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#9 Condensation Polymerization: Preparation of Nylon 6/10

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I. INTRODUCTION

Description

One of the classic experiments in the preparation of nylon is the interfacial polymerization. Students produce nylon 6/10 by the reaction between a diacid chloride and a diamine. This reaction occurs at the interface of two solutions. The material is wound up on a spool or other device and at some point it seems that the winding will never end.

Student Audience

This experiment is recommended for chemical technology students who have had organic chemistry. Students who have not taken organic chemistry will be able to perform the lab but will not get as clear an understanding of how the reaction is occurring.

Goals for the Experiment

By doing this lab the student will:

- demonstrate the synthesis of nylon 6/10,
- calculate the %-yield by mass,
- identify this polymer as thermoplastic or thermoset,
- write the formula for the repeat unit,
- describe the end product use of this polymer, and
- determine the melting point of the polymer.

Recommended Placement in the Curriculum

This experiment is recommended for use in the discussion of any of the following topics:

- condensation polymerization,
- %-yield,
- thermoplastic and thermoset polymers,
- melting point, and
- applications of polymers.

II. STUDENT HANDOUT

Condensation Polymerization, Preparation of Nylon 6/10

Scenario

The company you work for, Specialty Polymers Corporation, has just been awarded a government contract to develop a polymer formulation for an injection molded connector to be used in a new military jet engine. The tensile strength, stiffness, and dimensional stability requirements for this part suggest that a glass fiber reinforced nylon 6/10 formulation would meet the specifications. As part of this project, you have been asked to synthesize the nylon 6/10 base resin to be used in the formulations.

Safety, Handling, and Disposal

- Wear your safety goggles, gloves, and lab coat while working in lab.
- Hexamethylenediamine (1,6-diaminohexane) is irritating to the skin, eyes, and respiratory system.
- Sodium hydroxide is extremely caustic and can cause severe burns. Contact with the skin and eyes must be avoided.
- Sebacoyl chloride is corrosive and irritating to the skin, eyes, and respiratory system.
- Hexane and acetone are flammable. Both hexane and acetone vapors can irritate the respiratory tract.
- Should skin contact occur with any of these reagents, wash contaminated area immediately.
- Any unused polymerization mixture should not be poured into the sink but should be stirred until no further polymer is formed. Any remaining solvent/material will be disposed into a provided lab waste container.
- The polymer should be washed extensively with alcohol and water to get rid of HCl.

Materials

- sebacoyl chloride [$\text{ClCO}(\text{CH}_2)_8\text{COCl}$]
- hexane
- hexamethylenediamine (HMDA; 1,6-diaminohexane) [$\text{H}_2\text{N}(\text{CH}_2)_6\text{NH}_2$]
- sodium hydroxide
- 10-mL graduated cylinder
- 100-mL graduated cylinder
- funnel
- 3 250-mL beakers
- (optional) food color or phenolphthalein
- analytical balance
- 2 glass stirring rods
- forceps or tongs
- ethanol
- (optional) oven
- (optional) melting point apparatus

Procedure

1. To prepare the sebacoyl chloride solution, place 3.0 mL of sebacoyl chloride in 100 mL of hexane in a 250-mL beaker.

- To prepare the hexamethylenediamine (HMDA) solution, add 2.2 g of HMDA and 1.5 g of NaOH to 50 mL of distilled water into a 250-mL beaker. Stir to dissolve.

Caution! Always add NaOH pellets to the water and not the other way around. Considerable heat is generated when NaOH dissolves.

- (optional) Food color or phenolphthalein may be added to the aqueous hexamethylenediamine solution to enhance the visibility of the liquid interface.
- To layer the two solutions, pour the sebacoyl chloride solution slowly through a funnel placed such that its outlet is just over the surface of the hexamethylenediamine solution.
- Grasp the polymeric film which forms at the interface of the two solutions with forceps and slowly pull it from the beaker as a continuously forming fiber. The polymer will continue to form until one of the reactants is used up. *Which reactant is observed to be limiting? That is, which reactant is used up first?*
- Wash the polymer thoroughly with water and finally with ethanol, and air dry.
- (optional) The nylon can be placed in the oven at about 80°C to help speed up drying. The oven should be properly vented and caution should be used because the hexane and alcohol are flammable.
- Weigh the dry polymer.
- (optional) Determine and record the melting point of the polymer.
- Use the weight of the polymer (step 8), the limiting reagent (step 5), and the reaction equation to calculate the %-yield by mass of this product.



- Turn the sample into your instructor along with your results.

Questions

- What does the 6/10 mean in the name nylon 6/10? What is the scientific name for nylon 6/10?
- How do you know that the product is a polymer?
- How would you determine the number of repeat units?
- Is this polymer a thermoplastic or thermoset material? How do you know?
- Write the formula for the repeat unit for this polymer.
- What are the mass and melting point of your product?
- “Condensation” polymerization involves the formation and loss of a small molecule (such as water) as each monomer is added to the growing polymer chain. What small molecule would be formed by the reaction of sebacoyl chloride and hexamethylenediamine?
- Why is sodium hydroxide added to the HMDA?
- What are the major structural features of nylon 6/10 that influence its properties?

10. What is the commercial process for making nylon 6/10 fibers?
11. What are the commercial applications of this material?

III. INSTRUCTOR NOTES

Condensation Polymerization, Preparation of Nylon 6/10

Purpose

To produce nylon 6/10 by interfacial polymerization and identify its properties.

Time Required

This laboratory investigation should take 2.5-3 hours. See comments in “G. Procedural Tips and Suggestions” regarding drying time.

Suggested Group Size

It is recommended that each student do this activity individually.

Materials

Per class

- sebacoyl chloride [$\text{ClCO}(\text{CH}_2)_8\text{COCl}$]
- hexamethylenediamine (HMDA; 1,6-diaminohexane) [$\text{H}_2\text{N}(\text{CH}_2)_6\text{NH}_2$]
- sodium hydroxide
- ethanol
- (optional) oven
- (optional) melting point apparatus

Per student

- 10-mL graduated cylinder
- 100-mL graduated cylinder
- funnel
- 3 250-mL beakers
- (optional) food color or phenolphthalein
- analytical balance
- 2 glass stirring rods
- forceps or tongs

Safety, Handling, and Disposal

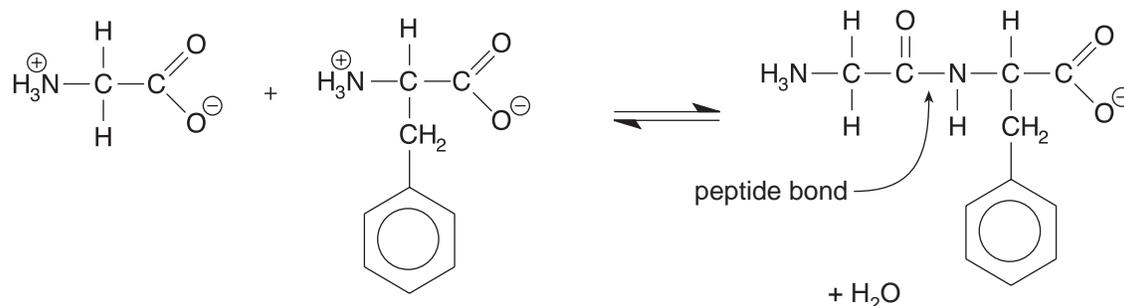
- Students should wear safety goggles, gloves, and lab coats.
- Hexamethylenediamine (1,6-diaminohexane) is irritating to the skin, eyes, and respiratory system.
- Sodium hydroxide is extremely caustic and can cause severe burns. Contact with the skin and eyes must be avoided.
- Sebacoyl chloride is corrosive and irritating to the skin, eyes, and respiratory system.
- Hexane and acetone are flammable. Both hexane and acetone vapors can irritate the respiratory tract and, in high concentrations, can be narcotic.
- Should skin contact occur with any of these reagents, wash contaminated area immediately.
- Any unused polymerization mixture should not be poured into the sink but should be stirred until no further polymer is formed. The solid may be disposed of along with other nonhazardous solid waste.
- Prepare a lab waste container for any remaining solvent/material. Dispose of this material according to local, state, or federal regulations.

Points to Cover in Pre-Lab

- Make sure students understand the safety, handling, and disposal procedures of this experiment.
- Explain to them that nylon is an example of a synthetic polyamide.
- Explain condensation and step-growth polymerization.
- If students will be taking melting points and are not familiar with the melting point apparatus, demonstrate how it works.

Procedural Tips and Suggestions

- Ligroin can be substituted for the hexane if desired.
- You may wish to have your students wind up nylon on their forceps, tongs, or stirring rod or wind around a soft drink bottle as it forms.
- Make sure the students wash their material well before handling it. Oven drying the product will speed up the investigation. If you have them air dry, they may have to complete the investigation another lab period.
- If the students are not familiar with condensation polymerization, it might help to provide an example which splits out water to introduce the topic. One possibility is the reaction of two amino acids to produce a dipeptide:



Plausible Answers to Questions

1. What does the 6/10 mean in the name nylon 6/10? What is the scientific name for nylon 6/10?
 A: Various nylons are described by a numbering system that indicates the number of carbon atoms in the monomers. Nylons from diamines and dicarboxylic acids use two numbers, the first representing carbons in the diamine and the second the carbons in the dicarboxylic acid. In this case, the diamine has a six-carbon formula and the dicarboxylic acid has ten, hence the name nylon 6/10. The scientific name is poly (hexamethylene sebacamide).
2. How do you know that the product is a polymer?
 A: This is not a trivial question. The definitive way to determine if it's a polymer is to run an IR or NMR and compare to a reference spectra. However, if instrumentation is not available, then one can infer that it may be a polymer from observations including that a viscous substance forms at the interface of the two immiscible solutions, this gooey substance can be drawn into a long rope or fiber, and the reagents being consumed can react to form a polymer. Of course, determining melting point and density and comparing it to known material would also help to identify the polymer.
3. How would you determine the number of repeat units?

A: The most often used way to determine the average number of monomers in a polymer is to measure the number average molecular weight (M_n) by size exclusion chromatography and divide by the molecular weight of the repeat unit. This number is an average because there are polymer chains different length (i.e., a molecular weight distribution) in the product.

If the molecular weight is not too high, it may be possible to determine M_n from the NMR spectrum of the polymer by comparing the relative peak areas for the end groups with the peak area for the backbone groups.

It may also be possible to use titration methods to quantify the end groups.

A newer, more accurate but more expensive method for obtaining absolute molecular weights of polymer chains (including proteins) is matrix assisted laser desorption/ionization mass spectroscopy (MALDI mass spec).

4. Is this polymer a thermoplastic or thermoset material? How do you know?

A: Nylon 6/10 is observed to be a thermoplastic polymer because it softens when heated and hardens when cooled in a reversible physical process.

5. Write the formula for the repeat unit for this polymer.



6. What are the mass and melting point of your product?

A: The mass will depend on the individual student. The melting point is approximately 130 °C.

7. “Condensation” polymerization involves the formation and loss of a small molecule (such as water) as each monomer is added to the growing polymer chain. What small molecule would be formed by the reaction of sebacoyl chloride and hexamethylenediamine?

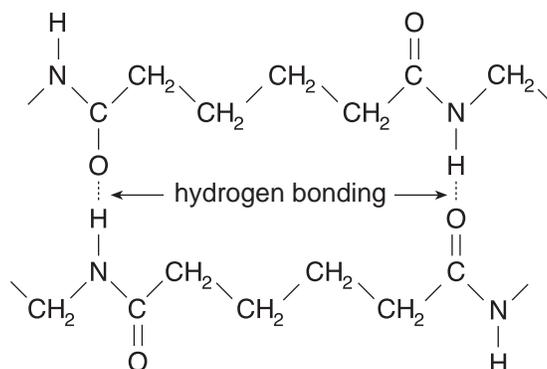
A: HCl, hydrochloric acid.

8. Why is sodium hydroxide added to the HMDA?

A: Sodium hydroxide is added to HMDA in order to help neutralize the HCl given off as a by-product during the formation of nylon 6/10.

9. What are the major structural features of nylon 6/10 that influence its properties?

A: The main structural feature is the amide functional group. In nylons, the hydrogen on the nitrogen can readily hydrogen bond to the carbonyl oxygen of the amide on an adjacent chain. (See figure below.) This allows the chains to all line up and pack very tightly imparting high crystallinity to the nylon. Because of this tight packing, the tough crystalline nylon fiber will not stretch very much. In addition, as the average molecular weight increases, so does the fiber tenacity and modulus; whereas the crystallization rate decreases.



10. What is the commercial process for making nylon 6/10 fibers?

A: Nylon fibers are formed by a melt spinning process in which the melted polymer is forced through a spinnerette, quenched, collected on a glass wheel, stretched to about four times its original length, and finally wound.

11. What are the commercial applications of this material?

A: Nylon 6/6, 6, and 6/10 can be used for injection molded articles such as “unlubricated gears, bearings, and antifriction parts, mechanical parts that must function at high temperatures and resist hydrocarbons and solvents, electrical parts subjected to high temperatures, and high-impact parts requiring strength add rigidity. Nylon is also used in packaging and for many general purpose applications.” (See Smith reference for more detail.) Nylon fibers (primarily nylon 6/6) are found in clothing, carpeting, etc.

References

- Shakhashiri, B.Z. *Chemical Demonstrations, Volume 1*; The University of Wisconsin Press: Madison, 1983; pp. 213-215.
- Smith, W.F. “Principles of Materials Science and Engineering,” Third Edition, pp. 374–377.