CAMURE-11 11th International Symposium on Catalysis in Multiphase Reactors
ISMR-10 10th International Symposium on Multifunctional Reactors
MILANO, ITALY - 21-24 March, 2021

Mechanism for nanoparticle synthesis by microwave and ultrasound irradiations

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1. Introduction

Quick response of microwave heating is attractive in the process of nano-particle synthesis because fine particle can be obtained. In our previous study [1], it was found that bubble is formed during the synthesis around nuclei of particle by microwave heating and the bubble growth can become initial stage of the superheating behavior. To prevent the behavior, the nucleation must be promoted. Heat generation per one particle becomes lower because the microwave absorbance energy is distributed to each particle. Accordingly, anti-solvent addition [2] and ultrasound irradiation [3] have been proposed for getting the higher suspension density. In this study, simultaneous effect of solute concentration on particle size and bubble during ultrasound irradiation and microwave irradiation were investigated.

2. Methods

Colloidal particle of ferric hydroxide was produced in small cell by heating ferric chloride solution (0.1, 0.01, 0.005 M) under the irradiation. In this study, the solution includes ethylene glycol (EG; 0, 5 and 10 vol.%). Microwave power (100 W) is irradiated for 30, 180 s. the Ultrasound (US) oscillator was installed by inserting it from the top of a microwave reactor as described in Fig.1, US is operated before microwave irradiation. The oscillator has frequency and power of 1.6 MHz and 1 W. Size profiles of particle and bubble before and after microwave were measured by DLS system [1].

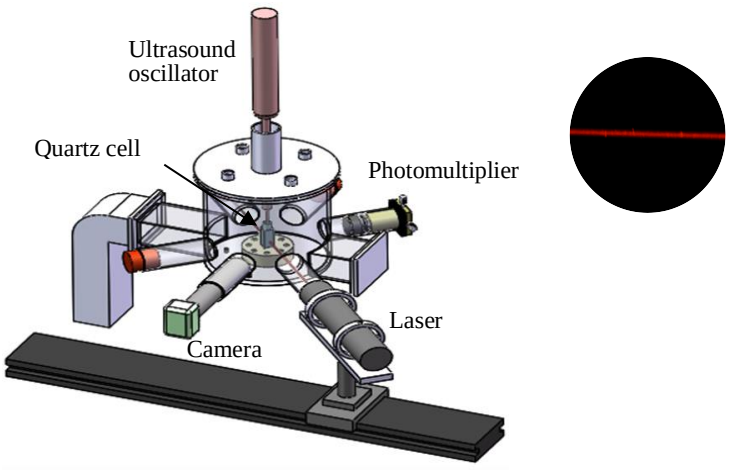

Figure 1. Microwave reactor equipped with DLS

3. Results and discussion

Figure 2 shows size and temperature profiles for each process during and after microwave irradiation. During the irradiation as shown in shade area, bubble size became larger. After microwave was turned off, bubble became smaller gradually, and the size became constant at room temperature. When Us is irradiated before

microwave operation, smaller bubble can be obtained. Microwave energy is distributed to much cavitation bubbles, which are produced by US in advance. Less energy concentration affects particle size as well, and bubble formation around particle can be prevented. Final particle size and maximum bubble size are plotted in Fig.3. When solute concentration is higher, US operation becomes more effective for bubble size and particle sizes.

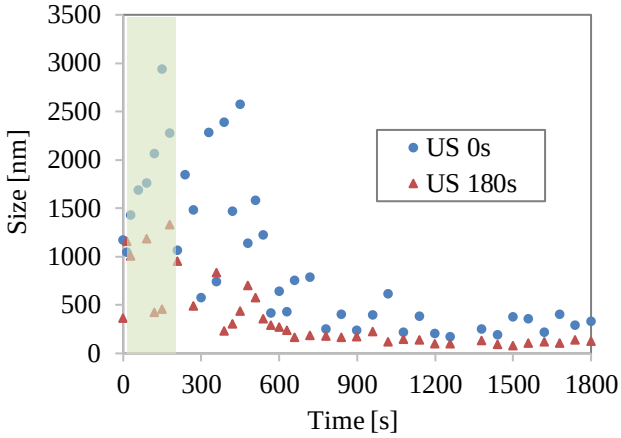

Figure 2. Example of size profiles with US and without US during microwave

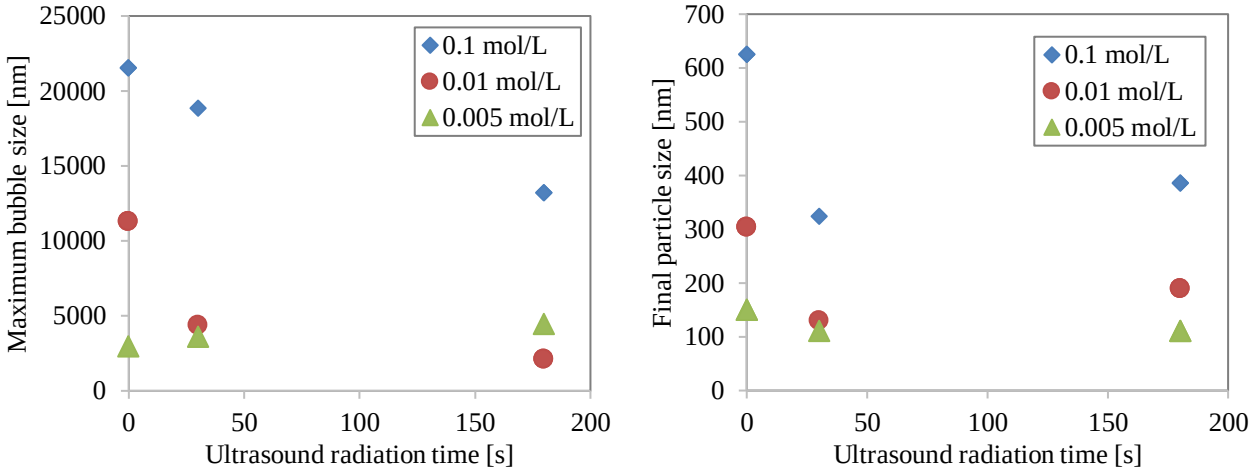

Figure 3. Summary of maximum bubble size and final particle size for different solute concentration

4. Conclusions

Nanoparticle synthesis process was investigated for combined operations of ultrasound and microwave where bubble and particle sizes were measured through in situ size measurement system. Based on size profiles, smaller particle sizes could be obtained by ultrasound operations before microwave. Cavitation bubbles produced by ultrasound plays an important role for the energy distribution of microwave energy, and it can prevent superheating behavior, which causes unstable operation. Moreover, ultrasound operation of 30 second is enough to get stable operation of microwave heating process because much cavitation bubbles are produced in advance. Thus, effect was enhanced by combination of ultrasound on microwave synergy in process of nanoparticle synthesis.

References

[1] Crystal Research and Technology, 52 (2017) 108-114
[2] Chemical Engineering and Processing: Process Intensification, 132 (2018) 11-15
[3] 2019 International Congress on Ultrasonics, (2019), 103