Form, formation, and deformation
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Summary

High shear granulation

The introduction of this thesis describes the high shear granulation process. High shear granulation is a commonly used unit operation to produce larger granules of primary particles. The granulation process has been described to consist of different stages.

The initial growth stage is the nucleation regime, where wetted particles stick together and form primary nuclei. Due to densification, the nuclei consolidate and coalescence of nuclei occurs; the start of the second growth stage. In the further course of this stage, growth stops and compaction of these agglomerates starts. Due to the collisions, the packing of the particles becomes denser and liquid moves to the granule surface. When enough binder liquid is present at the granule surface, further growth by coalescence is possible. After a successful collision, particle rearrangement to a sphere may occur. Coalescence promotes the growth of larger granules. At the end of the coalescence regime, crushing and layering will be the predominant process. The last growth stage is now reached: granules become too large to withstand the high shear forces. Breakage and attrition takes place, the broken pieces can be layered around existing granules, or can coalesce. In this way, an equilibrium between growth and breakage is reached (Chapter 1).

One of the advantages of high shear granulation is that spherical granules can be obtained. However, this goal is not always achieved. Therefore, the aim of this thesis is to investigate which material properties and process conditions are responsible for the shape of a granule.

Many parameters (e.g., impeller speed, massing time, powder properties, liquid properties, etc.) influence the granulation procedure. Since granule shape depends on the granule growth mechanism, these parameters also influence granule shape. Due to the complicated nature of the granulation process, it is not possible to predict the effects of changes in individual parameters on the granulation process. Therefore, more research is necessary to predict granule shape on the basis of the process conditions and the material properties of the material to be granulated (Chapter 1).

When looking at granule shape, a shape factor needs to be chosen to quantify granule shape (Chapter 2). In this thesis we aimed to create smooth, spherical granules, a shape characterised by the fact that the granules have the least surface area for a certain volume. Therefore, using the projection shape factor, together with a roughness factor was optimal. The projection shape factor compares the smoothed perimeter of the two dimensional picture to the area.
Equipment effects

It is known that equipment has an effect on the granulation process. In Chapter 3 the effect of vessel wall material on the nucleation process, and therefore on the final granule size distribution is described. Glass and stainless steel vessels produce similar granules, whereas PMMA and PTFE vessels give different granule size distributions. The contact angle of the vessel wall and the sorption rate of the powder used determine the type of nucleation process that will occur. A high contact angle of the vessel wall material, combined with a fast imbibing powder lead to a good distribution of liquid over the powder bed, thus resulting in a small particle size distribution.

In some cases, even when a vessel with a high contact angle and a fast imbibing powder is used, a broad size distribution is obtained (Chapter 4). Again the distribution of liquid over the powder bed is not homogeneous. When initially not all powder is taking part in the granulation process because it is sticking to the lid or because of overloading of the mixer, irreproducible granule size distributions may be obtained.

One of the items used to control the granulation process is torque (Chapter 5). It was found that whenever more binder liquid was used, larger granules and a higher torque were obtained. We have shown that the increased torque found when granule size increases is not caused by the higher mass of the individual granules. The major granule property determining torque is stickiness. Obviously stickiness links the granule size to torque. This implies that changes in composition or surface properties of the granules that affect stickiness will change the granule size-torque relationship.

Granule growth

Colour experiments can be used to reveal the granulation mechanism (Chapter 6). Exchange of solid material does occur during the equilibrium phase of wet granulation. Three different mechanisms of material exchange were identified; exchange by disintegration, where granules are rapidly crushed and formed to granules again, exchange by deformation, where abraded granule fragments immediately fuse with other granules, and exchange by distribution, a mechanism during which granules remain intact over a prolonged period during which hardly material exchange occurs which after a slight densification is followed by uncontrolled growth and exchange of material.

It was found that it was possible to shift between these mechanisms by changing the process conditions, which influences wet granule strength and deformability. For example, more liquid binder results in more deformable granules, which may result in a shift to the deformation mechanism.

Deformability and densification of MCC granules depend on the liquid amount. Chapter 7 shows how the granulation behaviour of MCC depends on the liquid content. The liquid should not only be regarded as a binder but also its effects on deformability of the granules should be taken into account, since the deformability
determines the granulation mechanisms occurring during the latter phases (consolidation and growth, and breakage and attrition) of the granulation process. When a low amount of binder liquid is available, densification of the granule will occur and the final phase of the granulation process will result in equilibrium between attrition and growth. Since the granules are no longer broken during this phase, spheronization of the material may occur. However, when an excess of binder liquid is used, the deformability of MCC granules increases, which will make them more easily subject to breakage upon continued granulation. Continued breakage will reduce the extent of densification of the granule core and the granules remain weak during the complete process. During the last (equilibrium) phase of the process, continuous breakage occurs. This breakage is now balanced by coalescence of the fragments with each other or with other granules. This results in weak, irregularly shaped granules.

**Growth regime map**

To understand the granulation process that occurs, the growth regime map is a useful tool. A quick method to assess the growth regime is possible by performing material exchange studies. When water is used as binder liquid, Stokes deformation number cannot be used as a predictive tool for the granulation regime. The method to calculate wet granule strength is not accurate when using low viscosity binders (Chapter 6). Therefore, the vertical drop experiments used to describe the deformability of wet material as performed by the inventors of the growth regime map were repeated (Chapter 8). Most powder-liquid combinations were found where expected. However, the experimental errors (inherent to the method) result in extremely large variations, overlapping all growth regimes. This shows that Stokes deformation number cannot be measured unambiguously. Until it has been improved, the growth regime map can only be used as a guidance to order different occurrences happening during granulation.