Chemistry of Fats

CHEM 131 Experiment 8

Chemistry of Fats – Soapmaking and Degree of Unsaturation

In this experiment, you will explore the chemistry of fats. In the first part of the experiment, you will perform a saponification (soap-making) reaction, which is an example of an important general reaction type in organic chemistry, the basic hydrolysis of an ester. In the second part of the experiment, you will use bromination to evaluate the relative degree of unsaturation in various different types of oils and solid fats. Bromination is similar to the process of hydrogenation, both of which are examples of addition reactions of alkenes.

BACKGROUND

Fats

Fats, also known as triglycerides or triacylglycerols, are biomolecules synthesized by the body from excess food intake (like after Thanksgiving dinner) in order to serve as energy reservoirs in times of need (as well as providing a little extra insulation from the cold). All fat molecules are synthesized from one glycerol molecule and 3 fatty acids, which are linked in an esterification reaction as shown below. Note that the glycerol molecule acts as a triple alcohol, each fatty acid terminates in a carboxylic acid group, and the condensation linkage results in a triple ester.

\[
\begin{align*}
\text{H}_2\text{C} &- \text{OH} \\
\text{HC} &- \text{OH} \\
\text{H}_2\text{C} &- \text{OH}
\end{align*}
\]
+ \[
\begin{align*}
\text{HO} &- \text{C} \\
\text{HO} &- \text{C} \\
\text{HO} &- \text{C}
\end{align*}
\]
\[
\begin{align*}
\rightarrow \text{H}_2\text{C} &- \text{O} - \text{C} \\
\text{HC} &- \text{O} - \text{C} \\
\text{H}_2\text{C} &- \text{O} - \text{C}
\end{align*}
\]
\[
\begin{align*}
\text{glycerol} & \\
3 \text{fatty acids} & \\
\text{triglyceride} 
\end{align*}
\]

The fatty acids are long-chain hydrocarbon molecules that terminate with a carboxylic acid group. The chains contain an even number of C atoms, varying typically between 12 and 18 C atoms, and may contain one or more C=C double bonds (unsaturated), or may contain only single-bonded carbons (saturated). The names, formulas, and sources of several common fatty acids are shown below. Most naturally occurring oils and fats actually contain a mixture of different fatty acids. For example, olive oil contains 73% oleic acid, 15% palmitic acid, and 12% linoleic acid.

<table>
<thead>
<tr>
<th># C</th>
<th>Structural Formula</th>
<th>Melting Pt. (°C)</th>
<th>Name</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saturated Fatty Acids</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>CH₃(CH₂)₁₀COOH</td>
<td>44</td>
<td>Lauric</td>
<td>Coconut</td>
</tr>
<tr>
<td>16</td>
<td>CH₃(CH₂)₁₄COOH</td>
<td>63</td>
<td>Palmitic</td>
<td>Palm</td>
</tr>
<tr>
<td>18</td>
<td>CH₃(CH₂)₁₆COOH</td>
<td>70</td>
<td>Stearic</td>
<td>Animal fat</td>
</tr>
<tr>
<td>Monounsaturated Fatty Acids</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>CH₃(CH₂)₇CH=CH(CH₂)₇COOH</td>
<td>4</td>
<td>Oleic</td>
<td>Olives</td>
</tr>
<tr>
<td>Polyunsaturated Fatty Acids</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>CH₃(CH₂)₉CH=CHCH₂CH=CH(CH₂)₂COOH</td>
<td>-5</td>
<td>Linoleic</td>
<td>Safflower</td>
</tr>
<tr>
<td>18</td>
<td>CH₃CH₂CH=CHCH₂CH=CHCH₂CH=CH(CH₂)₂COOH</td>
<td>-11</td>
<td>Linolenic</td>
<td>Corn</td>
</tr>
</tbody>
</table>
Chemistry of Fats

Because chains of single-bonded (sp$^3$ hybridized) C atoms pack together very efficiently (see figure below), the intermolecular forces between saturated fat molecules (found predominately in animal fats) are relatively strong, and thus they tend to be solid at room temperature. Chains that contain double bonds (sp$^2$ hybridized C), on the other hand, disrupt the alignment of the atoms in the fatty acid chains, thereby resulting in weak intermolecular forces. Therefore, unsaturated fats (found in vegetable oils) tend to be liquid at room temperature.

Hydrogenation of Fats

Food producers often use vegetable oils in many products that they wish to have a solid-like consistency (in addition to the consistency, another benefit is greater resistance to spoilage, and hence a longer shelf life). Consequently, they often hydrogenate (or partially hydrogenate) the unsaturated fats found in these vegetable oils so that they behave more like saturated fats. One example of this is the production of margarine.

Hydrogenation is one example of an addition reaction, in which an alkene is converted to an alkane by adding H atoms to the double-bonded C atoms. Bromination is another example of such an addition reaction, but in this case it is Br atoms that are added.

\[
\text{Hydrogenation of an alkene:} \quad \text{CH}_3\text{CH}=\text{CH}_2 \quad + \quad \text{H}_2 \quad \rightarrow \quad \text{CH}_3\text{CH}_2\text{CH}_3
\]

\[
\text{Bromination of an alkene:} \quad \text{CH}_3\text{CH}=\text{CH}_2 \quad + \quad \text{Br}_2 \quad \rightarrow \quad \text{CH}_3\text{CHBrCH}_2\text{Br}
\]

One interesting aspect of bromination is that Br$_2$ has a reddish-orange color, while the brominated hydrocarbon is colorless. As a result, bromine can be used to titrate the double bonds present in alkenes. This provides a very convenient method, which we will use in the second part of the experiment, to determine the relative degree of unsaturation (number of double bonds) in different types of fats.
Chemistry of Fats

Soapmaking

People have been making soap for many thousands of years. A Sumerian tablet dating to 2200 BC gives a formula for soap consisting of water, wood ashes, and oil. Excavations of Pompeii (79 AD) found a site that was either a soap factory, or a laundry, or both. Ancient Roman legend gives soap its name. From Mount Sapo, where animals were sacrificed, rain washed a mixture of melted animal fats and wood ashes down into the Tiber River. There the soapy mixture was discovered to be useful for washing clothing, skin, and hair.

Today, we consider soap a necessity, which we take for granted. But as late as the 1920’s, fine soap for bathing was considered a luxury, and was used only for special occasions or for medical purposes. In the home, soap was made from rendered animal fats saved over the year. The fat was melted and stirred with a quantity of water and ashes over a wood fire. The soap was not very good quality and did not smell very wholesome.

We are going to make a very mild moisturizing soap, using a method known as the “cold process”, by combining sodium hydroxide with your choice of oil or fat. Commercial soap is processed to remove the glycerol, which forms in the soap making process. But glycerol (also known as glycerin) is a wonderful moisturizer and is often added back to the final product of commercial soaps. We will leave the glycerol in our soap. Our soap will be “super fattted”, meaning that our recipe uses more fat than is needed to react with all of the lye.

The saponification reaction that occurs during soap making is an example of a type of reaction known as the basic hydrolysis of an ester. In this reaction, which is essentially an esterification reaction in reverse, an ester is split into an alcohol and a carboxylic acid. Since the solution is basic, the carboxylic acid actually exists as the carboxylate salt (remember that a salt is the product of an acid-base reaction). In the case of soapmaking, the alcohol produced is glycerol, while the carboxylate salts are the salts of the fatty acids, better known as soap!

The Soapmaking Reaction

$$\text{Fat} \quad + \quad 3 \text{NaOH} \quad \rightarrow \quad \text{Glycerol} \quad \text{Soap (Sodium carboxylate salt)}$$

Pre-Lab Questions

1. What 2 trends are apparent in the melting points of the fatty acids listed in the table on page 1? Explain the reason for each of these trends in terms of intermolecular forces.

2. Write the chemical reaction equation for the basic hydrolysis of methyl ethanoate (methyl acetate). Indicate both the chemical formulas and the names of each of the substances in the reaction.

3. a) Write the balanced chemical reaction equation for the bromination of a fat that has 1 stearic, 1 oleic, and 1 linoleic acid chain. Use organic line drawings to represent the fat.

b) How many moles of Br₂ must be added to react with 1 mole of this fat?
Experiment 8: Chemistry of Fats—Saponification and Bromination

Procedure

A. Making Soap

Soap making is a slow process. The reaction will keep going for several days to weeks. Next week, you can remove your soap from its mold. It may take an additional 2-3 weeks until it hardens completely.

The following is a generic recipe for about one 8 ounce bar of very mild soap. It is super fatted, which means it has a stoichiometric excess of fat. If you desire, you can scent the soap, add colorings, or use a mold with an unusual shape. You must follow the instructions exactly for usable soap.

Ingredients

- 23 g of lye (NaOH)  
  CAUTION: LYE IS VERY CAUSTIC!
- 61 g of distilled water (since 1 mL of water weighs 1 g, this is 61 mL of water)
- 166 g of total fat – this can be a single fat or oil, or it can be a mixture. Lard, Crisco, and safflower oil will be provided for you. Feel free to bring any other oils that you desire to use (mixtures that contain olive oil and/or coconut oil make particularly nice soaps). At least 1/3 of your fat should be saturated fat, in order for it to set properly.
- Optional: 7.5 g of essential scented oil (YOU PROVIDE THIS)
- Food coloring (will be provided in lab)
- Cookie cutters to use as molds; must be fairly deep (YOU PROVIDE THIS)

1. Liberally grease the sides and bottom of a paper cup with Crisco. This will be the mold for your soap. Be sure to record the number that is marked on the bottom of the cup. We will use this number to identify the different soaps.
2. Weigh the lye into a small beaker, and then carefully pour the water from a graduated cylinder into the beaker with the lye.
3. Put the beaker in an ice bath. As the lye dissolves, the solution gets hot.
4. Stir until all the lye is dissolved, and then set the small beaker aside.
5. If using any solid fats, weigh them into a large beaker, and melt them over low heat (warm water bath). When it completely melted, they should be transparent and yellow.
6. Add any liquid oils to the melted fats, and stir.
7. Measuring the temperature of the oil and lye solutions with two different thermometers, wait until both solutions are between 35°C and 37°C. (A warm bath and a cool bath are helpful for adjusting the temperatures of the solutions.) Then slowly pour a steady stream of lye into the oil mixture, stirring constantly. It should take at least 2 minutes to add all the lye.
8. Stir for at least 10 minutes, keeping the temperature between 35°C and 37°C. You want to keep stirring until the mixture traces. This means that you can see the pattern of your stirring, and that a small amount of the mixture drizzled on its surface can be seen for two seconds. It may take much longer than 10 minutes of stirring for your soap to reach the right consistency.
9. If you brought an essential oil to scent your soap, slowly add it now. You may also add a few drops of food coloring if desired (note that the colors often don’t turn out the way you expect, particularly since the oils themselves have a yellow color).

10. Pour the mixture into the greased paper cup or custom mold.

11. Place the paper cup full of the soap mixture in the insulated box (will be provided). Small volumes of soap can easily separate if allowed to cool too quickly, so set it in the box as soon as possible. Fill in the appropriate information about the ingredients of your soap on the chart provided.

12. The chemical reaction will occur slowly over the next few days, and the soap will be hardened by next week. The soap will continue to age over the semester break, and will be fully ready for use by the beginning of next semester.

B. Degree of Unsaturation of Fats

1. Place 5 drops, or an equivalent volume of solid, of each of the following into 5 separate test tubes: oleic acid, olive oil, safflower oil, lard, Crisco (you may also test any other oils that you brought with you).

2. Add 1 mL of methylene chloride to each sample. Note the solubility of each fat.

3. To each of the test tubes, add bromine solution drop by drop until a red-orange color is observed to persist for at least 1 minute. Record the number of drops required for each fat.