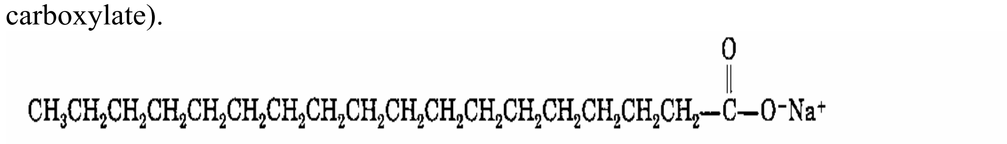
Organic Chemistry Lab
Experiment 4
Preparation and Properties of Soap

Introduction

A soap is the sodium or potassium salt of a long-chain fatty acid. The fatty acid usually contains 12 to 18 carbon atoms. The source of the fatty acids is either from animal fats or vegetables which are esters of carboxylic acids. They have a high molecular weight and contained alcohol and glycerol. Chemically, these fats and oils are called triglycerides.

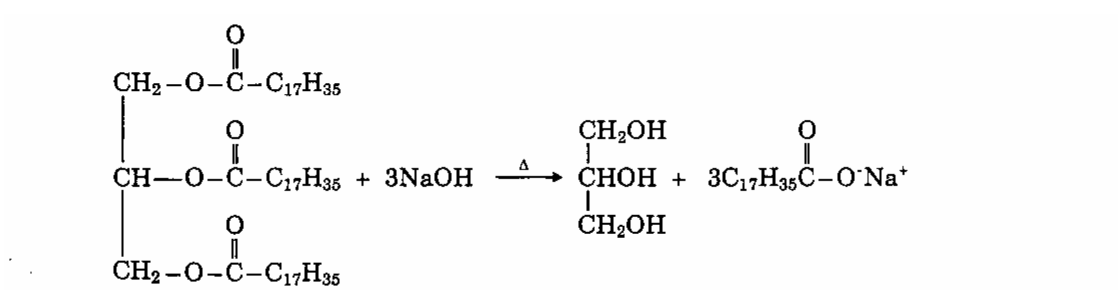
Solid soaps usually consist of sodium salts of fatty acids, whereas liquid soaps consist of the potassium salts of fatty acids. A soap such as sodium stearate consists of a nonpolar end (the hydrocarbon chain of the fatty acid) and a polar end (the ionic carboxylate).



nonpolar dissolves in oils) | polar (dissolves in water)

Because "like dissolves like/" the nonpolar end (hydrophobic or water-hating part) of the soap molecule can dissolve the greasy dirt, and the polar or ionic end (hydrophilic or water-loving part) of the molecule is attracted to water molecules. Therefore, the dirt from the surface being cleaned will be pulled away and suspended in water. Thus soap acts as an *emulsifying agent,* a substance used to disperse one liquid (oil molecules) in the form of finely suspended particles or droplets in another liquid (water molecules).

Treatment of fats or oils with strong bases such as lye (NaOH) or potash (KOH) causes them to undergo hydrolysis (saponification) to form glycerol and the salt of a long-chain fatty acid (soap).



Triglycerides | glycerol | sodium stearate (a soap)

Natural fatty acids are rarely a single type in any given fats or oil. In fact, a single tryglyceride molecule in a fat may contain three different acid residues (R_1COOH, R_2COOH, R_3COOH), and not every triglyceride in the substance will be identical.

The fats and oils that are most common in soap preparations are lard and tallow from animal sources and coconut, palm and olive oils from the vegetable sources. The length of the hydrocarbon chain and number of double bonds in the carboxylic acid salt of the carboxylic acid portion of the fat or oil determine the properties of the resulting salt. For example, the salt of a saturated long chain acid make a harder, more insoluble soap. Chain length also affects solubility.

Tallow is the principal fatty material used in making soap. The solid fats of cattle are melted with steam and tallow layer formed at the top is removed. Soap makers usually blend tallow with coconut oil and saponify this mixture. The resulting soap contains mainly the salts of palmitic, stearic and oleic acids from the tallow and the salts of lauric and myristic acids from coconut oil. The coconut oil is added to produce a softer, more soluble soap. Lard differs from tallow in that lard contains more oleic acids.

Pure coconut oil yields a soap that is very soluble in water. It is so soft that it will lather even in salt water. Palm oil contains mainly two acids, palmitic and oleic acid, in equal amount. Saponification of this oil yields a soap that is an important constituent of toilet soaps. Olive oil contains mainly oleic acid. It is used to prepare Castille soap.

Acid	Structure
Palmitic acid	$CH_3(CH_2)_{14}COOH$
Stearic acid	$CH_3(CH_2)_{16}COOH$
Oleic acid	$CH_3(CH_2)_7CH{=}CH(CH_2)_7COOH$
Lauric acid	$CH_3(CH_2)_{10}COOH$
Myristic acid	$CH_3(CH_2)_{12}COOH$

Table 1
Structure of Acids Commonly Found in Soap

Toilette soaps generally have been carefully washed free of any alkali remaining from saponification. As much glycerol as possible is usually left in the soap and perfumes and medicinal agents are sometimes added. Soft soaps are made by using potassium hydroxide, yielding potassium salts. They are used in shaving creams and liquid soaps.

Because soaps are salts of strong bases and weak acids, they should be weakly alkaline in aqueous solution. However, a soap with free alkali can cause damage to skin, silk, or wool. Therefore, a test for basicity of the soap is quite important.

Soap has been largely replaced by synthetic detergents during the last two decades, because soap has two serious drawbacks. One is that soap becomes ineffective in hard water; this is water that contains appreciable amounts of Ca^2+ or Mg^2+ salts.

$$\underset{\text{soap}}{2C_{17}H_{35}COO^-Na^+} + M^{2+} \longrightarrow \underset{\text{scum}}{[C_{17}H_{35}COO^-]_2\,M^{2+}\downarrow} + 2Na^+$$

$$M = (Ca^{2+} \text{ or } Mg^{2+})$$

The other is that, in an acidic solution, soap is converted to free fatty acid and therefore loses its cleansing action.

$$\underset{\text{soap}}{C_{17}H_{35}COO^{-}Na^{+}} + H^{+} \longrightarrow \underset{\text{fatty acid}}{C_{17}H_{35}COOH\downarrow} + Na^{+}$$

Procedure

Preparation of a soap

1. Measure 23 mL of a vegetable oil or 23 grams of lard into a 250-mL Erlenmeyer flask.
2. Add 10 mL of ethyl alcohol (to act as a solvent) and 20 mL of 25% sodium hydroxide solution (25% NaOH). While stirring the mixture constantly with a glass rod, the flask with its contents is heated gently in a boiling water bath.
3. A 600-mL beaker containing about 200 mL of tap water and a two boiling chips can serve as a water bath (Fig. 1).

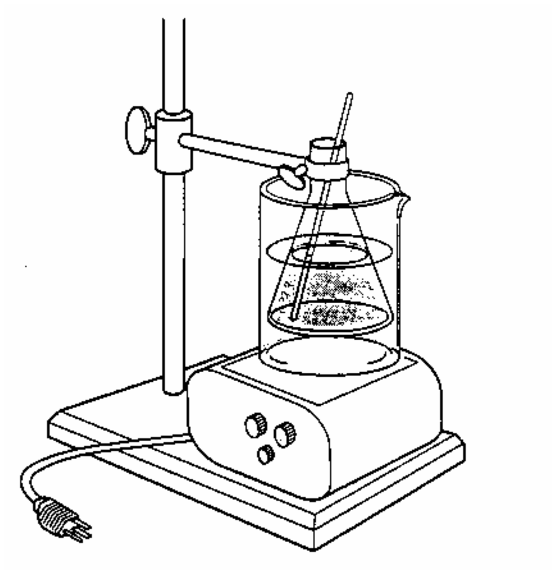


Figure 1

Set up for preparation of soap

Caution: Alcohol is flammable!

4. After being heated for about 20 min, the odor of alcohol will disappear, indicating the completion of the reaction. A pasty mass containing a mixture of the soap, glycerol, and excess sodium hydroxide is obtained.
5. Use an ice-water bath to cool the flask with its contents. To precipitate or "salt out" the soap, add 150 mL of a saturated sodium chloride solution to the soap mixture while stirring vigorously. This process increases the density of the aqueous solution; therefore, soap will float out from the aqueous solution.
6. Filter the precipitated soap with 4 ply cheese cloth on a gravity funnel and wash it with 10 mL of ice cold water. Observe the appearance of your soap and record your observation on the Report Sheet.

Properties of a soap

Emulsifying Properties.

1. Shake 5 drops of mineral oil in a test tube containing 5 mL of water. A temporary emulsion of tiny oil droplets in water will be formed.
2. Repeat the same test, but this time add a small piece of the soap you have prepared before shaking. Allow both solutions to stand for a short time.
3. Compare the appearance and the relative stabilities of the two emulsions.
4. Record your observations on the Report Sheet.

Hard Water Reactions.

1. Place about one-third spatula full of the soap you have prepared in a 50-mL beaker containing 25 mL of water.
2. Warm the beaker with its contents to dissolve the soap.
3. Pour 5 mL of the soap solution into each of 5 test tubes (nos. 1, 2, 3, 4, and 5).
4. Test no. 1 with 2 drops of a 5% solution of calcium chloride (5% $CaCl_2$), no. 2 with 2 drops of a 5% solution of magnesium chloride (5% $MgCl_2$), no. 3 with 2 drops of a 5% solution of iron(III) chloride (5% $FeCl_3$), and no. 4 with tap water. The no. 5 tube will be used for a basicity test, which will be performed later.
5. Record your observations on the Report Sheet.

Alkalinity (Basicity).

1. Test soap solution no. 5 with a wide-range pH paper.
2. What is the approximate pH of your soap solution? Record your answer on the Report Sheet.

Name:____

REPORT SHEET

Preparation

Describe the appearance of your soap.

Observation of the hard water reaction

No. 1 + $CaCl_2$____

No. 2 + $MgCl_2$

No. 3 + $FeCl_3$

No. 4 + tap water____

Alkalinity

pH of your soap solution (no. 5) ____

Name:____

POST-LAB QUESTIONS

1. When you made soap, first you dissolved vegetable oil in ethanol. What happened to the ethanol during the reaction?

2. What are the two main disadvantages of soaps versus detergent?

3. Soaps that have a pH above 8.0 tend to irritate some sensitive skins. Was your soap good enough to compete with commercial preparations.