Nucleation and Crystal Growth Phenomena of Sodium Chloride Using Ethanol as an Antisolvent

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Antisolvent crystallization experiments were carried out to observe nucleation and crystal growth phenomena. To observe the nucleation phenomena, supersaturation was created by the addition of ethanol (antisolvent method) or the addition of saturated sodium chloride solution (reverse antisolvent method) into the saturated solution with a high ethanol content. The crystals obtained were under certain operational conditions found to be unagglomerated and monodispersed. Observation of crystal surface showed that there was a difference between the crystals produced by both methods. The relationship between the number of produced crystals and the operational conditions was obtained. In another set of experiments, ethanol was added to a saturated sodium chloride solution whilst adding seeds. The grown crystals thus obtained were found to be grown by the attachment of fine crystals that had appeared by primary nucleation from the solution. The desired operational conditions were shown for obtaining crystals having little agglomerated form. A model of the crystal growth process of sodium chloride on seeds is suggested.

1. INTRODUCTION

Antisolvent crystallization is expected to be an alternative process to evaporative crystallization, to produce the crystals at lower temperature, and the research on antisolvent crystallization is developing. Antisolvent crystallization, however, often results in producing fine, variously shaped and agglomerated crystals. This fact may lead to a decrease in product quality and purity.

In this paper, nucleation phenomena and crystal growth phenomena were experimentally investigated under the conditions of high ethanol ratio.

2.EXPERIMENTAL

The antisolvent crystallization experiment of sodium chloride was carried out using ethanol as antisolvent on batchwise operation. [1], [2]

3.RESULT and DISCUSSIONS

3.1 Nucleation experiment

The obtained crystals by both methods shown in Fig.1 were found to be unagglomerated and monodispersed under certain operational conditions. From the observation of crystals, there was a difference between the crystals produced by both methods. In the r-antisolvent method, the small crystals were observed to be relatively wider in CSD range, and from surface observation, to have more often a hollow surface compared with antisolvent method in which obtained crystals have perfectly smooth surface. The difference of surface condition is considered to be (partly) due to the differences in solvent composition in the crystallization field and solution fluid condition.

Using the experimental data, the operational range of volume of feed and ethanol volumetric ratio of starting solution for obtaining monodispersed and unagglomerated cubic crystals of sodium chloride was experimentally determined, as shown in Fig.2.

From the crystals obtained under these conditions, the average crystal size and weight of obtained crystals were measured to calculate the number of produced crystals. From the relationship between the operational conditions, average crystal size and the number of produced crystals, the average crystal size seemed almost same under the operations of the same ethanol volumetric ratio of starting solution, which suggested that the average crystal size was affected by the supersaturation ratio of the feed and starting solution of determined ethanol volumetric ratio. The number of produced crystals seemed to increase with the increase in weight of crystals based on the feed volume.

3.2 Crystal growth experiment

Obtained grown crystals were found to be grown by the attachment of fine crystals that has appeared by primary nucleation from the solution.

The evaluation of obtained crystals were performed to suggest the desired operational conditions to obtain crystals with little agglomeration form, and resulted that the sufficient operational condition were in the

range of 0.4~0.6 on the starting ethanol volumetric ratio in the solution.

REFERENCES

1. Shogo Kaneko et al, Nihon Kaisuigakkai Dai50nenkai-yousisyu, (1999) 107
2. Shogo Kaneko et al, Nihon Kaisuigakkai Dai50nenkai-yousisyu, (1999) 65

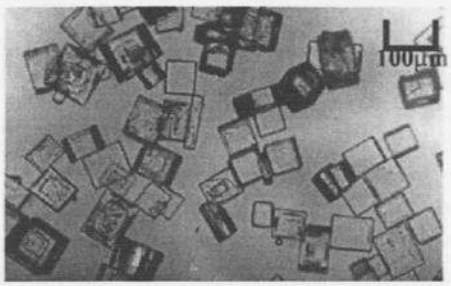

Fig.1 Photograph of obtained sodium chloride crystals

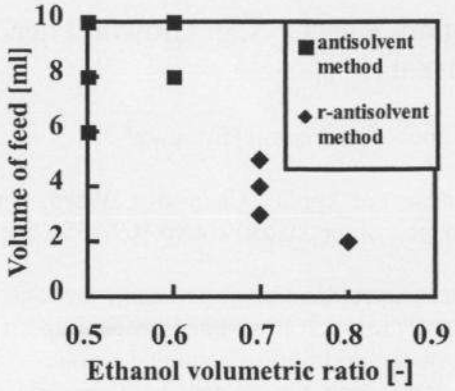

Fig.2 Operational range for obtaining monodispersed cubic crystals