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12 Condensation Polymerization: Preparation of Two Types of Polyesters

Submitted by: Arturo Contreras, Visiting Scholar, Center for Chemical Education, Miami University, Middletown, OH; 1996–1997.

I. INTRODUCTION

Description

In this lab, students will synthesize two examples of condensation polymers, a linear polyester and a cross-linked polyester. Both esters are produced by the reaction of an acid anhydride (phthalic anhydride) and an alcohol. Phthalic anhydride is the anhydride of phthalic acid, 1,2-benzenedicarboxylic acid. In the case of the linear polyester, the alcohol is a diol (ethylene glycol). Thus both the alcohol and the acid (anhydride) have two reaction sites and a linear polyester is produced. This polyester is similar to the more familiar Dacron.

When more than two functional groups are present in one of the monomers, the polymer chains can be cross-linked to make a three-dimensional network. The alcohol used in the synthesis of the cross-linked polymer is the triol, glycerol. This polyester (Glyptal®) has a structure which is more rigid than the linear structure and therefore has different properties.

Student Audience

This experiment is recommended for high school chemistry students and freshman or sophomore college chemistry students including chemical technology majors. Students who have taken organic chemistry will have a clearer understanding of how the reaction is occurring.

Goals for the Experiment

By doing this lab the student will:

- demonstrate the synthesis of two types of polyester,
- observe basic properties of both linear and cross-linked materials,
- identify the polymers as either thermoplastic or thermoset,
- write the equations for the reactions including the products' repeat units,
- determine the melting points of products, and
- describe end product uses of these polymers.

Recommended Placement in the Curriculum

This experiment is recommended to be used during a discussion of any of the following topics:

- linear versus cross-linked polymers,
- condensation polymerization,
- physical properties of polymers, and
- the relationship of properties to molecular structure.

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II. STUDENT HANDOUT

Condensation Polymerization: Preparation of Two Polyesters

Scenario

You work in the research lab of PolyEster Incorporated. PEI does not normally use phthalic anhydride in its production of polyesters. However, one of its suppliers, Acid Anhydrides, Inc. has overproduced phthalic anhydride and offered it to PEI at an excellent price. Your job is to use phthalic anhydride in the synthesis of several polyesters and determine the properties of these products. PEI management will use this information to decide if either or both will fit into its product line.

Safety, Handling, and Disposal

- Wear your safety goggles and lab coat while working in lab. Rubber gloves should be worn while performing this experiment.
- Read the Materials Safety Data Sheets for all chemicals used in this investigation.
- Phthalic anhydride is a skin and respiratory irritant. It is toxic if ingested.
- Ethylene glycol (antifreeze) is toxic by ingestion and inhalation. A lethal dose of ethylene glycol is about 100 mL.
- Glycerol can cause allergic skin and eye irritation in some people.
- Should skin contact occur with any of these reagents, wash contaminated area immediately.
- Use appropriate caution with the Bunsen burner.
- The polyesters may be difficult to remove from the test tubes. If you use organic solvents to clean the test tubes, do so in the hood and dispose of the solutions appropriately.
- Any unused polymerization mixture should not be poured into the sink. Any remaining solvent/material must be disposed of into the organic lab waste container.

Materials

- phthalic anhydride
- anhydrous sodium acetate
- ethylene glycol
- glycerol
- analytical balance
- 2 large test tubes (20- x 150-mm)
- 1-mL graduated pipette
- Bunsen burner
- ring stand
- 2 utility clamps (not rubber coated clamps)
- (optional) melting point apparatus
- (optional) small test tubes or spot plate
- (optional) assorted solvents such as water, alcohol, acetone, etc.

Procedure

1. Place both 2.0 g of phthalic anhydride and 0.1 g of anhydrous sodium acetate into each of the two test tubes.
2. To one test tube add 0.8 mL of ethylene glycol, and to the other add 0.8 mL of glycerol.

3. Clamp both tubes, at about a 45° angle, so that they can be heated simultaneously with the burner flame. Avoid rubber coated clamps.
4. Heat the tubes gently until the solutions appear to boil and continue to heat gently for about 3 minutes. Avoid overheating which may cause a dark mass to form in the glycerol reaction.
5. Allow the tubes to cool and compare the viscosities of the two polymers.
6. Remove the materials from the test tubes. This may be difficult. If so, heat the test tube and polymer in a hot water bath to reduce the viscosity of the polymer.
7. On a spatula, heat about 0.1 g of one polymer gently and determine if you can draw out a fiber with a glass stirring rod. Repeat with the second polymer.
8. (optional) Determine melting points for the two products.
9. (optional) Allow the polyesters to cure for 90 minutes or more and test properties such as hardness, scratch resistance, and solubility in different solvents (water, alcohol, acetone, etc.)
10. Turn in samples of your products along with the properties determined above and the answers to the questions.

Questions

1. Esterification is a condensation reaction producing a small molecule called the condensate along with the ester. What condensate is produced during the synthesis of the linear and cross-linked polyesters? What happened to the condensate?
2. Write the reactions for the production of the linear and cross-linked polyesters including the repeat units for the products.
3. What type of polymerization (step-growth or chain-growth) is occurring during the synthesis of these two polyesters? Explain.
4. Which one of these polyesters was thermoset? How do you know? Why is it a thermoset plastic?
5. If you were choosing a polymer to use under high temperature conditions, such as in an internal combustion engine, which of these polymers would you choose? Why?
6. What products could be made using each of these two polyesters?

III. INSTRUCTOR NOTES

Condensation Polymerization: Preparation of Two Polyesters

Purpose

To produce two polyesters (linear and cross-linked) and compare their properties.

Time Required

This lab requires about 1 hour (plus curing time if step 9 is done).

Suggested Group Size

It is recommended that each student do this lab individually or with a partner.

Materials

Per class

- phthalic anhydride
- sodium acetate
- ethylene glycol
- glycerol
- analytical balance

Per student or pair of students

- 2 large test tubes (20- x 150-mm)

  ➤ Disposable test tubes are recommended so they can be discarded at the end of the investigation. (See “Procedural Tips and Suggestions.”)

- 1-mL graduated pipette
- Bunsen burner
- ring stand
- 2 utility clamps (not rubber coated clamps)
- (optional) melting point apparatus
- (optional) small test tubes or spot plate
- (optional) assorted solvents such as water, alcohol, acetone, etc.

Safety, Handling, and Disposal

- Students should wear safety goggles and lab coats while working in lab. Rubber gloves are recommended while performing this experiment.
- Read the Materials Safety Data Sheets for all chemicals used in this investigation.
- Phthalic anhydride is a skin and respiratory irritant. It is toxic if ingested.
- Ethylene glycol (antifreeze) is toxic by ingestion and inhalation. A lethal dose of ethylene glycol is about 100 mL.
- Glycerol can cause allergic skin and eye irritation in some people.
- Should skin contact occur with any of these reagents, wash contaminated area immediately.
- Provide an organic laboratory waste container for any remaining solvent/material and any unused polymerization mixture. 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.
- Discuss the polyesters the students are going to produce including why one must be linear while the other is cross-linked.
- If students will be taking melting points and are not familiar with the melting point apparatus, demonstrate how it works.

Procedural Tips and Suggestions

- Disposable test tubes are recommended so they can be discarded at the end of the investigation. The polyesters are only soluble in organic solvents and difficult to remove from the test tubes. If students use organic solvents to clean the test tubes, be sure they do so in the hood and dispose of the solutions appropriately.

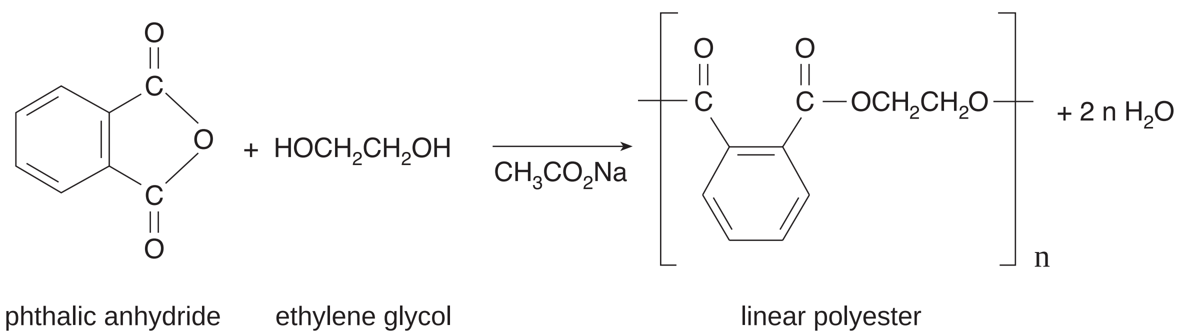
Plausible Answers to Questions

1. Esterification is a condensation reaction producing a small molecule called the condensate along with the ester. What condensate is produced during the synthesis of the linear and cross-linked polyesters? What happened to the condensate?

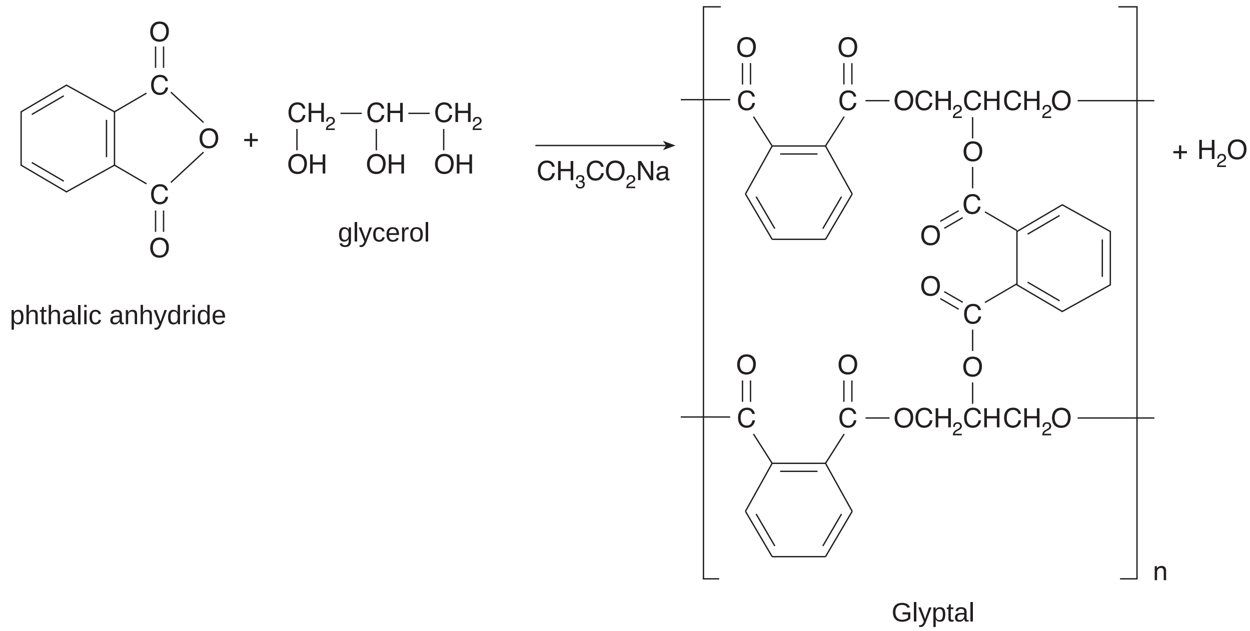
A: Water is the condensate in each of the reactions. It is vaporized under the reaction conditions and thus removed from the reaction. This prevents the reverse reaction (hydrolysis) from occurring and thus high molecular weight polymers can be made (Le Chatelier's principle).

2. Write the reactions for the production of the linear and cross-linked polyesters including the repeat units for the products..

A: linear:



A: cross-linked:



3. What type of polymerization (step-growth or chain-growth) is occurring during the synthesis of these two polyesters? Explain.

A: Both of these reactions are produced by a step-growth process and condensation polymerization. This refers to polymerization in which polymer molecular weight increases in a slow, step-like manner as the reaction proceeds. In condensation polymerization, the monomers are difunctional. As the monomers link together a small molecule, such as water, is released and must be removed to drive the reaction to completion. In a step-growth polymerization, the growing chains may react with each other to form even longer chains.

4. Which one of these polyesters was thermoset? How do you know? Why is it a thermoset plastic?

A: Glyptal, the cross-linked polyester, is the thermoset polymer. It did not soften when heated. The cross-linking keeps the polymer chains from moving with respect to one another when heated.

5. If you were choosing a polymer to use under high temperature conditions, such as in an internal combustion engine, which of these polymers would you choose? Why?

A: The cross-linked, thermoset polyester would be the correct choice for high temperature conditions as it will not soften. Note that if the temperature is too high, the polymer may char and begin to decompose.

6. What products could be made using each of these two polyesters?

A: The linear polyester can be formed into fibers and woven into cloth. This material is used in permanent press wear, rope, fish nets, and tire cord. The cross-linked polyester material is added to latex paints to improve washability and adhesion and is used as an embedding resin.

Variation

Glyptal, the cross-linked polyester made in the procedure, can be used as an embedding resin as described in the following variation. Refer to the main text for safety, handling, and disposal information.

Materials

Per class

- phthalic anhydride
- sodium acetate
- glycerol
- analytical balance

Per student or group of students

- 2 10-cm x 10-cm squares of heavy-duty aluminum foil
- 50-mL beaker
- 100-mL beaker
- large test tube (20- x 150-mm)

➤ Disposable test tubes are recommended so they can be discarded at the end of the investigation.

- 2 tin weighing pans
- 10-mL graduated cylinder
- (optional) food coloring
- Bunsen burner
- ring stand
- utility clamp (not rubber coated)
- small object to embed into the Glyptal

Procedure

a. Construct a sturdy aluminum foil cup by pressing two layers of heavy-duty foil around the bottom of a 50-mL beaker. Trim off excess foil. Place the foil cup inside a 100-mL beaker.
b. Measure out 10.00 g of phthalic anhydride in a tin weighing pan. Measure 0.50g of sodium acetate into another pan. Mix thoroughly together in one of the pans.
c. Carefully transfer the mixture into a 20- x 150-mm test tube. Add 4.0 mL of glycerol and a drop of food coloring, if desired.
d. Clamp the test tube near its top to a ring stand and adjust the inclination of the tube to an angle of about 45 °C. (Avoid rubber-coated clamps.)
e. Heat for 5 to 6 minutes with a low flame by continuously sweeping the flame over the surface of the test tube. Avoid leaving the flame in any one spot.
f. After the solids have dissolved to a clear liquid, heat for an additional 3 minutes. The liquid may darken.
g. Turn off the burner and carefully pour the contents into the cup you made out of foil.
h. Use tweezers to insert a small object, such as a coin, into the mold and allow the polymer to cool to room temperature.
i. After 90 minutes, remove the mold from the beaker and gently peel away the foil.

Reference

Sarquis, M., Ed. *Chain Gang—The Chemistry of Polymers*; Terrific Science Press: Miami University Middletown, Middletown, OH, 1995; pp. 65-68.