Preparation of Synthetic Rubber

Natural latex is found in the inner bark of many trees, especially those found in Brazil and the Far East. The white sticky sap of plants such as milkweed and dandelions is also a latex. Latex will turn into a rubbery mass within 12 hours after it is exposed to the air. The latex protects the tree or plant by covering any wound to the bark or tree surface with a rubbery material like a bandage.

Natural rubber is a polymer of isoprene (2-methyl-1,3-butadiene, see Figure 1) in the form of polymeric chains (See Figure 2) which are joined in a network structure of folded chains and have a high degree of flexibility (See Figure 3). Upon application of a stress to a rubber material, such as blowing up a balloon or stretching a rubber band, the polymer chain, which is randomly oriented, undergoes bond rotations allowing the chain to be extended or elongated (See Figure 4). The fact that the chains are joined in a network allows for elastomeric recoverability since the cross-linked chains cannot irreversibly slide over one another. The changes in arrangement are not constrained by chain rigidity due to crystallization or high viscosity due to a glassy state.

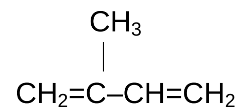


Figure 1. 2-methyl-1,3-butadiene

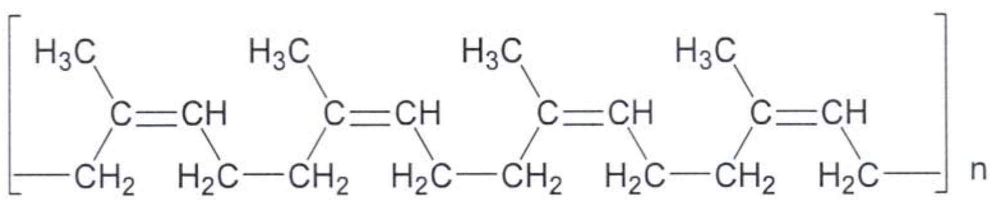


Figure 2. A rubber polymeric chain

Since latex will solidify in air, a stabilizer is added to prevent polymerization if the latex is to be stored or shipped in liquid form. The stabilizer is usually 0.5 to 1% ammonia. When the ammonia is removed by evaporation or by neutralization, the latex will solidify into rubber.

Figure 3. Schematic sketch of a typical elastomeric network

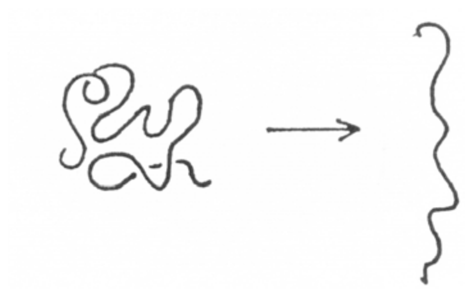
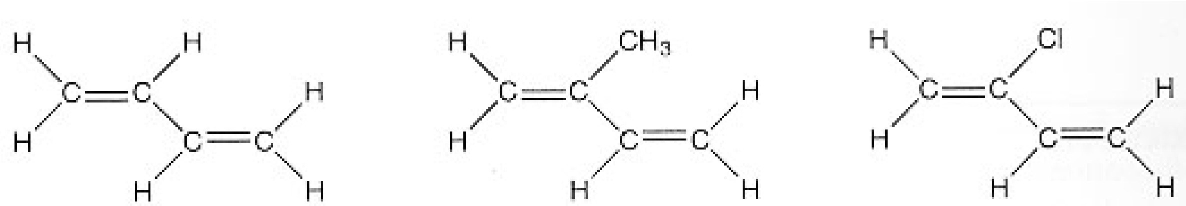


Figure 4. Elastic deformation of a rubber chain

Synthetic rubber is produced from petroleum-based hydrocarbons such as isoprene, butadiene, chloroprene, isobutylene, and styrene. (See Figure 5) Hundreds of synthetic rubber compounds have been developed for many diverse applications.



butadiene isoprene chloroprene

Figure 5. Some monomers used to produce synthetic rubbers

Polysulfide rubbers were among the first commercial synthetic rubbers produced. Two chemists, Joseph C. Patrick and Nathan Mnookin, were trying to invent an inexpensive antifreeze in 1926. In an experiment involving ethylene dichloride and sodium polysulfide, they created a gum whose outstanding characteristic was a terrible odor. Trying to dispose of it, the substance clogged a sink in the laboratory, and none of the solvents used to remove it were successful. The chemists realized that the resistance of the material to any kind of solvent was a useful property. They had invented synthetic rubber, which they christened "Thiokol," from the Greek words for sulfur (*theion*) and glue (*kolla*). The Thiokol Chemical Corporation was subsequently founded on December 5, 1929.

Thiokol rubber is resistant to natural oxidants such as oxygen and ozone and to organic solvents such as oils and gasoline. This makes it useful for engine O-rings, gaskets, and hoses which may come in contact with oils.

Thiokol can be prepared by the reaction of 1,2-dichloroethane and sodium polysulfide.

$$\text{Cl-CH}_2\text{-CH}_2\text{-Cl} + \text{Na}_2\text{S}_x \rightarrow \text{Cl-[-CH}_2\text{-CH}_2\text{-S-S-]}_n\text{-CH}_2\text{-CH}_2\text{-Cl} + 2n\ \text{NaCl}$$

In this experiment, you will prepare a sample of a polysulfide rubber. During the preparation, you will use some natural rubber latex to make a small rubber ball. After the preparations, you can compare the properties of the two types of rubber.

Part 1. The Small-Scale Preparation of Thiokol Rubber

This preparation of Thiokol rubber is a two-step process. The first step is the preparation of sodium polysulfide by the reaction of sulfur, S_8, with a strong base, sodium hydroxide, NaOH.

$$S_8 + 2\ NaOH \rightarrow Na_2S_8$$

At room conditions, sulfur is normally in the form of S_8 rings and chains. The reaction with sodium hydroxide produces a mixture of mostly Na_2S_8 with other chain lengths of sulfur present. That mixture is called sodium polysulfide.

The second step of this preparation is the reaction of sodium polysulfide with ethylene dichloride.

$$Cl\text{-}CH_2\text{-}CH_2\text{-}Cl + Na_2S_x \rightarrow [\text{-}CH_2\text{-}CH_2\text{-}S_8\text{-}]_n + 2n\ NaCl$$

Materials Needed

- Sodium hydroxide solution, 1M
- Sulfur
- 1,2-dichloroethane
- Distilled or deionized water
- Copper wire, approximately 6 inches long (15 cm)
- 2 10-mL vials with Teflon cap liners
- 2 400-mL beakers
- 10 mL graduated cylinder
- Glass pipette (dropper)
- Hot plate
- Chemical resistant gloves

Safety

Wear safety goggles at all times in the laboratory.

Sodium hydroxide solution is corrosive. Although the solution used in this experiment is dilute, if any splashes on the skin, immediately rinse well with room temperature water. If some redness of the skin occurs, seek medical attention.

1,2-dichloroethane is flammable, avoid any fires or sparks. It is also an irritant to the skin and eyes and is toxic by ingestion. Work with this only under a fume hood.

Disposal

Dispose of all liquid wastes in the proper waste bottle provided.

Dispose of the vials in the waste bucket provided.

Procedure

Prepare a water bath by filling a 400-mL beaker with approximately 350-mL of deionized water. Place the beaker on a hot plate and allow the water to come to a boil.

Weigh 0.5 g of sulfur and add it to the vial.

Add 5 mL of 1 M sodium hydroxide to the vial.

Seal the vial tightly. Shake the reaction mixture.
Wrap the copper wire around the vial leaving several inches to serve as a handle.

Place the vial in the boiling water. Heat for about 3 minutes.

Remove the vial from the water. Make sure the cap is tight. Invert the vial several times to mix the contents, then place it back into the boiling water.

Heat the vial for 5 minutes. Remove the vial, invert several times to mix the contents and replace it into the boiling water for an additional 5 minutes. The contents should turn red during this heating.

Remove the vial from the boiling water and allow it to cool for a few minutes. (Leave the beaker of hot water on the hot plate. It will be used again shortly.)

Place the vial in a beaker of cold water for two minutes. Move the apparatus to a fume hood.

Working under the hood, remove the vial from the cold water. Remove the copper wire handle and the cap and decant the red solution only into a clean vial, leaving any solid behind in the first vial. Reseal the first vial and place it in the waste bucket under the waste hood.

Using a glass dropper, add 1 mL of 1,2-dichloroethane to the red sodium polysulfide solution in the second vial. Seal it tightly. Reattach the wire handle.

Vigorously shake the reaction mixture. Then place it back into the boiling water bath. NOTE: If any bubbles begin to flow out of the vial cap during the reaction, remove the vial from the water, allow it to cool, and tighten the cap.

After about 3 minutes of heating, remove the vial from the boiling water. Make sure the cap is tight. Invert the vial several times to mix the contents, then place it back into the boiling water.

Continue to heat the vial for 20 minutes. Remove the vial every 5 minutes, invert several times to mix the contents and replace it into the boiling water. During this time a rubbery lump of yellow Thiokol will begin to form.

NOTE: During this heating step do Part 2 of this experiment. Always have one member of your group watch the vial in the boiling water.

After 20 minutes, remove the vial from the hot water and allow it to cool for a few minutes.

Place the vial in a beaker of cold water for two minutes.

Wear chemical-resistant gloves for the remaining steps.

Working under the hood, remove the vial from the cold water. Remove the copper wire handle and the cap. CAUTION: The reaction vial may be pressurized and some solution may squirt out.

Pour any liquid in the vial into a waste bottle in the waste hood. Keep the Thoikol product in the vial.

Rinse the Thiokol rubber at least twice with 4 to 5 mL of water. Dispose of the wash water in a waste bottle. Then, remove the rubber from the vial and rinse it very well with tap water.

Place the washed rubber on a paper towel, squeeze out any excess liquid, place under a hood to dry.

Once the rubber is dry, weight it and record the mass.

Part 2. Preparation of a Rubber Ball from Rubber Latex

Materials needed

two paper cups (5 ounce)
stirring rod (popsicle stick or equivalent)
small bucket or large beaker (1000 mL or larger)
15 mL rubber latex
15 mL vinegar
15 mL water

Safety Precautions

Wear safety goggles at all times in the laboratory.

The materials used in this experiment are considered to be non hazardous, however, some individuals may be allergic to rubber latex.

The odor of vinegar may be irritating to the nasal passages. Some individuals may want to work with it under a hood.

Disposal

Solid materials can be safely disposed of in the trash.

The latex solutions should be disposed of in the waste bottles provided. The solution will clog drains, do NOT pour latex solutions down the drain.

Clean-up:

Latex spilled on non-porous surfaces can be allowed to dry and then peeled off. If spilled on clothing, latex can be removed using a cleaning fluid such as Afta. A dry cleaner can often remove most of a latex stain.

Procedure:

Measure 15 mL of latex into a paper cup.

Pour a few drops of latex onto the palm of your hand. Spread it out using your finger. What does it smell like? Describe its properties. (Answer on data page.)

What happened to the stabilizer as you spread the latex around?

Obtain 15 mL of vinegar in a paper cup.

Dip the stirring rod into the vinegar, then into the latex, and then into the vinegar again. Remove the solidified latex. Stretch it. Describe the polymer.

What happened to the stabilizer when vinegar is added to the latex?

Add 15 mL of water to the latex and stir the mixture. Describe what happens.

Pour the 15 mL of vinegar into the cup of latex and stir the mixture. Describe what happens.

Remove the mass from the cup and stirring rod with your fingers. Carefully squeeze the mass while washing it under water in a small bucket. Squeeze and turn the mass repeatedly, forming the mass into a ball.

Dry it with a paper towel. Drop the mass on the floor and describe what happens.

References

Slater, Alan, *Making Thiokol Rubber*, Stratford Central Secondary School, Stratford, Ontario, Canada, 1995

Russo, Tom and Mark Meszaros, *Thiokol Rubber*, **Vail Organic II**, Flinn scientific, Inc., Batavia, IL, 2001

Katz, David A., *Rubber*, **Chemistry in the Toy Store**, 1985

Preparation of Synthetic Rubber

Data and Results

Names: ____

Date ________________ Course ____________________________ Section __________

Part 1. The Small-Scale Preparation of Thiokol Rubber

Mass of sulfur used	______________	g
Volume of sodium hydroxide used	______________	mL
Volume of 1,2-dichloroethane used	______________	mL
Mass of Thiokol rubber obtained	______________	g

Describe the properties of the Thiokol rubber (i.e., Color? Does it bounce? Does it stretch?)

Part 2. Preparation of a Rubber Ball from Rubber Latex

What does the rubber latex smell like? Describe its properties.

What happened to the stabilizer as you spread the latex around?

After dipping the stirring rod into the vinegar, then into the latex, Describe the polymer.

What happened to the stabilizer when vinegar was added to the latex?

Describe what happened when you added 15 mL of water to the latex?

Describe what happened when you added 15 mL of vinegar to the latex?

Describe the properties of the rubber (i.e., Color? Does it bounce? Does it stretch?)