# Specialized Experimental Module II

# Synthesis of Polyvinylpyrrolidone by an Energy-Saving Method

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## I. Objective

To synthesize polyvinylpyrrolidone using both traditional heating and microwave heating, and to compare the heating efficiency of the two methods.

## II. Technique

In this experiment, you will develop proficiency in essential laboratory techniques including accurately weighing chemicals, measuring temperature, and using a magnetic hot plate stirrer and a microwave oven.

## III. Introduction

Monomers are connected through covalent bonds to form molecules with very large molecular weights, which are called polymers. The polymer used in this experiment is polyvinylpyrrolidone (PVP), a macromolecular compound obtained by the polymerization of the monomer N-vinyl-2-pyrrolidone (vinylpyrrolidone, VP). In a dry environment, PVP appears as a white or pale-yellow powder. Because it has low toxicity and is soluble in water, ethanol, chloroform, and other organic solvents, it is often used as a food additive or a pharmaceutical binder. Common polymers in daily life include PET, PVC, and PTFE.

The two common heating methods are traditional heating and microwave heating. In traditional heating, heat from an external heat source is transferred by contact conduction through the container into the solution, and then transferred to the reactants by convection in the solution. During this process, part of the energy may be lost to the container or the surrounding air. In microwave heating, polar molecules in the reactants and solvent are irradiated by microwaves, causing molecular motion and mutual friction that generate thermal energy. Therefore, compared with traditional heating, microwave heating has the advantages of a faster heating rate, higher energy efficiency, and more uniform heating.

In this experiment, polyvinylpyrrolidone is prepared by both traditional heating and microwave heating, and the heating efficiencies are compared. Finally, polyvinylpyrrolidone is combined with silver nitrate, and qualitative analysis is performed using a UV-vis spectrometer.

## IV. Equipment

| Group Equipment/Materials | | |
|---|---|---|
| graduated cylinder | digital thermometer | 40 mL sample vial |
| hot plate stirrer | graduated cylinder | cuvette |
| lens-cleaning paper | dropper | UV-vis spectrometer |
| plastic wrap | 20 mL sample vial | microwave oven (1500 W) |
| | | |

## V. Chemicals

| |
|---|
| N-vinyl-2-pyrrolidone (VP) |
| 2,2-azobisisobutyronitrile (AIBN) |
| silver nitrate |

## VI. Procedure

### (A) Preparation of Polyvinylpyrrolidone (Microwave Heating)

1. Weigh 30 mg of AIBN into a 100 mL beaker. Add 10 mL of vinylpyrrolidone and 10 mL of deionized water. Stir at room temperature for 20 minutes to obtain a yellow solution.

2. Transfer 4 mL of the yellow solution from Step 1 into a 20 mL sample vial. Seal the vial with plastic wrap and place it into the microwave oven.

3. Set the microwave power to 750 W. Heat for 30 seconds and then stop. Allow the sample to stand for 30 seconds, then heat for another 60 seconds and stop.

4. Finally, place the sample in a 120 ℃ oven and bake for 10 minutes.

### (B) Preparation of Polyvinylpyrrolidone (Hot Plate Stirrer Heating)

1. Transfer 4 mL of the yellow solution from Step 1 into a 40 mL sample vial. Place the vial in an oil bath and heat it to 120 ℃ using a hot plate stirrer. Allow the reaction to proceed for 30 minutes.

2. After the reaction is complete, place the sample in a 120 ℃ oven and bake for 10 minutes

**(C) Qualitative Analysis of Polyvinylpyrrolidone**

1. Weigh 0.3 g of silver nitrate and 0.2 g of polyvinylpyrrolidone prepared by microwave heating into a 20 mL sample vial. Add 10 mL of deionized water, and then seal the vial with plastic wrap.

2. Place the sealed sample vial into the microwave oven and heat at 750 W for 20 seconds to obtain a yellow solution.

3. Allow the yellow solution to stand at room temperature. Then transfer part of the solution for qualitative analysis using a UV-vis spectrometer at 400-450 nm.

4. Replace the polyvinylpyrrolidone with the product prepared by hot plate stirrer heating, and repeat Steps 1-3.

Class _____ Name _____ Student ID _____ Date _____

# Synthesis of Polyvinylpyrrolidone by an Energy-Saving Method

## I. Experimental data

**(A)** Record in detail the observations made during the synthesis of polyvinylpyrrolidone by traditional heating.

**(B)** Heating record table for the microwave oven and the hot plate stirrer

| | **Microwave heating** | **Hot plate stirrer heating** |
|---|---|---|
| Power (W) | | |
| Heating time (s) | | |
| Energy consumed (kJ) | | |

**(A)** Record the absorbance of the PVP-stabilized silver nanoparticles.

| Wavelength (nm) | Microwave-heating absorbance (A) | Hot-plate-stirrer-heating absorbance (A) |
|---|---|---|
| 400 | | |
| 405 | | |
| 410 | | |
| 415 | | |
| 420 | | |
| 425 | | |
| 430 | | |
| 435 | | |
| 440 | | |
| 445 | | |
| 450 | | |

## II. Questions and Discussion

**(A)** Compare the efficiency of microwave heating and hot plate stirrer heating, and explain the reasons in detail.

**(B)** In addition to polyvinylpyrrolidone, the polymer synthesized in this experiment, provide three other examples of polymers and draw the structure of each corresponding monomer.

# III. References


1. Somenath Mitra, Nicholas P. Tworischuk. “Green Chemistry in Teaching Laboratory: Microwave Induced Reactions,” 2010.

2. Ashok K. Singh, Vijay S. Raykar, *Colloid and Polymer Science*, 2008, 1667-1673.

3. Slistan-Grijalva et al., *Materials Research Bulletin*, 2008, 90-96.