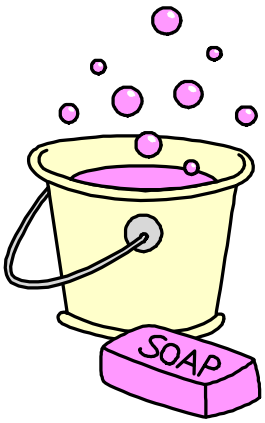
SAPONIFICATION:
Soap Making, Old Fashion Style

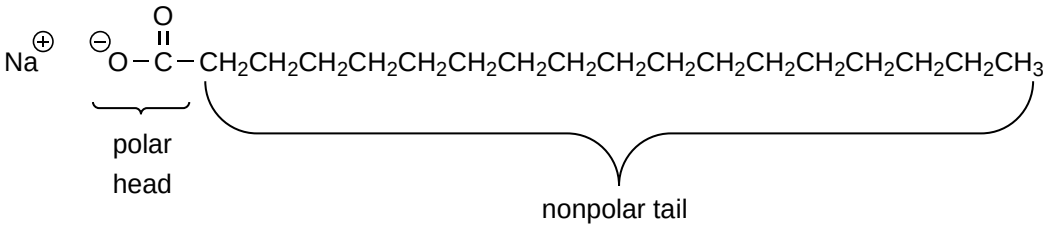


INTRODUCTION

Soap formulations have been known to exist since at least the second century AD. Frontier settlers dissolved wood ashes in rainwater and evaporated this mixture over a fire to make a concentrated solution known as “lye” (NaOH). This was mixed with waste animal fat and heated to form an impure but effective “lye” soap.

Modern “soap” formulations include synthetic detergents made from organic molecules that are less reactive towards the ions of hard water than lye soap and are less strongly basic on our skin. Other chemicals can be added to soap such as builders, bleaching agents, and enzymes. All of these can enhance cleansing or perceived cleansing actions.

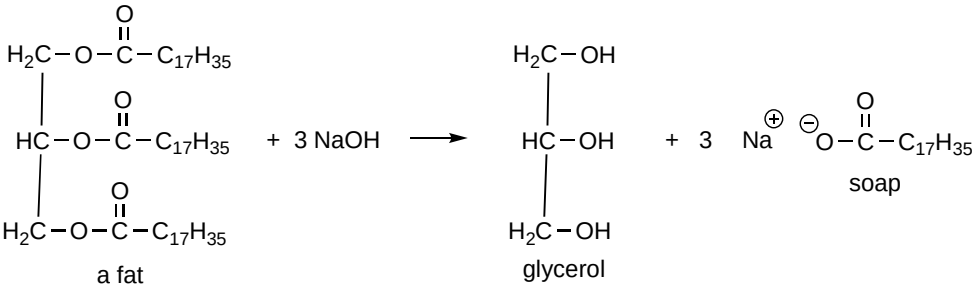
According to the rule “like dissolve likes”, soaps are able to clean because they have both a polar "head" (dissolves in water) and a nonpolar "tail' (dissolves in oils).



This soap is really a salt. The tail can be made of varying lengths and side branches, but should dissolve in nonpolar substances such as oily materials. While the tail gets into the dirt, the head dissolves in the water. Thus, emulsification of the "dirt" takes place, i.e. the dirt is suspended in a water solution.

Today you will make your own lye soap using a saponification similar to what our frontier settlers made. Fats and oils are triesters of glycerol, and they react with a strong base to form the sodium salt of the fatty acid, as shown here.

A saponification reaction:



TECHNIQUES

How to Decant

1. Allow the solid to settle to the bottom of the beaker. Setting the nonspout side of the beaker on a stirring rod or spatula will let it settle in the area below the spout. This will allow you to pour off more liquid.

2. Place your clean stirring rod in front of the spout of the beaker. Pour your solution down the stirring rod into a second beaker. Do this slowly so as not to disturb the settled solid.

SAFETY AND DISPOSAL

- Sodium hydroxide solution can burn your skin. If any should be spilled, first add water, and then wipe it up completely. If any should come in contact with your skin, wash with lots of water. Be very careful to wear your goggles at all times since the solution can splatter a good distance. Remove the beaker from the hot plate before the splattering becomes excessive.

- Use the beaker tongs (NOT crucible tongs) provided to handle hot beakers.

- Exercise caution around the hot plates. They remain hot for a period of time even after they have been turned off.

- Liquid waste may be flushed down the drain with lots of water. Solid waste may be placed in the trash.

- The lye soap should be discarded in the trash can when you are finished.

EXPERIMENTAL PROCEDURE

will appear to remind you of potential dangers and hazards.

will appear to indicate helpful hints, additional information, or interesting facts.

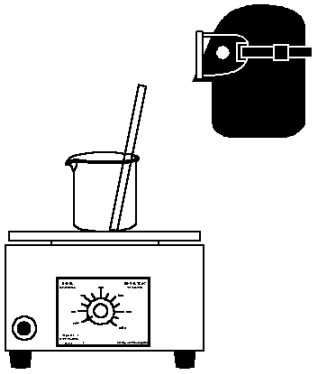
I. Saponification of vegetable oil

A. Place 10 mL of vegetable oil in a 250 mL beaker. Add 15 mL of 20% sodium hydroxide.

20% NaOH can burn your skin! Use care!

B. Place on a hotplate and heat with medium heat (a setting of 4-5). Stir with a long stirring rod. Hold paper towel around stirring rod to protect your hand. Boil and constantly stir this mixture until the solution has the consistency of peanut butter.

THIS MIXTURE SPLATTERS EASILY, BE SURE TO WEAR GOGGLES AND USE A LONG STIRRING ROD.



C. When the mixture starts to splatter, quickly remove the beaker from the hot plate using **BEAKER TONGS** and let it cool slightly while continuing to stir.

D. Continue stirring, place the beaker back on the hot plate again and boil until thick. The entire procedure may take as much as 1/2 hour.

E. When a peanut butter-like solid begins to form (which becomes harder upon further cooling), saponification is complete. If the mixture cools to a syrupy liquid, saponification is not complete and you must heat and stir again.

F. You may have to add 5 mL more of sodium hydroxide and boil again if the mixture does not thicken after three times.

G. When the saponification is complete, allow the crude soap mixture to cool to room temperature.

DO NOT HANDLE THIS -- IT STILL CONTAINS CONSIDERABLE AMOUNTS OF SODIUM HYDROXIDE. It also contains glycerol.

H. The excess sodium hydroxide and glycerol can be washed away with an aqueous sodium chloride (NaCl) solution.

1. Make the aqueous NaCl solution by weighing 18 g of sodium chloride in a 100 mL beaker. Add 60 mL of water and stir until dissolved.

2. Using your stirring rod, mix a 20 mL portion of solution into the soap. This permits maximum contact between the soap and the solution.

3. Decant the solution from the beaker leaving the soap in the beaker. (See techniques section.) You may also need to hold your watch glass over the top of the beaker to prevent your soap from leaving the beaker.

4. Repeat this process two more times using the rest of your NaCl solution. Remove the last traces of liquid by working the soap on a paper towel and shaping it to your liking.

II. Water hardness: Hard water contains calcium ions.

- To study the effect of calcium ions on a soap solution, take about one-tenth of your soap and shake it vigorously in a test tube with about 10 mL of tap water. (You can prevent the solution from touching your skin by putting a small square of Parafilm over the opening of the test tube before covering that with your thumb.) Record your observations
- Add several drops of 10% calcium chloride solution and shake again. Record your observations
- In another test tube, shake several drops of Alconox detergent with water and record your observations.
- Add several drops of 10% calcium chloride solution to this Alconox mixture and shake again. Record your observations.

Use Alconox -- this is the soap solution normally supplied in lab.

III. Emulsification

- Add 3 or 4 drops of mineral oil to 10 mL of water in a test tube. Shake the tube vigorously and record observations.
- Allow this mixture to stand a minute or so. Record your observations.
- Take another tenth of your solid soap and add it to the tube containing the oil and water, shake vigorously, and record your observations.
- Allow this mixture to stand a minute or so. Record your observations.

IV. Strong base-weak acid property of soap

- Dissolve several flakes or grains of Ivory in 5 mL of water
- Add 1 drop of phenolphthalein or use pH paper. (Ivory is used since your soap may have retained some of the NaOH. This would invalidate the experiment.)
- Record your observations and provide an explanation.