Experiment 15: Exploring the World of Polymers

Objective: In this experiment, you will explore a class of chemical compounds known as *polymers*. You will synthesize and modify polymers, test their properties and use a fabrication technique to produce an object from a polymer.

Introduction

(See Tro, pp 557-560.)

The importance of both natural and synthetic polymers in our lives cannot be overestimated. The desirable properties of these **macromolecules**, such as tensile strength and flexibility, make them extremely useful both in nature and in the manufacture of products that we use every day. Examples of naturally occurring polymers include such things as wood, cotton, paper and silk as well as proteins and DNA. Plastics, Teflon®, Plexiglass™ , and Mylar™ are all examples of commercially available synthetic polymers.

Polymers are made up of smaller repeating units, called **monomers**, which are linked together by covalent bonds. The **polymerization** processes by which polymers are synthesized fall into two categories.

Addition polymers are formed by addition reactions that link together monomers containing multiple bonds. One example of this process is the polymerization of styrene to form polystyrene.

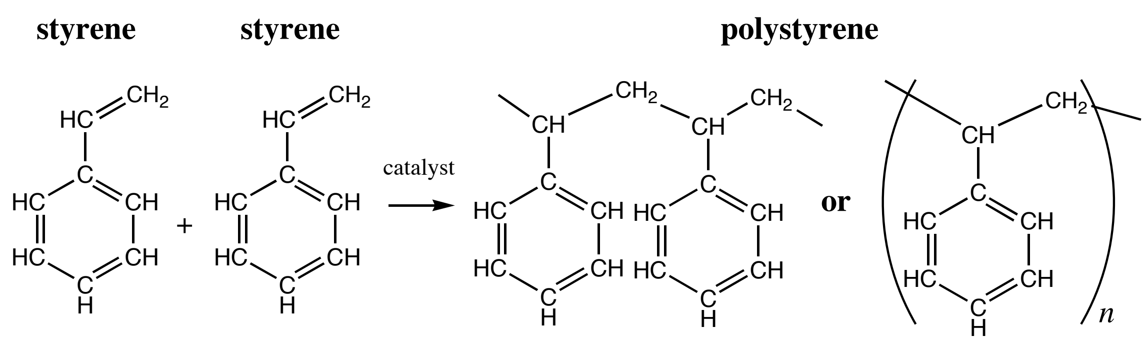


Figure 1. Reaction scheme for the preparation of polystyrene.

Polystyrene is used in molded plastics and as a foam insulator. You will synthesize polystyrene in today's experiment.

Condensation polymers are formed by linking together monomers with the release of a smaller molecule, such as water or hydrogen chloride. An example of this type of polymerization is the formation of a laminating resin called Glyptal from the reaction of glycerol and phthalic acid. The molecule that is eliminated in this case is water (see Figure 2 on top of next page). You will synthesize Glyptal in today's experiment.

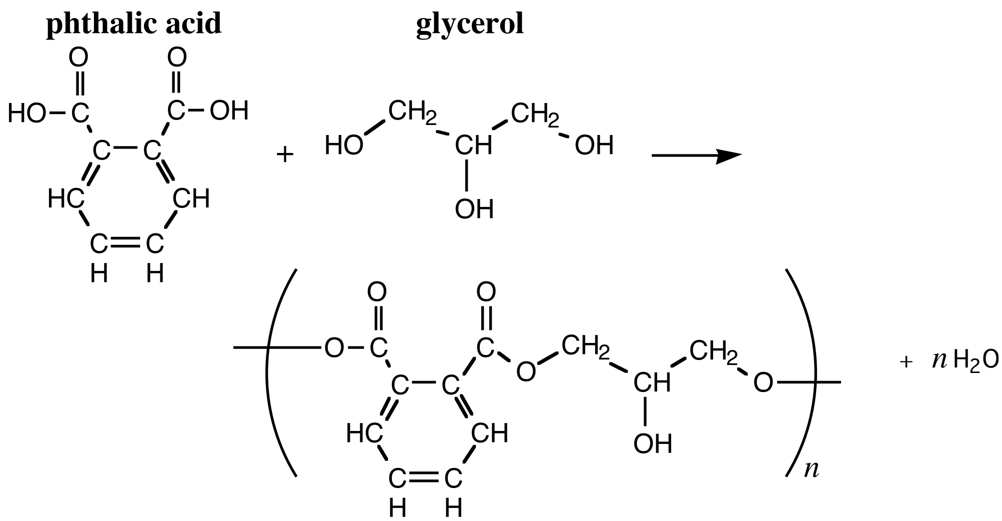


Figure 2. Reaction scheme for the preparation of Glyptal.

Glyptal has an additional feature to it in that its properties can be modified by a process known as **crosslinking**. A crosslinked polymer is one in which the long chains of molecules are joined to each other by covalent bonds. The link that is formed in Glyptal is shown below in Figure 3, along with a diagram which illustrates the cross-linking of several polymer strands.

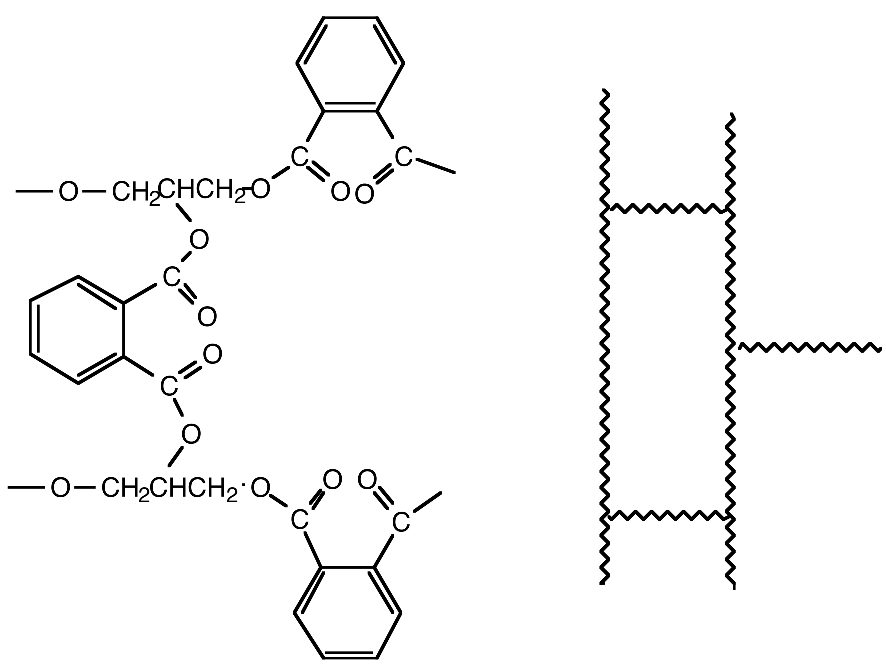
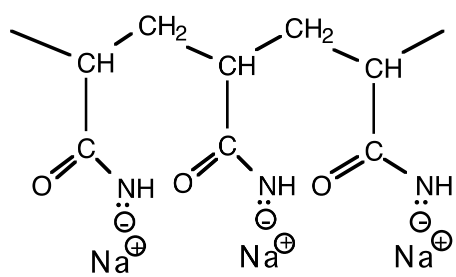


Figure 3. Crosslinking in Glyptal resin.

The properties of this polymer change after it is crosslinked so that it becomes irreversibly hardened. It is possible to modify the properties of many polymers by generating crosslinks such as these. You will experiment with crosslinking the polymer in Elmer's glue.

A number of different processes are used to turn synthetic and natural polymers into useful objects. These processes (along with some examples) include molding (toys, kitchen utensils), extrusion (toothbrush fibers, weather stripping), foaming (coffee cups, cushions) and casting (medical devices). In this experiment, you will use the technique of extrusion-blow molding in which a tube of polymer is blown up while in the molten state inside of a hollow mold. Almost all plastic bottles are formed by some variation of this method.

Throughout the experiment, you will test the various properties of the polymers that you synthesize and modify. Some polymers have exceptional properties and capabilities that can make them extremely useful to us. You will observe the property of one such polymer, sodium polyacrylate (the sodium salt of polyacrylamide).



This polymer can absorb up to 800 times its own weight of water. The ionic portion of the macromolecule is directed toward the center of the structure. When water is added to dry sodium polyacrylate, the water molecules are drawn into the center of the polymer in an attempt to dilute the salt, and the polymer expands.

It is very important that you record careful observations throughout the experiment. You will be asked to describe the appearances and properties of the various polymers in your lab report.

Procedure

Record detailed observations throughout the experiment.

Polymer synthesis

Addition polymer—Polystyrene

Place 20 drops of styrene into a disposable 13 x 100 mm test tube (provided by your TA). **Caution! Styrene is a flammable liquid! Keep styrene away from all Bunsen burner flames. It is also a suspected carcinogen. Wear gloves!** *Using a wooden or plastic utensil*, add a few grains of dibenzoyl peroxide and shake the tube to mix. **Caution! Dibenzoyl peroxide is explosive when heated. Use only a few grains!**

Label the test tube with your name, and place it in a sand bath that has been set up by your instructor. Allow the solution to heat for 90 minutes. You may proceed with the rest of the experiment as it heats.

When the material in the test tube has turned slightly yellow, remove the test tube from the sand bath and cool it on ice. Try to shake the polymer out of the test tube. If it has not separated from the glass sufficiently to do this, you may wrap the test tube in a cloth towel and *carefully* break it so that the polymer can be removed. Wear gloves when doing this! Describe the appearance and properties of your polymer.

Test the solubility of the polymer by grinding a few chunks in a mortar and pestle and placing the powder in two test tubes. Add water to one of the test tubes and acetone to the other test tube. Record your observations. Dispose of the acetone mixture in the appropriate **Laboratory Byproducts Jar**.

Discard the polystyrene in the appropriate **Laboratory Byproducts Jar**.

Condensation Polymer—Glyptal Resin

The following procedure must be carried out in a fume hood!

Obtain a paper cup. Place 5 g of phthalic anhydride and 0.25 g of sodium acetate in a disposable 18 x 150 mm test tube (provided by your TA). Add 2 mL of glycerol. Using your test tube clamp (*not* crucible tongs), *carefully* heat the mixture over a Bunsen burner flame (see TECH I.D). Heat the top of the contents first and work toward the bottom as the mixture melts. Move the test tube in and out of the flame as you do this. Note when the mixture begins to boil and then continue to heat for 3 to 4 minutes while moving the test tube in and out of the flame. The solution should turn yellow in color. If it begins to turn brown, stop heating.

Pour the solution into the paper cup. Allow the solution to thoroughly cool, then tear off the cup to recover your polymer. Dispose of the test tube in glass waste. Describe the appearance and properties of your polymer.

Test the solubility of the polymer as you did for polystyrene. Dispose of the acetone mixture in the appropriate **Laboratory Byproducts Jar**.

Modification of polymers

Experiment on crosslinking with Glue

> **It is recommended that gloves *not* be worn when working with Elmer's glue. It is easier to remove glue residue from your skin than from the gloves.**

Obtain a plastic cup and pour 15 mL of Elmer's white glue into it. Add 15 mL of water and stir well. Next, add 10 mL of saturated borax solution (sodium borate, $Na_2B_4O_7$) and stir well.

Remove the solid material and pull it off the stirrer. The material will be sticky for about one or two minutes. Pour any excess liquid left in the cup into the appropriate **Laboratory Byproducts Jar**.

Rinse the cup with warm water. Perform another crosslinking experiment using 15 mL of glue and different amounts of water and borax (from 5 to 15 mL of borax and from 0 to 30 mL of water). Compare the properties of the two polymers (strength, bounce, stretchiness, etc.). Observe the properties of at least two other students' polymers who used different amounts of water and borax in their second preparation. Record these observations.

Polymer fabrication

Polyethylene bottle

Heat one end of a 10-15 cm piece of polyethylene tubing in a Bunsen burner flame. When the polymer is in the molten state, crimp the end with crucible tongs to seal it off—allow it to cool somewhat before opening the tongs. Re-heat the tubing at the closed end by passing it in and out of the flame until 1-2 inches of the tubing is in the molten state (looks transparent instead of opaque). Place the molten tubing into a small *wide-mouth* jar or vial. Blow air into the open end of the tube so that it expands within the jar.

Let the expanded tubing cool in the jar for about 2 minutes. Because the polymer is flexible, you should now be able to easily remove it from the “mold”. Show your polyethylene bottle to your TA.

Polymer properties

Superabsorbers

Weigh 1 g of sodium polyacrylate and place it in a 150 mL beaker. Add 50 mL of water to the beaker from a graduated cylinder. Record your observations.

Add another 50 mL of water to the beaker. Record your observations.

Add 0.5 g of sodium chloride to the beaker and stir. Record your observations.

To recover the polymer so that it can be easily disposed of, collect it by vacuum filtration. Pour the water in the filter flask down the sink, and scrape the collected polymer off the filter paper and into the appropriate **Laboratory Byproducts Jar**. **Do not place any of the polymer in the sink as it will clog the drains!**

Questions

1. Describe the appearance of the polystyrene that you made. Describe the appearance of the Glyptal resin. Compare the properties of Glyptal with those of polystyrene.

2. Describe the changes in bonding and hybridization of the carbon atoms that take place during the polymerization of styrene to form polystyrene.

3. Summarize the various effects which you observed cross-linking to have on the properties of a polymer.

4. Calculate the mole ratio for the reactants used in the Glyptal resin preparation:

$$\frac{\textbf{number of moles phthalic anhydride}}{\textbf{number of moles glycerol}}$$

glycerol: 92.09 g/mol; density = 1.261 g/mL
phthalic anhydride: 148.12 g/mol

Should the mole ratio for this *crosslinked* polymer be greater than, less than or equal to 1? Explain.

5. Describe what you observed when different amounts of water and borax solution were used in preparing the cross-linked polymer from glue. Name two or three properties which you feel would be desirable to have in this polymer, and what recipe you would use to obtain this "best" polymer.

6. What are some possible commercial uses for sodium polyacrylate? Provide an explanation for what happens to the expanded polymer after salt is added to it.

7. Could ethylene glycol (structure shown) form a condensation polymer with phthalic anhydride in the same way that glycerol does? Explain.