

Introduction

Soap-making is an ancient craft and one of the oldest known chemical reactions involving organic compounds. Soaps are sodium and potassium salts of fatty acids. They are prepared by reacting fats and oils with a strong base, such as sodium hydroxide or potassium hydroxide.

Concepts

- Soaps and soap-making
- Saponification
- Triglycerides
- Surfactants

Background

Legend has it that the word **soap** comes from Mount Sapo, a hill in Rome that was the site of animal sacrifice. According to the legend, animal fat and ashes washed down the mountain with the rain, producing sudsy river water that was used to wash clothes. It's an interesting story, but there is no factual evidence for this legend. The earliest written reference comes from the Roman historian Pliny the Elder in the first century CE. Pliny described the preparation of **sapo** from goat fat and wood ashes and attributed the invention to the Gauls, who used it for hair treatment rather than for bathing or cleaning. Historical references to soap may be found in ancient Babylonian and Egyptian artifacts dating as far back as 2500 BCE.

Soap-making is also associated with colonial America and pioneers on the American frontier. The soap was made by boiling fat with a concentrated solution of potash (potassium carbonate) extracted from wood ashes with hot water. Potassium carbonate solutions are **caustic**—strongly basic and irritating to the skin and eyes. Soap made in this way was likely to contain excess (unreacted) potassium carbonate and was therefore quite harsh, leaving the skin rough and dry. This frontier method of soap-making may appear primitive, but it is still used in almost the same form today to make both commercial and handmade soaps. The methods are safer, however, and soaps are milder because the starting materials are pure, the chemistry is well-understood, and the reactants can be mixed in the right ratio.

The process of making soap is called **saponification** and is one of the earliest examples of using organic chemistry to produce a man-made product. Saponification involves the reaction of **triglycerides**—natural fats and oils—with sodium or potassium hydroxide.

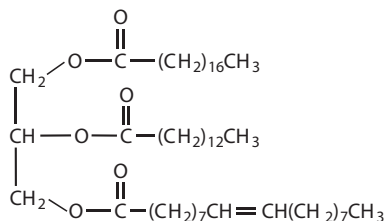
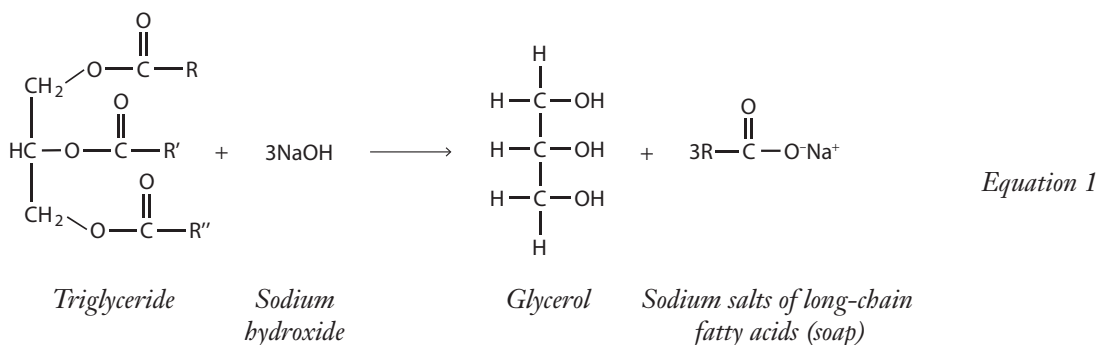


Figure 1. Structure of a Triglyceride

Triglycerides are **esters** containing three fatty acid groups attached via ester linkages to a glycerol backbone (Figure 1). The products of a saponification reaction are sodium or potassium salts of fatty acids and glycerol (Equation 1).



Most fats and oils contain a mixture of fatty acid residues of different chain lengths. The most common fatty acids have 12–18 carbon atoms and may be saturated or unsaturated. Unsaturated and polyunsaturated fatty acids contain one or more C=C double bonds, respectively, in their structures, while saturated fatty acids contain no C=C double bonds.

Soaps belong to a class of compounds called **surface-active agents** or **surfactants**, which also include detergents and emulsifying agents. A **surfactant** is defined as a compound that reduces surface tension when dissolved in water or in aqueous solutions. All surfactants have two basic features in common. One end of a surfactant molecule is usually a long, nonpolar hydrocarbon chain, resembling a “tail.” The hydrocarbon tail is said to be **hydrophobic** (water-fearing) because it tends to repel or exclude water and will not dissolve in water. The other end of a surfactant molecule is a small ionic or polar group that is hydrophilic (water-loving). The hydrophilic group will tend to be solvated or surrounded by water molecules and will dissolve in water. These two competing structural features give soaps and other surfactants their unique properties.

When dissolved in water, soaps and other surfactant molecules spontaneously self-associate to form spherical aggregates called **micelles** (Figure 2).

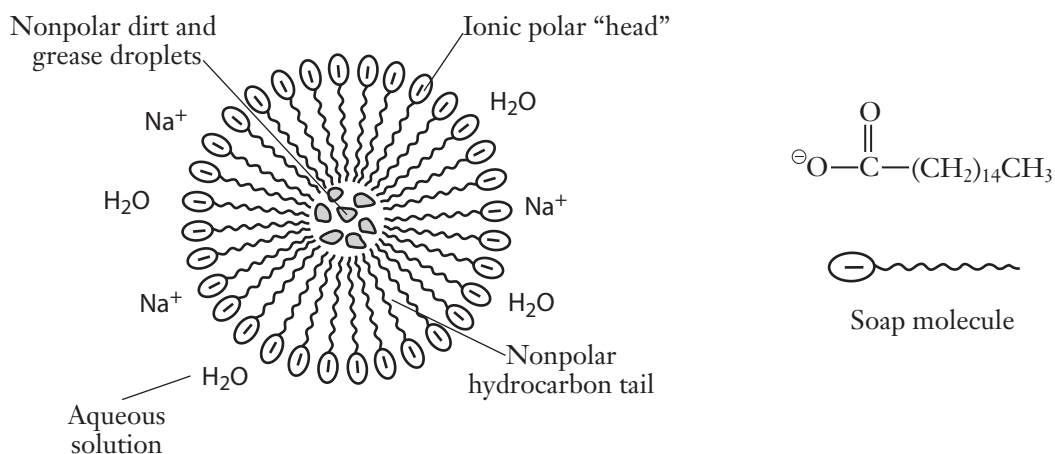


Figure 2. Structure and Properties of a Micelle

The nonpolar hydrocarbon tails in the soap molecules spontaneously arrange themselves toward the interior of the micelle, giving it a hydrophobic core that repels and thus excludes water. The ionic head groups are arranged on the outside surface of the micelle and are surrounded by water molecules. The ability of soap molecules to form micelles explains how and why soaps work. Dirt and grease are nonpolar, hydrophobic substances that are not soluble in water. If water alone were used for washing or cleaning, the hydrophobic dirt and grease molecules would not dissolve in the water. In soapy water, however, dirt and grease molecules become trapped or suspended within the hydrophobic core of a micelle. The soap thus disperses or breaks up the dirt particles and dissolves them, and the dirt-containing micelles are rinsed away in the wash. The formation of micelles is also related to the **emulsifying action** of soaps—their ability to form stable mixtures or suspensions of two or more immiscible liquids.

Experiment Overview

The purpose of this experiment is to make soap and study its properties. The soap will be prepared via saponification of a fat and oil with a solution of sodium hydroxide. The properties of the soap will then be investigated—its pH, texture, emulsifying action, and solubility in hard water.

Materials

Calcium chloride solution, CaCl_2 , 0.5 M, 1 mL	Graduated cylinder, 10-mL
Iron(III) chloride solution, FeCl_3 , 0.5 M, 1 mL	Hot plate
Magnesium chloride solution, MgCl_2 , 0.5 M, 1 mL	pH meter or pH paper
Olive oil or vegetable oil, 3 g	Spatula, metal
Sodium hydroxide solution, NaOH , 6 M, 5.8 mL	Stirring rod
Tallow or lard (solid fat), 7 g	Test tubes, small, 5
Water, distilled	Test tube rack
Balance, 0.1-g precision	Thermometer
Beakers, 50- and 250-mL	Wash bottle
Beral-type pipets, 4	Weighing dishes, small, 2

Safety Precautions

Sodium hydroxide solution causes severe skin burns and eye damage. Wear protective gloves and clothing and wash thoroughly after handling. Notify the instructor and clean up all spills immediately. Avoid contact of all chemicals with eyes and skin. Exercise care if you remove the prepared soap from the lab—it may be irritating to skin. Wear chemical splash goggles, chemical-resistant gloves, and a lab coat or chemical-resistant apron. Wash hands thoroughly with soap and water before leaving the lab.

Procedure

Preparation of Soap

1. Tare a 50-mL beaker on the electronic balance. Place about 7 g of tallow (solid fat) into the beaker using a metal spatula. The fat is messy to work with—try not to get any on the outside of the beaker.
2. Measure about 3 g of olive oil into the same 50-mL beaker.
3. Place the beaker on a hot plate **at the lowest setting** or inside a 250-mL beaker filled with hot tap water. Heat the contents of the beaker until the fat melts and the mixture of olive oil and fat forms a homogeneous solution.
4. Carefully remove the reaction beaker from the hot plate or warm water bath.
5. Measure 5.8 mL of 6 M sodium hydroxide solution into a 10-mL graduated cylinder and **carefully** add the sodium hydroxide solution to the melted fat and oil mixture. Heat gently on a hot plate or in a hot water bath to about 40°C. While heating, stir for 5 minutes.
6. After 5 minutes, carefully remove the reaction beaker and place into a larger, 250-mL beaker filled with cold water.
7. Continue stirring until the soap mixture gets thick—the product is ready to pour when the soap that drips back into the beaker from the stirring rod will trace a path (mark a trail) on the surface.
8. Label two small weighing dishes. Carefully pour the thickened soap solution from the reaction beaker into the weighing dishes.
9. Allow the soap to dry (cure) for several days. Describe the color, texture and appearance of the soap.

Properties of Soap

1. Add about 40 mL of distilled water to the leftover soap on the sides and bottom of the beaker. Gently scrape the soap into the water as needed.
2. Heat the beaker on a hot plate at a medium setting and stir gently until the leftover soap dissolves. Cool to room temperature. In the data table, describe the appearance of the soap solution.
3. Measure the pH of the soap solution using either a pH meter or pH paper. Record the pH of the solution.
4. Place three test tubes in a test tube rack and label them A–C. Pour about 3 mL of the soap solution into each test tube.
5. Add 5 drops of calcium chloride solution to test tube A, 5 drops of iron(III) chloride solution to test tube B, and 5 drops of magnesium chloride solution to test tube C. Swirl each test tube to mix the contents. Describe the color and appearance of the mixture in each test tube.
6. Place two clean test tubes in the test tube rack. Add 3 mL of distilled water to the first test tube and 3 mL of the soap solution to the second test tube.
7. Add one drop of olive oil to each test tube. Swirl or shake each test tube and then let sit for 5 minutes.
8. Describe the observations of this **emulsification** test in the data table.
9. When the soap bars are dry, measure and record the pH of the soap by placing a drop of water on pH paper and pressing the paper onto the top of the soap bar.

Pre-Laboratory Assignment

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Name: _____ Section: _____ Date: _____

Making Soap

1. Define the following terms: triglyceride, saponification, surfactant, micelle.
2. What is the principal safety hazard in this experiment? Explain why soap made by primitive methods was likely to be very harsh.
3. Olive oil has a **saponification value** of 190 mg KOH per gram. This means that it takes 190 mg of potassium hydroxide to react completely with 1 g of olive oil.
 - a. What is the mole ratio for the reaction of a triglyceride with KOH? (See Equation 1.)
 - b. Divide 0.190 g of KOH by its molar mass to calculate the number of moles of KOH that will react with 1 g of olive oil.
 - c. Use the answers to (a) and (b) to determine the number of moles corresponding to 1 g of olive oil. Divide 1 g by this number of moles to calculate the average molar mass (g/mole) of olive oil.

Laboratory Report

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Name: _____ Section: _____ Date: _____

Making Soap

Color, Texture and Appearance of Soap		
pH of Soap (after drying)		
pH and Appearance of Soap Solution		
Hard Water Tests	Ca²⁺ Ions	
	Fe³⁺ Ions	
	Mg²⁺ Ions	
Emulsification Test		

1. Compare the color, texture and appearance of the homemade soap versus your favorite brand of hand soap.

2. Is the homemade soap solution acidic or basic? Explain.

3. How do the reactions of the soap solution with calcium, iron and magnesium ions mimic what happens when soap is used in hard water? Write the equation, including the product formula, for the reaction of soap with calcium ions. Describe common observations around the home due to the reaction of soap with hard water.

- Compare the results of the emulsification test of olive oil with water and with the soap solution. Explain in terms of the ability of soap to form micelles.
- Explain why most soaps contain glycerol and describe the properties that glycerol adds to soap.
- Draw the general structure of a triglyceride containing both saturated and unsaturated fatty acids. Circle and label the glycerol backbone, an ester functional group, and an unsaturated fatty acid residue.
- Most homemade recipes for making soap use about 5% excess fat. Explain the benefits of using excess fat to make soap.

Supplementary Information

Table 1. Natural Fatty Acids

Name	Structure	Classification
Lauric Acid	$\text{CH}_3(\text{CH}_2)_{10}\text{CO}_2\text{H}$	Saturated
Myristic Acid	$\text{CH}_3(\text{CH}_2)_{12}\text{CO}_2\text{H}$	Saturated
Palmitic Acid	$\text{CH}_3(\text{CH}_2)_{14}\text{CO}_2\text{H}$	Saturated
Stearic Acid	$\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{H}$	Saturated
Oleic Acid	$\text{CH}_3(\text{CH}_2)_7\text{CH}=\text{CH}(\text{CH}_2)_7\text{CO}_2\text{H}$	Unsaturated
Linoleic Acid	$\text{CH}_3(\text{CH}_2)_4\text{CH}=\text{CHCH}_2\text{CH}=\text{CH}(\text{CH}_2)_7\text{CO}_2\text{H}$	Polyunsaturated
Linolenic Acid	$\text{CH}_3\text{CH}_2\text{CH}=\text{CHCH}_2\text{CH}=\text{CHCH}_2\text{CH}=\text{CH}(\text{CH}_2)_7\text{CO}_2\text{H}$	Polyunsaturated

Table 2. Composition of Triglycerides—Naturally Occurring Fats and Oils

Triglyceride	Percent Composition (g Fatty Acid/100 g Total Fatty Acids)*						
	Lauric	Myristic	Palmitic	Stearic	Oleic	Linoleic	Linolenic
Lard	–	1.3	28.3	11.9	47.5	6.0	–
Tallow (beef)	–	6.3	27.4	14.1	49.6	2.5	–
Coconut Oil	45.4	18.0	10.5	2.3	7.5	Trace	–
Corn Oil	–	1.4	10.2	3.0	49.6	34.3	–
Linseed Oil	–	–	6.3	2.5	19.0	24.1	47.4
Olive Oil	–	Trace	6.9	2.3	84.4	4.6	–
Palm Oil	–	1.4	40.1	5.5	42.7	10.3	–
Safflower Oil	6.8% saturated fatty acids				18.6	70.1	3.4

*Other fatty acids that may be present in amounts less than 1% include C_6 , C_8 , C_{10} , and C_{20} saturated fatty acids; C_{12} , C_{14} , and C_{16} monounsaturated acids; and C_{20} polyunsaturated acids.