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# Eagle Diesel Production Manual

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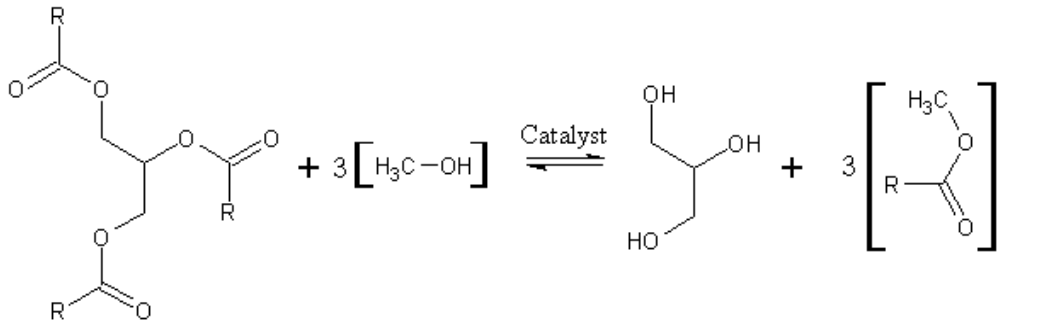
## Introduction to Biodiesel

Biodiesel is a biodegradable, combustible fuel made from vegetable oils and/or animal fats. It can be used in any combustion engine which accepts regular diesel fuel. Typical use is B20 (20% biodiesel) in the summer and B5 (5% biodiesel) in the winter for New York State.

Biodiesel is produced by using an alcohol and a caustic catalyst such as sodium hydroxide or potassium hydroxide. Methanol is the most commonly used alcohol since it is cheap and easy to obtain.

In the process, methanol and the catalyst are mixed together into a solution. The methyl catalyst is then slowly mixed into the vegetable oil for a certain period of time. Once a complete reaction is made, the methyl esters will replace the free fatty acids in the vegetable oil and drop out as a glycerin by-product. After a few hours of settling, you're left with biodiesel on the top and glycerin on the bottom.

When making biodiesel with used cooking oils and greases, the fats and oils react with methanol, using a strong alkaline catalyst (sodium hydroxide or potassium hydroxide) to produce mono-alkyl methyl esters (biodiesel) and glycerin (also called glycerol). Sulfuric acid acts as a catalyst to remove free fatty acids and is used as a pre-biodiesel reaction. NOTE: The equipment should be housed in a heated location.




Triglyceride Methanol (3) Glycerol Methyl Esters (3)

There are two waste products that come from this biodiesel production. The main by-product of the trans-esterfication process is glycerin. The second waste stream is from the final biodiesel wash cycle which removes residual methanol, glycerin, and acid salts. After residual oil removal the wash cycle water is safe to dispose of down the drain.

## Making Biodiesel

**Materials needed:**

Oil

Sulfuric acid

Methanol

Potassium hydroxide (KOH-catalysis)

**Equipment from the lab:**

(3) Glass pipets

(4) 250mL beakers

250mL round bottom flask

600mL waste beaker

Magnetic Stirrer

Scale

Weight boat

Condenser

Iron clamp

Heated oil bath

Seperatory funnel

**Safety hazards**

**17M Sulfuric acid:** This is a very strong acidic solution and very corrosive. This solution should not be kept in any metal containers. If spilled on your skin, wash the area with soap and water for several minutes. If spilled on any surface wipe it down with a wet sponge, and then rinse the sponge well.

**Methanol:** This is an alcohol and is extremely flammable; this should be keep away from any electrical outlet or heating device. If spilled on your skin or any surface, the area should be washed with a wet sponge. Methanol is not known to hurt your skin with immediate contact but with prolonged exposure it could cause irritation, this should be treated as any other hazard.

**Waste instructions**

All waste materials and leftover materials should be collected in the hazardous waste bottle.

**Scaled measurements for automated production**

Based on a 50 gallon batch (most automated Biodiesel production kits use 50 gal):

50 gal of oil

10 gallons of methanol

2350g of KOH

190mL of sulfuric acid ($H_2SO_4$)

1 gal = 3.78L/gal

50 gal x 3.78L = 189L of oil/per batch

Methanol per 50 gal batch:

10 gal methanol

10 gal x 3.78L= 37.8L/per batch

Based in an 80 gal batch:

80 gal of oil

16 gal of oil

3055g of KOH

247ml of $H_2SO_4$

80 gal x 3.78L= 302.4L of oil/per batch

16 gal x 3.78L= 60.5L of methanol/per batch

Based on a 100 gal batch:

100 gal of oil

20 gal of methanol

4700g of KOH

380mL of $H_2SO_4$

378L of oil/per 100 gal batch

75.6L methanol/per 100 gal batch

*Note any amount of oil is an estimate-the oil possibly needs to be drained of chucks of food. Should the amount be lower than 50 gals the amounts of methanol and catalysis needs to be changed. This is why most biodiesel processors put their oil in a filtering drum with amounts around 55 gals of oil. Filtering causes the loss several gallons in this step, leaving them with just about 50 gals of filtered oil to be processed give or take a few gallons. Also our oil that we will be using from the dining hall will be filtered very well so we might not need to do this step, but should be done as a precaution.

Methanol, like the catalysts, absorbs moisture from the air. Be sure that when working with methanol, to work quickly and seal the containers as soon as possible. The less moisture you have in your process, the less soaps you'll end up with in the finished product.

The amount of methanol required for the process is about 20% of the volume of the oil to be processed. Some brewers use 21% or even 22% to be sure there is enough. To be clear of how much methanol to use, if you were to process 100 liters of vegetable oil, the process would require 20 liters of methanol for 20%.

Once the methanol has been measured out, the catalyst now needs to be mixed into a solution with the methanol this step is the premixing of catalysis. When mixing chosen catalysis and methanol it gives off large amounts of heat, this is why it is suggested to add half of the catalysis mix well and add the other half when the heat is minimized. In small amounts like this lab scale experiment the heat given off in the premixing step seems to not be much of a factor.

## Basic step by step:

Process on the lab scale

- Weight out approximately 85g of both the canola and soybean oils in separate beakers, and then transfer the oils into your 250mL round bottom flask. The oils are much more viscous than water so pour out as much oil from the beakers as possible.

- Weight out 15g of Methanol (MeOH) in a spate beaker. Add the 15g of methanol to 170g of mixed oil in round bottom flask and 0.2mL of $H_2SO_4$. The 0.2mL is nearly impossible to measure out, therefore just add two drops of sulfuric acid to the round bottom flask.
- Place stirring device in the flask
- Put the flask in the heated oil bath which is heated at 65C; make sure the flask is tightly clamped. Because the boiling point of methanol is nearly 65C-we do not want this to evaporate-so keep the temperature constant and place a condenser on the flask as well. Start the stirring device at high speed because the oils and methanol do not mix well. The heating and reaction will take 50mins.
- While this is mixing weight another 15g of methanol in the same beaker as before and weigh out 2.35g of KOH on the weigh boat.
- Put the KOH in the methanol to dissolve-this is an exothermic reaction that gives off large amounts of heat when in greater quantities. This is why we do premixing of catalysis in methanol.
- When 50 minutes is up the first reaction should be complete. Let the flask cool. When cooled add premixed catalysis KOH that has been dissolved in the 15g of methanol to the flask.
- Again heat in oil bath at 65C for 50mins. Let it cool.
- Add cooled mixture to a separatory funnel to drain off lower phase (waste glycerin). When mixture is added to the funnel, you should wait until the phases are clearly visible before you start to drain and separate. The waste or glycerin will be the dark lower phase in this step. The glycerin should be drained in a 600mL beaker.
- Add approximately 50mL of deionized water and shake in the funnel. Be careful of this step make sure you hold the two ends where the oil can spill. Let it settle and drain off aqueous phase- repeat two or three more times-this removes any leftover impurities = higher quality biodiesel
- Drain the washed oil (biodiesel) into a breaker and place on a hot plate heated at 60C with a stirrer to evaporate unwanted water

## Testing Finished Biodiesel Product

Your oil can then be tested for quality via 27/3mL test. To be sure that the reaction went to completion and the oil has fully converted to usable biodiesel, testing should be done on a routine basis. The 27-3 test is a simple test to show whether the biodiesel reaction has gone to completion. To test, take 3 mL of biodiesel and then 27 mL of methanol. After shaking these two liquids together, fully reacted biodiesel will result in a clear solution immediately after shaking. If the biodiesel has not been fully reacted, the mixture remain cloudy for several minutes after being shaken and visible beads of oil will appear a few hours after being shaken.

*****Make note that this is a step by step process on a lab scale not using a biodiesel processor from a manufacturer

[1] National Renewable Energy Laboratory, Urban Waste Grease Resource Assessment, G. Wiltsee, 1998
[1] United States Department of Agriculture, Marketing Service Bulletin titled Per Capita Consumption of Fats & Oil in Food Products, 2000
[1] Environmental Protection Agency, Renewable Fuel Standards Program Regulatory Impact Analysis, released in February 2010 (http://www.extension.org/pages/Used_and_Waste_Oil_and_Grease_for_Biodiesel).