

THE „TRUE“ PORE SIZE OF TEXTILE FILTER MEDIA AND ITS RELEVANCE FOR THE FILTRATION PROCESS WITH RESPECT TO THE INTERACTION WITH APPARATUS AND SUSPENSION

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ABSTRACT

The success of a filtration process depends on the filter medium itself but also on the apparatus design and the process conditions. Here the discussion is focussed on woven media for cake filtration, but the principle findings seem to be transferable to vleece depth filter media or membranes.

Particle retention and the flow resistance are representing the main properties of a filter medium relevant for the filtration process. For an optimal filter operation further properties like mechanical strength, physical and chemical environment, wettability, tailormade fabrication and regenerability are of influence

Particle retention and resistance of a filter medium are determined by pore size and structure and the interaction with the slurry. To characterize the pore size, the pore size distribution and the flow resistance different measuring techniques are available. The pore size can be measured often not directly and the question has to be answered how to correlate an indirectly measured „hydraulic pore diameter“ and the „true“ pore size. The bubble point respresents a physically definite pore size measuring method enabling the direct correlation with a shpere of definite size. This had been validated by systematic investigations of different pore structures and in comparison to other measuring methods.

The flow resistance of a filter medium is characterized not adequate in the case of practical filtration by measuring only with particle-free fluid. How can be demonstrated, it must be measured by using the real suspension to be separated to get informations of practical relevance.

Finally process relevant phenomena like filtrate pollution by not separated fine particles or detachment of a filter cake from the filter cloth are influenced not only by the filter medium but also by design parameters of the apparatus and operational parameters of the process.

KEYWORDS

filter media, pore size analysis, bubble point, filter media resistance

1. INTRODUCTION AND DEFINITION OF THE LARGEST PORE

The success of a filtration process depends on the filter medium itself but also on the apparatus design and the process conditions. The filter medium acts as the decisive interface between slurry, filter apparatus and operational conditions. Only if these three aspects are well adjusted to each other, a filtration process will work properly. As [fig.1](#) illustrates, the filter medium interacts with slurry properties like particle concentration, particle size distribution, temperature or pH, with design and operation features of the filter apparatus, like filter type, batch or continuous operation, size and shape of the filter area, the kind of filter cake discharge, stress by pressure, drag or friction and operational parameters like vacuum or gas overpressure, rotation speed of the filter element or the cycle time of the filtration operation.

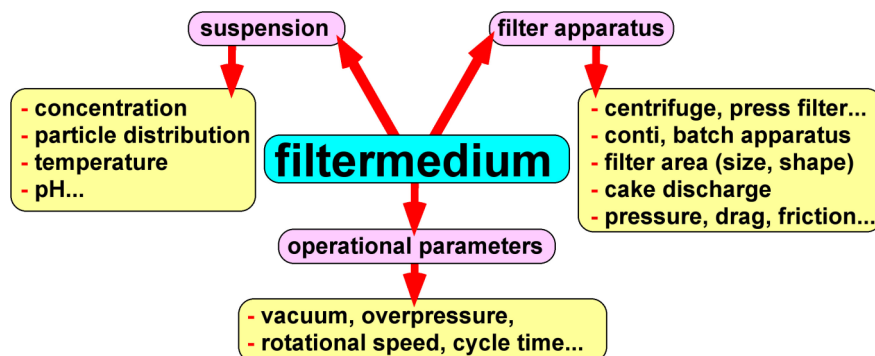


Fig.1: Function of filter media as interface

The focus in the here presented investigations was put on the characterization and filter efficiency of woven fabrics for cake filtration, but the findings seem to be transferable also to vleecees, membranes or other porous structures.

Particle retention and the flow resistance are representing the main properties of a filter medium relevant for the filtration process. The cut size should as small as possible to improve the filtrate clarity and the permeability of the filter medium should be as great as possible to improve the throughput. This seems to be a contradiction, but it will be shown, that both requirements could be realized simultaneously.

For a sufficient service life of a filter medium and a reliable, safe and optimal filter operation of course further properties like mechanical strength, physical (tensile strength...) and chemical (pH...) resistivity, wettability, precise manufacturing (correct fit, no visible needle wholes...) and regenerability (clogging...) are of important influence.

Retention and resistance of a filter medium are determined by pore size and structure and the interaction with the slurry. At first a definition is needed, what the pore size really means. [Fig.2](#) exemplifies the problems for an universal case.

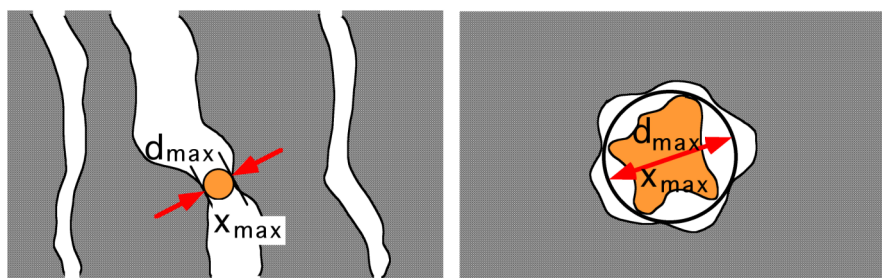


Fig.2: Definition of the maximal pore size

The pores in a three dimensional filter medium structure normally are not straight and cylindrical but wound and show constrictions and extensions. There is existing a certain pore size distribution and no pore looks exactly like the others. Things become more easy in the case of etched microsieves or monofilament fabrics, where the pore geometry is well defined. However it makes sense to define the pore size also for pore systems, like foams, vleeces or sinter materials, which are not so easy to describe.

If one looks to all pores, the largest relevant pore cross section for particle separation can be found as the largest cross section of all constrictions of throughgoing pore channels. This means, that this pore cross section determines the largest particle, which can penetrate the filter medium. To state more precisely the largest pore cross section is the largest circle, which can be drawn into this largest cross section of all constrictions. The diameter of this circle equals the smallest circle around the projection area of the largest particle, which can penetrate this pore. This particle diameter is not easy to determine, because each physically different particle size analyser gives a different equivalent particle size for the same particle. The equivalent particle diameter means the diameter of a sphere of the same physical property as the measured real particle of not spherical shape. For example the result of a sedimentation analysis gives the diameter of a sphere, which would settle with the same velocity like the investigated real particle.

This discussion makes clear, that pore size and particle size are not easy to determine. For practical use and corresponding to fig.2, the maximal pore size should be defined here as diameter of the largest sphere, that is able to penetrate the filter medium.

Beside the cut size of a filter medium the flow resistance is an important issue, because it determines the filter throughput. Similar to the discussion about the pore size also for the flow resistance the separated slurry has to be considered, because the relevant flow resistance of a filter medium originates from the interaction between filter medium and first separated particles.

Finally process relevant phenomena like filtrate pollution by not separated fine particles or detachment of a filter cake from the filter cloth are influenced not only by the filter medium but also by design parameters of the apparatus and operational parameters of the process. As illustrated in fig.1 all these parameters has to be coordinated to get an optimal final result.

2. METHODS OF PORE SIZE DETERMINATION

The pore size can be measured directly or indirectly. Direct measuring methods are the microscopic and image analysis or the sieving with spherical particles like glass beads. Indirect measuring methods like the capillary flow porometry [1, 2] or the bubble point test [3] provide hydraulic pore diameters, which can be interpreted like the equivalent particle diameter.

The microscopic analysis is relatively fast and the largest pore of the investigated sample can be identified clearly. In addition the whole pore size distribution can be measured. There is no limit of pore sizes, which can be measured by this technique. Unfortunately it is not possible to analyze complete filter cartridges and this analysis can be carried out only for filter media, which exhibit throughgoing pores vertical to

the filter area. Fig.3 shows as an example a plain weave, which can be analyzed with microscope and a twill weave, which cannot.

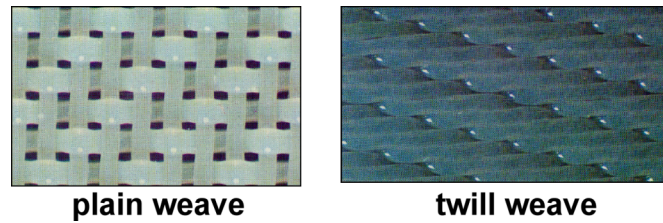


Fig.3: Fabrics in plain and twill weave

A screening test with spheres like glass beads would be able to analyse any structure of filter media [4, 5]. This test is relatively work intensive, can detect the largest pore approximately but not the pore size distribution. A lower limit for wet sieving is given at ca. 5µm, because van-der-Wals attractive forces are becoming more and more important for decreasing particle diameters in relation to the mass forces. The first precondition to find the largest pore is, that there is existing a sphere exactly of this diameter present in the sample. Secondly this sphere must find the largest pore. This pore must not be blocked before by a larger sphere and last but not least the decisive sphere has to be found again in the sieve underflow. To get the best results as possible, special standard fractions of glass beads are available.

An indirect pore size measurement is given by the capillary flow porometry or the bubble-point test. This method is applicable for any pore system, is relatively fast, the largest pore can be detected explicit, the pore size distribution can be measured and complete filter cartridges can be analyzed. The physical background of this method is the overcoming of the capillary pressure p_{cap} in wetted pores by an external applied gas pressure difference Δp . This capillary pressure is correlated with the hydraulic pore diameter d_{equ} , the surface tension of the liquid γ_L and the cosine of the wetting angle δ by the Laplace-equation.

$$d_{equ} = \frac{4 \cdot \gamma_L \cdot \cos \delta}{p_{cap}} = \frac{4 \cdot \gamma_L \cdot \cos \delta}{\Delta p} \quad (1)$$

The hydraulic diameter of a pore represents the diameter of a circular pore of the same capillary pressure like the investigated pore of any shape. The method is limited to great pores at ca. 500µm, because the precise measurement of very low capillary pressures is limited in the Pa range. On the other side the method is limited for pores in the nanometer range, because a gas flow occurs due to diffusion at high gas pressures in the MPa range and not by overcoming of the capillary pressure. This leads to a misinterpretation of the observed gas bubbles.

The principle arrangement of a bubble-point analyzer is shown in fig.4.

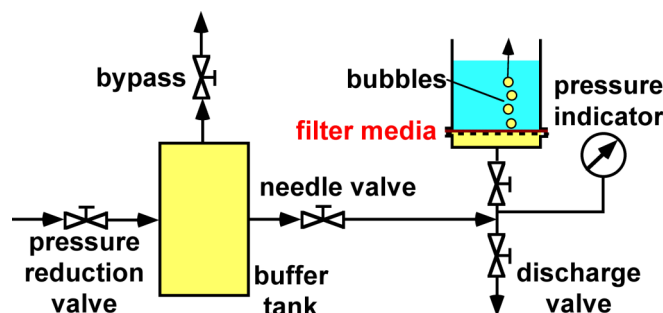


Fig.4: Bubble-point measurement apparatus

The filter media sample of a few cm² is fixed in a measurement cell. A liquid quantity of known surface tension, which must wet the sample perfectly, is given into the cell on top of the sample until a layer of about 1cm height has formed. The perfect wetting is necessary to have a contact angle of 0°. In this case $\cos\delta=1$, which makes equ.(1) more simple. Now a gas pressure is applied from the back side of the filter medium sample, which is increased very carefully by a needle valve. If the lowest capillary pressure of the largest pore is overcome by the gas pressure, gas flows in form of bubbles through this pore. Measuring the corresponding gas pressure the pore diameter can be calculated. It is important to know exactly the right value of the surface tension of the wetting liquid, which depends on the temperature. Eventually the surface tension should be measured in parallel.

3. THEORETICAL APPROACH TO CORRELATE BUBBLE POINT AND LARGEST PENETRATING SPHERE

The question is now how the indirectly measured „hydraulic pore diameter“ d_{equ} and the relevant pore diameter d_{max} of the largest sphere, which can penetrate the pore are correlating to each other. It can be assumed, that there will exist a certain conversion factor k , which allows to calculate d_{max} from the measured d_{equ} .

$$d_{max} = k \cdot d_{equ} \quad (2)$$

An approach to determine k is the formulation of a force balance vertical to the pore cross section.

At first this should be done for a circular pore like to be seen in [fig.5](#).

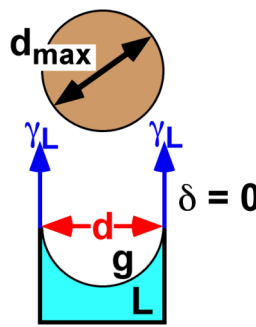


Fig.5: Circular pore

The real pore diameter d and the diameter of the sphere d_{max} obviously are the same. This can be demonstrated by the mentioned force balance.

$$p_{cap} \cdot \frac{\pi}{4} \cdot d^2 = \gamma_L \cdot \pi \cdot d \Rightarrow p_{cap} = \gamma_L \cdot \frac{4}{d} = \gamma_L \cdot \frac{4}{d_{equ}} \quad (3)$$

$$d_{equ} = d \quad d_{max} = d \quad (4)$$

$$\frac{d_{equ}}{d_{max}} = 1 \Rightarrow k = 1 \quad (5)$$

The calculated result correlates with the expectation. In a second step a square shaped pore like depicted in [fig.6](#) should be investigated.

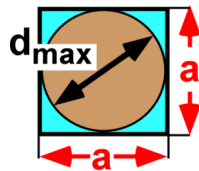


Fig.6: Square shaped pore

Analogous to equ.(3) the force balance has to be formulated in the first step.

$$p_{cap} \cdot a^2 = \gamma_L \cdot 4 \cdot a \Rightarrow p_{cap} = \gamma_L \cdot \frac{4}{a} = \gamma_L \cdot \frac{4}{d_{equ}} \quad (6)$$

Equivalent pore diameter and maximal diameter of the sphere correspond to the side length of the square a.

$$d_{equ} = a \quad d_{max} = a \quad (7)$$

$$\frac{d_{equ}}{d_{max}} = 1 \Rightarrow k = 1 \quad (8)$$

Also in this case the bubble point diameter is identical with the largest sphere, which can penetrate this pore.

A third theoretical experiment is made with a triangular shaped pore (equal sided) like shown in fig.7.

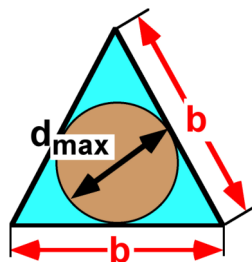


Fig.7: Triangular shaped pore (equal sided)

The calculation of k leads to the same result like in the cases before.

$$p_{cap} \cdot \frac{\sqrt{3}}{4} \cdot b^2 = \gamma_L \cdot 3 \cdot b \Rightarrow p_{cap} = \gamma_L \cdot \frac{12}{b \cdot \sqrt{3}} = \gamma_L \cdot \frac{4}{d_{equ}} \quad (9)$$

$$d_{equ} = \frac{\sqrt{3}}{3} \cdot b \quad d_{max} = \frac{\sqrt{3}}{3} \cdot b \Rightarrow \frac{d_{equ}}{d_{max}} = 1 \Rightarrow k = 1 \quad (10)$$

In the next step a non-symmetric geometry in form of a slot according to fig.8 was investigated.

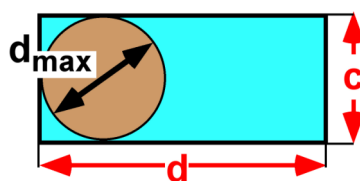


Fig.8: Slotted pore

Now the conversion-factor k depends on the ratio of length d and width c of the slot.

$$p_{cap} \cdot c \cdot d = \gamma_L \cdot 2 \cdot (c + d) \Rightarrow p_{cap} = \gamma_L \cdot \frac{2 \cdot (c + d)}{c \cdot d} = \gamma_L \cdot \frac{4}{d_{equ}} \quad (11)$$

$$d_{equ} = \frac{2 \cdot c \cdot d}{(c + d)} \quad d_{max} = c \Rightarrow \frac{d_{equ}}{d_{max}} = \frac{\frac{2 \cdot c}{\frac{c}{d} + 1}}{c} \Rightarrow k = \frac{\frac{2 \cdot c}{\frac{c}{d} + 1}}{c} \quad (12)$$

If $d=c$, the pore is a square again and $k=1$. If $d \rightarrow \infty$, $k=2$. This means, that in the extreme case of a slot of infinite length the bubble-point diameter is twice as large as the largest sphere, which can penetrate the slot. If c and d are known, k can be calculated exactly.

After pore geometries, which can be realized in form of etched screens weave structures of fabrics have been analyzed. In [fig.9](#) a lace weave can be seen.

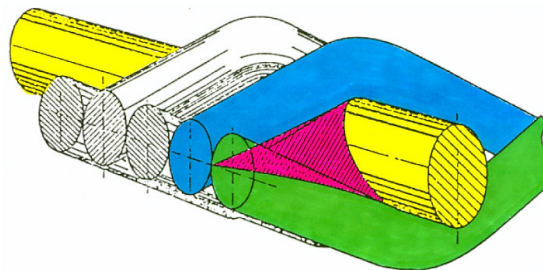


Fig.9: Lace weave

The cut size is given here for the smallest cross section of the greatest pore. From this follows, that one has to look for the smallest boundary surface, which can be placed into this cross section, because it determines the capillary pressure, which has to be overcome to desaturate the pore. It is given by the shortest connection between the contact points of warp and weft. The result is a triangle with slightly inward bent sides. The force balance leads to correlation factors k , which are near to 1 depending on the curvature.

$$\frac{d_{equ}}{d_{max}} \approx 0.95 \Rightarrow k \approx 0.95 \quad (13)$$

Due to the fact, that a fabric of monofilament wires is a geometrically well defined object, the exact geometry can be determined by knowing the weave parameters in every case. The results for a multifilament fabric may differ a little bit from those of a monofilament.

4. EXPERIMENTAL VALIDATION OF THE THEORETICAL FINDINGS

Different types of etched sieves and woven fabrics have been investigated with microscopic pore size analysis, sieving with glass beads and bubble-point measurement. The results have been compared to each other and the theoretical expectations.

In [fig.10](#) bubble-point and microscopic measurements for circular and square shaped pores of etched screens are plotted against each other. As can be seen clearly, all results are located with high accuracy on a 45° straight line. This means, that bubble-point measurements and image analysis give the same values for d_{max} .

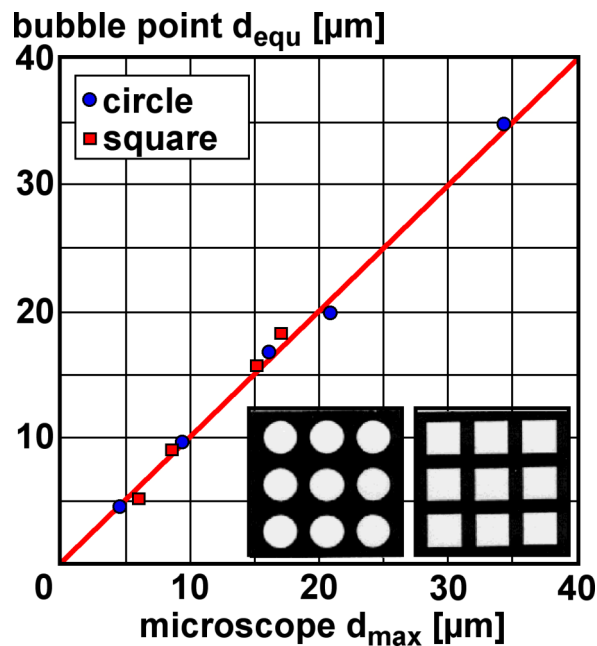


Fig.10: Bubble-point and microscopic analysis of circular and square shaped pores

The same can be observed for slotted screens with a different ratio of length d and width c in [fig.11](#). Again the results from the bubble-point measurement and the microscopic analysis are equivalent, if the factor k was calculated according to eq.(12).

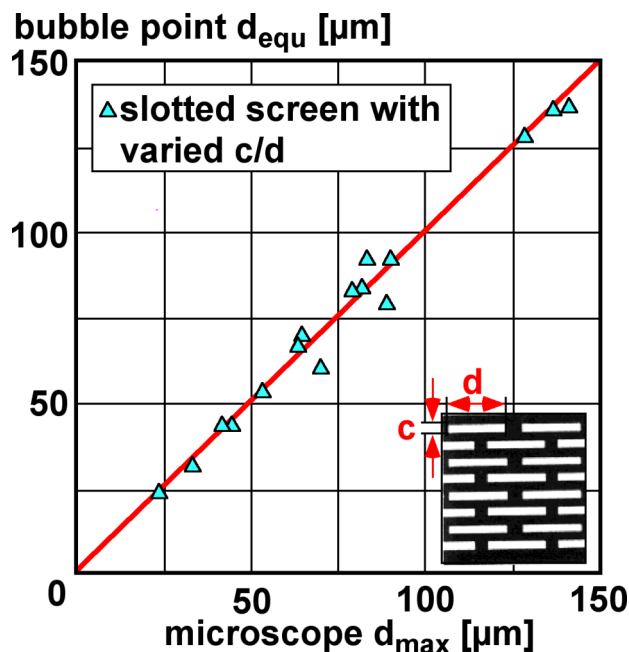


Fig.11: Bubble-point and microscopic analysis of slotted screens

After the validation of the theoretical findings for cylindrical pores of different shape (circle, square, slot) real three-dimensional monofilament fabrics have been investigated

In [fig.12](#) the results of bubble-point, microscopic and glass bead screening tests for plain weaves of different pore size have been plotted. All the methods are leading in principle to the same results. The results for sieving are shifted very slightly to smaller pore sizes in comparison to the bubble-point and microscopic measurements. This is a hint, that as discussed before, the largest pore only can be approximated by sieving.

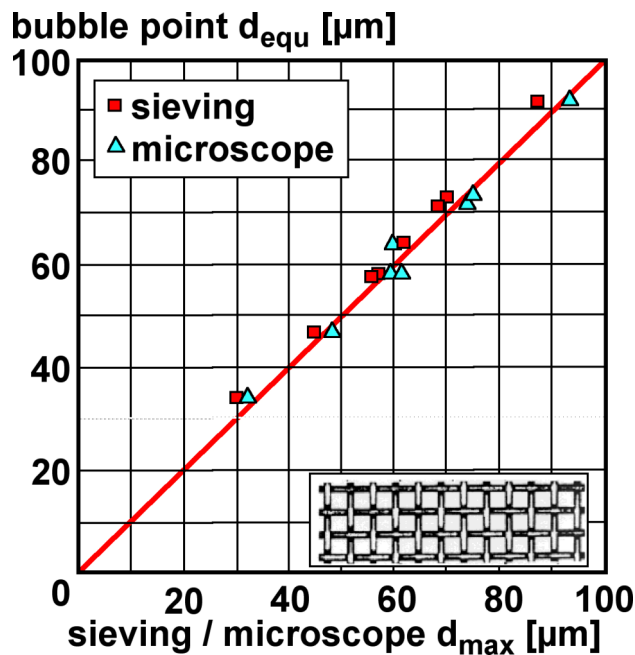


Fig.12: Bubble-point, microscopic and sieving analysis of plain weaved fabrics

The same result can be observed for bubble-point and sieving experiments with various lace weaved fabrics of different pore size. **Fig.13** documents, that it is indeed possible to determine the largest sphere, which can penetrate the fabric, precisely by the bubble-point measurement.

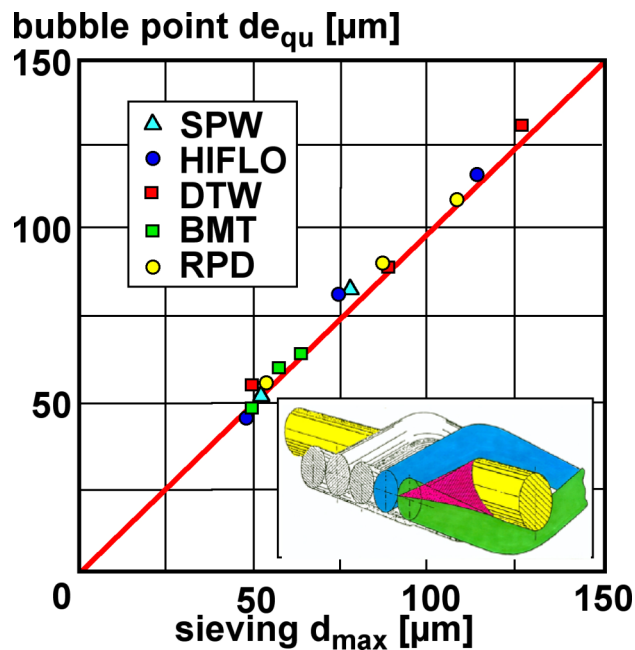


Fig.13: Bubble-point and sieving analysis of lace weaved fabrics

As could be seen from the theoretical approach and the experimental results, it is possible to determine the diameter of the largest penetrating sphere with the bubble-point method, if the geometry of the pore cross section is known. This is relatively easy in the case of fabrics, because there exists a defined weave structure. More difficult is the determination of the pore size for vleecees and felts, because the pore structure is much more complicated and formed by chance. Although it not has been done yet, it should be possible with modern simulation tools to generate non-woven structures and

to determine pore cross sections by connecting all contact points between the fibers, which form a pore cross section. One can assume, that different values for the transformation factor k will be result. For safety the lowest value should be taken.

5. FLOW RESISTANCE OF FILTER MEDIA

As stated in the introduction the flow resistance of a filter medium R_m is beside the pore size the second decisive parameter to describe the filter efficiency. Question is now, how to determine the flow resistance in the correct way to get results, which are relevant for the real filtration practice. **Fig.14** gives an answer to this question.

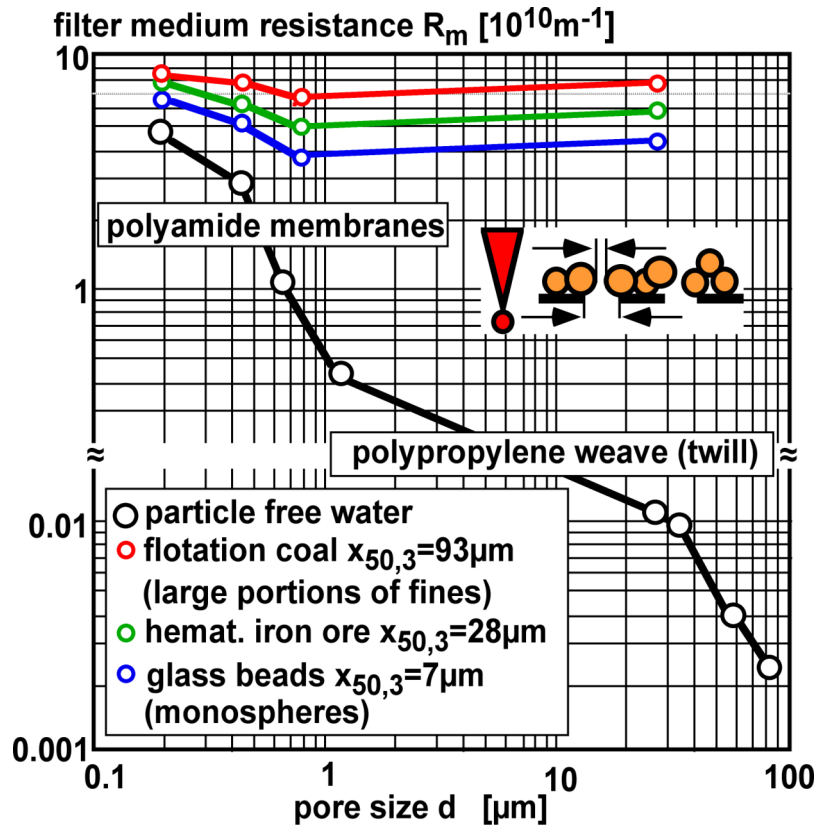


Fig.14: Filter media flow resistance

In fig.14 the filter media resistance is plotted against the pore size of different filter media. Different twill weaved fabrics and polyamide membranes for the sub- μm range had been investigated. Looking first to the black function for filtration with particle free water. As expected, the filter media resistance is changing dramatically with varied pore size. If not particle free water but slurries of flotation coal, hematitic iron ore and glass beads have been filtered, things are looking completely different. The filter media resistance seems to be now more or less independent of the pore size of the filter media. This can be explained by the fact, that first sealing particle bridges has to form over the pores of the filter media, before than the cake can growth on top of the bridges. Normally the pores of the filter media are chosen greater than the particles to prevent pore clogging. The real and relevant filter media resistance consists of the media structure itself, into the structure intruded single particles and the particle bridges. The resistance is dominated by the bottleneck in this system, which is located in the particle bridges. As a result, the filter media resistance is more dominated by the filtered particles than by the structure of the filter media itself. The filtration on a microporous

membrane can lead to the same cake formation performance like the filtration on a conventional filter cloth. If the pores of the membrane are smaller than the particles of the slurry, blocking is also prevented. As a rule the filter media pore size should not be chosen in the same size like the mean particle size, because this would lead to a maximal pore blocking probability.

As a consequence of the particle and media interaction the determination of the practice relevant filter media resistance a filtration experiment with the slurry of interest is indispensable. VDI-guideline 2762 Part 2 [6] gives an instruction, how to carry out this experiment in the correct way. To get the filter media and filter cake resistance, the experimental data of filtration time and filtrate volume have to be plotted according to [fig.15](#). A straight line is resulting with a slope, from which the cake resistance can be calculated and an axial section, from which the filter media resistance can be determined.

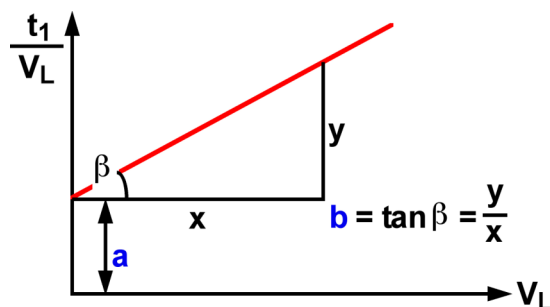


Fig.15: Determination of a practice relevant filter media resistance

The calculation can be carried out by using the filter cake forming equation in the linearized form.

$$\frac{t_1}{V_L} = \left[\frac{r_c \cdot \kappa \cdot \eta_L}{2 \cdot A^2 \cdot \Delta p} \right] \cdot V_L + \frac{R_m \cdot \eta_L}{A \cdot \Delta p} = b \cdot V_L + a \quad (14)$$

6. INTERACTION OF FILTER MEDIA AND FILTER APPARATUS

In fig.1 was shown, that the filter medium acts as an interface between slurry and filter apparatus. In the following two examples should be given to explain this more in detail. As mentioned above in the first moment of filtration particle bridges have to be formed over the pores of the filter medium. During this bridge forming phase particles are able to penetrate the medium and to pollute the filtrate. In a first assumption seems to be clear, that the filtrate pollution will increase with increasing pore diameter of the filter medium, because it needs more time to build the bridges. [Fig.16](#) is demonstrating this on the example of a flotation coal slurry, which was filtered with plain weaved fabrics of different pore size. Membrane filtration retains all particles and the filtrate pollution is zero in that case. For constant slurry concentration the filtrate pollution increases with increasing filter media pore size as expected. If the slurry concentration is increased, the filtrate pollution is reduced remarkably. If the solids concentration is increased from 10 to 45vol% the pore size of the media can be increased (less pore blocking!) without increased filtrate pollution. The reason for this is the much faster build-up of the bridges for the greater slurry concentration. After bridge formation the situation is sealed and no more particles are getting into the filtrate. By the way: If one observes in practice turbid filtrate although cake is forming, these particles could have been got lost in the piping

system and need some time to be swept out. But this is independent from the sealing function of the particle bridges.

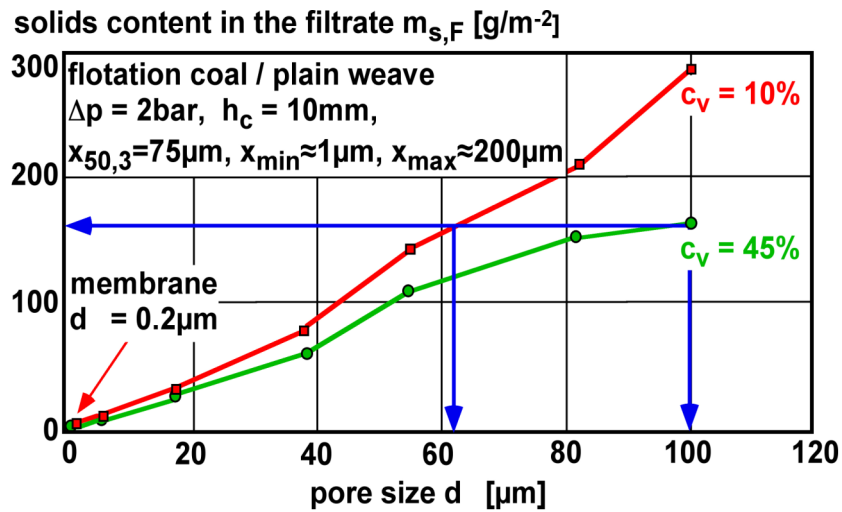


Fig.16: Filtrate pollution

The second example is related to the cake discharge from continuously operating vacuum disc filters. As can be seen from [fig.17](#) the filter cake is released from the filter cell by an air blow back.

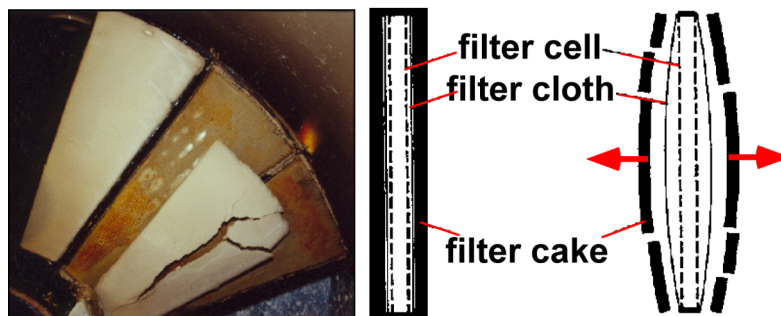


Fig.17: Filter cake discharge from disc filters

To guarantee a complete cake release filter media, filter apparatus design, cake properties and operational conditions must correspond to each other optimally. The cake must be accelerated as fast as possible and the adhering forces between cake and filter cloth have to be as low as possible. Precondition for a fast acceleration is a small cell volume and a tight mounted filter cloth to minimize the volume, which has to be pressurized. The filter cloth must have a high elasticity and plastic deformation is not allowed. The adhering forces between cloth and cake are influenced by the surface properties of the cloth and the moisture content of the cake. The surface of the cloth should be as smooth as possible and the cake should as dry as possible. A smooth surface of the filter cloth can be realized by calendering of the fabric. The moisture content of the cake influences the capillary forces between articles and cloth. A moist cake is sticky and pasty, whereby a more desaturated cake is brittle and is connected by less liquid bridges with the filter cloth.

7. CONCLUSIONS AND OUTLOOK

The definition and measurement of pore sizes in filter media as well as the flow resistance and interaction effects between filter media, slurry, filter apparatus and

operational parameters have been discussed. The largest pore of a filter media was defined as the largest sphere, which is able to penetrate the filter medium. Alternatively to direct measurement methods like microscopic or sieve analysis the bubble-point test as a physically distinct but indirect measuring method delivers an hydraulic pore diameter of a circular pore with the same capillary pressure like the investigated pore of any shape. It could be shown by a force balance, that under the precondition, that the geometry of the pore cross section is known the diameter of the largest penetrating sphere can be determined directly from the bubble-point test. This finding was validated by experiments with edged sieves of circular square and slot shaped pores as well as with monofilament fabrics in plain and lace weave. It seems to be possible to transfer this methodology to other filter media like membranes or fleeces and felts, if an information about the pore geometry would be available. Modern simulation techniques could help to solve this problem. The flow resistance of filter media, relevant for cake filtration processes cannot be measured by permeating the media with particle free liquid. It is indispensable to carry out a filtration test with original slurry, because the relevant media flow resistance results from the media structure itself, intruded particles and the particle bridges over the media pores, which are sealing the media from further particle penetration. This leads to the result, that the bottleneck in most cases is located in the particle bridges and the filter media resistance seems to be more or less independent from the media pore size. An example for the interaction between filter media and operational parameters is the choice of a more open filter media to prevent pore blocking and simultaneous reduction of filtrate pollution by increase of the slurry concentration. An other example is the cake discharge from vacuum disc filters. Only small cell volume, tight fit, high elasticity and smooth surface of the filter medium and preferably dry filter cake together guarantee the best filter performance.

8. NOTATION

latin characters

A	filter area	[m ²]
c _v	volume concentration	[-]
d _{equ}	hydraulic pore diameter	[m]
d _{max}	largest pore diameter	[m]
h _c	cake thickness	[m]
k	conversion factor	[-]
p _{cap}	capillary pressure	[Pa]
p _{cap,e}	capillary entry pressure	[Pa]
r _c	spec. cake resistance	[m ⁻²]
R _m	filter media resistance	[m ⁻¹]
t ₁	cake formation time	[s]
V _L	filtrate volume	[m ³]
x _{50,3}	mean volume related particle diameter	[m]
x _{max}	largest sphere diameter	[m]

greek characters

γ_L	surface tension	[Pa· m]
δ	wetting angle	[°]
Δp	pressure difference	[Pa]
ε	porosity	[-]
η_L	dyn. liquid viscosity	[Pa· s]
κ	concentration parameter	[-]

9. REFERENCES

- [1] J.K. Gupta, *Characterization of pore structure of filtration media*, Fluid/particle Separation Journal, **2002**
- [2] D. Li, M.F. Frey, Y.L. Joo, *Characterization of nanofibrous membranes with capillary flow porometry*, Journal of Membrane Science, Vol.286, Issues 1-2, **2006**, 104-114
- [3] A. Hernández, J.I. Calvo, P. Prádanos, F. Tejerina, *Pore size distributions in microporous membranes. A critical analysis of the bubble point extended method*, Journal of Membrane Science, Vol. 112, Issue 1, **1996**, 1-12
- [4] G.R. Rideal, J. Sorey, *A new high precision method of calibrating filters*, Journal of the Filtration Society, Vol 2(3),**2002**, 18–20
- [5] G.R. Rideal, *Analysis and monitoring: how to improve precision pore size measurement*, Filtration & Separation, Vol. 43, Issue 3, **2006**, 28-29
- [6] VDI-Guideline 2762, Part 2, *Mechanical solid-liquid-separation by cake filtration - Determination of filter cake resistance*, Beuth Verlag, Berlin **2010**

