

Experiment 31A

6/28/18

SOAP – A FATTY ACID SALT

MATERIALS: Soybean oil, 6 M NaOH, 100% ethanol, 150 mL beaker (2), 100 mL beaker (2), 10 mL (2) and 50 mL graduated cylinders, Buchner funnel, stir rod, small diameter filter paper, ice-cold saturated NaCl solution, Alkacid pH paper.

PURPOSE: The purpose of this experiment is to make soap from common household materials and study the physical and chemical properties of soap.

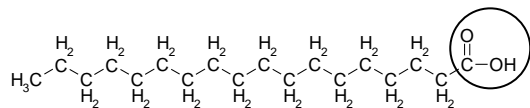
LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate these proficiencies:

1. Use intermolecular forces to explain why liquids are or are not miscible.
2. Apply understanding of molecular structure and intermolecular forces to describe how soap helps to remove greasy dirt in a water solution.
3. Understand features of structure and bonding in everyday organic materials.

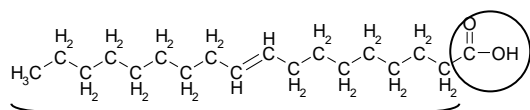
DISCUSSION:

Bathing and cleaning with soap were not always the everyday occurrences that we know them to be. Soap actually dates back to the Sumerians in 2500 BC, and was used sporadically in both Eastern and Western cultures over the ages. In colonial America, soap was typically made as a household chore, formed by combining soda ash (Na_2CO_3) recovered from fire pits with waste kitchen fats. The product was crude and fairly harsh, and was mostly used for cleaning clothes rather than skin. New processes made soap a factory-manufactured product around 1850, widely increasing its availability. However, not until the time of the Civil War were Americans convinced that daily use of soap in their bathing rituals was a good idea. The new focus on cleanliness resulted from the reduced number of war casualties due to disease, as compared to previous wars during which personal hygiene was virtually ignored. As the soldiers returned to their homes, they brought the idea of using soap with them (1). By the early 1900's, soap-making was such a commercial success that the Procter & Gamble Company was spending large sums of money advertising their brands of soap; and today, there are almost countless forms of soaps available (1). Some of them (especially laundry soaps) are even designed to remove calcium and magnesium ions from water (i.e. "soften" the water) to keep them from forming precipitates that would stick to clothing.

As indicated by the title, soaps are salts of fatty acids. Fatty acids are *carboxylic acids*, i.e., molecules with a —C(=O)OH acid fragment (circled in the structures below) attached to a long hydrocarbon tail (denoted below). The hydrocarbon portion could be *saturated*, containing only single carbon-carbon bonds as in stearic acid, or it might be *unsaturated*, as in oleic acid. Unsaturated fatty acids have carbon-carbon double bonds, either in one place (*monounsaturated*) or at multiple sites in the chain (*polyunsaturated*). In either case, the hydrocarbon chain is nonpolar.



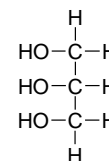
Stearic acid, a saturated fatty acid



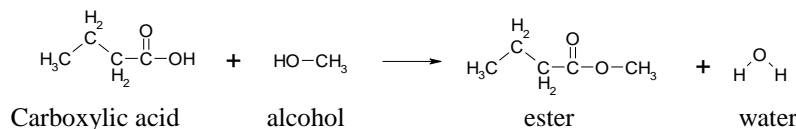
Oleic acid, a monounsaturated fatty acid

Nonpolar hydrocarbon chain

Fatty acids are the first building blocks of soaps. As indicated above, soaps were made from animal fats, and, more generally, can be made from fats or oils of plants or animals. Edible fats and oils are triglycerides, which are esters made from fatty acid molecules and glycerol, $\text{C}_3\text{H}_8\text{O}_3$, a trialcohol. (*Alcohols* are organic molecules with an —OH group. You can see that glycerol has three such segments.)

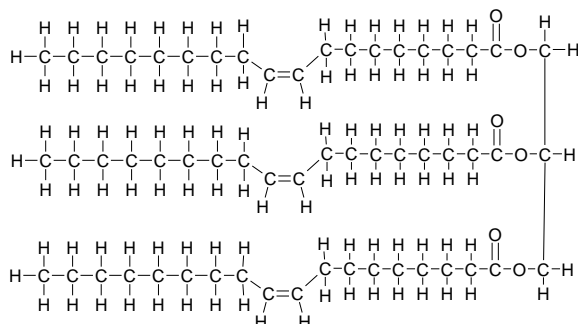


In a reaction that we shall see again in SC112, acids and alcohols can combine to form another class of molecules called esters.



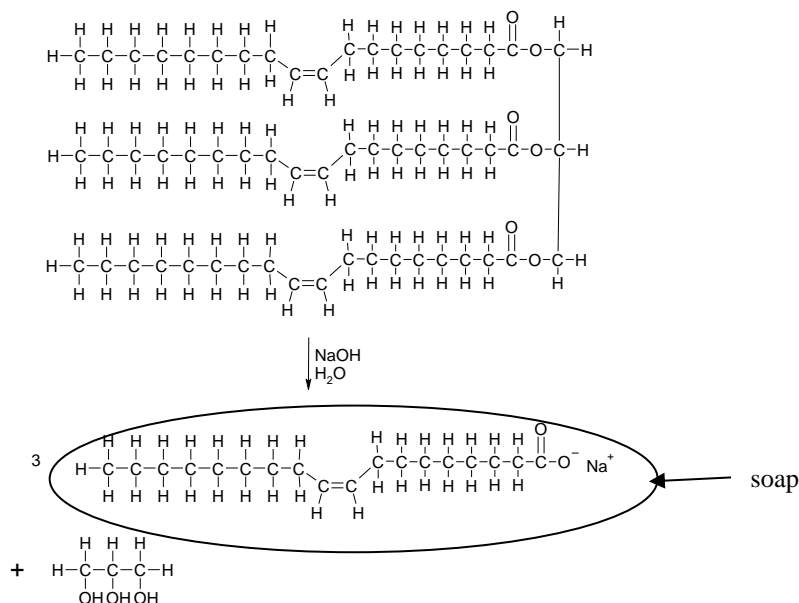
Esters have the general structure of $R_1-\overset{\text{O}}{\parallel}{\text{C}}-\text{O}-R_2$ where the R1 and R2 groups represent any organic moiety. (For example, R2 is CH₃ in the ester shown above.)

Since fats and oils are triglyceride esters, they must be esters made from the reaction of three fatty acid molecules and the glycerol molecule. One such example is the chief triglyceride in soybean oil. Looking at the molecular structure, see if you can identify the fatty acid chains, the glycerol backbone and the ester linkages.



“Oils” are triglycerides that are liquid at room temperature; typically they are composed of unsaturated fatty acids, such as the molecule above. “Fats” are triglycerides that are solids at room temperature; usually these involve saturated fatty acids. The three fatty acids in any triglyceride might be the same, as above, or they might be different.

What about soap-making? Typically, soap is made by the hydrolysis of a fat or oil (a process known as saponification). This is essentially the reverse process of ester formation. This involves reacting the hot fat or oil in an alkaline (basic) solution. Because the reaction takes place in a strongly basic solution, the fatty acid fragments are produced in the ionic form, with protons removed (as shown below), rather than the molecular form shown earlier. Essentially the base ionizes the fatty acid, and you are left with a solution of a salt (and an alcohol). The resulting salt (the soap, circled below) is then precipitated from solution using a saturated NaCl solution (2).



A similar process is carried out in clogged kitchen drains every day. Products like Drano[®] consist mostly of solid NaOH. When poured into a drain, it creates a strongly basic solution which hydrolyzes the grease or fat that forms the clog, rendering it more soluble so it can be washed away. (The shiny strips in Drano are aluminum chips; these produce H₂ by a redox reaction with the water, and the gas bubbles provide pressure and agitation to help the process along. Why should one avoid flames when using such products?)

PROCEDURE: (Work in pairs)

Part A: Making Soap

1. Make an ice bath and cool about 40 mL of saturated NaCl solution and 40 mL of water in separate 100 mL beakers.
2. Assemble your ring stand, small ring, large ring, wire mesh, 150 mL beaker, and Bunsen burner as shown to the right.
3. Using one 10 mL graduated cylinder for oil, and the other for ethanol or NaOH solutions, combine the following reagents in your 150 mL beaker:
 - 12.0 mL of soybean oil (or other oil that is available)
 - 12.0 mL of 6 M NaOH



Caution: NaOH is a strong base and very corrosive to living tissue. If you spill it on your skin, wash it off immediately; if you get it in your eyes, flush with water for at least 15 minutes.

 Answer In-Lab Question #1 on page E31A-5.

4. Now add 5.0 mL of 100% ethanol (C_2H_5OH) to your beaker. Remove any remnants of ethanol from the hood (since it's flammable).
5. After getting permission from your instructor to light a Bunsen burner, heat the mixture **gently** for about 10 minutes stirring constantly with a glass stir rod. When the mixture bubbles rapidly and then thickens to form a gel, the reaction is complete. Turn off the Bunsen burner. The resultant impure soap is a goopy, yellow gel. If your solution foams too much (over the beaker) during heating, the flame is too hot, so remove or lower the flame.
6. At this point 10 to 15 drops of scent oil can be added if desired, with moderate stirring. You can combine this step with Part B step 1.

 Answer In-Lab Question #2 on page E31A-5.

Part B: Purifying the Soap

Your Instructor will demonstrate the use of the Buchner funnel for vacuum filtration. It may not be possible to have more than 3 or 4 filtration set-ups per lab. Follow the directions of your Instructor.


1. Place the beaker containing crude soap in an ice bath for a few minutes.

 Answer In-Lab Question #3 on page E31A-5.

2. Add 40 mL of ice-cold saturated NaCl to the soap solution. Stir well. The soap will separate from the solution.
3. Use a Buchner funnel with filter paper to filter the soap crystals. Once the majority of the liquid has been removed rinse the paste with 10 mL of ice cold water. Again, remove the majority of the liquid and then place the filter paper with soap on a paper towel to allow for additional drying.

 Answer In-Lab Question #4 on page E31A-6.

4. Test the soap and the filtrate collected in the flask with pH paper.

 Answer In-Lab Questions #5, #6, and #7 on page E31A-6.

5. Place a pea-sized amount of your soap in the palm of your hand. Over the sink, wet your hands slightly and rub them together. Marvel at the quality of your prepared soap.

Clean Up:

1. Clean your glassware with soap and water. If a clean 150 mL beaker still appears cloudy at the bottom, the NaOH solution has etched the glass. It will still be useable for this experiment. All other glassware should clean up completely. It is already soapy so cleaning is easy, but you **HAVE TO RUB**, and rinse thoroughly, to get the film to come off.
2. Discard any used filter paper, pH paper and other debris. Empty the filtration flask and rinse it out.
3. To keep your soap sample, form it into a bar and wrap it in a piece of large filter paper. It will harden when dry.



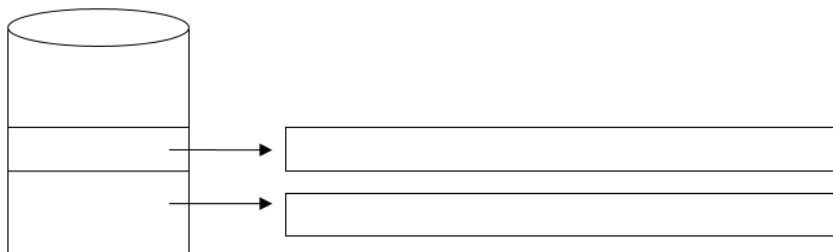
IN-LAB QUESTIONS

Experiment 31A



Part A: Making the Soap

1. a. In the boxes to the right below, list the contents of the layers that you observe in the mixture in your beaker.

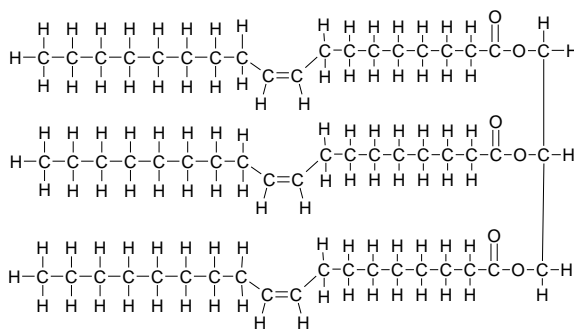


- b. Why do you observe two distinct layers in the mixture? (think about intermolecular forces)

Which layer is on the bottom of the beaker and why?

2. Describe the impure soap (color, odor, texture, etc).

3. a. Circle the three ester linkages in the triglyceride below.



- b. What are the bond angles around the carbon in the ester linkage $\begin{array}{c} \text{O} \\ \parallel \\ \text{---C---O} \\ \uparrow \end{array}$ of this molecule? _____

- c. What is the name of the arrangement of electron pairs around the carbon atom in the ester linkage?

- d. What is the hybridization of the carbon atom in the ester linkage?

- e. What is the hybridization of the first carbon in the triglyceride above?

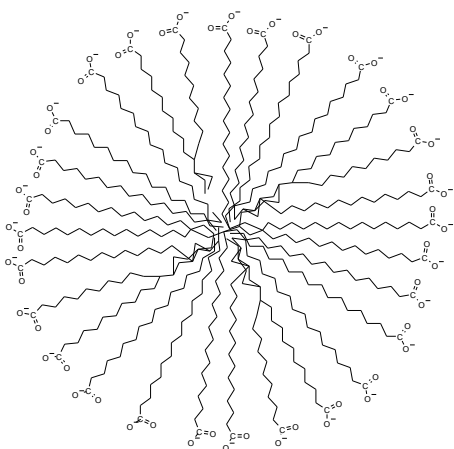
4. Describe the purified soap in comparison to the impure soap.

5. What is the pH of the soap? _____ What is the pH of the filtrate? _____

Are these pH values acidic or basic? _____

Why do you observe the pH value for the filtrate that you recorded above? What's in the filtrate?

6. Soap *in water* forms little "droplets" called micelles as shown below. The $\wedge\wedge\wedge\wedge\wedge$'s represent long hydrocarbon chains. The charged "heads" are on the outside of the micelle. Explain, in terms of intermolecular forces, why the micelle forms in this way.



Re-draw the picture above to represent a soap "droplet" if the soap was mixed with gasoline, a nonpolar liquid (instead of water).

7. How is soap able to remove dirt/grease from your skin? (Explain in terms of intermolecular forces.)

