Experiment 6.
Natural and Synthetic Polymers:
The preparation of Nylon

Experimental Procedure

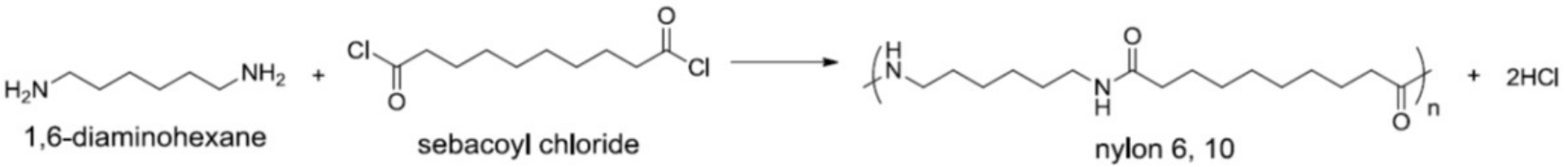
- Objectives
- Introduction
- Experimental Procedure

OBJECTIVES

- To learn some distinctions between natural and synthetic polymers
- To examine the monomers of two common fibers
- To become familiar with some of the physical and chemical properties of two common polymers
- To prepare the important polymer nylon

INTRODUCTION

In this experiment we use a polycondensation reaction, which involves the reaction of two multi–functional compounds (such as a diamine and a diacid chloride) to yield a polymer with a low molar mass by product; in our experiment, HCl is the by–product. If the monomers react between two immiscible phases (such as water and hexane), the reaction is called interfacial polycondensation.



EXPERIMENTAL PROCEDURE & RESULT VIDEOS

PART A. Hydrolysis of a Polymer, Cellulose: Formation of a Monomer

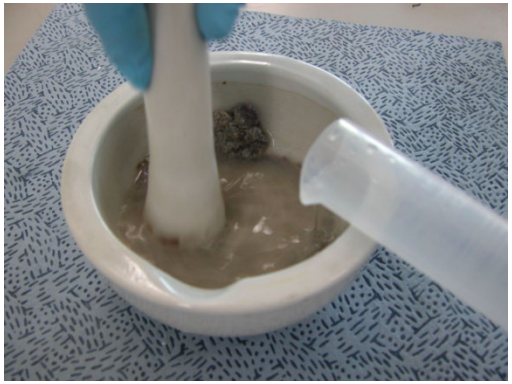
1. Make a compact wad of cotton (must be natural cotton) by compressing a cotton ball (about 4 cm in diameter), and place it in a large mortar.

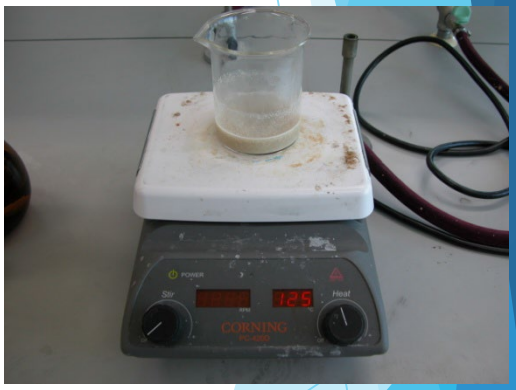
2. Pour 3 mL of concentrated sulfuric acid, H_2SO_4, over the cotton and grind it to a fine pulp with the pestle. Caution : Concentrated H_2SO_4 is dangerous! If you come In contact with acid, wash it off immediately.



3. Cautiously add 50 mL of water to the pulp, stir, and transfer all of the material to a 250 mL beaker. Boil the cellulose solution very gently for 30 minutes.

Replace any water that evaporates during the boiling process

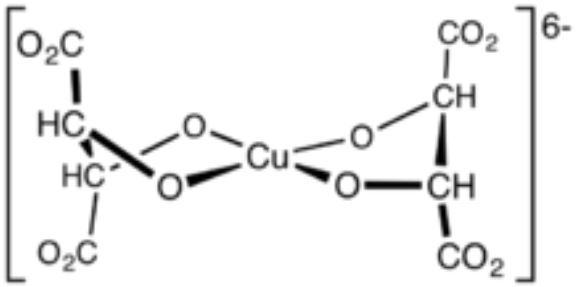




4. When the hydrolysis solution has cooled, neutralize it by stirring in 20% sodium hydroxide solution until the solution is just basic to litmus.

5. Confirm the presence of the monomer in the aqueous solution by means of Fehling's test as follows.

Fehling's solution is prepared by combining two separate solutions: Fehling's A, which is a deep blue aqueous solution of copper(II) sulfate, and Fehling's B, which is a colorless solution of aqueous potassium sodium tartrate (also known as Rochelle salt) made strongly alkali with potassium hydroxide.



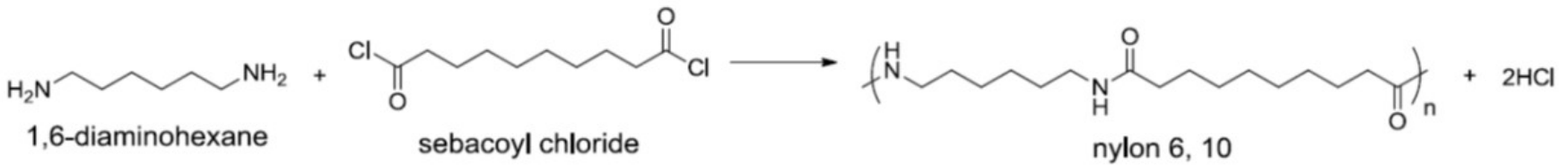
6. Mix 1 mL of the hydrolysis solution with 2 mL of Fehling's reagent and heat in a water bath for 10 to 15 minutes.

Formation of a reddish brown precipitate of cuprous oxide, Cu_2O, constitutes a positive test for the monomer.

PART B. Synthesis of a Polymer, Nylon

1. Clean and dry a 50 mL beaker. To the beaker add 10 mL of **a 2% solution of sebacoyl chloride in hexane.**

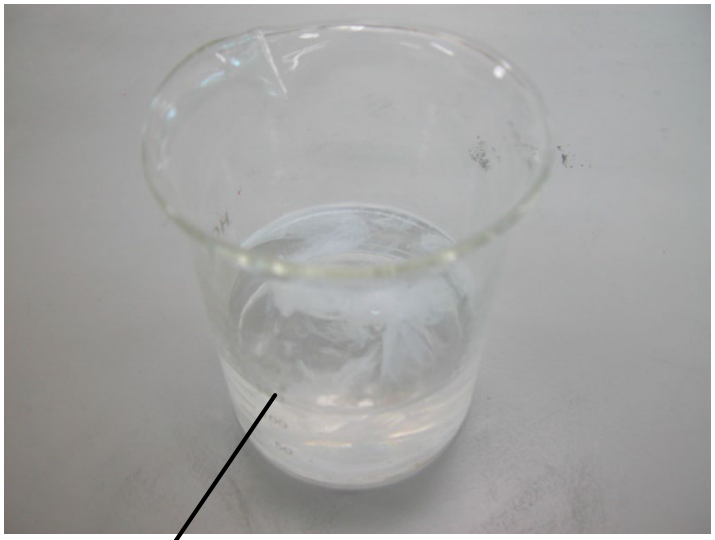
2. Gently pour the sebacoyl chloride in hexane solution on top of 10 mL of the **hexamethylenediamine (3.5% hexamethylenediamine + 3% NaOH)** in water solution in the beaker, using a glass rod to pour down; *the layers must not mix together*!. Put a piece of paper towel on the floor, and place the beaker on the towel.



3. Draw a thread out of this interface using a forceps, and draw the thread out of the beaker. Using a glass rod as a spool, slowly wind up the thread as you draw it out. (See below):

4. Pull out about 1m of rope and cut it with a pair of scissors. Put a piece of paper towel under the rope so that the chemicals do not drop on the desk.

5. Dip the rope into 50% aqueous alcohol solution (ethyl alcohol 20 mL + distilled water 20 mL), and thoroughly rinse it to remove all of the chemical reagents. Stretch out the nylon rope on a paper towel under the hood and press it with paper towels.



50% aqueous alcohol solution

6. Test the strength of your nylon by pulling on both ends of a piece.

7. Using forceps, hold a piece of nylon in a flame to see if it will burn.

8. Determine whether your nylon piece are soluble in, or are affected by, the following solvents: acetone, commercial liquid bleach, 25% sulfuric acid, 25% sodium hydroxide, and concentrated sulfuric acid (a small beaker of sulfuric acid will be provided under the hood–dip a piece of fiber Into the solution).

9. Now repeat all of these tests on cotton and compare the results with those for nylon.

10. Determine melting point of the nylon.

DISPOSAL

Discard the solution in the Waste Basic Solution container.