Basic vacuum biodiesel process.

Preamble:

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Purpose:

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

Scope:

This document covers a simple single stage base catalyzed reaction which is suitable for converting virgin or waste vegetable oil which has not been overused 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. A titration is done on waste oil to determine the extent of breakdown which occurs by high temperature use of the oil and depends on the length of time and temperature of oil use which makes the oil more acidic. Experimentation and quality testing with a particular reactor geometry determines what range of feedstock pH can be tolerated with this simple process in order to produce high quality fuel. More catalyst is required as the situation worsens which increases the cost of fuel and complicates the process. Conversion efficiencies begin to decline as input streams (both materials and energy) increase when using single stage process as feedstock pH progressively decreases. At some point it becomes more economical to use a more advanced two stage process which is more complicated but results in less chemical use and more complete reactions with highly saturated oils. See the advanced vacuum biodiesel process for details on this. In addition the advanced process describes other advanced techniques such as methanol recovery and glycerin separation which are not included in this document.

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. Potassium hydroxide (anhydrous)

Needed for Titration :

1. Isopropyl alcohol 99% purity
2. Distilled water
3. Phenolphthalein solution
4. Test tube
5. Stir bar
6. Retort stand
7. Mini magnetic stirrer
8. KOH minimum 85% assay.
9. 0.1% w/v KOH/DI water solution.
10. 10 ml x 0.1 ml pipette and control bulb.

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.

Process overview:

1. Vegetable oil is dried
2. Titration is done to determine how much catalyst is needed.
3. Exact quantity of potassium hydroxide and methanol are mixed to produce anhydrous potassium methoxide.
4. Vegetable oil is heated to 55 deg C and the potassium methoxide is added while recirculating tank contents and recirculated a further 60 minutes.
5. Settling and glycerin removal.
6. Wash test performed
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. 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. **Titration :** If necessary, prepare the solution for titration as follows. Once a stock of solution is prepared it can be used as described below. Dissolve 10 grams of KOH in 1 liter of distilled or de-ionized water and label this as 1.0% KOH solution. Measure 100 ml of the 1.0% KOH solution you just made and add it to 900 ml DI water and label this jar 0.1% KOH solution. Use this solution for titration. Use phenolphthalein solution to get end point. In the test tube, warm and dissolve 2 ml of dewatered oil in 20 ml of pure isopropyl alcohol. Warm the test tube gently using a mini magnetic stirrer until all the oil dissolves in the alcohol and the mixture turns clear. Add 6 drops of phenolphthalein solution.
   Using a pipette, add 0.1% KOH solution drop by drop to the oil alcohol phenolphthalein solution, stirring all the time, until the solution stays magenta for 10 seconds. Take the number of mls of 0.1% KOH solution you used and divide by two. This is the number of grams of KOH you add to the basic amount.
4. **Basic amount of KOH :** This depends on the purity of KOH. (Theoretical divided by purity percentage.)

If it is 100% purity use 4.9 g.

If it is 90% then use 5.5 g.

If it is 85% then use 5.8 g.

If it is less than 85% then do not use it!

5. **Calculate required catalyst :** Add the number from the titration step (which you have divided by two already) to the number for the basic amount above. The result is now multiplied by the number of litres of oil to be reacted. As an example where 90 litres of oil are in the reactor and the titration amount was 2.5 and we are using 90% purity KOH:

90 x (5.5 + 2.5) = 720 grams.

Since KOH adsorbs moisture from air it is beneficial to mix methoxide in small batches. Divide the amount into 3 equal amounts. Therefore in this case 240 grams of KOH would be dissolved in 6 litres of methanol for three times.

6. **Preparing methoxide :** Generally the amount of methanol needed is 20% of the vegetable oil by volume. This may vary depending on the density of the oil but is a general guideline. When transesterifying 90 liters of vegetable oil, use 18 litres (6 litres at a time) of methanol. Turn on the exhaust fan and add the methanol to the methoxide mixer. Using a lab scale which is accurate to $1/10^{th}$ of a gram, quickly weigh 1/3 of the total amount of KOH calculated. Turn on the methoxide

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.

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.

7. **Heating and mixing :** Pre-heat Vegetable oil 54-58 deg C. Add the potassium methoxide to the oil while recirculating the mixture until all the required methoxide is added. This step will be done in three stages with 1/3 of the total amount each time. This is accomplished with 3-4 inches Hg vacuum and careful adjustment of the methoxide delivery valve. Open the methoxide valve approximately 1 turn and observe the oil become cloudy and somewhat darker at the outlet side of the recirculation pump. Regulate the flow and vacuum until all the methoxide is added. Do not allow the vacuum to increase beyond 3-4 inches as it will cause cavitation in the recirculation pump and may also result in loss of methanol into the liquid trap. If the vacuum rises then reduce it by slightly opening the inlet valve until vacuum approaches normal range. If the pump cavitates and stops recirculating then close the methoxide valve, vent the system –which should result in the recirculation pump working properly again, and then start the process of adding methoxide over again. Do not allow the pump to cavitate by keeping reactor vacuum lower than 4" Hg. Too little vacuum will not allow the methoxide to enter the system. Repeat the procedure for the remaining two thirds or the required amount of methoxide. When methoxide is completely added, then release the vacuum by momentarily venting through the inlet valve and then close it and leave the system closed. Continue to heat and recirculate for an hour. The reaction is often complete in 30 minutes, but longer is better. The transesterification process separates the methyl esters from the glycerin. The OH from the KOH stabilizes the glycerin and CH_3O of the methanol forms the ester chains.
8. **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 color and the biodiesel is honey-colored. Keep a watch on what flows through the sight tube. When the lighter-colored biodiesel appears divert it to a separate container.
9. **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 liter 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 and the 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.
10. **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.

11. **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 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.
12. **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 1.5 ml of table vinegar (5% strength) per litre of oil originally processed. So in the case of 90 litres oil use 135 ml of table vinegar in the first wash. In the case of phosphoric acid use 0.2ml of 85% strength for each litre of oil originally processed, so for the 90 litre sized batch the amount of phosphoric acid to use would be 18 ml. 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.
13. **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.
14. **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 1 ml 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 on 0.05 ml for the first 0.5 ml

15. **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 (titration amount too high) or poor process conditions, low temperature, insufficient mixing, insufficient time, improper measurements, poor accuracy on measurements or loss of methanol during the process. Through a series of tests and experience you will determine what the upper titration amount is which works with your feedstock. The other parameters relate to care and discipline in your lab practice. Do not get lazy and cut corners, it does not work. If you have trouble finding feedstocks that titrate low enough to have success with these processes the easiest thing to do is try a different source for your waste vegetable oil. Failing that there is a more advanced two stage process which can be carried out on the reactor but it is covered in a separate document called Advanced vacuum biodiesel process.
16. **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. 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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