

*Greek symbols*

$\rho$	[kg/m <sup>3</sup> ]	density
$\zeta$	[V]	zeta potential
$\eta_{el}$	[kg/(m · s)]	dynamic viscosity with electric field
$\Delta p_E$	[bar]	electroosmotic pressure
$\Delta p_H$	[bar]	hydraulic pressure
$\eta_0$	[kg/(m · s)]	dynamic viscosity
$\epsilon_0$	[F/m]	electric field constant
$\epsilon_r$	[-]	dielectric constant
$\phi$	[V]	potential

**References**

- [1] H. Darcy, *Les Fontaines Publiques de la Ville de Dijon*, Dalmont, Paris **1856**.
- [2] H. J. Jacobasch, H. Kaden, *Z. Chem.* **1983**, 23 (3), 81.
- [3] A. M. James, *Surf. Coll. Sci.* **1979**, 11 (4), 121.
- [4] R. H. Müller, *Zetapotential und Partikelladung in der Laborpraxis*, Paperback APV, Band 37, Wissenschaftliche Verlagsgesellschaft mbH, Stuttgart **1996**.
- [5] M. von Smoluchowski, *Physikalische Z.* **1905**, 6, 529.
- [6] O. Stern, *Z. Elektrochemie* **1924**, 30, 508.
- [7] K. Weber, W. Stahl, *Separation and Purification Technol.* **2002**, 26 (1), 69.
- [8] H. Yukawa, K. Kobayashi, Y. Tsukui, S. Yamano, M. Iwata, *J. Chem. Eng. Jap.* **1976**, 9, 396.

**Prediction of Centrifugal Deliquoring For Changing Particle Composition\***

By Mark Stiborsky\*\* and Harald Anlauf

The influence of particle size distribution on mechanical deliquoring in the centrifugal field has been studied insufficiently until now. The objective of this paper is to contribute to a better understanding of these influences. Based on well-known models, the influence of the entire particle size distribution on filtering deliquoring in the centrifugal field has been studied. A second objective is to develop a procedure in order to minimize the necessary experimental effort to characterize materials with variable particle size distributions. In the case of a modification of the particle size distribution (for example due to modified settings of upstream devices) a prediction of the deliquoring results in filtering centrifuges can be made. No further deliquoring experiments have to be performed. On the other hand it is also possible to use this procedure for the optimization of the mechanical deliquoring process by changing the particle size distribution.

[\*] Presented at FILTECH EUROPA 16–18 Oct. 2001 Düsseldorf.

[\*\*] Dipl.-Ing. M. Stiborsky, Dr.-Ing. H. Anlauf, Universität Karlsruhe (TH), Institut für Mechanische Verfahrenstechnik und Mechanik, D-76128Karlsruhe, Germany.

**1 Introduction**

The geometrical characteristics of the particles to be separated are substantial influencing factors for the mechanical solid-liquid separation. This particularly applies for rougher particles within the range from some dozen to several hundred micrometers, which are deliquored in large quantities for example on filtering centrifuges.

The most important of these geometrical characteristics are the particle size and the particle size distribution. Usually a simple mean particle diameter is used for the characterization of the particle size distribution. However each mean particle diameter can be obtained by an arbitrary amount of different particle size distributions, which differ with respect to the mechanical deliquoring process.

The objective of this project was the systematic study of the influence of the entire particle size distribution of incompressible materials on the deliquoring results. The possibility of reducing the experimental effort for the characterization of materials with variable particle size distributions may result from this project. Conversion of one product to another is not considered here, since the different products differ also in other characteristics like particle shape or surface, which cannot be described by the particle size distribution.

The project was carried out in cooperation with the Laboratorio de Separaciones Mecánicas (LSM) of the Universidad Central de Venezuela in Caracas, Venezuela. The LSM investigated the influence of the particle size distribution on pressure filtration, while the Institut für Mechanische Verfahrenstechnik und Mechanik in Karlsruhe, Germany, studied the influence of particle size distributions on the centrifugal deliquoring in filtering centrifuges.

Until now the influence of the entire particle size distribution on the deliquoring results have not been fully evident. A number of models exist, but these are generally considered to be too imprecise for practical use. Thus, to study these influences, defined product fractions should be used. Characteristic parameters of these fractions are to be kept constant or should be varied in a clearly defined way. Characteristic parameters are usually mean parameters and parameters describing the distribution width.

**2 Methodology**

Deliquoring equilibrium and deliquoring kinetics generally describe the process of deliquoring in filtering centrifuges. In many previous publications it was shown that bulk parameters, which describe the equilibrium state of deliquoring also correctly describe the deliquoring kinetics [1]. Therefore, here the emphasis is put on the state of the deliquoring equilibrium.

In order to exclude undesired effects of particle characteristics, which cannot be completely considered, at first glass beads were selected, which are incompressible and show a defined particle shape and roughness. The material is available in relatively narrow fractions. The fractions were

additionally divided by sifting, in order to get narrower fractions. By mixing these initial fractions new fractions with predetermined particle size distributions could be produced. First a procedure was developed, allowing the precalculation of different proportions of the individual initial fractions for mixing fractions with defined particle size distributions.

First the particle size distributions of the initial fractions were measured. Using Microsoft Excel™ the characteristic parameters (compare Tab. 1) were calculated from the particle size distribution.

**Table 1.** Parameters of the fractions used.

$x_{3,2}$	$\sigma_{g,3}$	$x_{50,3}$	$\epsilon$	$d_h$
37.1 $\mu\text{m}$	3.62	158 $\mu\text{m}$	20.4 %	6.3 $\mu\text{m}$
40.9 $\mu\text{m}$	2.92	85.3 $\mu\text{m}$	23.7 %	8.5 $\mu\text{m}$
40.9 $\mu\text{m}$	2.60	79.6 $\mu\text{m}$	26.1 %	9.6 $\mu\text{m}$
42.7 $\mu\text{m}$	2.32	72.9 $\mu\text{m}$	26.5 %	10.3 $\mu\text{m}$
40.7 $\mu\text{m}$	1.92	65.5 $\mu\text{m}$	29.8 %	11.5 $\mu\text{m}$
38.5 $\mu\text{m}$	1.75	56.0 $\mu\text{m}$	32.4 %	12.3 $\mu\text{m}$

A calculation for the mixing of fractions has been developed. The characteristic parameters of the mixtures could also be determined by precalculation with this program. By variation of the proportions of the initial fractions, the characteristic parameters of the mixtures can be predetermined. The variation was automatically performed with the help of the Solver using Microsoft Excel. The mixtures were produced according to the precalculated proportions of the initial fractions and their particle size distributions were measured. Precalculated and measured values exhibited a sufficient fit. The measured values were always used for the analysis of the experimental results.

In order to determine the influence of the particle size distribution on the deliquoring behavior, characteristic parameters have to be used, which contain all necessary information about the particle size distribution. Since the simple mean values for cumulative distributions can be obtained from an arbitrary number of different distributions, which all have different characteristics concerning the deliquoring behavior, it is obvious that parameters, which integrally consider the entire particle size distribution should be used. It is assumed, however, that the distributions are completely measured, even the smallest particles. In order to receive universally valid results, approximation functions were not used, but the parameters were directly calculated by summation of the results of the particle size measurement.

### 3 Fundamentals

Particle size distributions are described by means of distribution functions. A distinction is made between cumulative distributions<sup>1)</sup>,  $Q_r$ , and density distributions,  $q_r$ . De-

pending on the principle of measurement different types of distributions are obtained, which describe the number ( $r = 0$ ), surface ( $r = 2$ ) or the volume ( $r = 3$ ) of the particles. The distributions are usually plotted versus the particle size. The different types of distribution functions can be converted with the help of the statistical moments,  $M_{k,s}$ :

$$M_{k,s} = \int_{x_{\min}}^{x_{\max}} x^k \cdot q_s(x) dx \quad (1)$$

It should be noted that particle sizes are always measured in intervals and thus the necessary integration for the calculation of the statistical moments is replaced by a summation. Particularly narrow distributions with very few measuring points can lead to uncertainties.

Characteristic parameters can easily be obtained from the cumulative functions. Most common are the median values, e.g., the median value of the cumulative distribution by volume  $x_{50,3}$  as a mean parameter or the relation of the particle sizes of 84 and 16 percentile by mass for the characterization of the distribution width. However these parameters are lacking as specified above.

Parameters, calculated integrally from the entire particle size distribution, are based on the entire distribution down to the finest particles and in the case of the conversion of distribution functions large errors may occur. However, these parameters characterize the entire particle size distribution.

The Sauter mean diameter  $x_{3,2}$  is suitable as the mean parameter, which corresponds to the particle size that represents the same volume-specific surface like the entire particle size distribution:

$$x_{3,2} = \frac{M_{3,0}}{M_{2,0}} = \frac{1}{M_{-1,3}} \quad (2)$$

The width of the distribution can be described, e.g., by the geometrically weighted standard deviation  $\sigma_{g,r}$ :

$$\left(\ln \sigma_{g,r}\right)^2 = \int_{x_{\min}}^{x_{\max}} \left(\ln \frac{x}{\bar{x}_{g,r}}\right)^2 \cdot q_r(\bar{x}) dx \quad (3)$$

This parameter is dimensionless and is well suited for the comparison of distribution widths of different fractions, since it indicates the distribution width relative to the logarithmically weighed mean value.

Dimensionless numbers are used to represent the deliquoring processes. For the deliquoring equilibrium the so-called Bond diagram is widely used, which displays the bulk saturation,  $S$ , versus the Bond number [2]. The bond number  $Bo_1$  is defined as:

$$Bo_1 = \frac{\rho \cdot g \cdot C \cdot H \cdot d_h}{\gamma \cdot \cos \delta} \quad (4)$$

In the Bond diagram all measured data points with different parameters coincide on one curve. However this applies only if there is still no deliquoring of the interstitial liquid between the particles. The bulk structure is only considered by the hydraulic diameter,  $d_h$ . Thus this parameter must contain all influences from the particle size distribution. The use of only

1) List of symbols at the end of the paper.

one parameter for the characterization of the bulk structure is indeed a major simplification, since bulks always exhibit a complex structure and a pore size distribution. The different pore sizes correspond to different deliquoring equilibrium values and kinetics.

There are various models for the calculation of the hydraulic diameter [3]. The most common one is a representation introduced by Carman and Kozeny. It is based on the definition of the hydraulic diameter as a ratio of the cross sectional drainage area and the moistened circumference:

$$d_h = \frac{2}{3} \frac{\varepsilon}{1-\varepsilon} \frac{x_{3,2}}{f} \quad (5)$$

This representation has the advantage that the influence of the particle size distribution is directly included in the Sauter mean diameter. Further parameters are the porosity,  $\varepsilon$ , and the shape factor,  $f$ , which is unity for spherical particles. Particle roughness is neglected. Thus, above all, the pore size depends not only on the volume specific surface but also on the bulk porosity. For random packings, volume porosity and surface porosity are related and are equal in all areas of intersection. Therefore also the drainage area decreases with smaller porosities. According to (5) this also leads to smaller hydraulic diameters.

Until now there has been no reliable computational model to precalculate porosity from the particle size distribution. It is not possible yet even with packings of monodisperse spherical particles. Random packings do not all inevitably have the same porosity. It is obvious that in bulks containing a broad particle size distribution storage effects can occur, which can strongly lower the bulk porosity. A prerequisite for the storage of particles is, that there must be substantially more smaller particles than larger ones. Furthermore the particle size distribution has to be very wide. Also the manner of bulk formation is important for the bulk porosity. Bulk formation by sedimentation, pressure filtration or centrifugation processes lead to different bulk structures and porosities. Therefore porosity is a parameter, which depends not only on geometrical but also on process parameters.

## 4 Materials and Experimental Procedures

Glass beads were used for this study. Using the procedure mentioned the Sauter mean diameter was kept constant, in order to detect influencing factors not considered in Eq. (5). Tab. 1 shows the characteristic parameters of the fractions used. The particle size distribution was determined by laser diffraction and the Coulter Counter. Fig. 1 shows the particle size distribution of the fractions used, obtained by measurement.

The experiments were carried out in a fast accelerating bucket centrifuge. Sample preparation was performed by means of continuous sample splitting. For sample suspension and bulk formation in the centrifuge buckets high solid concentrations were used, in order to avoid classifying effects.

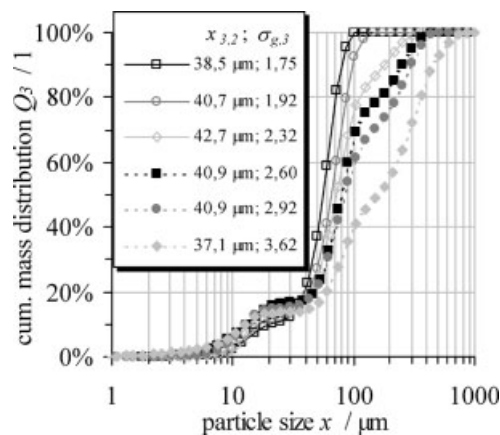


Figure 1. Particle size distribution of the fractions used.

The bulks were formed outside the centrifuge in the centrifuge buckets and afterwards conditioned to a defined initial state in a pressure nutsch filter. Very small differential pressures were used, in order to stay within the capillary threshold pressure.

After centrifuging the final cake thickness was measured and the residual moisture and saturation were determined gravimetrically.

## 5 Representation and Discussion of the Results

The porosity of the different fractions measured after centrifugation is represented in a dimensionless graph (Fig. 2), which was suggested by Wakeman [4].

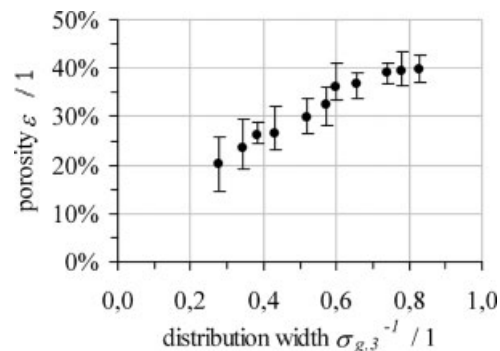


Figure 2. Porosity of different fractions after centrifuging as a function of the distribution width.

On the abscissa the entire possible range of distribution widths is represented by means of the reciprocal value of the geometrically weighed standard deviation. One refers to a packing of monodisperse particles and zero to a distribution of infinite distribution width. In Fig. 2 data points of additional fractions, which are not given in Tab. 1, are also displayed, in order to show the correlation between porosity and distribution width more clearly. Error bars for each data point indicate the range of measured porosity values for each fraction.

The data points indicate a clear trend. However due to the range for each distribution width no definite functional correlation can be derived. For random packings of very

narrow distributions a porosity value of approximately 40 % is expected. The tendency for a horizontal asymptote for large values is apparent in the figure. For infinitely broad fractions no lower limit for porosity exists theoretically. However values of reciprocal geometrical weighed standard deviations below 0.1 are improbable, as with very fine particles compressibility phenomena occur, additionally influencing the shape of the curve. In addition the size of the largest particles represent a boundary to the width of the particle size distribution.

Deliquoring results in the equilibrium state for the fractions given in Tab. 1 and these are presented in Fig. 3.

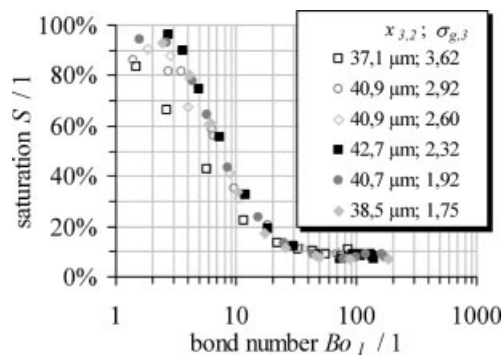


Figure 3. Bond diagram for the fractions used.

The Bond number was calculated by means of relations (4) and (5). The porosity was always calculated based on the cake thickness after centrifuging. All data points within the range of low saturation correspond very well. Within the range of the Bond plateau no substantial differences can be found for different distribution widths. In the range of a high degree of saturation it can be seen that the broadest fractions show an upper inflexion point at smaller Bond numbers than the narrower fractions. Obviously, due to the very broad particle size distribution of these fractions, bulks with a broader pore size distribution have also been formed. Accordingly, deliquoring starts at lower centrifugal forces than predicted by the hydraulic diameters used. However, for the deliquoring process this range is of minor importance, since most centrifuges are used for the reduction of the saturation to values on, or close, to the Bond plateau. In addition this effect doesn't occur until the particle size distribution gets very broad.

Clearly the model based on a single characteristic parameter for the bulk structure is applicable for practical use in centrifugal deliquoring, since at each centrifugal force an equilibrium is established and no complete drain off from the individual pores occurs. This is one of the major differences compared to differential pressure filtration. Thus the liquid levels in the pores of different pore size drop evenly with rising centrifugal forces, whereby the larger pores are deliquored to a greater extent.

## 6 Development of a Measuring Regulation for the Given Problem

The porosity graph in Fig. 2 gives the possibility for interpolation, in order to determine the porosity of a fraction with precalculated particle size distribution. The curve shown in Fig. 2 is not universally valid for all materials, because of the way that bulk formation as well as particle shape and roughness among other things, influence the curve. In order to obtain a sufficient representation for interpolation, both broad as well as narrow fractions should be experimentally analyzed. This can be done by analyzing a broad initial fraction and then classifying into several narrower fractions, which are also experimentally analyzed.

Figs. 4 and 5 show four selected fractions with different distribution widths (they are included in Figs. 2 and 3).

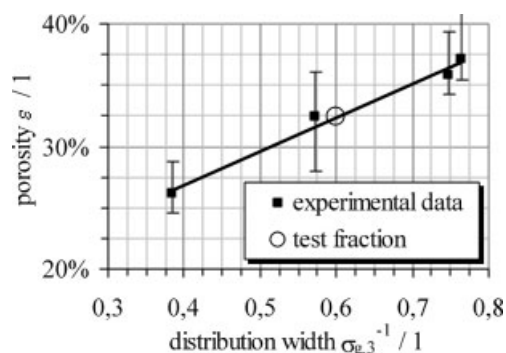


Figure 4. Diagrams for the prediction of porosity for fractions of known particle size distribution.

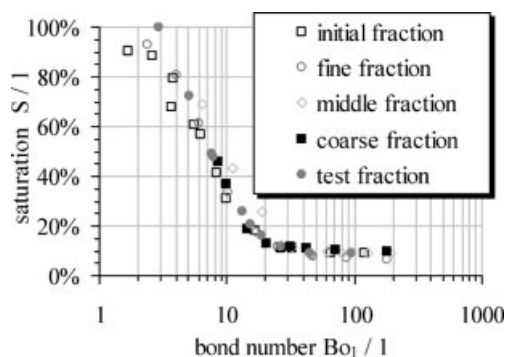
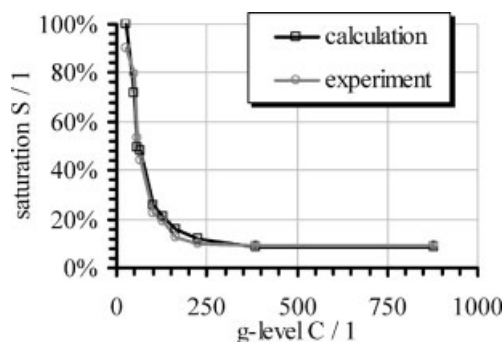


Figure 5. Bond diagram for the prediction of degree of saturation for fractions of known particle size distribution.

Fig. 4 shows the porosity plot corresponding to Fig. 2, and Fig. 5 illustrates the Bond diagram. The fraction with the black circle in Fig. 4 is the test fraction, which is used to compare the predicted deliquoring curve with the experimental data. The curve labeled "test fraction" in Fig. 5 is the fitting curve of the diagram. With the predicted porosity marked with a black circle in the porosity plot of Fig. 4, the Sauter mean diameter (2) and relation (5) the hydraulic

diameter can be calculated. From a fitting curve in the Bond diagram of Fig. 5, saturation degrees for different Bond numbers are obtained. With the definition of the Bond number, the predicted saturations for different g-levels can be compared with the experimental data. This comparison is shown in Fig. 6.



**Figure 6.** Comparison between predicted equilibrium saturations at different g-levels for a test fraction and experimental data.

Clearly the experimental and the predicted saturation degrees fit well. The curves match particularly well at the beginning of the Bond plateau, which is the range where centrifuges are usually operated.

In a Bond diagram the deliquoring results are supposed to form one single curve. It can be determined whether the described deviations from the curve occur for the broadest fractions or whether additional effects occur. For example if fine particles show different shape factors compared to bigger particles, the curves in the Bond diagram of these fractions can differ. This could become apparent in different values for the Bond plateau. In this case the Bond curve has to be interpolated.

## 7 Conclusion

It was shown, that the model for calculating the hydraulic diameter for different particle size distributions by Carman and Kozeny is very applicable. The entire particle size distribution has to be measured in order to calculate the necessary parameters. The influence of the shape factors is complicated, since small and large particles of a collective can differ concerning the shape factors. Thus by classifying the material the mean shape factor of each fraction can be changed.

The results led to a procedure, which allows the predetermination of the deliquoring results from the particle size distribution for a given product. Therefore, deliquoring experiments for only a few fractions with different widths of particle size distributions have to be performed. The results allow an estimation of the deliquoring behavior by interpolation.

## Acknowledgment

The authors would like to thank the German Science Foundation (Deutsche Forschungsgemeinschaft) and the Deutsche Gesellschaft für Technische Zusammenarbeit (GTZ) GmbH for their financial support of this research project.

Received: August 20, 2002 [K 3004]

## Symbols used

$Bo_1$	[-]	bond number
$C$	[-]	g-level
$d_h$	[m]	hydraulic diameter
$f$	[-]	shape factor
$g$	[m·s <sup>-2</sup> ]	acceleration due to gravity
$H$	[m]	cake thickness
$M_{k,s}$	[m <sup>8</sup> ]	statistical moment
$Q_r$	[-]	cumulative distribution
$q_r$	[m <sup>-1</sup> ]	density distribution
$S$	[-]	saturation
$x$	[m]	particle size
$x_{50,3}$	[m]	median value of the cumulative distribution by volume
$x_{max}$	[m]	maximum particle size
$x_{min}$	[m]	minimum particle size
$X_{g,r}$	[m]	geometrically weighted mean diameter
$x_{3,2}$	[m]	Sauter mean diameter
$\delta$	[-]	wetting angle
$\varepsilon$	[-]	porosity
$\gamma$	[Nm]	surface tension
$\rho$	[kg·m <sup>-3</sup> ]	liquid density
$\sigma_{g,r}$	[-]	geometrically weighted standard deviation

## References

- [1] R. Köhling, Zur Fliehkraftentwässerung von Steinkohle, *Dissertation*, TU Berlin **1972**.
- [2] C. Stadager, Die Entfeuchtung von Filterkuchen durch die Kombination von Zentrifugal- und Gasdruckkraft, *Dissertation*, Universität Karlsruhe **1995**.
- [3] W. Batel, *Chem. Ing. Tech.* **1959**, 33 (8), 541.
- [4] R. J. Wakeman, *Powder Technol.* **1975**, 11, 297.

This paper was also published in German in *Chem. Ing. Tech.* **2002**, 74 (4), 427.