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Original Research Paper

Influence of the initial volume fill ratio of the granulator on melt agglomeration behavior in a high-shear mixer

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ABSTRACT

The purpose of this study was to investigate the effects of different initial volume fill ratios of the granulator on granular agglomeration in a high-shear mixer. Calcium carbonate powders, with mean granule sizes of 75–150 μm were used as the raw material. Polyethylene glycol 6000 (PEG 6000) was used as the melting binder. The initial liquid to solid weight ratio was fixed at 0.15. Four different initial granulator fill ratios were used 11%, 14.7%, 18.4% and 22%. The results showed that the granules that formed during the nucleation stage were a little larger when the initial fill ratio was lower than higher. In the rapid-growth stage, the agglomeration growth rate increased as the initial fill ratio increased. The range in granule size at the end of agglomeration also increased as initial fill ratio increased. SEM images of the surface structure of the granules during the nucleation and final stages are shown.

1. Introduction

Agglomeration is a process whereby a liquid binder is used to help convert fine powder granules into larger ones, but the initial and primary particles can still be distinguished [1]. This process can be facilitated by the use of fluid beds, rotation drums, and high-shear mixers. These devices differ in the types of particle motion they induce, and for this reason, there are also differences in the mechanism of granule growth. In the pharmaceutical industry, a high-shear mixer is often used to reduce potential segregation, improve the dynamic properties of the fluid used, and mix the particles. This compaction and extrusion technique is achieved through mechanical collision and pressurization processes of particles which are bounded by other particles. The forces of repellence and attraction affect particle collisions during the process of wet-granulation. Repellence is produced by the elastic force. The attraction between a liquid and a solid are determined by the forces of cohesion and adhesion. The cohesive force comes from the attraction between the liquid molecules. The adhesive force is, however, a reciprocal attractive force at the interface between a liquid and a solid [2].

The process of wet granulation is very complicated and includes several simultaneous mechanisms. These mechanisms are: (i) wetting and nucleation; (ii) consolidation and coalescence; and (iii) attrition and breakage [3,4]. Nucleation is the mechanism by which small particles bind to others due to the forces of collision and adhesion. Schæfer and Mathiesen [5,6] considered the process of nucleation with a liquid binder to include both a distribution and an immersion mechanism. The state of these two mechanisms depended on the initial binder crystal size, the viscosity of the molten binder and the impeller speed. The distribution mechanism occurs when the binder droplets are smaller than the particles. The immersion mechanism occurs when the binder droplets are larger than the particles [7]. The particle-nucleation behaviors usually occur in the initial phase of granulation, although there are other mechanisms (e.g., consolidation, coalescence and attrition) that affect the deformability of the granules following the nucleation stage.

In efforts to determine the possible rate of consolidation of granules, Ennis et al. [8] established an expression which considers the relationship between granular formation and pendular bridges among the particles. Their results showed that high kinetic energy between the particles and a low binder viscosity would increase the consolidation rate, and that the frequency of collisions also influences the consolidation time. On the other hand, the impact behavior of wet granules not only makes for fewer granule voidages during the consolidation stage but also increases the probability of rebound [9]. Iveson and Litster [10] defined two growth behaviors based on the deformability of the system: steady growth and induction time behaviors. Their induction stage, however, is similar to the consolidation stage, where the collision forces compress the granules, squeezing the air and liquid binder from the interior to the granule surface. This is followed by a rapid-growth stage [11,12] where the constituent granules are drawn closer together and rearranged. After the consolidation stage, the surface of the

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