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Development of Scale-Up Method of Generation Rate of Attrition Fragments Due to Crystal Collisions with Impeller Blade in a Stirred-Type Crystallizer

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Summary

Stirring operations in a crystallizer often induce crystal particle abrasion caused by particle collision with impeller blades and subsequent secondary nucleation. This study developed a method for measuring size distribution and total number of attrition fragments generated by crystal collisions in a stirred vessel filled with anti-solvent (silicone oil) using potassium sulfate and potassium alum as the model crystals. Both of model crystals dose not dissolve and agglomerate in the oil. Therefore the change of the parent particle shape and attrition fragments caused by crystal collisions can be observed clearly.

A time series of SEM images of both the abraded parent crystals and the crystal attrition fragments were taken during several tens of hours. Time evolution of the total number N_f and size distribution of attrition fragments were analyzed based on an image processing algorithm using MATLAB® program. Furthermore, abraded volumes of the parent crystals were quantified using CAD software by modeling the abraded crystals. The relation between the generation rate of attrition fragments $B_{f,p}$ and ratio of the abraded volume to initial volume of a parent crystal r_a were examined.

In the case of potassium alum, N_f and r_a increase rapidly just after the stirring started, irrespective of the impeller speed and vessel size. Then they saturate about t = 20 h. Attrition fragments are generated mainly from cones of a parent crystal. In the case of potassium sulfate, N_f and r_a increase monotonically with stirring time until 40 h. Attrition fragments are generated from cones and edges of the parent crystals. In both the model cases, The attrition fragments generation rate $B_{f,p}$ decreases drastically as the parent crystal cones roundness increase, and was found to be correlated with r_a. $B_{f,p}$ of potassium sulfate is larger than that of potassium alum, and changes loosely with r_a compared to potassium alum. That means, it is important to consider the roundness of crystal cones when the generation rate of attrition fragments is estimated. And the attrition fragments from potassium sulfate are generated easily, and are continued to be generated longer compared to potassium alum.