51 PREPARATION OF SOAP

Text Reference
Section 24.4

Time Required
45 minutes

Objectives
- Saponify vegetable oil to form soap.
- Observe some of the physical and chemical properties of the soap product, including pH and solubility in aqueous solutions of calcium, iron(III), and magnesium salts.
- Compare the properties of the soap product to the properties of a commercial laundry detergent and a commercial hand soap.

Advance Preparation

0.1M calcium chloride
Dissolve 1.5 g of $CaCl{\bullet}2H_2O$ in 90 mL of distilled water. Dilute to 100 mL.

saturated sodium chloride solution
The solubility of NaCl is approximately 360 g/L H_2O at 25°C. It is estimated that 2.5 L of solution will meet the needs of 15 lab pairs.

0.1M iron(III) chloride
Dissolve 2.7 g of $FeCl_3{\bullet}6H_2O$ in 80 mL of distilled water, and dilute to 100 mL.

0.1M magnesium chloride
Dissolve 2.0 g of $MgCl_2{\bullet}6H_2O$ in 80 mL of distilled water, and dilute to 100 mL.

50% (v/v) ethanol-water mixture
Mix 150 mL of 95% ethanol with 150 mL of distilled water.

PURPOSE

To prepare and isolate a soap by saponification of a triglyceride.

BACKGROUND

Have you ever considered that soap is one of society's major defenses against disease? The cleansing power of soap helps rinse away many disease-causing organisms, making your home and school healthier places than they otherwise would be. Soaps are alkali metal salts of carboxylic acids. They are generally produced by the reaction of metallic hydroxides with animal fats and vegetable oils. The major components of these fats and oils are triglycerides, esters of glycerol, and various fatty acids. Typically, soaps are made by hydrolyzing the ester bonds of triglycerides with solutions of sodium hydroxide. This soap-making reaction is called saponification (in Greek, *sapon* means "soap"). The products of the hydrolysis reaction are soap and glycerol.

In this experiment, you will saponify a vegetable oil and examine some properties of your product. You will compare the properties of the soap you make with the properties of a commercial detergent and a commercial hand soap.

MATERIALS (PER PAIR)

safety goggles and apron
2 250-mL beakers
2 50-mL beakers
10-mL graduated cylinder
gas burner
ring stand
ring support
wire gauze
glass stirring rod
50-mL graduated cylinder
spatula
3 medium test tubes
centigram balance
glass-marking pencil
test-tube rack
dropper pipet
3 cork stoppers
plastic wash bottle
scoopula
vegetable oil
50% (v/v) ethanol-water mixture
sodium hydroxide, NaOH [C] [T]
saturated sodium chloride solution, NaCl
paper towels
laundry detergent [I]
hand soap
wide-range indicator solution or wide-range test paper
0.1*M* calcium chloride, $CaCl_2$ [T]
0.1*M* iron(III) chloride, $FeCl_3$ [T] [I]
0.1*M* magnesium chloride, $MgCl_2$ [T]

Name _____ Date _____ Class _____

Other materials

Corn oil used for cooking works well and is inexpensive. Lard or other vegetable oils may also be used.

Most laundry detergents are sodium salts of sulfonic acids (RSO_3Na) or sulfates ($ROSO_3Na$). Sodium dodecyl sulfate ($CH_3(CH_2)_{11}OSO_3Na$) is a common detergent.

SAFETY FIRST!

In this lab, observe all precautions, especially the ones listed below. If you see a safety icon beside a step in the Procedure, refer to the list below for its meaning.

Caution: Wear your safety goggles. (All steps.)

Caution: Wear your lab apron. (All steps.)

Caution: Sodium hydroxide is corrosive and can cause severe burns. Never handle sodium hydroxide pellets with your fingers; use a small beaker and a scoopula. Solid sodium hydroxide will absorb water from the atmosphere; do not leave the container of sodium hydroxide open. (Steps 2, 3.)

Caution: Aqueous iron(III) chloride will stain clothes permanently and is irritating to the skin. Avoid contact with this material. (Step 9.)

Caution: Keep ethanol and ethanol-water mixtures away from open flames. (Step 3.)

Note: Return or dispose of all materials according to the instructions of your teacher. (Step 12.)

PROCEDURE

Part A. Preparation of Soap

1. Pour 5 mL (5.0 g) of vegetable oil into a 250-mL beaker.
2. Measure 15 mL of 50% ethanol-water mixture into a 50-mL beaker. Slowly dissolve 2.5 g of NaOH pellets in the ethanol-water mixture.
3. Add 2–3 mL of the NaOH solution to the beaker containing the oil. **CAUTION:** *Keep your face away from the beaker*. Heat the mixture over a *low flame* while stirring. Every few minutes, for about 20 minutes, add a portion of the ethanol-water mixture while continuing to stir. Heat and stir for about 10 more minutes. The oil should be dissolved and a homogeneous solution should be obtained.
4. Add 25 mL of cold water to the hot solution. Using a towel "handle," as shown in Figure 30.1, pour this solution into a 250-mL beaker containing 150 mL of saturated NaCl. Stir this mixture gently and allow it to cool for several minutes.
5. Using a spatula, skim off the top layer of soap and place it in a 50-mL beaker.

Step 2.
The solid NaOH dissolves rather slowly. The dissolution is faster if the NaOH is first dissolved in 7.5 mL of water and 7.5 mL of ethanol is added. Use this alternative procedure if time is an important factor, but caution students that significant heat is generated when the NaOH pellets are added to such a small volume of water. The addition should be done slowly and with care.

Step 3.
Caution: The reaction mixture should be watched constantly during heating.

Step 5.
Soap is fairly soluble in water, but insoluble in saturated sodium chloride solution.

Step 9–11.
Typical reactions of soap and detergent with cations are as follows. The carboxylic acid salts are less soluble than the salts of the detergent acids.

$2RCO_2^-(aq) + Ca^{2+}(aq) \rightarrow (RCO_2)_2Ca(s)$

$2RCO_2^-(aq) + Mg^{2+}(aq) \rightarrow (RCO_2)_2Mg(s)$

$3RCO_2^-(aq) + Fe^{3+}(aq) \rightarrow (RCO_2)_3Fe(s)$

$2RSO_3^-(aq) + Ca^{2+}(aq) \rightarrow (RSO_3)_2Ca(aq)$

$2RSO_3^-(aq) + Mg^{2+}(aq) \rightarrow (RSO_3)_2Mg(aq)$

$3RSO_3^-(aq) + Fe^{3+}(aq) \rightarrow (RSO_3)_3Fe(aq)$

Part B. Properties of Soaps and Detergents

6. Place a pea-sized lump of your soap into a test tube. Use a scoopula to put a similar amount of laundry detergent in a second tube and a similar amount of hand soap in a third tube. Add 10 mL of water to each tube, stopper them, and shake the tubes thoroughly. In this step and throughout this experiment, use a test-tube rack as needed.

7. Estimate the pH of the solutions, using wide-range indicator solution or wide-range test paper. Record the results. Pour the contents of the test tubes down the drain. Rinse the test tubes and stoppers with water.

8. Mark three test tubes with the labels "$CaCl_2$," "$FeCl_3$," and "$MgCl_2$," respectively.

9. Prepare a detergent solution by dissolving 0.3 g of detergent in 30 mL of water. Divide this solution equally among the three test tubes. Add solutions to the test tubes as follows.

$CaCl_2$ test tube: 1.0 mL (or 20 drops) of 0.1*M* $CaCl_2$

$FeCl_3$ test tube: 1.0 mL of 0.1*M* $FeCl_3$

$MgCl_2$ test tube: 1.0 mL of 0.1*M* $MgCl_2$

Stopper each test tube and shake it to mix. Record your observations. Pour the contents of the test tubes down the drain. Rinse the test tubes and stoppers with water.

10. Repeat Step 9, but replace the detergent solution with a hand-soap solution of the same strength. Record your observations.

11. Repeat Step 9, but replace the detergent solution with a solution of your soap of the same strength. Record your observations.

12. Follow your teacher's instructions for proper disposal of the materials.

Use the following disposal methods for chemical waste.

Disposal 1: Soap in Part A.

Disposal 2: NaCl(*aq*) in Part A and the reaction solutions in Part B.

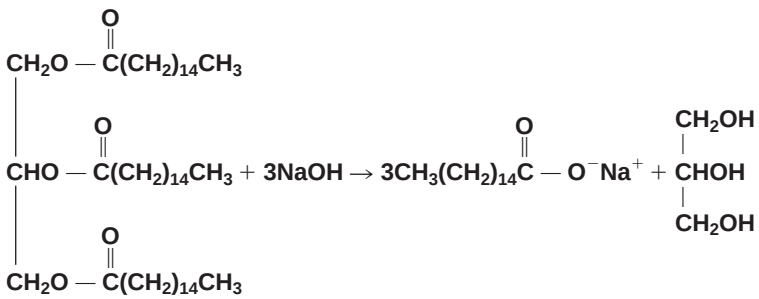
Name _____ Date _____ Class _____

OBSERVATIONS

DATA TABLE 1: PROPERTIES OF SOAPS AND DETERGENTS				
	pH of	Effect of Adding Salts		
Substance	Solution	$CaCl_2$	$FeCl_3$	$MgCl_2$
your soap	**10–11**	**heavy white ppt.**	**no ppt.**	**light white ppt.**
detergent	**10–11**	**no ppt.**	**no ppt.**	**light ppt.**
hand soap	**10–11**	**heavy white ppt.**	**light ppt.**	**white ppt.**

ANALYSES AND CONCLUSIONS

1. Write the reaction for saponification of a typical fat (or oil) with sodium hydroxide. Include structural formulas.



2. How does the pH of the soap solution that you prepared compare with those of the solution of commercial laundry detergent and the solution of hand soap? Which of these products would have the harshest effect on the skin?

The pH of each solution is about the same. If harshness to skin is caused by high pH, all would be equally harsh.

3. The metal ions Ca^{2+}, Fe^{3+}, and Mg^{2+} all contribute to the formation of *hard water*. What differences did you observe when the metal ions Ca^{2+}, Fe^{3+}, and Mg^{2+} were added to a soap or detergent? Do you think that a soap or detergent would make a better cleansing agent in hard water? Explain.

The detergent does not form insoluble salts (scums) with the ions Mg^{2+} and Ca^{2+} in hard water. This is the reason that detergents are often preferred to soaps as cleansing agents in hard water.

Name _____ Date _____ Class _____

GOING FURTHER

Develop a Hypothesis

Based on the results of this lab, develop a hypothesis to explain why commercial laundry detergents do not form insoluble salts when mixed with calcium, iron, or magnesium salts.

_____

_____

_____

_____

Design an Experiment

Propose an experiment to test your hypothesis. If resources are available and you have your teacher's permission, perform the experiment. Alternatively, do research to learn more about what properties of laundry detergent enhance their solubility in hard water.

_____

_____

_____

_____