

# Preparation and Properties of Soap

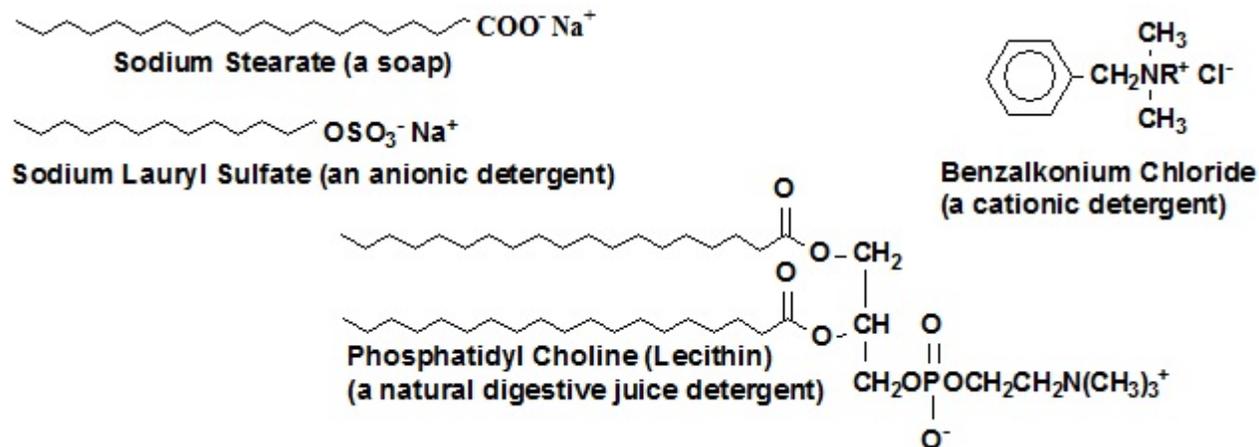
## Experiment #7

**Objective:** To prepare soap by alkaline hydrolysis (saponification) of natural fats and test some of the chemical properties and cleansing power of soap relative to detergent.

### Introduction

Soaps are the sodium and potassium salts of long chain fatty acids that are generally made by saponification (alkaline hydrolysis) of natural fats, such as animal fats or palm oil. Prior to World War II, most soap was prepared in the home by boiling animal fat with lye (commercial sodium hydroxide). Soap is not particularly good for cleaning in hard water (water with high concentrations of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and/or  $\text{Fe}^{3+}$  ions) because it forms insoluble complexes with divalent (or trivalent) cations. Organic sulfonic acid and phosphoric acid salts, commonly known as detergents, were found to be much more effective cleaning agents because they do not readily form insoluble complexes with the ions in hard water. Modern cleansers labeled "soft soap" are, in fact, detergents rather than soap. Check the labels for ingredients of some cleansers you have around the home, including shampoo.

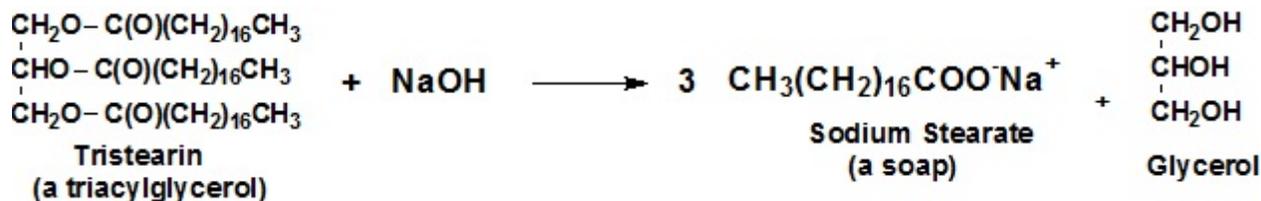
The phosphate detergents caused environmental problems in waterways, not because they are toxic, but because they are nutrients and resulted in an overabundance of plant growth, or algal blooms. Consequently phosphate detergents have been replaced with other alternatives. Today there are a wide variety of synthetic detergents available for various purposes. Some representative detergents and a soap are shown below.



In this experiment you will prepare soap from a fat and determine some of its properties relative to a detergent. See the chemical equation for saponification on the next page.

### Materials and Reagents

Shortening, lard or coconut oil, 30% sodium hydroxide solution, 95% ethanol, ice, universal pH paper or red litmus paper, 1 % sodium lauryl sulfate (detergent) solution, 5% calcium chloride solution, filter paper, filtration apparatus, dirty pump oil.



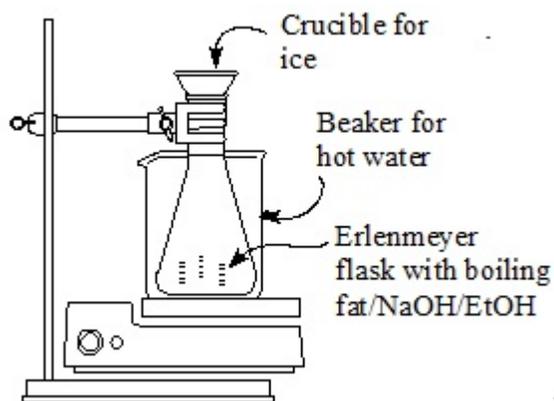
## Procedure

### Part A. Preparation of Soap.

1. Add about 150 mL water to a 400 mL beaker and heat the water on a hot plate to about 85°C. You will need to mount a clamp above the beaker to hold the Erlenmeyer flask upright in the water bath (see below).
2. Add about 1 g (approx. half a **level**, not heaping teaspoon) of shortening, coconut oil, or lard to a small beaker and dissolve it in 20 mL of 95% ethanol. Do not use too much fat or you will need more ethanol to dissolve it. Warm the ethanol and fat on the hotplate to get the fat to dissolve more easily. Do not allow the ethanol to boil.

**CAUTION!! SODIUM HYDROXIDE SOLUTION IS VERY CAUSTIC. AVOID GETTING IT ON YOUR SKIN OR CLOTHING. WASH IT OFF IMMEDIATELY WITH PLENTY OF WATER.**

3. When the fat is completely dissolved, pour the solution into a 125 mL Erlenmeyer flask and add 10 mL of 30% NaOH solution. Swirl the flask to mix the solution and add a few (3 to 5) boiling chips (Boileezers) to the flask.
4. Place the flask containing the fat solution in the water bath and fasten it with a clamp to hold it upright in the water bath. Place a ceramic crucible over the mouth of the flask to minimize evaporation of the alcohol. Place an ice cube in the crucible to more effectively condense the alcohol.
5. Allow the the fat-alcoholic NaOH solution to boil for at least 30 min. [**Note: do not boil the water in the water bath**]. If you observe undissolved fat in the flask, add a little more ethanol to the flask and stir to dissolve the fat. Do not allow the hot water bath to get too hot, keep it just hot enough to have controlled boiling of the fat solution.



6. While the saponification proceeds, prepare a salt solution by completely dissolving 12 g sodium chloride (NaCl) in 60 mL deionized water in a 250 mL beaker. After the salt completely dissolves, transfer about half of this salt solution to another beaker and place both beakers of salt solution on ice to cool them.
7. After 30 min, test the fat solution to see if saponification is complete by placing a few drops of the solution in a test tube of deionized water. If you see fat droplets float to the top, the saponification is not complete and allow it to boil for an additional 10 min.
8. When saponification is complete, carefully pour the hot reaction mixture into one beaker of salt solution and stir for a minute or two. Place the mixture on ice to cool it before filtering. The soap should solidify before filtering this mixture.
9. Set up a suction filtration apparatus with a Buchner funnel and filter paper (see Appendix I for a diagram of the vacuum filtration apparatus). Filter the mixture from step 8, and wash the soap (remaining in the Buchner funnel) twice with ice cold salt water (from the second beaker). Draw air through the soap for a few minutes to remove most of the water.
10. Remove the soap cake from the Buchner funnel and save it for the following tests.

## **Part B. Comparison of the Properties of Soap with Detergent.**

### B-1. Alkalinity

1. Prepare a 1% soap solution by dissolving about 0.5 g of the soap you just made in 50 mL of deionized water. It may help to heat the water to get the soap to dissolve completely.
2. Use a glass stirring rod to place a drop of the soap solution on a piece of universal pH paper to determine whether the solution is alkaline, neutral or acid. Record your observation on the Report Sheet.
3. Add about 5 mL of detergent (e.g., sodium lauryl sulfate) solution to a beaker or test tube. You will compare this solution with your soap solution for the remaining tests.
4. Test the detergent solution to see if it is alkaline, neutral or acidic by placing one drop of the detergent solution on a strip of pH paper or red litmus paper. Record your observation on the Report Sheet.

### B-2. Lathering Power

1. Add about 2 mL of deionized water to two large test tubes.
2. Add an equal amount of soap solution to one test tube of water and shake vigorously by

placing a stopper in the tube. This should give a permanent lather (sudsing) that lasts for at least 30 sec. If the lather doesn't last, add another 10 drops of soap solution and shake vigorously.

3. Add an equal amount of detergent solution to another test tube of water and shake vigorously. This should give a permanent lather. If not, add another 10 drops and shake vigorously. Record the results on the report sheet.
4. Add 2 mL of 5% calcium chloride solution to each of two clean test tubes.
5. Add an equal amount of soap solution to one of the tubes containing calcium chloride and shake vigorously. Notice whether this solution forms a permanent lather and note whether there is any flocculent precipitate in the tube. Does the precipitate float or sink?
6. Add an equal amount of detergent solution to the other tube containing  $\text{Ca}^{2+}$  ion and shake vigorously to get a permanent lather. Does the lathering power of the detergent differ from the soap when  $\text{Ca}^{2+}$  is present? Is there any precipitate in this solution? Record the results on the report sheet.

### B-3. Cleansing Power

1. Place a drop of used dirty oil or smear some grease or fat, such as shortening, on four separate thin strips of filter paper. Make sure the strips of filter paper will fit in the test tubes used in part B-2.
2. Place one filter paper with oil spot in the tube containing soap in water. Place another in the tube containing detergent and water. Place a third strip in the tube containing soap in calcium solution. Place the fourth strip of oily paper in the tube containing detergent and calcium solution. Shake each one well and make sure the dirty spot on the filter paper is immersed in the solution.
3. Remove the filter paper and rinse with tap water. Did the dirty oil or fat get washed out of the filter paper strip? You can discard the solutions in the sink. Throw the paper strips in the trash can. Compare the cleaning power of soap vs detergent on the Report Sheet, indicating whether calcium ion has any effect on the cleaning power of either.

**Preparation and Properties of Soap**

Experiment #7

Pre-Lab Exercise

1. What is the chemical nature of fats and oils, *i.e.*, what class of chemical compounds make up the bulk of fats and oils? Are they alcohols, aldehydes, ketones, acids, esters, or amines?
2. Describe the chemical reaction that takes place in the process of saponification. What are the reactants? What are the products? Show a representative chemical reaction for saponification.
3. How do soaps differ from detergents? (See text book or check the internet)
4. Briefly describe how soap and detergent work to clean your body or to clean other surfaces, such as dishes. Why are these agents (soap or detergent) needed in order to have thorough cleaning?

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Name \_\_\_\_\_

Section \_\_\_\_\_

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| <p><b>Preparation and Properties of Soap</b><br/>                 Experiment #7</p> | <p>Data &amp; Report Sheet</p> |
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A-1. Describe the look and feel of the soap you have made, compared to the fat you started with.

A-2. How does your soap compare with the look and feel of commercial soap?

Table B1. Comparison of Properties of Soap vs Detergent

|           | Alkalinity test with<br>pH indicator paper |                                    | Lathering and<br>solubility |              |
|-----------|--|------------------------------------|-----------------------------|--------------|
|           | Color of<br>test paper                     | Is it alkaline,<br>neutral or acid | no calcium                  | with calcium |
| Soap      |  |                                    |                             |              |
| Detergent |  |                                    |                             |              |

B-1. What conclusion would you make with regard to the harshness of soap vs detergent on skin from the alkalinity test?

B-2. Compare the lathering power and solubility of soap vs detergent in hard water (calcium solution).

B-3. Would you expect soap or detergent to be a more effective cleanser (dissolving oils) in hard water (water with high level of calcium or magnesium ions)? Explain.

B-4. Describe the oil/grease spot on the filter paper after cleansing with soap vs detergent in water. Is there a noticeable difference? Which is the more effective cleanser in deionized water?

B-5. Describe any differences in the cleaning power of soap vs detergent in calcium solution.