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CHARACTERIZATION OF PACKED BEDS OBTAINED BY FILTRATION OF COLLOIDAL SUSPENSIONS

Mark Hieke, Harald Anlauf, Hermann Nirschl
Institut für Mechanische Verfahrenstechnik und Mechanik, Universität Karlsruhe (TH), Straße am Forum 8, 76131 Karlsruhe

ABSTRACT

Particles in the colloidal size range gain more and more importance. The solid liquid separation of these particles is a challenge, which is up to now not solved satisfactorily. In order to understand the mechanism of filtration with these particles a method to describe the packed bed is needed. By means of Magnetic Resonance Imaging (MRI) it is possible to gain information of the structure of the packed bed. In this study we compare the structure of packed beds formed by two compressible materials. The difference in compressibility can clearly be seen in the porosity profiles.

KEYWORDS

Filtration, MRI, Porosity Gradient, Colloidal Systems, Compressibility

1. Introduction

The wide range of applications and nowadays widespread use of products with particles in the colloidal size range, imply the need for efficient separation processes. In order to develop such a process a fundamental understanding of the phenomena occurring during filtration of these particles is needed. Interparticulate forces dominate the suspension behaviour, while mass forces can be neglected within this size range. With this drastic change in behaviour the question arises, whether the established filtration laws are still valid for colloidal suspension.

Packed beds obtained by filtration of colloidal particle suspensions usually show compressible behaviour. The filtration is characterized by very high resistances and thus very long filtration times. With the development of an efficient separation process in mind, the packed beds are kept low in height (<5 mm). These small heights required the development of a new technique to determine the height dependent porosity. A gravimetrical determination by cutting and analyzing separate slices is hardly feasible for these thin packed beds.

To analyze these comparatively thin layers with respect to its porosity, a non-invasive magnetic resonance imaging (MRI) method was used. With this method the filtration is conducted within the magnetic resonance tomograph and the packed bed is analyzed in-situ without the peril of a change in structure by handling the sample. With the current setup filtration and consolidation pressures of up to 600 kPa were investigated.

The procedure was developed with an aluminium oxide hydroxide in aqueous solutions. A validation of this method was conducted by a comparison of the determined mean porosity value from MRI experiments and mean porosity from classical filtration experiments. The resolution of approx. 65 microns proved to be sufficient to resolve all relevant effects in this particle system, even though the resolution is not sufficient to visualize single particle layers. Soot was used as a second, more compressible particle system.

2. Experimental Procedure

Materials

Two different particle systems were investigated in this study. On the one hand we used Disperal 20 from Sasol. Disperal is a high-purity, highly dispersible boehmite alumina powder. From this suspensions with a volume concentration of 5% were prepared by a combination of mechanical energy input by stirring and manipulation of interparticulate forces to repulsion by choice of pH value. Disperal showed a comparatively small compressibility.

The other particle system is soot 'FW200' from Evonik. This soot was reported by Alles 2000 to show a strong compressibility. The soot consists of nano scale primary particles and shows due to the production process a good wettability and acid reaction in aqueous solutions. The primary particles form agglomerates with a high porosity. The soot suspensions were set to a volume concentration of 3% and the dispersion was achieved by stirring.

Sample Preparation

In order to eliminate the influence of the sample preparation method, a batch preparation was conducted. A fixed volume of suspension was prepared and exposed to identical dispersion routines. From this batch the required amount of suspension was taken for the filtration experiment. This procedure has proven reliable in the past and is necessary because of the sensitivity of the particle systems in regard to differences in the sample preparation. Due to the non-linear correlation between volume and stirring time, different sample volumes would result in different agglomerate sizes and structures in the suspension.

Method

Filtration experiments with a simple laboratory nutsche device only yield mean values of porosity as results. The porosity of a packed bed is constant only for incompressible material, the investigated materials both show compressible behaviour and thus form a porosity profile within the packed bed.

Due to the comparatively small overall cake heights a gravimetrical method is hardly feasible. In order to resolve the porosity profile within these thin packed beds Magnetic Resonance Imaging (MRI) techniques were used. By this means it is possible to run the filtration and analyse the packed bed in-situ without any alterations of the sample due to handling procedure.

3. Results and Discussion

With the MRI technique it is possible to virtually divide the sample into slices and to analyse each slice individually. A height resolution of 65 microns can be achieved with this technique. This resolution is too low to distinguish between single agglomerates, but it is sufficient to show the porosity profile within a packed bed.

During filtration of a compressible material a porosity profile within the filtercake is formed. The layer next to the filter media shows the lowest porosity and the porosity increases with the cake height. The shape of the porosity profile is determined by the compressibility of the sample. Figure 1 shows the exemplary porosity profile for samples with different compressibilities. The shape of the curves is similar, the difference is in the extent of the compressed lower part of the filtercake.

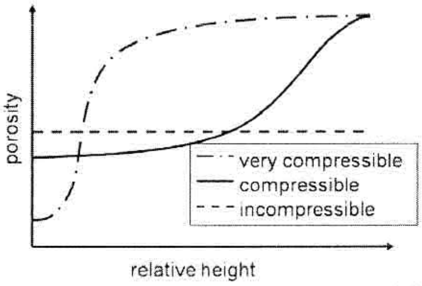

Figure 1 Porosity profiles for different compressibilities

The higher the compressibility is, the smaller the compressed layer at the bottom. In combination with the steep slope and high porosities in the layers above, which show almost porosities equal to suspension concentration, the mean porosity of compressible material is significantly higher. This high porosity implies a larger amount liquid left in the packed bed.

A validation of the MRI technique was done via the comparison of the mean porosity determined by laboratory nutsche filtration and the calculation of the mean porosity from the porosity profiles obtained by MRI experiments. To validate the method data for Disperal was used. For the given pH value of 4, classical filtration experiments yielded a mean porosity of 74% at a pressure of 300 kPa. The calculation of the mean porosity from the porosity profiles led to porosities between 71% and 73% for identical parameters. Both methods are in good agreement and the MRI can thus be used for the determination of the porosity profile.

Figure 2 shows the porosity profiles for the filtration of aqueous soot suspensions with a volume concentration of 3% at different filtration pressures. The profiles show a very high overall porosity with an almost indiscernible region of low porosities next to the filter media. The packed beds have a mean porosity of approx. 95% for all pressures. Increasing pressure results in smaller porosities at the very bottom of the filtercake, but this low porosity region is confined to the lower 20% of the overall cake height. This very small extension of highly consolidated material is characteristic for very compressible material, Tiller and Green 1973 predicted this behavior as well.

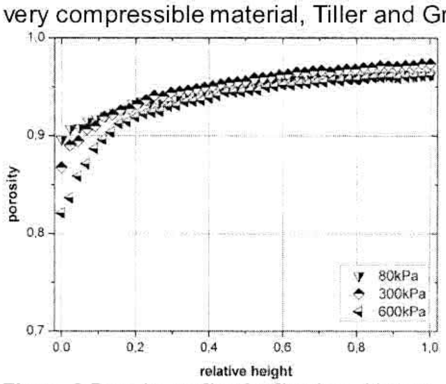

Figure 2 Porosity profiles for filtration with soot at pressures between 80kPa and 600kPa

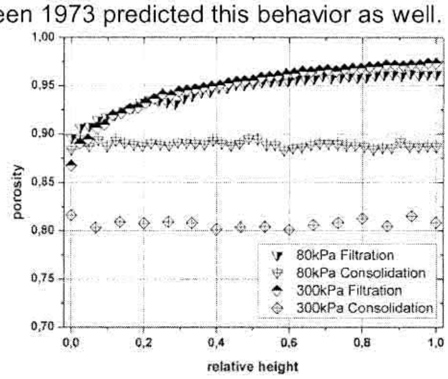

Figure 3 Comparison of porosity profiles from filtration and consolidation at different pressures

Figure 3 depicts the porosities determined during filtration and the porosity obtained after consolidating the filtercake. During consolidation the filtercake is homogenized, the differences in porosity are reduced to the pressure dependent equilibrium value.

This value equals the porosity in the lowest layer of the filtercake during filtration. If the filtration and consolidation value coincide the resolution is sufficient. If the values diverge, as they do for 300 kPa, the extension of the compacted lower layer is below the resolution of 65 microns and the porosity profile needs to be extrapolated to the consolidation value. This inaccuracy in measurement is confined only to the lowest layer/first data point. In this case there is an increase in porosity from approx. 80% (consolidation value) to approx. 88% within the first 65 microns of the filtercake. As the data points depict the mean value for the entire layer the deviation can be explained.

A comparison of porosity profiles for different compressibilities is shown in figure 4. The porosity profiles for soot and Disperal are plotted for a filtration pressure of 300 kPa. There are multiple lines for each particle system, the difference between the lines is the original filtercake height. By using the relative cake height the porosity profiles for different cake heights coincide in a mastercurve for the filtration parameters. So during the filtration process there is a constant rearrangement of particles occurring within the packed bed, resulting in a height independent porosity profile.

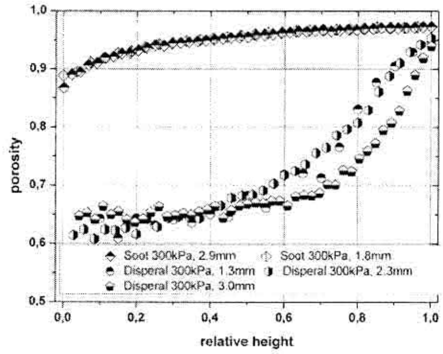

Figure 4 Comparison of porosity plots for Disperal and soot at 300kPa

For Disperal the steep slope in the profile occurs approx. at 60% of the overall cake height. Whereas with soot, which is very compressible, this steep increase in porosity occurs within the lower 10% of the filtercake. The changes in porosity throughout the remaining 90% of the filtercake are very small. A filtercake of this kind resembles more a thickened sludge than a packed bed.

The extent of the area showing almost constant low porosities is a measure for the compressibility. The further this area stretches out, the less compressible the material. The extreme value for this behavior is incompressibility, where the low porosity area covers the whole packed bed.

4. Conclusions

By using MRI techniques the porosity profiles of comparatively thin filtercakes can be determined in-situ. With the current setup the filtercake is virtually divided in layers of 65 microns. The maximum filtration pressure is 1000 kPa, which covers most application purposes.

The technique was validated by comparing the results from MRI experiments with classical nutsche filtration. For a direct comparison the mean value of porosity was

calculated from the obtained porosity profile in MRI experiments. The results were in good agreement, the MRI technique proved feasible and accurate.
With the two particle systems Disperal and soot we were able to measure the different porosity profiles according to the theoretical profiles depicted in figure 1. Disperal has a smaller compressibility, which can be seen in the large extent of the lower porosities in the filtercake. The consolidation porosity is not necessarily the key indicator of compressibility. Disperal has much lower consolidation porosities compared to soot, but nevertheless soot is far more compressible. The compressibility is dependent on the extent of the almost consolidated lower part of the packed bed. The sooner the large increase in porosity occurs, the more compressible the material.
The porosity profile is almost independent of the cake height. There needs to be a constant rearrangement of particles within the packed bed to achieve this. The porosity in each layer of the packed bed is continuously reduced, until it reaches the consolidation value. This rearrangement process begins with the start of filtration and not only after cake formation.

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