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#21 Films, Fibers, and Solubility

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I. INTRODUCTION

Description

These activities will introduce how the differences in solubility of materials are used in the manufacture of fibers and films. The various processes used to make films and fibers will be introduced to the student and completed on a small scale.

Student Audience

This investigation is appropriate for high school chemistry, chemical or material technology students, and university chemistry students.

Goals for the Experiment

The student will:

- prepare fibers using wet spinning and melt spinning techniques,
- prepare cast and blown film samples,
- relate solubility to dry and wet spin methods of fibers,
- relate solubility to casting of films,
- describe variations in polarity and the resulting solubility, and
- compare and contrast materials produced.

Recommended Placement in the Curriculum

This investigation can be used in discussions of

- solubility and polarity,
- physical and chemical properties,
- polymers and forms of plastics, and
- materials and methods of production.

II. STUDENT HANDOUT

Films, Fibers, and Solubility

Scenario

After working as a chemical technician for a number of years, you have decided to take some time off to try out another career as a fashion designer. You know that to be successful in this career, you will have to be both creative and innovative. In fact, you are concerned that your unique design ideas are not enough to get you noticed in this very competitive industry. With this in mind, you have decided that one approach that may help you to make your mark in the fashion world would be to make new and different uses of films and fibers in your designs. You decide to first make some sample films and fibers and investigate their properties before you go further with the design process.

Industrial Application

Scientists have investigated methods for producing manufactured fibers as early as 1664 when Robert Hooke suggested the possibility of making artificial fiber. In the history of polymers, the trend has gone from the production of natural materials to chemically modified natural products to man-made or synthetic materials. The history is not different for fibers. The first patent for "artificial silk" was granted in England in 1855 for a cellulose based material. By 1889 French chemist Count Hilaire de Chardonnet started the first commercial production of modified natural fibers and established a company in France in 1891. Soon after in 1893, the same process used to make modified natural fibers was used to create a similar cellulose acetate for making motion picture film and toiletry articles. Modified natural fiber production came to the United States in 1910 with the commercial production of cellulose nitrate. Because cellulose nitrate is flammable, it was soon replaced by cellulose acetate and rayon (regenerated cellulose). Cellulose acetate was introduced in 1924 for commercial textile production.

The advantage of chemical modification of natural fibers could be seen in the variety of textures and characteristics obtained. Cellulose acetate could be made to be like linen or fine silk, from a dull surface to shiny. The cellulose was treated with acetic acid and acetic anhydride to produce the cellulose acetate. By varying the number of hydroxyl groups acetylated, the chemist varied the resulting characteristics. For example, when increasing the ratio of acetate groups to cellulose, cellulose acetate becomes cellulose triacetate. The resulting materials had to be processed to create the fibers or film desired. Cellulose acetate dissolved in acetone can be cast to form a film or extruded through a spinneret to form fibers. The viscous material can be dry spun by extruding it from the spinneret, then evaporating the solvent in warm, dry air. The alternative is wet spinning in which the viscose is again extruded into a solution (in this case water) where the fiber is regenerated, then drawn. The solution is chosen based on solubility and insolubility of the materials. In this case, cellulose acetate, which is a moderately polar material, dissolves in the moderately polar acetone but not in the more polar water. Even though both water and acetone are polar, polymers will only dissolve in very similarly structured solvents. The cellulose acetate dissolves in acetone since both have carbonyl groups but regenerates when in contact with the water which does not have a similar structure.

Solubility is often used to identify specific polymers within a family. These experiments will use the highly specific solubility of two polymers and the various methods of fiber and film production to create a wide range of samples.

Safety, Handling, and Disposal

While the chemicals and procedures in this experiment are not extremely hazardous proper laboratory precautions are necessary.

- When using chemicals, read the MSDS and label then follow any warnings or suggestions for use.
- Since acetone is flammable, work in a fume hood away from open flames, sparks, etc. to limit fire hazards.
- When using the hot plate, take precautions to prevent serious burns by using tongs and heat resistant gloves at all times.
- Dispose of any used reagents according to local ordinances.

Materials

- 1/2 teaspoon powdered cellulose acetate
- acetone
- 20 ml 4% aqueous solution of polyvinyl alcohol (PVAl)
- 2 50-ml glass beakers
- foil or foil pans
- food color
- dish pan
- tweezers
- glass rod or stir stick
- hot plate
- small amount of various plastic pellets
- Bunsen burner
- tongs
- heat resistant gloves
- 12-inch polyethylene tube with 1/2-inch inner diameter
- optional: microscope, IR spectrophotometer

Procedure

This procedure will produce fibers and films by a variety of methods. The first part will use the different solubilities of cellulose acetate and polyvinyl alcohol (PVAl). Cellulose acetate is a polar material and dissolves in acetone but not water. This difference will be used to regenerate fibers of cellulose acetate. The opposite is true of PVAl. Although PVAl is polar it is not polar due to carbonyl groups but hydroxyl groups. PVAl will dissolve in hot water due to similar hydroxyl structures, but in acetone it will regenerate. These examples show the principle on which wet spinning fiber production is based. Dry spinning uses a dissolved polymer but the solvent is allowed to evaporate to form the fiber or to cast films.

Melt spinning is another method of fiber production in which the melted plastic is extruded and then drawn and cooled. Production of melt spun fibers began in the 1930’s with the production of nylon. Film can be similarly extruded through a die to form a sheet of film or a tube that is then blown into a film. The procedure will produce both melt spin type fibers and blown film.

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1. Place a level 1/2 teaspoon of powdered cellulose acetate in a glass beaker. Dissolve in acetone by slowly adding small amounts of acetone, then stirring until completely incorporated. Continue adding acetone until material is dissolved and the viscose (the solution of cellulose acetate) is the consistency of oil.
2. Label one foil pan CA. Pour half the viscose into the foil pan and set in the fume hood to evaporate.
3. Pour the other half in a very thin stream into a dish pan of water and retrieve the precipitated cellulose acetate fiber. A paper cup with pin holes punched in the bottom can be used to ensure a very thin stream of viscose. Examine (under a microscope, if available) and describe the resulting fiber.
4. Label the second foil pan PVAl. Pour approximately half of the PVAl solution into the second foil pan. Set in the fume hood to evaporate.
5. Add a few drops of food color to the remaining PVAl. Add an equal amount of acetone carefully over the PVAl by decanting the acetone down the inside wall of the beaker. PVAl will precipitate at the interface of the two solutions.
6. Pull the fibrous rope of PVAl from the beaker using tweezers. Lay it out on toweling or on foil sheets to dry as the acetone evaporates. Remember, do not wash the PVAl because it is water soluble. Examine the dry fiber and compare and contrast with the precipitated cellulose acetate for tenacity (its ability to hold together), color, fineness, etc.
7. Once the solvents in the foil pans have completely evaporated, peel the cast films off the foil pans. Examine and compare the two samples for color, tensile strength (resistance to a force tending to tear it apart), and transparency. If an IR spectrophotometer is available, run and obtain representative spectra to confirm the identity of cellulose acetate and PVAl.

Caution: The remaining steps require that extra caution is taken with hot equipment and materials. Wear heat resistant gloves and use tongs.

8. To create the blown film, heat the end of the polyethylene tubing over a low flame of a Bunsen burner until it becomes clear. (The flame should be blue in color and approximately 1 inch high.) Be careful with the molten plastic as it can cause severe burns. Use the tongs to clamp shut the end of the tubing completely while the material is still transparent. As the material cools, it will become translucent again.
9. Heat the tube again a few inches above the clamped end. Turn the tubing constantly while heating until the material is transparent along a 2–3 inch segment. Be careful to not let the sides of the tube collapse. While the walls are clear, remove from the flame and gently blow into the open end. To help in blowing the bubble of film slightly stretch the tube at the mouth and clamped end while blowing.

Caution: Don’t breathe in!

10. Cut the bubble open to get the sheet of film. Compare and contrast with the cast films.
11. To make melt fibers, heat a foil pan on the hot plate on a medium to high heat. Place 4–5 pellets of one material on the foil to melt. When the material turns transparent, remove foil from the hot plate. Grab the molten plastic with tweezers or tongs and pull a fiber. Continue drawing the fiber out until it is oriented and very tenacious.

12. Repeat with a variety of different types of plastic pellets to obtain a variety of different fiber samples.

Questions

1. Draw the repeat units of the polymers used in this investigation.
2. What are some commercial uses of cellulose acetate and PVA1?
3. Describe how differences in solubility, as in fiber production, could be used in other areas.
4. Describe the differences observed in making the various melt fibers.
5. Create a fiber chart and a film chart that compare and contrast at least five characteristics of the materials made.
6. Research the development of fibers from the 1930's to the present and their chemistry.

References

Carley, J.F., ed. *Whittington's Dictionary of Plastics;* Technomic Publishing Company: Lancaster, PA, 1993.

Gruenwald, G. *Plastics: How Structure Determines Properties;* Hanser Publishing Company: New York, 1993.

Manufactured Fiber Fact Book.; American Fiber Manufacturers Association, Inc.: Washington, D.C.

Shah, V. *Handbook of Plastics Testing Technology;* John Wiley & Sons: New York, 1984.

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III. INSTRUCTOR'S NOTES

Films, Fibers, and Solubility

Purpose

These activities will introduce methods of producing fibers and films based on solubility and other properties.

Time Required

To complete all portions of this activity will take several hours depending on the extent of testing and comparisons made between the fibers and film. If comparing and contrasting is more qualitative in nature an extended lab period of approximately 2–3 hours will be needed.

Suggested Group Size

This can be done with any size class. To facilitate a large class size and limited equipment, several stations for each component can be set up so students can rotate through the work areas.

Materials

- 1/2 teaspoon powdered cellulose acetate (Cellulose acetate is available through most chemical supply and science education catalogs. Cellulose acetate can be easily ground in a small household coffee grinder.)
- acetone (Amounts will vary depending on the amount used to dissolve the cellulose acetate. No more that 40–50 mL per student will be needed. Reagent grade acetone is not needed; the acetone available in paint and hardware stores is more than suitable.)
- 20 mL 4% solution of polyvinyl alcohol (PVAl; PVAl solution can be made by dissolving 4 grams of PVAl in 100 mL of distilled water. Heat, stirring constantly, until dissolved. To decrease evaporation prepare solution in an Erlenmeyer flask with an inverted beaker over the opening. Use a stirring hot plate. To enhance the solubility portion of the lab activity, you may want students to prepare their own PVAl solution. This is quite time consuming but will also show the students the difference in solubility and the need to increase the temperature for the PVAl to dissolve.)
- 2 50-mL glass beakers
- foil or foil pans (These are used for casting films. Pans can be made out of sheet aluminum foil by tearing a sheet, folding to make a 4 layer square, then form over the bottom of a 400 mL beaker creating a smooth pan.)
- tweezers
- glass rod or stir stick
- hot plate
- small amount of various plastic pellets (Include olefins and nylons which are common commercial fibers. Melt index of materials which are easiest to use should be over 6 g/10 min. The pellets can be obtained from plastic processors or can be cut from polymer products. Try them before student use.)
- Bunsen burner
- tongs
- heat resistant gloves

- polyethylene tubing with 1/2 inch ID (PE or PP tubing works well. This can be purchased at hardware stores in the plumbing supplies. PE and PP tubing is white. Do NOT purchase the clear PVC tubing for this activity. Each student will need about 1 foot, therefore precut to avoid waste. Small inner diameters will work but not as well as 1/2 inch or larger.)

Safety, Handling, and Disposal

While the chemicals and procedures in this experiment are not extremely hazardous proper laboratory precautions are necessary.

- When using chemicals read the MSDS and label then follow any warnings or suggestions for use.
- Since acetone is flammable, work in a fume hood away from open flames, sparks, etc. to limit fire hazards.
- When using the hot plate take precautions to prevent serious burns by using tongs and heat resistant gloves at all times.
- Dispose of any used reagents according to local ordinances.

Points to Cover in Pre-Lab

- Discuss crystallinity and morphology.
- Discuss the relationship between polymer structure and properties.
- Discussion of solubility and polarity tends to be better prior to lab activity.
- Safety concerns include using acetone which evaporates rapidly and is highly flammable. This reagent should be used in the fume hood only. Severe burns could also result with the equipment used and molten plastic. Tongs and heat resistant gloves should be used.

Procedural Tips and Suggestions

- Adding cellulose acetate to acetone will result in lumps and it is very difficult to dissolve the material. All lumps should be dissolved before continuing the activity.
- Foil can be sprayed with silicone release spray or vegetable spray coating such as Wesson No Stick or PAM to help students release the film more readily.
- To increase evaporation, the pan can be put in a 50 °C oven.
- The food color is only added to make the differences in the interfaces more pronounced. It is not actually needed.
- Steps #7 to the end of the lab require that extra caution is taken with hot equipment and materials. Wear heat resistant gloves and use tongs.
- This is a skill that may take some practice to get a bubble approximately 6 inches in diameter. Since only a small amount of film is needed for observation, students should be able to get a sample from no more than 2 tubes. If one student is very successful, their film can be shared with others also.

Sample Results

Fibers:

- Cellulose acetate fiber will be very fine with a great deal of tenacity. The fibers will be short in length and white in color.
- PVAl fiber will have a fine texture and be very weak. The fibers will be longer than the cellulose acetate and also white in color.
- The appearance and properties of melt plastic fibers will vary depending on the polymer used.

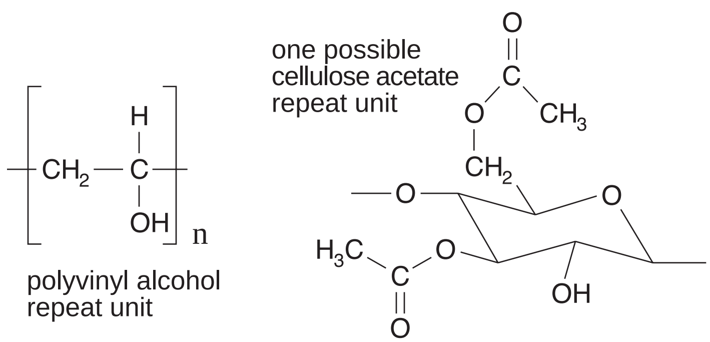
Films:

- Cellulose acetate will be transparent and clear if the material was completely dissolved. If not, the film will be white or have areas of white. The film will be very brittle.
- PVAl will be a transparent film that is brittle.
- Blown film will be thicker than the cast films and very flexible. Transparency will depend on the thickness of the blown sample.
- Uniformity in thickness will vary dramatically.

Plausible Answers to Questions

1. Draw the repeat units of the polymers used in this investigation.

A:



The number of acetate groups will vary. For commercial CA, initial acetylation is often 100% and then partial hydrolysis is used to bring the acetyl value to 52–56% or whatever value gives the desired properties.

2. What are some commercial uses of cellulose acetate and PVA1?

A: PVAl is used for water soluble laundry bags, pesticide and herbicide containers. PVAl solution is also used as a glue (Glue Gel®). Cellulose acetate replaced cellulose nitrate, which is very flammable, for photographic and movie film. Now newer polyesters are replacing celluloid film. Cellulose acetate is also used as fiber for clothes such as acetate graduation gowns.

3. Describe how differences in solubility, as in fiber production, could be used in other areas.

A: Polymer identification can be based on solubility and also comparison of polarity of materials. Solubility is also used extensively in the adhesive industry. Many adhesives are polymer specific because of solubility such as PVC dope is only for PVC and ABS dope is used only for ABS.

4. Describe the differences observed in making the various melt fibers.

A: Students will notice a variety of differences in making fiber. For example, some plastics are very easy to pull and draw and others are very difficult to work with. Also, olefins have broad Tg and nylons have very narrow Tg. Students have more working time with the broader the Tg.

5. Create a fiber chart and a film chart that compare and contrast at least five characteristics of the materials made.

A: Some characteristics that can be suggested are as follows:

For fibers:

- lineal density (mg/m of fiber), Denier (g/9000 m of fiber), TEX (g/1 km),
- relative tensile or tenacity, and
- texture, color, water absorbency, stretch, elasticity.

For films:

- thickness, color, uniformity, transparency, shear, tensile, IR spectra.

6. Research the development of fibers from the 1930’s to the present and their chemistry.

A: Various chemistry, fiber, art, and textile texts are available for students to research this topic. Chemistry has allowed for the development of a variety of synthetic fibers that are engineered with a variety of physical and chemical characteristics. Studies of the hydrophobic and hygroscopic nature of fibers can be done based on the molecular structure and the chemistry of fiber and the ability to be pigmented or dyed.

Extensions and Variations

1. Create larger quantities of film material and complete various tests such as impact, drop dart, sheer, or tear. Industrial standards are available for film testing through ASTM and ISO. Have students replicate testing equipment from the standards for further testing.

References

Carley, J.F., ed. *Whittington’s Dictionary of Plastics;* Technomic Publishing Company: Lancaster, PA, 1993.

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