EXPERIMENT 12

Saponification and Soaps

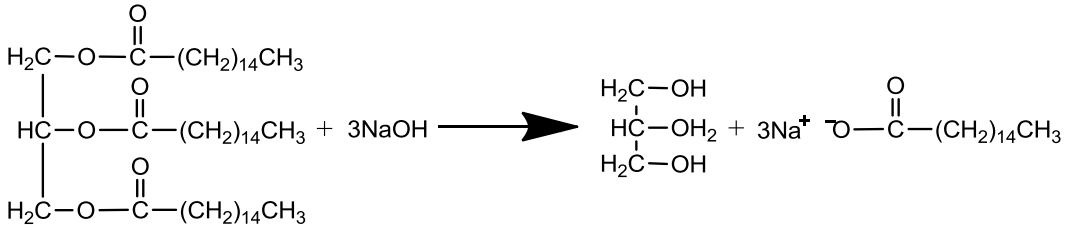
Goals

- Prepare soap by the saponification of a fat or oil.
- Observe the reactions of soap with oil, $CaCl_2$, $MgCl_2$, and $FeCl_3$.

Discussion

A. Saponification: Preparation of Soap

For centuries, soaps have been made from animal fats and lye (NaOH), which was obtained by pouring water through wood ashes. The hydrolysis of a fat or oil by a base such as NaOH is called *saponification* and the salts of the fatty acids obtained are called *soaps*. The other product of hydrolysis is glycerol, which is soluble in water.



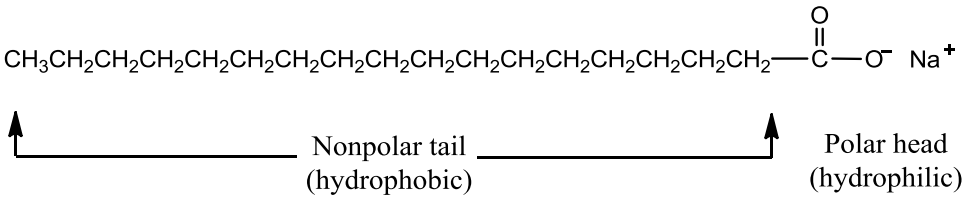
Fat (tripalmitin) Base Glycerol Soap (sodium palmitate)

The fats that are most commonly used to make soap are lard and tallow from animal fat and coconut, palm and olive oils from vegetables. Castile soap is made from olive oil. Soaps that float have air pockets. Soft soaps are made with KOH instead of NaOH to give potassium salts.

B. Properties of Soaps and Detergents

A soap molecule has a dual nature. The nonpolar carbon chain is hydrophobic and attracted to nonpolar substances such as grease. The polar head of the carboxylate salt is hydrophilic and attracted to water.

The dual polarity of a soap (salt of a fatty acid)

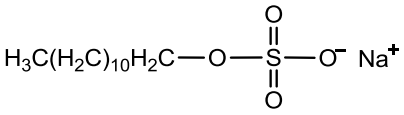


Nonpolar tail (hydrophobic) Polar head (hydrophilic)

When soap is added to a greasy substance, the hydrophobic tails are embedded in the nonpolar fats and oils. However, the polar heads are attracted to the polar water molecules. Clusters of soap particles called *micelles* form with the nonpolar oil droplet in the center surrounded by many polar heads that extend into the water. Eventually all of the greasy substance forms micelles, which can be washed away with water. In hard water, the carboxylate ends of soap react with Ca^{2+}, Fe^{3+}, or Mg^{2+} ions and form an insoluble substance, which we see as a gray line in the bathtub or sink. Tests will be done with the soap you prepare to measure its pH, its ability to form suds in soft and hard water, and its reaction with oils.

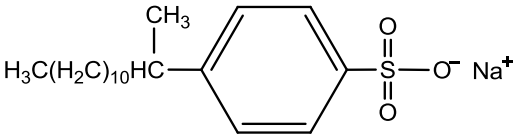
Detergents or "syndets" are called synthetic cleaning agents because they are not derived from

naturally occurring fats or oils. They are popular because they don't form insoluble salts with ions, which means they work in hard water as well as in soft water. A typical detergent is sodium lauryl sulfate.



Lauryl sulfate salt,
a nonbiodegradable detergent

As detergents replaced soaps for cleaning, it was found that they were not degraded in sewage treatment plants. Large amounts of foam appeared in streams and lakes that became polluted with detergents. Biodegradable detergents such as an alkylbenzenesulfonate detergent eventually replaced the nonbiodegradable detergents.



Laurylbenzenesulfonate salt,
a biodegradable detergent

In addition to the sulfonate salts, a box of detergent contains phosphate compounds along with brighteners and perfumes. However, phosphates accelerate the growth of algae in lakes and cause a decrease in the dissolved oxygen in the water. As a result, the lake decays. Some replacements for phosphates have been made.

Lab Information

Time: 2 hr

Comments: You will be working with hot oil and NaOH. Be sure you wear your goggles. Tear out the report sheets and place them beside the matching procedures.

Related Topics: Esters, saponification, soaps, hydrophobic, hydrophilic

Experimental Procedures — Wear your safety goggles!

A. Saponification: Preparation of Soap

Materials: 150-mL beaker, hot plate, graduated cylinder, stirring rod or stirring hot plate with stirring bar, large watch glass, 400-mL beaker, Büchner filter system, filter paper, plastic gloves, fat (lard, solid shortening, coconut oil, olive or other vegetable oil), ethanol, 20% NaOH, saturated NaCl solution.

Weigh a 150-mL beaker. Add about 5 g of fat or oil. Reweigh.

Add 15 mL ethanol (solvent) and 15 mL of 20% NaOH. ***Use care when pouring NaOH.*** Place the beaker on a hot plate and heat to a gentle boil, stirring continuously. A magnetic stirring bar may be used with a magnetic stirrer. Heat for 30 minutes or until saponification is complete and the solution becomes clear with no separation of layers. Be careful of splattering; the mixture contains a strong base. Wear disposable gloves, if available. Do not let the mixture overheat or char. Add 5-mL portions of an ethanol-water (1:1) mixture to maintain volume. If foaming is excessive, *reduce* the heat.

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

Obtain 50 mL of a saturated NaCl solution in a 400-mL beaker. (A saturated NaCl solution is prepared by mixing 30 g of NaCl with 100 mL of water.) Pour the soap solution into this salt solution and stir. This process, known as "salting out", causes the soap to separate out and float on the surface.

Collecting the soap Collect the solid soap using a Büchner funnel and filter paper. See Figure 32.1. 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 prepared for the next part of this experiment. Describe the soap.

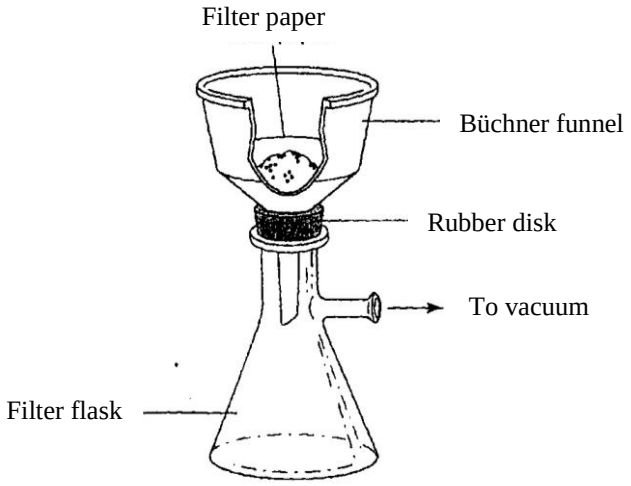


Figure 32.1. Apparatus for suction filtration with Büchner funnel.

B. Properties of Soaps and Detergents

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 g of each in about 50 mL of distilled water. If the soap is a liquid, use 20 drops.

B.1 **pH test** Place 10 mL of each soap solution in separate test tubes. Use 10 mL of water as a comparison. Label. Dip a stirring rod into each solution, then touch the stirring rod to pH paper. Determine the pH. *Save the tubes for part B.2.*

B.2 **Foam test** Stopper each of the tubes from B.1 and shake for 10 seconds. The soap should form a layer of suds or foam. Record your observations. *Save the tubes for part B.3.*

B.3 **Reaction with oil** Add 5 drops of oil to each test tube from B.2. Stopper and shake each one for 10 seconds. Record your observations. Compare the sudsy layer in each test tube to the sudsy layers in part B.2.

Report Sheet—Lab 32

Date	Name
Section	Team
Instructor	

Pre-Lab Study Questions

1. What happens when a fatty acid reacts with NaOH?

2. Why is ethanol added to the reaction mixture of fat and base in the making of soap?

3. What is the product of saponification of a salt?

A. Saponification: Preparation of Soap

Describe the appearance of your soap.

Questions and Problems

Q.1 How would soaps made from vegetable oils differ from soaps made from animal fat?

Q.2 How does soap remove an oil spot?

B. Properties of Soaps and Detergents

Tests	**Water**	**Lab Soap**	**Commercial Soap**	**Detergent**
B.1 pH				
B.2 Foam				
B.3 Oil				
B.4 $CaCl_2$				
$MgCl_2$				
$FeCl_3$				