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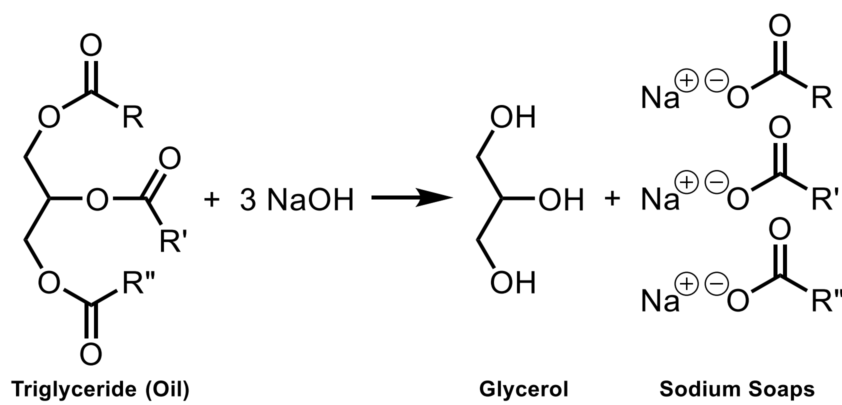
Experiment 19 – Synthesis and Characterization of Soap

In this experiment, you will prepare a soap fro vegetable oil. The properties of your soap will be compared to those of commercial soaps and detergents under various conditions.

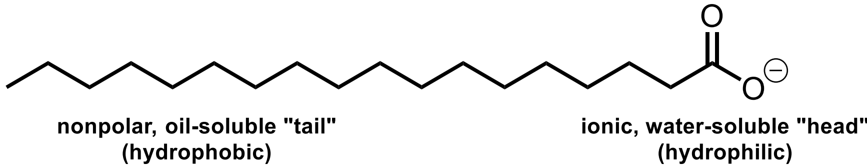
<u>Discussion</u>

Common experience has shown us that oil and water do not mix. This behavior is due to the fact that oil molecules are nonpolar, while water is very polar. In fact, oil and water repel one another, making the removal of oil stains very difficult with water alone. To remove an oil stain on a piece of clothing, it is necessary to add soap or detergent in order to make the oil dissolve into the wash water. The unique chemical structure of soap allows it to act as a "go-between" for the water and oil.

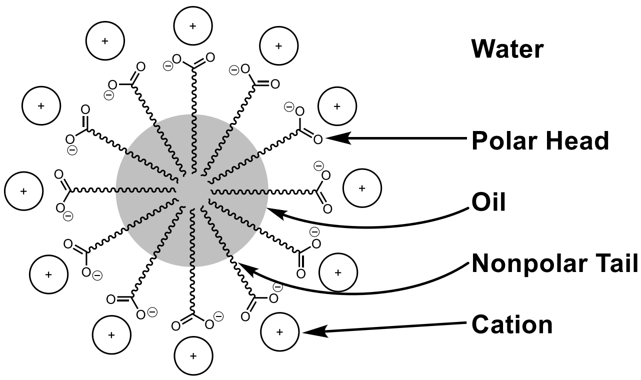
Most water-soluble soaps are sodium or potassium salts of long chain organic acids. Soaps are prepared from natural fats or oils with strong base such as NaOH. All fats and oils have the general structure shown below, with the R, R', and R" signifying hydrocarbon chains of varying length. Glycerol and soap salts (e.g., sodium salts of fatty acids produced from oil and sodium hydroxide) are the products.



Sodium stearate is a common commercial soap and has the formula $NaC_{18}H_{35}O_2$. In water, this compound dissolves to form sodium ions and stearate ions (see structure below). The stearate ion consists of a long, nonpolar hydrocarbon chain that dissolves well in oil, and a polar, negatively charged end that dissolves in water.



The nonpolar chain and the charged (ionic) tail are referred to as the "hydrophobic" (water-fearing) and "hydrophilic" (water-loving) parts, respectively. When washing oil-stained clothing, the hydrophobic tails of the soap are attracted into the oil spot. As motion of the water molecules tugs the charged end back and forth, the oil is pulled free from the fabric in small droplets that become surrounded by soap ions. These spherical beads, called micelles, repel one another due to the charged ends on the surface (see figure below) and remain dispersed throughout the wash water. Such a dispersion is called an ***emulsion***.



For a soap to function well as a cleaning agent, it must be dissolved in water. In hard water, ions such as Ca^{+2} and Mg^{+2} will precipitate with the soap anions as an insoluble solid. Since this precipitate is not very dense, it usually floats and is usually visible as soap scum or bathtub ring. As soap scum removes soap ions from solution, cleaning in hard water is difficult unless more soap is added to compensate for the loss. Another alternative is to "soften" the water by removing most of the troublesome metal ions.

Soap ions must also retain their charged ends in order to remain functional. In acidic water, the soap anion will act as a base, accepting a hydrogen ion to form the neutral fatty acid with the end group, COOH. Without the charged end, the soap will not be attracted as strongly to water, will not form micelles, and thus will not remove and emulsify the oil. In some cases, the acid form of the soap may be insoluble if sufficient polarity is lost. One solution to this problem is to use detergent instead of soap. A detergent consists of a long hydrophobic chain with a hydrophilic head, but the head is a less reactive [$—SO_3^-$] group instead of the [$—CO_2^-$] found in soap. These detergent ions will remain soluble in hard and acidic water, and thus are more effective under such conditions. Sodium lauryl sulfate, $CH_3(CH_2)_{11}OSO_3^-Na^+$ (abbreviated $ROSO_3^-Na^+$), is a common detergent used in shampoos. Also, some commercial soaps and detergents contain additives such as phosphates to reduce the acidity of the water used.

Cautions

Ethanol is flammable, so no open flames are allowed. Sodium hydroxide solutions are corrosive to skin and clothing, so avoid contact. If contact occurs, rinse with lots of water.

Procedure

1. Place 5 g of vegetable oil in a 125 mL Erlenmeyer flask. Add 5 mL of ethanol. Swirl the solution to mix the layers together.

2. Add 7 mL of 5 M NaOH. Swirl the solution well and pour into a 100 mL beaker. Stir with a long glass rod while heating gently (setting at 3–4) on a hot plate. Continue to heat while stirring frequently, until the mixture is a stiff paste. This may take up to 30–40 minutes. After the paste has formed, allow the beaker to cool for 15 minutes until slightly warm to the touch.

3. Add 25 mL of saturated NaCl solution to the cooled paste and stir, breaking up the large chunks of paste by pressing them against the side of the beaker. This procedure is known as "salting out". Be careful not to overdo the breaking up, or your soap won't filter well.

4. Filter the soap mixture by suction filtration. (NOTE: Pour most of the liquid through first because the soap may clog the filter.) Wash once with 10 mL of ice water through the filter, then allow it to drain for 5 minutes with the aspirator on.

5. Make separate solutions of 1) your prepared soap; 2) a commercial soap; and 3) a commercial detergent as follows: Warm about 300 mL of D.I. water on a hot plate until it reaches about 60 °C. Dissolve about 0.25 g of each of the test materials in 100 mL of the warm water in 250 mL beakers. Stir well to ensure that all of the soap or detergent has dissolved. These will serve as your test solutions for the following steps.

6. **pH**: Test the pH of each solution and D.I. water using universal indicator paper. For each solution, use a clean glass rod to transfer a drop of solution to the paper. Record your results.

7. **Emulsifying properties**: Label 4 clean, dry test tubes to correspond with each of your test solutions plus D.I. water. Place 10 mL of each test solution into its labeled test tube, and 10 mL D.I. water into the fourth. Add 10 drops of paraffin oil to each test tube and shake, and immediately after shaking, record how uniformly the oil and water are distributed throughout the mixture. Leave the tubes undisturbed for 1 minute, then again record your observations on how well the oil and water remain mixed, and how much oil has recollected at the top.

8. **Effect of Hard Water**: Label 4 clean, dry test tubes to correspond with each of your test solutions plus D.I. water. Place 10 mL of each test solution into its labeled test tube, and 10 mL D.I. water into the fourth. Shake each tube to observe the head of "suds" produced (the D.I. will provide you with a comparison with no suds). Add 2 mL of "hard water" provided (contains Ca^{+2} and Mg^{+2} ions) to the first tube, shake well, and observe any changes, looking for cloudiness, precipitate, lack of suds, etc. Record you observations. Allow to stand for 5 minutes and then observe again. Record any further changes. Repeat for the remaining test solutions.

9. **Cleaning Properties**: Use tap water to wash your hands with some of the soap you made. Note how well (or how poorly) it lathers. If there was an excess of oil used in making the soap, it may leave a greasy film on your skin. If there was an excess of NaOH used in making the soap, your hands may feel slippery during washing and rinsing, but not greasy. Rinse your hands well and dry them. Record your observations on the lathering properties and the "feel" of your soap. Dispose of your product in the trash.

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Data and Calculations for Experiment 19

A. pH

Solution	pH
1. Prepared Soap	________
2. Commercial Soap	________
3. Commercial Detergent	________
4. D.I. Water	________

B. Emulsifying Properties

Solution	Initial Observations	Observations After 1 Minute
1. Prepared Soap		
2. Commercial Soap		
3. Commercial Detergent		
4. D.I. Water		

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C. Effect of Hard Water

Solution	Initial Observations	Observations After 5 Minutes
1. Prepared Soap		
2. Commercial Soap		
3. Commercial Detergent		

D. Describe the washing properties of your soap in terms of lathering ability and feel.

Questions

1. Which solution was most basic according to pH measurements?

2. Which solution(s) show(s) the best ability to emulsify oil?

3. Explain your observations for the addition of hard water. What is happening in each test tube and why?