Improvement of viscosity method for viscosity-average molecular weight of PVA

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Abstract: The measurement of viscosity-average molecular weight by viscosity method is a basic experimental operation in polymer physics. However, the experiment for determining the viscosity-average molecular weight of polyvinyl alcohol by viscosity method has several urgent problems, including long instrument drying time, fragile Ubbelohde viscometer and easy foaming during measurement. Combined with the author's years of experience in guiding student experiments, this paper puts forward simple and feasible solutions to several problems that have troubled teachers and students in the experiment.

Keywords: experimental teaching, viscosity-average molecular weight, Ubbelohde viscometer, polymer physics

1. Introduction

The viscosity method is the most used technique for determining polymer molecular weight. Featuring simple equipment, convenient operation, short processing cycle and high experimental accuracy, it serves as a compulsory basic experimental item for junior college and undergraduate students majoring in chemistry, chemical engineering, papermaking, polymer materials and other related disciplines in universities. Once the solvent and temperature Tare fixed, the intrinsic viscosity of a given polymer depends solely on its molecular weight. Under such circumstances, the viscosity-average molecular weight can be obtained only by measuring the intrinsic viscosity of the polymer via the viscosity method [1]. To guarantee the accuracy of measurement results, the "extrapolation method" (also known as the dilution method) is adopted in this experiment to measure the intrinsic viscosity of the test solution. Afterwards, the value of intrinsic viscosity is substituted into the Mark-Hou wink nonlinear equation to calculate the viscosity-average molecular weight of the test polymer [1].

In traditional polymer experiment textbooks, to determine the viscosity-average molecular weight of polystyrene, toluene is used as the solvent in this experiment. Compared with water, organic solvents have drawbacks such as high cost, troublesome instrument cleaning and environmental pollution, so they tend to be replaced by aqueous polyvinyl alcohol solution at present [1-2]. In addition, the experiment of measuring viscosity-average molecular weight of polyvinyl alcohol by viscosity method still has problems including long drying time of residual water in instruments, bubble formation which affects the measurement of flow time, and the common fragility of Ubbelohde viscometers. Combined with the author's years of teaching experience in polymer physics experiments, this paper puts forward some improvement suggestions targeting the above problems.

2. A minor improvement for protecting Ubbelohde viscometer

The schematic diagram of the Ubbelohde viscometer is shown in Figure 1. Tubes A, B and C are connected and fixed at the upper part by two thin glass tubes. When students suck the test liquid into bulb G directly through tube A with a rubber pipette bulb or block the opening of tube A by hand after sucking the solution into bulb G, improper operations can easily break the thin glass connectors among tubes A, B and C, and even cause fracture of tube A, resulting in heavy experimental loss. If a long rubber hose is used to

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connect tube A with the rubber pipette bulb, direct contact between the rubber pipette bulb and tube A can be avoided. Practices by teachers and students in our university verify that damage to Ubbelohde viscometers hardly occurs after adopting the rubber hose connection.

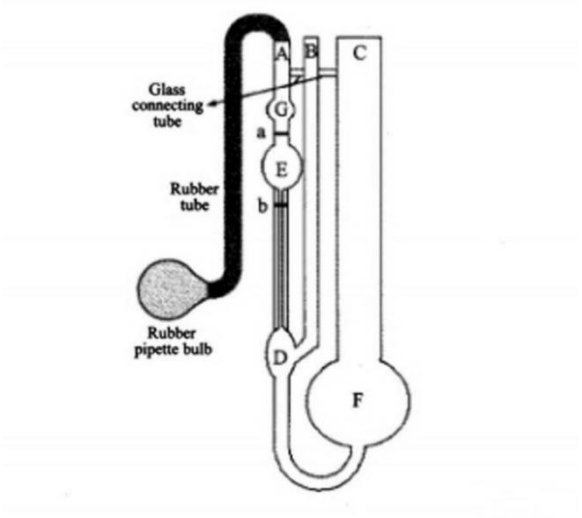

Figure 1 Schematic of Ubbelohde viscometer

3. Solution to long instrument drying time

The traditional experimental procedure for viscosity-average molecular weight measurement by viscosity method is generally as follows: measure the flow time of solvent → dry the instrument → measure the flow time of solution → clean the instrument → dry the instrument for the next group of students. The advantage of this procedure is that no instrument cleaning is required during the experiment. When organic solvents are adopted, instrument cleaning is relatively troublesome, so this step is arranged at the end of the experiment. However, instrument drying in the experiment consumes plenty of time. When water-soluble polyvinyl alcohol is used in the experiment, water serves as the solvent and is harder to dry than volatile organic solvents. If this testing sequence is still adopted, much more time will be spent on instrument drying.

To save experimental time, we revised the measurement sequence as follows: measure the flow time of aqueous polyvinyl alcohol solution → clean the instrument → measure the flow time of solvent (water) → dry the instrument for the next group of students. Compared with the original sequence, the revised procedure eliminates the intermediate instrument drying step and thus saves experimental time. After finishing the measurement of flow time of aqueous polyvinyl alcohol solution, the Ubbelohde viscometer only needs to be rinsed with tap water and then washed with distilled water. Instrument cleaning is much simpler than that for oil-soluble polymers, so the flow time of the solution can be measured first.

4. Bubble elimination of PVA solution without defoamer

Bubble formation has long troubled teachers and students in the viscosity measurement experiment for viscosity-average molecular weight using aqueous polyvinyl alcohol solution. Bubbles can be sucked into bulb G by the rubber pipette bulb, seriously interfering with the judgment of liquid level passing through graduations and b. Some teachers add n-butanol to water for defoaming [1-2] or even replace the experimental system with aqueous polyethylene glycol solution or dilute hydrochloric acid solution of chitosan [3-4]. However, the addition of n-butanol for defoaming inevitably affects experimental results. Moreover, the constants K and α in the Mark-Hou wink equation are not corrected during data processing; instead, the K and α values for aqueous PVA solution are still adopted for calculation. This misleads students into believing that a polymer has identical K and α in different solvents. Defoamer is still required for aqueous polyethylene glycol solution [3], while purification of chitosan and preparation of its solution are relatively cumbersome [3-4].

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After repeated experimental exploration, we have developed a method to eliminate the effect of bubbles in aqueous polyvinyl alcohol solution without adding defoamers. As shown in Figure 2, simply seal tube B of the Ubbelohde viscometer and tilt it to one side, then blow air into tube A with a rubber pipette bulb. All bubbles on the liquid surface of the left branch tube can be blown to the liquid surface of the right branch tube, where bubbles will not interfere with the measurement of flow time. Thus, the adverse effect of bubbles on the experiment is completely removed. This bubble-eliminating method is applicable to all viscosity-average molecular weight measurement experiments using Ubbelohde viscometers.

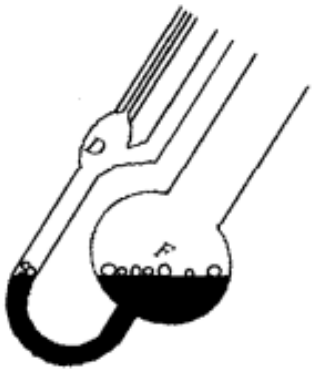

Figure 2 Schematic Diagram of Bubble Elimination

5. Other practical problems

The water bath temperature was controlled at 25 ℃ in the experiment. The polyvinyl alcohol adopted in our experiment was produced by Sinopharm Chemical Reagent Co., Ltd., with the grade 175050. The constants in the Mark-Hou wink equation were in mL/g, and the obtained molecular weight results were very close to those in the product specification. 2. The capillary of the Ubbelohde viscometer with a specification of 0.5–0.6 mm was selected, which can generally ensure that the flow time of water is longer than 100 seconds. 3. The initial concentration of polyvinyl alcohol was 0.005 g/ml. At this initial concentration, the flow time of the transferred 10 mL solution was about 3 minutes. After sequentially adding 5, 5, 10 and 10 mL of solvent, the final measured t5 was approximately 110–140 s, indicating that the initial concentration of 0.005 g/mL was appropriate.

6. Conclusion

This paper proposes improved methods targeting the common problems in the experiment for determining the viscosity-average molecular weight of polyvinyl alcohol, including fragile Ubbelohde viscometers, lengthy drying time and easy forming of solutions. A rubber hose is used to connect the rubber suction bulb to protect the viscometer; the measurement sequence is adjusted to eliminate intermediate drying and save experimental time. The bubble removal method of sealing tube B and blowing air with inclined operation requires no defoamer, avoiding measurement errors caused by changes in the solvent system. Appropriate experimental temperature, capillary specification and initial sample concentration are determined, and the measured results are basically consistent with the nominal values of the reagent. The improved protocol features simple operation and low cost without altering the experimental principle. It can reduce instrument loss and shorten class hours, help students understand the experimental mechanism, and is suitable for promotion in polymer physics experimental teaching at universities.

7. References

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