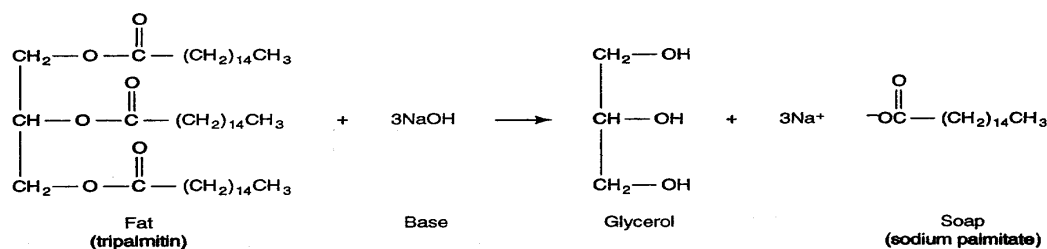


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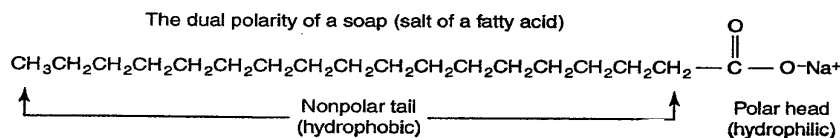
Experiment 10: Preparation of Soap

Saponification: For centuries soaps have been made from animal fats and lye (NaOH), which was obtained by pouring water through wood ashes. The hydrolysis of a fat (a triacylglycerol or triglyceride) with a base is called *saponification*, and the resulting salts of the fatty acids are called

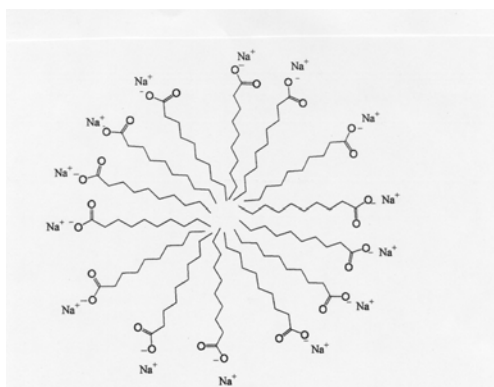


soaps. Glycerol (which also forms in the hydrolysis) is soluble in water. The most commonly used triacylglycerols are lard and tallow from animal fat and coconut, palm, and olive oils from vegetables. Castile soap is made from olive oil, soft soaps are made with KOH as the base and therefore yield potassium salts. Soaps that float have trapped air pockets within.

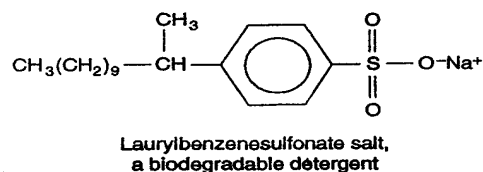
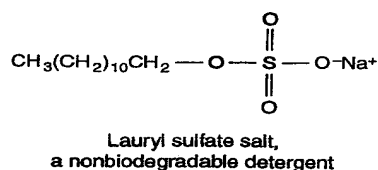
Properties of Soaps and Detergents: A soap molecule has dual properties. The non-polar hydrocarbon chain tail is hydrophobic and therefore attracted to other non-polar substances like



grease. The polar (actually ionic) head of the carboxylic acid salt is hydrophilic and therefore attracted to water (which is polar). When soap is added to a greasy substance, the hydrophobic tails are embedded in the non-polar fats and oils. And the polar heads are attracted to the polar water molecules. Clusters of soap aggregates called *micelles* form with the non-polar oil droplet in the center surrounded by many polar heads that extend into the water - eventually all the greasy material is encased in micelles, and these are then washed and rinsed away. In “hard water” the carboxylate ends of the soap react with the Ca^{+2} , Fe^{+3} , or Mg^{+2} ions in the water to form insoluble salts, which we see as the scum ring around the sink or bathtub.



A micelle and Synthetic Detergents



Synthetic detergents are not derived from natural fats and oils, but they function in the same manner. Their popularity arose because they do not form insoluble salts with hard water so they always work well. However, as detergents replaced soaps for cleaning, it became apparent that they were not degraded in sewage treatment plants since large amounts of foam appeared in streams and lakes - as they became polluted with detergent (not to mention the interference with the ecosystem of the body of water). Biodegradable detergents eventually have replaced the original detergents.

Experimental Procedure:

Wear your protective goggles !!!

Safety: Protective eyewear and gloves should be worn while doing this experiment! Handle the sodium hydroxide solution carefully! And while stirring is necessary do not splash the liquid out of the beaker!

Saponification Procedure:

1. Place about 12 mL of the liquid fat (or 10 g of the solid fat) into a 150 mL beaker. Add 15 mL of ethanol and stir to dissolve the fat. Slowly and carefully add 8 mL of 8 M. sodium hydroxide solution.
2. Place the beaker on the hotplate and heat to a ***gentle boil*** while stirring continuously with a glass stirring rod. Continue heating and stirring until the triglyceride has completely reacted with the base (about 30 minutes). Stir carefully; be careful of splattering. If foaming becomes excessive (that is, it may overflow out of the beaker!), remove the beaker from the hotplate for a short time. Be careful not to “burn” the soap mixture.

3. When the mixture becomes a waxy consistency, take the beaker off the hotplate and allow it to cool somewhat and then add 20 mL of water and stir. Allow this mixture to reach room temperature. (Please note that there is still an excess of NaOH present.) Describe the solid that formed:

4. While your soap is cooling, in a *different beaker*, dissolve 18 g of sodium chloride to 60 mL of water.

5. We will now attempt to remove the excess NaOH within the soap (using a “salting out” process). Wash the soap *three times* - by adding 20 mL of the sodium chloride solution, stirring thoroughly and breaking up any lumps with the stirring rod, and then carefully pouring off the liquid. You need to keep your solid soap while pouring off the liquid! After the third wash, collect your solid soap on a paper towel and “pat it dry.” Be careful: the solid still contains NaOH, which can irritate the skin.

Properties of Soap:

1. pH Test - Place a small piece (about the size of a pea) of your soap into a test tube. Place about 20 drops of the commercial soap in a second test tube. Add about 10 mL of water to each test tube and shake to dissolve. Determine the pH of each resulting solution by dipping a stirring rod into the solution and then touching the rod to pH paper. *Save these solutions.* Record your results:

pH: your soap _____; commercial soap _____

2. Sudsing - Stopper each of the test tubes (from part 1 above) and shake the tube vigorously. Each should form a layer of suds or foam. Compare the sudsing and record your results:

your soap

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commercial soap

3. Hard Water Test -

a) In *three different* test tubes, place 5 mL of tap water, 5 mL of distilled water, and 5 mL of 1% CaCl_2 solution (ie. “hard” water).

b) To a *fourth* test tube, add about “three peas” quantity of your soap and about 7 mL of distilled water, and shake to dissolve most of the soap.

c) Now add about 2 mL of the soap solution made in b) to each of the original three part a) test tubes. [You could just “eyeball” about one-third of the b) solution into each of the a) test tubes.] Shake well and record your results.

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tap water

distilled water

“hard” water

Write a balanced equation for the reaction which occurs with the CaCl_2 solution.

[Please use fatty acid- $\text{CO}_2^- \text{Na}^+$ as the structure for your beginning soap.]
