CHEM 321  Synthesis of Two Polymers: Plexiglass and Nylon-6,6

Polymers are large molecules made up of many repeating groups of atoms called subunits. They can be extremely useful materials because they can be made to have an enormous variety of physical and chemical properties. Modern life depends heavily on such materials. Examples of useful polymers include fibers like nylon and polyesters from which clothing, carpets and upholstery can be made; rubbers of various kinds; poly(ethylene) and poly(propylene); plexiglass; foams; adhesives; … The list is endless. Life itself depends on biological polymers like DNA, RNA, and proteins.

Certain simple organic chemicals that possess suitable functional groups (an atom or group of atoms covalently bonded in a specific order that confers unique chemical properties on the molecule) can be easily transformed from liquids into high molecular weight solids. The liquid or liquids are monomer units that can be thought of as building blocks from which the polymer is made. In this experiment, two types of polymers will be formed: poly(methylmethacrylate) — trade name Plexiglass, also known as Lucite or Perspex — and Nylon-6,6.

Poly(methylmethacrylate) is formed by a process known as addition polymerization (a stepwise addition of monomers to a lengthening chain with no loss of atoms in the process). The functional group that permits one monomer to connect with another is the carbon-carbon double bond, which is the distinguishing feature of the class of organic compounds called alkenes. The structures of the monomer and polymer are shown below.

![Diagram of methyl methacrylate and poly(methyl methacrylate)](image)

Nylon-6,6 is formed by a process known as condensation polymerization (bifunctional monomers react with each other in such a way that the elements of a small molecule such as water or HCl are eliminated). In the case of nylon, the functional groups that permit the monomers to connect to one another are (a) the acyl chloride functional group (carbon connected to chlorine and double bonded to oxygen in the first monomer) and (b) the amine functional group (nitrogen connected to carbon and two hydrogen atoms in the second monomer). The structures of the monomers and polymer are shown below.

![Diagram of adipoyl chloride, 1,6-diaminoehexane, and nylon-6,6](image)

The objectives of this experiment are to learn how to measure and mix reactive chemicals, arrange for control of reaction temperature, and clean crude products by removing spent reagents.

Be sure to follow Pre-lab requirements and your Table of Properties should have entries for methyl methacrylate, benzoyl peroxide, hexane, adipoyl chloride, and 1,6-hexanediameine (including hazards of all of these).
**Experimental Protocols**  [There is a lot of waiting while methyl methacrylate polymerizes. Start it first, then make the nylon.]

1. **Polymerization of methyl methacrylate**

   A water bath at 60 – 70 °C will be used to warm the reaction mixture. If none is available, heat water in an almost full 250 mL beaker covered with a watch glass using a hot plate. Adjust the heating rate to maintain a water bath temperature between 60-70 °C (stir to measure). As the beaker heats, obtain a clean, dry, disposable small test tube that is a little taller than the beaker. Find a cork (not made of rubber) that fits the neck. Temporarily store the test tube in a 50 mL Erlenmeyer flask (to hold it upright).

   Use a ceramic spoon to weigh approximately 0.12 g of benzoyl peroxide on a piece of weighing paper using the method demonstrated in lab— NO METAL SPATULA! (The compound is explosive and metal can trigger the reaction). Partially fold the paper so as to make a pour spout and gently tap it until all the powder falls into the test tube stored in the Erlenmeyer flask. Using a funnel, pour methyl methacrylate (monomer) from the stock bottle into the test tube until the test tube is just over ½ full. Close the tube tightly with a stopper and repeatedly invert it to thoroughly mix the contents until all the solid dissolves.

   Fill the E-flask with water from the bath, put the test tube in it, and make sure that the cork is very loose! Incubate in the water bath for about 20 - 30 minutes. Maintain the temperature between 60 – 70 °C. If it gets too cool, the polymerization takes forever, and if it gets too hot, lots of bubbles form in the plastic. During this time the polymerization starts, and the solution starts to become viscous toward the end of the incubation time. At this point, objects can be placed into the polymerizing mixture to embed them (i.e., paper clip, plant seed or leaf, rock, etc.). Replace the cork loosely. Let the tube incubate until the contents solidify (another 25 - 35 minutes). The polymerization can be detected as a change in the index of refraction that grows upward from the bottom of the test tube.

   Once the plastic is solid, remove the tube from the water bath. Let it cool to room temperature. Wrap it in several layers of paper towel and break the glass (small hammer, step on it, etc.). Be careful not to hurt yourself! Dispose of glass fragments in the box at the front of the lab.

2. **Preparation of Nylon-6,6**

   **Caution:** Both monomer solutions are skin irritants so handle them carefully and wear goggles. Place 25 mL of adipoyl chloride solution (1% by volume in a solvent of hexanes) in a 250 mL beaker. To a second 250 mL beaker, add 25 mL of hexamethylenediamine solution (IUPAC name 1,6-hexanediame; 1.15% by volume in water). Add 1 ml of 1 M NaOH solution to the hexamethylenediamine solution and mix well. The purposes of the NaOH are to make sure that all the amine functional groups are unprotonated (electrically uncharged) and to react with protons from the HCl that will be released when the polymerization reaction occurs.

   Tilt the beaker containing the hexamethylenediamine at a 45° angle and gently pour all of the adipoyl chloride solution onto the aqueous surface of the hexamethylenediamine. Return the beaker to its upright position. Water and hexanes do not mix. The polymerization takes place at the organic/aqueous interface where the monomers dissolved in each of the liquids meet – in other words, the nylon polymer forms only at the interface between the solutions.

   Use a small wire with a hook at the end to gently snag the nylon at the middle of the interface and pull it slowly straight up out of the solution. Try to keep the emerging strand centered in the 250 mL beaker as you drape the end of the nylon over the outside of a 400 mL beaker. Slowly rotate the 400 mL beaker to form a continuous coil of nylon around it until no more nylon is present at the interface in the 250 mL beaker. Rinse the nylon coil with 40 mL of 50% ethanol/water and then with flowing tap water. Slowly peel the strand of nylon off of the beaker and squeeze dry between two paper towels.
Report/Summary  See the lab syllabus for Week 2’s post-lab assignment description and a list of what is due at the beginning of Week 3.

Note:
Remember to cite the source in American Chemical Society (ACS) format. The direct link on our website which can help with proper citation format is:
http://www.linfield.edu/chem/assets/files/Courses/CHEM%20321/ACSQuickRefGuide.pdf

To cite an internet source, use the following format:
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You will also need to cite the references where you found the physical properties for your pre-lab (or any additional external sources utilized as you write your summary).