

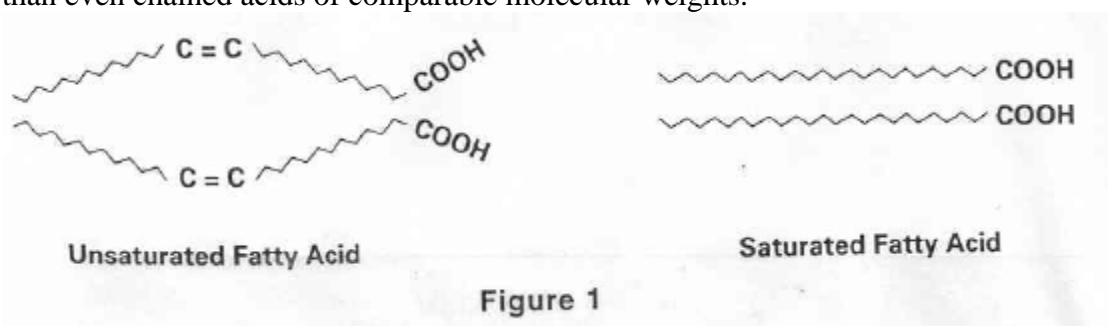
Preparation of Soap

By Walter Scharf and Charles Malerich
Natural Sciences/Chemistry
Baruch College
New York, NY 10010

Introduction

Soap, from a chemical standpoint, is a salt (or a mixture of salts) of fatty acids. As with all salts, soap contains a positive ion, usually Na^+ or K^+ , and a negative ion, usually the anions of long-chained carboxylic acids obtained by the hydrolysis of animal or vegetable fats. These fatty acids, of which there are about 20 naturally occurring members, are carboxylic acids containing 14, 16, or 18 carbon atoms in an unbranched chain. The even numbered chains result from the fact that fats are synthesized in cells by the polymerization of a 2-carbon acetate unit.

In addition to the normal (straight chain) saturated acids, there occur several with hydroxyl groups, and/or one or more double bonds in the carbon chain. the presence of unsaturation (double bonds) in molecules of fatty acids, fats, or soap tend to lower the melting point of these compounds and to cause them to be in the liquid state at room temperature. Thus, vegetable fats are relatively unsaturated and liquid under the ordinary conditions, while animal fats, being relatively more saturated, are solid, or semi-solid, at the same temperature. For this reason, vegetable fats are commonly referred to as vegetable oils. (We say relatively saturated, or unsaturated because both vegetable oils and animal fats contain saturated and unsaturated chains). The reason why double bonds lower the melting point of a fatty acid chain, is that the sections of the chain attached to the double bond are attached cis-wise to each other (probably because the double bond cis configuration produces a bent chain which does not easily adhere to a neighboring chain by a Van der Waals attraction hence a lower temperature (lower mobility) is required for these molecules to adhere in order to form a solid crystal lattice. It is interesting to note, that straight chained carboxylic acids having an odd number of carbon atoms melt lower than even chained acids of comparable molecular weights.



The most commonly encountered fatty acids in natural fats are given below:

Saturated Acids

Lauric Acid (Dodecanoic Acid)



Myristic Acid (tetradecanoic Acid)



Palmitic acid (hexadecanoic acid)



Stearic acid (octadecanoic acid)

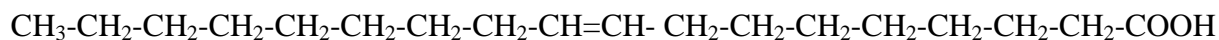


Unsaturated acids:

Oleic acid (octadec-9-enoic acid)

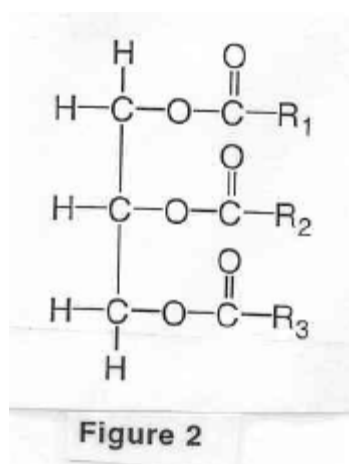


Linoleic acid (octadec-9, 12-dienoic acid)



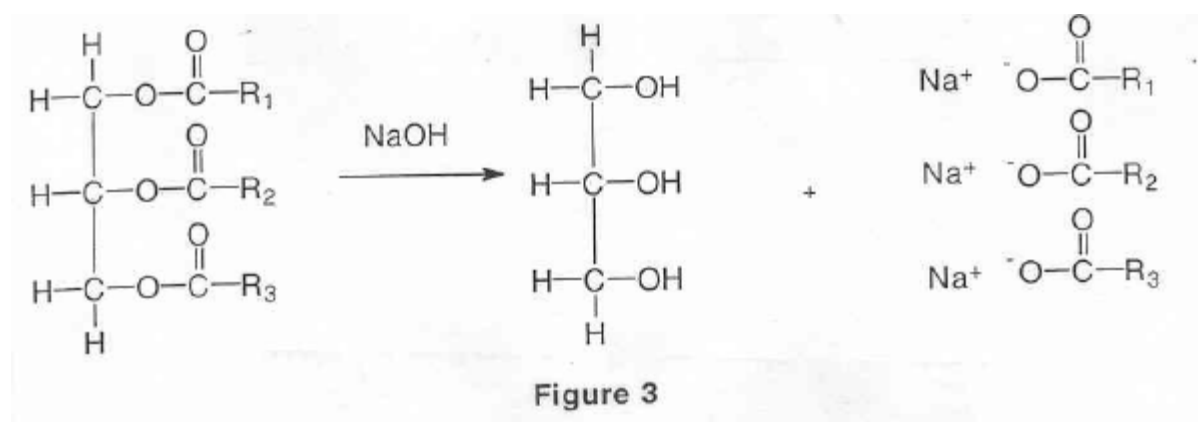
All fats are esters (though, not all esters are necessarily, fat!) of the trihydroxy alcohol, glycerin, and fatty acids. Since each molecule of fat contains three ester groups, fats are often referred to as triglycerides to distinguish them from other materials having a fatty or greasy texture, such as waxes (mono esters of long chain alcohols are carboxylic acids) mineral oil, and petroleum jelly (long-chain hydrocarbons).

If R-COOH represents a generalized fatty acid (R is a hydrocarbon chain of 13, 15, or 17 carbons) then, the general formula for a fat is



Note that the fatty-acid residues in a typical fat molecule are usually different! Fats from different sources have different percentages of the common fatty acids, and can be distinguished from one another by a quantitative analysis of these constituents.

Soap is prepared by hydrolyzing a fat under alkaline (basic) conditions. The reaction is called saponification, and produces one molecule of glycerin and three molecules of soap, for each molecule of fat taken.



The fats and oils most commonly used in soap preparation are lard and tallow from animal sources, and coconut, palm and olive oils from vegetable sources.

Tallow and lard are the fats separated from solid beef fat (suet) and hog fat, by the process of rendering. Rendering consists of boiling the animal fat in a pot with an equal volume of water until a layer of melted fat collects on top. (when the animal tissue is heated, the fat in the cells melts, expands, ruptures the cell membrane- and then flows out. Since the fat is immiscible with water, and has a lower density, it forms a floating layer on the top of the water in the pot.) the rendered fat is then drawn off, and allowed to solidify by cooling.

The length of the hydrocarbon chain and the number of double bonds in the carboxylic-acid portion of the fat or oil determine the properties of the resulting soap. For example, a salt of a saturated, long-chain acid makes a harder, more insoluble soap. Chain length also affects solubility. Tallow is the principal fatty material used in soap making, with coconut or palm oil

blended in, to produce a softer (more soluble) soap which will readily lather. Soaps made with KOH (caustic potash) instead of NaOH (caustic soda) are liquid rather than solid, at room temperature. (potassium salts have a lower melting point than sodium salts.)

Toilet soaps are generally carefully washed free of any remaining alkali used in the saponification procedure to avoid irritation and drying of the skin. As much glycerine as possible is usually left in the soap, and perfumes, coloring, and medicinal agents are sometimes added. Also added, are antioxidants to prevent the soap from turning rancid. Many soaps are superfatted, i.e. not all of the fat is hydrolysed, in order that the final soap have some fat remaining to lubricate and smooth dry and sensitive skin.

Besides tallow and coconut oil, there are many other natural fats and oils which can be economically employed to make soaps of varying degrees of quality. The less pure the raw material is, the more likely the soap will have an off-color (generally yellow or brown), and an undesirable odor. (impure fats are sometimes treated with bleaching agents before saponification, but this adds to their cost.) Examples of such other starting materials in soap manufacture are: coconut oil, palm oil, olive oil, cottonseed oil, sesame oil, corn oil, Soya bean oil, hydrogenated vegetable oils, rosin, naphthelinic acids, and greases made from animal skins, bones, tankage and garbage. The lower grades of soaps are used as yellow laundry soap, soap powders, cleansers, and industrial soaps.

Below is a short glossary of terms employed to describe special types of soap:

Castile soap -- a mild soap originally made in Spain with pure olive oil. Today many "castile" soaps are made with other vegetable oils. Castile is a marvelous cleanser, producing a rich lather.

Cream soaps – soaps containing cold cream materials, moisturizers and emollients. Cream soaps are particularly good for dry and delicate skin

Deodorant soaps – soaps to which antibacterial agents have been added to reduce odor-causing bacteria.

Floating soaps – soaps having air bubbles incorporated to lower the density. This causes the bar to float.

Hypo-allergenic soaps – Mild formula soaps, low in potential irritants. They usually produce a poor lather.

Medicated soaps – soaps containing medications such as tar, sulphur, or antibacterial ingredients, used to treat acne and other skin disorders. Medicated soaps require thorough rinsing and are not recommended for sensitive skin.

Milled soaps – these are the most commonly used, mass produced soaps. Milling refers to the mixing of color, perfume and soap flakes.

Oatmeal soap – A rough –textured soap to which oatmeal has been added as a mild abrasive and lather. Good for oily, dry, and normal skin.

Procedure

Two procedures for soap making are given, a “cold” and a “hot” process. The cold process, suitable for homemade soaps, produces soap bars which retain the glycerine by-product, and if the amount of alkali employed in the saponification is limited, an excess of fat (superfatted). The hot process, more suitable for laboratory or industrial preparation, yields a more chemically pure powder; by-products and excess starting materials are separated.

I. Cold process

You must wear goggles or glasses.

When using a thermometer in this experiment, never use it to stir liquids. Instead, use a fire polished glass rod. to properly measure the temperature of a liquid, hold the thermometer so that its bulb is suspended in the center of the liquid while reading the mercury level. If the liquid is being heated on a hot plate, do not the thermometer bulb to rest on the bottom of the container, as it will then be overheated. Remove the thermometer from the container after each reading. Note: the thermometer supplied does not require shaking down before or after reading. Plug in a hot plate and set to high.

Weigh a 150 ml beaker on the trip scale and add 8g of NaOH pellets to it. Handle NaOH with care as NaOH can burn the skin and is especially harmful to the eyes. In the hood, add 25 ml of cold water to the beaker. Stir the mixture of NaOH pellets and water, until a clear solution results. Caution: the beaker will become very hot as the NaOH dissolves.

Weigh a 250ml beaker on the trip-scale and add 58g of fat (Crisco vegetable shortening works well) to it. Then place the beaker on the hot plate with low heat and with occasional stirring, melt the fat to melt completely. Warm the melted fat to between 40-50 °C. Remove the fat from the hot plate and add the lye solution to the fat with stirring. Stir the fat and NaOH mixture continuously and until an emulsion is formed. Your mixture should look like a thick, light yellow milk shake and should stay emulsified (should not separate into a fat and NaOH layer). If your emulsion separates, the fat is too hot and needs to cool. Let the mixture cool on the bench top with occasional stirring until an emulsion, which does not separate, is formed. You may want to add perfume or other additives at this point. Pour the emulsion into a plastic cup and place in your drawer for the reaction to run (it takes about 24 hours for the reaction to be complete). Take the soap home with you and let age for about two weeks. During this time a powdery layer of NaCO₃ (soda ash) will form on the surface, as residual NaOH reacts with CO₂ in the air while the soap is drying. This powdery layer should be sliced off and the soap is ready to be used.

II. Hot process

You MUST wear goggles or glasses!

Plug in a hot plate and set it on HIGH. Half fill a 400 ml beaker with hot water from the sink and place on the hot plate. When the water begins to boil, adjust the heat, so that the water boils gently, but continuously. While waiting for the water to boil, weigh a 250ml Erlenmeyer flask on a triple beam balance and add 10g of fat (Crisco, spry, or lard) to it. In a 150ml beaker prepare 100 ml of a 50-50 solution of alcohol-water, by mixing 50 ml of water and 50 ml of 95% ethanol or methanol. Weigh another 150 ml beaker on the triple beam balance, and add 10g of NaOH pellets to the beaker. Handle NaOH with care as NaOH can burn the skin and is especially harmful to the eyes. In the hood, mix the NaOH with 36 ml of the of the 50-50 alcohol-water solution to it. Stir the mixture until a transparent solution is formed. Caution: the beaker will get very hot as the lye dissolves.

Pour the lye solution into the Erlenmeyer flask containing the fat and mix well using a stirring rod or by swirling with a beaker tongs. Clamp the Erlenmeyer in the boiling-water bath and with occasional stirring, allow it to cook for at least 30 minutes. While the mixture boils, some foam will form (due to soap formation). Try to minimize excessive foaming, by adding small adding small portions of your alcohol-water solution. The reaction is complete when oil globules are no longer visible when the reaction mixture is stirred.

Half fill a 600 ml beaker with 300 ml of clear (filtered) saturated salt (NaCl) solution and 50 ml of water. Pour the still hot reaction mixture containing soap, glycerine, excess NaOH, and alcohol into the salt solution. Stir the resulting mixture and allow to stand for 5-10 minutes. The soap will collect as a white layer on the surface of the salt water in the beaker.

Prepare a suction trap from the vacuum flask, which will be used later to filter the soap preparation. Bend a long piece of glass tubing into a right angle (instructor will demonstrate), and push one leg of the bend through a one-hole rubber stopper fitted for the mouth of the flask. When inserted, the glass tube should extend to about ½ inch from the bottom of the flask. The exposed leg of the bend should be cut off at about 3 inches from the bend, and, by means of a piece of rubber connector tubing, be attached to a 12-inch length of glass tubing. The side-arm of the vacuum flask is now connected to the vacuum outlet with pressure tubing. When the suction is turned on, this device will act like a vacuum cleaner, sucking up liquid instead of dust.

Be sure the suction is OFF, before proceeding. Bring the beaker containing the soap preparation next to the suction trap, and place the “spout” of the trap into the beaker having it touch the bottom. Slowly turn on the suction, and watch the bottom (aqueous) layer being drawn into the vacuum flask. Draw off most, but not all, of the bottom layer. Be ready to stop the suction instantly, if it appears that some of the soap crystals are being sucked up. You can stop the suction quickly by pulling the pressure tubing away from the outlet, then turning off the vacuum. Caution: At no time should the vacuum flask be allowed to fill more than half-the trap, and pour out its contents.

When most of the aqueous layer in the beaker has been drawn off, filter the remaining mixture of soap and liquid through a Buchner funnel (with filter paper attached) to the suction flask. Wash the crystals on the filter with 5 ml portions of ice-cold water. Press out any remaining moisture from the cake of filtered crystals on the funnel with the flat end of a clean cork, or the clean bottom of a small beaker. Allow the product to suck dry for 10-15 minutes.

Turn off the suction, detach the Buchner funnel, and with the help of a clean spatula, transfer the soap crystals to a large sheet of smooth paper. Pick up the sheet and slide the crystals into a clean, dry 150 ml beaker. Leave the beaker stand, uncovered in your drawer for several days to air-dry the product.

Weight the soap powder, determine the yield, and hand it to your instructor.

Report Sheet Cold Process

Name _____ lab section _____ Date _____

Type of fat used _____

Weight of fat taken _____ g

Weight of NaOH used _____ g

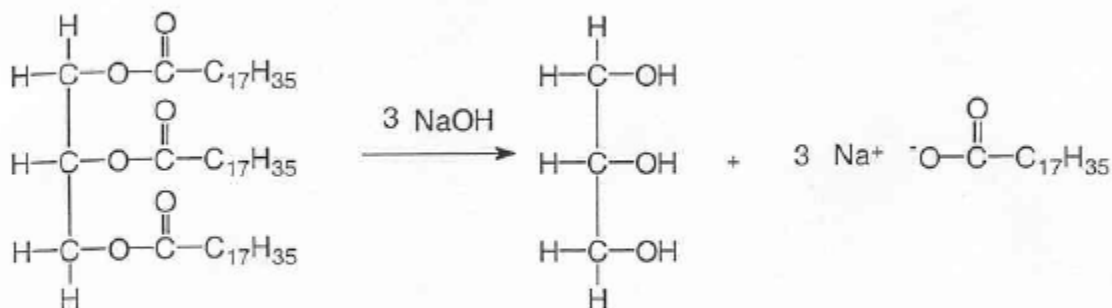
Weight of soap formed _____ g

Use the weight of fat taken and the reaction to find the theoretical yield (weight) of soap bar?

Theoretical yield _____ g

% yield = (actual yield (weight of soap formed)/theoretical yield) X 100= _____ %

Saponification Reaction of Glyceryl Tristearate



Weights: 854 amu

3 x 40 amu

92 amu

3 x 306 amu

Report Sheet Hot Process

Name _____ lab section _____ Date _____

Type of fat used _____

Weight of fat taken _____ g

Weight of NaOH used _____ g

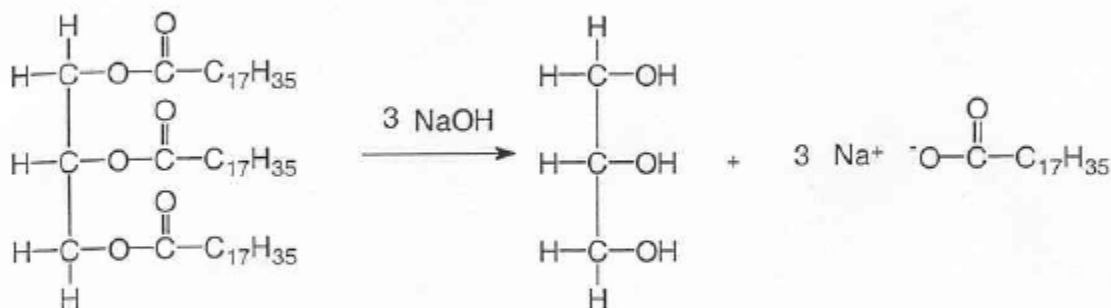
Weight of soap formed _____ g

Use the weight of fat taken and the reaction to find the theoretical yield (weight) of soap bar?

Theoretical yield _____ g

% yield = (actual yield (weight of soap formed)/theoretical yield) X 100= _____ %

Saponification Reaction of Glyceryl Tristearate



Weights: 854 amu

3 x 40 amu

92 amu

3 x 306 amu

Questions

- 1) Why do long chain fatty acids melt higher than short chain acids?
- 2) Why may you use the same weight of fat in your soap preparation regardless of the type of fat-saturated or unsaturated- employed?
- 3) Why are vegetables fats (oils) liquid, while animal fats are solid?
- 4) Why are naturally occurring fatty acids even-chained?
- 5) Why do waxes, Vaseline and mineral oil all feel greasy like fats, even though they are not triglycerides?
- 6) What is soda ash? How does it form? Why is it useful in water softening?
- 7) Using the same weights of fat and alkali, would you get more or less soap by using KOH instead of NaOH?
- 8) Why can you unclog a kitchen-sink drain by pouring lye down into it?