EXPERIMENT 32

Preparation and Properties of a Soap

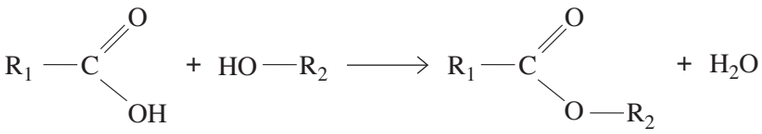
Performance Goals

32–1 Starting with a vegetable oil, prepare a soap in the laboratory.

32–2 Examine the chemical properties of the soap you prepared.

CHEMICAL OVERVIEW

An ester is the product of the reaction between an alcohol and a carboxylic acid. The typical equation for the formation of an ester is

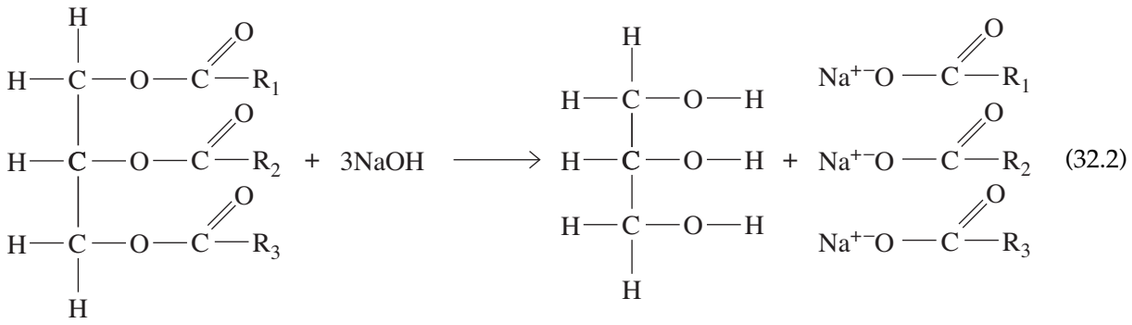


(32.1)

Acid *Alcohol* *Ester*

where R_1 and R_2 are general symbols for *alkyl* groups containing only hydrogen and carbon. They may be the same group or they may be different. If the alcohol is glycerol, $C_3H_5(OH)_3$, and the acid is a long-chain fatty acid such as stearic acid, $C_{17}H_{35}COOH$, the ester is typical of those found in fats and oils. These esters can be reacted with strong bases to yield glycerol and the salt of the fatty acid. This process is known as

saponification, and the sodium (or potassium) salts of the fatty acids are called *soaps.* The soap-making process may be written as



(32.2)

Ester + *Base* ⟶ *Glycerol* + *Soap*

As you can see from Equation 32.2, the anions of soaps contain both polar groups (—COO^-) and nonpolar groups (long chains of carbon atoms). Polar compounds (or groups) are attracted to water and are called **hydrophilic.** Nonpolar compounds (or groups) are water repelling or **hydrophobic,** and are soluble in or miscible with nonpolar compounds, such as fats, grease, oil, or other ''dirt.'' This dual characteristic of soaps is the reason behind their cleaning action. The fat or oil is displaced from the fiber by the soap solution to form large globules that can be detached by jarring (rubbing) and then dispersed (emulsified) in the aqueous solution. **Emulsions** consist of fine droplets of one liquid dispersed in another liquid, in which the first is insoluble (like oil in water). The better the emulsion characteristics of a soap, the better it can clean.

Generally, soaps made from liquid fats (or oils) are more soluble than those made from solid fats. In the laboratory, you will prepare a soap by saponifying a vegetable oil with sodium hydroxide. Ethyl alcohol will be added to serve as a common solvent for the reactants. You will also investigate the characteristics of soaps formed from fatty acids and some divalent and trivalent cations. These cations are commonly encountered in areas where there is hard water and in industry. Their soaps are usually referred to as **metallic soaps.**

SAFETY PRECAUTIONS AND DISPOSAL METHODS

Alcohol vapors are highly flammable. Keep the flame away from the top of the beaker. Have an asbestos square handy to cover the beaker if the vapors should ignite. Also, sodium hydroxide causes severe burns when in contact with the skin. If a slippery feeling is observed on the hands or other parts of the body, rinse with large amounts of cold water immediately. Wear eye protection at all times during this experiment.

Dispose of excess vegetable oil and ethyl alcohol in separate bottles. Excess NaOH should be diluted with water and then poured down the drain. Collect solutions containing kerosene.

PROCEDURE

1. Preparation of a Soap

A. Weigh a 150-mL beaker on a decigram balance and weigh into it 18 to 20 g of vegetable oil. Add 20 mL of ethyl alcohol and 25 mL of 20% sodium hydroxide solution.

B. Stir the solution and support the beaker on an asbestos gauze on a tripod. Heat the beaker and its contents gently. Continue the heating until the odor of alcohol is no longer apparent and a pasty mass remains in the beaker. The reaction product is a mixture of the soap and the glycerol freed in the reaction (see Equation 32.2).

C. Allow the soap mixture to cool; then add 100 mL of saturated sodium chloride solution and stir thoroughly with a glass rod. This process is called *salting out* and is used to remove the soap from water, glycerol, and any excess sodium hydroxide present.

D. After the mixture has been stirred and mixed completely, filter off the soap on a Büchner funnel, using suction, as illustrated in Figure 31.2. Rinse with two 10-mL portions of ice-cold water, drawing this water through the funnel. Allow your soap to dry by spreading it out on a paper towel.

2. Properties and Reactions of Soaps

A. Washing properties. Take a small amount of your soap and wash your hands with it. In soft water, it should lather easily. If any oil is left over, the soap will feel greasy. Describe the washing properties of your soap on the work page. Rinse your hands several times after the test.

B. Basicity. A soap that contains free alkali is harmful to the skin, silk, or wool. To test for the presence of free base, dissolve a small amount of your soap in 5 mL of ethyl alcohol and add two drops of phenolphthalein. If the indicator turns red, free alkali is present. Record your observation.

C. Reaction with multivalent cations. Dissolve about 1 g of your soap in 50 mL of warm water.Pour about 10 mL of soap solution into each of three test tubes. To the first test tube, add 8 or 10 drops of 5% $CaCl_2$; to the second, 8 or 10 drops of 5% $MgCl_2$; and to the third, 8 or 10 drops of 5% $FeCl_3$. Record your observations on the work page. (Does this remind you of the ''scum'' that forms when you wash in hard water?)

D. Emulsification. Put 5 to 10 drops of kerosene in a test tube containing 8 to 10 mL of water and shake it. An emulsion or suspension of tiny oil droplets in water will form (the solution will look cloudy). Let this solution stand for a few minutes. Prepare another test tube with the same ingredients, but add about 0.5 g of your soap to it before shaking it. Compare the stabilities of the emulsions in the two test tubes. Which emulsion seems to contain smaller droplets? Which emulsion clears up first? Explain. Record your answers on the work page.

Name *Date* *Section*

Experiment 32

Advance Study Assignment

1. What is a soap?

2. What does the term *saponification* mean?

3. What does the term *emulsion* mean?

4. What do the terms *hydrophilic* and *hydrophobic* mean?

Name _____ Date _____ Section _____

Experiment 32

Work Page

PART 2—Properties and Reactions of Soaps

Step A—Washing Properties

Soap lathers a lot _____, a little _____, not at all _____.

Soap feels oily: yes _____, no _____.

Step B—Basicity

Soap solution + indicator: turns pink _____; remains colorless _____

Step C—Reaction with Multivalent Cations

Cation Added	*Observation*
Ca^{2+}	
Mg^{2+}	
Fe^{3+}	

Step D—Emulsification

Emulsion containing smaller droplets:

Kerosene in water_____; kerosene + soap in water _____.

Emulsion that clears up first:

Kerosene in water_____; kerosene + soap in water _____.

Explanation:

Name _____ Date _____ Section _____

Experiment 32

Report Sheet

PART 2—Properties and Reactions of Soaps

Step A—Washing Properties

Soap lathers a lot _____, a little _____, not at all _____.

Soap feels oily: yes _____, no _____.

Step B—Basicity

Soap solution + indicator: turns pink _____; remains colorless _____

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Step D—Emulsification

Emulsion containing smaller droplets:

Kerosene in water _____; kerosene + soap in water _____.

Emulsion that clears up first:

Kerosene in water _____; kerosene + soap in water _____.

Explanation: