Experiment 31A

FV 4-7-15

SOAP – A FATTY ACID SALT

MATERIALS: Soybean oil, 6 M NaOH, 100% ethanol, 150 mL beaker, 50 mL beaker, 10 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.

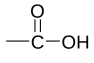
LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate these proficiencies:

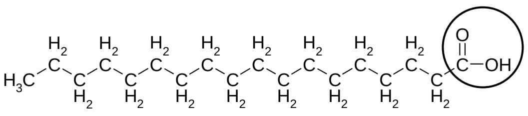
1. Use intermolecular forces to explain why liquids are or are not miscible.
2. Apply understanding of molecular structure and intermolecular forces to describe how soap helps to remove greasy dirt in a water solution.
3. Understand features of structure and bonding in everyday organic materials.

PRE-LAB: Complete the pre-lab on page 8 **before** lab. Bring your goggles to lab.

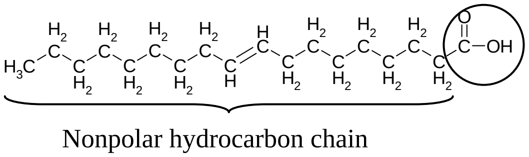
DISCUSSION:

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  $-\overset{O}{\overset{\|}{C}}-OH$ 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.

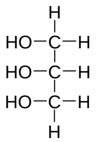


Stearic acid, a saturated fatty acid

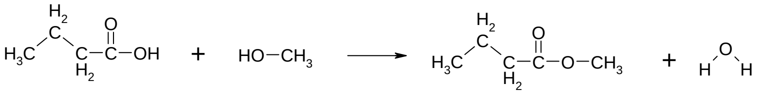


Oleic acid, a monounsaturated fatty acid

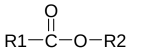
Fatty acids are the first building blocks of soaps. As indicated above, soaps were made from animal fats, and, more generally, can be made from fats or oils of plants or animals. Edible fats and oils are triglycerides, which are esters made from fatty acid molecules and glycerol, $C_3H_8O_3$, a trialcohol.



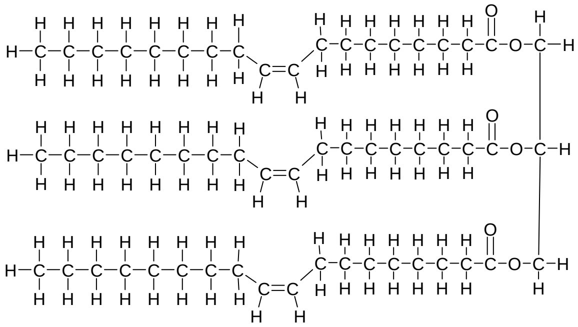
(*Alcohols* are organic molecules with an –OH group. You can see that glycerol has **three** such segments.) In a reaction that we shall see later, acids and alcohols can combine to form another class of molecules called *esters*.



Carboxylic acid alcohol ester water

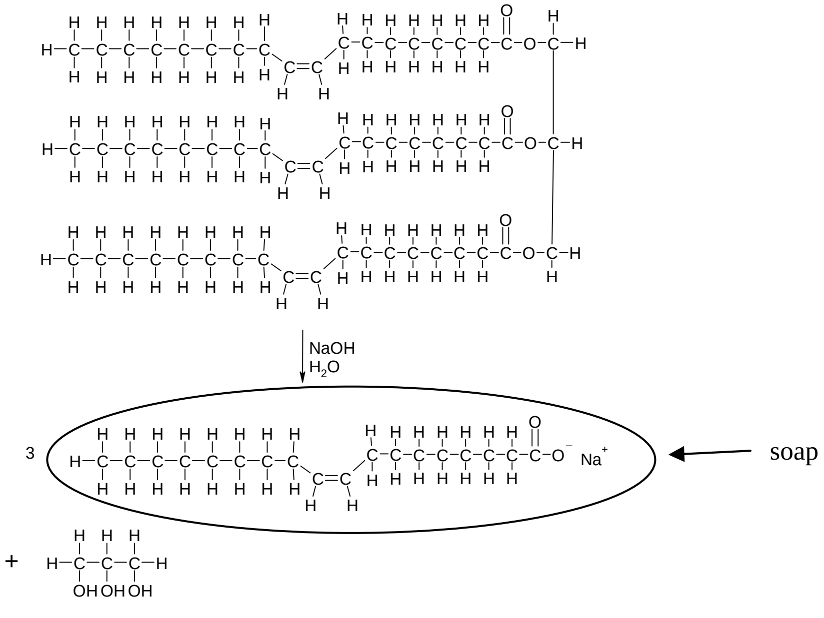
Esters have the general structure of  $R1-\overset{O}{\overset{||}{C}}-O-R2$ where the R1 and R2 groups represent any organic moiety. (For example, R1 is $CH_3CH_2CH_2$, and R2 is CH_3 in the ester shown above.)

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.



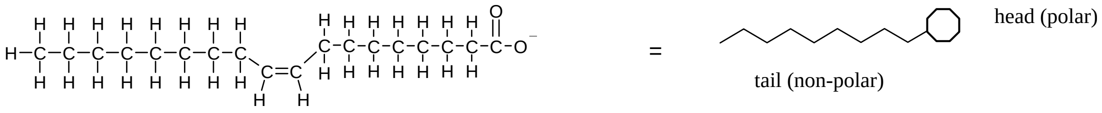
"Oils" are triglycerides that are liquid at room temperature; typically they are composed of unsaturated fatty acids, such as the molecule above. "Fats" are triglycerides that are solids at room temperature; usually these involve saturated fatty acids. The three fatty acids in any triglyceride might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



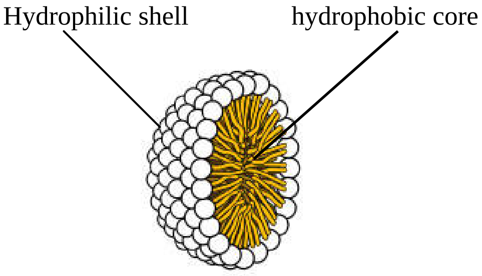
A similar process is carried out in clogged kitchen drains every day. Products like Drano® consist mostly of solid NaOH. When poured into a drain, it creates a strongly basic solution which hydrolyzes the grease or fat that forms the clog, rendering it more soluble so it can be washed away. (The shiny strips in Drano are aluminum chips; these produce H_2 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?)

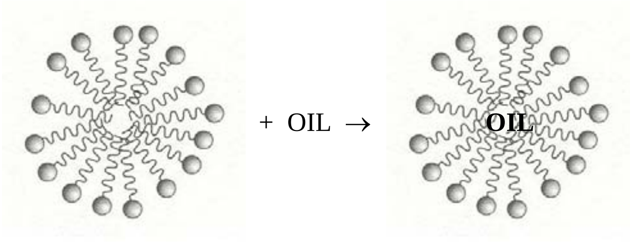
Why do soaps work? It all has to do with intermolecular forces. Molecules that have a non-polar "hydrophobic" part (the hydrocarbon tail) AND a polar "hydrophilic" part (the ionic head) are called *surfactants*, which is short for "surface-active agents". Frequently such structures are qualitatively represented as:



"Soap"

In water solution, surfactants tend to aggregate to form hollow colloid-sized particles called *micelles* that can transport various materials within them. In water solution, the hydrophobic tails of the surfactant point inward towards the center of the micelle, while the hydrophilic heads point outward, so they can interact with the water (a cutaway view of a spherical micelle is shown below). Soaps cleanse because oily dirt on clothes or skin joins the hydrophobic centers of the soap micelles and is rinsed away. The whole process is illustrated in the figure below.





Soaps generally cannot be used with "hard" water (water containing +2 ions like Ca^{2+}, Mg^{2+} or Fe^{2+}), because they tend to precipitate out as Ca, Mg or Fe salts, which are less soluble than sodium salts. (You should recall this general feature of solubility from earlier work.) "Soap scum" is that precipitate. "Detergents" are synthetic molecules that have similar chemical structures and cleanse in the same way as soaps do, but tend not to form such precipitates.

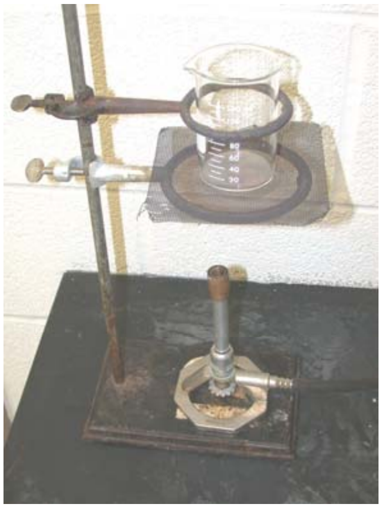
In this experiment, soybean oil will be reacted with sodium hydroxide and the soap product precipitated using saturated sodium chloride. We will synthesize the soap and purify it.

REFERENCES:

1. Kostka, K.; McKay, D. J. Chem. Educ. 2002, 79(10), 1172-1174.
2. Phanstiel IV, O.; Dueno, E.; Wang, Q.X. J. Chem. Educ. 1998, 75(5), 612-614.
3. Moore, J.W.; Stanitski, C.L.; Jurs, P.C.; *Chemistry, the Molecular Science, 2nd ed*., Brooks/Cole-Thomson Learning, Belmont, CA, 2005.

PROCEDURE: (Work in pairs)

Part A: Making Soap



1. Make an ice bath and begin cooling about 50 mL of saturated NaCl solution.

2. Assemble your ring stand, small ring, large ring, wire mesh, 150 mL beaker, and Bunsen burner as shown in the figure to the right.

3. Combine the following reagents in a 150 mL beaker:

   8.0 mL of soybean oil (or other oil that is available)
   4.0 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. Do not get this strong NaOH on your skin.

✍ Answer In-Lab Question #1 on page E31-6.

4. Now add 8.0 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.

✍ Answer In-Lab Question #2 on page E31-6.

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. Add 15 mL of distilled water to the reaction mixture. Stir to dissolve the soap. Heat the solution gently to aid in the dissolution process. If the soap does not completely dissolve, add just enough additional distilled water to get all of the soap into solution (no more than 5 mL). Remove the Bunsen burner from beneath the beaker. Place the beaker on the lab bench and allow the soap solution to cool in the beaker for 5-10 minutes.

✍ Answer In-Lab Question #3 on page E31-6.

2. Add 25 mL of ice-cold saturated NaCl to the soap solution. Stir well. Pure soap crystals will precipitate out of solution.

3. Use a Buchner funnel with filter paper to filter the soap crystals. Be sure to empty the flask before using the suction filtration apparatus. To aid in the filtration process, rub the stirring rod gently along the filter paper to dislodge the solid. BE CAREFUL NOT TO TEAR THE FILTER PAPER. Once the majority of the liquid has been removed (soap will still be quite fluid) place the filter paper with soap on a paper towel to allow for additional drying.

✍ Answer In-Lab Question #4 on page E31-7.

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

✍ Answer In-Lab Questions #5, #6, and #7 on page E31-7.

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 <u>clean</u> 150 mL beaker still appears somewhat cloudy at the bottom that is because the NaOH solution has etched the glass. It will still be useable for this experiment. All other glassware should clean up completely. Since it is already soapy, 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.

3. Empty the filtration flask and rinse it out.

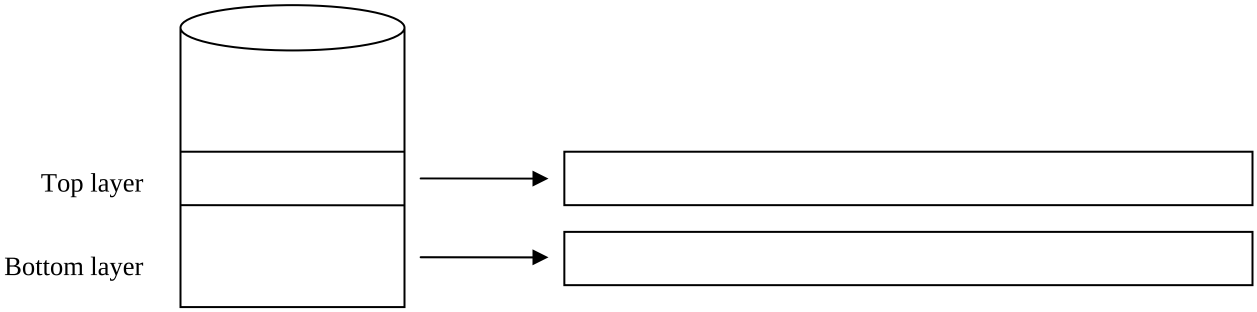
4. If you wish to take your soap sample with you, you might form it into a bar and wrap it in a piece of large filter paper. It should harden when sufficiently dry.

Name ______________________________________ Section _____________

✍ 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.

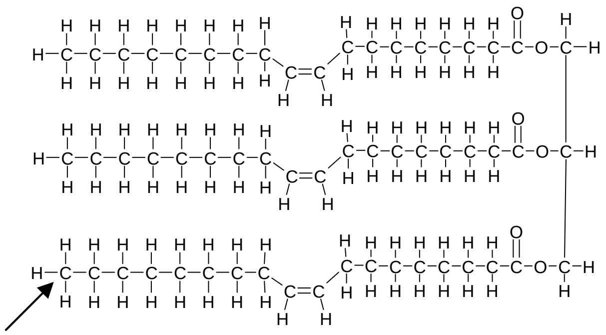


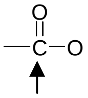
b. Why do you observe two distinct (immiscible) layers in the mixture? (think about intermolecular forces)

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

2. Describe the <u>impure</u> 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  $-\overset{\overset{\displaystyle O}{\|}}{C}-O$ 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 (see arrow)? ________________

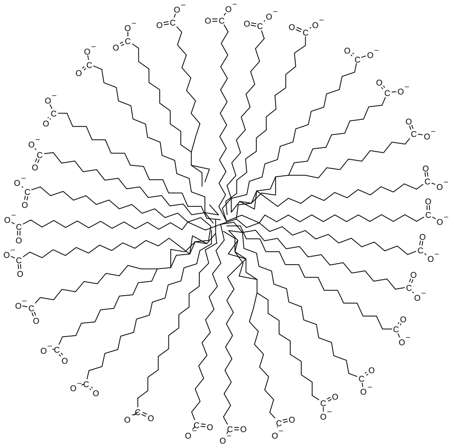
4. Describe the purified soap in comparison to the impure soap.

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 /\/\/\/\/\/'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 from your skin? (Explain in terms of intermolecular forces.)

Expt. 31A – Soap: A Fatty Acid Salt Name ____
PRE-LAB

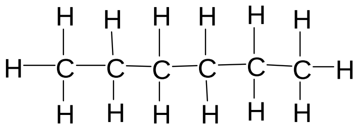
1. What everyday substance will you be preparing in the Expt. 31A? _________________________

2. The process of soap-making is known as saponification (*hydrolysis* of a fat or oil in an *alkaline* solution).

   Will your prepared soap be acidic, basic, or neutral? _______________________

   Does this correspond to a pH that is < 7, > 7, or = 7? _________________

3. Hexane (C_6H_{14}), a saturated hydrocarbon, has the following structure:



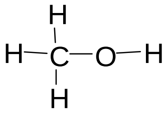
   a. Is hexane a polar or nonpolar molecule?

   ____

   b. List all of the intermolecular forces present in hexane.

   ____

4. The simplest alcohol is methanol (CH_3OH). An alcohol is an organic function group, R-OH.



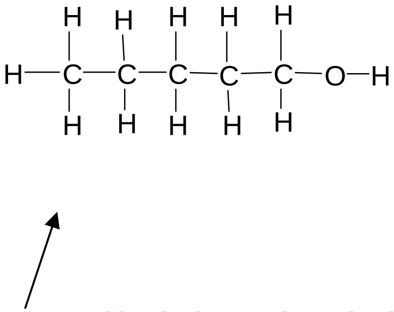
   a. Is methanol a polar or nonpolar molecule?

   ____

   b. List all of the intermolecular forces present in methanol.

   ____

5. The 1-pentanol structure is given below:



   a. Is 1-pentanol a polar or nonpolar molecule?

   ____

   b. List all of the intermolecular forces present in 1-pentanol.

   ____

1-pentanol has both nonpolar and polar components (like soap). *In the structure above*, **circle** the *nonpolar* component of 1-pentanol. Draw a **box** around the *polar* component of 1-pentanol.

6. Order the 3 substances above (hexane, methanol, 1-pentanol) in order of *increasing* overall polarity:

   ______________________ < ________________________ < __________________________
   Least polar Most polar

   Which of these 3 substances would dissolve *best* in water? _________________________