Lab: Soap or Fuel?

FOR THE TEACHER

Summary
In this lab, students will transform vegetable oil into a soft soap and into biodiesel fuel. The two reactions emphasize that the products of a chemical reaction are under the control of the chemist. By noting the relationship of the reaction product to the reactants, students will gain a deeper understanding of the law of conservation of matter.

Grade Level
High School

NGSS Alignment
This activity will help prepare your students to meet the performance expectations in the following standards:

- **HS-PS2-6**: Communicate scientific and technical information about why the molecular-level structure is important in the functioning of designed materials
- **Scientific and Engineering Practices**:
  - Asking Questions and Defining Problems

Objectives
By the end of this lab, students should be able to
- Recognize carboxylic acid and carboxylate ester functional groups.
- Write balanced chemical equations for saponification and transesterification of a generic fatty acid ester.

Chemistry Topics
This lab supports students’ understanding of
- Chemical Reactions
- Renewable Energy
- Organic Chemistry
- Molecular Structure
- Density

Time
**Teacher Preparation:** 30 minutes
**Lesson:** Two 45-50 minute class periods

Materials
- test tubes, 150 mm (2 per group)
- hotplate (1 per group)
- thermometer (1 per group)
- glass stirring rods (1-2 per group)
- graduated cylinder, 10 mL (1 per group)
- vegetable oil (25 mL per group)
- sodium hydroxide (30% solution in water, 5 mL per group)
- sodium hydroxide, pellet (1-2 pellets per group)
- methanol (3-5 mL per group)
- water
- aluminum foil, 5 cm - 10 cm (per group)
- beaker (400-mL) (1 per group)
- beaker (50-mL) (1 per group)
• pH paper

Safety
• Always wear safety goggles when handling chemicals in the lab.
• Students should wash their hands thoroughly before leaving the lab.
• When students complete the lab, instruct them how to clean up their materials and dispose of any chemicals.
• Exercise caution when using a heat source. Hot plates should be turned off and unplugged as soon as they are no longer needed.
• Sodium hydroxide is used in this laboratory experiment; note that it is corrosive and damaging to skin and mucous membranes. If any solution gets on students’ skin, they should immediately alert you and thoroughly flush their skin with water.
• Methanol is used in this laboratory experiment; note that it is flammable and volatile. Parts of this experiment are to be done in a fume hood. Keep it in a sealed container in the fume hood and away from heat sources.
• Vegetable oil is used in this laboratory experiment, please note it may make surfaces slippery.
• Wear gloves as required during this experiment.

Teacher Notes

Background
• Fats have been used as raw materials for many purposes over the years. Their natural lubricity suggests their use as lubricants. The presence of long hydrocarbon chains suggests their use as fuels in combustion. Animal and vegetable oils have long been used as lamp fuel, for example. Rudolph Diesel used peanut oil to fuel the original diesel engine. Simple modifications to the molecule make it more suitable as fuel by reducing its viscosity and increasing its volatility. Alternatively, the molecule can be made amphiphilic (simultaneously hydrophilic and hydrophobic) by modifying the chemical group at one end of the molecule. This modification is the basis for making soap from fats and oils: the nonpolar hydrocarbon portion of the molecule dissolves greasy, oily substances and the polar, ionic portion of the molecule dissolves in water to suspend the dirt.

• In this experiment students will use vegetable oil as a reactant to carry out two transformations: saponification and transesterification. In saponification (literally, soap-making) students will combine vegetable oil with sodium hydroxide to convert the fatty acid esters into carboxylic acid salts, or soaps as shown schematically in Figure 1. Note that sodium hydroxide is a reactant in this transformation and is therefore consumed. This is basically (pun intended) the same reaction carried out for hundreds of years before the large-scale manufacture of soap.

\[
\begin{align*}
\text{CH}_2\text{O} & \quad \text{C(\text{CH}_2)_{14}\text{CH}_3} \\
\text{CH} & \quad \text{C(\text{CH}_2)_{14}\text{CH}_3} \\
\text{CH}_2\text{O} & \quad \text{C(\text{CH}_2)_{14}\text{CH}_3} \\
\end{align*}
\]

\[+ \quad 3\text{NaOH} \quad \rightarrow \quad \begin{align*}
\text{CH}_2\text{OH} \\
\text{CH}_2\text{OH} \\
\end{align*}
\]

\[+ \quad 3\text{CH}_3(\text{CH}_2)_{14}\text{CO}_2\text{Na} \]

Figure 1. Saponification of a Fatty Acid Ester with Sodium Hydroxide (By Leijonhufvud (Own work) [CC BY-SA 3.0 (http://creativecommons.org/licenses/by-sa/3.0)], via Wikimedia Commons)

• In the second reaction students will use the same vegetable oil as a reactant, but in this case they will convert the viscous glyceryl triester to the less viscous methyl ester. This conversion is important because unconverted vegetable oil must be heated to reduce its viscosity in order use
it as biodiesel fuel, while the methyl ester may be used as a direct replacement for conventional diesel fuel. This conversion is illustrated schematically in Figure 2. Note that the sodium hydroxide is used as a catalyst only; it is not consumed in the reaction and does not appear in the products.

\[
\begin{align*}
\text{CH}_2\text{-O-}\text{C}\text{(CH}_2\text{)}_{14}\text{CH}_3 + 3\text{CH}_3\text{OH} & \xrightarrow{\text{NaOH}} \text{CH}_2\text{-OH} \quad \text{CH}_3\text{-OH} \quad + 3\text{CH}_3\text{(CH}_2\text{)}_{14}\text{CO-CH}_3 \\
\end{align*}
\]

**Figure 2. Transesterification of a Fatty Acid Ester with Methanol catalyzed by Sodium Hydroxide**

**References**

**Tips**
- Depending on time and student ability, it may be useful to assign saponification to half the class and transesterification to the other half. Students in each group should be given the opportunity to share their observations with the other group. More advanced learners might explore the effects of type of oil used, amount or type of catalyst, reaction time and temperature, etc.
- Students should exclude water from the transesterification reaction as well as possible, as the presence of water will cause saponification in competition with transesterification. It is important that all equipment be dry prior to adding reactants and catalyst. Care should be taken not to splash any water from the water bath into the test tube. Good agitation is important for both reactions. Allowing both reactions to stand overnight will give the best and clearest results.

**Preparation**
- Prepare 30% (10 M) sodium hydroxide solution in advance of class by adding solid sodium hydroxide to water. Dissolving 40 g NaOH in about 93 mL of water will give 100 mL of solution, enough for about 20 groups of students. Allow sufficient time for the solution to cool before class.

**Cross-Disciplinary Extensions**

**Connect to Math**
Students can calculate the stoichiometry of each reaction. They will have to search online for an equivalent molecular weight for the particular vegetable oil they are using, or the teacher can provide it. Students should see that the amount of sodium hydroxide in the saponification reaction is approximately equal to (actually, a little less than) the amount of ester groups. For the esterification, students should see that methanol is in excess and the amount of sodium hydroxide is only catalytic.

**Connect to Reading and Writing**
Students could research the pros and cons of manufacturing biodiesel fuel from plant oils. Some questions to consider:
- What is the most appropriate source of oil to be converted to biodiesel?
- Are there any technical challenges preventing use of biodiesel to replace petroleum diesel? If so, what are they and how might they be overcome?
Connect to Social Studies
One of the by-products of making soap and biodiesel fuel is glycerol, also known as glycerin. This product was an essential reactant for the manufacture of explosives and munitions during World War II. Have students respond to the video and the short article at this link.

FOR THE STUDENT
Lesson

Soap or Fuel?

Background
The ability to transform one substance into another is at the heart of chemistry. In this experiment, you will transform vegetable oil into two substances with very different properties: soap and biodiesel fuel. Both transformations rely on chemical reactions of the ester functional group. In the soap making (or saponification) reaction, the ester is split into a carboxylic acid and an alcohol by addition of a molecule of water. The carboxylic acid is then further neutralized by base (sodium hydroxide) present in the reaction. In the second reaction, one alcohol group is exchanged for another in a process called transesterification. Both of these transformations are shown schematically below.

![Figure 3: Saponification of a Carboxylic Ester with Water and Base](image)

![Figure 4: Base-catalyzed Transesterification of a Carboxylic Ester with Methanol](image)

Safety
- Always wear safety goggles and lab apron when handling chemicals in the lab.
- Wash your hands thoroughly before leaving the lab.
- Follow the teacher’s instructions for cleanup of materials and disposal of chemicals.
- Exercise caution when using a heat source. Hot plates should be turned off and unplugged as soon as they are no longer needed.
- Sodium hydroxide is used in this laboratory experiment; note that it is corrosive and damaging to skin and mucous membranes. If any solution gets on your skin, immediately alert the teacher and thoroughly flush your skin with water.
- Methanol is used in this laboratory experiment; note that it is flammable and volatile. Parts of this experiment are to be done in a fume hood. Keep methanol in a sealed container in the fume hood and away from heat sources.
- Vegetable oil is used in this laboratory experiment; note it may make surfaces slippery.
- Wear gloves as required during this experiment.

Materials
- test tubes, 150 mm (2)
- hotplate
- thermometer
- glass stirring rods
- graduated cylinder, 10 mL
- vegetable oil
- sodium hydroxide (30% solution in water)
- methanol
- water
- aluminum foil, 5 cm × 10 cm
- beaker (400-mL)
- beaker (50-mL)
- pH paper

Procedure
1. **Read the entire procedure before obtaining any chemicals.**
2. Place about 300 mL of tap water into the 400-mL beaker and place it on the hotplate. Preheat the water to 60-65°C.
3. Add 10 mL of vegetable oil to each test tube. Clean the graduated cylinder.
4. Label one test tube “Saponification” and the other test tube “Biodiesel.”

**Saponification reaction**
1. Add 3.9 g (or 3.0 mL) of 30% sodium hydroxide to the test tube. DO NOT ALLOW THE SODIUM HYDROXIDE TO CONTACT YOUR SKIN. Wear gloves if you are handling the sodium hydroxide.
2. Observe the mixture and record your observations in your notebook. (Which solution is on the bottom of the test tube? Why?)
3. Stir the mixture with a glass stirring rod. Rinse the stirring rod with water when finished. (Why?)
4. Cover the test tube with a 5 cm square of aluminum foil.
5. Place the test tube in the warm water bath. Stir it occasionally over the remainder of the class period (the test tube should spend about 20-30 minutes in the warm water bath). Rinse the stirring rod after each agitation. Record any observations in your notebook.

**Transesterification Reaction**
1. Mass a small pellet of sodium hydroxide. Your target is 0.1 g, but anything less than 0.5 g will work. Record the mass of the pellet. Wear gloves if you are handling the sodium hydroxide.
2. In the fume hood, measure 2.5 mL of methanol into a clean, dry 10-mL graduated cylinder. Replace the cap to the methanol immediately. Do not remove the methanol from the fume hood.
3. Add the pellet to the graduated cylinder with the methanol. Swirl the graduated cylinder to dissolve the sodium hydroxide.
4. When the sodium hydroxide appears to be mostly dissolved, add it to the test tube labeled “Biodiesel.” Note: some degree of cloudiness may remain in the methanol. The undissolved material may be sodium carbonate from the reaction of sodium hydroxide with carbon dioxide from the air. It will not affect the experiment.
5. Stir the methanol/sodium hydroxide solution into the oil. Record any observations in your lab notebook. (Which layer is on the bottom of the test tube? Why?)
6. Cap the tube with a 5 cm square of aluminum foil and place the tube into the water bath. Continue to stir or swirl the mixture occasionally throughout the remainder of the class period (the test tube should spend about 20-30 minutes in the warm water bath).
7. Clean up your work area while the two reactions proceed. Rinse any equipment or laboratory surfaces that contacted sodium hydroxide thoroughly with water.
8. During the clean up, turn off the hotplate and allow it to cool. Transfer the water bath with the two test tubes to a storage location designated by your teacher. Be sure to label your experiment with your name, the date, and the contents of the tubes as instructed by your teacher.

**Analysis**
1. After allowing the products to stand overnight, record any observations about their appearance in your lab notebook.
2. Remove a small amount of the saponification product and place it against the pH paper. Record the pH (acid, basic, or neutral is sufficient) in your notebook. Place a small portion (perhaps 1/10 of the total amount) of the product into a small (50-mL) beaker and add some water. See if you can froth or foam the product by stirring it with the glass stirring rod.
3. Observe the layers in the biodiesel product. With a plastic pipet, remove the top layer to another test tube. How does the viscosity of this biodiesel product compare with the vegetable oil reactant? Record these observations in your notebook.
4. Add a small amount of water to your biodiesel product. See if you can froth or foam the mixture. Record your observations in your notebook.

**Post-Lab Questions**
Answer all of the questions that appear in the procedure for this experiment in complete sentences.
1. In the saponification reaction, the oil was the top layer; in the transesterification reaction, the oil was the bottom layer. Explain this observation using the concept of density.
2. After the transesterification reaction was complete, a dark reddish layer of glycerol (also known as glycerin) formed on the bottom of the test tube. What can you conclude about the density of glycerol based on this observation? Find the density of glycerol in a reference book or online. Is the reported value consistent with your explanation?
3. If sodium hydroxide contacts the skin, the skin begins to feel slippery or “soapy.” Given what you just learned about the reaction of sodium hydroxide with vegetable oil and what you know about human skin, explain this observation.