Indices

B breakingm mass specificP particle0 reference state

#### References

- K. Borho, R. Polke, K. Wintermantel, H. Schubert, K. Sommer, Chem. Ing. Tech. 1991, 63, 792.
- [2] M. Bentz, W. Stahl, Filtr. Sep. 2001, 38 (6), 42.
- [3] H. Rumpf, K. Schönert, 3rd Eur. Symp. Zerkleinern, Cannes, 1971, 27.
- [4] H. Rumpf, 1st Eur. Symp Zerkleinern, Amsterdam 1962, 1, 108.
- [5] H. Rumpf, Aufbereitungstechnik 1973, 2, 59.
- [6] W. Stahl, M. Bentz, Final Report AiF Research Project No 11958, Universität Karlsruhe (TH) 2001.
- [7] W. Weibull, in *Proc. Ingeniörs Ventenskaps Akademien*, Generalst. Litogr. Anst. Förl., Stockholm 1939, No 153.
- [8] W. Weibull, Trans. Royal Inst. Technol., Stockholm, Sweden, 1949, No
- [9] M. Ghadiri, J. A. S. Cleaver, N. Rolfe, *Powder Technol.* **1993**, 76, 15.
- [10] W. Peukert, L. Vogel, Chem. Ing. Tech. 2001, 73 (4), 327.

This paper was also published in German in Chem. Ing. Tech. 2003, 75 (9), 1265.

# The Influence of Particle Collective Characteristics on Cake Filtration Results

By Harald Anlauf\* and José Angel Sorrentino

Several years of collaborative work, undertaken mainly at the LSM in Caracas, have been performed in order to achieve a better knowledge of the correlation between filter cake properties and the characteristics of the cake forming particle collective.

Common sense suggests that a connection should exist between cake properties and particle characteristics. However, due to its complex character, it has been the prevailing opinion that the only way to know cake properties is to measure them, which is a severe drawback for process engineers who are compelled to make forecasts about what happens if changes in particle size occur during a technical process. In this context, the results of this work provide some progress to overcome the described difficulties.

By comprehensive investigation of various particle size distributions and different materials it could be demonstrated that the cake properties like porosity, permeability, irreducible saturation, void size distribution index and mean capillary pressure can be successfully correlated with particle collective characteristics like mean particle size, particle size distribution width and particle shape. These relationships are strongly dependent on the cake-building mode and are substance-specific.

Once the correlations are established, predictions of the filter performance under conditions of changing particle size are possible using existing kinetic models that have been extensively studied by other authors.

The results of this work claim to offer process engineers an advanced way to understand the behavior of the substance they are dealing with, i.e., perform some selected measurements, fit them in a proper way and make reasonable predictions about the behavior of a substance, no matter its particle size distribution. It will always be necessary to perform experiments and to fit some parameters, but it will be possible to reduce the total amount of work necessary to get extensive information about the filtration behavior of slurries in the case of changing particle size.

#### 1 Introduction

The work under consideration represents the results of several years collaborative research between the Laboratorio Separaciones Mecánicas (LSM) in Caracas and the Institut für Mechanische Verfahrenstechnik und Mechanik (MVM) in Karlsruhe.

The aim of the work, mainly carried out in Caracas [1], was to gain an improved quantitative knowledge about the relationships between the properties of particle collectives and their filtration behavior. Therefore, a method useful for the practical prediction of filtration results under conditions of changing particle properties needs to be developed. For changing particle properties, the particle size distribution and the particle shape were considered here.

The starting point was the very unsatisfying situation for process engineers of not being able to give sound statements about the consequences of changing particle properties in the feed of a separation apparatus. Information about the particle size, and eventually the particle shape, were (and are) mostly used only to characterize the suspension to be separated and not for direct estimation of separation results.

Changes in the particle properties in the feed of a separation apparatus can be caused by changes of a natural feed product composition or by the operation of upstream process steps. When considering upstream process steps, principally particle production processes, e.g., crystallization or precipitation, agglomeration or comminution, and processes like fractionation or thickening of diluted slurries are relevant here.

As well as the practical aspects for the operation of the separation apparatus itself, quantifying the influence of the particles on the results of the separation step acts as an important element for the still newly evolving computational simulations of complex solids handling.

<sup>\*]</sup> Dr.-Ing. H. Anlauf (harald.anlauf@mvm.uni-karlsruhe.de), Universität Karlsruhe (TH), Institut für MVM, D-76128 Karlsruhe, Germany; Prof. Dr.-Ing. J. A. Sorrentino (sorrentj@camelot.rect.ucv.ve), Universidad Central de Venezuela, Laboratorio Separaciones Mecánicas, Apartado 17278, Caracas 1015-A, Venezuela.



Fig. 1 illustrates these correlations for the example of a milling circuit, including the subsequent concentration step and filtration.

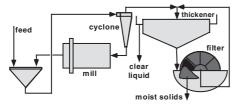


Figure 1. Milling circuit including classification, thickening and filtration.

As indicated in Fig. 1, the work presented has focused (for the present) on the area of cake filtration using gas differential pressure, and is therefore limited to more or less incompressible cake structures. Variation of the physico-chemical conditions of the suspension and the resulting stabilizing or destabilizing effects, have not been investigated.

Target functions for solving the problem in question are the filter cake formation and demoistening kinetics. Under conditions of constant material, well-proven methods for the mathematical formulation of these functions already exist [2], and are not discussed here for that reason.

The key parameters, which contain the particle properties and are, therefore, of central interest here are the cake permeability, the porosity and the capillary pressure distribution.

With regard to the particle influence on the filter cake properties, the first question to arise was which measuring method should be applied to get relevant particle size results.

Secondly, a decision had to be made as to how many parameters of a particle distribution are necessary to get sufficient information for the description of the filtration properties. For this reason mean size, standard deviation and symmetry of the distribution, as well as the particle shape had to be investigated independently from each other.

#### 2 Materials and Methods

Fractions of lime stone, magnesite, aluminiumhydroxide and glass beads with mean particle sizes in the range of about  $x_{50} = 5 \div 200 \,\mu\text{m}$  and geometrical standard deviations up to about  $\sigma_{g,3} = 2.5$  were chosen for the investigations.

To get specifically predefined particle size distributions in a wide range of particle sizes very narrow fractions were first prepared by sieving and sifting. These fractions were then proportionally remixed to yield the desired mean size and particle size distribution shape. For the particle size analysis electro-zone, laser-diffraction, image analysis and small angle photometry were used.

The filter experiments were performed by a laboratory pressure filter cell of the type "Filtratest" (BOKELA GmbH, Karlsruhe), which corresponds to VDI-guideline 2762. This apparatus with 20 cm<sup>2</sup> filter area is able to realize

gas pressure differences of up to  $\Delta p = 0.4$  MPa and a cake thickness up to  $h_K = 25$  mm.

## 3 Measurement of the Relevant Particle Properties

In addition to considering the particle shape, the investigation of different particle size distributions requires the definition of a representative mean particle diameter and a parameter to describe the width of the distribution.

As a key parameter to describe the particle size, the socalled "Sauter diameter",  $x_{SV}$ , proved useful: this is the equivalent spherical diameter by surface area per unit volume to the full distribution, i.e., the particle diameter that has the same specific surface as that of the full distribution. The correct determination of the value of this parameter is imperative for its meaningful utilization in the related calculation methods.

The different devices for particle size analysis quantify the number of particles and determine an equivalent particle diameter corresponding to the respective physical measuring principle.

The device-dependent (index I) Sauter diameter,  $D_{3,2,I}$ , is to be calculated, without information about the particle shape, from the number  $(q_0(x))$ , area  $(q_2(x))$  or volume  $(q_3(x))$  particle frequency distributions according to Eq. (1):

$$D_{3,2,i} = \frac{\int\limits_{0}^{\infty} x_{i}^{3} q_{0}(x_{i}) dx_{i}}{\int\limits_{0}^{\infty} x_{i}^{2} q_{0}(x_{i}) dx_{i}} = \int\limits_{0}^{\infty} x_{i} q_{2}(x_{i}) dx_{i} = \frac{1}{\int\limits_{0}^{\infty} \frac{1}{x_{i}} q_{3}(x_{i}) dx_{i}}$$
(1)

The correlation between the real  $x_{SV}$  and the parameter  $D_{3,2,i}$ , is given by the introduction of a special shape factor  $\psi_{SVI}$ , as can be seen in Eq. (2):

$$x_{SV} = \psi_{SV} D_{3.2.i} \tag{2}$$

As will be shown below, this shape factor, valid for a certain material and measuring method, is determined by a simple experiment on the filter cake permeation.

For practical application, it can be concluded that every particle analyzing method can be utilized for the determination of the Sauter diameter. This is especially interesting for the application of quick methods like laser diffraction.

As a parameter to characterize the width of the investigated particle collectives, the geometrical weighted standard deviation of the particle volume distribution,  $\sigma_{g,3}$ , according to Eq. (3) was chosen:

$$\ln(\sigma_{g,3}) = \sqrt{\int\limits_{0}^{\infty} \left\{ \ln \frac{x}{\bar{x}_{g,3}} \right\}^{2} q_{3}(x) dx}$$
 (3)

This parameter is dimensionless and very well suited to the comparison of the distribution width of different particle fractions because the width is given relative to the logarithmic weighted mean value.

## 4 Influence of the Particle Properties on Cake Permeability and Porosity

The cake height,  $h_K$ , of a homogeneous and approximately incompressible filter cake which is forming in a fixed time  $t_1$ , can be calculated for negligible filter medium resistance from the constant applied pressure difference  $\Delta p$ , the liquid viscosity,  $\eta_L$ , the cake porosity,  $\varepsilon$ , and the slurry concentration,  $\varepsilon_{\nu}$ , according to Eq. (4):

$$h_{\rm K} = \sqrt{\frac{2p_c \varepsilon \Delta p t_1}{\eta_L (1 - \varepsilon - c_v)}} \tag{4}$$

The particle collective influences the structure of the filter cake and thus its spec. permeability,  $p_c$ , and its porosity,  $\varepsilon$ .

After an analysis of different concepts to describe the permeability, the model of Kozeny-Carman given in Eq. (5) was selected:

$$p_{c} = \frac{1}{180} \frac{\varepsilon}{(1-\varepsilon)^{2}} x_{SV}^{2} = \frac{1}{C\varphi(\varepsilon)} \eta_{SV,i} D_{3,2,i}^{2}$$
 (5)

According to Eq. (6) and on the basis of the Kozeny-Carman model, a new pore-particle-shape-factor,  $\eta_{SV,I}$ , is defined which considers, in addition to the aspects of the particle size analysis given by the shape factor,  $\psi_{SV,I}$ , (see Eq. (2)), further particle specific influences on the pore structure of the filter cake, which are caused by deviation from the ideal model of straight and parallel arranged cylindrical capillaries:

$$\eta_{SV,i} = \frac{\psi_{SV,i}}{\lambda \beta} \tag{6}$$

The factor  $\lambda$  takes into account the fact that due to particle contact inside the filter cake, not all the total particle surface acts as a pore surface. The tortuosity factor,  $\beta$  corrects the deviation of the real flow length through the filter cake compared with the straight cylindrical capillary.

From the point of practical application, this differentiated view is efficient because due to different filter cake forming procedures the cake structure can change for the same particle system.

To determine the expected spec. permeability of the filter cake as a function of the particle properties, the filter cake and particle properties included in Eq. (5) are separated according to Eq. (7):

$$p_c C \varphi(\varepsilon) = \eta_{SV,i}^2 D_{3,2,i}^2 \tag{7}$$

As can be seen in Fig. 2, the results of the filtration experiments can be correlated with the data of the respective particle size analysis.

In a double logarithmic diagram for every particle system a straight line of gradient 2 results, which is shifted by the factor  $\eta_{SV,i}$  parallel to the straight line of Kozeny/Carman.

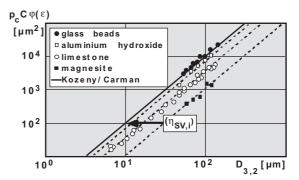


Figure 2. Correlation of filter cake and particle properties.

As expected, the results of the glass bead fractions are placed very near to the Kozeny/Carman straight line.

The validity of this concept to determine the shape factor was confirmed independently by particle size analysis on the basis of a small angle photometer. The shape factor can be calculated directly from the photometric measurement of the spec. particle surface and corresponds to the results evaluated from Eq. (7).

For a practical application of the relations shown in Fig. 2 the particle influence on the porosity  $\varepsilon$  has to be known explicitly.

The expected porosity of a filter cake can be calculated very exactly, but is relatively work intensive, on the basis of a model proposed by Yu and Standish [3]. The respective results are valid only for a constant cake forming procedure.

The basic idea is that the porosity of a packing results from overlaying and weighing the contribution of every involved particle fraction. The weighing function takes into account the existence of interactions between the particles. These interactions result in the fact that particles of different sizes contribute to the porosity not by their volume ratio, but corresponding to a so-called "packing ratio".

As a first step the dependence of the porosity,  $\varepsilon_I$ , of monodisperse packings on the particle size  $x_{\nu}$  and the fitting parameters a and b has to be determined according to Eq. (8):

$$\frac{\varepsilon_i - \varepsilon_0}{1 - \varepsilon_0} = \exp\left\{-ax_v^b\right\} \tag{8}$$

Towards larger particle diameters the porosity function approaches a lower limit,  $\varepsilon_0$ , which is reached if interparticulate adhesion forces become negligible due to the dominant particle weight.

In a second and relatively work intensive step, the packing parameter, p, has to be determined according to Eq. (9), which correlates the packing ratio,  $r_{ij}$ , of different sized particles  $x_i$  and  $x_j$  with their volume ratio,  $R_{ij}$ :

$$r_{ij} = \left(R_{ij}\right)^p = \left[\frac{x_i}{x_i}\right]^p \tag{9}$$

To determine p, several binary particle fractions with different mixing ratios have to be investigated. If for a particle

system and a cake forming procedure the parameters a, b,  $\varepsilon_0$ , and p are known, the cake porosity for any particle size distribution can be calculated with high precision. Past analyses have yielded a prediction accuracy in the limits of about  $\pm$  5 %, as can be seen in Fig. 3 for the example of lime stone distributions.

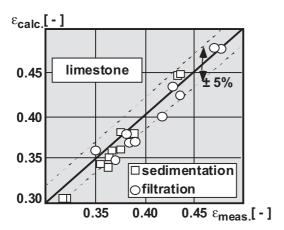


Figure 3. Comparison of calculated and measured filter cake porosities

The filter cakes used here were formed by sedimentation under gravity or, alternatively, by a gas pressure difference.

A less labor-consuming procedure to estimate the filter cake porosity from a pragmatic point of view is given by drawing measured porosity values of cakes from different particle compositions over the reciprocal of the geometrical standard deviation  $\sigma_{g,3}$  of their particle size distribution. Fig. 4 illustrates this using results from centrifugal experiments.

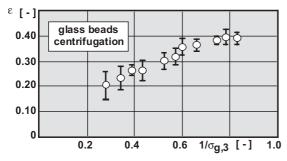


Figure 4. Dependency of porosity on the width of the particle size distribu-

Towards larger porosities the curve reaches a maximum value for monosized particles. Moving towards the opposite direction the porosity decreases due to the increasing width of the particle collective.

All necessary information is now available to be able to simulate the filter cake formation for any particle size distribution under the conditions of a defined procedure.

## 5 Influence of the Particle Properties on the **Capillary Pressure Distribution**

The reduction of the filter cake saturation, S, during the demoistening time, t2, can be formulated according to Eq. (10):

$$\frac{S - S_{\infty}(\Delta p)}{1 - S_{\infty}(\Delta p)} = \left[ 1 + a \left\{ \frac{p_c(\Delta p - \bar{p}_K)}{\varepsilon \eta_L h_K^2} t_2 \right\} \right]^b$$
 (10)

In addition to the cake thickness,  $h_{\rm K}$ , and the porosity,  $\varepsilon$ , the pressure dependent equilibrium saturation,  $S_{\infty}(\Delta p)$ , and a mean capillary pressure,  $\overline{p}_{K}$ , are parameters influenced by the particle collective.

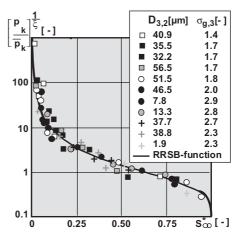


Figure 5. Generalized presentation of the capillary pressure distribution.

As demonstrated in Eq. (11), and especially in Fig. 5, the correlation between capillary pressure and equilibrium saturation can be approximated in a generalized form much better by the Rosin-Rammler-Sperling-Bennett (RRSB)-distribution than by commonly used power functions.

$$S_{\infty}^{*} = \frac{S_{\infty}(\Delta p) - S_{r}}{1 - S_{r}} = 1 - \exp\left[-\left\{\frac{p_{K}}{\bar{p}_{K}}\right\}^{\frac{1}{\bar{\xi}}}\right]$$

$$(11)$$

The three parameters to be approximated are the mechanical irreducible saturation,  $S_r$ , the pore size distribution index,  $\xi$ , and the mean capillary pressure,  $\overline{p}_{\kappa}$ .

The mean capillary pressure represents the pressure at which 63 % of the mechanically drainable liquid still remains in the filter cake.

To predict demoistening results it is again necessary to estimate the particle size influence on these three decisive parameters individually.

For  $S_r$  the following correlation with the Sauter diameter has been found according to Eq. (12):

$$S_r = \frac{1}{(1 + x_{sv})^b} \tag{12}$$



As given in Eq. (13), the pore size distribution index correlates with the geometrical standard deviation of the particle collective:

$$\xi = aln(\sigma_{g,3}) \tag{13}$$

and for the characteristic mean capillary pressure, Eq. (14) seems to be promising:

$$\bar{p}_{k} = \frac{\left[\frac{6}{x_{sv}} \frac{1-\varepsilon}{\varepsilon}\right]^{b}}{(1-0.5\xi+0.5\xi^{2})}$$

$$(14)$$

#### 6 Conclusions

The work presented offers a coherent and praxis oriented concept to predict and simulate filter cake formation and demoistening in the case of changing particle size in a suspension to be separated.

Model equations to correlate particle and cake properties are formulated. For the calculation selected experimental data of different sized solids samples are needed.

The feed material must firstly be fractionated. After particle analysis of the fractions filtration experiments are carried out, which have to follow a constant and application oriented procedure.

On the basis of the current results, further work to validate these findings, especially the correlations found for the cake demoistening, and systematic guidelines for the general utilization of the method have to be formulated.

Received: May 26, 2004 [K 3248]

#### References

- J. A. Sorrentino, Dissertation, Universität Karlsruhe (TH) 2002, Shaker-Verlag, Aachen 2003.
- I. Nicolaou, Fortschritte in Theorie und Praxis der Filterkuchenbildung und -entfeuchtung durch Gasdifferenzdruck, Fortschr.-Ber. VDI, Reihe 3,VDI-Verlag, Düsseldorf 1999.
- [3] A. B. Yu, R. P. Zou, N. Standish, Ind. Eng. Chem. Res. 1996, 35, 3730.

This paper was also published in German in Chem. Ing. Tech. 2003, 75 (9), 1254.

# Comparison of Sedimentation Behavior and Structure Analysis with Regard to Destabilization Processes in Suspensions

By Marc Beiser\*, Götz Bickert, and Philip Scharfer

#### 1 Introduction

At the Institute of Mechanical Engineering and Applied Mechanics, Karlsruhe University (TH), Germany, studies on sedimentation in the centrifugal field have been conducted for several years. In 1992, a manometric sedimentation centrifuge was developed which can be used for analyzing the sedimentation behavior of fine disperse products in the centrifugal field. Bickert [1] studied the sedimentation behavior of stabilized mono- and bidisperse suspensions and, finally, polydisperse systems. Ettmayr *et al.* [2] investigated the sedimentation behavior of different polydisperse solids in stabilized and destabilized suspensions, however, without evaluating the effect of physicochemical suspension properties. The progress of these studies is presented in this work which deals with the influence of electrolyte concentration on the sedimentation behavior of quartz.

The critical ionic strength necessary for the destabilization of the suspension was determined by two different methods: centrifugal settling and FBRM (focused beam reflectance measurement). The sedimentation behavior of quartz particles depending on the electrolyte concentration was detected in the well-proven manometric centrifuge. A laser reflectance measurement (FBRM, Lasentec) determining the cord length distribution was used to detect changes in the state of aggregation. FBRM studies were carried out at the School of Chemical Engineering and Industrial Chemistry, UNSW, Sydney, Australia.

### 2 Experimental Setup and Material

#### 2.1 Sedimentation Analysis

The sedimentation behavior of solids in the liquid phase is characterized by its sedimentation velocity distribution. As mentioned above, the sedimentation velocity distribution can be detected with a sedimentation centrifuge. The machine used is designed up to an acceleration of 300 g. The principle of this analytical centrifuge is based on the measurement of the hydrostatic pressure difference between the

<sup>\*]</sup> Dipl.-Ing. M. Beiser (marc.beiser@mvm.uni-karlsruhe.de), Institut für Mechanische Verfahrenstechnik und Mechanik, Dipl.-Ing. P. Scharfer, Institut für Thermische Verfahrenstechnik, Universität Karlsruhe (TH), Kaiserstr. 12, D-76128 Karlsruhe, Germany; Dr.-Ing. G. Bickert, School of Chemical Engineering and Industrial Chemistry, UNSW Sydney NSW 2052, Australia.