



Detailed insight into microscopic phenomena using 3D-tomography data to develop a better model for dead-end filtration

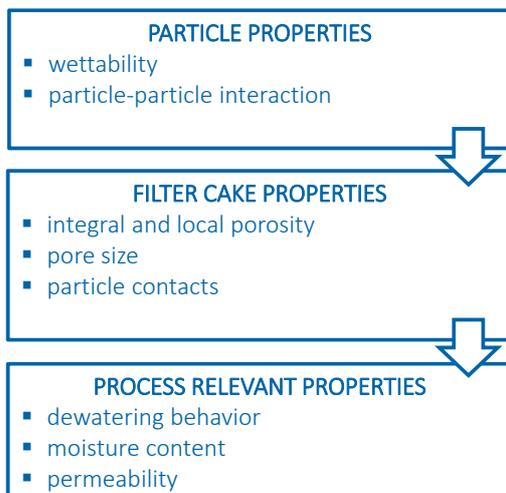
E. Löwer, M. Brockmann, Dr. Th. Leißner, Prof. Dr.-Ing. Urs A. Peuker

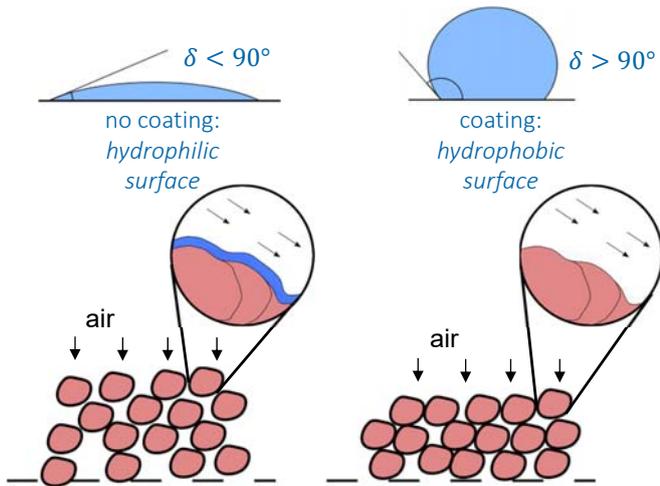


Objectives of the project



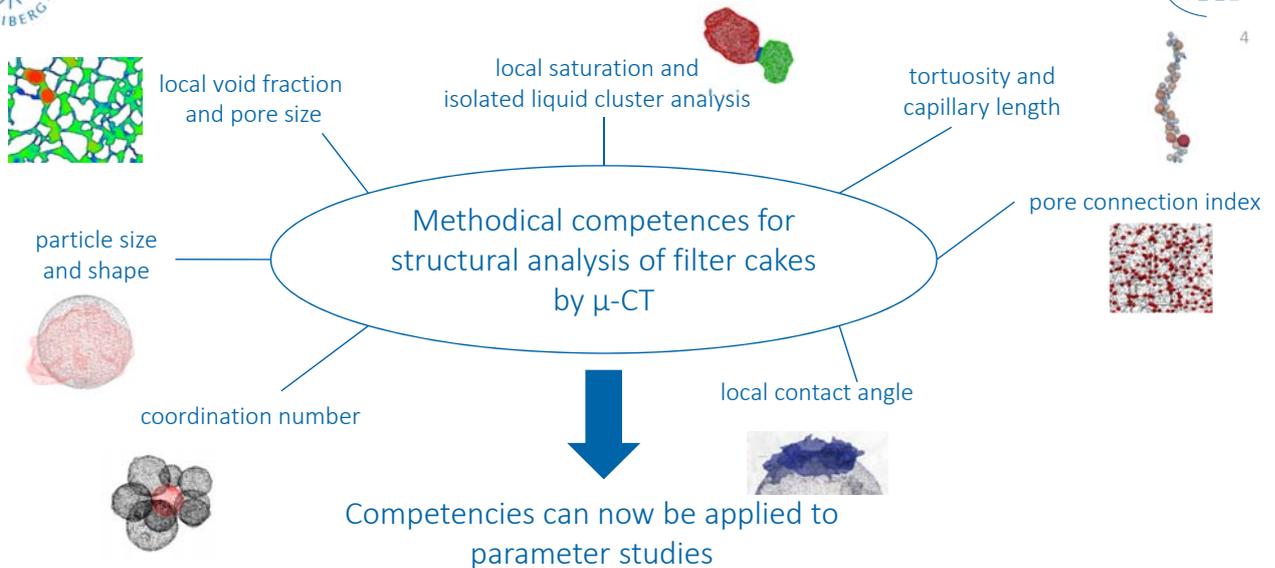
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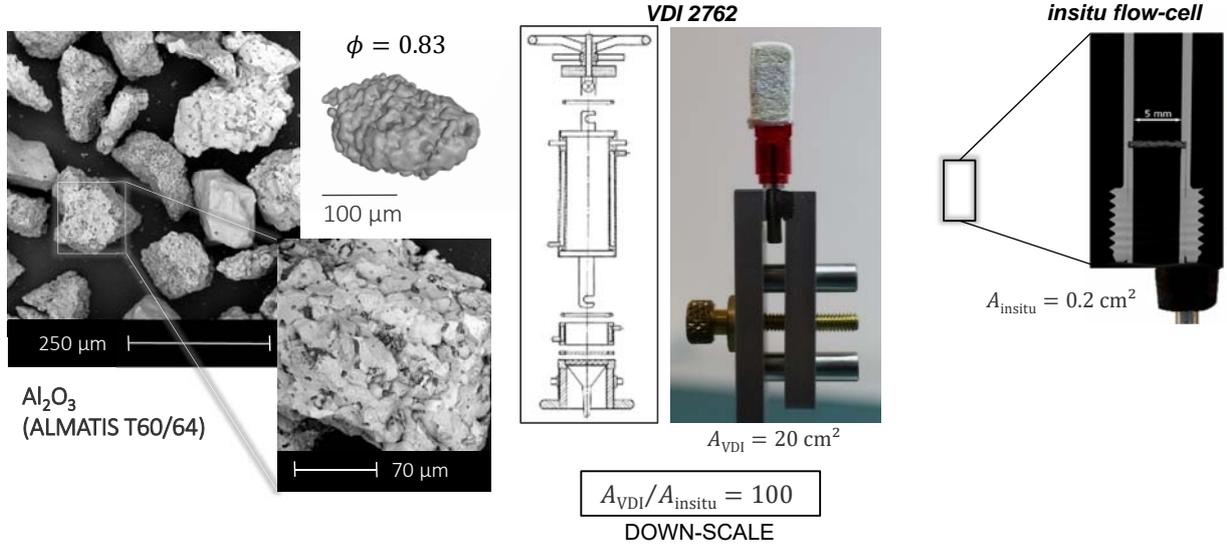




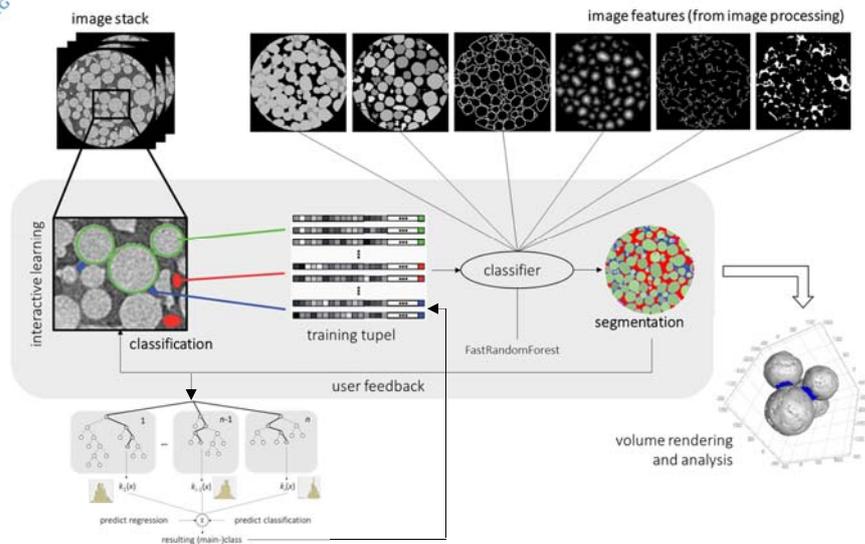
The wetting influences both the particle-particle interactions and the de-watering behavior of a porous network. At poor wetting we observe:

- Gas-bridges (capillary forces) increase the attractive forces between particles
- Liquid repels from solid surface





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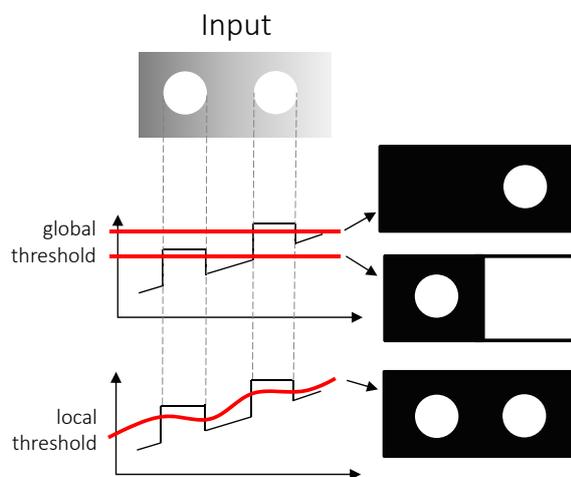


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Methods I – determining parameters form the two phase system (solid-liquid)

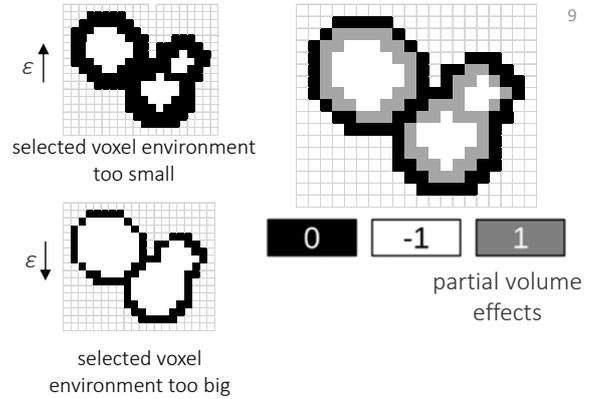
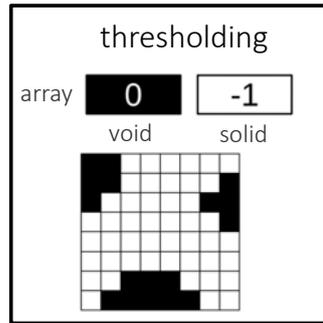
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Identification & quantification of the porosity

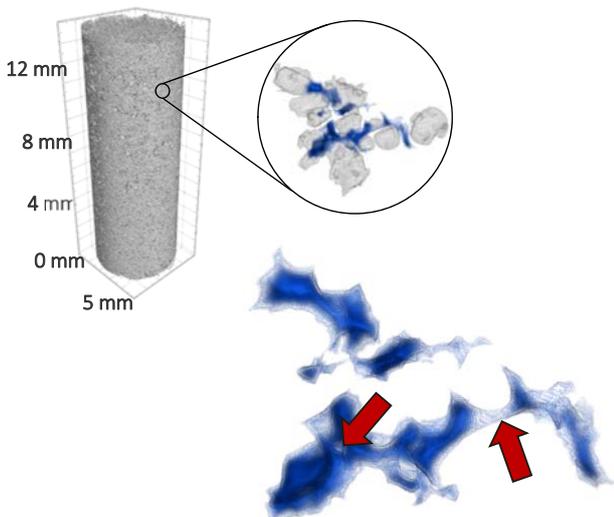


partial volume correction
depending on

- particle size and shape
- x-ray attenuation
- resolution

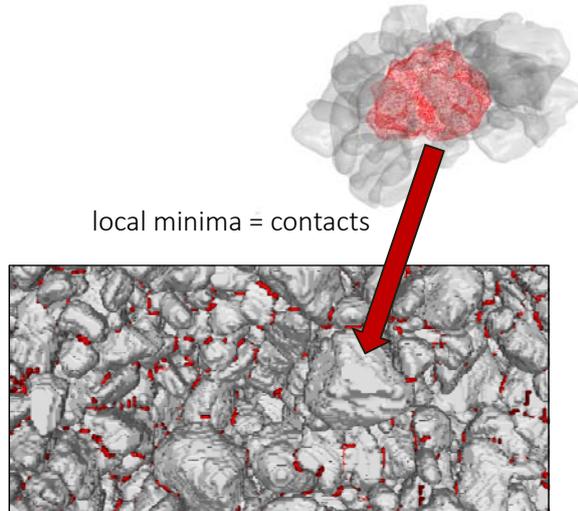


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- Resolution of the measurements is not able to correlate several voxels to the particle-particle contact.
- The alternative definition of a contact point uses the distance map between the particles.
- The minimum of distance is defined (threshold) as contact point.

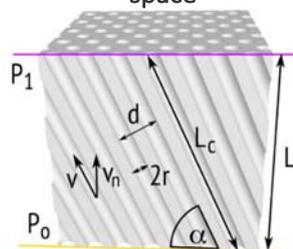
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- The visualization of a single irregularly shaped particle shows how it is embedded into the filter cake.
- The red marked positions are the contacts between the individual particles according to the definition (minimum of the distance map).

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N identical capillaries with the same alignment in space



P_i – pressure
 L_c – capillary length
 α – alignment angle

pressure loss can be determined using the laws of classic fluid dynamics

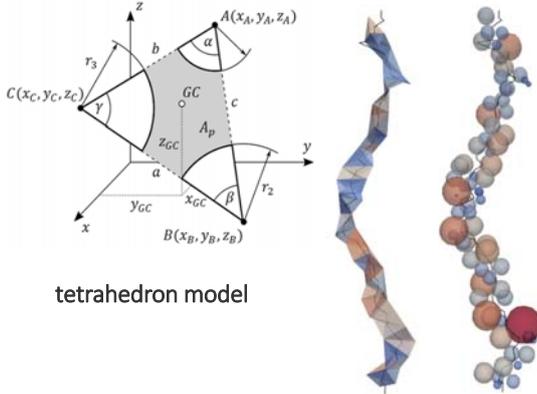
BUT

disregarding:

- capillary crosslinking
- inlet effects
- changes in cross sectional area
- surface wettability
- Always assumption:
 $S_V = \text{const.}$
 $\epsilon = \text{const.}$
 $r_C = \text{const.}$

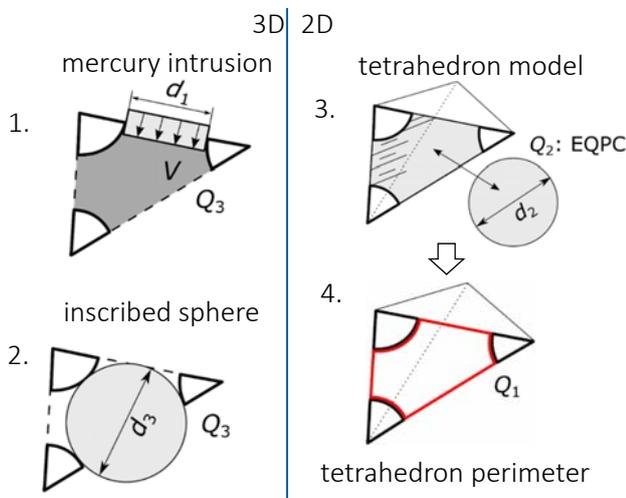
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capillary length L_c



tetrahedron model

The capillary length, i.e. the tortuosity, depend on the individual way we are walking through the filter cake. This length also differs with the starting point at the filter cake surface. We defined the capillary length, by connecting the balance points of the triangles, which have been constructed between the particle centers.



To apply the LAPLACE equation

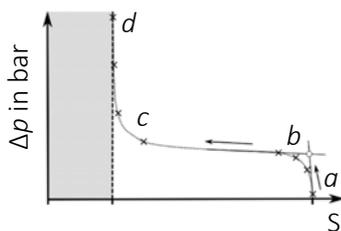
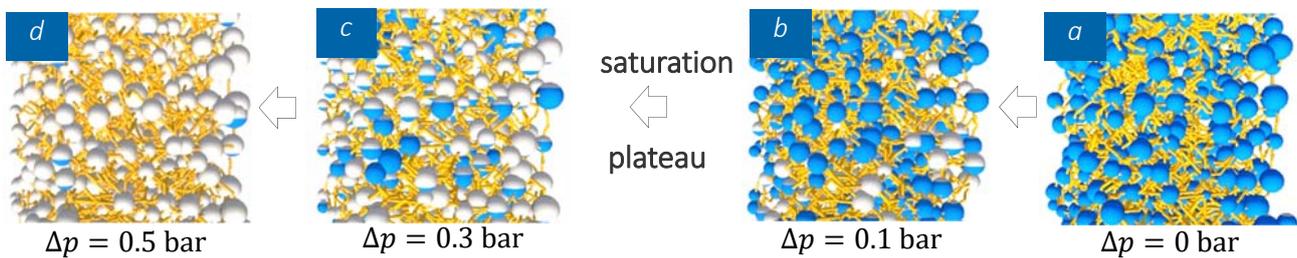
$$\Delta p = \frac{U}{A} \cdot \gamma_l \cdot \cos \delta$$

we need a detailed view into the local geometry of the pore. There are different options to quantify the required geometric parameters. U reflects length of the three phase contact line.

Methods II – determining parameters form the three phase system (solid-liquid-gas)

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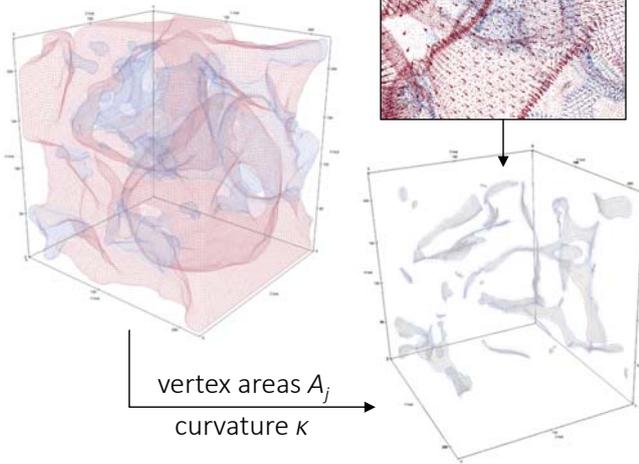
Determination of the liquid saturation (balancing)



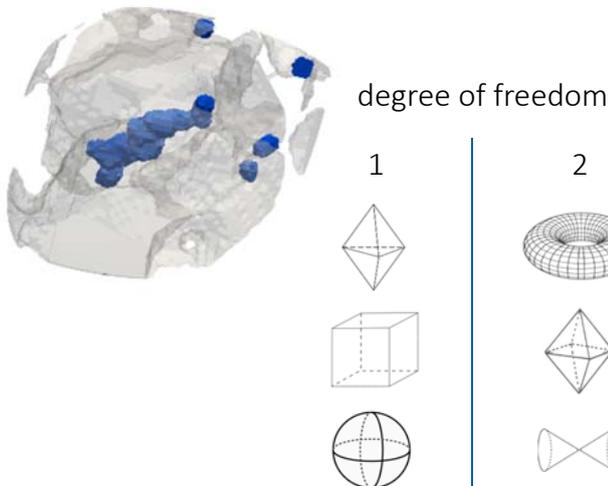
$$S = \frac{V_l}{V_{void}} = \frac{V_l}{V_{cake} - V_s}$$

SINGLE “LIQUID BRIDGE”

surface vector map



The three phase contact line of the liquid bridges is far below the resolution of the CT-measurements. Therefore, the wetting angle as been extrapolated by using a vectoral approach using the scalar product of the perpendicular vectors on each plane. Due to the local curvature to the liquid we measure a wetting angle distribution.



The tomograms show the challenges in the definition of the characteristic size of the pore liquid. The remaining liquid has quite irregular elongated shape, thus we are far from defined liquid bridges, which are common in the 2D-models.



Validation

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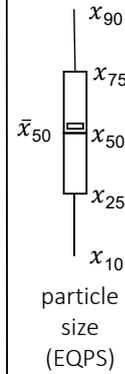
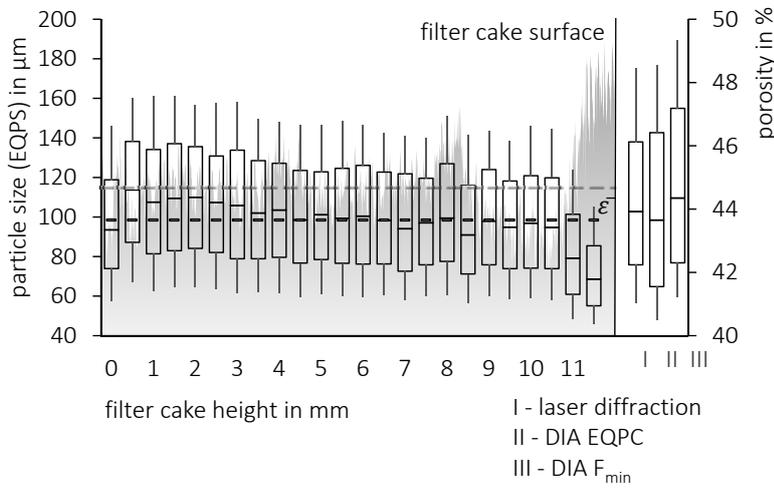


Validation

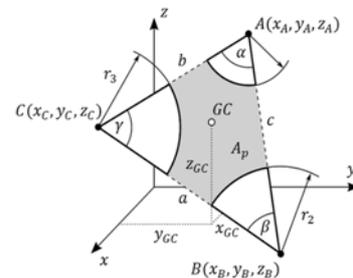
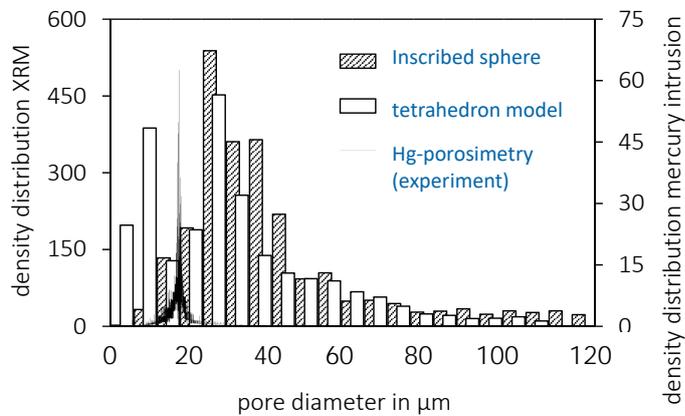


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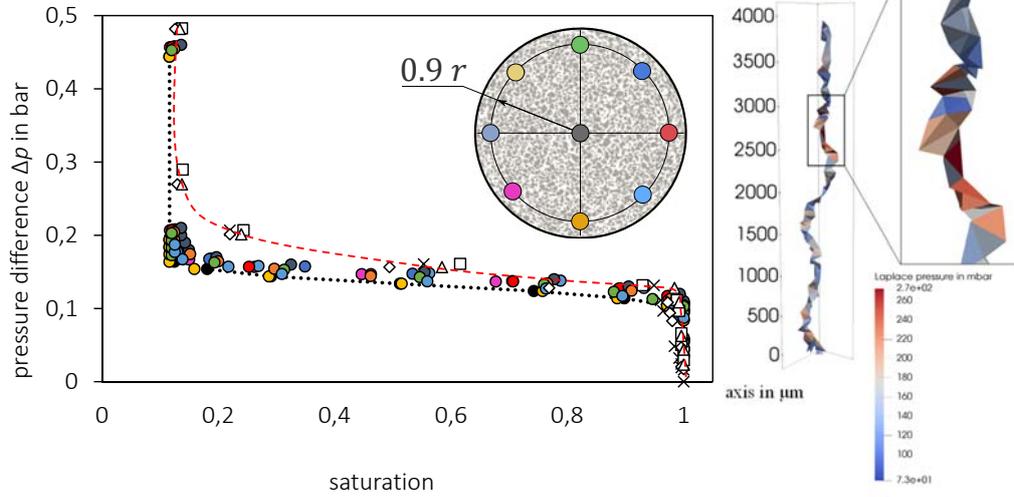
CONCERN	VALIDATION METHOD
structure effects	<ul style="list-style-type: none">local particle sizelocal porosity
particle morphology	<ul style="list-style-type: none">2D and 3D shape parameters
pore size discription	<ul style="list-style-type: none">mercury intrusioninscribed sphere modelpore wetting line
local contact angle	<ul style="list-style-type: none">static and dynamic sessile drop
capillary distribution	<ul style="list-style-type: none">perculating sphere modellaboratory pressure tests



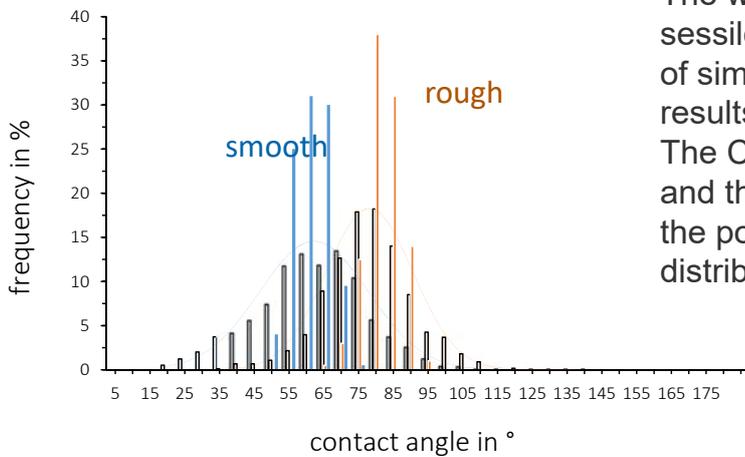
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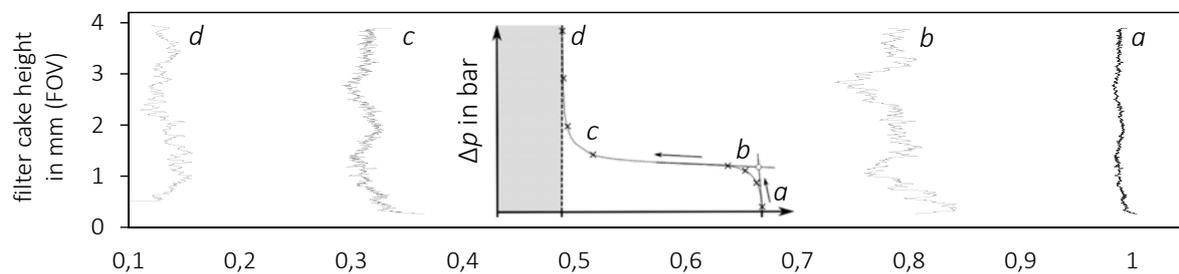
The wetting angle measured with the sessile drop method on a planar surface of similar roughness, fits to the CT-results, especially with the modal values. The CT-results allow a better resolution and therefore and due to the curvature of the pore geometry they have wider distribution.

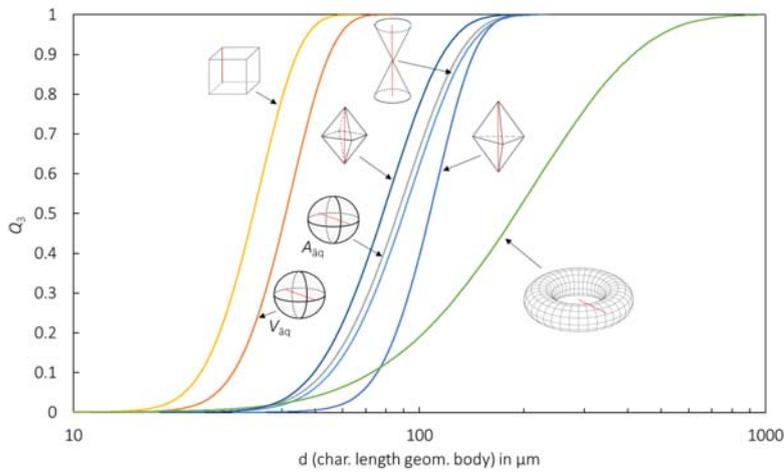
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Results – what we can further learn form CT-data?

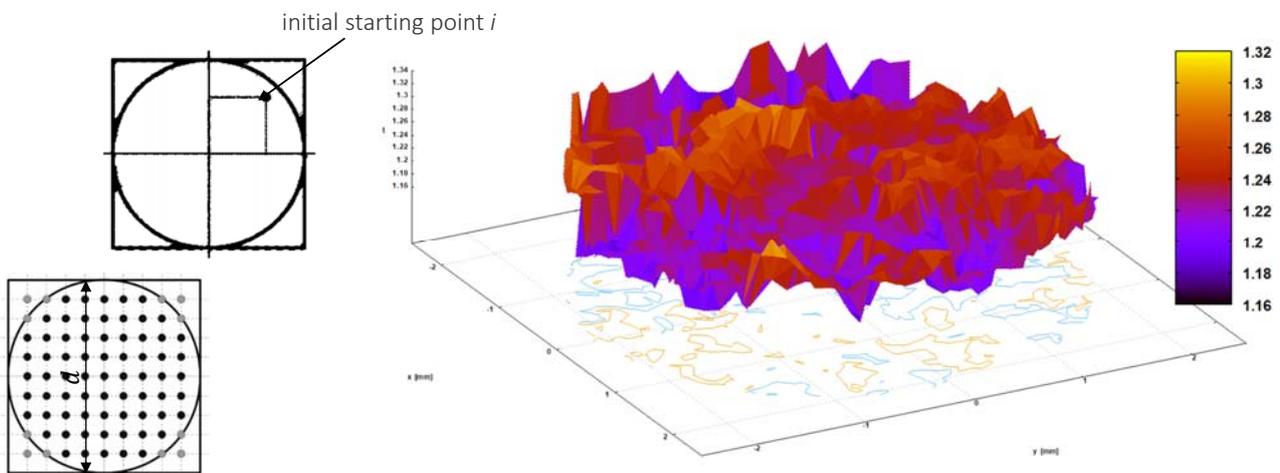
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Saturation – liquid distribution over the cake height.

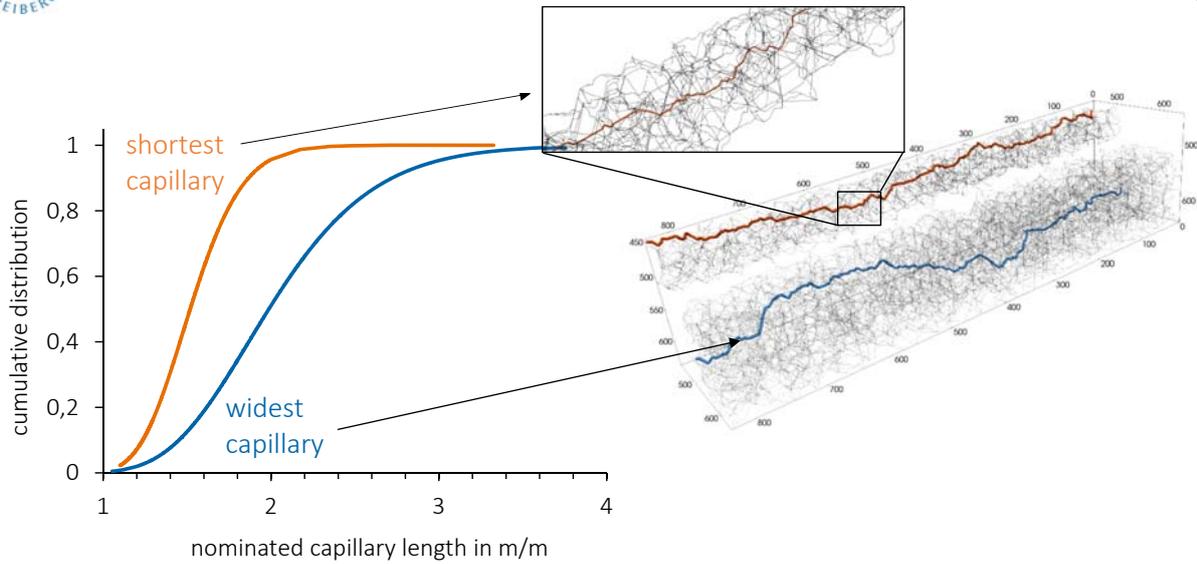




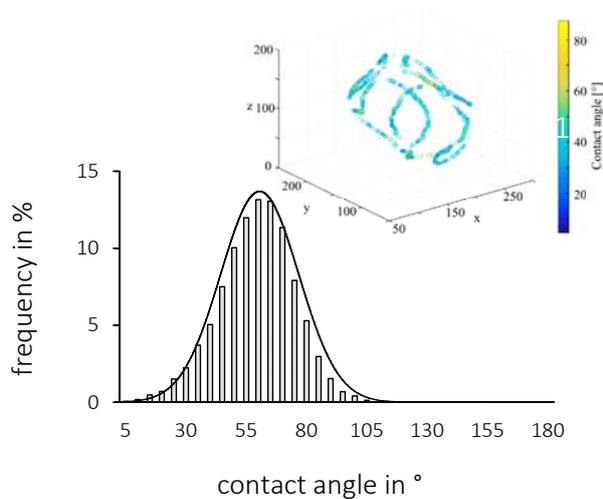
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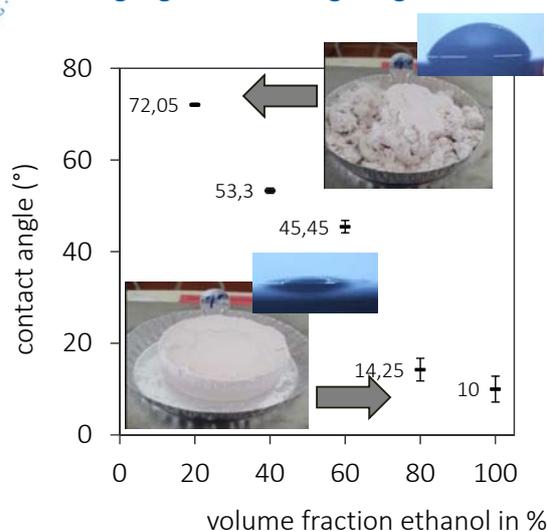
The contact angle on real (rough) curved particle surfaces shows a large variation, which is due to the complex geometry of the pore system formed by the particles.

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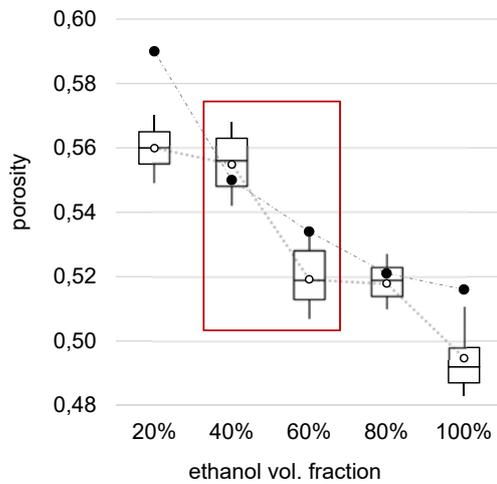
Results – what we can further learn form CT-data concerning wetting effects.

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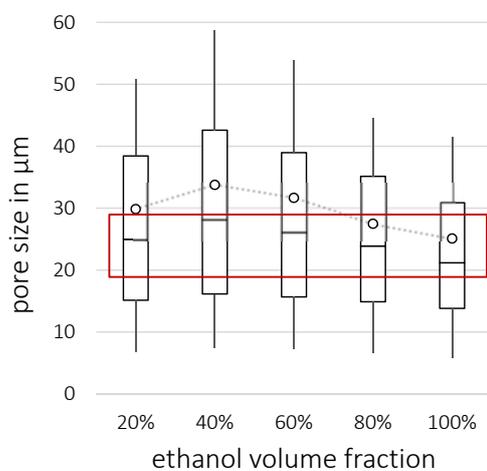
Changing the wetting angle



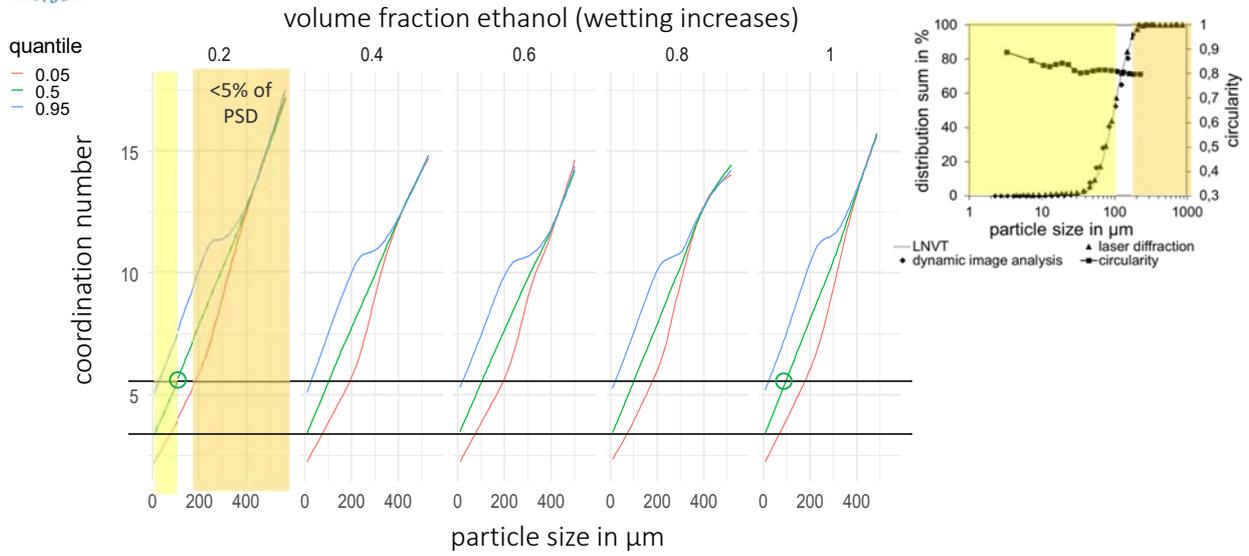
From the macroscopic point of view we see significant changes in the tensile stress and the flowability of a filter cake, when the wetting between mother liquid and solid changes. (The more ethanol is in the liquid phase the better the wetting becomes.)



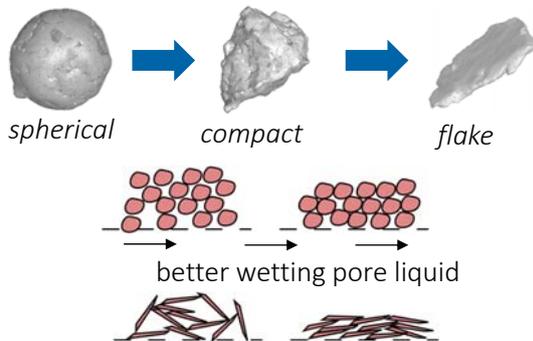
The effect of the wetting also can be seen in the structuring of the cake itself. The integral porosity decreases by about 6 %-points. The fluctuations within the porosity are larger in the range between 40-60 % of ethanol, where the largest change in porosity occurs.



The pore size (using the inscribed sphere model) does not change much as a function of the wetting properties. It just can be stated that the span on the extreme values increases at 40 and 60 % of ethanol. The median (-) of the PoreSD decreases with the higher packing, with the lower porosity, respectively.

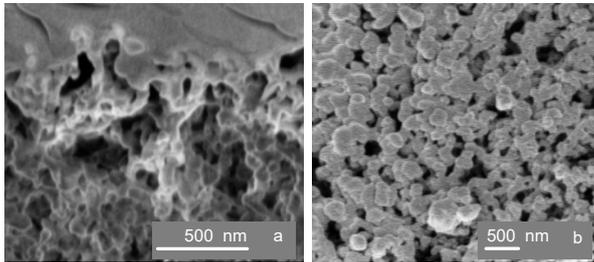


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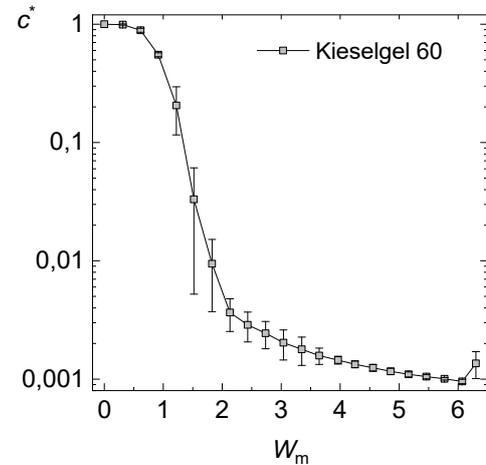


Having now in place a reliable data processing strategy, which bases on self-coded software, we be able to finally address strongly non-ideal particles and their structuring as a function of wetting.

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FIB-Analysis of filter cake made of porous particles



Washing curve for porous product (pore width 6 nm)

Seupel, S 2020 (paper submitted to WFC)

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On strategy concerning second period:

- Centrifugal de-watering / sediment formation (sedimentation)
- Centrifugal de-watering (de-watering / Bond-diagram)
- Filter cake washing and structural effects

Multi-Scale structural analysis of pore networks, e.g. combination of FIB and μ -CT