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Three dimensional characterization of morphology and internal structure of soft material agglomerates produced in spray fluidized bed by X-ray tomography

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ABSTRACT

Food powders such as maltodextrin are often produced in agglomerate form in spray fluidized beds in order to enhance their user properties. These agglomerates mostly have complex structures and irregular shapes. The internal structure and morphology of food agglomerates have rarely been investigated at the microscopic scale. In this work, a nondestructive X-ray micro-computed tomography technique is used as an appropriate experimental method to overcome this lack of data by a thorough characterization of the three-dimensional internal structure of maltodextrin agglomerates. A sequence of image processing steps is applied to the X-ray images in order to obtain 3D views and to extract data for the morphological characterization. The internal porosity as well as the size and spatial distribution of the pores inside the agglomerates are evaluated. Open pores formed during the agglomeration process are also determined from the X-ray images. The agglomerate shape is investigated and compared by 2D and 3D image analyses. Maltodextrin primary particles with non-spherical shape have a broad size distribution, and they may deform and overlap as they go above the glass transition temperature during the agglomeration process. A comprehensive methodology is developed based on the preflooded watershed segmentation of X-ray images to distinguish the primary particles in maltodextrin agglomerates. On this basis, the radius of gyration and the fractal dimension are calculated. A low fractal dimension of 1.8 is found, which proves that the structure of maltodextrin agglomerates is more open and fluffier than the structure of insoluble hard material agglomerates.

1. Introduction

In various industries fine powders are produced in agglomerated form in a fluidized bed in order to improve their flowability, instant properties or simply to improve the optical appearance of the product [1]. Fluidized bed agglomeration is a complex process with many interdependent factors that influence the quality of the end product. In spray fluidized bed agglomeration, the primary particles are fluidized by blowing hot air from the bottom of the granulator, while a binder solution or suspension is sprayed as small droplets onto the particles, creating liquid bridges, which finally lead to agglomerates. Our understanding of the physical phenomena that occur during spray fluidized bed agglomeration has been significantly enhanced by recently developed Monte Carlo simulation [2] and population balance models [3]. Despite this progress, it still remains a challenge to characterize the structure of agglomerates (especially food agglomerates) produced in a spray fluidized bed. A successful characterization should allow a better understanding of process–structure or structure–property relationships. The quantification of the internal microstructure of agglomerates is also crucial for setting up processing maps and for describing agglomeration patterns and mechanisms.

Most of the food, pharmaceutical and chemical powders which are agglomerated are water-soluble. Maltodextrin may serve as a model substance for many amorphous-water soluble food powders. Agglomeration of this kind of material, which has a low glass transition temperature, is more difficult due to the strong adhesive forces between moist amorphous particles. This leads to a rather broad particle size distribution and to the formation of a crust on the equipment surface and around the spraying nozzle [1].

Several studies were performed on the effect of process parameters such as fluidizing air flow rate, temperature, agglomeration time and binder spray rate on the growth kinetics of maltodextrin agglomerates, and on their physical, mechanical and rheological properties [4–6]. Moreover, in recent publications on the agglomeration of amorphous material, the fluid, particle and collision dynamics inside a fluidized bed granulator was described in detail using coupled DEM–CFD simulations. Different process variables and granulator configurations (i.e., top spray, Wurster coater, spouted bed) were compared in terms of the agglomeration probability, the breakage and growth rate as well as the agglomerate strength [7,8].

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Nomenclature

A	area [m^2]
C	circularity [−]
d_A	equivalent projected area diameter of primary particle [m]
d_{FeMin}	minimum Feret diameter of primary particle [m]
d_V	equivalent volume diameter of primary particle [m]
D_f	fractal dimension [−]
D_{FeMin}	minimum Feret diameter of agglomerate [m]
I_G	mass moment of inertia [kg m^2]
k_g	fractal pre-factor
M	mass [kg]
N_p	number of primary particles in agglomerate [−]
P	perimeter [m]
r	radial coordinate [m]
r_G	position vector of center of mass [m]
r_i	position vector of primary particle center [m]
$\bar{r}_p$	mean radius of primary particles [m]
R_e	equivalent radius [m]
R_g	radius of gyration [m]
S	surface area [m^2]
V_{agg}	total volume of agglomerate [m^3]
V_b	bulk volume [m^3]
V_{cp}	volume of closed pores
V_s	volume of compact solid phase in agglomerate [m^3]
$V_{s,cp}$	volume of solid phase including internal pores in agglomerate [m^3]

Greek letters

ε_b	bulk porosity [−]
ε_{cp}	porosity of closed pores [−]
ε_{op}	porosity of open pores [−]
ρ	mass density [kg/m]
ϕ_S	sphericity [−]

The physical properties of a material are strongly influenced by its internal microstructure, which is created during processing [9]. Despite the amount of research on maltodextrin agglomeration, the internal microstructure and morphology of this kind of agglomerates have rarely been investigated, especially in three dimensions. Most of the available studies were performed with non-soluble particles and only a few of them investigated the microstructure of particles undergoing glass transition [10,11]. The first systematic studies on various morphological descriptors for fluidized bed agglomerates were published by Dadkhah et al. [12] for hard non-porous and porous primary particles (glass beads and γ-Al_2O_3, respectively). The primary particles in [12] are insoluble in the binder, however the maltodextrin is amorphous material that can absorb water and deform during agglomeration. The methodology and image processing sequences developed in [12] are merely effective for agglomerates made from primary particles which do not deviate too much from the spherical shape. Whereas, the methodology developed in this work can be used for agglomerates with irregular shape.

Common analytical techniques applied to the study of agglomerate microstructure are restricted to two-dimensions. The advantage of these techniques is that they provide rather inexpensive and rapid quantitative analysis. The drawback is that the sample preparation for such techniques is often destructive and the techniques do not provide direct information in the third dimension. Therefore, such 2D data may not fully represent the true 3D structures [13]. X-ray micro-computed tomography (μ-CT) on agglomerate specimens provides an opportunity to completely analyze the structure of the sample in three dimensions. X-ray μ-CT is a nondestructive 3D imaging technique which uses a set of two-dimensional shadow X-ray images of an object to reconstruct its three-dimensional structure using a mathematical algorithm [14].

In this study, suitable process parameters were determined for the production of maltodextrin agglomerates in a spray fluidized bed. In two-dimensional analyses, the size and shape of the primary particles and of the agglomerates were studied with a Camsizer. The results are presented as the evolution of the median diameter, particle size distribution and circularity. The microstructure of individual agglomerates (obtained by X-ray micro tomography) is visualized down to details that contain valuable information such as the actual morphology and spatial distribution of primary particles and pores, which cannot be assessed by other techniques. By further processing of the X-ray image sequences, the micro-scale morphology of soft agglomerates made of maltodextrin particles is studied and the results are evaluated quantitatively. The internal porosity and the pore size distribution of the primary particles as well as of the agglomerates are obtained and evaluated. The open pores of agglomerates, which comprise relatively large cavities and channels, are also determined from the X-ray images. The open porosity of agglomerates is calculated by three different methods, i.e. convex hull, dilation and radius of gyration, and the results are compared. The bulk porosity is also measured for maltodextrin particles before and after agglomeration. The shape of the agglomerates is analyzed in terms of sphericity and compared with two-dimensional values. Moreover, a comprehensive methodology is developed based on the segmentation method using preflooded watershed transform to distinguish and separate the primary particles in maltodextrin agglomerates. The gyration radius and fractal dimension of agglomerates are also calculated based on the separated primary particles.

2. Materials and experimental methods

2.1. Spray fluidized bed agglomeration

In order to obtain a narrow size distribution of primary particles used in the agglomeration process, maltodextrin powder (DE 12, Glucidex, Roquette, France) was sieved in the range of 300 to 500 μm. The agglomeration was performed in a lab-scale batch fluidized bed granulator (GPCG 1.1 LabSystem) with a transparent, cylindrical fluidization chamber made of Plexiglas with 152 mm inner diameter and 450 mm height (Glatt GmbH, Germany). For amorphous polar (water-soluble) powders it is mostly sufficient to atomize water on the fluidized particles [1]. Therefore, pure water was sprayed as a binder (plasticizing agent) with a two-fluid nozzle (model 940) provided by Düsen-Schlick GmbH (Untersiemau, Germany). The nozzle was placed on top of the chamber at the height of 150 mm from the distributor plate and operated with relative air pressure of 0.5 bar. For each experiment, 50 g of powder was fluidized using a constant fluidization air flow rate of 70 kg/h, heated by an electrical heater up to 50 °C before it enters into the chamber. The temperature sensor was located below the distributor plate. The maltodextrin powder was preheated before spraying the water. With a piston pump the water was sprayed at a constant rate. The process parameters for agglomeration are summarized in Table 1. The total agglomeration time was about 5 min and after that a sample

Table 1
The values of process parameters used for the agglomeration of maltodextrin DE 12.

Parameter	Value
Primary particle diameter (μm)	300–500
Bed hold up mass (g)	50
Air flow rate (kg/h)	70
Inlet air temperature (°C)	50
Binder spraying rate (g/min)	1.75
Atomization air pressure (bar)	0.5