Morphological Characterization of Spray Fluidized bed Agglomerates by X-ray Tomography

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Introduction. Agglomeration occurs when a wet particle collides with another particle and gets bound with it by a liquid bridge which solidifies during drying. Although spray fluidized bed agglomeration is widely used, industry is still confronted with problems of poor product quality control. Experimental investigations have been conducted on agglomerates produced in spray fluidized beds using different primary materials (non-porous glass, porous ceramic) and HPMC (hydroxyl-propyl-methyl-cellulose) as the binder, under varying operating conditions, inlet gas temperature and binder mass fraction. X-ray µ-computed tomography, µ-CT, was utilized to evaluate the three dimensional micro-structure of the agglomerates and spatial distribution of each constituent primary particle in the aggregate. The measurements performed at different process conditions could be used to develop correlations of macroscopic properties with micro-structural features of the agglomerates. In this way, formation of agglomerates with desired morphologies under controlled conditions can be achieved.

Problem Definition

Agglomeration is referred to be more an art than a science, since process parameters selection is still mostly empirical. To enhance and control properties of aggregates and be able to predict product properties, it is essential to establish a link between the product properties and the operating conditions.

Objectives

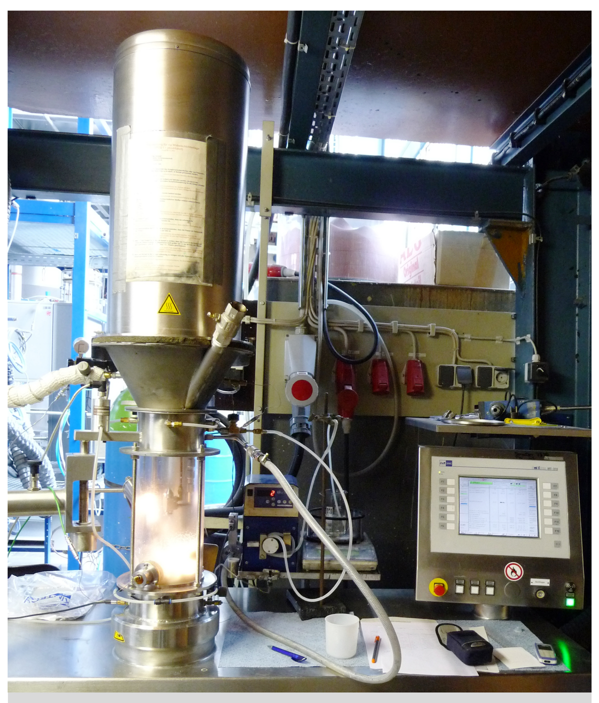
Aim of this study is to analyze the morphology of agglomerates experimentally and find correlation between primary material properties, process parameters, and resulting product properties, enhancing the existing process in terms of control and prediction of the final product properties.

Cooperation

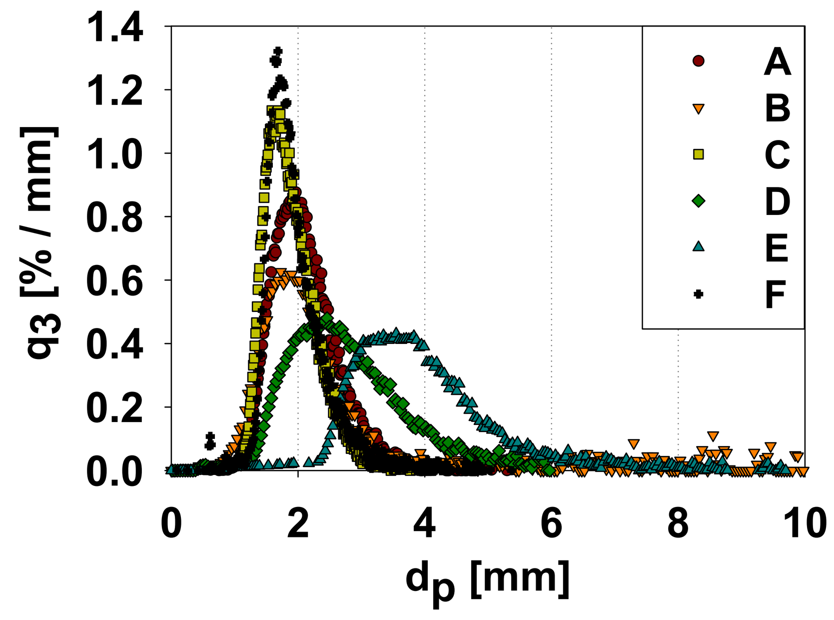
- Dr.-Ing. Peter Müller granule center coordinates
- M.Sc. Yujing Wang image processing
- M.Sc. Nicole Vorhauer pore network drying

Experimental Setup

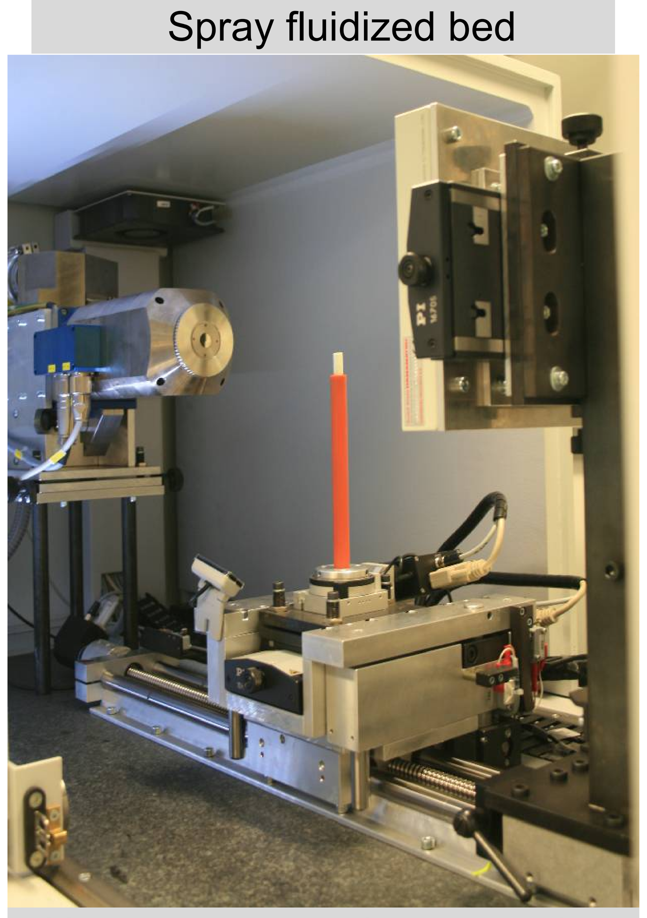
The dependence of the internal morphology of agglomerates produced in a spray fluidized bed dryer on the operating conditions (inlet gas temperature and binder mass fraction) was investigated by means of X-ray µ-CT. Six experiments were carried out, denoted by A to F. All the experiments were performed with the same total amount of sprayed solid binder, namely 0.5% of the total mass of dry primary particles.



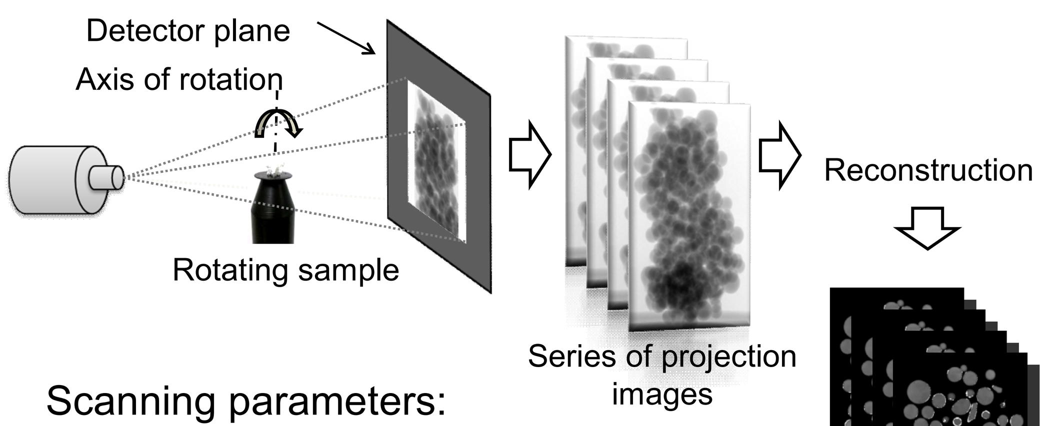
Spray fluidized bed



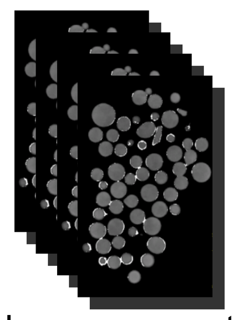
	Particles	Binder mass fraction, X_b	$T_{g,in}$
A	Glass	2%	60 [°C]
B	Glass	2%	30 [°C]
C	Glass	2%	90 [°C]
D	Glass	6%	60 [°C]
E	Glass	10%	60 [°C]
F	ɣ-Al_2O_3	2%	60 [°C]



X-ray µ-computed tomography

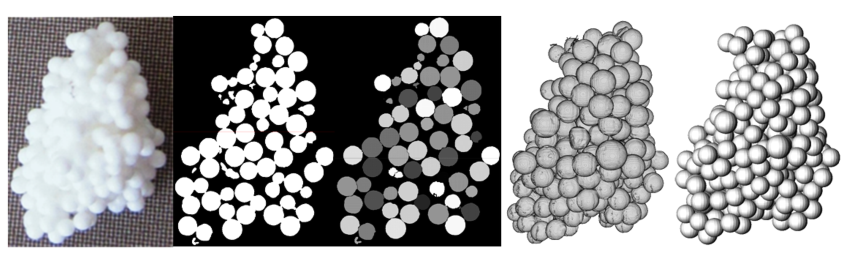


Scanning parameters:
- Exposure time 2 s
- Rotation step 0.45°
- Frame averaging 3
- Voltage 45 kV, current 170 µA
- Acquisition time 1.5 h



3D Image represent as series of 2D slices

Sequence of image processing involves:
- Extraction of the region of interest
- Removal of measurement noise
- Image binarization
- Separation of constituents

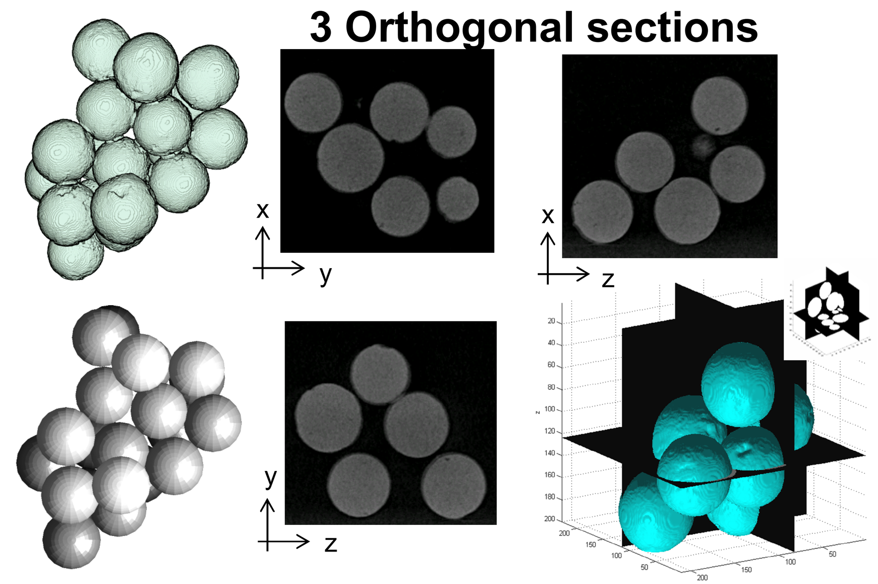


For each aggregate a matrix is created, providing the number of primary particles per agglomerate and storing the center coordinates, the radius and the volume of each particle.

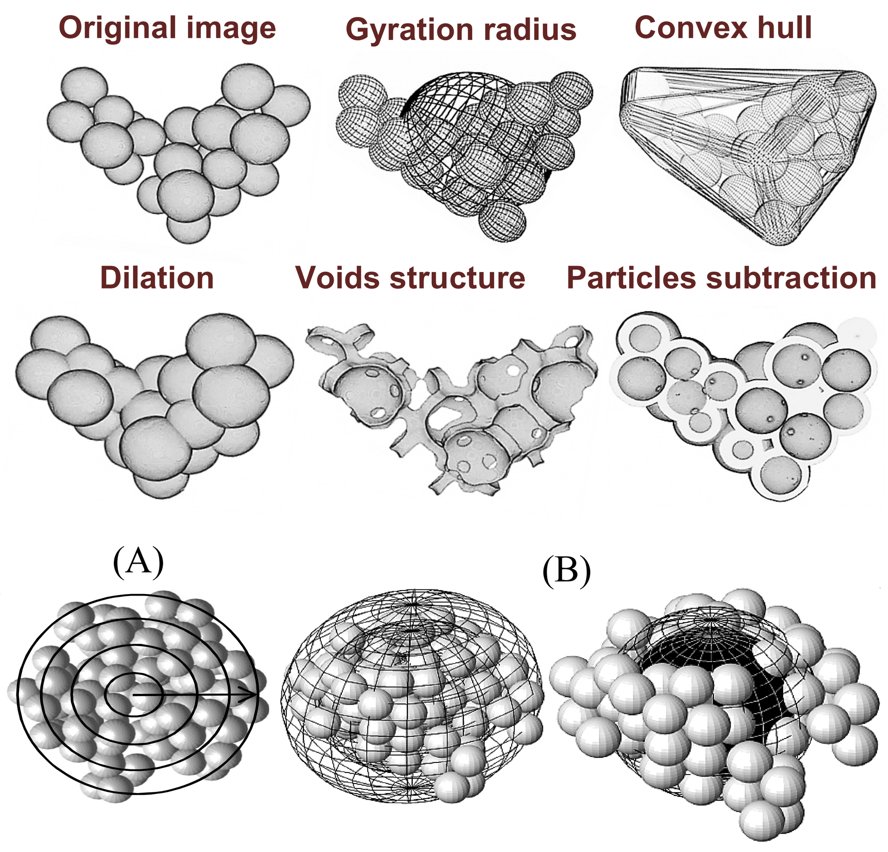
Analytical Description

Morphological descriptors
- Number of primary particles
- Radius of gyration
- Aggregate porosity (gyration radius, convex hull, and dilation)
- Coordination number
- Fractal dimension, pre-factor
- Binder characterization
- Void characterization
- Particle radial distribution
- Porosity radial distribution
- Coordination angle

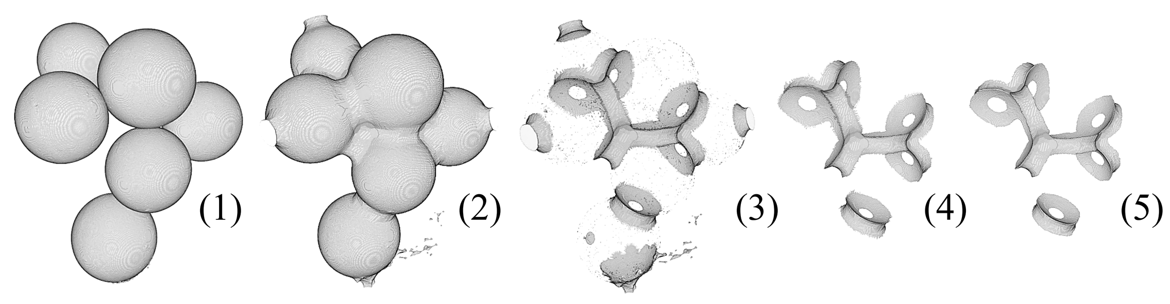
3 Orthogonal sections



Distributions of particles are evaluated by 2 different approaches:
(A) A sphere with radius R starts growing with the thickness ΔR, and in each step the number of centers included in the sphere is calculated.
(B) Two spheres are defined, one sited inside the other one. In this case all centers located in the free volume between the two spheres were calculated for each step. The reference point of both methods is the gravity center of the aggregate.

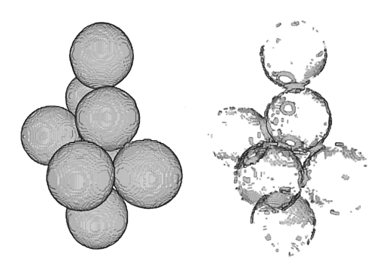


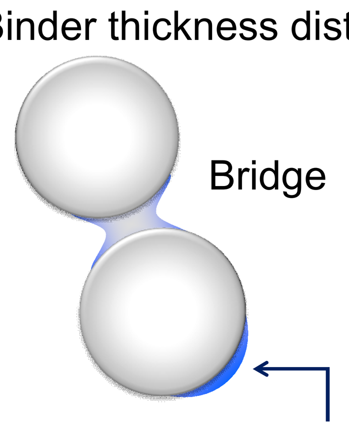
Void characterization steps



1. Volume image
2. Closing
3. Subtract particles
4. Objects filtering
5. Noise removal

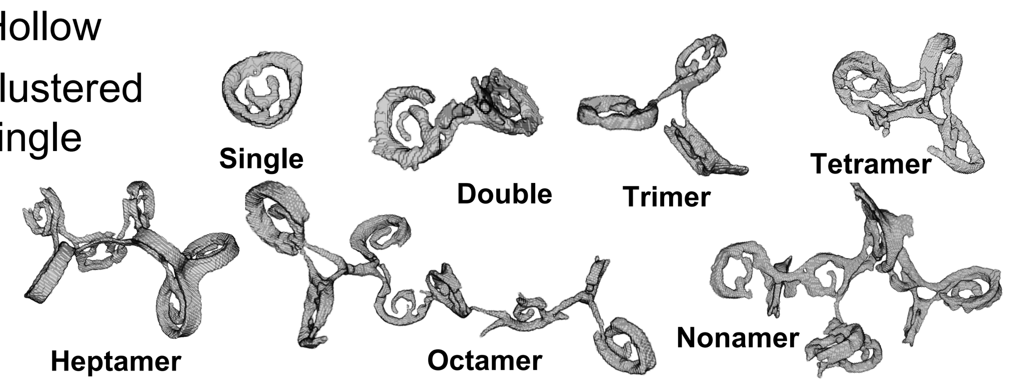
- ✓ Local distribution of droplets on particle surface
- ✓ Porosity for individual bridges
- ✓ Binder thickness distribution
  - Spherical granolometry
  - Volume of each bridge
  - Discrete directions





- Full
- Hollow
- Clustered
- Single

Unsuccessfully dried sessile droplets or collapsed bridges



Results and Discussion

- Increase in gas inlet temperature, $T_{g,in}$, produces more dense structure and is accompanied by an increase in the fractal dimension, D_f, and the mean coordination number.
- Increase in the sticking capability of the spray solution improves the growth rate of particles and decreases their D_f and MCN.
- Coordination angle is found to be rather independent of agglomerate size, $T_{g,in}$ and binder viscosity.

Experiment	A	B	C	D	E	F
$T_{g,in}$ [°C]	60	30	90	60	60	60
X_b [%]	2	2	2	6	10	2
Growth rate	1.07	1.15	0.63	3.79	8.20	0.60
D_f	2.44	2.31	2.94	2.28	2.09	2.45
K_g	1.77	2.01	0.98	1.86	2.24	1.60
MCN	3.32	3.10	4.02	2.92	2.87	3.16
Porosity	0.57	0.62	0.53	0.58	0.63	0.62

Conclusions

Results show that the operating conditions of the production process have a significant influence on the internal morphology of the produced agglomerates:

$T_{g,in}$ ↑
- Growth rate ↓
- D_f ↑
- MCN ↑
- Porosity ↓

X_b ↑
- Growth rate ↑
- D_f ↓
- MCN ↓
- Porosity ↑

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