

Synthesizing a Biofuel

Student Activity

What is this activity about?

Kristala L. Jones Prather enjoys teaching at MIT where she researches genetically engineered microorganisms to produce medicines, biofuels, and other important substances. In this activity, you will make a biofuel from household vegetable oil by converting triglycerides into long chain methyl esters. Bioenergy is derived from biofuels and provides approximately 10% of the total world energy with biomass providing the main source of energy in developing countries. New technologies are providing a wide variety of biofuels that are used for cooking, heating, and transportation. Renewable biofuels reduce dependence on imported oil and recycle the carbon dioxide that is produced in their combustion for photosynthesis.

What Materials do I need?

Chemical Splash goggles	Hot Hands™ or Thermal Gloves
Chemical-resistant apron	Thermometer
Chemical-resistant rubber gloves	2-100 mL graduated cylinders
Balance	100 mL beaker
Weighing paper	250 mL beaker
Spatula	Wide mouth jar with lid (at least 200 mL)
Hot plate	Canola oil
Glass stirring rod	Potassium hydroxide
Plastic pipet	Methanol (100%)

What safety precautions and disposal actions must I take?

- 1) The methanol and potassium hydroxide are both skin and eye irritants. Wear chemical splash goggles, chemical-resistant apron, and chemical resistant rubber gloves during the entire procedure.
- 2) Label all containers with their contents.
- 3) Take care in working with the hot oil to prevent burns.
- 3) Follow your teacher's directions for proper storing and/or disposal of chemicals.

What procedure must I follow?

Day 1: Mixing the raw ingredients

- 1) Put on chemical splash goggles, chemical-resistant apron, and chemical resistant rubber gloves. Both the methanol and potassium hydroxide are skin and eye irritants.
- 2) Use a graduated cylinder to measure 20 mL methanol. Pour into the 100 mL beaker.
- 3) Mass out 0.70 grams KOH and add to the beaker of methanol. Gently stir to dissolve. This may take 10 minutes. Be careful, this solution is caustic and dissolving of KOH in methanol is exothermic.
- 4) Use a graduated cylinder to measure 100 mL of canola oil and pour into the 250 mL beaker.

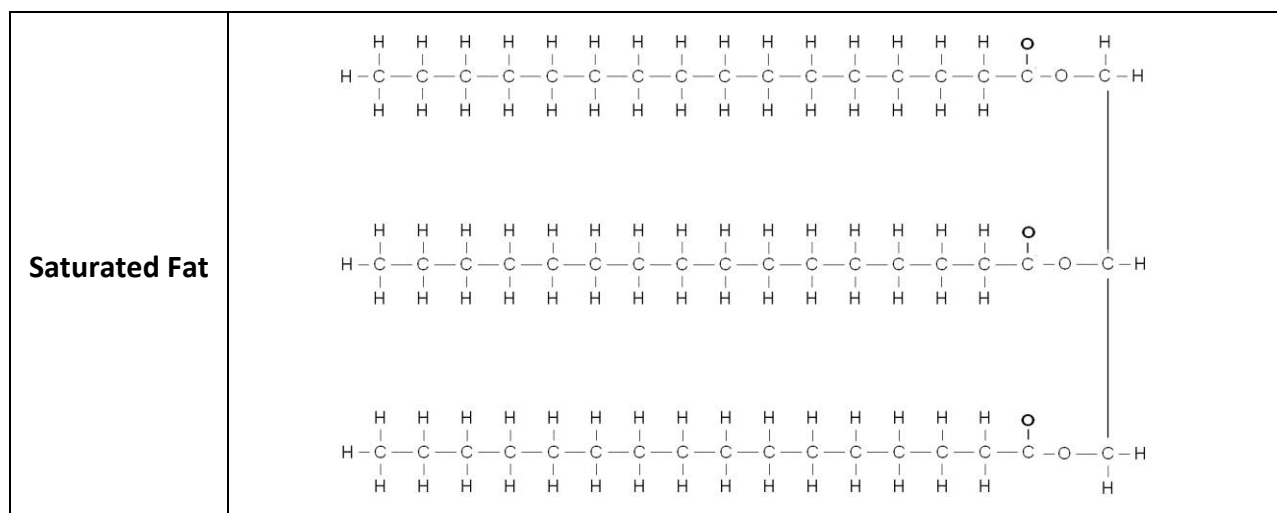
- 5) Place the beaker of oil on a hot plate. Monitor the temperature with the thermometer. When the temperature reaches 120°C, remove the beaker and turn the hot plate off.
Caution: keep the methanol solution away from the hot plate.
- 6) Carefully pour the hot oil into the jar. Slowly add the methanol/KOH solution. Put the lid on the jar and shake the mixture for 15 minutes. Allow the mixture to sit for a minute. Loosen the lid slowly to vent the mixture and then tighten the lid again.
- 7) You now have a cloudy mixture of biofuel and glycerin. Let this sit overnight.
- 8) Clean your workstation and wash your glassware.

Day 2: Separating the biodiesel from the glycerin.

- 1) Put on your safety glasses, apron, and rubber gloves.
- 2) Examine the contents of the jar. The brownish liquid at the bottom is glycerin and the colorless liquid on top is your biofuel.
- 3) Decant the biofuel (top layer) into a graduated cylinder. Record the volume of biofuel.
- 4) Leave the glycerin in the jar, replace the cap, and return the jar to your teacher. Be careful with the glycerin as it still contains methanol.
- 5) Follow your teacher's instructions for the storing/disposal of the biofuel. Follow your teacher's instructions for clean-up.

How is this activity related to my knowledge of science/chemistry?

Oil molecules contain three fatty acids bonded to a glycerol molecule. Some oil molecules are saturated (all carbons are single bonded); while others are unsaturated (some carbons are double bonded). When oil molecules react with methanol and a catalyst, biofuels are made. Biofuels are methyl esters.



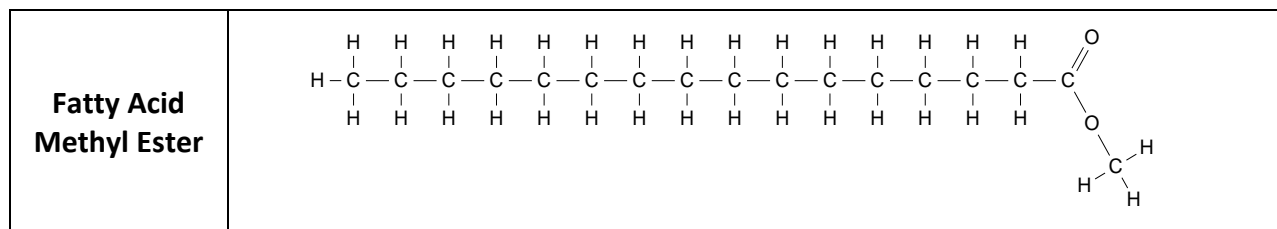


Table 1. A saturated oil and its methyl ester

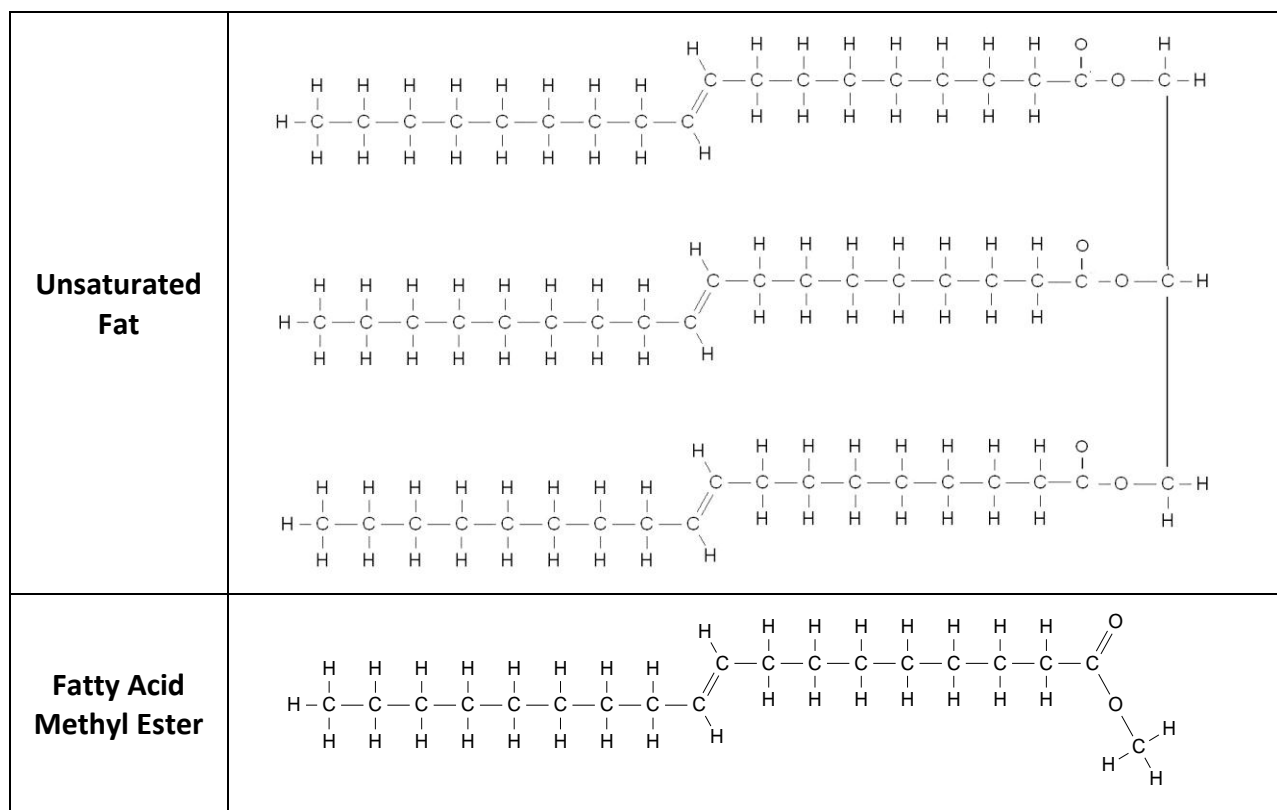


Table 2. An unsaturated oil and its methyl ester

The chemical structure of an oil affects its properties. Saturated oils have higher melting points because the molecules are straighter and therefore can pack together more tightly.

The quality and efficiency of biofuels are related to their chemical structures and there are various tests that chemists can perform on oils and biofuels to measure their performance:

Iodine Number – Oils that contain double bonds react with the oxygen in the air to produce peroxides. These peroxides can polymerize to produce materials that can “gum” and clog engine parts. The amount of iodine in grams per 100 mL of oil is the Iodine Number and it indicates the degree of double bonds. The higher the Iodine Number, the more double bonds are present; thus, there is a greater potential for engine gumming.

Cetane Number - This rates the burning properties of biofuels. Diesel engines run best with fuels that have Cetane Numbers between 40-55.

Cloud Point - One of the disadvantages of biofuels is that they tend to gel in colder weather. The Cloud Point identifies the lowest temperature at which the fuel can be used because it will not gel.

Properties of Some Oils and Their Esters

Type of Oil	Melting Temperature (°C)		Iodine Number	Cetane Number	Cloud Point (°C)
	Oil	Methyl Ester			
Palm Oil	30-38	14	44-58	65	8
Soybean Oil	-12	-10	125-140	53	2
Sunflower Oil	-18	-12	125-130	52	0
Lard	32-36	14	60-70	65	~12

Table 3. (Data from <http://journeytoforever.org>)

- 1) In this activity you used 100. mL of canola oil.
 - a) How many mL of biodiesel did you make?
 - b) What is your percent yield?
- 2) What is the formula of the methyl ester functional group?
- 3) Based on Melting Temperature and Iodine Number data, rank the oils from most saturated to least saturated.
- 4) Which oils(s) would not be practical for making biofuels in colder climates?
- 5) Which is the most efficient burning fuel? Is it saturated or unsaturated?
- 6) What is an advantage of using palm oil to make biofuel?
- 7) Name two disadvantages of using palm oil to make biofuel.
- 8) Explain which oil in Table 3 is the best for making biofuel.

How can I extend my learning with this activity? (Extensions)

- 1) Use sunflower, soybean, or another oil in place of canola oil and compare your percent yield.
- 2) Make biodiesel from used cooking oil.
- 3) Use the glycerin by-product to make soap. Be careful to remove the methanol first.

Synthesizing a Biofuel

Teacher's Guide

Concepts:

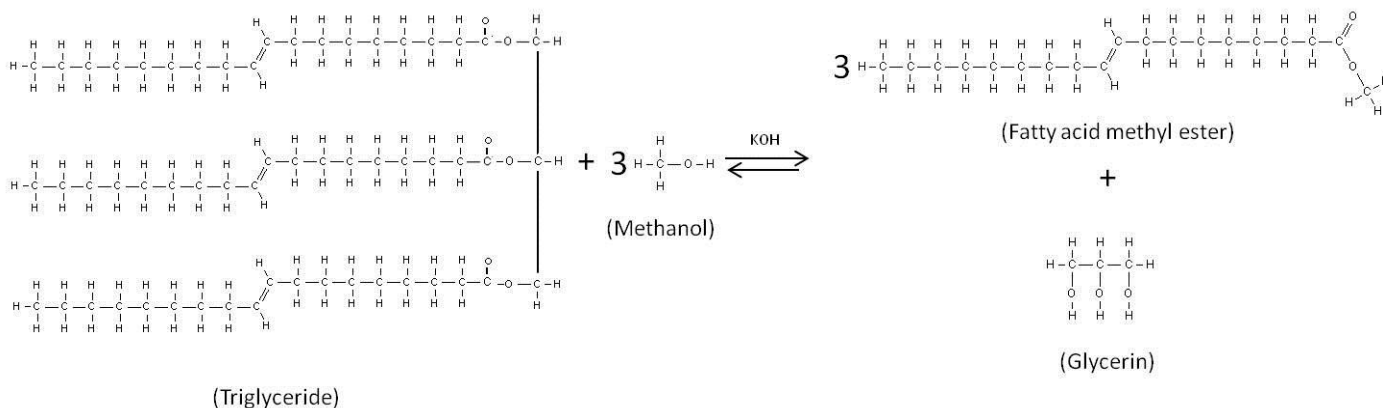
- Biofuels
- Transesterification
- Organic nomenclature

Background:

In this activity, students synthesize biodiesel from canola oil. They will then examine several properties of oils and their fatty acid methyl esters to determine which oil makes the best biofuel.

Chemistry:

During trans-esterification of a triglyceride, a basic catalyst breaks the fatty acids from glycerin. If a methanol contacts a fatty acid, they bond and form a methyl ester. The hydroxide ion from the KOH stabilizes the glycerin.



Materials:

Safety goggles
 Lab apron
 Rubber gloves
 Balance
 Weighing paper
 Spatula
 Hot plate
 Glass stirring rod
 Plastic pipet

Hot Hands™
 Thermometer
 2-100 mL graduated cylinders
 100 mL beaker
 250 mL beaker
 Wide mouth jar with lid (at least 200 mL)
 Canola oil
 Potassium hydroxide
 Methanol (100%)

Procedure:

The procedure for making a biofuel from canola oil can be viewed at <http://www.utahbio.com>

Soybean oil, sunflower oil, and others that can be purchased in grocery stores can be substituted for the canola oil.

0.56 g of NaOH can be substituted for the KOH.

Safety and Disposal:

Wear chemical splash goggles, chemical-resistant apron, and chemical resistant rubber gloves when working with the methanol, KOH, and glycerin by-product.

Care must be taken in working with the heated oil.

The biofuel made in this experiment is non-toxic, biodegradable, and safe to flush down the drain. It also has a high flash point, so it is safe to store in properly labeled containers.

The glycerin by-product from this activity is about 25% methanol and is basic. Check state and local regulations for disposal of the methanol product. You may be able to flush down the drain with large amounts of water.

If you choose to use the glycerin by-product to make soap, you will need to distill or evaporate the methanol. This must be done in a well-ventilated area.

How is this activity related to my knowledge of science/chemistry?**(Questions/Targeted Answers)**

- 1) In this activity you started out with 100. mL of canola oil.
 - a) How many mL of biodiesel did you make?
Example (75 mL)
 - b) What is your percent yield?
 $(75 \text{ mL}/100 \text{ mL}) \times 100\% = 75\% \text{ yield}$
- 2) What is the formula of the methyl ester functional group?
-COCH₃
- 3) Based on Melting Temperature and Iodine Number data, rank the oils from most saturated to least saturated.
Palm oil, Lard, (Sunflower oil and Soybean oil are about the same)
- 4) Which oils(s) would not be practical for making biofuels in colder climates?
Palm oil and lard have high Cloud Points.
- 5) Which is the most efficient burning fuel? Is it saturated or unsaturated?
Soybean oil and sunflower oil have Cetane Numbers of 52. Efficient fuels have cetane numbers between 40-55. These are unsaturated.
- 6) What is an advantage of using palm oil to make biofuel?
Low Iodine Number indicates low gumming of engine.
- 7) Name two disadvantages of using palm oil to make biofuel.

Not good in colder weather (Cloud Point) Cetane Number is above the optimal range.

8) Explain which oil in Table 3 the best for making biofuel.

*Soybean and Sunflower are good because of low Cloud Point and Cetane Number is good.
Lard and Palm oil are good because the Iodine Number indicates less gumming of engine parts.*

Extensions:

- 1) Use sunflower, soybean, or another oil in place of canola oil and compare your percent yield. The recipe used in this activity will work.
- 2) Make biodiesel from used cooking oil. Students get a chance to practice their titration and stoichiometry skills. Directions can be found at <http://www.utahbiodieselsupply.com/tutorialvideos.php#smallbatch4>.
- 3) Use the glycerin by-product to make soap. Be careful to remove the methanol first. Directions for making soap can be found at <http://www.home-made-biodiesel.com/biodiesel-soap.html>

References:

<http://www.utahbio.com>

<http://journeytoforever.org>

<http://www.greeningschools.org/docs/BiodieselUsingRenewableResources.pdf>

Wagner, E.P.; Koehle, M.A.; Moyle, T.M; Lambert, P.D. *J. Chem Educ.* **2010**, 87, 711-712