P-4: Synthesis and Characterization of Polypyrrole Particle using Ultrasound

Daisuke Kobayashi*[1], Tadaaki Sakamoto[1], Tomoki Takahashi[1], Katsuto Otake[1] and Atsushi Shono[1]

[1] *Department of Industrial Chemistry, Tokyo University of Science*
Kagurazaka 1-3, Shinjuku-ku, Tokyo 162-8601, Japan, dkobayashi@ci.kagu.tus.ac.jp
[2] *Department of Chemical Science and Technology, Tokyo University of Science*
Kagurazaka 1-3, Shinjuku-ku, Tokyo 162-8601, Japan

Ultrasound was applied to chemical oxidative polymerization process of polypyrrole, and effects of frequency on characteristics of synthesized polypyrrole particles were investigated. The spherical polypyrrole particles were synthesized by chemical oxidative polymerization assistance of ultrasound, and ultrasound improve the polymerization reaction.

1.Introduction

Many researches have been carried out on synthesis and characterization of conducting polymers such as polyacetylene, polyaniline, and polythiophene. Polypyrrole is one of the most standard conducting polymers because of its environmental stability and relative ease of synthesis (Wang *et al.*, 2001). However, the poor processability of polypyrrole limits the application, and the method for synthesis of polypyrrole as fine particle using chemical oxidative polymerization is required. In this study, we focused on the control of polymerization rate, and ultrasound was applied to chemical oxidative polymerization process of polypyrrole as the model conducting polymers. The water dispersed polymerization of polypyrrole with ultrasonic irradiation was investigated in the condition of low oxidant concentration. The effects of ultrasonic frequency and reaction time on polypyrrole particle size and morphology were investigated.

2. Experimental

2.1. Experimental apparatus

Figure 1 shows the entire experimental apparatus. A stainless steel vibration plate attached with a PZT transducer (Honda Electronics Co., Ltd.) was installed in the center of the water bath at the bottom. The temperature of the water bath was maintained constant.

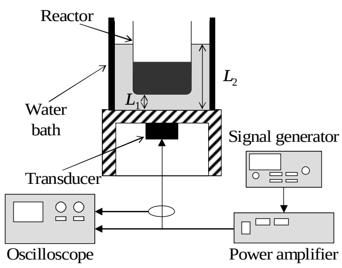

Figure 1: Experimental setup

2.2. Synthesis of polypyrrole particle

Pyrrole monomer was dispersed into water by stirring, and ammonium persulfate (APS) solution was added with ultrasonic irradiation. Total volume of sample solution, reaction temperature, and ultrasonic power were 30 mL, 298 K, and 6.5 W, respectively. Ultrasonic frequency was changed between 20 kHz and 1.6 MHz. An oxidative polymerization without ultrasonic irradiation was also investigated for the comparison.

2.3. Analysis

After the polymerization reaction was stopped, polypyrrole particles were separated from water by centrifugal separation, and washed with water and ethanol. The morphology and size of synthesized polypyrrole particle were analyzed by scanning electron microscope. And, the particle size distribution was determined by dynamic light scattering method. The ultrasonic power in the reactor was measured by calorimetry (Contamine et al., 1995).

3. Results and discussions

3.1. Morphology and size of synthesized polypyrrole particles

Figure 2 shows the SEM micrographs of synthesized polypyrrole particles for various frequencies. The concentration of APS and the volume fraction of pyrrole were 0.043 M and 0.1, respectively. The reaction time was 1 h except for condition of mixing without ultrasonic irradiation (Fig. 2 (e)). The spherical polypyrrole particles were synthesized by chemical oxidative polymerization assistance of ultrasound. The control of reaction rate is an important factor to synthesis spherical fine polypyrrole particles.

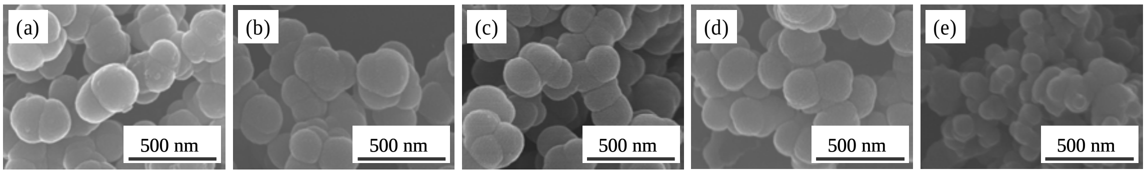

Figure 2: SEM micrographs of synthesized polypyrrole particles for various frequencies: (a) 22.8 kHz, (b) 490 kHz, (c) 940 kHz, (d) 1.6 MHz, and (e) mixing (without ultrasonic irradiation, and reaction time was 24 h)

3.2. Effects of frequency on characteristics of polypyrrole particles

Figure 3 shows the effects of ultrasonic frequency on polymer yield and average particle diameter of polypyrrole. On condition without ultrasonic irradiation, polymer yield and average particle diameter were 0.98 g and 190 nm. Polymer yields under ultrasonic irradiation were larger than that without ultrasonic irradiation. Ultrasound is considered to improve the polymerization reaction, and the enhancement of reaction was influenced by frequency.

Figure 4 shows the relationship between sonochemical efficiency (Koda *et al.*, 2003) and polymer yield. When the frequency is 22.8 kHz, the polymer yield is high in spite of low sonochemical efficiency. It is found that not only chemical effect but also physical effect has influenced activation of the polymerization reaction.

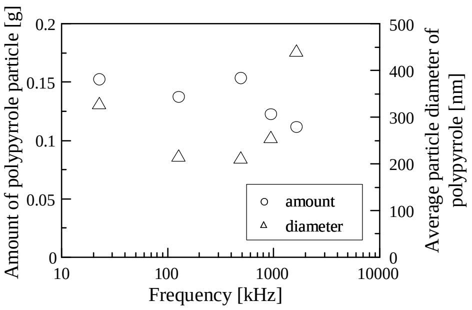

Figure 3: Effects of frequency on polymer yield and average particle diameter of polypyrrole

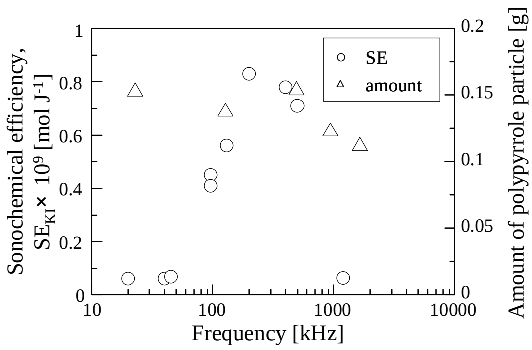

Figure 4: Relationship between sonochemical efficiency and polymer yield

4. Conclusions

The spherical polypyrrole particles were synthesized by chemical oxidative polymerization assistance of ultrasound, and the yield of polypyrrole particles under ultrasonic irradiation were larger than that without ultrasonic irradiation for the same reaction time.

References

Contamine R. F., Wilhelm A. M., Berlan J., and Delmas H., 1995, Power measurement in sonochemistry, Ultrasonics Sonochemistry, 2, S43-S47.

Koda, S., Kimura, T., Kondo, T. and Mitome H., 2003, A standard method to calibrate sonochemical efficiency of an individual reaction system, Ultrasonics Sonochemistry, 10, 149-156.

Wang L.-X., Li X.-G., and Yang Y.-L., 2001, Preparation, properties and application of polypyrroles, Reactive & Functional Polymers, 47, 125-139.