

마이크로파 반응하에 폴리올방법과 은거울반응을 이용한 은나노입자 합성

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Microwave-Polyol Process for Silver Nanoparticles

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1. INTRODUCTION

In many industrial field, silver nano particle have been proven as a effective material. The reports on new applications and technology for nanoscale semiconducting¹⁻⁶ and etallic nanoparticles has grown in the past decade due to advancements in the chemical synthetic methodologies for their preparation. When microwaves was irradiated into mixtures of $AgNO_3$ in ethylene glycol(EG)solution with a assistance of PVP as a capping reagent ,silver nano particles were preapred. When PVP was used, silver particles were prepared whose size were 100~200nm. We found that the key to the formation of ananomaterials is the use of PVP as a protecting reagent. Effect of PVP were examined by comparing with citrate which was used in silver mirror reaction. At the same time we examined the effect of microwave When we use microwave instead of oil bath heating method, the reaction time was shorter than previous method. These results indicated that microwave-polyol method is more effective synthesis way to make smaller and even silver nano particles.

2. EXPERIMENTAL

Materials. Silver nitrate, Ethylene Glycol, Trisodium citrate, Formaldehyde, Distilled water, PVP(Mw.20,000), Acetone.

In each synthesis, 5 mL ethylene glycol was first heated in an oil bath at 148 C for 4 h to remove trace amounts of water. Injection of two 3-mL EG solutions into the hot EG at a rate of 45 mL per hour. One of the solutions contained 0.94 M silver nitrate, and the other one contained 0.375 M poly(vinyl pyrrolidone) (PVP, Mw 20 000). Magnetic stirring was applied throughout the entire synthesis. The typical synthesis went through a number of color changes before the color became stable at approximately 3 h. A set of samples were taken in the course of each synthesis using a glass pipet. To minimize temperature perturbations during sampling, the glass pipet was held just above the solution and preheated for 30 s

before immersion. The samples were washed with acetone and then with water to remove most of the EG and PVP. During the washing process, the suspension was centrifuged at 3,000 rpm for 10 min or 1 h (depending on whether acetone or water was used) to make sure that most of the silver particles taken from the reaction were recovered. Finally, the sample was dispersed in water for further characterization. A reference synthesis was also performed under the same conditions, with microwave and ultrasonic sound. And then we used UV spectro scope and SEM.

A drop of the aqueous suspension of particles was placed on a piece of silicon wafer (for SEM) and dried in the fume hood. After that, the sample was transferred into a gravity-fed flow cell and washed for 1 h with deionized water to remove the remaining PVP. Finally, the sample was dried and stored in a vacuum. Then SEM images were taken. Photographs were captured. UV-visible extinction spectra were taken at room temperature using a quartz cuvette with an optical path of 1 cm. All solutions were diluted with water before spectral measurements.

3. RESULTS AND DISCUSSION

The reaction could be easily followed through its distinctive color changes. Within the first minute of injection, the solution became light yellow in color, indicating the formation of silver nanoparticles through polyol reduction. The yellow color kept increasing in intensity until $t = 8$ min, after which it maintained its appearance for another 15 min. As the reaction proceeded, the yellow color started to fade due to the dissolution of silver particles into the solution and the deposition of particles onto the inner wall of the flask. Figure 1C shows that the solution was nearly colorless at $t = 30$ min. however, the stirring bar was barely visible due to the deposition of silver nanoparticles on the flask.

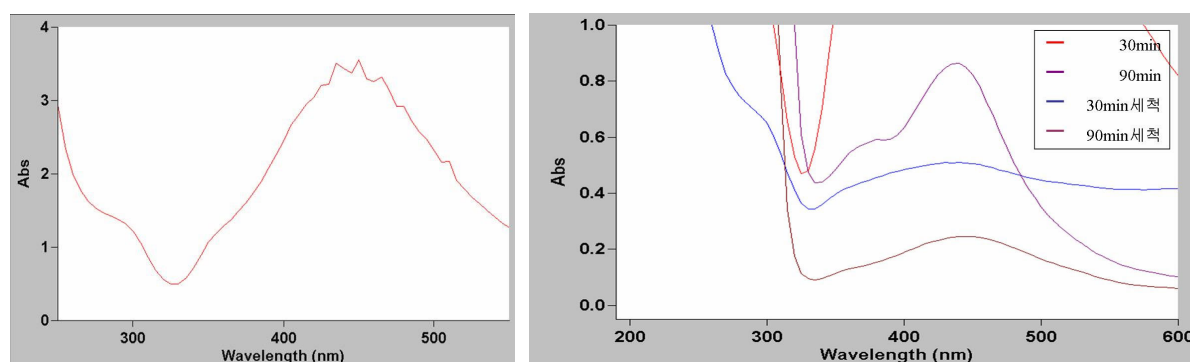


Figure 1. UV-vis spectra of samples taken from the reaction solution at different growth stages of silver nanoparticles.

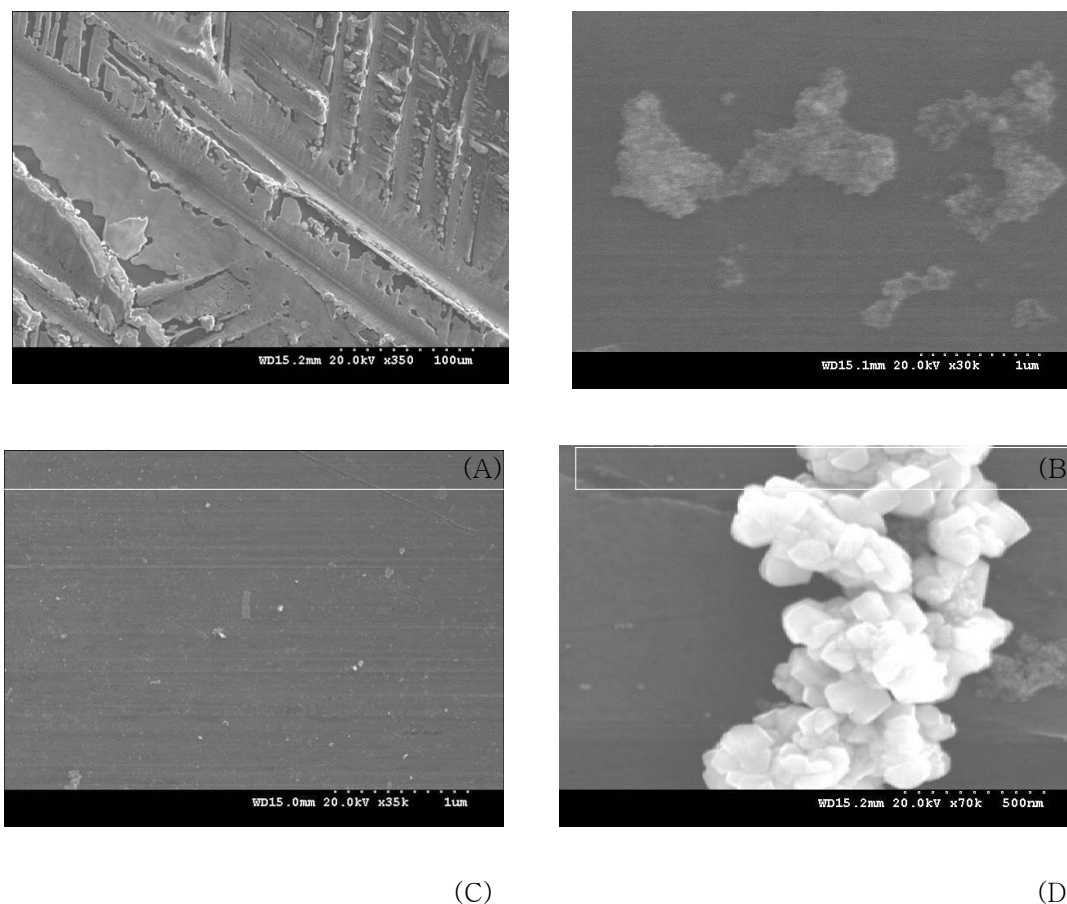


Figure 2. (A),(B) SEM image of a sample taken at 1min. without PVP(10~ μm) particles. (C),(D) SEM image of a sample taken at 1min. (~500 nm) particles.

| | Reacting time (min) | Temperature ($^{\circ}\text{C}$) | Power (W) |
|-----------------------------|---------------------|------------------------------------|-----------|
| Oil bath | 10 | 130~150 | . |
| Polyol synthesis With MW | 1~1.5 | 300 | 300 |

4. CONCLUSIONS

Silver nano particles of average size in the range of less than 100nm have grown in large scale at room temperature by reducing silver nitrate in polyol solution using the electrochemical method in the presence of PVP. PVP is electrochemically stable and the use of PVP as a stabilizer facilitated the formation of well defined nanosize silver particle in MW-assisted process. The use of the microwave eliminates thermal gradients by volumetric heating, eliminates the need for high-temperature injection for size focusing, and is scalable for commercialization.

5. REFERENCES

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