Biofuels from plant oils

Focus question	How do plant oils become fuel?
Vocabulary	Transesterification, biodiesel, biodiesel, petroleum diesel, miscible, glycerin, catalyst, ester, fatty acid chains, glycerol, hygroscopic

Diesel engines such as trucks, tractors, and heavy motors rely on No. 2 diesel for power. Diesel is commonly made by petroleum distillation. Renewable substitutes for petroleum diesel are growing in popularity. These biofuels can be easily made from corn, soy and other plant oils, animal fats, and waste grease through chemical reactions.

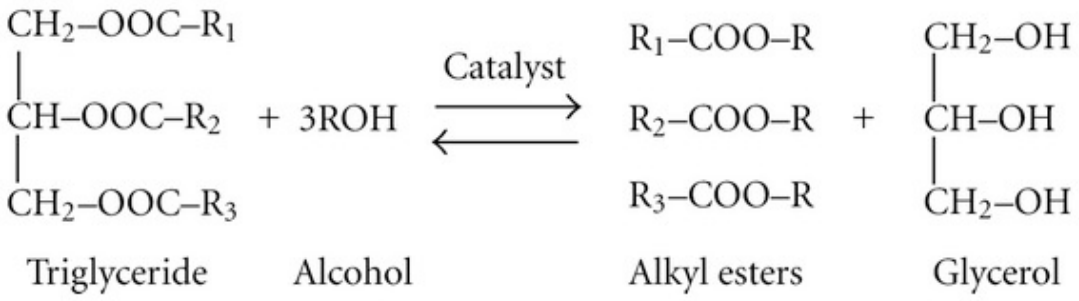
Biofuels are liquid or gaseous fuels commonly used for transportation. These are referred to by the United States Department of Agriculture as “drop-in fuels”, requiring no engine modification when blended with refined petroleum products. Biofuels derived from plant materials such as corn and soy are among the most rapidly-growing renewable energy technologies. While this fuel has less energy per unit volume than traditional diesel fuel, it results in much less air pollution due to its higher oxygen content and lack of "aromatic compounds” and sulfur.

Biofuels from plant oils include biodiesel and renewable diesel (eia.gov/energyexplained/biofuels/).

- **Biodiesel** is the term used for fuel made from soybean oil and is typically blended with petroleum diesel.
- **Renewable diesel** refers to biofuel mainly from corn oil and can be used as a replacement fuel with no blending.

Plant oils are triglycerides that 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. Biodiesel is produced using the chemical process known as transesterification. Transesterification occurs when one type of ester, an oil molecule in this case, exchanges an R group with an alcohol.

Making biodiesel in the classroom requires methanol. We will also use a catalyst, potassium hydroxide, to speed up the reaction. The combination of catalyst and methanol is called methoxide. The end product is a combination of biomass-based diesel, unreactive methanol, glycerin, and soap.



Koohikamali, Sara & Tan, Chin & Ling, Tau. (2012). Optimization of Sunflower Oil Transesterification Process Using Sodium Methoxide. TheScientificWorldJournal. 2012. 475027. 10.1100/2012/475027.

The products separate into two layers with the biodiesel on top. The biodiesel is removed and washed, then it is ready for product evaluation. In industrial applications, the oil is then refined through a process that we cannot replicate in the lab. Biodiesel undergoes a refinery process similar to petroleum diesel.

Renewable diesel results in biodiesel that is stable at low temperatures using a different process that creates a reaction with the feedstock and hydrogen called hydrotreating. Gasification, pyrolysis, and other biochemical and thermochemical technologies can also be used to create renewable diesel. Renewable diesel has lower production volumes than biodiesel in the U.S.

Materials

- Methanol or HEET (yellow bottle)
- Sodium hydroxide or Potassium hydroxide
- Glass jar/lid
- 200 mL beaker
- Magnetic stir bar
- Hot plate/stir option
- Separatory funnel, 250 mL or pint-sized jar with lid
- Ring stand with ring (not needed if using jars)
- Graduated cylinder
- Serological pump and pipettes
- Distilled water
- Weigh boats
- Scales
- Corn oil
- Vegetable (soybean) oil
- Other oils, if desired
- Disposable pipettes

Materials for testing

- Pop pop steam boats (1 per group)
- Lighter or matches
- Disposable pipettes
- Clear test tube
- Liquid food coloring
- Microtubes
- Access to refrigerator/freezer
- Balance/scale
- pH test strips or pH meter

Procedure

Part 1: Making biodiesel (Day 1)

1. Under a fume hood, measure out 30 mL of methanol or HEET and add to glass jar, then seal jar quickly.
2. Weigh out 0.75 g of KOH (potassium hydroxide) and quickly add it to the jar of methanol. Seal jar immediately and shake to dissolve. Make sure to recap the KOH because it is hygroscopic. Your mixture is now called methoxide.
3. At the lab station, in a clean beaker, warm 75 mL of oil sample to 50° C.
4. Add warmed oil sample to methoxide mixture in jar.
5. Add magnetic stir bar to the jar; loosely place lid back on jar; set stir to high speed and stir for 15 minutes. (Alternatively, tighten the lid, then shake vigorously for 15 minutes.)
6. Allow to sit for 24 hours.

Data for Part 1 (Day 1): visual observations from part 1 of Making biodiesel

1. Immediately upon adding the methoxide, what did you notice about the oil? Was there a change in the color of the sample?

2. What did the solution look like after it began stirring?

Part 2 (Day 2)

Data from Washing biodiesel

1. Now that the biodiesel has rested for 24 hours, describe your sample.

2. Record the following characteristics of your biodiesel sample: color, consistency, and odor.

Initial removal of glycerin

1. Drain the glycerin from the biodiesel sample by removing the bottom layer. You may release the glycerin from the separatory funnel into a beaker or jar. If your sample is in a jar, remove the glycerin by using disposable pipettes or a pipette pump and serological pipette. Empty the glycerin into a graduated cylinder and record the amount of glycerin retrieved from sample. *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

1. Using a serological pipette, slowly add a total of 20 mL distilled water down the side of the separatory funnel or jar.
2. Pick up the jar and gently rock back and forth for five minutes to wash the biodiesel. (Do not shake!)
3. Let jar stand and wait 10 minutes for the mixture to separate into two layers. Pipette off the bottom "soapy" layer. Remove soap/glycerin waste into a waste flask or jar.
4. Using pH paper or probe, test the pH of the "soapy" layer and record below.
5. Repeat washing procedure steps 1–4 for a second washing.
6. Allow to settle overnight.
7. Measure the quantity of biodiesel in a graduated cylinder and record.
8. Calculate the percentage yield of your biodiesel production using the following equation:

$$\% \text{ yield} = [\text{volume biodiesel} / (\text{volume biodiesel} + \text{volume glycerin})] \times 100\%$$

Data

Oil	pH	Color	Odor	% Yield
Corn				
Soy				

Reflection

1. What tests might you want to perform on your diesel fuel? (Think about various climate zones in the U.S.)

2. Is your biodiesel ready to put into an engine? If not, what other processes might be necessary before it is ready?

Testing biodiesel

Various tests can be performed on the biodiesel to compare different fuels from different plant oils.

Using pop pop steam boats

1. Remove the spoon/candle holder from the boat. Prepare the boat for running by using a disposable pipette to add water to the pipes on the underside of the boat. This water is necessary for propulsion of the boat through the water.
2. Remove the candle from the spoon or holder. You will need a wick in a holder or a piece of string to light.
3. Add 1 ml of your fuel into the spoon/holder. Add a wick.

4. Light the wick, then insert the spoon/holder into the hull of the boat under the tin plate. *Be careful not to push it under the plate too far or the flame will not stay lit.*
5. It may take a few moments for the boat to heat enough to make steam that will propel the boat in the water. You will hear the pop pop that indicates the boat will move.
6. Time how long the fuel burns or set up races against other groups to test speed.

Using food coloring

This qualitative test measures the amount of water in your sample.

1. Place 3 ml of a sample into a clear test tube.
2. Put one drop of liquid food coloring into the sample of biodiesel.
3. Observe the behavior of the drop. Does the color drop diffuse through the sample?
4. The less it diffuses into the mixture the less water there is in the sample.

Using temperature

Cold flow test

1. Place a 1 mL sample of fuel into each of three clear microtubes.
2. Place one sample in a refrigerator for fifteen minutes
3. Place another sample in the freezer for fifteen minutes.
4. Keep the third sample at room temperature to use as a control.
5. Remove the samples from the refrigerator and freezer. Invert the tube to see if the biodiesel flows.

If the sample does not flow, brainstorm additives that could be used to keep the fuel from freezing without affecting performance.

Using density

1. Weigh a microtube and record weight.
2. Add 1 mL of biodiesel to the microtube and re-weigh sample. Subtract the weight of the empty microtube. This will give the mass of 1 mL of biodiesel.
3. Divide mass of the biodiesel (g) by volume of biodiesel (1mL) to calculate density of the biodiesel (g/mL).

Using pH

1. Use the pH test strips to determine the pH of each biodiesel sample.

Rubric for self-assessment

Skill	Yes	No	Unsure
I can explain the difference between petroleum diesel, biodiesel, and biodiesel.			
I can explain the process of transesterification.			
I can perform at least three procedures to test the quality of biodiesel.			