

Saponification: The preparation of Soap

Few college students today can recall watching soap being made. Only a few generations ago, people routinely made soap by boiling beef tallow (a triglyceride) with lye (impure sodium hydroxide). When the top layer cooled and solidified, it was cut into cubes of yellow soap (sodium stearate). This soap may have been hard on hands and clothes, but it had good cleaning action, particularly with soft water. In this experiment, you will make soap as your great-grandmother may have, and examine some of its properties.

Saponification of triglycerides. Triglycerides are high molecular mass fats and oils (esters) which can be saponified (hydrolyzed) in basic solution to give soap and glycerol.

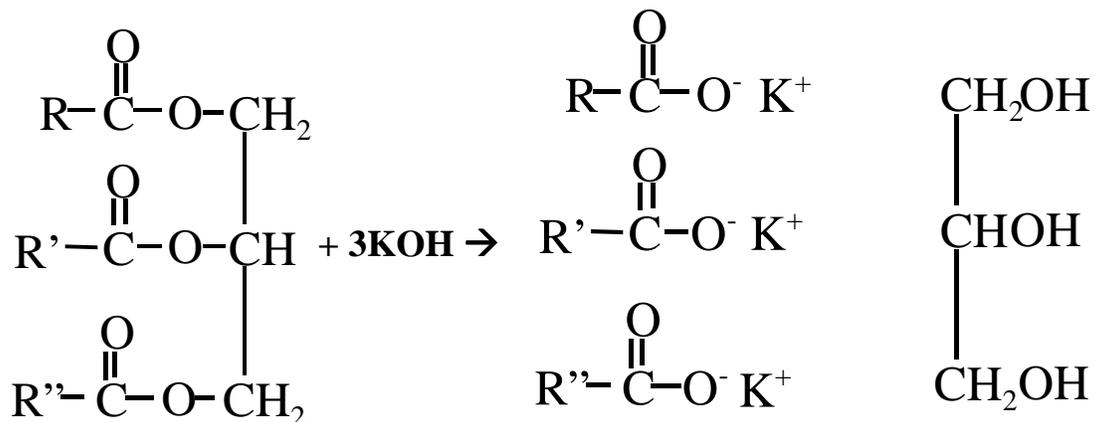
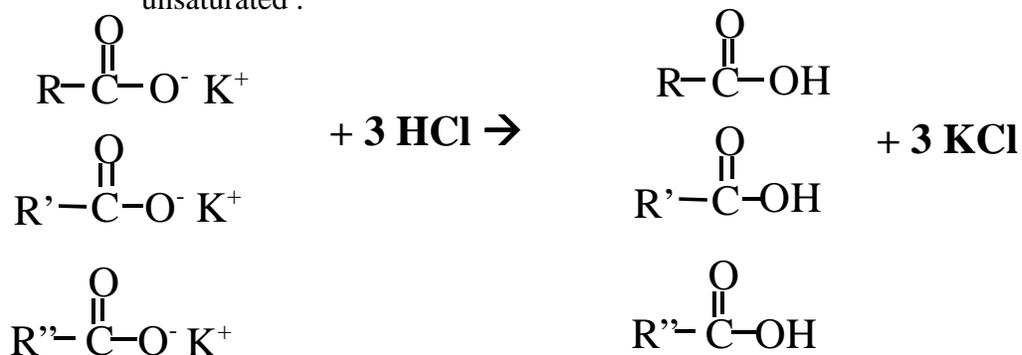


Figure 1. A triglyceride reacts with KOH to form a mixture of potassium carboxylates and glycerol.

As suggested in the equation, soap is a salt composed of a mixture of carboxylate anions and a univalent cation. A mixture of anions is formed because each triglyceride molecule contains a variety of fatty acid residues and because a particular fat or oil is itself a mixture of molecules.

Potassium soaps are more soluble than sodium soaps and readily produce lather. Therefore, potassium soaps are used to make liquid soap and shaving cream. Soaps from highly saturated, solid fats, such as tallow, lard, or shortening, are hard. Saponification of an unsaturated oil, such as olive oil, gives a soft soap.

Treatment of a soap solution with dilute hydrochloric acid produces a mixture of fatty acids. Fatty acids are long-chain carboxylic acids (C_{10} to C_{18}) which may be saturated or unsaturated.



Synthetic detergents differ from soaps in that they are salts of long-chain alkyl sulfuric acids or alkylbenzenesulfonic acids, rather than carboxylic acids.



Figure 2 Alkyl sulfuric acid (left) and alkylbenzenesulfonic acid (right)



Figure 3 Alkyl sulfate salt (detergent) at left. Alkylbenzenesulfonate salt (detergent) at right

The function of soaps and detergents is to remove grease and dirt by emulsifying the grease (bringing it into suspension). Dirt adheres to clothing and to skin primarily by being “glued” to these surfaces with a thin film of oil or grease; the oil (lipid) on the skin is generally secreted during perspiration. The soap or detergent removes the oil film and the dirt can be washed away.

How do soaps and detergents “dissolve” non-polar substances such as fats, oils, and greases? Molecules of soaps and detergents contain a non-polar (hydrophobic) hydrocarbon end, and a polar (hydrophilic) end that is usually ionic. The non-polar ends of the molecule surround the tiny oil droplets and are partially dissolved in them (like dissolves like). The polar ends of the molecules, which are extremely soluble in water, solubilize or emulsify the entire droplet.

Soaps do not work well in hard water because the divalent cations of dissolved minerals (Ca^{+2} , Mg^{+2} , and Fe^{+2}) form precipitates with the carboxylate (fatty acid) anions of soaps. Consequently, a scum of calcium stearate (and other salts) is found as a ring in the bathtub or as a dingy film on clothes, hair, and skin.

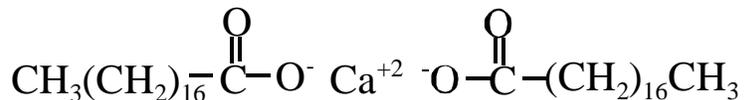


Figure 4 Calcium stearate (a scum in hard water)

On the other hand, the alkyl sulfate and alkyl sulfonate anions of detergents do not form precipitates with these cations, so they are quite effective in hard water.

In today’s experiment you will prepare a potassium soap by the saponification of a fat with potassium hydroxide. You will use the potassium soap to make a sodium soap and

to obtain a mixture of fatty acids. You will also compare some of the properties of soaps and detergents.

Hydrophilic end ▲ Hydrophobic end ●

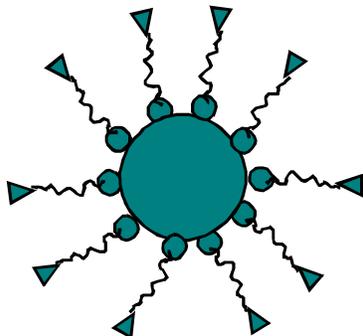


Figure 5 A soap micelle. Soap molecules surround the oil droplet. Their hydrocarbon (hydrophobic) ends are attracted to the oil, while the hydrophilic ends are left on the exterior. The entire micelle now appears to be hydrophilic, and dissolves in water.

Procedure:

A. Saponification of a fat; preparation of a potassium soap.

Mass about 1.5 g of solid fat (tallow, lard, or shortening) in a large test tube. (It is not necessary to force the fat to the bottom of the test tube, since it will melt and run down when the test tube is heated.) Add 10 mL of a 10% solution of KOH in 95% ethyl alcohol. Place the test tube in a 250 mL beaker half-filled with boiling water. Replace any alcohol that evaporates with ethyl alcohol. (Remember that alcohol is flammable)

After heating the tube for 15 minutes, test for completeness of saponification by adding a few drops of the reaction mixture to water. Do you see droplets of fat? If not, saponification is complete. If droplets of fat are visible, continue to heat the tube for another 15 minutes and test again.

When the saponification of the fat is complete, pour the contents of the tube into a 100 mL beaker. Cautiously evaporate the alcohol until the residue becomes viscous and tacky; do not overheat it or the soap will darken. Now add 30 mL of distilled water and heat the mixture briefly, while stirring it until a solution is obtained. The product is a solution of potassium soap.

B. Properties of Soaps and Detergents:

Test a 1-mL portion of your potassium soap solution for sudsing action. Smear some of the starting fat on the bottom of a watch glass, and determine whether your potassium soap solution removes the grease. Perform the same tests with a little of your sodium soap added to water.

To 1 mL portions of your potassium soap solution in three separate test tubes, add 1 mL of each of the following solutions: Calcium Chloride (0.1%) Magnesium Chloride (0.1%), and Iron(II) Chloride (0.1%). Stir the solutions. Do precipitates form? Repeat this test with a synthetic detergent such as Tide™ or Dreft™. Prepare a solution of the detergent by dissolving 0.5 g of detergent in 50 mL of distilled water.

Now mix equal portions (1 mL of each) of your potassium soap and tap water. Does a precipitate form? Repeat with the detergent solution.

Post-Lab questions:

1. What is the difference between a detergent and a soap?
2. Why do detergents typically not form soap scum?
3. What is a micelle?
4. Indicate the polarity differences that occur in a soap molecule? (Draw a picture.)
5. What is a saturated fatty acid?
6. What is an unsaturated fatty acid? (Are double bonds cis or trans?)
7. List the percentages and draw the major fatty acids that make up Butter? Olive oil?
8. Draw a mechanism for the saponification. (Just one acid will be fine.)