Dewatering mechanisms of compressible filter cakes

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For designing filtration processes, a correlation between applied pressure and filter cake saturation is required. This correlation is provided by the capillary pressure curve, and depends on the pore size distribution of the filter cake. Many materials form compressible filter cakes: The porosity - and pore size distribution - changes with applied pressure. To determine the capillary pressure curve, a saturated filter cake is dewatered by gradually increasing the gas pressure. Below the capillary entry pressure the filter cake will consolidate. Above, desaturation of pores will occur in addition to further consolidation. In industrial filtration processes in contrary, the filter cake is subjected to constant gas pressure and therefore consolidation and desaturation occur simultaneously. With desaturation though, the tensile strength of the filter cake and therefore the resistance to consolidation increases. It is therefore interesting to investigate, if desaturation of an already consolidated cake, as in a capillary pressure experiment, will actually result in the same porosity and saturation as in a conventional filtration experiment, where both mechanisms occur at once.

In this work, filter cake porosity, saturation and residual moisture are compared for two sets of filtration experiments. The first experiment is a standard filtration test, where the filter cake is formed and desaturated by gas pressure. In the second experiment a filter cake is first formed and consolidated and subsequently desaturated by applying gas pressure to the filter cake.

The obtained results show a difference in porosity for the two types of experiments with in all other respects comparable filtration conditions. Porosities for standard filtration tests are higher and independent of applied pressure. We attribute this to the increasing tensile strength of the cake during desaturation which prevents further consolidation. Residual moisture and saturation for both types of experiments show slight differences that can be explained by the different dewatering mechanisms.

Keywords: Cake Filtration, Capillary Pressure, Compressible Filter Cakes, Desaturation, Porosity

Introduction

In modeling desaturation processes, filter cakes are usually considered incompressible, meaning their structure is locally and temporarily constant and not dependent on process parameters [1]. A lot of materials, however, show compressible behavior, leading to a dependency of cake porosity on the filtration pressure and also to a porosity gradient within the cake after cake formation is completed [2]. For the determination of the capillary pressure curve, the differential pressure is increased step by step. When regarding compressible filter cakes, for pressures smaller than the capillary entry pressure the filter cake will be consolidated at each pressure stage. As soon as the capillary entry pressure is exceeded, desaturation of pores will occur in addition to further consolidation. In conventional filtration tests, however, the filter cake is subjected to a constant gas differential pressure and consolidation and desaturation of pores will occur simultaneously. As described by Schubert [3], desaturation of granular material will lead to a fast increase in tensile strength. The material properties of the filter cake and therefore its resistance to consolidation will change during the desaturation process. This leads to the question, whether the correlation between saturation or rather porosity and applied pressure, is independent of the mechanism of dewatering. If there is an effect, desaturation behavior obtained from capillary pressure experiments might not be transferable to a conventional filtration process.

In this work, we investigate the effect of the dewatering mechanisms – consolidation and desaturation – on filter cake porosity and residual moisture. For practical reasons, we simulate capillary pressure experiments by consolidating the filter cake with a piston and then applying gas pressure to the consolidated cake. A comparison of the obtained results with filtration experiments conducted in a pressure nutsche at constant gas pressure follows.

Method

For the filtration experiments precipitated calcium carbonate with a density of 2610 kg/m³ and a mean particle size $x_{50,3}$ of 5.2 µm was suspended in deionized water. The particle size distribution is relatively narrow with a span $\frac{x_{90}-x_{10}}{x_{50}} = 1.4$ as shown in Fig. 1.



Fig. 1: Particle size distribution of the precipitated calcium carbonate used (Distribution measured by laser diffraction)

Conventional filtration experiments were conducted in a stainless steel pressure filter nutsche with a filtration area of 16.62 cm². As filter medium we used a hydrophilic polyethersulfone membrane with a nominal pore size of 0.1 µm. The membrane is not permeable to gas in the pressure range considered, thus thermal drying of the filter cake was avoided. The filter medium was supported by a stainless steel mesh. At the end of desaturation time, the stainless steel mesh was dried off before the gas pressure p_a was turned off in order to prevent resaturation of the filter cake. The filter cake exhibited pronounced wall detachment and cracking during filtration which complicated determination of cake geometry (Fig. 2). To accurately determine porosity and saturation of the filter cake, a stainless steel sample taker was used to obtain samples of defined geometry (compare Fig. 3). The sample takers had a diameter of 12.07 mm. Sample height was measured with a laser displacement sensor. The number of samples that could be taken from a filter cake was limited by the crack pattern and varied between one and three samples. The crack-free samples and the residual filter cake were dried to determine the residual moisture content.





Fig. 2: Filter cake after filtration at 4 bar, showing pronounced wall detachment and cracking

Fig. 3: Stainless steel sample taker with calcium carbonate sample

A compression-permeability-cell (CP-cell) with a filtration area of 51.53 cm² was used to determine the change of porosity and cake resistance with applied pressure. This offers the opportunity to characterize the compressibility of the filter cake. Further the CP-cell was used to conduct combined consolidation-desaturation experiments. The cake height at any time during the experiment can be determined from the piston position. The sample was subjected to piston pressures p_p from 0.4 to 9.5 bar. For measuring the mass specific flow resistance α_m , deionized water was passed through the cake after each consolidation step. The piston pressure is maintained and the driving fluid pressure is set to 0.2 bar.

Compressibility was determined according to the method described by Alles [4]. Pressure-dependent values of porosity ε and mass specific flow resistance α_m are fitted with the following empirical material functions:

$$(1 - \varepsilon) = (1 - \varepsilon_0) \cdot \left(1 + \frac{p_p}{p_0}\right)^{\beta}$$
$$\alpha_m = \alpha_{m.0} \cdot \left(1 + \frac{p_p}{p_0}\right)^n$$

 ε_0 is the porosity measured in the sediment after sedimentation of the suspension and $\alpha_{m,0}$ is calculated from the sedimentation velocity of the suspension [5]. The normalization pressure p_0 was set to 0.1 bar. The compressibility of the filter cake is then evaluated by the sum of the exponents β and n. For the combined consolidation-desaturation tests, the piston was equipped with a gas-permeable monofilament nylon filter medium with twill-weave pattern and a mesh opening of 5 μ m. The suspension was first subjected to piston pressure until a filter cake had formed and a degree of consolidation U_c of 99% was reached. The cake was then dewatered by applying gas pressure p_g to the cake while the piston pressure was maintained. The gas pressure corresponds to the piston pressure. As shrinkage and cracking were strongly reduced in this configuration, saturation and porosity could be calculated from the filter area and cake height. Residual moisture was determined by drying the cake. All samples were dried at a temperature of 108 °C for 24 hours.

Results and Discussion

Fig. 4 and 5 show the progress of porosity and mass specific flow resistance with piston pressure. The material shows compressible behavior, which is evidenced by a decreasing porosity and an increasing flow resistance with increasing piston pressure. Evaluation of the compressibility, as previously described, resulted in a compressibility index β +n of 0.3. Thus the compressibility of the material is low according to the classification by Alles [4].



Fig. 6 shows the porosity of the samples for the two types of filtration experiments at different gas pressures and piston pressures respectively. Two regimes can be recognized: Below the capillary entry pressure, pressure nutsche experiments yield the same values of porosity as experiments conducted in the CP-cell. It can be

concluded that in this case, the gas pressure has the same effect as a piston and compresses the filter cake.

Above the capillary entry pressure, results for porosity obtained from both types of experiments differ. Porosity values after filtration in the pressure nutsche are higher in value and are also approximately constant with a value of 0.61 ± 0.01 . The combined consolidation-desaturation experiment yields lower values of porosity that decline with increasing piston pressure, and are in agreement with the values obtained from compressibility tests. We conclude that in nutsche experiments, the increase in tensile strength of the filter cake due to desaturation prevents further consolidation. Porosity values corresponding to pressure in CP-cell tests cannot be obtained.



Fig. 6: Comparison of porosity obtained for different mechanisms of dewatering the filter cake

Fig. 7 show residual moisture of the filter cake for the two different types of experiments. For the region below the capillary entry pressure, the same trend as in porosity can be observed: Both types of experiments yield the same results. Filter cakes are also fully saturated (compare Fig. 8) indicating that the gas pressure indeed has the same effect as a mechanical piston. Above the capillary entry pressure, the values of residual moisture are slightly larger for the combined experiment. This can be explained by the different mechanisms of dewatering and how it influences porosity and the desaturation process. In CP-cell tests, on the one

hand residual moisture is largely reduced by consolidating the cake. On the other hand this leads to a compact filter cake structure that provides more resistance to the subsequent desaturation step. In nutsche experiments, the residual moisture reduction from the consolidation step is largely omitted. However the overall filter cake porosity is higher and therefore desaturation should be facilitated. From an industrial point of view, the difference in residual moisture seems negligible and it can be concluded that the two effects compensate each other. The difference might be more pronounced for materials of higher compressibility though.

Larger differences can be seen in filter cake saturation (Fig. 8). Saturation is in general higher for the combined consolidation-desaturation experiment. With the water content being comparable for the two types of experiment as stated above, this can be attributed to the lower porosity of the filter cake.



Fig. 7: Comparison of residual moisture obtained for different mechanisms of dewatering



Fig. 8: Comparison of filter cake saturation obtained for different mechanisms of dewatering

Conclusions

It was observed that the parallel mechanisms of dewatering in a nutsche experiment – consolidation and desaturation – lead to a different porosity of the filter cake at the end of filtration compared to experiments where the cake is consolidated first. For gas pressures above the capillary entry pressure, porosities obtained from nutsche experiments are in general higher than porosities obtained from mechanical consolidation and subsequent desaturation. We attribute this effect to the rise in tensile strength during desaturation of the cake that prevents further consolidation.

The difference in cake structure at the onset of desaturation leads to a difference in desaturation behavior for the two types of experiments. Due to the more open structure, filter cakes from nutsche experiments yield lower residual moisture and lower saturation. For a weakly compressible material as used in this work, the difference in residual moisture and saturation for both types of experiments are negligible though. In this case the correlation of gas pressure and saturation/residual moisture obtained from a capillary pressure curve can be transferred to the modeling and design of filtration processes. Possibly the difference will be more pronounced for materials of higher compressibility. This will be subject of further investigations. Furthermore, a reduction of shrinkage cracking could be observed under CP-cell test conditions. This method might provide a feasible means of dewatering materials that are prone to shrinkage cracking. Investigating the details of this effect is part of ongoing research.

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