Advanced vacuum biodiesel process.

Preamble:

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Purpose:

The purpose of this document is to describe an advanced process for converting waste vegetable oil to methyl ester biofuel.

Scope:

This document covers a two stage acid -base catalyzed reaction which is suitable for converting waste vegetable oil which has been over used to biofuel. Over use results in the oil breaking down and becoming converted to highly saturated fatty acids which are very unhealthy for the body and not particularly pleasant for making fuel. When feed oil is known to be too acidic to be practical for single stage conversion it is advantageous to use this process to get more complete conversion of the input stream with less chemical use. There is a limit to even the use of multistage processes if cooking oil is abused to a high degree. Experimentation and quality testing with a particular reactor geometry determines what range of feedstock pH can be tolerated with this process in order to produce high quality fuel. It is always better to start with the best quality input oil to convert to high quality fuel. It is worth the effort to find better oil if you find that you need to use two stage processing.

Preparation:

Personal protection :

1. Rubber gloves.
2. Lab coat.
3. Safety glasses.
4. Cold running water.

Reactants :

1. Vegetable Oil
2. Methanol (CH_3OH) 98% purity or better.
3. Concentrated Sulfuric acid 93% or better
4. Potassium hydroxide (anhydrous) 85% or better

Needed for Washing :

1. Water
2. Table vinegar (5%) or phosphoric acid (85%).

Needed for final quality test :

1. Methanol same as above.
2. 100 ml KIMAX graduated oil tube.

Needed for glycerin separation :

1. Phosphoric acid (85%)

Needed for Methanol recovery :

1. 3A molecular sieves.

Process overview

1. Vegetable oil is dried
2. Calculated quantities of methanol and sulfuric acid are mixed and added while recirculating heated oil. Recirculation is continued for at least 60 minutes.
3. Calculated quantities of potassium hydroxide and methanol are mixed and then added while recirculating heated oil. Recirculation is continued for at least 60 minutes.
4. Settling and glycerin removal.
5. Wash test performed
6. Excess methanol is recovered from fuel (recommended option)
7. Biodiesel is then washed and dried.
8. It is then checked for quality.

Process details

1. **Fill** the reactor with a known quantity of oil no more than the maximum mark as follows. Connect the inlet hose to the inlet valve and turn on the exhaust and vacuum pump. Insert the end of the inlet hose into the oil keeping it above the level of any sludge settled in the oil reservoir. It is recommended to allow waste oil to settle before using it. Open the vacuum valve to draw oil into the system. Observe the level rising in the sight tube. Fill to the required height. Close the inlet valve, turn off the vacuum pump and exhaust then disconnect the inlet hose.
2. **Heat** the oil to remove any water content. Waste oil will probably contain water, which will lead to soap formation in the process. Water is the enemy of this process so it must be eliminated at every point. Allow the reactor to reach full temperature of at least 58 degrees Celsius. The thermostat clicks on and off once the working temperature is reached. Open the inlet valve to allow air to enter the tank and drain any water which has collected in the tank bottom with the drain valve. Recirculate the oil to homogenize any remaining traces of water to avoid steam pockets forming below the oil and exploding, foaming oil up to the vacuum port. Save any oil that comes out with the water to separate and reuse later. Evacuate the tank to remove remaining adsorbed water from the oil and collect it in the liquid trap. Water will begin to be removed when the temperature of the vacuum port begins to rise. Water will begin to drop from the end of the coaxial gas drier. At this point pumping should be throttled so that rate of drip does not become a steady stream. If water removal proceeds too quickly the oil will foam up and foul up the coaxial gas drier. Continue to remove water until vacuum improves to 27” Hg and drip rate slows. The

vacuum port temperature will also fall. At this point the oil is dry. Virgin oil does not need to have the water boiled off, in which case do not do it, boiling means unnecessary extra energy and time input into the process. Remove a sample from the bottom of the tank. Discard any liquid water and titrate the oil. After this step it will be required to have 3-4 in Hg vacuum in the reactor to begin the next step. Exhaust and vacuum pump may be shut down until it is time to add methoxide to the reactor.

3. **Acid stage:** Multiply the number of litres of oil to be converted by 0.12 to obtain the amount of methanol to be used in the acid stage of the process. For the example of 90 litres this would be 90 x 0.12 = 10.8 litres. The stainless mixing vessel used in the 90 litre reactor cannot hold this much methanol so the acid stage of the process will be done in two steps requiring 5.4 litres of methanol each. For each litre of oil 1 ml of concentrated sulfuric acid will be used and this will also be divided in half and done in two steps with half of the methanol as outlined above. So in the 90 litre case use 45 ml of acid in each step. Ensure that the oil has reached at least 58 degrees Celsius. Turn on the exhaust fan and add the methanol to the mixing pot. Turn on the mixing motor before adding the acid to the methanol. Carefully measure the acid in a graduated cylinder and slowly pour it in to the methanol. Evacuate the reactor to no more than 3 to 4 inches of mercury. Turn on the recirculation pump and open the chemical injection valve approximately one turn. Carefully regulate the vacuum valve to maintain no more than 4" Hg vacuum in the tank. Excess vacuum will cause pump cavitation and loss of mixing action. If this happens immediately close the chemical injection valve, then vent the reactor vacuum which will result in the recirculation pump returning to normal operation. Then begin again with gentle vacuum and chemical delivery. Continue until all the first half of the acid/methanol mixture is added. Repeat for the second half. Momentarily open the inlet valve to release any remaining vacuum and continue with the recirculation for at least 60 minutes while maintaining the temperature at 58 degrees minimum.
   CAUTION : Sulfuric acid is a dangerous chemical which causes severe chemical burns if spilled on the skin. Use personal protective equipment as outlined in the introduction of this document and work in a location where you can immediately rinse any spilled acid with running water. Sulfuric acid and methanol can produce small amounts of dimethyl ether which is poisonous and explosive. Forced exhaust is mandatory.
4. **Base stage:** For the base stage the amount of methanol needed is 80 ml for each liter of oil to be processed. For the 90 litre example the methanol amount would be 90 x 0.08 = 7.2 litres. As in the acid stage, the base stage will be done in two steps with 3.6 litres each. Turn on the exhaust fan and add the methanol to the mixing pot. Using a lab scale which is accurate to $1/10^{th}$ of a gram, quickly weigh 1/2of the total amount of KOH calculated. The KOH amount for the base stage is dependant on base purity and is calculated as the stoichiometric amount plus the amount needed to neutralize the acid from stage one, 5 + 1.9 gives 6.9 grams in the case of pure KOH or for 90% pure KOH use 7.6 grams per litre, or for 85% KOH use 8.1 grams for each litre of oil. So in the case of a 90 litre batch with say 90% pure KOH, , use 90 x 7.6 = 684 grams. Each step of the base process would then use 342 grams. Turn on the mixer and pour in the KOH. Keep the KOH container closed as much as possible while doing all of this. The methanol is mixed into a solution with the KOH, creating potassium methoxide in an exothermic reaction (it warms up). With large batches it may be necessary to watch the temperature rise when mixing methoxide to avoid thermal runaway which could result in boiling of the methoxide when starting temperatures are high as they might be in the summer for example. The KOH should be dissolved in less than 10 minutes. Keep all utensils the KOH comes in contact with as dry as possible. Repeat for the second half of the methanol and KOH amounts, and then as in the acid stage continue to recirculate for 60 minutes or more while maintaining a minimum temperature of 58 degrees Celsius.
   CAUTION : Treat potassium methoxide with extreme caution! Do not inhale any vapors! Methoxide kills nerves immediately. If any potassium methoxide gets splashed on your skin, it will burn you without your feeling it. Wash immediately with lots of water. Always have a hose running when working with potassium methoxide. Potassium methoxide also reacts with aluminum, tin and zinc and will destroy paint. Use plastic, glass, enamel or stainless steel utensils for handling KOH and methoxide.
5. **Settling and separation :** Allow the solution to settle and cool for at least eight hours, preferably longer. The methyl esters (biodiesel) will be floating on top while the denser glycerin will have

collected at the bottom of the tank. Then carefully remove the glycerin. This can be done by draining it out of the bottom of the reactor through a transparent hose. Remember that the inlet valve must be open to allow air to enter the tank to replace liquid volume which is being removed. The semi-liquid glycerin has a dark brown colour and the biodiesel is honey-coloured. Keep a watch on what flows through the sight tube. When the lighter-coloured biodiesel appears divert it to a separate container.

6. **Glycerin cocktail :** The glycerin from oil is brown and may turn to a solid below about 20 deg C. Glycerin (glycerol) is the main co-product of making biodiesel. Theoretically 79 ml of glycerin per litre of oil used, 7.9% glycerin should be produced. The cocktail drained from the system is not pure glycerin. What sinks to the bottom of the biodiesel processor during the settling stage is a mixture of glycerin, methanol, soaps, water and the excess KOH catalyst. Most of the excess methanol and most of the catalyst remains in this layer. Once separated from the biodiesel, adding phosphoric acid to the glycerin layer precipitates the catalyst out as potassium phosphate which is useful as a fertilizer, and also converts the soaps back to free fatty acids (FFAs), which float on top. You are left with a light-colored potassium phosphate precipitate on the bottom, glycerin/methanol/water in the middle, and FFA (free fatty acid) on top. The excess methanol can be recovered similarly to the way water is removed from oil when drying. Recovered methanol must be dried for reuse in the process which is an advanced technique. Another idea for disposing of the glycerin is breaking it down, with an anaerobic digester to produce methane gas. This is mentioned here for reference but is covered in detail in the advanced process documentation.
7. **Soap :** Suspended in the biodiesel will also be some soapy residues. These are the result of K^+ ions from the KOH reacting with water created when the methanol bonds with the ester chains along with any other water that was suspended in the oil. If the reaction produces more than the usual amount of soap, this is due to KOH coming into contact with water before it has a chance to react with the oil. In this case the excess water should have been removed first.
   The part of the process where it is vital to keep all water out of the reaction is when making the potassium methoxide. Keep the vessels KOH comes in contact with as dry as possible. The chances of a good clean splitting of ester from glycerin with little soap by-product are much better on a warm dry summer day than on a damp rainy day. Soaps and excess catalyst must be removed from the fuel by washing.
8. **Wash test :** Before proceeding with the fuel washing stages at this point a crude quality test is performed to determine if the process proceeded correctly. A small sample (100 ml) is drained into a clear plastic bottle and the same amount of water is added and then the bottle is vigorously shaken for a few seconds. The bottle is left to stand and the water and fuel should separate within a minute or two. If not, a few ml of vinegar may be added to encourage separation but the need to use it suggests that the reaction was incomplete. Incomplete reactions leave some amount of partially reacted or unreacted oil molecules in the fuel. This is not immediately disastrous to an engine and may not be noticed but long term use can result in a reduction of the engine service life and may violate engine warranty conditions.
9. **Washing :** If the wash test passed then add an amount of water equal to 20% of the fuel volume (assume same as original oil volume) to the tank. The fuel will contain some excess methanol which can be removed in the liquid trap as per drying. Methanol recovery is an advanced technique. Hard water is fine for the purpose of washing. Soft water may result in emulsion problems when reactions have not gone close enough to completion. Highly complete reactions will not require any vinegar or phosphoric acid in the first wash to separate well. Washing with phosphoric acid can reduce the amount of water consumed and the number of required washes. If you wish to use an acid wash the quantities are 8 ml of table vinegar (5% strength) per litre of water used for washing. In the case of phosphoric acid use 2ml of 10% strength for each litre of wash water. The acid brings the pH of the solution closer to neutral because it neutralizes and drops out any KOH suspended in the biodiesel. The recirculation pump is turned on and the sight tube at the bottom is observed until all the water is evenly mixed with the fuel. The recirculation pump is then switched off and the system is allowed to sit while the water settles. After settling the wash water is drained by opening the inlet and drain valves as in draining glycerin. The wash water ends up with the soaps, excess catalyst, and traces of glycerin which were not drained as well as methanol if not recovered from fuel. First wash water is discarded and is not environmentally dangerous. Second and third wash water is reclaimed and used as the first and

second (respectively) washes for the next batch of fuel.
At least three washes are required to remove close to 100% of soaps. The second and third washings can be done with water alone. After the third washing the drained water should be clear and have the same pH as it did prior to the wash. The finished fuel should have a pH of 7. If the reaction was complete and wash water splits quickly, the settling time for wash cycles can be reduced. Two hours should be held as the minimum settling time per wash cycle.

10. **Drying :** Final wash can be done with hot water and the heater may be turned back on. After settling and draining the temperature should be raised to 55 degrees C and the exhaust and vacuum pump are turned on and water is then removed from the fuel just like removing water from oil at the start of the process. Take care not to dry too quickly. As the fuel dries it will become crystal clear in the sight tube. When a vacuum of 27" Hg is reached at 55 deg. C. then the drying process is complete and the fuel may be drained from the system for use. As a precaution the first bit of fuel should be drained into a small container (in case of any water in the bottom of the reactor) and returned to the second or third wash water. Check that the fuel runs consistently clear before diverting it to a fuel can.
11. **Methanol test :** To check how close to complete the reaction was, another quality test can now be performed. Biodiesel is soluble in methanol but unreacted components are not. Add 10 ml biodiesel to 90 ml methanol. Each 1ml of liquid which drops out represents 10% contaminant in the fuel. This is not a totally conclusive test and does not define specifically what contaminants are present in the fuel but passing this test will give you confidence that your fuel is of reasonably high quality. Many of the various international quality standards for biodiesel state that the fuel must be more than 96.5% ester content by mass. Accordingly there should be less than 0.35 ml of liquid which drops out of the methanol test. The graduated oil tube has markings that represent 0.05 ml for the first 0.5 ml.
12. **Reprocessing :** A 1 litre sample of the fuel may be tested by treating it as if it was virgin oil and reprocessing it. In this case no titration is required and only the basic amount of catalyst is used along with only 10% methanol by volume for the methoxide. Process as usual. If the reaction was complete there will be no further glycerin produced. Fuel which fails the methanol test can be reprocessed with this method to yield high quality product. If this happens it is likely that the process did not go far enough for one or more of several reasons. The most likely are due to poor feed stock quality or poor process conditions, low temperature, insufficient mixing, insufficient time, improper measurements, poor accuracy on measurements or loss of methanol during the process. The first thing to check is process temperature and try increasing it by a degree or two. Methanol boils at 64 degrees Celsius and going above 60 degrees increases the probability of methanol evaporation during the process, which becomes counterproductive. Next thing to try is extending the reaction time by recirculating longer at temperature.
13. **Glycerin separation :** Initially a test will need to be done to determine how much phosphoric acid will be required to split the glycerin. Put one litre of glycerin cocktail into each of four containers. To each of the containers add 30, 31, 32and 33 ml of phosphoric acid respectively. Allow the mixtures to sit in a warm room overnight and use the jar which required the minimum quantity of phosphoric acid as a guide to determine what ratio to use for a larger batch. The FFA (free fatty acid) layer is darkest and floats on top. The glycerin/water/methanol fraction is lighter and settles below the FFA. On the bottom is a white potassium phosphate layer. Read the section on the journey to forever website for more detail on this process.
14. **Methanol recovery from glycerin :** The purified glycerin can be collected and when a large amount is ready it can be returned to the reactor and heated to remove the methanol.
15. **Acknowledgements :** Much of the information in this document was obtained from the Journey to forever website www.journeytoforever.org. The methanol test was developed by Jan Warnqvist and is reprinted from the journey to forever site with permission here. The two stage acid –base process was developed by Bob Allen of www.ozarker.org . My thanks to these sources and others which through many online discussions have contributed to the development of the reactor and the process that runs on it.

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