國立中正大學

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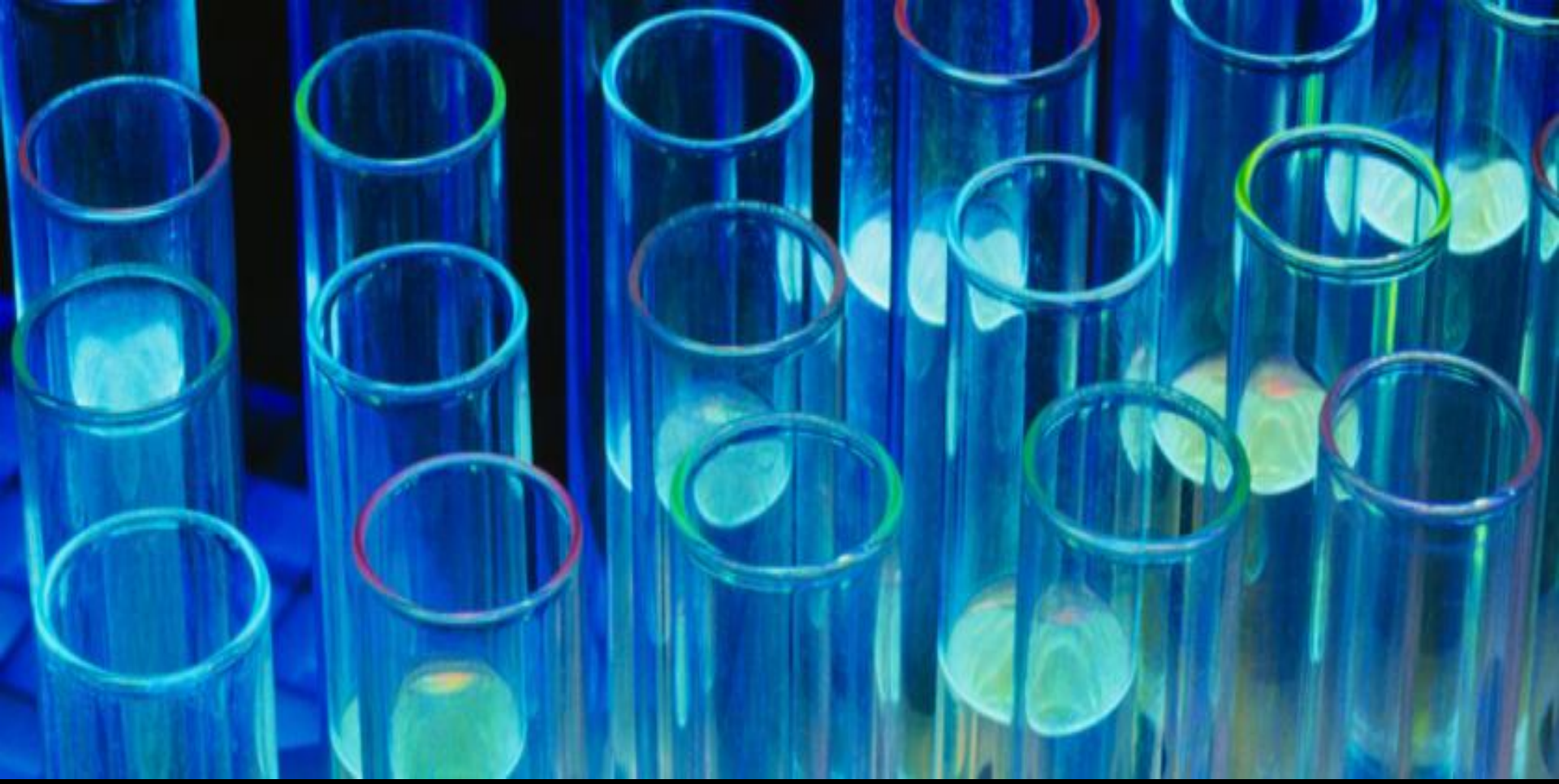
積極創新 修德澤人

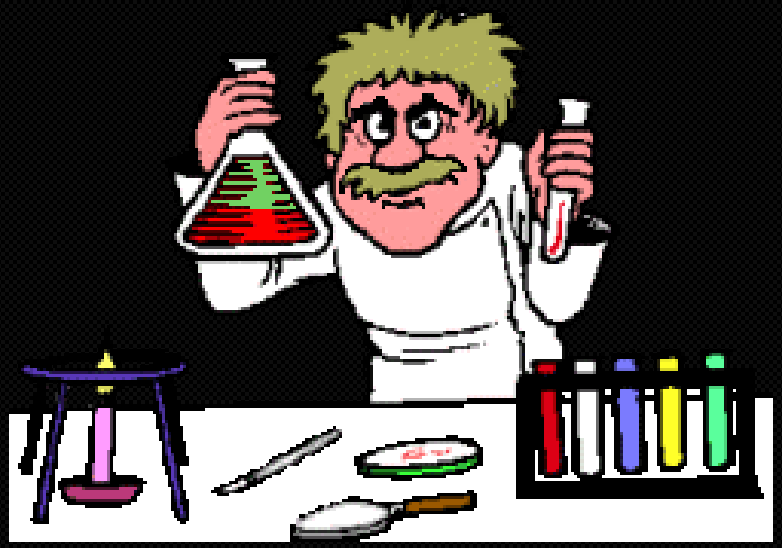


114-2
Specialized Experimental Module II : Energy-Efficient Synthesis of Poly(vinylpyrrolidone)

115.05.10 ~ 115.05.16 (time required：2 hours)

1. Objective

- Poly(vinylpyrrolidone) (PVP) was synthesized using both conventional heating and microwave-assisted heating, and the heating efficiencies of the two methods were compared.



2. Principle

- **Polymers** : They are macromolecular compounds composed of numerous repeating monomers or structural units linked together through covalent bonds. This process, known as polymerization, results in the formation of long-chain or network structures that endow polymers with distinctive physical and chemical properties.

2. Principle

- **Based on their structure and origin, polymers can be broadly classified into :**

1. **Natural polymers, such as proteins, DNA, cellulose**
2. **Synthetic polymers, such as polyethylene, polypropylene, polystyrene, and (PVP)**

- **According to the mode of polymerization, polymers are mainly formed through :**

1. **Addition polymerization**
2. **Condensation polymerization**

2. Principle

- **Poly(vinylpyrrolidone) (PVP):** Is a macromolecular compound obtained through the addition polymerization of the monomer N-vinyl-2-pyrrolidone (VP). Under dry conditions, PVP appears as a white or pale yellow powder. Owing to its low toxicity and its solubility in water, ethanol, chloroform, and other organic solvents, it is widely used as a food additive and as a pharmaceutical binder. Common polymers encountered in daily life include PET, PVC, and PTFE.

Conventional vs Microwave Heating

- **Conventional heating：** Heat from the external source is conducted through the container into the solution and then transferred to the reactants by convection. During this process, some energy may be lost to the container or the surrounding air.

Conventional vs Microwave Heating

- **Microwave heating：** Microwaves are low-energy electromagnetic waves that cause polar molecules in the reactants and solvent to vibrate rapidly under irradiation, generating heat through molecular motion and friction. Compared with conventional heating, microwave heating is faster, more energy-efficient, and more uniform.

Principles of Conventional and Microwave Heating

- The principle of microwave heating relies on the presence of an electric field, which interacts with molecules in the following two ways to generate thermal energy:

1. **Dipolar Rotation:** Molecules continuously rotate, causing their dipoles to repeatedly realign with the direction of the electric field. The friction between rotating molecules generates thermal energy.
2. **Ionic Conduction:**
   One or more free ions move through the space and attempt to align with the changing direction of the electric field. Similar to dipolar rotation, friction between these moving species generates thermal energy. As the temperature of the reaction mixture increases, the efficiency of energy transfer also increases.

3. Instruments and Chemicals

Instruments

- Except for the graduated cylinder, all equipment and materials were either shared laboratory items or provided by the teaching assistant.

Hot plate stirrer (shared use)	microwave oven (1500 W) (shared use)	spectrophotometer (shared use)
vials (40 mL)	vials (20 mL)	droppers
plastic wrap	lens paper	cuvettes
aluminum foil	graduated cylinders (10 mL)	

Chemicals

Azobisisobutyronitrile (AIBN)***	silver nitrate ($AgNO_3$)***
vinylpyrrolidone (VP)**	

* : corrosive * : irritant * : flammable * : toxic * : light-sensitive and should be protected from light by wrapping with aluminum foil

References：https://pubchem.ncbi.nlm.nih.gov

4. Experimental Procedure

Step 1 : Preparation of (PVP) by Microwave Heating

- **Weighing:** Accurately weigh 0.03 g of AIBN into a 100 mL beaker.
- **Solubilizing:** Add 10.0 mL of deionized (DI) water and VP to the beaker.
- **Stirring:** Mix the solutions and stir at room temperature for 20 minutes to obtain a pale yellow solution

4. Experimental Procedure

Step 1 : Preparation of (PVP) by Microwave Heating

- **Transfer:** Pipette 4.00 mL of the pale yellow solution into a 20 mL sample vial.
- **Sealing:** Seal the vial with plastic wrap and place it in the microwave oven.
- **Heating:** Set the microwave power to 750 W. Heat for 30 s, pause for 30 s, and then heat again for 60 s.

4. Experimental Procedure

Step 2 : Upon completion of the reaction (as shown in Figure 1), transfer the sample to an oven and heat at 120.0 ˚C for 10 min.

Figure 1. PVP solid prepared by microwave heating.

4. Experimental Procedure

2. Preparation of (PVP) by Heating with a Hot Plate Stirrer

- **Transfer:** Pipette 4.00 mL of the step 1 pale yellow solution into a 40 mL sample vial.
- **Heating:** heat and stir the pale yellow solution in an oil bath to 120 ˚C for 30 min (figure 2)
- **Baking:** After completion of the reaction (Figure 3), transfer the sample to an oven and heat it at 120˚C for 10 min.

Figure 2. Experimental setup for conventional heating.

Figure 3. Poly(vinylpyrrolidone) (PVP) solid prepared by conventional heating.

4. Experimental Procedure

3. Qualitative Analysis of the Prepared (PVP)

① Silver nitrate (0.300 g) and 0.200 g of the PVP prepared by microwave heating (as shown in Figure 4) were weighed and placed in a 20 mL sample vial. Subsequently, 10.00 mL of deionized water was added, and the vial was finally sealed with plastic wrap.

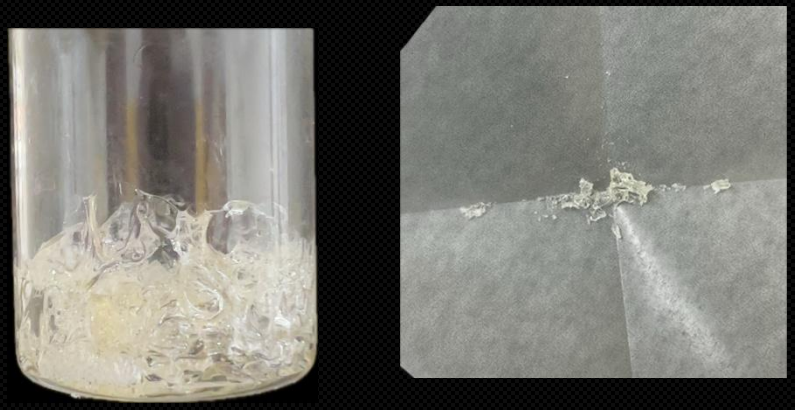


Figure 4. Solid poly(vinylpyrrolidone) (PVP).

4. Experimental Procedure

② Heating: use microwave oven and heated at 750 W for 20 s, yielding a yellow solution (Figure 5).

② Qualitative analysis: cool the yellow solution to room temperature and use a spectrophotometer at 400–450 nm.

③ Reapting: Use the PVP prepared by heating with a hotplate stirrer, and repeat Steps ①-④

Figure 5.
Appearance of the yellow solution.

Introduction to Microwave Oven Operation

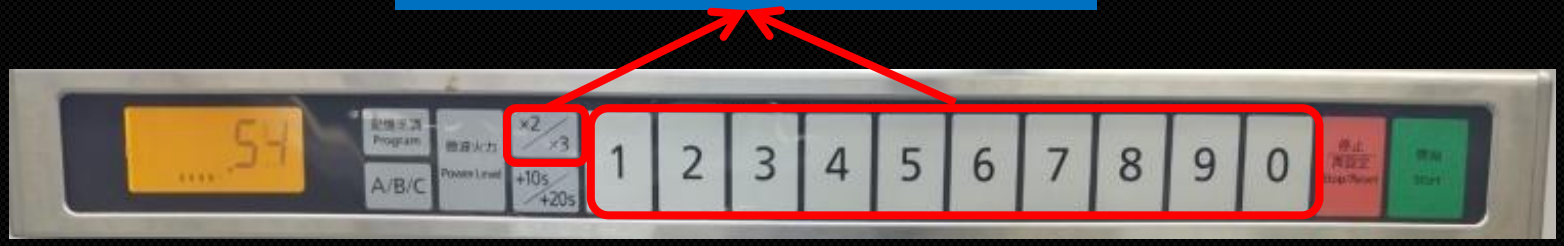
Selection of Power Level

Start and Stop

- The control panel located on the upper part of the microwave oven allows the selection of power level, as well as the start and stop functions.
- To adjust the power level, press “Microwave Power”, and the setting will sequentially decrease from P10 (1500 W) to P1 (300 W). Since the required power for this experiment was 750 W, the microwave power was set to P5.

Introduction to Microwave Oven Operation

Setting the Heating Time



- **The heating time should be set in advance by entering the desired duration using the numeric keypad, or by pressing "+10 s" to adjust the required heating time.**

- **After heating is completed, the container should not be handled directly, as it may be hot enough to cause burns. Heat-resistant gloves must be worn before removing the container.**

5. Safety Precautions (1/3)

1. **Weighing:** Accurately weigh of AIBN to prevent influence on the experimental results.
2. **Cooling:** The PVP removed from the oven should be cooled to room temperature before collection, otherwise, it may stick to the tools.
3. **Do not microwave:** Wrap the sample vial with aluminum foil when measuring silver nitrate, and remove the foil before microwaving.
4. **PVP Collection:** Use smaller PVP particles when collecting the sample to prevent undissolved particles from remaining at room temperature.

5. Safety Precautions (2/3)

5. **PVP Crushing:** Place a cloth under the vial and wear cotton gloves when crushing hard PVP, to prevent injury from vial breakage.
6. **Conventional Heating :** Ensure the oil bath reaches the set temperature before starting the reaction; otherwise, the failure rate may increase.
7. **Microwave Safety:** Keep away from the side and rear vents of the microwave. Inform the TA immediately if any liquid splashes inside the microwave.
8. **PVP Collection:** Use smaller PVP particles when collecting the sample to prevent undissolved particles from remaining at room temperature.

5. Safety Precautions (3/3)

9. **End of Experiment:** After the experiment, place the sample vials in the front plastic bottle for TA disposal.
10. **Spectrophotometer Use :** When measuring at a different wavelength, remeasure the blank to ensure accurate results.
11. **Mandatory PPE:** Full protective gear (lab coat, goggles, gloves, and mask) is required at all times.
12. **Safety Compliance:** Strictly follow all laboratory regulations and TA instructions regarding hazardous chemicals.
13. **No Removal Policy:** Taking any chemicals or equipment out of the laboratory is strictly prohibited for safety and security reasons.

6. Experimental Data and Results

1. Please record in detail the phenomena observed during the synthesis of polyvinylpyrrolidone by conventional heating.

a. Conventional heating required a longer reaction time, and the network structure formed after the solution reached boiling was less dense than that obtained by microwave heating.

b. During collection, the PVP prepared by conventional heating was found to be more viscous.

c. In the preparation of the qualitative PVP solution, the PVP produced by conventional heating required a longer time to dissolve

6. Experimental Data and Results

2. Table of Heating Records for Microwave and Hot Plate Stirrer Methods

	Microwave Heating	Hot Plate Stirrer Heating
Power (W)		
Heating Time (s)		
Energy Consumed (kJ)		
Relative Efficiency (%)		

6. Experimental Data and Results

3. The absorbance of the complex formed between (PVP) and silver nitrate should be recorded.

Wavelength (nm)	Absorbance (A) of Microwave-Heated Sample	Absorbance (A) of Hot Plate Stirrer-Heated Sample
400		
405		
410		
415		
420		
425		
430		
435		
440		
445		
450		

7. Questions and Discussion

1. This experiment focuses on the energy-efficient synthesis of PVP. Based on the data recorded in the tables in Section VI, Experimental Data and Results, calculate the amount of energy (kJ) saved by microwave heating relative to hot plate stirrer heating, and estimate the corresponding Energy Saving Rate (%).

7. Questions and Discussion

2. **Besides the poly(vinylpyrrolidone) (PVP) synthesized in this experiment, list three additional examples of polymers and illustrate the structure of the monomer corresponding to each polymer.**

7. Questions and Discussion

3. **Assuming a 200 W heater operating at an efficiency of 30% is used to heat 1 kg of water from 30 ˚C to 100 ˚C, calculate the time required for the heating process.**

7. Questions and Discussion

4. **Explain why poly(vinylpyrrolidone) (PVP) has good water solubility, whereas certain other polymers, such as poly(vinyl chloride) (PVC), are insoluble in water.**

8. Grading Criteria

1. Whether the preparation was successful was determined from the morphology of the student-prepared PVP, specifically the presence of a network structure.
2. For the spectrophotometric analysis, an absorbance value within the range of $0.2 < X < 0.6$ was regarded as meeting the criterion for a score of 80 or above.

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