2013)

**ASTM Quality Standard Methods**
In order to efficiently recognize and acknowledge quality biodiesel, it is necessary to meet the ASTM D6751 standards. These standards are there to provide a uniform quality within the industry and to remove harmful effects if used in an engine. There are numerous criteria to assess from viscosity to cetane number (see Appendix 1). These criteria as a whole, if well managed, should give a decent quality that will meet the standards. Notice that there are other standards in Europe and Asia, but it shall be necessary to follow this one given the North American context of this application.
For the purpose of this paper, three major features will be addressed and recommended for further analytic manipulation; the glycerin content, the alcohol and catalyst residual and the water content. These methods were not conducted during the assessment of our biodiesel sample batch since they required resources, time and money which were not available for this project time line. Nevertheless these three analyses are recommended for future biodiesel usage within the campus.

**Glycerin Content**
Glycerin content is an important test of biodiesel quality. Even after the transesterification reaction is complete, there are still some tri-di and monoglycerol that were not converted and remained in the biodiesel. The same issue can occur with free glycerol after settlement that is still within the biodiesel solution. It remains in suspended droplets that are dissolved in the biodiesel. This total glycerin (sum of these two factors) content is generally due to the incompleteness of the reaction. To effectively measure its proportion a gas chromatograph (GC) or a high-performance liquid chromatography (HPLC) are usually the norm, but requires knowledge and money. Since the ASTM standard requires a maximum value of total glycerin < 0.24% in the final biodiesel product, it would be necessary to evaluate this value using Dr. Lefsrud’s HPLC. This could enable attestation and an iterative reaction response, if standards are not met, for a better transesterification reaction. Recall that a fuel with glycerol enhances mixture viscosity and clogs filters or injectors in the engine as well as permitting poor cold flow properties. (Davis, 2010)

**Alcohol and Catalyst Residuals**
Even if methanol, ethanol and the alkali catalysts are more soluble with glycerol due to its high polarity, it should not be accounted that it should all be removed with glycerol settlement. After separation, 2-4% of methanol can still be within the biodiesel. This causes the biodiesel to have a lower flashpoint of 40°C instead of 170°C which can present potential safety hazard issue. Since the ASTM required a flashpoint of 93°C, the residual methanol has to range around 0.1%. To disable this issue, the methanol as well as the alkali catalyst remaining should be removed by a water washing process (This will be explained later in the optimization section). An excess of catalyst creates ash deposit which can act as an abrasive in the engine. To test the flashpoint a Tag flash point testing device should be used in laboratory. The sample would be heated in a vessel and then ignite. The temperature would be recorded at this time. Unfortunately it is not know if this device is available on campus. (Knothe, 2005)

**Water Content**
Water can be present in two different forms in the end-product; dissolved water and suspended water droplet (biodiesel is insoluble in water). Standard from ASTM limit the amount of water to 500 ppm and if there is too much, water corrosion within engine equipment and microbial growth in the fuel can occur. To effectively determine the amount of water, biodiesel should be centrifuged. Usually 100ml suffice at 800rcf for 10 minutes in a calibrated tube. This could be performed in the Dr. Lefsrud laboratory where a centrifuge is
available. It is important to test this factor since biodiesel can absorbs 1500 ppm. With this same test, sediment quantity can also be assessed. (Burton R, 2008)

For complete analyses of all parameters necessary to fulfill a quality biodiesel, it shall be also recommended to assess a sample, after processing, from an external analytic laboratory within the region of Montreal. It might be costly but resources from the university, in term of personnel and equipment, might not be available to test all criteria necessary to ensure safe product for future biodiesel user. There was a viscosity test run on one of the biodiesel batches using resources available within the department, to see what range of values could be expected from such a test, with further details in the Appendix 3.

Risk analysis

Despite the spills that occurred with the prototype as well as other little adjustments, they would not be required on the large-scale model, as pipes are directly attached to the processors and other components. In the event that the large-scale model would be implemented, then further testing would be required to make sure everything is safe by studying the failure modes.

Failure modes

The heating element is the most dangerous part in this system, hence why there is the need for a fire extinguisher. Since the element is directly in the oil, the overheating could lead to a slight risk of fire as it was mentioned before, yet the thermostat regulating the heating element reduces the fire hazard as long as it functions properly. This could be made worse by a pump malfunction, which would then reduce the heat transfer from the element in the oil by limiting the amount of oil flowing by the element. This could then cause local overheating of the oil around the element, and causing some oil to cook around the element. Since the element is immersed in the oil and controlled by the thermostat, there would still be convection in the tank, yet a further heat transfer analysis would be required to see if there would actually be a serious fire hazard in the event of a failure. There would be no danger if the heating element was to fail, since the mixture would simply cool down and the processing reaction would slow down.

If the pipes fail, then the contents of the tank would drain all the way down in the barrel to the elevation of the break, and there would be a local spill of the WVO or biodiesel, depending at which point the reaction is. Since there are very little concentrated chemical substances running in the pipes, there is no serious hazard in the spilled material. There would be further precautions to be taken with the handling of a methoxide spill, which would most likely be done by human error.

Other sources of human error could be present in the mixing of the methoxide, where wrong proportions of reactant and catalyst could be mixed. Too much or little methanol leads to a
proportional change in processing efficiency, whereas incorrect amounts of lye would lead to an incomplete reaction or jellification of the biodiesel. These errors would either be picked up in the quality testing afterwards, or at least be inoffensive.
Biodiesel Neutralization and Washing

Hence, after the separation of the glycerol and methyl esters neutralization should be performed. An acid should be added in order to neutralize any residual catalyst and to separate soap that might form during the reaction. During this reaction soap will react with the acid and form salt and FFA. It follows the following reaction:

\[ R - COONa + HAc \rightarrow R - COOH + NaAc \]

Once done, the water washing step will remove salt. Hence the neutralization minimizes the potential for emulsion to form from the soap and biodiesel during the washing test. It is then no oblige but shall be considered if there is emulsion issue. Recall that the water washing is intended to remove the remaining catalyst, soap, salt methanol and free glycerol that might stay in the biodiesel. After this process the remaining water in the biodiesel can be removed by evaporation or vacuum flash process (Knothe, 2005).

The washing step required to have a 3:1 biodiesel to water volume ratio. More or less water could be used for each wash; decision according to the size of the washing has to be made. This step will be held in the same tank that the transesterification occurs. Thus right after the reaction was completed; water would be poor inside the tank. The pump could be activated to enhance water molecules contact with the biodiesel. This operation though should not be longer than 5mins, until the mixture looks homogenous. After let it settles for an hour or until the two phases (oil and water) reach equilibrium and remove the water phase. The washing step needs to be done at least 5 times to effectively remove all possible impurities. Water could be reused in between each wash but not after the first wash batch. At this early point the water in the first batch might be already saturated (Addison, 2013).

Methanol Recovery

Excess of methanol is usually present in the glycerol. In fact, glycerol leaving is usually around 50% glycerol content. Hence it is possible to recycle this methanol present in the glycerol and reused it in another transesterification. At first, to extract this methanol, acid addition to split the soap into FFA and salts need to be accounted to further separate the methanol in the glycerol by an evaporator or a vacuum flash process. The glycerol should have a purity of 85% and could be sold to a glycerol refiner. The methanol recovery might have some water present; hence a distillation should be performed before usage in another transesterification batch reaction (Knothe, 2005).
Acid Pretreatment

In order to improve the poor biodiesel quality which resulted from the methanol test provided in the testing section, an acid pre-treatment would need to be attempted. In fact, feedstock’s with high FFAs (>2%) have some difficulty to be converted by the alkali transesterification since the FFA reacts with the catalyst and form soap. If this soap is not released it will interfere in the washing process by forming emulsion and resulting in yield reductions. Although it is true that this step could have been avoided by integrating an acid rather than a base catalyst the long reaction time involved with the acid catalysed method favors the two step pre-treatment method. By doing a pre-treatment, the acid added to the oil converts the FFA’s into methyl esters therefore removing the potential soap formation problems. The resulting mixture can therefore be used in an alkali base transesterification (Godlisten, 2008).

The acid pre-treatment step is not complicated to perform. According to literature, sulfuric acid (H$_2$SO$_4$) is generally used at a 1% acid to oil weight ratio but this value may differ from experimentation protocol by different authors. This acid is poured into methanol and oil solution which has a 3:10 methanol to oil volume ratio. It needs to react for about an hour or two. The protocol performed in the laboratory experiment is shown in the appendix (Godlisten, 2008).

After the pre-treatment was done, a titration was performed to assess the potential benefit. The table 3 below shows what results were obtained.

<table>
<thead>
<tr>
<th>Table 3: Titrations of WVO</th>
</tr>
</thead>
<tbody>
<tr>
<td>[KOH] (M)</td>
</tr>
<tr>
<td>WVO</td>
</tr>
<tr>
<td>WVO after esterification (1% H$_2$SO$_4$)</td>
</tr>
</tbody>
</table>

From this table it can be seen that there is around 350% reduction in percentage of FFA content where it drops to a value beneath 1% of 0.628% of weight. Also the extra KOH needed to overcome FFA presence is diminished by almost 3g. Hence there is a net advantage in term reduction of KOH in the reaction and also the reduction in potential soap formation that could create emulsion during the wash test.
Expandability

The way this reactor works makes the expansion of the system quite easy. Since most of the elements are in line with each other, any component can easily be replicated and added to the system. They would mostly require additional valves, tees and fittings to connect. This means that the capacity of this system could be doubled or tripled, considering there is a lot of operation time dedicated to drying or settling. By adding tanks, raw oil could be stocked up for a little while before transforming it, pretreated oil could rest in a separate container while another batch is processed, and finished biodiesel could settle independently. The reactor downtime would thereby be reduced, and the utilization of the whole system would be maximized.

Sizing the heating element

Heat needed to raise the temperature of the oil:

\[ Q = m \times c_p \times \Delta T \]

- \( Q \) = heat required (J)
- \( c_p \) = specific heat capacity (J/g/°C)
- \( m \) = mass (g)
- \( \Delta T \) = temperature rise in °C

Amount of heat added by the element to the oil:

\[ Q = P \times t \]

- \( P \) = wattage of the element (W)
- \( t \) = time the element operates (s)

Since the heat needed to heat the oil is equal to the heat added by the element the formulas can be combined, eliminating \( Q \):

\[ P \times t = mc_p \Delta T \text{ or } t = mc_p \Delta T / P \]

Furthermore,

\[ m = \rho V \]

- \( \rho \) = density (kg/L)
- \( V \) = volume (L)

Giving:
\[ T = c_p \cdot \rho \cdot V \cdot \Delta T / P \]

Properties of vegetable oil (Make Biodiesel, 2013):

- \( c_p \) = between 1.7 and 2.2 J/g°C. Use the mean: 2 J/g°C.
- \( \rho \approx 920 g/L \)

Substituting into the formula:

\[ t = (2 J/g°C) \cdot (920 g/L) \cdot V \cdot \Delta T / P \]

\[ t = 1840 \cdot V \cdot \Delta T / P \]

Converting \( t \) from seconds to hours:

\[ t = 0.511 \cdot V \cdot \Delta T / P \cong 0.5 \cdot V \cdot \Delta T / P \]

**Heating element**

Screw plug immersion heaters are ideal for immersion into liquids and all types of oils, where space and connections are a problem. A 2-inch NPT Screw Plug Immersion heater (source below) will be screwed on the side of the first tank.

The installation of a thermostat to control the temperature of the heating system is recommended.

**Specifications** (AccuTherm, 2013)

- Brass plug, ideal for oils and alkali solutions
- 3 elements
- Watt density of each element = 23 W/in²
- Wattage = 3000 W
- Volts = 240V
- Length of element = 171/2 in
- Net weight = 9 lbs
Approximation of time required to heat the oil from 22°C to 35°C

\[ t = \frac{0.5 \times V \times \Delta T}{P} = \frac{0.5 \times 160L \times 13}{3000W} = 0.35\text{hr} \approx 21\text{min} \]

Approximation of time required to heat the oil from 22°C to 60°C

\[ t = \frac{0.5 \times V \times \Delta T}{P} = \frac{0.5 \times 160L \times 38}{1000W} \approx 1\text{hr} \]

**Sizing the pump**

**Assumptions**

A system of valves and a pump to circulate the oil, methanol and catalyst mix and the biodiesel are in the design of a small-scale biodiesel production on Macdonald campus. Depending on the configuration of the valves in the system, the liquids can circulate from a tank to another, and the biodiesel can be mixed by recirculation. The oil and the methanol-catalyst blend need to be mixed thoroughly in order to have a good quality biodiesel. To have a proper functioning system, a good flow rate for the mixing and sizing of the pump need to be determined. The Home-Made Biodiesel website gives some rules of thumbs concerning small scale biodiesel production. According to them, the optimal recirculation time of the biodiesel in the mixing tank is every five minutes. The volume of the mixing container is 40 gallons, which is about 151.41 Liters. It would be rare that we process 40 gallons of biodiesel per batch, but we size the pump accordingly in order to have a functioning system with a full tank. Therefore, the ideal flow rate in the system with a complete recirculation every five minutes would be around 0.0005 m³/s. The piping used in the system has an internal diameter of one inch (0.0254 m), ball valves, a positive displacement pump, nipple joints and flow reducers are also used. This fluid mechanics analysis was done with the information provided by the Introduction to Food Engineering textbook (Singh et al., 2009, pp. 117-118).

**Computation of the mean velocity**

\[ Q = A \times \bar{u} \]

Therefore the average velocity in the pipe would be 0.99 m/s.

**Computation of the Reynolds number**

\[ Re = \frac{\bar{u} \times D}{v} \]

The viscosity used in this fluids mechanics analysis is the highest viscosity of biodiesel at 40 degrees Celsius accepted by the ASTM D 6751-07b standard (Biofuel Systems Group Ltd, 2012), which is 0.000006 m²/s. From this assumption, the Reynolds number is 4191, which
gives a turbulent flow. The flow in a pipe is laminar if the Reynolds number is under 2300. Between 2300 and 4000, the flow is characterised as transitional. When the Reynolds number is more than 4000, the flow is turbulent.

**Biodiesel recirculation**

It is assumed for the fluids mechanics analysis that what is requiring the most power is mixing the biodiesel. Because of the energetic mixing, it will be the highest flow rate in the system. It is the most critical part in the fluid circulation system. Figure 14 shows the recirculation part of the system.

**Bernoulli equation**

It is the general equation for a steady state flow of an incompressible fluid with no heat added or work done in the fluid system (turbine). It is assumed for this design that even if there is heat transfer to keep the moving liquid at a constant temperature and heat loss through the insulated pipes, the viscosity of the fluid remains the same during the mixing. The Bernoulli equation used to find the power requirement of the pump contains terms for pressure energy, kinetic energy, potential energy and friction losses.

\[
Ep = \frac{p_2 - p_1}{\rho} + \frac{1}{2} \left( \bar{v}_1^2 - \bar{v}_2^2 \right) + g(z_2 - z_1) + Ef_{major} + Ef_{minor}
\]
The biodiesel reaction tank has a protective lid to prevent splashing but it is not sealed on the tank. Therefore it can be assumed that the pressure at point 1 is the atmospheric pressure, and so is the point 2 when the fluid falls into the tank ($P_1=P_2$). The pressure part of the equation can be considered as zero. Also, the mean velocity is assumed to be constant through the pipe as the pipe diameter stays constant. The mean velocity at point 1 can be considered as 0 m/s because the fluid is resting in the tank waiting to be emptied ($\bar{u}_1=0$). The mean velocity at point 2 is 0.99 m/s. Assuming that the zero elevation line is at the bottom pipe, point 1 is at 1.17m and point 2 is at 1.45m. From all these assumptions, the equation becomes:

$$g(1.17) + E_p = g(1.45) + \frac{0.99^2}{2} + E_{major} + E_{minor}$$

**Friction losses**
The equation for major friction losses is:

$$E_{major} = \frac{\Delta P}{\rho} = 2f \frac{\bar{u}^2 L}{D}$$

The total length of the piping is 2.35m in the recirculation system only. Based on the Reynolds number and assuming we are using a smooth piping system made out of PVC plastic, it is possible to find the Fanning friction factor using the Moody diagram. It is assumed that the relative roughness of the piping system is 0.000275 ($0.007*10^{-3}$ m being the roughness of a PVC pipe), and from the Moody diagram the Fanning friction factor is close to 0.01. From the equation the major friction is 1.81 J/kg.

For minor losses, contraction, expansion and fittings need to be considered. The equation to use is:

$$E_{minor} = \frac{\Delta P}{\rho} = C_f \frac{\bar{u}^2}{2}$$

Where $C_f$ is a friction coefficient proper to contraction, expansion or fittings.

**Expansion friction coefficient**
There is sudden expansion as the fluid falls into the reaction tank. The diameter of the pipe is much smaller than the tank diameter, therefore the ratio of the two can be considered as zero. In that case, the friction coefficient is 1.0. The expansion friction is 0.49 J/kg.

**Contraction friction coefficient**
In this design case there is contraction from the liquid in the tank going down the pipe. However, the contraction is not sudden because the fluid goes down a funnel shape and a fluid reducer is added to make sure there is no sudden contraction. In that case, the contraction friction will be ignored.
**Pipe fittings**
In the recirculation part of the system, there are 6 ball valves ($C_f$ of 0.05), 9 threaded unions ($C_f$ of 0.8) and 3 regular 90 degrees threaded elbows ($C_f$ of 1.5). Therefore the total minor friction is 7.86 J/kg.

**Power requirement of the pump**
Including the friction, the power requirement equation is now:

$$g(1.17) + Ep = g(1.45) + \frac{0.99^2}{2} + 1.81 + 8.35$$

$$Power = Ep \times \dot{m}$$

Assuming the biodiesel density is 900 kg/m$^3$, the mass flow rate is 0.45 kg/s. Therefore the power required for pumping the system would theoretically be 6 watts. Assuming most pumps have efficiency between 50% and 60%, it would be correct to suggest a 10 Watt pump for the system.

**Cavitation**
A positive displacement pump would be ideal for our system, and it would also prevent cavitation. Cavitation is a phenomenon where vaporization of the flowing fluid occurs and forms gas bubbles. When these bubbles collide in an impeller pump it can cause damage to the pump. Because the vapor pressure of the biodiesel that has been made at the Macdonald Campus is unknown, it is difficult to say if cavitation will occur in the system. However, positive displacement pumps are less affected by cavitation and can deal with a mixture of gas and liquid.

**Dimensional analysis**
In the case of a small scale biodiesel system, there is no need to build a significantly smaller model than the actual model. Testing the system with cheaper materials more reasonable than building the same system but significantly smaller. Therefore, in our case, we do not need to consider dimensional analysis.
Revised Biodiesel Production Steps

After the optimization described previously, the new biodiesel production steps can be integrated in this new flowchart (Figure 15).

Figure 15. Final biodiesel production flowchart
**Automation**

In order to ease the process and make it user-friendlier, the system could be automated. There are unit operations in each of the steps illustrated in Figure 15, and we can see an example of a few of the pretreatment unit operations in Figure 16. The use of a microcontroller board and various electrical devices could make the process as simple as pressing a button. In order to keep it simple and affordable, the ideal system would require a little more attention, yet be very straightforward and easy to use. Installing an electrical relay for the pump, electrical element and thermostat would give control of the pumping, stirring and heating to the microcontroller. Since the system already has a pump and valves, then installing electric valves and level sensors would allow almost fully automatic operation. Going further with temperature sensors instead of a thermostat would allow data logging for further fine tuning of the system.

*Figure 16. Example of unit processes for an acid pretreatment*
Discussion and Conclusion

This project was mainly intended to design a small-scale biodiesel production system and from what was assessed it can be reasonable to state that this type of system could probably be implemented on campus. Even though it wasn’t actually built, the prototyping gave a rough idea of what main causes of errors might be reflected and what efficiently works. However, there are some issues that arose which have to be corrected to further advance the potential for implementation.

In term of feedstock, the WVO from residences downtown can be reasonably declared as a reliable source. There is no evidence that shows that this essential component may not be available in the future. There are still some concerns about transportation but this can most likely be managed well without having negative impact. However, the biodiesel reaction process (transesterification) was more complicated than expected. The titration result is clearly carrying out the need to perform an acid pre-treatment, and this correlates also to what the downtown team, working on a pretty similar project, discovered and recommended. Regarding results from tests (methanol and water test), there are obvious problems in the chemical reaction itself. This was probably due to an inaccurate protocol, but perhaps more importantly by deficient manipulation skills and practice times. It has to be also noticed that literature assesses biodiesel production from many different sources and therefore recommends different proportions with respect to; oil to methanol ratio, processing time, amount of lye to added etc. Therefore it is difficult to directly use recommendations from literature given our unique feedstock source. Hence, it would be beneficial to further study the transesterification in more depth with the given feedstock in order effective us in the biodiesel production system designed.

The location for our system for now would be located in the Bioresource Engineering shop in a room that is currently available. This room would however, need to be properly ventilated before we could install our system. As the system is fairly small in size, other potential locations could probably be made available in the future.

With respect to the design of the system, two final designs were presented which different based on their materials and approximate prices. As it was shown, both designs respect all design considerations, with the exception of the plastic design not being fire proof. The ideal design composed of mainly stainless steel is much more expensive due to the significant costs of the tank, the pump, the fittings, etc. Considering the systems application at Mac Campus, these economic considerations make the ideal system unfeasible unless second hand material could be acquired. The plastic design would work well but would not be fire proof. However, with proper operation there is little risk for fires, as the main fire hazard results from an open system in a poorly vented room.
The prototype was a good example of how to properly select adequate equipment when designing a system, as there were many limitations to be applied with the given budget. It was also difficult to find affordable smaller size components and acquire the different parts missing, to then assemble them into a system. Once done, scaling up the experiment proved to be as difficult as expected, yet the processor did function to the desired extent. The system of valve and pipes was easy to use when well secured, and the pump and heating element did exactly what was expected.

The biodiesel quality test, as shown, was not very conclusive. As stated to fulfill ASTM standard requirement the transesterification reaction must be further investigated. Also other more accurate testing methods underlining; completeness of the reaction with an HPLC, alcohol and catalyst content as well as water and sediment content needed to be accounted in the future. Moreover there are other parameters which would have to be assessed; hence a complete analysis on this could be done. Most probably, an investigation by subsequent future engineers from the department could be available only on this subject. If not, the ASTM standard in this case can be extensively overviewed by external chemical lab.

Optimization proposes how to implement acid pre-treatment which, as previously mentioned, is necessary considering the high FFA content in the WVO. Even if it didn’t result in a better transesterification after the reaction, it was still necessary to attempted. Other than that, to provide a good quality the neutralization and the washing of the biodiesel have to be generated. These steps are not complicated to implement but are required and should be performed. Additionally, since methanol involves an inevitable cost, alcohol recovery via glycerol treatment prior to transesterification is a possibility, although it would require equipments (vacuum flash process or evaporator) and complexity.

From this project, there are a couple of major points that can be concluded. Firstly, even if biodiesel seems to be easily processed on paper, it still has to be done with rigorous discipline and diligence. Designing a system to process is feasible but making sure the reaction will process as well as to be able to have a decent product quality are factors much more difficult to achieve. Active research and further studies are still needed to fulfill this task within campus, but there are no signs after conducting this assessment which would seriously impede the feasibility of implementing a biodiesel processing system at Macdonald campus.
References


Blayney, A., 2012, The Optimization of Biodiesel Production, Department of Chemistry, McGill University, Montreal, Québec, Canada


Godlisten, G., 2008, High Free Fatty Acid (FFA) Feedstock Pre-Treatment Method for Biodiesel Production, Second International Conference on Advances in Engineering and Technology.


Appendix 1: Biodiesel Data

Free Fatty Acid Content and KOH Extra Lye Calculation

1. Molarity of KOH and NaOH

KOH specifications: (According to what is published by the company)

- 85% wt/wt
- Molecular weight = 57.11 g/mol

When preparing the titration 1g of KOH at 85% purity was mixed into 1L of water. So there is 0.85g of KOH in 1L of water. In terms of mol there is:

\[
0.85 \text{ g} \div 57.11 \text{ g/mol} \rightarrow 0.01488 \text{ mol of KOH in 1 liter}
\]

So the molarity (M) is 0.01488.

If NaOH is used (M) is approximately 0.025 at 99% purity.

2. Determination of FFA content in oil sample

During the titration of WVO 5.346ml of KOH solution at 0.01488M was used to neutralize the acid. Hence the amount of mol of KOH use for titration is:

\[
0.01488 \text{ mol/l} \times 0.005346l = 7.95 \times 10^{-5} \text{ mol}
\]

Since we titrated 1ml of WVO, and assuming the number of mol of KOH added in the titration is equal to the number of mol of FFA, there are 7.95×10^{-5} mol of FFA per ml of oil.

3. Determination of percent weight FFA in the WVO

Molar mass of FFA = 261.8 g/mol  (Blayney A, 2012)

FFAs content in gram first need to be known

\[
FFAs(g) = FFAs(mol) \times FFA \text{ molar mass}
\]

\[
FFAs(g) = 7.95 \times 10^{-5} \text{ mol} \times 261.8 \frac{\text{g}}{\text{mol}} = 2.08 \times 10^{-2} \text{ g}
\]

Knowing the mass of oil added.

\[
\text{Mass of oil} = \rho \text{ of oil} \times \text{volume of oil in the titration}
\]
\[ Mass \ of \ oil = 0.9243385 \frac{g}{ml} \times 1ml = 0.9243385 \ g \]

Hence the weight to weight percentage of FFAs to oil is:

\[ \frac{W}{W} \% = \frac{Mass \ FFAs(g)}{Mass \ oil(g)} \times 100 = \frac{2.08 \times 10^{-2} g}{0.9243285 g} \times 100 = 2.25 \% \]

4. Extra lye to be added
To account the extra added, if it is known that you need \( 7.95 \times 10^{-5} \) mol of KOH to neutralized FFAs per ml of oil, then at least 4.54 g of KOH is needed since:

\[ Extra \ KOH = 7.95 \times \frac{10^{-5} \ mol}{oil \ ml} \times \text{Molecular Weight of KOH} \frac{g}{mol} \times \frac{1000 \ ml}{L} \]

\[ Extra \ KOH = 7.95 \times \frac{10^{-5} \ mol}{oil \ ml} \times 57.11 \frac{g}{mol} \times \frac{1000 \ oil \ ml}{oil \ L} = 4.54 \ g \ per \ oil \ L \]

**Transesterification and Titration Reaction: Steps in point-form**

1. Determine the amount of oil to transesterify
2. Take down potential impurity within the oil through filters
3. Heat the chosen amount of oil at 60°C
4. Titration
   4.1. Mix 1g of lye (catalysis) (NaOH or KOH) with 1L of water (titrant)
   4.2. Mix 1ml of oil in 10ml of isopropanol (titrand)
   4.3. Add 2 or 3 drops of phenolphthalein in the oil and isopropanol mixture
   4.4. Add slowly the lye mixture with a pipette in the titrand
   4.5. Stop adding when the solution turn pinkish
   4.6. Measure titrant quantity
5. Use a 6:1 methanol to oil molar ratio
6. In the methanol mix the 1% w/w KOH to Oil ratio
7. Add to this the extra amount of lye needed
8. Once the KOH is dissolve in the methanol, poor the solution in the oil container
9. Mix the solution during 1h
10. Settle for 24h
Acid Pretreatment Point Form Steps

1. Determine the volume of oil pretreated
2. Heat the oil to about 35°C
3. Determine the volume of methanol need according to a 3:10 methanol-to-oil volume ratio
4. Pour 1% sulfuric acid-to-oil volume ratio (H₂SO₄) in the methanol solution
5. When the oil is at temperature, incorporate the methanol mixture in the container
6. Let the mixture stir for an hour or two
7. When the chemical reaction done, titrate the solution and proceed to transesterification

ASTM D6751 standard

ASTM standard was taken from internet since it costs money to buy this standard chart from ASABE organization.

<table>
<thead>
<tr>
<th></th>
<th>Method</th>
<th>Limits</th>
<th>Units</th>
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<tbody>
<tr>
<td>Flash Point, Closed Cup</td>
<td>ASTM D93</td>
<td>93 min</td>
<td>°C</td>
</tr>
<tr>
<td>Water and Sediment</td>
<td>ASTM D2709</td>
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<td>% volume</td>
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<tr>
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<td>mm²/s</td>
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<td>Sulfated Ash</td>
<td>ASTM D874</td>
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<tr>
<td>Sulfur</td>
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<td></td>
<td></td>
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<td>S 15 Grade</td>
<td>ASTM D5453</td>
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<td>% mass</td>
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<td>S 500 Grade</td>
<td>ASTM D5453</td>
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<td>Copper Strip Corrosion</td>
<td>ASTM D130</td>
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<td>Alcohol Content</td>
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<tr>
<td>Methanol Content</td>
<td>EN 14110</td>
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<td>% volume</td>
</tr>
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<td>°C</td>
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<tr>
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<td>ASTM D613</td>
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<td>Cloud Point</td>
<td>ASTM D2500</td>
<td>Report to Customer</td>
<td>°C</td>
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<tr>
<td>Carbon Residue</td>
<td>ASTM D4530</td>
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<td>% mass</td>
</tr>
<tr>
<td>Acid Number</td>
<td>ASTM D664</td>
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<td>mg KOH/g</td>
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<td>Free Glycerin</td>
<td>ASTM D6584</td>
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<td>Vacuum Distillation End Point</td>
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<td>Oxidation Stability</td>
<td>EN 14112</td>
<td>3 min</td>
<td>hours</td>
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<tr>
<td>Calcium &amp; Magnesium (combined)</td>
<td>EN 14538</td>
<td>5 max</td>
<td>ppm</td>
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</table>

Available at: http://www.eurofueltech.com/Upload/File/American_and_European_Biodiesel_Quality_Standards.pdf
Appendix 2: Chosen Components
Appendix 3: Viscosity Measurements

Experimental viscosity measurements of a batch of biodiesel made using the Brookfield viscometer

<table>
<thead>
<tr>
<th>Biodiesel viscosity at room temperature – Brookfield viscometer measurement</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>20 rpm</td>
<td>7.6%</td>
<td>22.5 cP</td>
</tr>
<tr>
<td>30 rpm</td>
<td>17%</td>
<td>33.8 cP</td>
</tr>
<tr>
<td>50 rpm</td>
<td>35.2%</td>
<td>42.1 cP</td>
</tr>
<tr>
<td>60 rpm</td>
<td>44.6%</td>
<td>44.6 cP</td>
</tr>
<tr>
<td>100 rpm</td>
<td>82%</td>
<td>49.02 cP</td>
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</table>

<table>
<thead>
<tr>
<th>Biodiesel viscosity at 40 degrees Celsius</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>30 rpm</td>
<td>0.8%</td>
<td>1.6 cP</td>
</tr>
<tr>
<td>50 rpm</td>
<td>2.7%</td>
<td>3.2 cP</td>
</tr>
<tr>
<td>60 rpm</td>
<td>2.6%</td>
<td>2.5 cP</td>
</tr>
<tr>
<td>100 rpm</td>
<td>8.0%</td>
<td>4.8 cP</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Waste vegetable oil viscosity at 40 degrees Celsius</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>30 rpm</td>
<td>2.0%</td>
<td>4.0 cP</td>
</tr>
<tr>
<td>50 rpm</td>
<td>7.0%</td>
<td>8.0 cP</td>
</tr>
<tr>
<td>60 rpm</td>
<td>9.0%</td>
<td>9.0 cP</td>
</tr>
<tr>
<td>100 rpm</td>
<td>15.5%</td>
<td>9.25 cP</td>
</tr>
</tbody>
</table>

From these viscosity measurements, the three liquids are non-Newtonian and they all seem to express a shear thickening behavior. This behavior is often due to particles in suspension in the liquid, which would make sense because the waste vegetable oil is opaque.

**Signification of all the symbols**

- $Q$ is the flow rate (m$^3$/s)
- $A$ is the cross-section area of the pipe (m$^2$)
- $\bar{u}$ is the average velocity of fluid in the pipe (m/s)
- $\nu$ is the kinematic viscosity (m$^2$/s)
- $\rho$ is the density (kg/ m$^3$)
- $g$ is the acceleration due to gravity (m/s$^2$)
- $E_P$ is the energy requirement of a pump per unit mass (J/kg)
- $P$ is the pressure at the corresponding location on the system (Pa)
- $z$ is the elevation at the corresponding location on the system (m)
- $E_f$ major is the sum of all the major friction forces in the system (J/kg)
- $E_f$ minor is the sum of all the minor friction forces in the system (valves, fittings, etc.) (J/kg)
- $f$ is the Fanning friction factor obtained from the Moody diagram
- $L$ is the length of the pipe (m)
- $D$ is the diameter of the pipe (m)