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#11 Condensation Polymerization: Preparation of Thiokol (Polysulfide Rubber)

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I. INTRODUCTION

Description

Students will prepare a synthetic elastomer called Thiokol®. This polysulfide was the first synthetic rubber produced by Dr. Joseph C. Patrick in the 1920s. Students will synthesize this material by combining an alkylene dichloride and sodium polysulfide. This is an interfacial polymer reaction but unlike the nylon 6/10 the polymer forms at the bottom of the beaker.

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.

There are serious chemical hazards associated with this lab. Use it only if all safety measures can and will be strictly followed.

Goals for the Experiment

By doing this lab the student will:

- demonstrate the synthesis of a polysulfide rubber,
- calculate the weight %-yield,
- determine the density of product,
- determine the melting point of product,
- determine the solubility of product in various solvents,
- write the repeat unit of the product, and
- describe the end product uses of this polymer.

Recommended Placement in the Curriculum

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

- polysulfide synthesis,
- physical and chemical properties of polysulfides, and
- industrial applications.

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II. STUDENT HANDOUT

Condensation Polymerization: Preparation of Thiokol (Polysulfide Rubber)

Scenario

Along with the economics of manufacturing a chemical, its physical and chemical properties will dictate its end-use applications. A major chemical company has contracted the research company you work for to develop polysulfide coatings and sealants for tanks and pipelines for a variety of solvents under varying climactic conditions. As part of this project, you have been asked to synthesize a polysulfide and investigate its physical and chemical properties.

Safety, Handling, and Disposal

- Wear your safety goggles and lab coat while working in the lab**. Rubber gloves should be worn while performing this experiment.**
- Should skin contact occur while performing this experiment, wash contaminated area with water immediately.
- **This lab should be performed in a fume hood.**
- **1,2-dichloroethane is highly flammable and is toxic by ingestion, inhalation, and skin absorption. It is also considered a strong irritant to eyes and skin, and may reasonably be anticipated to be a carcinogen. Handle with extreme care.**
- Dispose of any solvents into laboratory waste container provided for that purpose. Dichloroethane must be disposed of as a chlorinated compound. This includes both unreacted and waste materials.
- Sodium hydroxide is corrosive to tissue in the presence of moisture, and is considered a strong irritant to eyes and mucous membranes.
- Sulfur is a fire risk in finely divided form. It may cause irritation of the skin and mucous membranes.
- Acetone and ethanol are extremely flammable.
- Sulfuric acid is a strong irritant to tissue. Use great care in mixing with water due to heat evolved. Always add sulfuric acid to water rather than the other way around.
- Nitric acid is corrosive to skin and mucous membranes.
- Thermometers are delicate; they contain mercury, which is toxic.

Materials

- sodium hydroxide pellets
- hot plate
- stirring rod
- sulfur
- ethylene dichloride (1,2-dichloroethane)
- acetone
- nitric acid (conc.)
- sulfuric acid (conc.)
- 6M sodium hydroxide
- methyl alcohol
- analytical balance
- 250-mL beaker

- 50- or 100-mL graduated cylinder
- course filter paper
- funnel
- 10- or 25-mL graduated cylinder
- thermometer, -20 °C to 110 °C
- forceps
- (optional) melting point apparatus
- 5 test tubes

Procedure

Caution! Wear goggles, gloves, and lab coat while performing this experiment.

1. Dissolve 2.0 g of sodium hydroxide in 50 mL of distilled water and heat the solution to the boiling point. Place a stirring rod in the solution to prevent bumping.

   Caution! Always add NaOH to water, not the other way around.

2. In the hood, add 4.0 g of sulfur and stir until all or most of the sulfur has dissolved. The liquid will turn from light yellow to dark brown as the sulfur content of the sodium polysulfide increases.
3. After 5 minutes, allow the solution to cool and decant the dark brown liquid from the undissolved sulfur. If much of the sulfur remains undissolved, it can be more effectively removed by filtration through filter paper.
4. Add 10.0 mL of ethylene dichloride (1, 2-dichloroethane) to the liquid. Warm this mixture to 70–80 °C and stir for 10 minutes. While stirring, a rubbery polymer will form at the interface between the two immiscible liquids and will collect as a lump at the bottom of the beaker.
5. Remove the polymer from the solution and wash it under tap water. Blot dry using paper towels. Note the amount and color of the liquids remaining in the beaker. Dispose of the liquids in the laboratory waste container.
6. Record the physical properties of the product.
7. Find the dry mass of the product and determine the %-yield.
8. Determine the density of the polymer.
9. (optional) Measure the melting point of the product.
10. Labeled 5 test tubes: acetone, conc. nitric acid, conc. sulfuric acid, 6 M sodium hydroxide, and methyl alcohol. Place 1 mL of the appropriate solvent into each test tube. Add a small amount of your polymer product. Shake gently to mix. Record your observations.
11. Turn in the remainder of the sample to your instructor along with your results and the answers to the questions.

Questions

1. Will this material bounce? Do its elastic properties resemble that of a rubber band?
2. Does the polymer have an objectionable feature?

3. There are two types of Thiokol produced. Identify and describe these two types. Which type did you produce?
4. What is the repeat unit of your polymer product?
5. What can be said about the solubility of this polymer?
6. Under what conditions could your product be used as a coating or sealant for tanks and pipelines? Explain your reasoning.
7. Based on your observations (and research), what end product applications would this material fit?

References

Budavari, Susan, Ed. *The Merck Index, Twelfth Edition*; Merck Research Laboratories: Whitehouse Station, 1996; p. 1593.

Shakhashiri, B.Z. *Chemical Demonstrations, Volume 1*; The University of Wisconsin Press: Madison, 1983; pp. 245-246.

III. INSTRUCTOR NOTES

Condensation Polymerization: Preparation of Thiokol (Polysulfide Rubber)

Purpose

To produce a polysulfide rubber by interfacial polymerization and identify its properties.

Time Required

This laboratory procedure should take 2-3 hours.

Group Size

It is recommended that each student do this activity individually.

Materials

Per class

- sodium hydroxide pellets
- hot plates
- stirring rod
- sulfur
- ethylene dichloride (1,2-dichloroethane)
- acetone
- nitric acid (conc.)
- sulfuric acid (conc.)
- 6M sodium hydroxide
- methanol
- analytical balance
- (optional) melting point apparatus

Per student

- 2 250-mL beakers
- 50- or 100-mL graduated cylinder
- stirring rod
- course filter paper
- funnel
- 10- or 25-mL graduated cylinder
- thermometer, -20 °C to 110 °C
- forceps
- 5 test tubes

Safety, Handling, and Disposal

- Students should wear safety goggles and lab coat while working in the lab. **Rubber gloves should be worn while performing this synthesis.**
- Should skin contact occur while performing this experiment, wash contaminated area with water immediately.
- **This lab should be performed in a fume hood.**
- **1,2-dichloroethane is highly flammable and is toxic by ingestion, inhalation, and skin**

absorption. It is also considered a strong irritant to eyes and skin, and may reasonably be anticipated to be a carcinogen. Handle with extreme care.

- Provide a laboratory waste container for the disposal of any excess solvents. Dichloroethane must be disposed of as a chlorinated compound. This includes both unreacted and waste materials.
- Sodium hydroxide is corrosive to tissue in the presence of moisture, and is considered a strong irritant to eyes and mucous membranes.
- Sulfur is a fire risk in finely divided form. It may cause irritation of the skin and mucous membranes.
- Acetone and ethanol are extremely flammable.
- Sulfuric acid is a strong irritant to tissue. Use great care in mixing with water due to heat evolved. Always add sulfuric acid to water rather than the other way around.
- Nitric acid is corrosive to skin and mucous membranes.

Points to Cover in Pre-Lab

- Make sure students understand the safety, handling, and disposal procedures of this synthesis and investigation.
- Thiokol® is a trademark used for any of various polysulfide polymers in the form of liquids, water dispersions, and solids.
- Explain the production of polysulfide by the interfacial polymerization method.
- If students are not familiar with the Merck Index, discuss it briefly.
- If students will be taking melting points and are not familiar with the melting point apparatus, demonstrate how it works.

Procedural Tips and Suggestions

- The trick in making the polysulfide sample almost round is stirring. You might have student use stirring hot plates set at constant speed. Try not to stir too fast.

Plausible Answers to Questions

1. Will this material bounce? Do its elastic properties resemble that of a rubber band?

A: If the material is nearly round, the students might get some bounce out of it. They will also see that the material is somewhat elastic but more rigid than a rubber band.

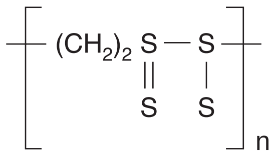
2. Does the polymer have an objectionable feature? Explain.

A: Yes, this particular material has a very strong unpleasant odor.

3. There are two types of Thiokol produced. Identify and describe these two types. Which type did you produce?

A: The two types of polysulfide are Thiokol A and Thiokol FA. Thiokol A has about 85% sulfur content and when cured retains its unpleasant odor. Thiokol FA has about 47% sulfur and when cured does not have an odor. Thiokol A is produced in this lab.

4. What is the repeat unit of your polymer product?

A: 

Thiokol A

5. What can be said about the solubility of this polymer?

A: This answer should correspond to the results your student observe in their solubility testing. In general, Thiokol A is stable to the usual organic solvents, oils, and dilute mineral acids. It is unstable to strong bases and oxidizing agents.

6. Under what conditions could your product be used as a coating or sealant for tanks and pipelines? Explain your reasoning.

A: Thiokol A could be used as coatings or sealants for tanks and pipelines which contain liquids which do not dissolve or react with the Thiokol. If students have checked in Merck, they will also be aware that Thiokol A is not recommended for used in tropical or Arctic climates.

7. If you were marketing manager for this material, how would you sell it to prospective customers? That is, what properties make it valuable and for what uses?

A: You would want your customer to overlook the unpleasant odor and look at the positive application of such material. It has an outstanding resistance to most solvents and could be used to transport solvents in rail cars if rail cars were lined with this material. Such applications as gasoline and oil-loading hoses, sealants and adhesive compositions, binder in solid rocket fuel propellants, gaskets and paint spray hoses are just a few end product applications.

Extensions

1. Additional solvents can be tested for their impact on Thiokol. These might include THF, formic acid, gasoline, oil, or others.
2. Other types of polymers might be tested with the same solvents and their resistance compared with that of Thiokol.

References

Budavari, Susan, Ed. *The Merck Index, Twelfth Edition*; Merck Research Laboratories: Whitehouse Station, 1996; 9475, p. 1593.

Shakhashiri, B.Z. *Chemical Demonstrations, Volume 1*; The University of Wisconsin Press: Madison, 1983; pp. 245-246.