Solid/liquid separation through cake filtration

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Outline

Among the processes for mechanical separation of particles from liquids, the method of cake filtration offers a vast range of physical and technical possibilities. In addition, cake filtration achieves the lowest degree of residual solid substance moisture using mechanical methods. Particle sizes, which range across several decimal powers; suspension quantities of a few liters up to many cubic meters per hour; the most varied solid substance concentrations, chemical composition of the suspension, process conditions and demands on the separation result lead to the development of a correspondingly large number of separation methods and apparatuses in the field of cake filtration. Under consideration of countless new challenges posed by separation tasks, which are presently not yet or insufficiently solved, there is a substantial development potential for cake filtration methods in the future. This concerns both the theoretical description of the methods as well as the development of apparatuses and processes.

INTRODUCTION

As shown in Fig. 1, cake filtration represents a surface filtration method. Due to a pressure difference (p1-p2), particles move towards a porous filter medium with the velocity v. While the liquid can penetrate the filter medium, the particles form bridges across its pores and lead to the growth of a filter cake. In most cases, individual particles are able to penetrate the pores of the filter medium. This is why the particle concentration in the suspension must exceed a critical value to enable the formation of bridges. The faster the bridges form, the fewer particles reach the filtrate in this first filtration phase. Depending on the specific nature of the separation task, the hydrostatic or centrifugal pressure, a mechanical or hydraulic pressure and a differential gas pressure can be used as driving force.

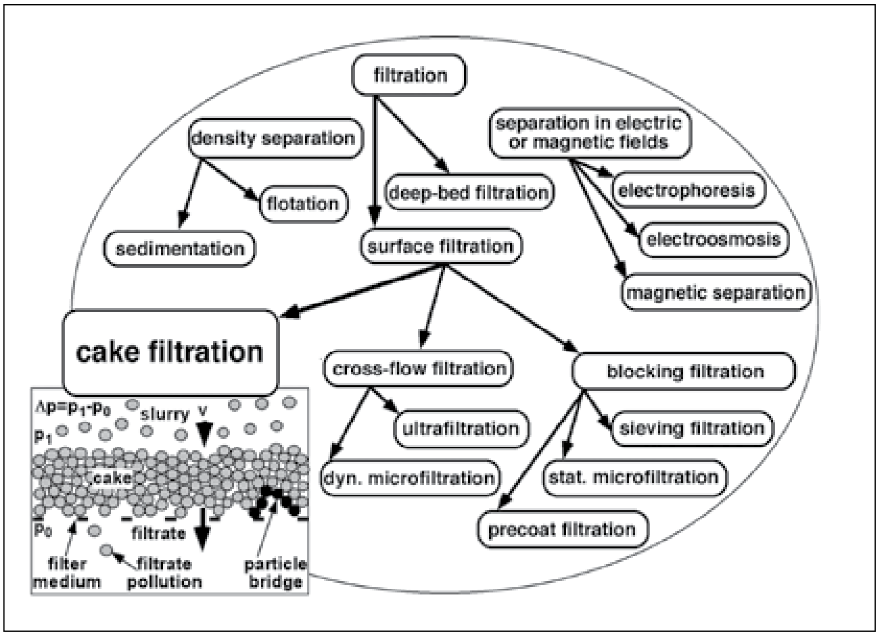

Fig. 1: Cake filtration in the diagram of mechanical solid/liquid separation methods

Various pre-treatment methods are available to improve the filtration properties of suspensions, e.g. agglomeration, concentration, screening or mixing with filter aids. After its formation, a filter cake can be washed and/or dehumidified through post-treatment. Compared to all other mechanical separation methods, cake filtration offers the best possibility for liquid separation through undersaturation or squeezing the cake(1,2,3).

Different separation techniques and the most varied apparatus constructions can be identified for each of the physical separation principles, density separation or filtration. This is due to the fact that one has to overcome an extreme variability with reference to particle size, suspension concentration, volume flow, chemical composition of the suspension, process-related boundary conditions and demands on the separation result.

Fig. 2 shows some separation apparatuses for cake filtration and an orientation of their operating range.

For all filtration methods, the filter medium is the decisive interface between apparatus and suspension. The most varied filter media were developed depending on the apparatus construction, the suspension composition and the operating conditions. For example, the strong friction between filter cake and filter medium in continuously working pusher centrifuges requires the application of robust metallic slotted

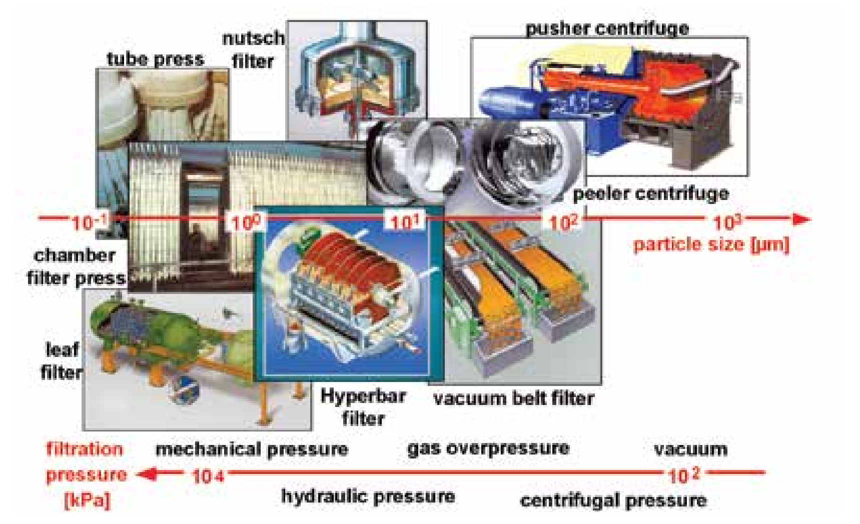

Fig. 2: Selection of different apparatuses for cake filtration

screens. Vacuum disk filters on the other hand require woven filter cloths with a high degree of circumferential elasticity for perfect cake discharge through compressed air blasts. The selection of the material, the construction, the surface treatment and the design allow the best possible adaptation of the filter medium to the demands of the separation process. A suitable filter medium is decisive for the success or failure of each filtration process(4).

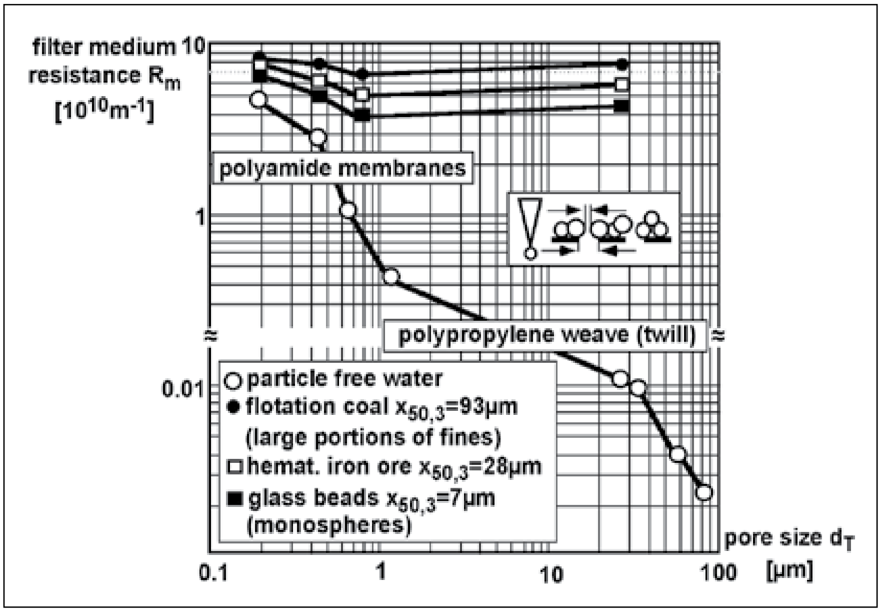

Fig. 3: Filter medium resistance for filtering particle-free liquids and suspensions

Physical phenomena

Initial cake filtration phase – interaction between particles and filter medium

The initial cake filtration phase defines the effective filtration resistance of the filter medium. As Fig. 3 shows, the filter medium resistance in filtration with a suspension cannot be compared to that of filtration with a particle-free fluid. With cake filtration, the resistance of the filter medium is defined by the structure of the filter medium itself, the particles penetrating this structure and the structure of the particle layer forming the bridge. Generally, the pore sizes in this bridge layer are decisive for the size of the filter medium resistance. This explains why the effective resistance of the filter medium is nearly independent of its pore size in the illustrated example. One practical conclusion of this phenomenon is the possibility to replace wide-mesh filter fabrics with microporous membrane filter media at the same cake formation rate but with a much clearer filtrate. In the meantime, a number of such products are available on the market, which promise sufficient service times under technical operating conditions(5).

Formation of incompressible filter cakes

The mechanisms of incompressible filter cake formation are relatively known and can be described with simple equations. The prerequisite for the practical application of the theory in practice on a specific suspension is the knowledge of the actual cake permeability value pc and the filter medium resistance Rm. These parameters must be measured in line with a specific set of regulations (VDI guideline 2762(6) in the laboratory with original suspension and filter medium under practical conditions. In addition, it would be interesting for engineers to be able to estimate in which way the filtration results would shift with respect to quantity in case of a change of the particle size distribution in upstream process stages, e.g. crystallisation or comminution. In principle, the Kozeny/Carman equation can provide an answer to this question. But which numerical value is to be applied for the characteristic average particle diameter? In line with the physical principle, each measuring method leads to a different equivalent diameter. A remarkable step towards solving this problem was developed by

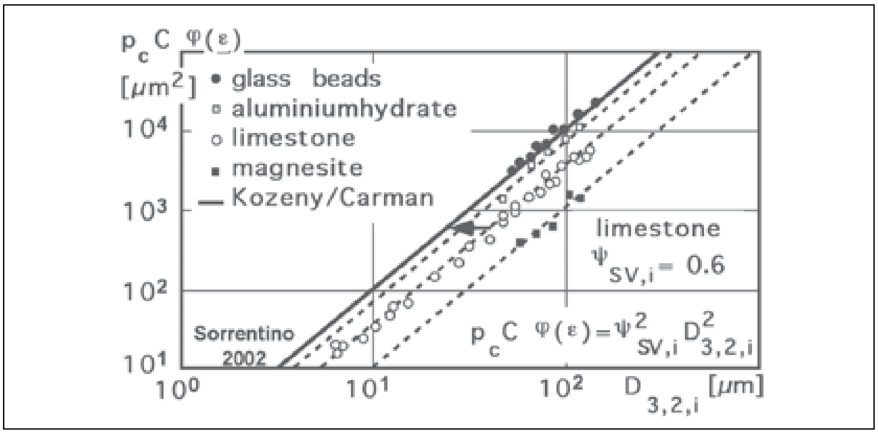

Fig. 4: Determination of the shape factor ψSV,i to determine xSV,i

Sorrentino(7) with the calculation of a "true" Sauter diameter x_{SV} from a Sauter diameter $D_{3,2,i}$ measured with any chosen technology and a corresponding particle shape factor $\psi_{SV,i}$. This shape factor can be determined in filter experiments and particle analysis using a special data analysis as shown in Fig. 4. The product of the specific cake permeability pc, a constant C (180) and a porosity function φ(ε) is applied above the practically measured Sauter diameter $D_{3,2,i}$ for this purpose. Fig.4: Determination of the shape factor ψSV,i to determine xSV,i The shape factor for each test product corresponds to the factor by which the real measuring curve is shifted compared to the ideal theoretical curve according to Kozeny/Carman. And finally, a correlation between particle size and porosity function φ(ε) is required to predict the filtration result of a forecast particle size distribution. This correlation can be calculated from experimental data according to a rule of mixtures by Yu/Standish(8).

Undersaturation of incompressible filter cakes

To be able to displace capillary fluid from the pores of a filter cake with the help of a gas, the pressure difference Δp must be larger on both sides of the filter cake than the capillary pressure effective in the pores p_k. A capillary pressure distribution $p_k(S)$ results in the filter cake due to the pore size distribution. As can be seen in Fig. 5, each pressure difference Δp above the capillary entry pressure p_{ke} allows a fluid displacement up to a corresponding degree of saturation S in equilibrium state.5

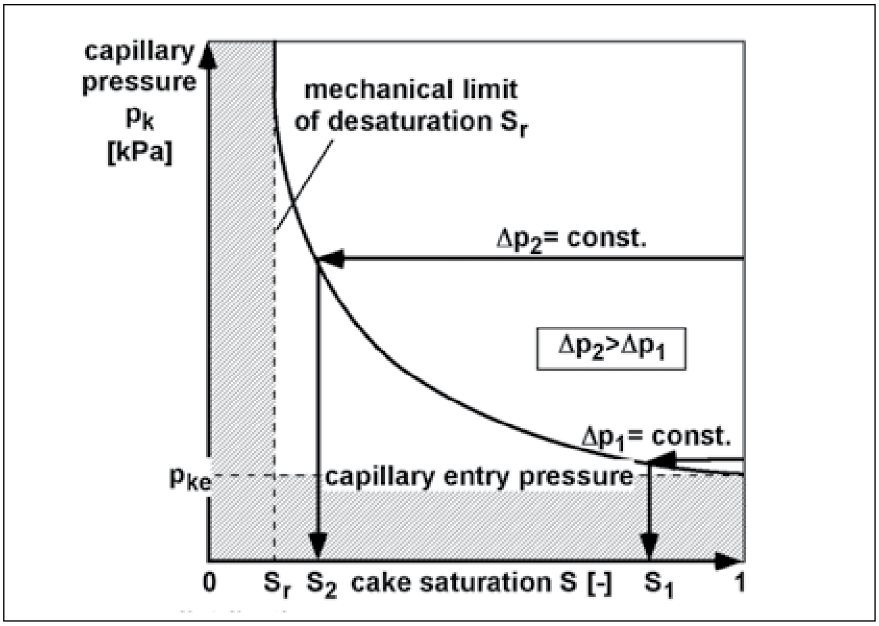

Fig. 5: Capillary pressure distribution

The more homogeneous the cake structure, the better the dehumidification conditions. De-mixing effects through sedimentation-related classification in the suspension lead to the formation of ultrafine grained particle layers with a high capillary pressure on the surface of the filter cake. This has a negative impact on the attainable residual filter cake moisture. A filter cake cannot be completely dehumidified mechanically. Liquid bridges on the contact points of the particles, liquid adhering to the particle surfaces or enclosed in micropores of the particles represent the mechanical demoisturing limit S_r, which can only be undercut using thermal drying. Desaturation of a filter cake can be achieved using differential gas pressure or centrifugal pressure. Both alternatives have advantages and disadvantages, which can be attributed to their physical principles. In case of shrinkage cracks in the cake during demoisturising, it is difficult to maintain the differential gas pressure due to the high gas volume flows. Here, a filter centrifuge would probably be the better choice, because no compressor has to compensate a gas flow through the filter cake. On the other hand, the centrifugal pressure depends directly on the height of the liquid column in the cake. With desaturation of the filter cake in the centrifuge, the capillary system drains, the liquid level sinks and the centrifugal pressure reduces continuously. As soon as the centrifu-

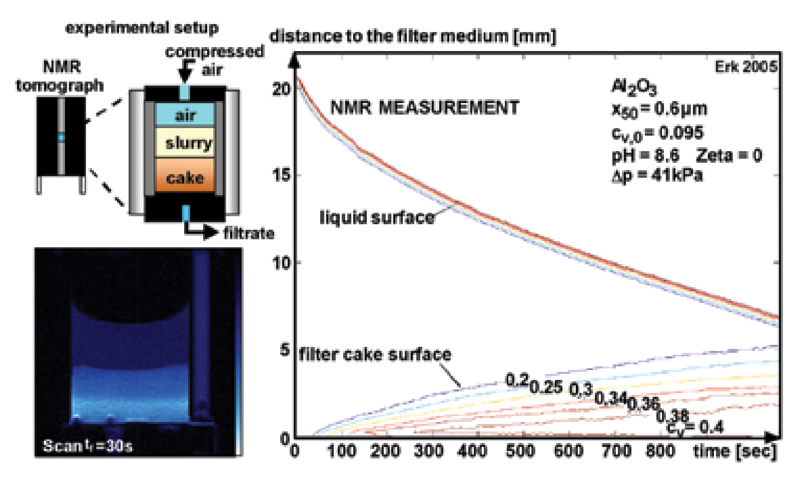

Fig. 6: NMR analysis of a compressible cake filtration

gal pressure and the capillary pressure are equal, the dehumidification process stops and leaves a cake layer completely saturated with liquid. A differential gas pressure on the other hand remains constant during dehumidification and all capillaries in the cake, which can be dehumidified with this pressure, are completely drained(9).

Formation and consolidation of compressible filter cakes

If the particle diameter sinks to less than ≈10µm, the influence of forces on their surfaces and the physical chemistry of the suspension move increasingly into the foreground. The zeta potential is a parameter for the surface charge of the particles and decisive for the stability or agglomeration of a suspension. At the isoelectric point, the zeta potential turns to zero, there are no repulsive electrostatic forces in effect between the particles and the effect of the van-der-Waals adhesive forces can unfold completely. A filter cake with maximum permeability builds up in this case. On the other hand, the filter cake structure is not longer incompressible in this area due to the present pressure difference. This is why a porosity gradient with low porosity forms on the filter medium across the height of the cake and a high porosity on the top side of the cake. In line with the individual product behaviour, compressibility effects are more or less strong. If a filter cake is compressible, rising filtration pressure generally leads to a higher filtrate flow. Compared to an incompressible structure, however, the filtrate flow is reduced, because the cake is compressed at the same time with increasing pressure and its filtration resistance grows.

Therefore, the higher the compressibility, the lower the effectiveness of a pressure increase with respect to the filtrate performance. With ideal compressible systems and relatively high pressures, both tendencies cancel each other and then it is no longer possible to observe a rise of the filtrate flow with increasing pressure. To be able to examine these phenomena in detail and validate theoretical models for their description, more precise and sensitive measuring technologies are required. A very elegant but at the same time extensive technology in this case is NMR (Nuclear Magnetic Resonance). It allows in-situ measurements during a filtration or sedimentation process and a three-dimensional analysis of the filter cake structure and the liquid distribution(10).

Fig. 6 shows NMR measuring results for the filtration of an Al_2O_3 suspension.

In this case, the filter cell was installed directly in the NMR tomograph and the filtration process observed *in-situ*. As shown in Fig. 6, the liquid level in the measuring cell sinks during filtration, while the filter cake grows. Decisive in this case is a direct look at the filter cake structure. A pronounced porosity gradient with a highly compacted layer forms on the filter medium and a very loosely packed cake surface. This was an excellent way to validate mathematical models describing these processes.

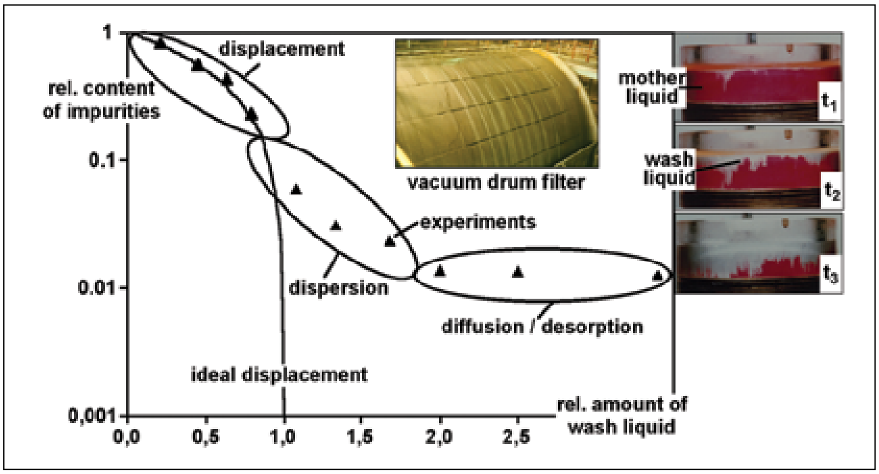

Fig. 7: Results of a permeation washing

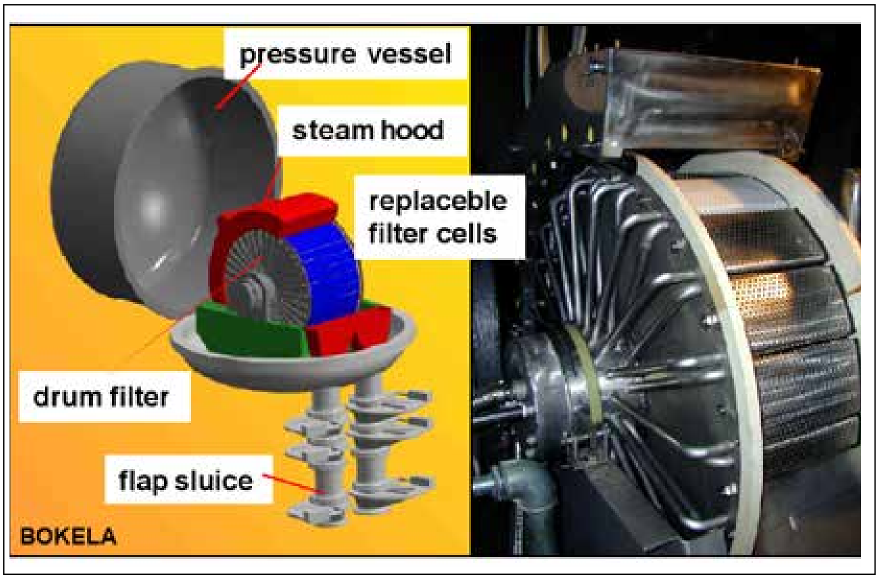

Fig. 8: Steam pressure filtration with a HiBar-Oister filter

Filter cake washing

After the formation of a filter cake, the solid substance must often be post-cleaned in a washing process. This filter cake washing can either be realised via permeation or dilution. With dilution washing, the initially formed and then dehumidified filter cake is resuspended in washing liquid and then filtered again. This process is repeated as long as the demanded purity of the particles is achieved(11).

The most suitable method for the respective separation task must be determined through individual case analysis. The results of a washing process can be illustrated in a washing diagram (see Fig. 7).

In this diagram the remaining content of impurities in the filter cake is plotted as function of the amount of consumed washing liquid. After an initial mere dispersion of the original filtrate (mother filtrate), the washing curve deviates from the ideal curve of mere dispersion due to dispersion effects. Such a dispersion effect can be observed as “fingering” on the right side of the image in Fig. 7.

The likewise displayed image of a vacuum drum filter in Fig. 7 shows another dispersion effect due to the irregular application of the washing liquid. After a certain permeation time, remaining impurities, e.g. from the inside of porous particles or agglomerates, can only be removed through diffusion and/or desorption.

These processes require time and therefore a lot of washing liquid in case of continuous permeation. In order to reduce the washing liquid quantity especially in this last washing stage, it may be advisable to use a dilution washing, interrupt the permeation with a “washing break” (filter presses) or a counter-flow of the washing liquid (vacuum belt filter).

Alternative technologies

To perform a solid/liquid separation task, physically completely different methods and the corresponding separation apparatuses can be applied. The final decision for the optimum system is then made on the basis of technical, economic and other aspects. For example, in order to separate suspensions containing very small particles with a diameter of a few μm in moderate concentrations, there are many different technologies available. Chamber or diaphragm filter presses can be mentioned in the context of cake filtering technology.

Alternatively, decanter centrifuges or disc stack separators can also be applied. The application of crossflow filters would also be feasible. It would only be possible to use depth filters if the particle concentration was extremely slight and the liquid the valuable substance. In case of cake filtration, there is a larger selection of different methods.

Besides the mentioned filter presses, it could be feasible to use a double-belt press, an inverting filter or scraper centrifuge, a pressure nutsch filter, a pressure leaf filter or a hyperbar filter. If a continuous vacuum filter is selected for separating relatively easy to filter suspensions, applicants principally have the choice between drum, disk, belt or pan filters. In the end, different special versions are available for each separation apparatus to allow optimum adaptation to the respective separation task. With a vacuum drum filter, for example, this would be different filter cake removal options like scraper and compressed air blast, roller, leaving filter belt, chains and strings or a progressing knife in case of precoat filtration.

Improvement possibilities for the separation result

Besides suspension pre-treatment methods, e.g. agglomeration, cake filtration may also be substantially improved using a suitable combination of apparatuses. To improve the operating results of a continuously operating pusher centrifuge, it is of advantage to pre-concentrate the suspension by connecting the centrifuge with a static thickener. In this case, the suspension volume to be separated in the centrifuge is reduced through the economic separation of clear liquid from the overflow of the thickener. At the same time, a filter cake can form in the highly concentrated suspension, washed if required and then dehumidified. This is due to the fact that the residence time of the solid substance in pusher centrifuges is generally below 30 seconds. The filtrate, which is cloudy due to the relatively permeable slotted screens in such centrifuges, is returned to the thickener so that the solid substance is completely retrieved.

This so-called cross-arrangement secures the function of the centrifuge on the one hand and improves the separation results with respect to residual product moisture and filtrate clarity on the other. Other principles of combined apparatuses, for example parallel arrangement of identical filters for increasing the throughput or the serial alignment of congeneric apparatuses to improve the separation effectiveness can also be taken into consideration. For complex and frequently changing separation tasks, which occur especially in the pharmaceutical industry, alternatively a process integration, e.g. in a filter reactor, is required. The reduction of the required apparatuses and the integration of as many process steps as possible into one single process chamber minimises hygienic problems and product losses, which are otherwise unavoidable from one process stage to the next.

Another way to improve processes is to improve the construction of the separation apparatus itself. A good example for the latest developments is redesigning the construction of vacuum disk filters for high solid substance throughputs and low residual solid substance moisture(12).

A higher number of filter cells (up to 30) and a deep immersion of the disks into the suspension lead to a filter cake with a uniform thickness and optimum demoisturising conditions. An extremely fast application of the compressed air blast and specially woven filter cloths with a high circumferential elasticity and smooth surface ensure the complete removal even of thin cakes. This allows the operation of such filters at high rotation speeds. Filtrate pipe systems with expanded capacity result in substantially increased solid substance throughputs. Hyperbar filters were developed to separate suspensions effectively, which are comparatively hard to separate with vacuum rotation filters. For hyperbar filtration, modern drum or disk filters are completely installed in a pressure chamber. This makes it possible to realise filtration pressures of up to ≈0.8MPa using gas pressure in the chamber.

Fig. 8 shows one of the most modern types of these apparatuses using the example of a so-called "HiBar-Oister" filter. The variety presented here also contains the option of steam pressure filtration, which has only been available for a short time(13).

As Fig. 8 shows, a part of the filter drum is closed off by a steam hood on the emergence side. Overheated steam with a pressure that corresponds to the internal chamber pressure mechanically disperses the pore liquid from the filter cake and heats it up. A forming condensation layer moves piston-shaped through the cake towards the filter medium. Besides almost ideal piston-shaped and homogeneous liquid dispersion, this method results in an excellent washing effect by overflowing the particle surfaces with hot condensate. After the steam breakthrough on the filter medium, the filter cake leaves the steam hood and air flows through the heated cake. This results in the very effective thermal post-drying of the particles. A remotely similar variant of the combination of mechanical and thermal filter cake removal is used in diaphragm filter presses, where the filter cake can be post-dried thermally after squeezing through application of a vacuum on the partially heatable filter chambers.

Outlook

Although a lot is known about the physical mechanisms of cake filtration as well as the pre and post-treatment methods, a lot of questions are still unanswered due to the complex interaction of the large number of influence parameters and effect mechanisms. This especially concerns the µm and sub-µm range of particle diameters and the hybrid processes in which additional effects through thermal influences, electric or magnetic fields, etc. have to be considered besides the conventional filtration mechanisms. There is a large demand for reliable, predictive process simulation also in the field of cake filtration methods. However, this requires an understanding of the quantitative correlations between parameters like particle size distribution, the physical chemistry of the suspensions, etc., which still has to be intensified.

New application fields for cake filtration methods for the selective separation of nanoparticles in the field of biological and pharmaceutical products, energy saving for reducing CO_2 production or the global demand for clean water pose great challenges on the development of improved or new cake filtration methods. Examples for innovative new processes, which are currently being developed and so far not applied on a large scale, are cake filtration with superimposed magnetic fields for selective active substance separation, cake filtration with semi-permeable microporous membrane filter media for suppressing the gas throughput that occurs in the dehumidification phase or continuous very thin layer filtration methods for particles in the µm and sub-µm range(14).

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