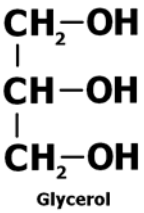
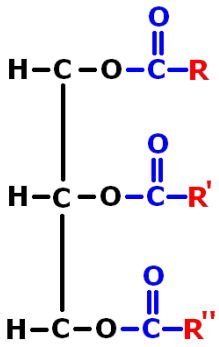
Preparation and Comparison of Soaps
Minneapolis Community and Tech. College
C1152 Principles of Chemistry II
v.4.16

I. Introduction



NOTE: The laboratory quiz contains questions about the saponification mechanism. View & study the saponification video available on the Principles of Chemistry 2 laboratory website before coming to lab.



Vegetable oil, like all animal and vegetable fats, are made up of a mixture of triglycerides. Triglycerides are esters of glycerol that contain long hydrocarbon chains (**R**, **R'** and **R"**) whose lengths vary (figure at right). Biologically, their primary purpose is chemical energy storage.

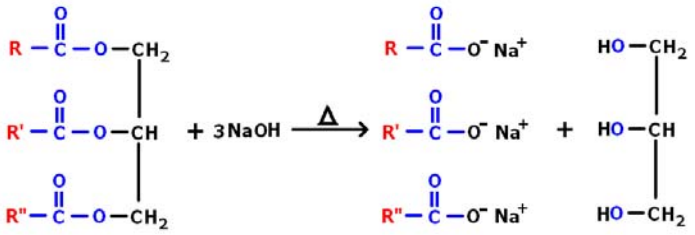
When the R groups contain only singly bonded carbons, they are said to be saturated. R groups containing one or more doubly bonded carbons are said to be monoUNsaturated and polyUNsaturated respectively. Because of their double bonds, the unsaturated hydrocarbon chains exhibit "bends" that prevent adjacent chains from approaching too closely. Thus the intermolecular forces between unsaturated chains is limited and a lower melting point temperatures observed. Unsaturated fats are usually liquids at room temperature while saturated fats, whose molecules can get closer, have higher melting point temperatures and are found in the solid state at room temperature.

The "R" group can be any fatty acid. In the table below are typical examples of fatty acids whose "**R**" group contribution is shown in **red.**

Fatty Acid	Structural Formula	Formula	Molecular Weight (g/mol)
Butyric Acid	$CH_3(CH_2)_2COOH$	$C_4H_8O_2$	88.10
Caproic Acid	$CH_3(CH_2)_4COOH$	$C_6H_{12}O_2$	116.16
Capric Acid	$CH_3(CH_2)_8COOH$	$C_{10}H_{20}O_2$	172.26
Lauric Acid	$CH_3(CH_2)_{10}COOH$	$C_{12}H_{24}O_2$	200.32
Myristic Acid	$CH_3(CH_2)_{12}COOH$	$C_{14}H_{28}O_2$	228.36
Palmitic Acid	$CH_3(CH_2)_{14}COOH$	$C_{16}H_{32}O_2$	256.42
Linoleic Acid	CH_3-$(CH_2)_4$-$(CH$=CH-$CH_2)_2$ -$(CH_2)_6$-$COOH$	$C_{18}H_{32}O_2$	280.45
Oleic Acid	CH_3-$(CH_2)_7CH$=$CH(CH_2)_7COOH$	$C_{18}H_{34}O_2$	282.46
Stearic Acid	CH_3-$(CH_2)_{16}COOH$	$C_{18}H_{36}O_2$	284.47
Ricinoleic Acid	$CH_3(CH_2)_5CH(OH)CH_2CH$=$CH(CH_2)_7COOH$	$C_{18}H_{34}O_3$	298.46
Erucic Acid	$CH_3(CH_2)_7CH$=$CH(CH_2)_{11}COOH$	$C_{22}H_{42}O_2$	338.56

Information compiled from Wikopedia and The Soapmaker's Companion by Susan Miller Cavitch (ISBN# 0-88266-965-6) Storey Publishing, Copyright 1997

In today's experiment, we will hydrolyze the triglycerides found in vegetable oil with concentrated sodium hydroxide to make soap. The reaction occurs when the δ- side of OH^- is attracted to the δ+ of the carboxylic **C**arbon atom:

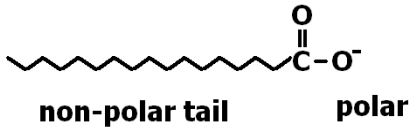


a triglyceride ***3 soaps*** ***glycerol***

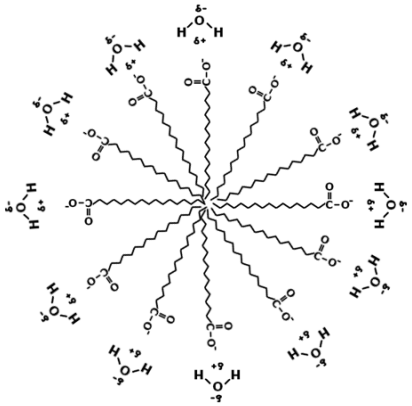
Below is a breakdown of three common oils and the fatty acids they contain. Longer, saturated hydrocarbon chains will produce soaps where significant intermolecular forces exist. In these cases, a more solid soap should be observed.

	Arachidic (313)	Stearic (284)	Oleic (282)	Linoleic (280)	Linoleic (280.4)	Palmitic (256)	Palmitoleic (254)	Myrstic (228)	Lauric (200)
Corn oil	0	3.0	49.6	34.3	0	10.2	1.5	1.4	0
Olive Oil	0.1	2.3	84.4	4.6	0	6.9	0	trace	0
Soybean Oil	0.9	2.4	28.9	50.7	6.5	9.8	0.4	0.1	0.2

The soap molecules that form have long non-polar hydrocarbon chains whose length is determined by the original "R" group. The soap molecule also has a polar end associated with the COO^- group.

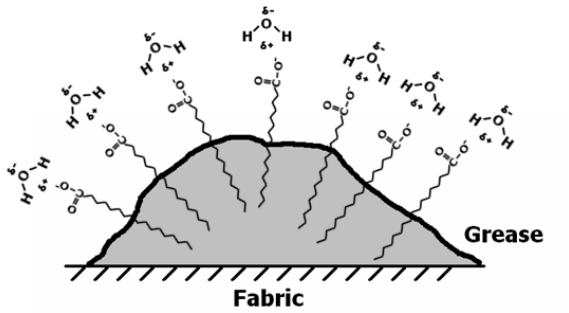


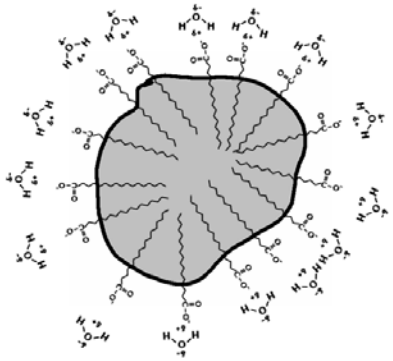
Soap, Water and Cleaning



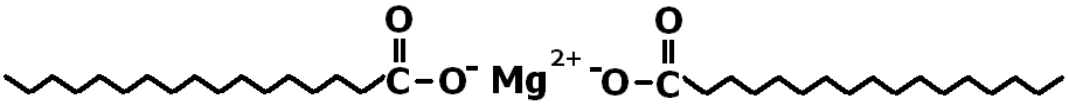
In an aqueous (and therefore polar) environment, the soap molecule's non-polar end is insoluble. This leaves the negatively charged oxygen atom at the end to interact with the surrounding water molecules (figure at right). This end is said to be hydrophilic (*water loving*). The hydrophobic (*water hating*) non-polar tails are forced inward, left to interact only with themselves. The spherical structure that forms is known as a micelle.

Water by itself cannot effectively clean grease from clothing and surfaces since the grease is hydrophobic. In these situations, soap is used to bridge the gap between the non-polar grease and the polar water solvent. The soap molecules imbed their hydrophobic ends in the grease leaving the hydrophilic ends free to interact with water. A micelle is formed with the grease droplet at its center that can now be rinsed away.





Soap scum forms when soap is used in water containing divalent (2+) cations. Two soap molecules are attracted by these ions creating a structure that is essentially non-polar (figure below using Mg^{+2} as the divalent cation) . As it is non-polar, it precipitates out of the polar water solvent as a solid (a.k.a. soap skum).



Water softeners exchange Na^+ ions for the divalent Ca^{2+} and Mg^{2+} ions eliminating the formation of these structures thus improving the cleaning ability of soaps.

Procedure

Warning: Sodium Hydroxide is corrosive and will produce burns to the eyes and skin. Use eye protection at all times and avoid contact with any material that may contain NaOH.

Construct a hot water bath using a 600 mL beaker. Choose one of the several oils (or bring one from home) available in the lab, record its name in your notebook and dispense 15 mL in a clean 150 mL beaker.

Add 10 mL of ethanol and 20 mL of 6 M sodium hydroxide to the reaction container. Note the liquid level on the side of the beaker as well as the number of liquid layers that initially form in your notebook.

Secure the beaker in the hot water bath using a ring stand, utility clamp and rubber band.

Heat the water bath at the highest setting available on the hot plate. As soon as you observe tiny bubbles rising from the solution turn the hot plate down the 300 setting. Vigorous heating is required to obtain the soap product. It is impossible to cook the mixture too long but don't let it boil over into the water bath.

Heat the mixture for a total of 30 minutes <u>after the reaction mixture comes to a boil</u>. Stir continuously with your glass stirring rod.

If necessary, reduce heat to the reaction mixture by moving the clamp/beaker out of the hot water bath.

Maintain the liquid level in the reaction container throughout the heating process by adding small amounts of 1:1 ethanol/distilled water as needed (already mixed and available at your bench top).

Turn off the hotplate, add 25 mL of distilled water to the beaker and place the beaker on the bench top to cool for about 5 minutes. Label the 150 mL beaker and then place it in the Styrofoam ice chest for 10 minutes.

At the end of the soap cooling process, decant any excess liquid from the 150 mL beaker being careful not to discard the soap layer.

Now add 50 mL of **cold** saturated NaCl solution to the soap mixture and stir thoroughly with a glass stirring rod. Use the entire bottle of $NaCl_{(aq)}$ but don't pour in the solid salt at the bottom.

You may at this point add up to 2 drops of essential oil to your soap mixture to make it smell good. We have a collection of essential oils available in the laboratory or you may bring your own. Stir well.

Use the Buchner funnel apparatus to separate the soap from the residual liquid. Two Kim Wipe tissues (one on top of the other) will be used instead of filter paper. Leave the tissue corners hanging out of the funnel to make it easier to remove the tissue & soap after the following washing steps. Removal of your product from the 150 mL beaker can be aided by the use of your rubber policeman.

While filtering, slowly wash the soap twice with small portions of ice cold distilled water available at the filtration stations. Dry the soap for several minutes by drawing air over the product using the Buchner filtration apparatus and vacuum pump.

When no further filtration is necessary, remove the soap cake from the Buchner funnel and pat dry with paper towels. Avoid touching the soap cake directly with your fingers (Why? What is present?). Record the physical characteristics of the soap cake in your lab notebook (consistency, color, texture, etc).

Press your soap into a soap mold. If your soap is especially crumbly, you will have to warm/melt it slightly in the microwave oven before transferring it to the mold. Do this in 5 second increments. Typically 10 – 15 seconds are required to melt the soap granuels together. Cool the soap bar in the Styrofoam ice chest.

Soap Evaluation

Place approximately 5 mL(approximately half full or the top level of the test tube rack) of distilled water in a test tube (13x100 mm). Use a semi-micro spatula to extract a small soap sample and add it to the test tube. Stopper the test tube and shake thoroughly.

Obtain a drop of the solution that forms at the end of a clean stirring rod and apply it to a piece of pH paper that you have placed on a watch glass using a tweezers.

Compare the color of the paper to the color key provided and determine the approximate pH of the soap solution. Repeat this procedure using a small amount of commercially available bar soap.

Record all observations in your lab notebook.

Prepare three clean 13 x 100 mm test tubes as follows (half full):

- Test tube #1: ~5 mL distilled water
- Test tube #2. ~5 mL tap water
- Test tube #3: ~5 mL 1% calcium chloride solution (Very hard water)

Use a semi-micro spatula to add a small piece of your soap to each test tube. Stopper and shake each thoroughly. *(Be sure to rinse the stopper after each use)*

Carefully compare the three test tubes and record your observations. Pay special attention to the amount of lather or foam that forms for each and the appearance of the solution.

Acknowledgements:

This procedure is based on an experiment created by Dr. Michael Eugene Pugh, Bloomsburg University, Bloomsburg, PA 17815

Thanks to Emily Arachtingi and Tom Ofstad for their help testing the recipe.