Micro-structure of particles produced by fluidized bed agglomeration of soft materials

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Introduction. Though spray fluidized bed agglomeration is a widespread process in the food, pharmaceutical and chemical industry, the structure of the respective agglomerates had hardly been investigated until recently. The first systematic results were published by Dadkhah et al. (2012). In this work, the positions of primary particles in the agglomerate were determined by means of X-ray micro-tomography, and various morphological descriptors were evaluated. However, only hard primary particles (glass, ceramic) have been used, which stick together by spraying into the fluidized bed the aqueous solution of a binder and evaporating the water. This investigation shall, therefore, be continued with soft materials that have a low glass transition temperature, agglomerated by spraying a binder solution, pure water, or without any spraying. Structural characterization shall be based on the results of X-ray micro-CT scans.

Problem Definition

Macroscopic features of agglomerates are directly influenced by microscopic features like micro-structure, size and shape. In order to enhance or control properties of aggregates and be able to predict the structure of final products, it is essential to establish a link between the product properties and the operating conditions.

Objectives

The morphological evaluation of the micro-structure of soft agglomerates made of maltodextrin particles will be studied, in order to correlate process parameters with the properties of real products. To this purpose, already developed morphological descriptors will be modified to incorporate the overlap and deformation of primary particles.

Cooperation

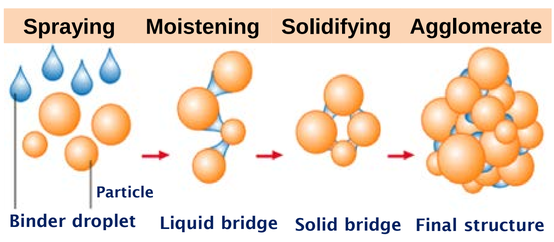
- Dr.-Ing. Peter Müller: particle size and shape measurements, preparation of binder solutions, SEM, DEM
- M.Sc. Maryam Dadkhah: operation of spray fluidized bed agglomerator and X-ray micro tomograph, image analysis

Experimental Setup

Stepwise evaluation procedure:

1. Fluidized bed agglomeration

In spray fluidized bed, primary particles are fluidized and a binder solution or suspension is sprayed onto the fluidized particles, creating liquid bridges which form agglomerates. As soon as a desired size of agglomerates is achieved, spraying is stopped and further evaporation of liquid may lead to the solidification of binders at contact points between primary particles (Figure 1).



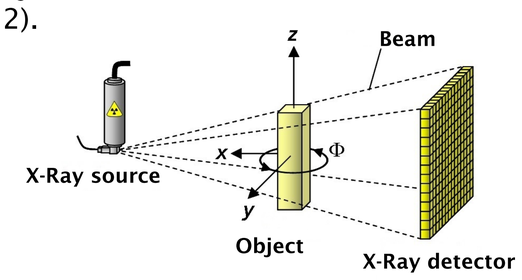
From Glatt Co., Weimar

2. X-ray µ-computed tomography

The µ-CT generates a 3D images of an object from a series of 2D X-ray images (Figure 2).

Scanning parameters:

- Exposure time
- Rotation step
- Frame averaging
- Voltage, current
- Acquisition time



3. Image processing algorithm

- Cropping of gray-level images
- Binarization of gray-level images (distinguish between solid particles and binder bridges)
- Filtration of binarized images (noise elimination)
- Segmentation, using pre-flooded watershed (particle center coordinates)

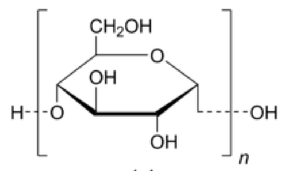
Figure 1. Spray fluidized bed

Figure 2. X-ray µ-computed tomography

Materials

Maltodextrin

$C_{6n}H_{(10n+2)}O_{(5n+1)}$



α-1,4

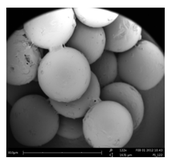
$2 < n < 20$

The material that will be used in this study for agglomerate production is maltodextrin (MD). MD is produced from starch by partial hydrolysis and is usually found as a white hygroscopic spray-dried powder. Maltodextrins are classified by DE (dextrose equivalent) and have a DE between 3 to 20.

Material	Bulk density (gr/cm^3)	Glass transition. tem. (°C)	Water solubility	Sorption behavior
Glass bead	1.5-1.6	520-650	insoluble	non-hygroscopic
Maltodextrin (DE:10-15)	0.30-0.50	100-150	soluble	hygroscopic

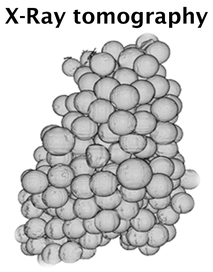
Hard agglomerate
(made of glass beads)

SEM

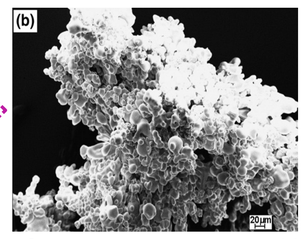


Ref. M. Dadkhah

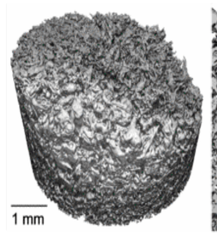
X-Ray tomography

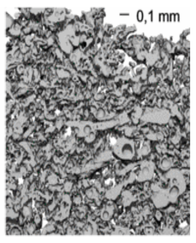


Soft agglomerate
(made of maltodextrin)



Ref. P.C. Martins, T.G. Kieckbusch





Ref. L.Fries, M. Heine

The major structural differences between hard and soft agglomerates:

- ✓ Sphericity
- ✓ Void space geometry
- ✓ Particle deformation
- ✓ Overlap between particles

Ongoing Work

The following **morphological descriptors** shall be developed to characterize the structure of maltodextrin agglomerates produced by a spray fluidized bed:

- ✓ Number of primary particle
- ✓ Radius of gyration
- ✓ Porosity
- ✓ Fractal dimension
- ✓ Pre-factor (structural coefficient)
- ✓ Coordination number
- ✓ Distribution of angles among particle neighbors

Conclusions

A structural characterization tool based on X-ray images shall be provided to analyze the microstructure of maltodextrin agglomerates produced in a spray fluidized bed. By this tool, process parameters may be identified to produce agglomerates with desired end-user qualities.
To this goal, the already available image processing algorithms as well as the morphological descriptors will be modified and extended to soft agglomerates.

DFG-Graduate School 1554 “Micro-Macro-Interactions in Structured Media and Particle Systems”