# 8.2: Saponification Procedure

## Learning Objectives

- Understanding the chemical process of saponification
- Identifying the key components of soap
- Evaluating the properties of different soaps based on their ingredients
- Gaining practical laboratory skills

## Safety

- Oil and ethanol will be hot, and may splatter or possible catch fire.
- Keep a watch glass nearby to smother any flames.
- NaOH is caustic and can cause burns and eye damage.
- Wear goggles at all times.
- Wear disposable gloves.

## Introduction

Saponification of an ester occurs when a hydroxide attacks the carboxyl carbon breaking the ester linkage to produce a carboxylate and an alcohol:

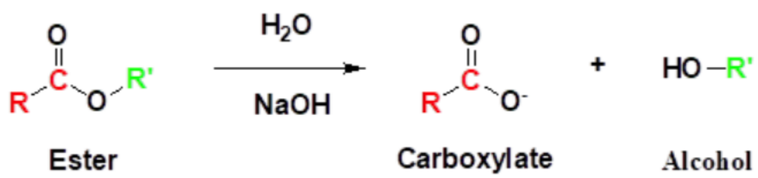


Figure 8.2.1. Saponification. Reaction of an ester with NaOH.

Using long chain acids in which the R group can have 14 to 24 carbon atoms attached to a glycerol molecule, is an example of a triglyceride called a fat as in the example below, to react with a strong base or “lye” to produce a soap through this saponification process.

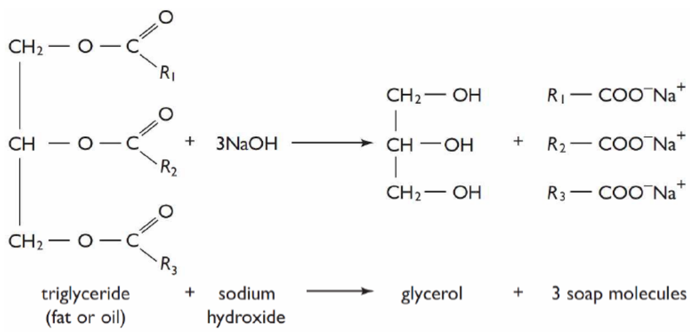


Figure 8.2.2. Saponification of a triglyceride.

Soap is the salt of a fatty acid. Sodium salts are known as “hard” salts found in most cake soaps. Potassium salts are the “soft” soaps used in shaving creams and liquid soaps.

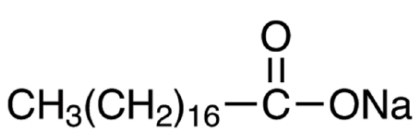

Figure 8.2.3. Sodium stearate is a typical soap.

Historically the preparation of soap began by using the extract of wood ashes as the base, and animal fats. The art of soap making was lost during the Middle Ages after the fall of the Roman Empire.

As you study the long chain soap molecule you can see that there is a nonpolar "hydrophobic" end and, the other end is the carboxylate ion which is ionic and "hydrophilic" or water loving. The nonpolar ends dissolve the grease or oils that accompany dirt, the water loving ends extend outside where they dissolve in water. The clusters that form from this action are called "micelles" .The small globules of oil and fat that are coated with soap molecules are pulled into the water layer and rinsed away.

One problem of using soaps is that the carboxylate end reacts with ions in the water such as $Ca^{2+}$ or $Mg^{2+}$, to form insoluble substances. (Recall those solubility rules?)

$$\underbrace{2\,C_{17}H_{35}COO^{-}Na^{+}}_{\text{soap}} + M^{2+} \rightarrow \underbrace{[C_{17}H_{35}COO^{-}]_{2}M^{2+}}_{\text{scum}} + 2\,Na^{+} \tag{8.2.1}$$

$M = Ca^{2+}\text{or } Mg^{2+}$

Another important test for soaps is the acidity. If a soap is too alkaline it can cause damage to skin and clothes. It the solution is too acidic the sodium salt is reverted back to the fatty acid, thus losing the cleansing action.

$$C_{17}H_{35}COO^{-}Na^{+} + H^{+} \rightleftharpoons C_{17}H_{35}COOH + Na^{+} \tag{8.2.2}$$

## Procedure:

### Part A: Making the Soap

**Read All the safety warnings above.**

1. Prepare a hot water bath using a 250 mL beaker.
2. Weigh *approximately* 5-10 g of fat or vegetable oil into a clean 150 mL beaker.
3. Using a graduated cylinder, add 15.0 mL of ethanol, $CH_3CH_2OH$, and stir using a glass rod.
4. Warm the beaker in the hot water bath until the lard dissolves, remove from heat.
5. **Carefully** add 15.0 mL of 30**%** NaOH, stirring constantly. ***Use care when pouring NaOH!***
6. Place the beaker in the water bath on a hot plate and heat to a gentle boil and stir continuously. Heat the mixture for about 30 minutes, or until saponification is complete and the solution is almost a solid. **Be careful of splattering, the mixture contains a strong base. Wear disposable gloves. Do not let the mixture overheat or char.**
7. Allow the mixture to cool for about 10 minutes, until it is slightly warm to touch.
8. Obtain 50 mL of a saturated NaCl solution in a 400-mL beaker. Pour the soap solution into this salt solution and stir. This process, known as "salting out," increases the density of the solution, which causes the solid soap curds to float on the surface.
9. Place the beaker in an ice bath until the solution equilibrates to the ice bath temperature.
10. Collecting the soap. Collect the solid soap using a Buchner funnel and filter paper. Wash the soap with two 10-mL portions of cold water. Pull air through the product to dry it further. Place the soap curds on a watch glass or in a small beaker and dry the soap until the next lab session. Use disposable, plastic gloves to handle the soap. **Handle with care: The soap may still contain NaOH, which can irritate the skin. Save the soap you prepare for the next part of this experiment**

### Part B: Testing Soap and Detergent:

> **Materials:** Test tubes, stoppers to fit, droppers, small beakers, 50- or 100-mL graduated cylinder, stirring rod, laboratory-prepared soap (from part A), commercial soap product, detergent, pH paper, oil, 1% $CaCl_2$, 1% $MgCl_2$, and 1% $FeCl_3$

Prepare solutions of the soap you made in part A, a commercial soap, and a detergent by dissolving about 1 gram of each in 50 mL of warm distilled water. If the soap is a liquid, use 20 drops instead of 1 gram.

**Record your observations in the Data Table below for steps 1-4.**

1. **pH test**: Place 10 mL of each soap solution into a separate test tube. Use 10 mL of water as a comparison. Label. Stir each solution with a stirring rod. Touch the stirring rod to pH paper. Determine the pH. Save tubes for part step .2
2. **Foam test**: Stopper each of the tubes from step 1 and shake for 10 seconds. The soap should form a layer of suds or foam. Record your observations. Save tubes for step 3.
3. **Reaction with oil**: Add 5 drops of oil to each test tube from step 2. Stopper and shake each one for 10 seconds. What happens to the oil layer? Record your observations. Compare the sudsy layer in each test tube to the sudsy layers in part step 2. Which substance dispersed (emulsified) the oil?

4. **Hard water test**: Place 5 mL of each soap solution in three separate test tubes. Add 20 drops of 1% $CaCl_2$ to the first sample, 20 drops of 1% $MgCl_2$ to the second tube, and 20 drops of 1% $FeCl_3$ to the third tube. Stopper each test tube and shake 10 seconds. Compare the foamy layer in each of the test tubes.