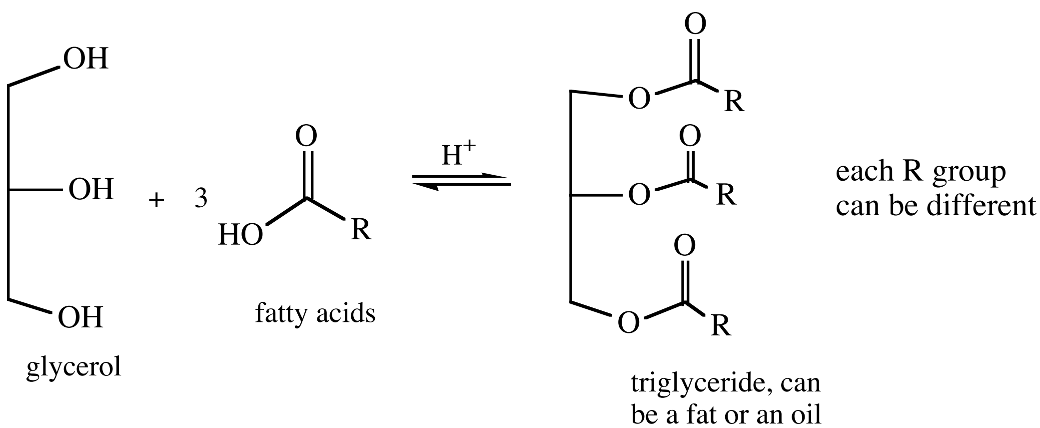
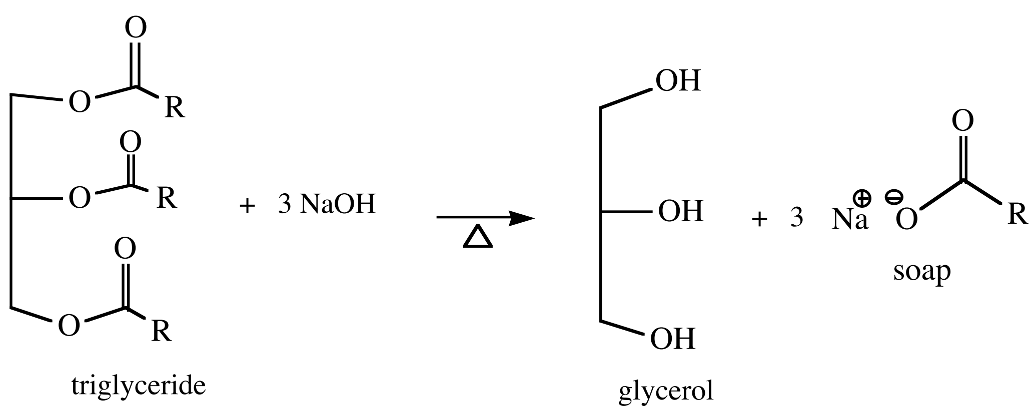
Synthesis of Soap

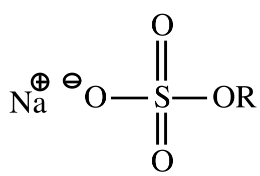


Fats and oils have the general structure shown above. Glycerol combines with 3 fatty acids to form the fat or oil (triglyceride), which has 3 ester groups. Fats are solids at room temperature, and oils are liquids. A "fatty acid" is a carboxylic acid with a long chain, typically containing 14, 16, or 18 carbon atoms; but this number can vary from 4 to 26 carbon atoms. Interestingly, this chain always has an even number of carbon atoms. This is because the biosynthesis of these molecules adds carbons in groups of 2. Saturated fatty acids (and the corresponding fats) contain all single bonds, and unsaturated fatty acids contain one or more double bonds which are almost always *cis* double bonds. Generally, oils are made from unsaturated fatty acids. The "kink" in the chains prevent them from packing tightly together and becoming a solid. Fats, in contrast, are made from mainly saturated fatty acids.

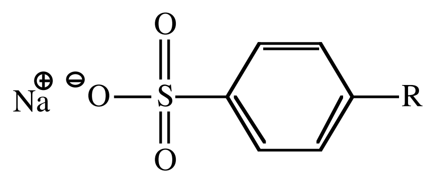


Soaps are the products of the reaction of triglycerides with hydroxide salts. Soaps are usually the sodium or potassium carboxylate salts of the fatty acids. This basic cleavage of the ester bond is called a "saponification". Due to the stability and lack of reactivity of the carboxylate anion with most nucleophiles (in this reaction, the hydroxide or alkoxide ions), this reaction is <u>not reversible</u>, as opposed to the acid catalyzed mechanism.

The nature of the metal used in the hydroxide salt (usually sodium or potassium) can have an effect on the texture of the soap and on its solubility in water. Sodium and potassium salts are generally soluble in water, and potassium soaps are usually more soft than sodium soaps. When these metals are replaced by calcium, iron, or magnesium ions, the new carboxylate salts are no longer soluble in water; and form what we lovingly refer to as "soap scum". You may have heard of the terms "hard water" or "soft water". Hard water is water that contains many metal ions such as iron, calcium, and magnesium ions. Soft water contains less of these ions. Therefore, when we use soap with hard water, the sodium or potassium ions get replaced by these ions, and we get lots of soap scum on our sinks and bathtubs. Which you then have to scrub off. Such fun! In order to avoid this problem, many synthetic detergents have been produced which have many of the same structural features of normal soap, and are less likely to form these insoluble salts. Common ones are the sodium alkyl sulfates or sodium aryl sulfonates (shown below). Note the polar end and non-polar ends of these molecules. While normal soaps made from triglycerides are easily biodegradable, the aryl sufonates are only biodegradable if the R group is a straight chain. Can you propose a synthesis of a sodium aryl sufonate using benzene as a starting material?



sodium alkyl sulfate
R ~ 12-16 carbons
biodegradable



sodium aryl sulfonate
R ~ 8-12 carbons
biodegradable only if R is a straight chain

Many of these alternate detergents also have additives which will bind to the metals that can create soap scum, preventing them from reacting with the soap. Unfortunately, many of these binders have environmentally damaging effects. One of which is that some of these additives are nutrients for algae in lakes and other bodies of water. The algae grows very quickly, and then when this algae begins to die and decompose, they consume so much dissolved oxygen from the water that no other life can exist in that water. The lake then rapidly dies. This process is called "eutrophication". Which is a good word to impress people with.

Experimental Procedure

Place 2.5 g of solid sodium hydroxide pellets into your 50 or 25 mL round bottom flask. (Sodium hydroxide is caustic and can react with your skin, making soap out of the fats and oils in and on your skin. If you do get any on your fingers, just rinse them off in the sink with lots of running water. Use your scoopula to measure out the sodium hydroxide.) Add 5 mL of distilled water, and swirl this mixture to dissolve the NaOH. Feel the outside of the flask, and note how exothermic this dissolving process is. Then add 5 mL of denatured ethanol. This will help dissolve the fat or oil once you add it to the solution. Add approximately 2.5 mL of your oil, or 2 g of your fat. Add 2 boiling chips and place your water cooled condenser (water in at the bottom of the condenser, out from the top) on the flask. Reflux the mixture for about 30 minutes. You may need to ocassionally swirl the flask to wash the salts and oils back into the reaction mixture.

While this reaction is refluxing, prepare a concentrated sodium chloride solution by dissolving 15 g of ground up sodium chloride (you may have to grind it up yourself) in 50 mL of distilled water. After the NaCl is dissolved, cool this solution in an ice bath. When your reaction mixture is done refluxing, pour the <u>hot</u> solution into your ice-cold NaCl solution. This helps make the soap precipitate out as a solid. (Remember the common ion effect from 101B?) Stir this mixture for 2 minutes, and allow it to stand for 10 minutes in the ice bath. Then collect your soap by suction filtration and allow the suction to partially dry your soap. Wash the soap with 15 mL of cold distilled water.

Make a solution by dissolving 0.5 g of your soap in 25 mL of distilled water. Heat this to boiling to dissolve as much of your soap as possible. Cool this solution to room temperature. Place 5 mL of this solution in a test tube, add 4-5 drops of mineral oil, shake the tube for a minute, allow it to stand for a minute or so, and then observe how well the oil is dissolved. The following procedure is designed to show you what happens when soap is used in hard water. Take 3 test tubes and add 5 mL of your soap solution to each. Add 5 mL of 0.05 M $CaCl_2$ to one tube, 5 mL of 0.05 M $MgCl_2$ to another test tube, and 5 mL of 0.033 M $FeCl_3$ to the third test tube. Shake each test tube and record your observations. Test the soap properties of the contents by adding 4-5 drops of mineral oil to each test tube, shaking each tube for one minute, and observing how well the oil is dissolved.

Now you can save your soap to clean all of your dirty glassware!