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Effect of microwave radiation on diffusion behavior of anti-solvent during crystallization

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ABSTRACT

The effect of microwave radiation on anti-solvent crystallization was investigated by in situ observation of nanoparticle size in a reactor. The precipitation of sodium chloride by the addition of ethanol was accelerated by the increase in molecular diffusion caused by microwave radiation. The microwave power controlled the particle size because increasing the power increases the temperature, which leads to the dissolution of the precipitate. The salt concentration in the solution, and the anti-solvent concentration and volume, were important factors in anti-solvent crystallization under microwave radiation.

KEYWORDS: A1.Diffusion, A2.Growth from solutions, B1.Sodium chloride

1 INTRODUCTION

Crystallization under microwave radiation has been extensively investigated because of its higher yields, polymorph selectivity, rapid growth, and nanocrystalline and monodispersed crystal size [1-3]. There are crystallization techniques that use homogeneous heating and induction heating through the molecular rotation of polar molecules; however, the mechanisms are not clear. Consequently, it is difficult to identify the optimum conditions for obtaining a desired size distribution. In our previous study, we observed the behavior of nanoparticles in situ under microwave radiation in a nonreaction system to clarify the microwave effect on the nanoscale [4]. The temporary aggregation of nanoparticles or bubble formation around particles caused by microwave radiation was observed by dynamic measurements in the microwave reactor [4], and nucleation induction by instantaneous radiation was observed for saturated solutions [5]. In contrast, anti-solvent crystallization is often used to obtain fine crystals and rapid growth rates [6,7]. In this study, we have combined the anti-solvent method with microwave radiation to achieve a stable crystallization process. Sodium chloride was precipitated by feeding alcohol into aqueous sodium chloride solutions under microwave radiation in order to understand how to control and predict the crystal size distribution. Important factors, such as the microwave power, salt concentration, and the concentration and volume of the anti-solvent, were investigated to clarify the mechanism of anti-solvent crystallization under microwave radiation.

2 EXPERIMENTAL

2.1 Materials

The sodium chloride and ethanol used in this experiment are analytical grade. Sodium chloride is used as a solute because the temperature dependence of its solubility in water is weak, and the effect of temperature rises caused by microwave radiation on precipitation is small. Moreover, sodium chloride has no crystal polymorphs. Ethanol is used as an anti-solvent because it completely dissolves in water and the solubility of sodium chloride in ethanol is negligible.