Neighborhood Relationships of Widely Distributed and Irregularly Shaped Particles in Partially Dewatered Filter Cakes

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Abstract

A more thorough understanding of the properties of bulk material structures in solid–liquid separation processes is essential to understand better and optimize industrially established processes, such as cake filtration, whose process outcome is mainly dependent on the properties of the bulk material structure. Here, changes of bulk properties like porosity and permeability can originate from local variations in particle size, especially for non-spherical particles. In this study, we mix self-similar fractions of crushed, irregularly shaped Al_2O_3 particles (20 to 90 μm and 55 to 300 μm) to bimodal distributions. These mixtures vary in volume fraction of fines (0, 20, 30, 40, 50, 60 and 100 vol.%). The self-similarity of both systems serves the improved parameter correlation in the case of multimodal distributed particle systems. We use nondestructive 3D X-ray microscopy to capture the filter cake microstructure directly after mechanical dewatering, whereby we give particular attention to packing structure and particle–particle relationships (porosity, coordination number, particle size and corresponding hydraulic isolated liquid areas). Our results reveal widely varying distributions of local porosity and particle contact points. An average coordination number (here 5.84 to 6.04) is no longer a sufficient measure to describe the significant bulk porosity variation (in our case, 40 and 49%). Therefore, the explanation of the correlation is provided on a discrete particle level. While individual particles $< 90 \mu\text{m}$ had only two or three contacts, others $> 100 \mu\text{m}$ took up to 25. Due to this higher local coordination number, the liquid load of corresponding particles (liquid volume/particle volume) after mechanical dewatering increases from 0.48 to 1.47.

Article Highlights

- **High-resolution X-ray tomography measurements grant insights into 3D filter cake structures on particle level**
- **Coordination number distribution is strongly dependent on local variation in particle size and porosity**

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- **Fine particles cause higher particle liquid load after dewatering, but smaller hydraulic isolated liquid areas**

Keywords Computed tomography · Cake filtration · Filter cake dewatering · Image processing · Microporous structure · Coordination number

Abbreviations

FOV	Field of view
PMMA	Polymethyl methacrylate
SNR	Signal-to-noise ratio
XRM	X-ray microscope

List of Symbols

A	Filter area (m^2)
c_v	Solid volume fraction related to the total suspension volume ($\text{m}^3 \text{m}^{-3}$)
$c_{v,F}$	Volume of fine particles related to the total solid volume ($\text{m}^3 \text{m}^{-3}$)
D	Distance (m)
d	Diameter (m)
h_{Cake}	Filter cake height (m)
k	Coordination number (–)
m	Filter cake mass (kg)
Δp	Pressure difference (Pa)
Q_3	Cumulative mass-related distribution (–)
q_3	Mass-related density distribution (1/m)
q_3^*	Normalized mass-related density distribution (–)
S	Saturation ($\text{m}^3 \text{m}^{-3}$)
S_{ire}	Irreducible saturation ($\text{m}^3 \text{m}^{-3}$)
V_L	Liquid volume (m^3)
V_S	Solid volume (m^3)
$V_{P,L}$	Liquid pore volume (m^3)
$V_{L,\text{dis}}$	Displaced liquid pore volume
V_P	Pore volume (m^3)
x	Particle diameter (m)
x_{ii}	Characteristic values of the cumulative particle size distribution (m)
x_m	Modal value (m)
ε	Porosity ($\text{m}^3 \text{m}^{-3}$)
η	Dynamic viscosity (Pas)
μ	Linear X-ray attenuation coefficient (m^{-1})
ρ	Density (kg m^{-3})

1 Introduction

A detailed understanding of permeability and capillary effects has been of particular interest in fundamental and applied research of mechanical separation techniques (Iglauer et al. 2013). Cake-forming filtration represents the basic principle of many industrial applications where liquids are to be separated from solids. In these processes, a feed stream

consisting of solid particles dispersed in a continuous fluid medium is passed through a semipermeable filter cloth. The filter medium allows the continuous phase to pass through almost unhindered, while at the beginning individual solid particles build up a rising structure on the filter cloth via blocking mechanisms, which increasingly retains the solids themselves. The filter cloth only initiates the build-up of the so-called filter cake. As the filter cake height increases, the continuous phase's pressure drop increases, which is why the filter cake must be removed when a certain height is reached. This pressure drop, which determines the process throughput, is determined by the bulk material properties. In contrast to the pressure drop, permeability is often cited in this context, which unites the bulk material properties, such as porosity, as a macroscopic quantity.

Most popular cake filtration models often assume homogenous cake structures with constant porosity and, therefore, globally estimated permeability (Tien and Ramarao 2013; Matsumura et al. 2015; Endo et al. 2009). Particle properties such as shape, size and wetting behavior are not only a single bulk property but distributed within the filter cake (Löwer et al. 2020b; Hwang et al. 2018; Perini et al. 2019). They significantly affect the porous network formation during cake filtration with possible local deviation in vertical (axial) or horizontal (radial) orientation. Since microscopic local parameters are inaccessible in conventional filtration experiments, cake structure models rely on average numbers or neglect some influencing factors, e.g., geometric particle–particle relationships, like particle contacts and corresponding contact area, thus simplifying process relevant phenomena within the filter cake significantly.

By combining both conventional macroscopic laboratory-scale experiments (nutshell filter) and X-ray microscopy (XRM) in situ experiments, we can connect particle properties with pore properties to gain a detailed insight into the filtration process. The resulting correlation of locally distributed and bulk parameters contributes to the development of suitable filtration models connecting micro and macro processes (Esser et al. 2021; Löwer et al. 2020a).

Particle–particle relationships provide information about bulk structures resulting in stable particle arrangements (Karamchandani et al. 2019). Microscopically, the coordination number as a measure for particle contacts depends on the packing density, represented by the corresponding porosity, and the shape and size of the neighbors.

Besides porosity, the coordination number is also related to other properties of a packing structure according to the application, such as tensile strength (Breuer and Khalifa 2019), thermal (Kovalev and Gusarov 2017; Gusarov and Kovalev 2009) and electrical conductivity (Anderson 1986), permeability (Rabbani and Babaei 2019) and hydrodynamic isolated pore liquid (Andersson et al. 2018).

As known from crystallography (Wan et al. 2018), the coordination numbers for an ideal regular cubic, orthorhombic, tetragonal–sphenoidal and rhombohedral packing are 6, 8, 10 and 12 (Cooke and Rowe 1999; Dullien 1975). The corresponding porosity values ε are 0.4764, 0.3954, 0.3019 and 0.2595, respectively. Randomly arranged particle packings in cake filtration rarely achieve these ideal structures. For a random packing of monodisperse spheres, the overall average coordination number k is ideally assumed to be 6 (Suzuki et al. 1981). In reality, random particle packings of monodisperse spheres have coordination numbers ranging from 5.5 for "loose packings" to about 6.5 for "tight packings" (German 2014). Further predictions of the characteristic properties of packing structures of non-uniformly sized particles in terms of porosity and coordination number were part of many investigations (Zou et al. 2003; Du Toit et al. 2009; Smith et al. 1929; Rumpf 1958; Yang et al. 2000; Zhang et al. 2001), summarized by (Van Antwerpen et al. 2010). Various authors also developed several theories to estimate coordination numbers for randomly

arranged binary mixtures of rigid spheres of uniform size (Suzuki and Oshima 1983; Bouvard and Lange 1991; Chen et al. 2009).

Figure 1 presents an overview of the coordination number predictions as a function of the bulk porosity. The named empirical and numerical studies focus on the relationship between the particles' packing properties and the controlling parameters. These parameters include the primary particle size (Langston and Kennedy 2014; Bertei and Nicolella 2011; Suzuki et al. 1999) and shape (Nam et al. 2019; Nan et al. 2019), the binding mechanism between particles (Cheng et al. 2000) and the type of external forces (Breuer and Khalifa 2019) applied. These available correlations from the literature mainly depend on a single bulk porosity, whereby the currently existing studies often neglect the local porosity distribution. The analytical model equations shown in Fig. 1 rely on the experimental/theoretical principles summarized in Table 1.

Additionally, most investigations do not rely on a statistically representative number of particles. Corresponding simulation calculations often contain only several hundred, e.g., the discrete element method (Dong et al. 2009, 2007; de Bono and McDowell 2015). In contrast to the location of a single particle within the bulk, the simulation cannot represent the variation of size and shape distributions with sufficient accuracy. However, this is essential for cake filtration because the corresponding distribution tails substantially impact the coordination number and thus the pore flow behavior (Ding et al. 2018). Minor amounts of fines, which the distribution reflects only at the tail end, can already significantly determine the porosity, locally significantly reduce the pore size and lead to a decreased permeability of the bulk material, which permanently limits the throughput of the industrial process. Mathematically, these distribution tails are difficult to account for in simulation calculations since many approximation functions cover distributions only to a certain degree and are limited to the most frequently occurring particle sizes and shapes.

A real filter cake structure consists of several hundred thousand to millions of particles. The corresponding very high number of contact points between these particles preferably

Fig. 1 Summary of the empirical correlations for the average coordination number k as function of the bulk porosity ε (Smith et al. 1929; Rumpf 1958; Ridgway and Tarbuck 1968; Meissner et al. 1964; Nakagaki and Sunada 1968; Haughey and Beveridge 1969; Gotoh et al. 1978) besides monodisperse ideal sphere arrangements

Table 1 Additional information to Fig. 1 about corresponding particle properties and experimental design

Author	Particle size	Particle shape	Particle–particle interaction	External forces
Smith et al. (1929)	7.56 mm	Spherical	Cohesion	Shaking, tapping, tamped with piston
Rumpf (1958)	0.5...50 mm	Almost spherical	Adhesion	Shaking, pressing
Meissner et al. (1964)	0.25...1 mm	Spherical	Cohesion	Rotating, tumbling
Ridgway and Tarbuck (1968)	9 mm	Spherical	Cohesion	partial tapping
Nakagaki and Sunada (1968)	5, 6, 7, 10 (theoretical)	Spherical	Adhesion	None (particle concentration in suspension)
Haughey and Beveridge (1969)	Uniform (review paper)	Spherical	Cohesion	Fluidized, loosely poured, shaking
Gotoh et al. (1978)	Uniform (simulation)	Spherical	cohesion	None (variation number of particles)

retain liquid after mechanical filter cake dewatering as a drainage process, resulting in residual moisture as a characteristic measure (cf. Fig. 2) (Batel 1954; Xu and Louge 2015). If the formed filter cake is subsequently further pressurized with compressed air, the gas phase penetrating the pores displaces the contained liquid. For industrial applications in which the pure solid or liquid represents the final product, further processing is essential for the process's success. The displacement of the liquid is only successful if the capillary inlet pressure can be exceeded at the individual pore. Pores that are too small or hydraulically isolated remain liquid-filled. Understanding the liquid entrapment at the microscopic scale provides the key to further industrial process optimization.

The analysis of heterogeneous filter cakes built up from irregularly shaped particles with widely distributed particle sizes constitutes the focus of this research to investigate the macroscopic influence of the mentioned microscopic particle properties on filter cake structure and dewatering behavior. The use of X-ray microscopy grants us the needed high-resolution access to the particle–particle contact region and the hydraulically isolated liquid volumes trapped in the immediate environment of the contacts.

2 Material and Methods

Sections 2.1. to 2.3 introduce the materials used, describe their properties and explain their importance for industrial processes. Subsequently, we present the experimental setup for cake formation and dewatering for both the laboratory-scale and the small-scale experiments performed in situ within the XRM. We already describe the following basics of filter cake tomography in (Löwer et al. 2020b), but we give a more detailed insight into image processing at the end of the chapter.

Fig. 2 Left: Schematic view of progress in filter cake dewatering with detailed view of remaining liquid distribution between particles according to Batel (1954). Right: Several particles and remaining liquid inside a scanned filter cake after mechanical dewatering; liquid bridges connects particle neighbors to the central particle (red)

2.1 Material Characterization

For all filtration experiments, we use crushed and classified Al_2O_3 particles ($\rho = 3.82 \text{ g cm}^{-3}$, in size range of 20 to 300 μm (Fig. 4), creating different mixtures from the two primary particle collectives ($20 < x < 90 \text{ }\mu\text{m}$ ($c_{\text{V,F}} = 1$), $55 < x < 300 \text{ }\mu\text{m}$ ($c_{\text{V,F}} = 0$)) according to their content of fine particles ($c_{\text{V,F}}$ = volume fine fraction/total solid volume). We have chosen these particles because they serve as raw materials in many industrial processes (Ashwath and Xavier 2016; Gogolewski et al. 2009; Fei et al. 2014; York et al. 1996). They have a non-spherical but compact shape and, in addition to the liquid used, offer good contrast for tomographic measurements, indicated by the mass-related X-ray absorption coefficient μ/ρ ($\mu/\rho = 2.14 \text{ cm}^2/\text{g}$ at 20 keV for Al_2O_3) (Gerward et al. 2004). The particles are suspended in a liquid phase of 24 m.% glycerin ($\text{C}_3\text{H}_8\text{O}_3$, $\mu/\rho = 0.64 \text{ cm}^2/\text{g}$ at 20 keV) and 76 m.% dist. water (H_2O , $\mu/\rho = 0.79 \text{ cm}^2/\text{g}$ at 20 keV) with 25 mM KI (density of the solution 1.06 g/cm^3 , dynamic viscosity of 1.98 mPas at 20 °C, $\mu/\rho = 0.84 \text{ cm}^2/\text{g}$ at 20 keV).

Contrast agents like KI enhance the achievable contrast between liquid, solid and gaseous phases in the final gray value images. The implication of contrast agents simplifies the image analysis and virtual phase separation. We keep the salt concentration of KI below a critical value in order for the ζ -potential to stay above $|\pm 30 \text{ mV}|$ and, consequently, to prevent van der Waals driven agglomeration effects. Any pre-structuring of the suspension due to particle–particle interactions at low ζ -potentials might significantly influence the subsequent cake structure and the associated parameters.

2.2 Experimental Procedure and Setup

The filtration tests are performed in a laboratory-scale pressure nutsch as well as in a small-scale filtration cell. A pressure nutsch consists of a vertical cylindrical vessel into which the suspension is filled in. At the bottom, the filter cloth closes the nutsch. At the top, a pressure-tight lid with lighting and a sight window covers it. After the lid and the filter cloth are mounted and the suspension is filled in, the vessel can be pressurized.

For the tomography experiments inside the XRM ZEISS Xradia 510 Versa X-ray microscope, we build a custom-designed in situ flow cell (Fig. 3). Based on a standardized batch pressure nutsch from laboratory scale, we downscale the cell to the desired volume due to handling reasons and small dimensions inside the X-ray microscope. For data acquisition at high resolution and moderate measuring time, the sample must also be of smaller dimensions due to the measuring principle. The self-developed flow cell (ii) consists of polymethyl methacrylate (PMMA) with low X-ray attenuation. The dimensions correlate with the laboratory equipment (i) at a downscale factor of 100 ($A_{\text{f}}A_{\text{ii}}^{-1} = 100$, A means filter area). The effective filter area amounts to 20 mm^2 with an internal nutsch diameter of 5 mm. To validate the XRM data, we also carried out experiments on a laboratory scale at 500 hPa with a standardized laboratory pressure nutsch (VDI-2762/1 2006). Detailed information about the experimental setup and the measurement principle is available in (Ditscherlein et al. 2020; Löwer et al. 2020b). Figure 3 illustrates both experimental setups schematically.

We investigate the filtration characteristics for bimodal systems by varying the volume fraction of fines $c_{\text{V,F}}$ inside the feed suspension from 0 to 1 (Fig. 4). The normalized particle density distribution $q_3^*(\lg x)$ (Frank et al. 2019) of the particle system is plotted in

Fig. 3 Used filtration equipment for all investigations. Left: Laboratory pressure nutsch and surface of filter cake before and after dewatering ($c_{V,F}=0.2$). Right: In situ flow cell with filter cake inside (Löwer et al. 2020b) and tomographic projection of a filter cake ($c_{V,F}=0.2$) before and after dewatering. At the top pressurized air/liquid inlet, at the bottom filtrate outlet, which is equipped with a filtrate collection chamber

Fig. 4 Normalized particle size distribution $q_3^*(x)$ (Frank et al. 2019) measured by laser diffraction (Sympatec HELOS) with a scanning electron microscopy image of single particles at $c_{V,F}=0.4$

Fig. 4 measured by laser diffraction (Sympatec HELOS). Equation (1) defines the density distributions for each particle size class i according

$$q_{3,i}^*(\lg x) = q_{3,i}(x) \frac{x_{m,i}}{\lg e} \quad (1)$$

$x_{m,i}$ is the mean value of the corresponding particle class i . The ratio of the modal values $x_{M,coarse}/x_{M,fine}$ ranges between 3 and 4.

The feed suspension with a total solid volume fraction of $c_v=0.35$ (c_v =solid volume/total suspension volume) is gently stirred at 250 rpm for 4 min and afterward filled into one of the filtration units. The suspension volume inside the in situ nutsch amounts to 330 μL ; for the laboratory scale, we use 52.4 mL. Filtration tests stop at complete saturation ($S=1$), where liquid still fills all voids within the cake. This point is determined by the time when the reflection on the surface of the filter cake disappears.

Afterward, the filter cakes are slightly mechanically consolidated with a perforated piston at 100 hPa and dewatered at 800 hPa to the final irreducible saturation ($S=S_{ire}$). To avoid an early gas breakthrough, we use a semipermeable membrane made of PVDF (Merck Durapore PVDF, average pore size of 0.45 μm) with a gas capillary entry pressure of 1500 hPa as a filter cloth. Thus, only hydraulic isolated liquid regions remain inside the pore structure.

We conduct the tomography measurements at a voxel size of 4.17 $\mu\text{m voxel}^{-1}$ (camera binning 2), at an acceleration voltage of 45 kV at 3.5 W with 2001 projections per scan. The effective field of view (FOV) amounts to $4.2 \times 4.2 \text{ mm}^2$ with a $4 \times$ microscopic objective. The limited FOV requires a combination of multiple measurements along the filter cake height called vertical stitching. By vertical stitching, we can image the built filter cakes with heights between 11 and 16 mm.

With an increasing fraction of fines, identifying of the solid phase boundary from the image data becomes more complex due to the higher number of small particles and the reduction of void space. Therefore, we set the camera binning to 1 (instead of 2) for the highest fraction of fines ($c_{v,F}=1$), which improves the resolution from 4.17 to 2.08 $\mu\text{m pixel}^{-1}$ at constant FOV. We choose the higher resolution for the mixture at $c_{v,F}=1$ at the expense of much higher measurement time and lower signal-to-noise ratio (SNR). This procedure also requires significantly increased computing time due to the eight times larger dataset, so we avoided the setting for the other measurements.

2.3 Image Processing

We reconstruct the volume data using the *filtered back-projection* algorithm implemented in the ZEISS Xradia reconstruction software (Xradia XMReconstructor 11.1.8). It includes an automatic center shift and beam hardening correction with a factor of 0.05. We smooth the projection data with a Gaussian filter with a kernel of 0.7.

Artifacts related to the XRM measurements necessitate the implementation of a reliable and reproducible image data analyzing procedure. Filtering in ImageJ (Fiji 1.53c) includes additional smoothing, edge enhancement and iterative phase segmentation. A *histogram equalization* (0.3% saturated pixels) and a *non-local means denoising* (standard deviation 25, smoothing 1) (Coll and Morel 2011; Darbon et al. 2008) are applied to the image data for the entire image stack. Then, a *Linear Kuwahara* smoothing filter (angles 11, line length 3, criterion Variance Mean⁻²) (Kuwahara et al. 1976) on every single image reduces the remaining image noise but preserves the edges. The subsequent *Unsharp Masking* for edge enhancement (radius 1, mask weight 0.6) increases the gray value gradient at phase boundaries. To separate the image into grayscale classes, a *Statistical Region Merging* (25 intervals) (Nock and Nielsen 2004) decreases the number of mismatched voxels. It enhances the phase separation in the following image processing workflow. For the solid phase, an *Auto Local Threshold* (Niblack, radius 40) (Niblack 1986) generates a binary

image stack. A *Watershed Irregular Features* algorithm separates connected features as third step segmentation (erosion 4, convexity threshold 0.9 with a minimum size filter of 20 μm , no maximum size filter) using a *Euclidian Distance Transformation* (Brocher 2014) of the solid phase. Finally, the labeling of every feature in the RGB spectrum enables object identification afterward (Videla et al. 2006). After image data treatment, size and shape analysis of the individual identified particles and isolated liquid areas is carried out (Videla et al. 2007).

The presented data processing principle is discussed in Schlüter et al. (2014) and already applied for the present setup by Löwer et al. (2020a, b) and Esser et al. (2021).

To determine the coordination number, the following analysis uses the binarized image stack, processed by the watershed algorithm. Figure 5 explains the procedure schematically, showing the simplified two-dimensional case. Based on the distance field (*Euclidian Distance Transformation*), a new gray value is assigned to each voxel of the void space (binary value 1) on the binarized image stack, depending on the distance to the solid phase (binary value 0) (Borgefors 1986). This new array of gray values consists of four values in the example [-1:3]. In the analyzed data sets, the values vary between [-1:18]. Only voxels that directly share an edge length were included for the neighborhood relations, while we do not consider diagonally connected voxels with only common nodes.

To determine a local minimum or maximum, a working environment of $5 \times 5 \times 5$ voxels in i -, j - and k -direction around a voxel is determined and separated (Fig. 5a). Then a flood filling algorithm was applied (Fig. 5b) (Torbert 2016). Starting from a voxel within the pore volume, the algorithm tests whether the neighboring voxels also contain the same

Fig. 5 Image processing and analysis workflow. Top: two-dimensional representation of the procedure in three-dimensional space (a–c) with example distance to surface array [-1:3] (higher number means larger distance). Bottom: Visualization of the distance field in three-dimensional space in the section of a rendered filter cake scan. Local minima of the field marked as red connections between the particles

corresponding gray value. If the gray value corresponds to the gray value of the starting voxel, it is labeled with the same value. Afterward, the connected volumes are transferred to a new 0/1 mask with the value 1 if the adjacent volume consists of smaller voxel values (Fig. 5c). Otherwise, the connected volume receives the value 0. Each iteration step generates the distance field by a final dilation process, whereby the lowest corresponding gray values form the local minima. For analysis purposes, a unique value from the RGB space labels the resulting contact points according to the corresponding particles.

In the lower part of Fig. 5, different shades of blue visualize the resulting distance field for a few particles. Local maxima are colored dark blue. Local minima appear light blue. This representation leads to the final coloring of the particle contacts in red. In our calculations, we do not only consider points of contact but also near points within the range of one (camera binning 2) and two (camera binning 1) voxels, which is due to the partial volume artifacts during the segmentation (Wang et al. 2015). Here, this consideration may lead to overestimating the number of contact points and uncertainties in porosity for $c_{V,F} = 1$, compared to the fine fractions $c_{V,F} < 1$ (Guan et al. 2019).

3 Results and Discussion

Due to the simple determination in a laboratory experiment, the bulk porosity often serves as a structural key parameter for describing porous media or filter cakes, respectively. By XRM images, the bulk value and the local porosity are directly quantifiable, which reveals information on inhomogeneities within the filter cake structure. Particle–particle interaction forces influence the void volume fraction, and thus, the void fraction depends on the surface-to-volume ratio of the particles forming the solid matrix.

Figure 6 contains the porosity distributions dependent on the fraction of fines. On the left side in Fig. 6a, the boxplots represent experimental data according to the laboratory experiments, weighing the dry cake and relating the resulting solid volume to the total cake volume (Eq. (2)). Twelve filtration experiments for each fine fraction result in the distributions shown in Fig. 6a. These values are the only representative for the entire filter cake as the porosity is measured for the total bulk. Five distinctive quantiles (ϵ_{10} , ϵ_{25} , ϵ_{50} , ϵ_{75} , ϵ_{90}) represent the deviation of porosity as cumulative distribution, visualized as boxplots. The calculation of the bulk porosity for the laboratory experiments in Fig. 6a, b results for every experiment j from

$$\epsilon_{\text{bulk},j} = \frac{\text{void volume}}{\text{total filter cake volume}} = \frac{A\bar{h}_{\text{cake}} - m_{\text{dry cake}}\rho_{\text{Al}_2\text{O}_3}}{A\bar{h}_{\text{cake}}}, \quad (2)$$

where we average the cake height h_{cake} over five values at different locations on the surface.

On the right side of Fig. 6c presents the local porosity values for the tomographic data measured by in situ experiments within the XRM. The in situ experiments are chosen to be representative within the error range of the laboratory tests, and their corresponding bulk porosity (red circles) is shown in Fig. 6b for all fine fractions individually concerning transparency. Figure 6c contains the local porosity distribution ϵ_{local} of the image data by counting the background voxels and then relating them to the total number of voxels of each slice i along with the filter cake height:

Fig. 6 Porosity distributions ε versus the volume fraction of fines $c_{v,F}$. **a** Deviation in bulk porosity of 12 laboratory experiments represented by mean value and standard deviation according to Eq. (2). **b** Measured bulk porosity of in situ experiments for XRM measurements. **c** Local porosity distribution of filter cakes measured by XRM according to Eq. (3) represented by boxplots with ε_{10} ε_{90} as whiskers, the box frame by ε_{25} and ε_{75} with the median value ε_{50}

$$\varepsilon_{local,i} = \left(\frac{\text{background voxels}}{\text{total number of voxels}} \right)_i \quad (3)$$

The voxel edge length defines the spacing of the individual slices.

For isotropic and statistically homogeneous samples, the cross-sectional porosity is equal to the volumetric porosity (Elkhourey et al. 2019), and thus, the porosity results according to Eq. (3) are comparable to Eq. (2).

A distinction has to be made between the distribution over the height of the filter cake (XRM measurements, local porosity ε_{local} in (c)) and multiple measurements for the experimental laboratory tests (bulk porosity ε_{bulk} in (a) and (b)).

Figure 6c shows that the broader variation in porosity is noticeable because the boxplots consider the total height's local distribution. Within the cake structure, some regions are more or less densely packed due to the arrangement of the fines during cake formation. In Fig. 6a, this fluctuation is less evident since the laboratory experiment always only provides an average of the porosity value over the cake's entire height. Here, deviations only occur due to the repetition of the individual experiments.

For $c_{v,F} < 0.3$, the addition of fines slightly reduces the void space between particles from 0.43 without fines to 0.42 with a void fraction minimum at $c_{v,F} = 0.3$. An increase in the fine fraction above 0.3 leads to a growing porosity. The porosity amounts to 0.49 for only fines are present in the structure ($c_{v,F} = 1$). We observe a local difference in the void fraction, which occurs between the homogeneous core volume up to $< 80\%$ of the filter cake height and the upper area, ranging from about 0.44 to 0.50 on average.

This trend of first decreasing and then increasing porosity is well known for bimodal size distributions of sediments, latest by El-Husseiny et al. (El-Husseiny et al. 2019), whereby their lowest value is reached earlier at approx. $c_{v,F} = 0.3$. The results presented

here are more similar to tapped particle beds as Fathollahi et al. (Fathollahi et al. 2020) and Glover and Luo (Glover and Luo 2020) investigated for binary particle mixtures. Responsible for achieving these results is the pressure difference during filtration in contrast to sedimentation. Due to the significantly smaller fine fraction in particle size (Fathollahi et al. 2020) or due to the ideal spherical shape of the particles (Glover and Luo 2020), they achieved $\varepsilon < 0.40$ at $c_{V,F} = 0.4$.

The trend between laboratory data and tomograms shows similar results. In detail, there are deviations at $c_{V,F} = 0.2$ and 0.4 . In the XRM tests, the deviations in porosity are small as function of the cake height. Both measurements' median is also below the laboratory tests between 0.41 and 0.42 . In contrast, the laboratory values remain above 0.42 . The porosity for $c_{V,F} = 0.6$ in the tomogram increases to $\varepsilon_{50} = 0.45$, and the value in the laboratory remains at 0.44 . For $c_{V,F} = 0$ and $c_{V,F} = 1$, very similar porosities are visible. These medians are 0.42 to 0.43 for $c_{V,F} = 0$ and 0.48 to 0.49 for $c_{V,F} = 1$. Notably, the largest local porosity variations occur at 0.3 , although the median value remains almost constant between 0.2 and 0.4 . At this fine particle fraction of 0.3 , some fine particles integrate into the coarse particle structure during cake formation. However, there are also individual fine layers without coarse material that tend to have higher porosities. Figure 7 illustrates this effect on individual cross sections of the measured filter cakes: In particular, at $c_{V,F} = 0.3$, there are significant fluctuations in particle size both in the vertical, i.e., flow direction, and in the horizontal planes, as highlighted in the figure. Densely packed areas with high local fines content exist directly next to coarse material dominated sections with large voids. At $c_{V,F} = 0.3$, locally lowest porosities occur since the integration into the coarse material skeleton works best due to the not too highly deposited fine particle numbers on the filter cake surface.

In addition to porosity, the mentioned coordination number provides further information about the filter cake's particle structure. Figure 8 shows an example of the neighborhood relations for a selected particle of $150 \mu\text{m}$. This particle size is present in every investigated bulk structure except in the bulk built out of the fine fraction. For this reason, there is no

Fig. 7 Cross sections of filter cakes measured by XRM as function of the fines content within the cake structure. At $c_{V,F} = 0.3$, the alternating densely and loosely packed regions are visualized

Fig. 8 Example of neighbors for every fine fraction $c_{V,F}$. k means the coordination number. Top left: 150 μm large particle with its neighbors has been cut out of the data set at $c_{V,F}=0.4$. The size of the red center particle is 150 μm for all solid volume fractions. For better visualization, volume equivalent spheres represent particles in their geometric center

visualization for $c_{V,F}=1$, as there are hardly any particles of this size left in the filter cake. For simplification and clarity, the visualization only adopts the particles' geometric centers of gravity, and volume equivalent spheres represent the particles in the illustration. The results show an increase in the coordination number associated with an increase in the fines content. Several smaller particles replace larger neighbors. From $c_{V,F}=0.5$ on, there are no more neighbors of the same size of the 150 μm center particle.

In the literature, a coordination number of 6 is reported for monodisperse spherical systems, which almost only applies to the coarse fraction $c_{V,F}=0$, shown in Fig. 8 with $k=7$. With all other particle size distributions, significantly higher coordination numbers occur. Some authors also published that the particles' shape and size distribution strongly influence the coordination number (Alonso et al. 1997; Wetter et al. 2018). Karamchandani et al. (Karamchandani et al. 2019) state for widely distributed particle sizes that a stable structure formation starts with a coordination number equal to 8, whereby the theoretical upper value 14 serves for a thoroughly compacted bulk material, which was also proved by other authors (German 2014; German et al. 2009; Bjørk et al. 2012). It is also possible to obtain higher coordination numbers than 14 for particles with different dimensions and shapes (Kristiansen et al. 2005).

Rumpf (1958, 1974) found experimentally that in general, for a cubic packing of uniform spheres, k correlates with the porosity ϵ according to Eq. (4):

$$k\epsilon \approx 3.1 \approx \pi \quad (4)$$

Table 2 Average coordination number k for all mixtures $c_{V,F}$ and the relationship from the literature, Eq. (4) (Rumpf 1974)

$c_{V,F}$ ($\text{m}^3 \text{m}^{-3}$)	Number of ana- lyzed particles (–)	Average k (–)	$k \varepsilon_{50} \pi^{-1}$ (Eq. (4)) ($\text{m}^3 \text{m}^{-3}$)
0	237,963	5.84	0.80
0.2	216,123	5.96	0.80
0.3	204,865	6.04	0.80
0.4	206,563	5.93	0.79
0.5	208,973	5.99	0.84
0.6	303,153	5.97	0.84
1	1,007,768	6.02	0.93

Fig. 9 Distribution of the coordination number k as boxplots dependent on the solid volume fraction of fines ($c_{V,F}$). Width of boxplot represents number of data points, the boxplot whiskers show k_{10} and k_{90} , and the box frame symbolizes k_{25} and k_{75} with the median value k_{50} . Black circle inside the box shows the mean value and circles outside the box occurring outliers

For a large array of randomly packed spherical particles, the coordination number is expressed by Eq. (4) while ignoring the dependency of porosity and coordination number on the extremes $k=1$ and $\varepsilon=0$.

Table 2 includes the average coordination number k , the total number of particles analyzed for all samples and the results according to Eq. (4). In Table 2, the product of $k \varepsilon$ always remains below π , but approaches the literature value with increasing fineness.

Figures 9 and 10 indicate the fine fraction's influence and the particle size on the coordination number distribution. In the first diagram, boxplots represent the distributions of k .

The overall distribution reveals hardly any differences between the different mixtures. The individual particles own between 5.84 and 6.04 points of contact in average

Fig. 10 Distribution of the coordination number k as a function of the particle size x . 0.05 (red), 0.5 (green) and 0.95 (blue) quantiles summarize the contact point distributions in their particle size class. The yellow area indicates the particle size range in which all particles are located between 10 and 90% of the cumulative particle size distribution Q_3 (Frank et al. 2019)

with other particles if only the entire distribution is considered. Comparing the particle size-independent values in Fig. 9 with the global coordination number in Table 2, it is noticeable that the significant variation of the particle size does not manifest itself in k . This value fluctuates only slightly. The coordination number increases with the addition of fine particles up to $c_{V,F}=0.3$. With the further addition of fine particles to $c_{V,F}=0.4$, the average coordination number drops again, but then settles at around $k=6$.

Even if the median and mean values of the overall distribution change only slightly, the fines' influence on the lower and upper part of the distribution is more decisive. The distribution of the coordination number changes with increasing fines content. However, the coordination number's fluctuation range is only determined by very few individual particles, whereas the most significant number of particles has a coordination number around 6.

Figure 10 provides information about the coordination number in more detail. The quantiles of the coordination number distribution 0.05, 0.50 and 0.95 reflect the dependency of k on particle size. While the coordination number for the smallest but in number dominating particle size classes $<50\ \mu\text{m}$ is within the range of 2 to 6 contacts, the addition of fine particles has a more substantial effect on the contacts of larger particles $>100\ \mu\text{m}$. In this case, up to 25 particles surround a single one. As $c_{V,F}$ increases, these particle sizes appear with decreasing frequency and occur only sporadically, so the quantiles end up with the same values for the largest particles.

From $c_{V,F}=0.3$ to 0.4, the lowest porosity within the filter cake is present (cf. Fig. 6). Fine and coarse materials complement each other to form the densest possible packing. The largest particles mainly build the solid skeleton. Smaller ones fill the gaps and have direct contact or form narrow pore throats to the coarse fraction, which results in an average of 5 to 20 contacts per particle increasing with particle size. With a further increase of $c_{V,F}$, the fine particles replace the coarse ones and disrupt the coarse skeleton, making the coordination number decrease again until $c_{V,F}=1$. In pure fractions,

this value is in a range of 4 to 10 for $55 < x < 300 \mu\text{m}$ and 5 to 15 within the fraction $20 < x < 90 \mu\text{m}$. We expect similar coordination number distributions here due to the similar porosity distributions in the filter cake for $c_{V,F}=0$ and $c_{V,F}=1$. However, the decrease in the contact number for large particles $> 100 \mu\text{m}$, in particular, is not of such significance for $c_{V,F}=1$.

We improve the resolution for the sample with fine fraction $c_{V,F}=1$. By setting camera binning from 2 to 1, we reduce the voxel size from 4.17 to $2.08 \mu\text{m}$. Possibly, characteristic deviations at small particle sizes for $c_{V,F}=1$ in Fig. 10 may result from a different resolution which needs to be proved. To achieve the same FOV while keeping the average voxel number per object constant, this verification has to be carried out, but is not possible with the measurement technology and approach we use here. Contacts for small particles might result from the lower SNR at the higher camera binning. The reconstructed image set's noise intensifies with every pixel used on the detector and cannot be easily distinguished from properly assigned voxels.

Figure 11 highlights the importance of particle contacts for cake dewatering after cake formation. On the left in Fig. 11a, the displayed boxplots illustrate the hydraulically isolated liquid areas' distribution as a function of the fine particle content. The left ordinate shows the individual liquid volumes, and the right ordinate with the black squares indicates the average liquid volume per particle volume, i.e., which particle volume binds how much liquid volume.

Fig. 11 Results of the liquid volume and pore size analysis. Left diagram **a** Distribution of the isolated liquid volumes V_L within the filter cake as a function of the fines volume fraction $c_{V,F}$ on the left ordinate. The boxplot whiskers show V_{10} and V_{90} , the box frame symbolizes V_{25} and V_{75} with the median value V_{50} . The right ordinate indicates the ratio of average liquid volume V_L to average particle volume V_S . Center diagram **b** irreducible saturation S_{ire} after filter cake dewatering. Besides the experimental laboratory values S_{exp} according Eq. (8) (with occurring min and max values) the analyzed remaining saturation S_{XRM} from the XRM scans are shown according Eq. (7). Right diagram **c**: Pore throat distribution as a function of the fines volume fraction $c_{V,F}$, defined as shortest solid surface-to-solid surface distance at a local minima of the pore space distance field, which are not defined as contact (distance > 1 voxel). The boxplot whiskers show D_{10} and D_{90} , the box frame symbolizes D_{25} and D_{75} with the median value D_{50} of the cumulative distribution

The liquid distribution shows that the individual size of an isolated liquid volume decreases with increasing fines content. The hydraulically isolated regions resolvable by XRM occur primarily at the particle junctions and contacts, characterized in the previous Figs. 9 and 10.

Focusing on the individual, contact-bound volume as a function of the fines content, a decreasing trend is evident up to a fine particle content of 0.6, after which the volume increases again only slightly. On the one hand, this means that smaller particles can also bind less liquid, and thus, the liquid bridges between the particles become smaller. On the other hand, since most contacts per particle are present at $c_{V,F}=0.3$ and 0.4 (cf. Fig. 10), the total liquid volume per particle volume can also be higher since the coarse material's coordination number $x > 90 \mu\text{m}$ increases strongly. With few contacts per particle as with $c_{V,F}=0$, the individual liquid volumes per particle volume are larger, but the particles also have fewer contacts. Not all particle contacts bind the liquid volume completely; it is also possible for individual volumes of liquid to fill entire pore chambers. These chambers are only accessible through particle proximities or contact points to which the liquid remains entrapped.

In support of achieving a better understanding of this particle–liquid relationship, Fig. 11 presents the liquid volume to the corresponding particle volume ratio in addition to the size distribution of the pore liquid. We calculate the values on the right ordinate according to Eq. (5):

$$\frac{\bar{V}_L}{\bar{V}_S} = \bar{k}_{c_{V,F,i}} * \frac{\frac{1}{N} \sum_{c=1}^N V_{L,c}}{\frac{1}{M} \sum_{j=1}^M V_{S,j}} \quad (5)$$

In this equation, the mean coordination number $\bar{k}_{c_{V,F,i}}$ is used to relate the mean liquid volume $\bar{V}_L$ of the distribution shown in Fig. 11 to the mean particle volume $\bar{V}_S$ of the corresponding particle fraction $c_{V,F,i}$.

In the present case, the decreasing particle size dominates the approximately constant coordination number from Table 2 and the liquid volume, which also becomes smaller. This correlation leads to strongly increasing values of the individual particles' liquid loading from 0.48 for the coarse to 1.47 for the fine fraction. The bimodal mixtures of both fractions show liquid loads between 0.6 and 0.9 with an increasing tendency in the presence of more fines.

In Fig. 11b, the data points indicate the remaining and irreducible saturation S_{ire} after mechanical dewatering. The figure compares the saturations from the tomographic images and those from the laboratory experiments with each other. The saturation values result from Eqs. (7) and (8). All values are between 0.18 and 0.31, which means that 18 to 31% of the liquid volume still occupies the void space.

$$S = \frac{\text{pore liquid volume}}{\text{total void volume}} = \frac{V_{P,L}}{\varepsilon h_{\text{Cake}} A} \quad (6)$$

$$S_{\text{XRM}} = \frac{1}{N_{\text{slices}}} \sum_{i=1}^N \frac{\text{pore liquid voxels}}{\text{background voxels}} \quad (7)$$

$$S_{\text{exp}} = \frac{\varepsilon h_{\text{Cake}} A - V_{L,\text{dis}}}{\varepsilon h_{\text{Cake}} A} = 1 - \frac{V_{L,\text{dis}}}{\varepsilon h_{\text{Cake}} A} \quad (8)$$

In Eqs. (6) and (8), $V_{P,L}$ means liquid pore volume and $V_{L,\text{dis}}$ the displaced liquid volume during the experiment, weighted after the filter cake reaches $S=1$ and before $S=S_{\text{ire}}$.

Equation (7) summarizes all N slices' saturation values to the presented mean value in Fig. 11b.

The laboratory values with their maximum and minimum occurring values show that the saturation reaches its maximum value with the densest packing. Although the individual liquid volumes are highest at $c_{V,F}=0$, the lowest saturation can be reached here since less but larger hydraulically isolated liquid pore clusters.

If the liquid volumes from a) decrease continuously with increasing fines content, two different saturation effects occur. First, the saturation increases until $c_{V,F}=0.5$, when the lowest porosities are just passed. Here the large particles $> 100 \mu\text{m}$ have the most contact points ($k > 20$). Although the liquid volume is minimal due to much smaller neighboring particles, many contacts mean many bound and individual liquid volumes. There are no more contacts between large particles among themselves that could bind large liquid clusters. The solid skeleton of coarse material is disturbed. These numerous neighbors connected with small liquid bridges impair mechanical dewatering and negatively affect the process result.

The second effect manifests itself starting at $c_{V,F}=0.5$: The saturation decreases again to a value of 0.26. The packing structure becomes looser, on the one hand, due to particles of the same size and, on the other hand, due to arising surface forces, which also result in increasing porosities (cf. Fig. 6). The penetrating gas experiences a narrower capillary pressure distribution, which results in lower saturation levels. However, since the overall capillary pressure increases with smaller pore sizes, the initial values of saturation like at $c_{V,F}=0$ cannot be obtained again.

Corresponding results from the XRM measurements are very similar. The saturation is determined as a function of cake height and then averaged according to Eq. (7). Substantial local deviations as a function of height do not occur. The most significant deviations from the average of the 12 laboratory values are found at $c_{V,F}=0.5$. The measured XRM saturation is 0.31, clearly above the mean value of the laboratory data of 0.28. The reason for this might be the precise phase classification during image segmentation discussed in (Oesch et al. 2019), but the overall trend, as shown above, is the same.

Finally, Fig. 11c includes the results of the pore size analysis. A reasonable approach is to look only at the pore space's narrowest parts where the locally highest capillary pressure has to act to displace liquid bridging. Thus, the investigated liquid volumes are mostly found at those pore throats. To define a pore throat, we choose the shortest distance between two solid surfaces passing through a local minimum of the pore space's distance field, at which no contact per definition exists (more than one voxel).

The distribution of these distance lengths plotted as a boxplot diagram in the figure is a function of the fines content. Since characteristically only very few voxels or voxel diagonals are available for this dimension, the distributions, especially between $c_{V,F}=0.2$ and 0.6, are very similar. The only distribution at $c_{V,F}=0$ shows larger pore throats at $11 \mu\text{m}$, subsequently becoming smaller to 8 and $9 \mu\text{m}$ and reaching their minimum at $5 \mu\text{m}$ for $c_{V,F}=1$. Plotting as a boxplot reveals that the broader distribution tends to become narrower with increasing fineness.

At a higher resolution, the downward trend in the pore structure's size could also provide more insight into the correlation between pore throat size and liquid bridge, but further conclusions are not possible at this point.

With these findings, we can conclude that although from a microscopic point of view, larger volumes of liquid are present at each particle contact, from a macroscopic point of view, a lower final saturation is possible after dewatering. Smaller hydraulic isolated liquid regions correspond to a lower local saturation, but an increase in these liquid bridges

results in a higher final bulk saturation for the entire filter cake. Thus, the advantage of a narrow particle size distribution for dewatering becomes evident from a microscopic view as well.

4 Conclusion

Investigating the cake void properties by increasing the fines content results in different characteristics of coordination number, porosity and hydraulically isolated liquid regions. In this study, we verified that the particle size has a strong influence on the particle contacts on the microscale, which has an enormous impact on the process result of filter cake dewatering.

As a function of the percentage of fines, the coordination number is always between 5.84 and 6.04, which coincides with the literature results for other bulk structures. In contrast, significant deviations from the literature become apparent when considering the coordination number as a particle size function. The most significant differences exist for particle sizes above 100 μm . We count up to 25 interparticular contacts with a maximum for mixtures containing 0.3 to 0.6 fines. For particles < 100 μm , the coordination number stagnates at 2 to 5 contacts.

Concerning porosity, the void fraction determines whether large particles still have contact with large particles or whether smaller particles shield them completely. Between a fines content of 0.3 and 0.4, the transition to the shielding effect occurs, and the porosity reaches a minimum. By a further increase of fines, large particles start to be isolated from each other in the filter cake. The higher amount of fines influences the generation of particle–particle interactions at the microscopic level and leads to a shift toward a throat-dominated inner pore structure and smaller pore sizes. This pore throat-dominated structure favors the formation of many but very small hydraulic isolated areas, which hinder the cake structure's dewatering and lead to high saturation levels. By filling the voids with fine material, the resulting increased number of particle contacts intensifies this effect. We were also able to demonstrate a significantly higher loading of liquid versus the volume of the solids, which negatively affects the removal of moisture. A higher ratio of the modal values of the size distributions of coarse and fine material will further pronounce the investigated characteristics.

In the perspective of future studies, a direct correlation on the microscale between bound liquid and contact point for each particle would be desirable. This information would indicate whether an isolated liquid volume belongs to multiple contact points or fills a pore, which would allow an estimation of the binding strength of the respective liquid volumes.

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Authors' contributions Erik Löwer was involved in concept and methods, experiments, data handling (processing, analysis, visualization) and writing the original draft. Florian Pfaff carried out data analysis and experiments. Thomas Leißner took part in supervision, writing the review and editing, and validation. Urs Peuker had contributed to supervision, writing the review and editing, funding acquisition, project administration and resources.

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Data Availability The tomographic datasets generated and analyzed during the current study are available from the corresponding author on reasonable request.

Code availability Custom code in the open-source software applications: Fiji 1.53c (image processing); ParaView 5.8.0 (visualization).

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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