FEEDBACK to IFPRI General Meeting questions

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- **How small can microCT resolve?**

Theoretically, voxel sizes of up to 300 nm can be achieved with sufficiently small samples. Thus, spherical particles from 3 µm size can be imaged. If the shape of the particles deviates more from that of a sphere, more voxels per particle are required. When using the Insitu filtration cell with a diameter of 5 mm and thus 5 mm radiated length, spherical particles from **20 µm** can be imaged sufficiently well. For smaller particles there is only the possibility to reduce the size of the filtration cell or to switch to sampling of filter cakes formed in the laboratory. This would make smaller samples and better resolutions possible.

If, however, voids or liquid bridges should also be imaged, their characteristic lengths should not be less than 20 µm in the Insitu cell. Since their size is usually well below the particle size, all phases can only be imaged satisfactorily from spherical particle sizes of **40 to 50 µm**.

- **Do you think this method can be applied to ensembles of smaller particles?**

Smaller particles in filter cakes can only be measured by switching to smaller sample sizes. This is currently not compatible with the current In-situ filtration cell and only partially feasible with the XRM-tomography used.

For the investigation of particle below 20 µm having liquid bridges at about below 4 µm we would need a different setup. Either we have to use a synchrotron or a high resolution XRM. For those machines we will have to develop a entirely new sample preparation and validation method. Furthermore, it takes time and costs to get access to this type of equipment, which goes beyond an IFPRI project. Nevertheless, we will develop our fundamental research (independently from IFPRI) into this direction. Another option is the application of HR-SEM-TEM combinations with FIB sampling, which locally provide access to the cake structure. This method does not provide any real 3D-information and would need a lot of assumptions and validation efforts to come to reliable pore data.

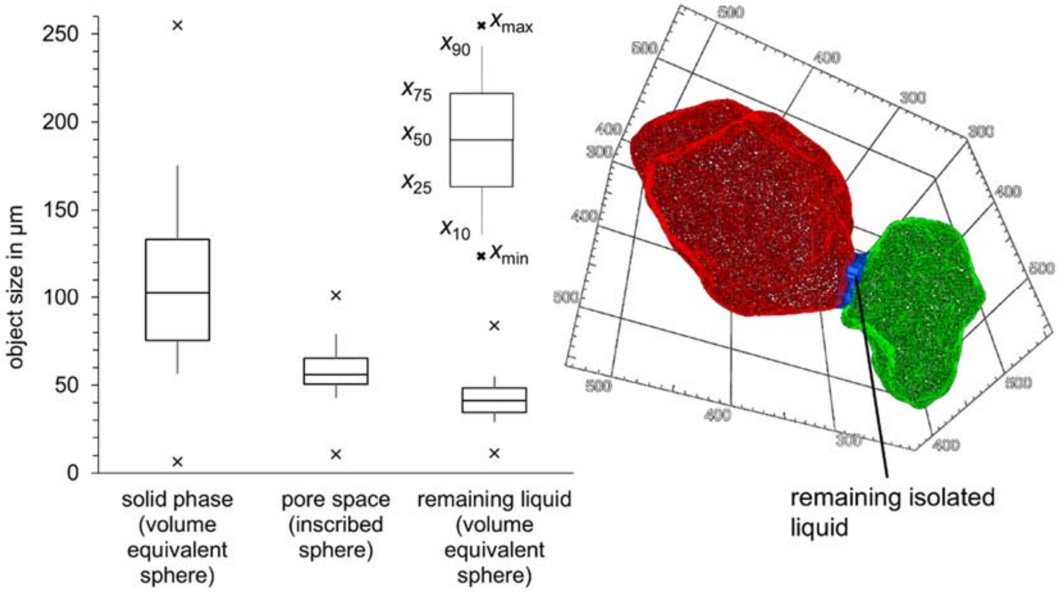

Fig. Size distribution and relation of particle, characteristic pores and liquid bridges.

- **How do ions effect the wettability of the alumina surface such as sulfates and chlorides? Or cations?**

The sintered alumina particles have already undergone a heat treatment of 1600 °C and are very stable against external influences. Since the particles are washed both before the coating process and afterwards, there is hardly any contamination or remaining silanes on the particle surface. At the natural pH value of 9.5 these particles in aqueous systems and at room temperature it can be assumed that no special interaction processes of the surface with chlorides take place. In the case of sulphides, the pH value will be decisive for a possible change in the contact angle of the solution. Under the given filtration conditions this is extremely unlikely. However, no tests have been carried out to this effect.

In further investigations we have resolved the interaction of hydrophobic and DLVO-interaction effects. It can be stated that both effects can be superimposed. The particle surface has to be considered a s layered system of silane coating and bulk material, since the coating is too thin to shield the van-der-Waals interaction of the bulk aluminum oxide. With this mixed Hamaker constant (combination rules) we are able to see the influence of coating on van-der-Waals interactions. The electrostatic (repulsive) interactions can use a typical approach describing the Debye-length to quantify their contributions. The hydrophobic effects, e.g. nano-bubbles can be implemented using capillary forces and they also have to be considered in the calculation of the Hamaker-constant using the layer model with an air layer.

To conclude, yes, the role of ions can be investigated as well in this context, the principal influence on the electrostatic interactions remains unchanged even in the hydrophobic system.

- **How does temperature play a role in these systems?**

Concerning the thermal stability of the materials, we can state: At temperatures above 120 °C the silane coating is no longer stable. If the material is exposed to temperatures of 200 °C for a longer period of time, the coating process can even be completely reversed.

Concerning processing effects, we can state: Moderate temperature increases above room temperature were not investigated yet in the filtration experiments, so we cannot make any statements about the change in wetting behavior. If requested we can use in a first step the sedimentation measurements (see below) we are planning to reflect the temperature influence on suspension stability in the window 10 – 70 °C.

- **The SEM shows the sintered Alumina particles having a rather rough surface. To which reference plane is the modelled contact angle defined?**

This is a critical question. The tomograms will definitely not resolve the entire surface geometry, since the characteristic length is at or even below the image resolution. We can say that we are measuring the “effective” wetting angle, which incorporates both the material and the geometrical properties of the particle surface. Since we are measuring the 3D-case, i.e. the wetting angle at every point of the three-phase line, we get a wetting angle distribution, which reflects all local properties. This shows us again, that in the future we have to calculate using different distribution functions for our process parameters instead of defined values.

The contact angle is obtained directly from the tomograms. For this purpose, the three-phase line is determined from the data sets and the vectors at this line of the surface normal of solid and liquid are calculated. The scalar product of both vectors at the same vertex of the polygon mesh gives the local contact angle. For reference measurements, both sessile drops on planar surfaces were measured in the laboratory using contour analysis, as well as in µCT. For the reference surfaces a plate sintered from Alumina was used.

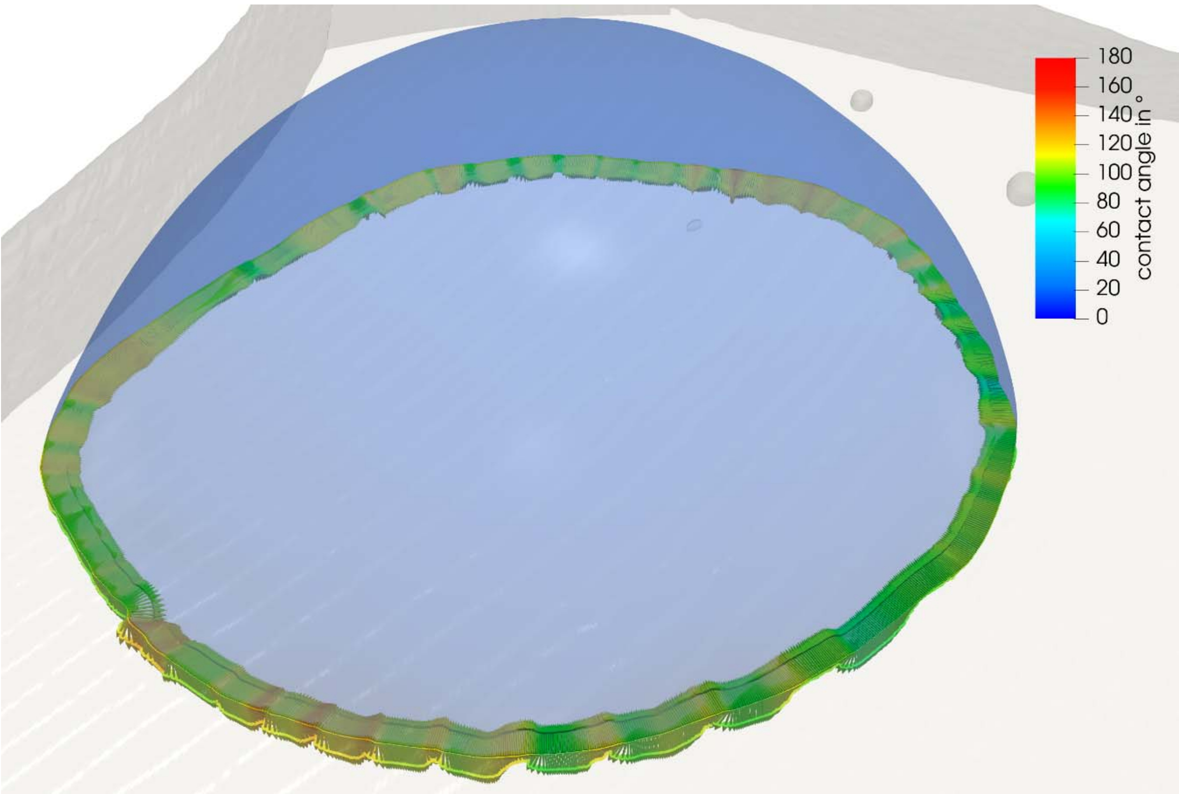

Fig.: Local effective contact angle at the 3D-contact line of the drop.

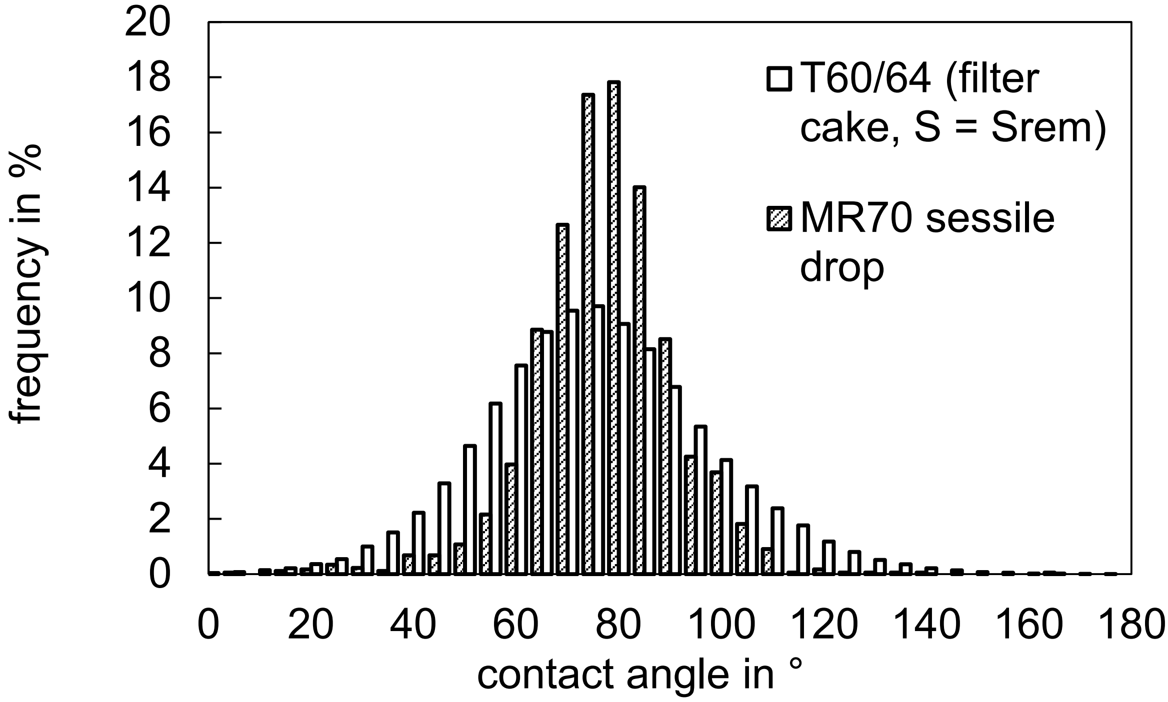

Fig.: resulting contact angle distribution along the three phase wetting line, incorporating both material and structural (roughness) effects.

- **The modelled capillary pressure curve appears to be a static representation of the moisture distribution. How does this compare with the equilibrium moisture in a dynamic dewatering process at constant pressure as it is typically done in the lab filter cell?**

By measuring on µCT we can only represent static states of the dewatering process. Due to the long measuring times the dynamic process, which in this case takes place in a few seconds, cannot be detected. In principle, however, we measure similar saturation curves in comparison to the laboratory values, since the same state of equilibrium is reached in every pressure level. The progression of the liquid front and the resulting receding angle cannot be measured. Here we can only display the liquid in the cake without the influence of external forces. Capillary forces, which may move the liquid into other pore areas and only occur due to the long measuring time, cannot be excluded. To get access to these transient parameters, we can only take the highly resolved inner geometry of the cake and implement a sophisticated de-watering model. The tomography provides high quality data for model validation.

To come to the desired time scales the resolution has to be decreased significantly, which at our point of view is not able to provide any addition insight compared to other methods, e.g. electrical tomography or 1-D X-ray adsorption using a line sensor.

- **The decreased porosity with better wettability did not quite correlate with pore size and coordination number. Have you observed a variation of the hydrophobic agglomeration status in the suspension (sedimentation rate, sediment volume) with the wetting behavior?**

We have not yet carried out any studies on the sedimentation behavior of the suspension, but it is a good point for future studies, which we will definitely investigate. In an other project we carried out hydrophobic agglomeration and we measured in-situ in a laser diffraction cell the transient particle size (0-20 µm and 0-2 µm particles). Here we clearly observed the hydrophobic agglomeration – the media of the PSD increased by the factor 2.5 to 3.5 in the defined shear field. The larger mass and inertia of the particles used here probably will lead to weaker agglomeration effects.

Sedimentation analysis will be more suitable, du to the settling effects compared to in-situ laser diffraction measurements