Fundamentals of the high-shear pelletisation process
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Document Version
Publisher's PDF, also known as Version of record

Publication date:
2001

Link to publication in University of Groningen/UMCG research database

Citation for published version (APA):
In this thesis, several aspects of the pelletisation process in a high-shear mixer were discussed. The nucleation stage, as the first stage of the pelletisation process, was investigated in two Gral high-shear mixers (chapter 2). As soon as the first liquid drops fall on the bed of dry powder particles (a mixture of lactose and microcrystalline cellulose), the first primary nuclei are formed. These nuclei consist of many powder particles and one liquid drop. This stage of growth is characterised by the increasing $d_{90}$ of the particle size distribution, while the $d_{50}$ and $d_{10}$-values remain rather unchanged. Furthermore, these nuclei are relatively large and too weak to withstand the impact of the impeller. The primary nuclei will be densified and broken due to the actions of the impeller and the chopper. As a result of this breaking, secondary nuclei are formed. These secondary nuclei bring about the distribution of the binder liquid. Nucleation ends when exponential growth of the particles is observed. Exponential growth of pellets occurs due to the densification-action of the impeller. The porosity of pellets decreases resulting in liquid (which was occupying the pores) to be squeezed out to the surface of the pellet. Subsequently, the surface of the pellet is surrounded with a small liquid layer, and growth can proceed. Finally, when the minimal porosity of the pellet is reached, no more densification can occur and the growth rate diminishes. Equilibrium between the growth rate and breakage rate of pellets has been reached. Growth and breakage of pellets still occur during this equilibrium stage, but the mean pellet size and the pellet size distribution remains constant.

The suitability of the coffee grinder as a downscaled high-shear mixer was demonstrated in chapter 3. Growth mechanisms comparable to the larger-scale high-shear mixer were found as well as comparable results of tracer experiments. A similar flow profile of pellets was found in the downscaled high-shear mixer. The total velocity of the pellets at the top of the torus showed to be independent of the impeller rotational speed.

Tracer experiments were performed in order to investigate the transport characteristics of pellets and the growth mechanisms of pellets. By replacing a part of the batch of wet pellets by coloured wet pellets, the exchange of the colour with the other particles could be measured. It was shown that within a few seconds a homogenous colour distribution was
obtained, demonstrating that growth and breakage of pellets still occur during the equilibrium stage of growth at an amazing rate. The rate of change of the colour concentration of a specific size class is expressed with the conversion rate constant. High conversion rate constants were found for small particles, whereas for particles larger than the mean particle size, constant but lower conversion rate constants were found. Also, at a higher impeller rotational speed, a homogeneous colour distribution was found resulting in a shorter time. This resulted in larger conversion rate constants.

The influence of the impeller rotational speed on the conversion rate constants could not be related to the relative swept volume, because the impeller acts as a rotating disk. Therefore a higher impeller speed not directly results into a higher swept volume of the pellets. The influence of the impeller rotational speed on the conversion rate constants showed to be dependent on the power input. More power input at higher impeller speeds caused larger conversion rate constants.

The mechanism generating the flow profile of pellets in the high-shear mixer was investigated in chapter 4. Small-scale experiments showed that the wet pellets stay inside the torus, even without the lid on the coffee grinder. Dry pellets flew away immediately when the lid was removed. The visco-elastic properties of the pellets showed to have a great influence on the stability of the torus. Comparison of some possible mechanisms by means of some order of magnitude calculations showed that the flow profile of pellets in the high-shear mixer could be explained by the propulsive mechanism. The propulsive mechanism exists of a combination between the centrifugal forces (moving the pellets towards the wall at the bottom of the torus), accumulation (at the wall, responsible for the upward motion at the wall), and a force due to gravity (causing the downwards motion of the pellets from the top of the torus).

The deformability of wet pellets was measured with horizontal impact experiments (chapter 5). Wet pellets were connected to a cotton thread and were impacted on a vertical plate. From the impact velocity, rebound velocity and impact radius, the coefficient of restitution (0.2-0.3), yield pressure (0.1-0.9 MPa) and elasticity modulus (10-130 MPa) of pellets could be calculated, depending on the moisture content. A differentiation between viscous deformation (velocity dependent) and plastic deformation (velocity independent) could be made. At moisture contents of $H = 1$ (moisture content at which pellets normally are made), a more plastic deformation was found, whereas at higher moisture contents, a more viscous deformation of the pellets was obtained.

In chapter 6, growth of pellets was modelled with the population balance. The population balance was presented in mass distributions. And breakage of pellets was included as well, because breakage of pellets highly affects the pellet size distribution during pelletisation in a high-shear mixer. Three different breakage mechanisms - fragmentation, shattering, and abrasion - were modelled. For the coffee grinder experiments, shattering could be excluded as a possible breakage mechanism. For the Gral experiments, no distinction could be made. All
breakage mechanisms were supposed to occur simultaneously, each mechanism occurring at different preferable locations in the bowl. Fragmentation and shattering are likely to occur at the impeller site, whereas abrasion occurs inside the torus due to pellet-pellet collisions or due to pellet-wall collisions.

In this thesis, a fundamental study of the high-shear pelletisation process is presented. Pelletisation was described by looking at the process, which provides more knowledge in order to understand what really goes on. Pellet growth mechanisms are described, especially at the nucleation stage and the equilibrium stage.

In order to gain more insight in the reason why microcrystalline cellulose is such an excellent pelletisation excipient, modelling of the pellet deformation is performed. This resulted into the determination of the coefficient of restitution and the yield pressure of wet pellets. With these results one can search for more pelletisation excipients, with similar wet-mass properties.

Furthermore, population balance modelling at the equilibrium stage of growth has been performed. In addition to established modelling, three different breakage mechanisms of pellets were defined and included in the population balance.

With the results described in this thesis, more insight in the pelletisation process in the high-shear mixer is obtained. This results in the possibility to change the pelletisation process in such a way that the final pellets are in agreement with the required properties.