Saponification Laboratory

By Emily Morales

WELCOME!

Welcome to the Organic Chemistry Saponification Laboratory.

The earliest recorded evidence of soap production goes back as long as 5000 years ago in ancient Babylon. No one really knows how anyone figured out that if you combine animal fat with a type of ash, an effective cleaning product resulted. There is speculation that someone may have discovered an odd substance at the sites where animals were killed and cooked; perhaps some of the fat off the animal seeped into ashes and formed what we now know to be soap.

Up until about 150 years ago, many families made their own soaps from products they kept around the farm and home. Ben Franklin's father was a soap and candle maker (both products use the same substance - beef tallow, in their manufacture). Today, large corporations like Proctor and Gamble employ research scientists and chemical engineers to develop very sophisticated cleansing products.

Over the next few weeks, you will be the chemists, as you prepare your own fine quality soaps, in a base-promoted ester hydrolysis reaction. But before you put your chemist hat on (really, goggles and gloves), it is important for you to understand some facts concerning saponification chemistry. Soap making can be dangerous; we will use some very caustic (not to mention interesting) chemicals to make mild, gentle soaps, so you need to be aware of how to handle these safely.

In the coming weeks, we are going to perform a chemical hydrolysis reaction of an ester which produces both a glycerol and carboxylate anion. This carboxylate anion will then combine in an ionic bond with a sodium cation, producing “soap.”

Chemical Reactions

When substances undergo a chemical reaction, they completely abandon many of their own properties and take on completely new ones. The fats and oils we will be using are slippery and will go rancid (stinky) if left at room temperature for very long. Lye or sodium hydroxide, is used commercially with water as a drain cleaner, and in the past has been used to “digest” the tissues of dead animals on farms. It is an extremely dangerous chemical and should be handled with great care. Figure 1 shows how sodium hydroxide is packaged commercially - as a drain opener!

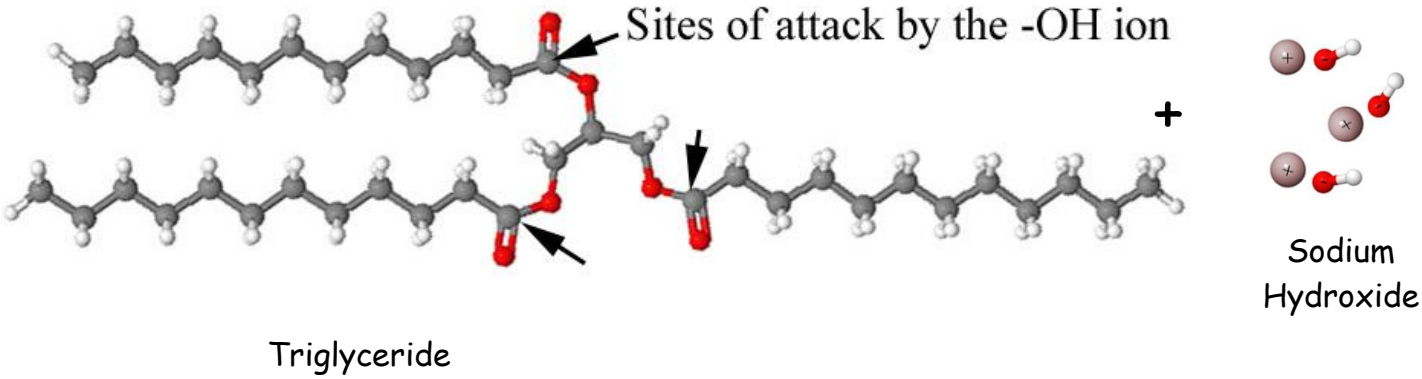
Figure 1. Sodium hydroxide.

Amazingly, when something as caustic as sodium hydroxide is mixed with something as slippery and greasy as hard fats and oils, the products that form result in a useful, high quality soap. In this chemical reaction, sodium hydroxide loses its identity as a caustic substance that can burn your skin and eyes, while fat loses the properties that subject it to easy decay and two

completely new chemicals are formed: glycerol and soap.

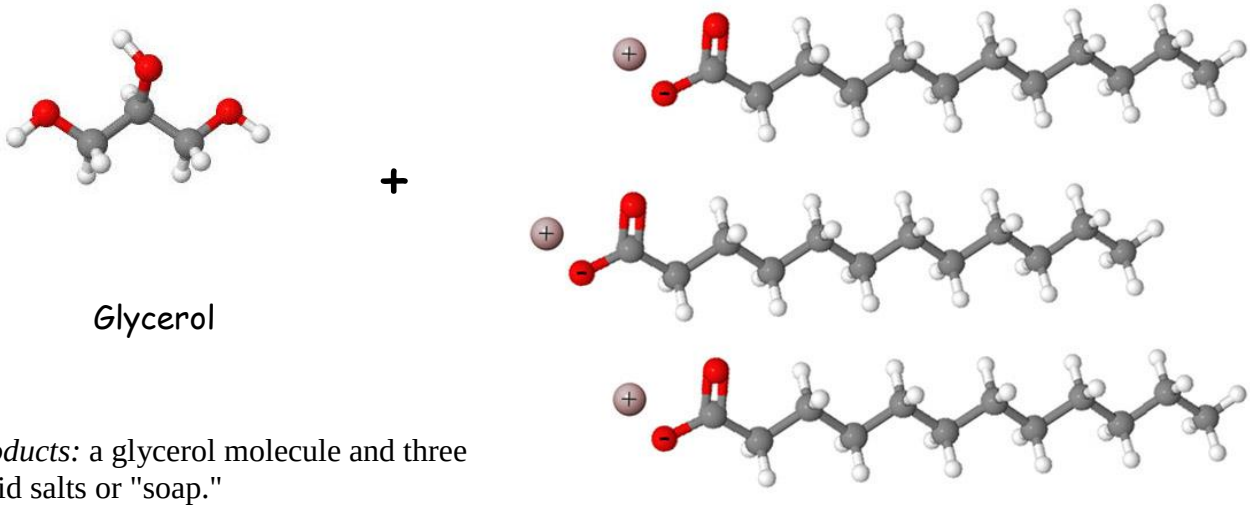
These new products that form will NOT burn your skin if you use them, and will NOT go rancid if left at room temperature! Rather these products combined in a solid bar, leave your skin clean and soft and are almost always left at room temperature!

The chemical reaction between fat and sodium hydroxide can be represented as follows:



Triglyceride

The reactants: triglyceride and sodium hydroxide. The mechanism: In water, the sodium ions (Na^+) dissociate from the hydroxide ions (OH^-), then the three negatively charged hydroxide ions attack the triglyceride at the slightly positively charged points indicated by the arrows. The hydroxide ions end up cleaving the large triglyceride into four new molecules.



Glycerol

Fatty Acid Salt or "Soap"

The products: a glycerol molecule and three fatty acid salts or "soap."

The glycerol is a natural emollient, and the soap has wonderful cleansing properties. Soap owes these properties to the fact it is now an amphipathic molecule – having both hydrophilic and hydrophobic regions, so it loves both oil and water.

The soap-making or saponification reaction typifies what happens when two or more substances undergo a chemical reaction, the reactants (sodium hydroxide, fats) lose their former physical and chemical identities and take on new physical and chemical properties as new products (glycerol, soap).

Procedure 1 - Example of Saponification Reaction in Soap-making

Materials

Liquid Fats:

- 300g Olive Oil
- 80 g Sweet Almond Oil

Hard Fats:

- 250g Coconut Oil
- 100 g Palm Oil
- 70g Cocoa (or Shea) Butter

Lye Solution:

- 250ml Distilled Water
- 105g Sodium Hydroxide

Equipment/ Supplies:

- Ohaus Triple Beam Balance
- Polypropylene Bottles
- Magnetic Stir Plate
- Polypropylene Mold (1.2L)
- Saponification Oven
- Measuring Cups, Spatula
- Instant Read Thermometer

Additives:

- Fragrance oil
- Herbs, cocoa powder, oatmeal

Protocol I - Preparation of Lye Solution

Station L1a: Weigh out 105 g NaOH crystals

□1. Weigh your container

□2. Add 105 to the weight of your container and set the triple-beam balance to that weight.

□3. Slowly pour NaOH crystals into the container until the scale is balanced.

□4. If you spill any crystals, let Mrs. Morales know.

□5. Cap up the NaOH crystals container when done.

Station L1b: Measure out 250 mL of water

□Using the Rubbermaid plastic container, pour 250 mL of distilled water into the container.

Station L2: Mixing the solution

□1. Place water container on magnetic stir plate, and place magnetic stir bar in stirrer.

□2. Turn on the magnetic stir plate to get the bar spinning.

□3. With goggles on, slowly pour lye crystals in the water, allowing each addition to be fully dissolved before adding any more.

□4. When all crystals are dissolved, carefully remove stir bar with another magnet, and rinse off with water immediately. Do not touch this stir bar with your fingers, you could get burned.

□5. Place the red cap on your solution, but keep the tab open until cool.

□6. When done, rinse out the empty container that held the NaOH crystals and place on pad.

Be cautious, your container will get very hot.

Protocol II - Preparation of Hard Fats

Station HF1: Weigh out coconut oil

□1. Place container on scale and weigh.

□2. Add 250 to the weight of your container, and set the scale to that amount.

□3. Using a knife, place enough coconut oil in the container until the scale balances out.

□4. Move to next station.

Station HF2: Weigh out palm oil

□1. Place container on scale and weigh.

□2. Add 100 to the weight of your container, and set the scale to that amount.

□3. Using a knife, place enough palm oil in the container until the scale balances out.

□4. Move to next station.

Station HF3: Weigh out either cocoa butter (warm recipe) or shea butter (cool recipe)

□1. Place container on scale and weigh.

□2. Add 70 to the weight of your container, and set the scale to that amount.

□3. Drop pieces of cocoa butter or shea butter in your container until scale balances out.

□4. Move to next station.

When you have all three hard fats in your container, seal it up and write your name on it. Next week we will weigh out the liquid oils and combine.

The Saponification Reaction

□Step 1. Heat the container with your solid fats in the microwave until they are completely melted. Do not overheat, check after each minute, stir, and reheat if necessary.

"Trace" occurs when about 50 percent of the fats/ oils have successfully reacted with the lye to produce soap and glycerol. At trace, the solution looks more like a milk shake and coats the sides of the bowl like a thick malt.

□Step 2. Pour the melted fats into the container holding your liquid oils, and gently stir the oils, breaking up any solids that may remain. The mixture should be translucent, if it is opaque there are too many solids remaining. Once you know that everything is in liquid phase, set aside and cool to 100˚F.

□Step 3. Before we mix the fats with the lye, make sure you have all dyes, additives (herbs, honey, oatmeal), fragrances, containers for mixing colors, and soap mold at your station.

□Step 4. With goggles on, pour the melted fats into the metal bowl, making sure you empty the container completely. Slowly pour the lye solution into the fats, taking care to not splash any of the lye – it is dangerous. Before you even stir that lye solution, rinse out the lye container in the sink.

□Step 5. With a spatula, carefully stir the lye into the fats until combined. Then, with a stick blender, and with a pulsing action at lowest speed, blend until you see first signs of "trace."

□Step 6. If you want to add colors or additives, this is the time to do it. If not, skip to next step.

Adding Colorant

□Step C1. Divide your soap into containers for the addition of color.

□Step C2. Add colorant and stir with a spatula to incorporate. Keep adding colorant and stirring until you achieve the desired degree of color saturation. Take care to not overstir at this point.

□Step C3. Once all the colors have been added, and you are happy with the color saturation, pulse the soaps in the containers with a stick blender to mix well, and get ready to add fragrance. Set containers aside and move on to the next step.

Cocoa, French clay, ground walnut hulls, sweet paprika, are all treated as colorants.

Additives

□A1. Divide your soap into containers (if necessary)for the addition of additives (herbs, poppy seeds, oatmeal, honey).

□A2. Add the additives and stir with spatula to desired consistency. Set container(s) aside for final step of adding fragrance.

□Step 7. Measure out fragrance oil and cap the bottle immediately (it will evaporate very quickly because it has a low heat of vaporization. Add to your soap. Keep in mind that once fragrance is added, the soap often will react very quickly so have mold ready to pour.

□Step 8. Pour soap into mold, and marble, swirl or decorate according to taste.

Soap embellishment Plan

How many colors do you want in your batch? _____

What colors? _____

Fragrance name: _____ How much? _____ ml

Additives? (mint, poppy seed, oatmeal, honey, rosemary)

_____

How much per 2 lb batch? _____

Anything on top? (oatmeal, poppy seed, oats, rosemary, coffee grounds)

_____