A Polymer in Everyday Life: The Isolation of Poly(vinyl alcohol) from Aqueous PVA Glues

An Undergraduate Chemistry Experiment

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Synthetic polymers are found everywhere in our daily lives. However, laboratory experiments involving polymers in undergraduate chemistry curricula are not in proportion to the prevalence of polymer materials in modern society *(1)*. Reported here is a simple undergraduate-level polymer-based experiment that involves poly(vinyl alcohol), PVA, a synthetic polymer contained in some glues. Instead of the gelation of PVA, which has been widely discussed in the literature *(2–9)*, we propose a different aspect of the study on PVA in this experiment. The goal of this laboratory is to prepare PVA via an ester hydrolysis reaction using the polymer contained in an aqueous PVA glue as the precursor. Aqueous PVA glues contain PVA with residual acetate groups, which serve to increase the solubility of the PVA in water. With the procedures provided, the preparation of PVA can be accomplished by the students in the lab, and the problems inherent in the synthesis of the polymer are avoided *(10, 11)*. Since the reaction involves the conversion of acetate groups to hydroxyl groups, IR spectroscopy is employed as a tool to monitor the reaction. The IR spectra of PVA polymers are recorded and the corresponding functional groups are identified. This experiment does not use special materials or organic solvents. If a student-operable IR spectrometer is available, the experiment is suitable to be included in the undergraduate chemistry laboratory.

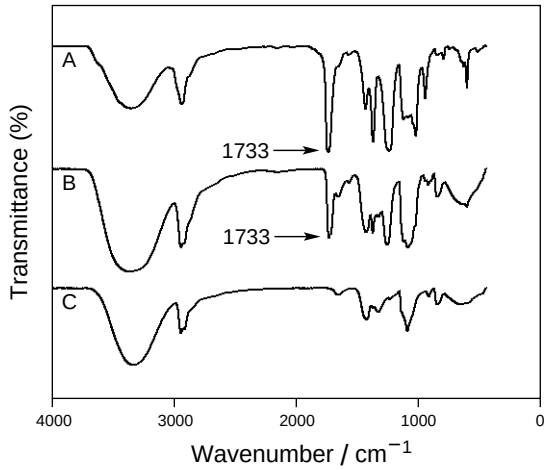
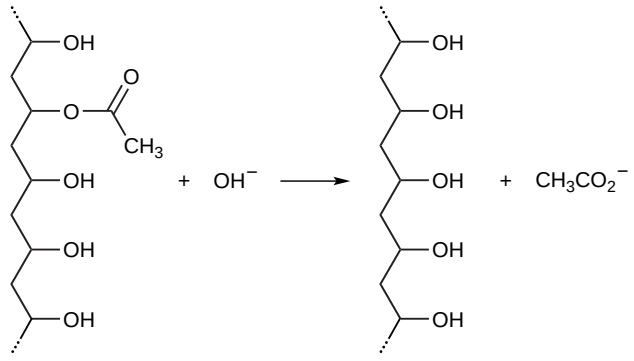


Figure 1. The IR spectra of (A) white glue, (B) the aqueous PVA glue, and (C) the PVA resulted from the hydrolysis of the aqueous glue.



Scheme I. Base hydrolysis of the residual acetate groups in PVA-based glue.

Although most of the aqueous PVA glues[1] contain the polymers of PVA with residual acetate groups, white glue is a poly(vinyl acetate)-based emulsion. The carbonyl stretch at 1733 cm^{-1} appears in the IR spectra of the polymers in both glues, but the intensity is markedly decreased for the polymer in the aqueous PVA glue (Figure 1). Unlike the acetate groups in poly(vinyl acetate),[2] the residual acetate groups of the PVA are readily hydrolyzed via the reaction with sodium hydroxide in aqueous solution (Scheme I). The disappearance of the carbonyl stretch at 1733 cm^{-1} in the IR spectrum of the product provides the evidence for the occurrence of the hydrolysis (Figure 1). The solubility in water of the resulting PVA is thus decreased owing to the strong interactions between the hydroxyl groups in the molecules. As the temperature elevated, the solubility in water of the polymer increases.

In our curriculum, this experiment is performed near the end of the second-semester general chemistry laboratory course. The topics on covalent bonding, molecular structures, and introductory organic chemistry including ester chemistry have been covered in general chemistry lecture. Prelab discussion begins with a comparison of the molecular structures of three related polymers: poly(vinyl acetate), PVA, and the PVA with residual acetate groups. A short review of base hydrolysis of esters is given for the conversion of acetate groups to hydroxyl groups and students learn to identify them. Since IR spectroscopy is utilized for monitoring the hydrolysis of acetate groups, the vibrational modes of the hydroxyl bond and the carbonyl bond are discussed and the corresponding IR absorptions are noted. Example IR spectra of alcohol–acetate derivative pairs are shown to the students

to imply the relationship between the functional groups and the corresponding spectral absorptions.

Experimental Procedure

This is a three-hour laboratory exercise. Before the lab, the students were informed to bring their own aqueous PVA glues. A solution mixture containing about 30 g of the aqueous PVA glue in 100 mL of water and 10 mL of 2 M NaOH is heated to a gentle boiling on a hot plate with continuous stirring for about 25 minutes. After the reaction, the product is precipitated in a saturated NaCl solution. The resulting polymer is removed from the solution with a glass stirring rod and washed with large quantity of water. Subsequently, part of the PVA product is dissolved in water to form a viscous solution.

Students working in pairs perform the IR spectroscopy with Spectrum One FTIR spectrometer from Perkin-Elmer Instruments. They prepare thin films of white glue, the aqueous PVA glue, and the PVA resulting from the hydrolysis. The IR spectrum of each thin polymer film is recorded. The film[3] is prepared by spreading few drops of the glue or the PVA solution on an aluminum foil. It is then dried by hot air from a hair dryer and carefully peeled off the aluminum foil with a pair of sharp-ended tweezers. The transparent polymer film is then taped on a film card[4] for the spectrum recording.

The solubility in water of the polymer is investigated qualitatively. Using a pair of sharp-ended tweezers, a piece of polymer film is placed evenly on the surface of water in a Petri dish. Whether or not the polymer film dissolves in water is recorded.

Hazards

Sodium hydroxide is caustic and produces a tremendous quantity of heat when dissolved in water. It should be handled with care. Students should wear goggles and gloves to prevent contact with the splashed reaction mixtures. Care must be taken while the hot plate is in use. Other materials such as the aqueous PVA glues and NaCl are safe and nontoxic.

Students Results

Data have been collected for over fifty student samples. The yields of the resulting PVA in its dry form ranged from 1.8 g to 2.8 g on the basis of 30 g of various brands of aqueous PVA glues. The results of the solubility test were consistent for all the students. While the film of the polymer contained in the aqueous glues quickly collapsed and redissolved in water at room temperature, the film of the resulting PVA expanded and stayed as a film. However, as the temperature of water was elevated, the film dissolved. Although the thickness of the films varied, the IR spectra of the three related polymers showing significant differences in the intensity of the peak at 1733 cm^{-1} were successfully obtained by the students (Figure 1).[5]

For the past two years, this experiment has been successfully performed in our chemistry laboratory courses by approximately 100 students. Although this experiment was originally designed for general chemistry laboratory, it is also appropriate for organic chemistry or polymer chemistry laboratory. It should also be advantageous to supplement this experiment with related activities, such as the molecular weight determination of the PVA polymers, the viscosity of PVA solutions, and the swelling of PVA films.

Summary

This activity provides students the opportunity to become familiar with the molecular structures of the PVA related polymers and the chemistry involved in the PVA preparations. The students observe one way in which PVA can be prepared as they conduct a common organic chemical reaction, base hydrolysis of esters. They also observe how a physical property, the solubility in water, of the PVA polymer is affected by functional group modification. In this experiment, the IR spectral analysis is focused on the absorption corresponding to the stretching of the carbonyl bond. The students understand that the hydrolysis reaction converts the acetate groups in the polymer molecules to hydroxyl groups. Accordingly, the disappearance of the carbonyl group absorption in the IR spectra of the PVA product indicates a complete hydrolysis of the acetate groups.

At this point students may not understand IR spectroscopy in detail, but learn how to verify the presence of a carbonyl group in a molecule using IR spectroscopy. Most importantly, the students get a chance to operate the IR spectrometer and experience how the IR spectral information is used in a chemistry lab. In addition, most of the students enjoyed the real-world nature of this experiment. One student stated, "It's amazing that the glues have so much to do with chemistry." Another student even stated, "This experiment really made me feel like I had learned something." The success of this simple experiment involving polymers in everyday lives would encourage the wider integration of polymer chemistry into the undergraduate chemistry laboratory.

Acknowledgment

The authors would like to express gratitude to the reviewers for constructive suggestions regarding the manuscript.

[W]Supplemental Material

Instructions for the students and notes for instructor are available in this issue of *JCE Online*.

Notes

1. In Taiwan, as in the United States, most of the aqueous PVA glues contain PVA with residual acetate groups. Five different aqueous PVA glues purchased from stores in the United States (clear Elmer's washable school glue, MonoAqua liquid glue, Staples liquid glue, Dab'N Stic paper glue, and Pentel brush glue) were tested in this experiment and gave similar results.

2. In this experiment, white glue is not used for the preparation of PVA because poly(vinyl acetate) is much more difficult to be hydrolyzed in aqueous solution. We have tried the hydrolysis in ethanol using strong base as the catalyst. A barely complete hydrolyzed PVA was obtained by refluxing the mixtures of 10 g of white glue and 2.6 g KOH in 100 mL of ethanol for 2.5 hours. This

procedure is relatively time consuming. The use of a large quantity of strong base and the flammable solvent are also major disadvantages of this procedure.

3. Since the IR spectral analysis in this lab is a qualitative one, the quantity of the glue solution used for the film preparation is not exact. However, the film must be sufficiently transparent for good peak identifications.

4. The film card used for IR spectral analysis is originally purchased from PIKE Technologies, Inc. (part number: 162-5400). Alternatively, our students use cardboard to duplicate the film cards for their own samples.

5. The IR spectrum of commercially available PVA (99+%, hydrolyzed) is recorded and included in the Instructor's Notes in the Supplemental Material.[W]

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