

Experiment 15

It's a Soap Opera!

OUTCOMES

After completing this experiment, the student should be able to:

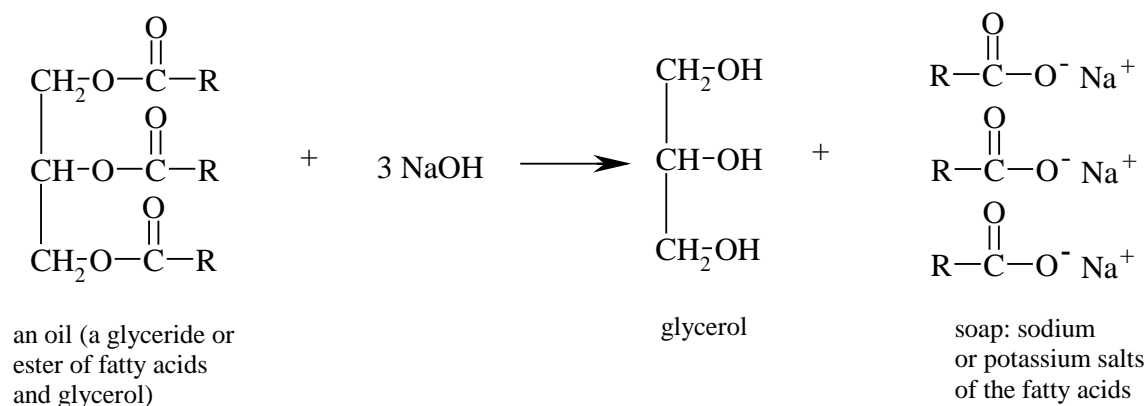
- prepare a soap starting with animal lard.
- examine the chemical properties of the soap that is made.
- understand the result of a saponification reaction.
- understand how soap cleans.
- use vacuum filtration as a separation technique.

BACKGROUND

A soap is the sodium or potassium salt of a long chain fatty acid (at least 12 carbons and a carboxylic acid group). Solid soaps typically consist of sodium salts of fatty acids and liquid soaps consist of potassium salts of fatty acids. Carboxylic acids with their acidic H removed by a strong base such as NaOH or KOH are known as carboxylate salts. Compare the general formula for a carboxylic acid with the general formula for a soap, shown below.

Soaps are made from the **saponification** (also known as basic hydrolysis) of a fat or oil (such as lard, cooking oil, or cottonseed oil) with concentrated sodium hydroxide (NaOH, also known as lye). Historically, soap was made from lard, an animal fat, and lye, a mixture of aqueous NaOH and KOH obtained by leaching wood ashes with water.

A general saponification reaction is shown below:



Note: the R groups in the general saponification reaction above represent alkyl chains with 12-18 carbons. Saturated fats have all C-C single bonds in these chains. Unsaturated and trans fats have some C=C double bonds in their alkyl chains.

Soap molecules have a polar (charged) end that is **hydrophilic** and a long, nonpolar, hydrocarbon chain that is **hydrophobic**. Soap is able to dissolve (actually to suspend or emulsify) oils or other nonpolar substances in water in the following way: the polar ends of the soap molecules dissolve in water, and the nonpolar ends encase or surround the small globules of oil or dirt. This makes the dirt more soluble in water. This spherical aggregate is called a **micelle**. In this way, soap helps remove dirt from things we want to clean, like clothes or the skin.

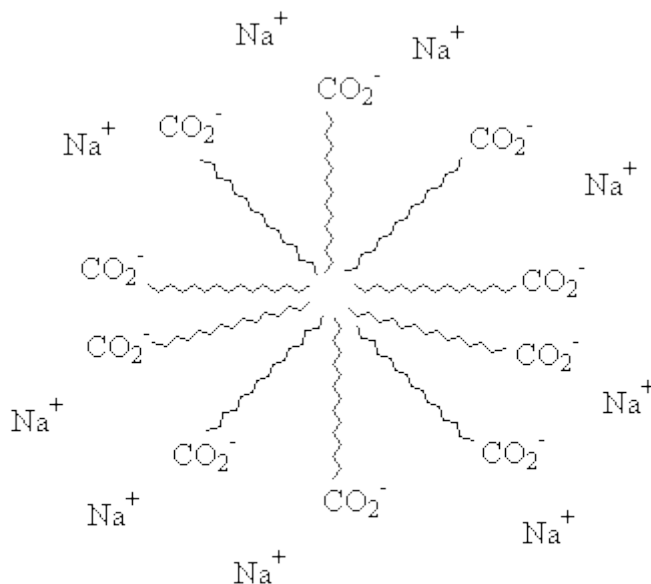


Diagram of Soap Micelle

If the water in which the soap is dissolved contains appreciable amounts of ions such as calcium, magnesium or iron, (that is, the water is **“hard”**), these ions replace the sodium ions in the soap molecule and the resulting compound becomes insoluble forming bath tub ring or soap scum. Water softeners replace these hard water ions with sodium ions.

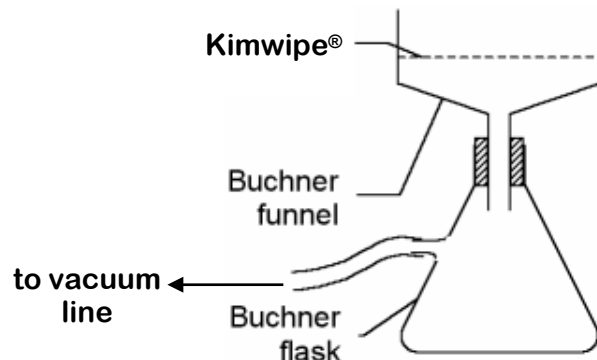
When we shake a mixture of oil and water vigorously, the oil forms very small droplets that disperse uniformly throughout the water. When we stop shaking the oil/water mixture, the oil and water separate back into their layers. If we add a few drops of oil to water containing some soap and shake the mixture vigorously, the oil drops disperse through the solution as before. However, when we allow this solution to stand, the oil droplets do not easily coalesce to form separate layers as before. This new solution is an **emulsion**. An emulsion contains fine droplets of one liquid suspended in another liquid. In this case the oil is suspended in water. The hydrophobic end of the soap molecule interacts with the nonpolar oil, and the hydrophilic end (COO^-) of the soap molecule dissolves in water. The oil droplet has been emulsified by the soap solution. The better emulsion characteristics of soap, the better it can clean.

PROCEDURE

- ⚠ **Wear large chemical splash goggles at all times for this experiment.**
- ⚠ **Wear gloves while working with the chemicals in this experiment. Sodium hydroxide causes serious chemical burns. If a slippery feel is noted on the skin, immediately **rinse with cold water** and report it to your instructor.**

Preparation of Soap

1. Tare a 400 mL beaker on the balance and then measure 18 g of lard into it. Using a graduated cylinder, add 20 mL of ethanol and 20 mL of 20% sodium hydroxide. Stir the mixture.
- ⚠ **Perform step 2 in the fume hood. Make sure the hood sash is as low as possible.**
⚠ **Stir constantly while heating to avoid bumping or splattering.**
2. Place the beaker on a hot plate and heat gently for 15-30 minutes while stirring constantly. Continue heating until odor of alcohol dissipates and a pasty mass remains (similar to the consistency of melted marshmallows). This pasty mass is the soap plus the glycerol produced in the reaction.
- ⚠ **Turn off and unplug the hotplate when you're finished with it.**
3. Allow the soap mixture to cool for a few minutes; then add 150 mL of saturated NaCl solution and stir thoroughly with a glass rod. This process is referred to as "salting out" (a form of precipitation) and is used to remove the soap from the water, glycerol and unreacted NaOH.
4. After the mixture has been stirred and mixed completely, filter out the soap using a Buchner funnel. Use a Kimwipe® rather than filter paper in the Buchner funnel. Rinse with two 10 mL portions of chilled ethanol while the suction is still on. Allow the soap to dry by spreading it out on a paper towel.



Properties and Reactions of Soap

1. *Washing properties.* (If you have dry or sensitive skin, you may want to wear gloves for the first test.) Take a small amount of your soap and wash your hands with it. In soft or deionized water, the soap should lather easily. If any oil is left over, the soap will feel greasy. Too much NaOH will also result in a slick, greasy feel and will roughen your hands after awhile. Describe the washing properties of soap on your report sheet. Rinse your hands several times with clean water after the test.
2. *Alkalinity (basicity).* A soap that contains free alkali (excess NaOH) can damage your skin. To test whether the soap contains free alkali, dissolve a small piece in 5 mL of ethanol and add 2 drops of phenolphthalein indicator. If the indicator causes the solution to remain colorless, there is no free alkali; if the indicator causes the solution to turn purple, pink or red, free alkali is present. Record your observation.
3. *Hard-water reactions.* Dissolve about 1 gram of your soap in 50 mL of deionized water. Pour 10 mL of soap solution into three test tubes. Add 8-10 drops of 5% CaCl_2 to test tube 1; add 8-10 drops of 5% MgCl_2 to test tube 2; add 8-10 drops of 5% FeCl_3 to test tube 3. Record your observations. (Does this remind you of the “scum” that forms when you wash in hard water?)
4. *Emulsifying properties.* Add 15-20 drops of mineral oil in a test tube containing 8-10 mL of deionized water and shake it. A temporary emulsion or suspension of oil droplets in water should form. Let this solution stand for a few moments. Prepare another test tube with the same ingredients, and add 10 mL of soap solution before shaking the test tube. Compare the stabilities of the emulsions in the two test tubes. Which emulsion separates back into two clear layers first? Which is the more stable emulsion? Record your observations.

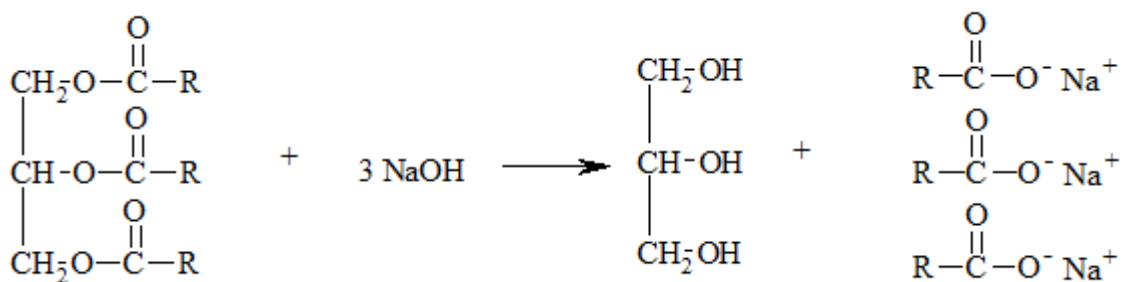
⚠ Dispose of all chemicals in the proper waste container.

Name _____

Lab Section _____

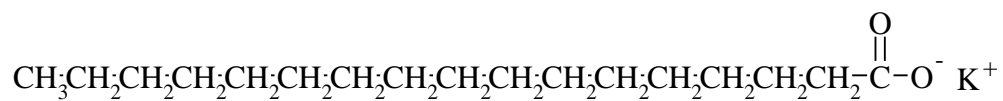
PRELAB QUESTIONS

1. What is soap?
2. What does the term saponification mean?
3. What does the term emulsion mean?
4. What do the terms hydrophilic and hydrophobic mean (you may need to consult a dictionary)?
5. Circle the ester and alcohol functional groups that appear in the following molecules:

*(more on back)*

6. For typical soaps, what do the "R" groups in the previous equation consist of?

7. Indicate the polar and nonpolar portion of the soap shown below:



8. What problem(s) may result from using soap in hard water?

Name _____

Lab Section _____

Partner's Name _____

RESULTS

1. Washing properties. Does the soap lather a lot, a little, or not at all? Does the soap feel oily?

2. Alkalinity (basicity): Soap solution + phenolphthalein indicator

turns red or pink _____

remains colorless _____

3. Hard water reactions

Cation Added	Observations
Ca^{+2}	
Mg^{+2}	
Fe^{+3}	

4. Emulsifying properties

a. Which mixture, **oil-water** or **oil-water-soap**, forms a more stable emulsion?

b. Which emulsion separates back into two layers first?

c. Explanation:

POST LAB QUESTIONS

1. Explain how soap acts as a cleansing agent? Include terms used in this lab including hydrophilic, hydrophobic, polar, and non-polar.
2. Write a balanced chemical equation for the preparation of a typical soap from fat using the condensed structures of all reactants and products.
3. What is one thing that can cause a soap not to clean properly?
4. Why is it important that excess lye (NaOH) be removed from soap after it is made?
5. What is the difference between a carboxylate salt and a carboxylic acid? Give an example from this lab.