Preparation and Properties of a Soap

Adapted from Bettelheim, et al.

OBJECTIVE:

To understand the process of soap production and study the properties of soap.

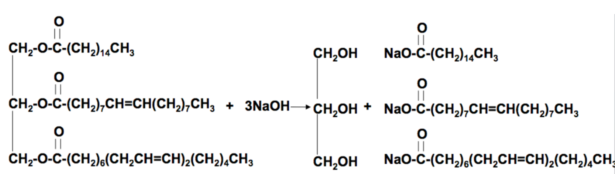
BACKGROUND:

Soap- and detergent-containing products are found in many colors, fragrances, and textures. Years of research have led to the incredible selection of soaps that we encounter in the market today, but the basics of soap formation have not changed over the years; the synthesis begins with fats and oils.

On the chemical level, fats and oils are referred to as triglycerides. Triglycerides contain the ester functional group, which comes from the reaction of the alcohol, glycerol, with three long-chain carboxylic acids, the fatty acids.

When the alkyl groups (R groups) of a triglyceride come from mostly saturated fatty acids such as stearic acid, the fat is a solid at room temperature; this is usually a characteristic of animal fats. If the alkyl groups are mainly unsaturated fatty acids, the fat is a liquid at room temperature. Example of fatty acids found in these oils include oleic and linoleic acid.

When a triglyceride is hydrolyzed in a basic medium, the products of the reaction are glycerol and the salts of the respective fatty acids. This type of reaction is often termed saponification. The fatty acid salts that form make up the substance soap. More often than not, the soap consists of a mixture of fatty acid salts. Because soaps are salts of strong bases (usually NaOH, lye, or KOH, potash) and weak acids, they should be weakly alkaline in aqueous solution. However, soap with excess free alkali can cause damage to skin, silk, or wool.



Saponification

One way to help ensure that soap has a pH close to neutral is to use a stoichiometric amount of lye in the preparation. Excess sodium hydroxide will speed the reaction rate and ensure that no oil remains unconverted to its free fatty acids but results in a finished product too basic to use safely. Home soap makers typically take advantage of tables of saponification values, which estimate the amount of lye needed for a stoichiometric ratio, and slowly bake their reactions for several hours in a warm oven to ensure complete conversion of the oils to soap. In order to speed up the process, we will use higher temperatures but still make an effort to make soaps with close to neutral pH by calculating appropriate amounts of sodium hydroxide to use for near stoichiometric ratios of oil to lye. The table below provides alkali ratios, which are similar to tradition saponification values, for a number of possible common oils. Saponification values for soaps made from a mixture of oils must be calculated based on the ratio of each of the oils used in the preparation.

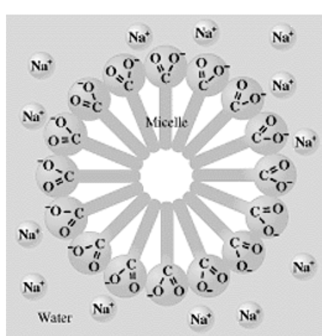
Oil	**ppt alkali**
Palm oil	135
Lard (pork fat)	136
Olive oil	131
Corn oil	133
Safflower oil	132
Coconut oil	176
Soybean oil	134

Table 1. Alkali ratios for several different types of oils. Amounts of NaOH are given in parts per thousand, the number of grams NaOH needed for 1000 grams of oil.

A major drawback associated with the use of soap is the soap scum that can form in hard water. Calcium, iron, and magnesium ions are abundant in hard water. These ions form an insoluble precipitate with soap. A second disadvantage is that, in acidic solution, soap is converted to free fatty acids and therefore loses its cleansing action. Consequently, soaps have been largely replaced by synthetic detergents.

Soaps and detergents possess a type of structure that allows for dual cleaning action. One end of the compound is a long hydrocarbon chain that is water insoluble. The other end of the compound is a very water-soluble salt structure. Dirt and oils are similar in structure to the hydrocarbon portion of the soap molecule and are dissolved. At the same time, soap is water-soluble because of its hydrophilic portion. So, once the soap dissolves the dirt and oils, it can then be washed off with water.

In the process described above, the formation of micelles takes place. Micelles are very small droplets that form by the action of soaps. The hydrophobic part of the soap surrounds the dirt, which the polar head of the soap remains on the outside in the water. Since soap can form micelles, it has the ability to form semi-stable emulsions. Emulsions are mixtures of two immiscible solvents; the greasy layer of the dirt is dispersed in the water layer via the soap micelles.



PROCEDURE:

A. SYNTHESIS OF SOAP:

1. Measure approximately 25 g of oil into a 250-mL Erlenmeyer flask, recording the exact mass used. Add 20 mL of ethanol and an appropriate amount of 25% (m/v) NaOH based on the saponification value of your oil.
2. Heat the mixture in a hot water bath for about 20 min with constant, vigorous, stirring. Keep a close watch on your flask, being prepared to lift the flask out of the water bath for a moment if it looks as though it might be about to boil over.
3. After the odor of alcohol has disappeared and the reaction mixture is viscous with a soap-like appearance, place the flask in an ice-water bath.
4. To precipitate 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, solidifies the soap and removes some excess sodium hydroxide from solution.
5. Filter the precipitated soap using vacuum filtration and wash it with two 10 mL portions of ice-cold water.
6. Observe and record the appearance of the soap.
7. You may choose to mold your soap or add fragrance. If so desired, gently melt your soap, add fragrance or dye and pour into a mold. It will take at least a week for the soap to finish drying in the mold; when dry, your instructor will return your sample.

B. PROPERTIES OF SOAP

Emulsifying properties - 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. Repeat the same test, but this time add a small piece of the soap you prepared before shaking the test tube. Allow both test tubes to stand for a short time. Record your observations

Hard water reactions and alkalinity test - Place some soap in a 50-mL beaker containing 25 mL of water. Warm the beaker to dissolve the soap. Pour 5 mL of the soap solution into each of five test tubes.

Add the following:
Test tube 1 – 2 drops of a 5% $CaCl_2$ solution
Test tube 2 – 2 drops of a 5% $MgCl_2$ solution
Test tube 3 – 2 drops of a 5% $FeCl_3$ solution
Test tube 4 – 2 drops of tap water
Test tube 5 – determine the pH of the solution using pH paper as an alkalinity test