# Make Biodiesel from Vegetable Oil (Chemistry)

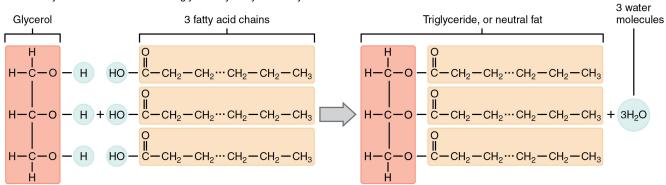
# How can vegetable oil be used as fuel?

## Background

Diesel engines such as locomotives, trucks, tractors, heavy construction equipment, and oil-heating systems rely on No. 2 diesel for power. Diesel is made from petroleum by distillation. A renewable substitute for fossil diesel is biodiesel, which can be easily made from vegetable oils (soybean oil, canola oil, etc.), animal fats, and waste grease through chemical reactions.

Biodiesel burns cleaner than its petroleum counterpart and is derived from entirely biological sources. It is completely miscible with petroleum diesel, allowing for blending and more stable storage at low temperatures. Biodiesel can be combusted in any diesel engine, without needing to modify the engine.

Vegetable oils are triglycerides and they have a standard structure. A molecule of any given vegetable oil consists of two parts, a glycerol backbone and three distinct fatty acid chains that stem from the glycerol. (Figure 1) Biodiesel is produced using the chemical process, transesterification. Transesterification occurs when one type of ester, a vegetable oil molecule in this case, is changed into another (Figure 2). Today, we will be making biodiesel using this process by employing the following reagents; vegetable oil and methanol (a single carbon alcohol). We will also use a base catalyst, potassium hydroxide or sodium hydroxide, to speed up the reaction. The combination of catalyst and methanol is called methoxide. The end product is a combination of biodiesel, unreacted methanol, base, glycerin, and soap.



Three fatty acid chains are bound to glycerol by dehydration synthesis.

Figure 1: http://cnx.org/content/m46008/latest/220\_Triglycerides-01.jpg

$R_1 - COO - CH2$		Catalyst	$R_1 - COO - CH_3$	HO — CH2
			D 000 01	
R <sub>2</sub> – COO – CH	+ 3 CH₃ OH		$R_2 - COO - CH_3$	+ HO-CH
$R_3 - COO - CH_2$			$R_3 - COO - CH_3$	HO – CH <sub>2</sub>
				_
Triglyceride	Methanol		Methyl esters	Glycerol

# R 1, R 2, R 3 = Hydrocarbon chain of 15 to 21 carbon atoms

Figure 2: https://commons.wikimedia.org/wiki/File:Transesterification\_reaction.pn



# Making Biodiesel from Soybeans

#### **Materials for Class**

250mL graduated cylinder 100mL graduated cylinder Scale or balance KOH(flake or pellet)

### **Group Materials**

250 mL separatory funnel w/ stopper
~125 mL vegetable oil (soy oil is preferred)
~0.5gKOH
50 mL Methanol
Ringstand and ring
Ball Jar or Mason Jar with lid (An Erlenmeyer flask can be substituted.)
pH paper

Students should also utilize a lab notebook to record observations, take notes, and record data so as to be able to reflect on mistakes or miscalculations.

#### **Initial Calculations and Preparations**

You will be responsible for determining the amounts of catalyst/reagent you will need as well as the physical mixing of the two. The ratio of reactants follows a very simple formula:

1 L of vegetable oil + 0.2 L methanol + 4.9 g KOH 1 L biodiesel + 0.2 L glycerin + salts We will be using 125 mL of vegetable oil. Use the space below to calculate the amount of reagent and catalyst

you will need.

125 mL vegetable oil	mL methanol	g KOH Procedure
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#### Day 1

- 1. Under a fume hood, measure out the calculated volume of methanol required into a 100 mL graduated cylinder. Place the methanol in your ball jar and seal tightly.
- 2. Using a balance, mass out the calculated amount of KOH required and **immediately** replace the lid to the KOH container. Do all of this **quickly** as KOH is hygroscopic, meaning that it absorbs moisture from the atmosphere and thus distort your mass reading. Place the KOH in the ball jar with the methanol. Shake vigorously until no KOH remains (this may take some time as KOH can be a stubborn dissolver). Congratulations, you've made methoxide.

#### In your notebook record the following observations.

I.a. Approximately how long did it take for all the KOH to dissolve?

I.b. Did you notice a change in temperature when shaking the jar? Why might that be?

I.c. Did any methanol accidentally spill when transferring containers? If so, what did you notice about the liquid? Did it evaporate quickly?

3. Using the class 250 mL graduated cylinder, obtain 125 mL of vegetable oil. Add the vegetable oil to your Ball jar and shake for 2 minutes. After two minutes take your Ball jar and 250 mL separatory funnel to the hood and transfer the contents. MAKE SURE THE VALVE ON THE FUNNEL IS CLOSED AND THE FUNNEL IS DRY!!! Shake for another 8 minutes, for a total of 10 minutes.

## In your notebook record the following observations.

II.a. Immediately upon adding the methoxide, what did you notice about the vegetable oil?

- II.b. Was there any change in color?
- II.c. What did the solution look like after you began shaking?
- II.d. If you paused shaking for a moment, what was the characteristic of the solution?



## Making Biodiesel from Soybeans

4. Place your separatory funnel in the ring stand and let it sit overnight. Step 5 will take place during the next class day. Congratulations, you've just made biodiesel!

#### Day 2

- 5. Before moving on to step 6, record your observations of your sample. Specifically, consider what changed from the previous class to now?
- 6. When the separation is complete, the top layer will be fairly clear. At this point, discharge the bottom glycerin layer into a 50 ml graduated cylinder and record the volume: \_\_\_\_\_ mL. Store the glycerin in a corked 150 ml Erlenmeyer flask for further use or soap making. (Your instructor may want to store all of the classroom glycerin in one container.)

Note: Crude biodiesel contains impurities such as soap, incompletely transesterified glycerides, and methanol and must be cleaned/washed prior to use.

#### Wash and Dry Biodiesel

- 7. Add 50 mL tap water to the raw biodiesel in the separation funnel. Mix the water with biodiesel by gently rolling biodiesel and water back and forth for 5 minutes. Wait for the mixture to separate into two layers, and discharge the bottom "soapy" layer. Using the pH paper, test the pH of the "soapy" layer. Record in your observations.
- 8. Wait for the mixture to separate into two layers (about 10 minutes), then discharge the bottom "soapy" layer.
- 9. Using the pH paper, test the pH of the soapy layer. Record your observations.
- 10. Repeat the washing procedure 3-4 times until the bottom water layer becomes clear and there is a sharp boundary between the water and the biodiesel layers.
- 11.Repeat the pH test with each "soapy" layer. Record your observations.
- 12. After removing the last water wash, remove the biodiesel and place into a 250 mL beaker.
- 13.Heat the beaker in a microwave to 100°C. Use a glove to remove the beaker of oil from the microwave. Let the sample sit uncovered overnight. The remaining water will be boiled off or evaporate.

#### Day 3

- 14. Measure the quantity of biodiesel in a graduated cylinder: \_\_\_\_\_ mL
- 15.Calculate the % yield of your biodiesel production.

Volume of glycerol generated. \_\_\_\_\_ml glycerol Volume of biodiesel generated \_\_\_\_\_ml biodiesel

Calculate the % yield of biodiesel: %Yield = volume biodiesel/(volume biodiesel +volume glycerine) x 100%

#### Analysis Questions

- 1. When producing biodiesel, what are the reactants and what are the products? What was the catalyst? Why is a catalyst necessary for the reaction?
- 2. How did the clarity of the solution change as you mixed the sample?
- 3. Take a moment to clarify your observations about what happened to your sample overnight? Consider

color, layers, apparent viscosity, etc.)

- 4. Why did the two substances separate?
- 5. How did the pH of the "soapy" layers change with each subsequent washing? If you saw a change,

why would that occur?

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