

## **Chapter 2**

### **Mechanical solid-liquid separation processes and techniques**

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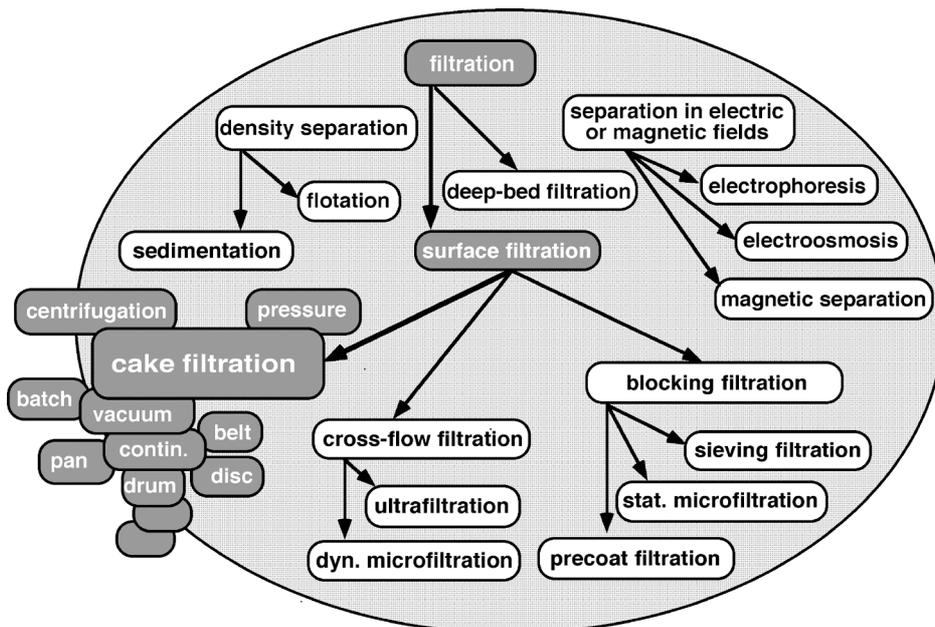
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## 2.1 Introduction and overview

The more or less entire separation of a suspension into the continuous liquid and the disperse solid phase can be realized in principle by thermal or mechanical means. One important aspect of modern drying technology consists in the integration of the process steps before and behind the thermal dryer to gain an overall optimal process design. Thus the mechanical liquid separation as an important process step, which is located upstream the thermal dryer and should be discussed here in more detail. The thermal drying procedures are not discussed further on in this chapter. As a rule the thermal methods are usually quite energy-intensive compared with the mechanical liquid separation because they require a phase transition from the liquid to the gaseous aggregate state and the appropriate vaporization enthalpy must be raised. For this reason and also because of the often undesirable load of the product to be separated at higher temperatures it is mostly advantageous to separate as much liquid as possible mechanically. For physical reasons a final rest of liquid remains in every case after the mechanical liquid separation in the particle structure. This portion of liquid however can only be removed from the solid material by thermal means. If a completely dry powder is required as the final product, one of the tasks for the optimization of the whole separation process consists in determining the most favourable point of transfer from the mechanical to the thermal separation step. This interconnection point is very variable and defined by the requirements of the selected thermal drying process. For a spray drying a still pumpable and sprayable slurry is necessary, whereas the solids should be dehumidified down to the mechanical limit for a fluidized bed drying.

At the interface of these two basic processes for solid-liquid separation also combined mechanical-thermal processes have been developed and established like centrifuge dryers, nutsch dryers and in recent time steam pressure filters (see chapter 2.5). The advantages of these systems consist in synergies, which result in energy conservation, compact and simplified process design.

Figure 2.1 gives a schematic survey of the physical possibilities for mechanical solid-liquid separation.



**Fig. 2.1** Survey of the physical processes for mechanical solid-liquid separation

For each of the shown physical principles different separation techniques can be identified and for each of these an almost unclear number of different separation apparatuses. This is exemplary represented by the cake filtration, which can be carried out for example in the centrifugal field, under the influence of a mechanically produced pressure or by generation of a vacuum behind a filter medium. The respective cake filter can be operated discontinuously or continuously. If one picks out exemplary the family of continuously operating rotary vacuum filters, one can find as basic types drum, disc, belt and pan filters. Each of these apparatuses can be still optimally adapted by special constructive means to the individual task of a separation problem. The reason for this variety is caused by the fact that it is a matter to master mechanical separation processes in an extreme range concerning particle size, size distribution and shape, spec. weight of solids and liquid, suspension concentration, suspension and liquid rheology, flow rate, chemical composition of the suspension, process and technical boundary conditions and last but not least demands on the separation results. For instance the particle diameters can vary from few nanometers to some centimeters. In addition, the general principle of mechanical separation of particles from liquid allows a further differentiation into different special settings of tasks. The primary goal of a mechanical solid-liquid separation process can be distinguished as follows:

- Dehumidification of the solids  
As much liquid as mechanically possible should be displaced from sediments oder filter cakes by squeezing or desaturation.
- Concentration of suspensions  
Only a part of clear liquid is to be withdrawn from a diluted suspension in order to relieve following apparatuses or to make them applicable at all.
- Clarification of a liquid  
As much as possible particle free liquid should be withdrawn from a suspension.
- Classification of solids according to particle size  
A particle collective is to be separated at a defined cut size into a fine fraction and a coarse fraction or a defined particle fraction has to be produced.
- Sorting of solids according to material properties (spec. weight, shape, colour...)  
A particle collective has to be separated according other properties than particle size.
- Washing of solids to remove soluble substances  
To purify a particle system from solute substances a liquid, which has to be molecularly miscible with the suspension is used. One differentiates basically into the permeation washing and the dilution washing. During the permeation washing the wash liquid is filtrated through a packed bed of already separated particles. In the case of the dilution washing the beforehand separated particles are resuspended with wash liquid and separated again. In multistage processes the mode of wash liquid application can be realized as co-current flow or counter-current flow. By counter-current flow operation wash liquid can be saved in comparison to the co-current flow operation, but it is not applicable in every case.
- Three-phase separation of solids and two molecularly not mixable liquids  
In general for this setting of tasks the necessary difference in spec. weight of the materials involved are utilized.

- Extraction and following phase separation

Here the same density separation processes are applied like for the three phase separation. Additionally an intensive mixing of extraction agent and suspension to be extracted has to be realized.

Figure 2.1 illustrates, that the apparative variety can be arranged nevertheless systematically into physically against each other well defined separation processes. The mechanical separation processes can be split basically into three main groups.

The density separation processes are utilizing a difference in the specific weight of solids and liquid for the separation of particles by sedimentation in the direction of and by flotation against the earth gravity or a centrifugal field (see chapter 2.2).

The filtration processes are characterized basically by the flow of the liquid phase through a porous filter medium due to a pressure difference, whereas the solid particles are held back. One differentiates thereby between the procedures of depth filtration (see chapter 2.3.4) with which the particles to be separated are deposited inside the structure of a filter layer and the procedures of surface filtration with which the particles are retained at the surface of a filter medium. The surface filtration procedures are made up of cake, cross-flow and blocking filtration.

The cake filtration is characterized by the fact that the solids are deposited as a macroscopic porous layer on the filter medium, while the liquid must flow through the already formed filter cake and the filter medium. In subsequent post-treatment steps the filter cake can be washed first and afterwards desaturated in the case of incompressible or squeezed in the case of compressible cake structure. In comparison to other filtration procedures the cake filtration permits extensive dehumidification of the solids. In order to be realized economically, cake filtration requires however the fulfillment of certain boundary conditions like sufficiently high suspension concentration and particle size (see chapter 2.3.1).

In comparison to cake filtration the procedures of cross-flow filtration are based on tangential suspension flow over the filter medium, which consists usually of a microporous membrane. In this manner a filter cake formation is avoided nearly totally and the suspension can be concentrated up to its limit of flowability. This technique is applied to separate very small particles and highly diluted suspensions, which would lead to uneconomically high filter cake resistances and long filtration time in the case of cake filtration (see chapter 2.3.3).

As an alternative to filter highly diluted suspensions containing very small or even coarse particles the blocking filtration can be applied. Here one permits the gradual blockage of the filter medium pores by single particles and after reaching a defined critical pressure loss, the particles are detached for example by a back-flushing procedure (see chapter 2.3.2).

Finally the particle separation can be influenced by applying an additional electric or magnetic field, if the particles show an appropriate material behavior (see chapter 2.4).

Modern mechanical solid-liquid separation technology includes beside the versatile apparatus spectrum further important possibilities to optimize the separation process. One of these possibilities consists in a suspension pre-treatment to make the subsequent separation easier (see chapter 2.6.3). Furthermore has to be checked whether a batchwise or a continuously operating separation process should be chosen (see chapter 2.6.1). Both modes of operation feature special advantages and disadvantages. In the rarest cases a solid-liquid separation process is based on

one single apparatus. Very often an apparatus combination is the best choice to gain the physically and economically best solution despite comparatively larger expenditure for equipment (see chapter 2.6.2).

Last but not least nearly for each separation problem several alternatives are existing and only a careful and deep analysis of all relevant aspects lead to optimal results. For the choice of a separation process in a first step the separation problem has to be analyzed and a requirement specification regarding the separation results has to be formulated. This leads to first ideas for eventually suited processes. The second step includes generally bench scale separation experiments, to verify the principle feasibility of these hypotheses and to get the necessary data basis for scaling up considered types of apparatuses. On the basis of the laboratory results and already comprehending economic and other superordinate aspects in most cases a pilot scale test must be carried out. Now specific apparatus parameters can be investigated, which can not be captured by the simple bench scale test. From the successful realized pilot test the final quantitative scale up to the process scale can be done.

The procedures and apparatuses for the mechanical solid-liquid separation are under permanent research and development in view of new challenges and requirements to the separation results (Anlauf, 2006; Anlauf, 2007a; Anlauf, 2007b; Anlauf, 2008; Höflinger, 2008; Kopf et.al., 2008; Lyko, 2008; Ripperger, 2008). Actual results from the research are used permanently for the apparatus construction. New materials and production engineering is used. Modern sensor and data transfer technology allows the remote monitoring of separation processes and a result-dependent control and regulation. The trend goes to the „intelligent“ machine, which reacts to changes of the feed conditions automatically. However on the way to that target a certain number of today still unsolved questions of basic research in this field have to be answered. One of these questions is the quantitative correlation between filter separation results and real particle collective characteristics (Sorrentino, 2002).

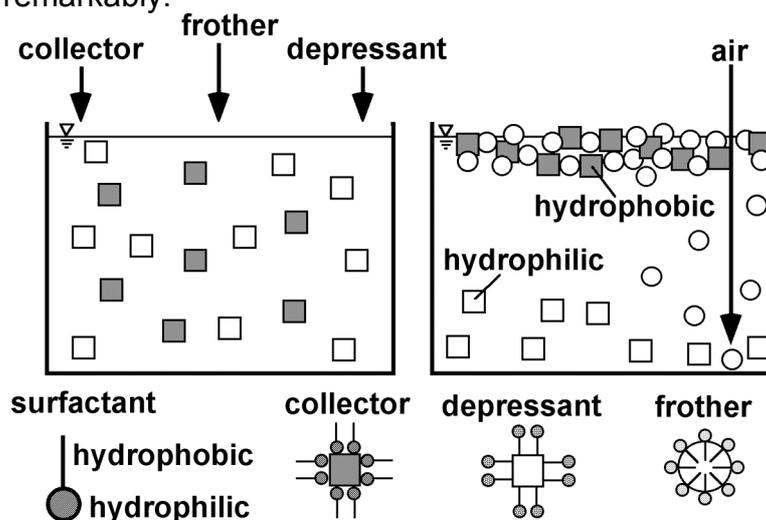
General information about theory, processes and apparatuses of mechanical solid-liquid separation in english language can be found in several books (Jornitz, Meltzer, 2001; Dickenson, 1997; Leung, 1998, 2007; Rushton, Ward, Holdich, 1996; Sutherland, 2005; Svarowski, 2000; Wakeman, Tarleton, 2005a, 2005b, 2007).

## **2.2 Density separation processes**

### **2.2.1 Froth flotation**

Flotation processes generally are based on the fact, that suspended particles have a smaller spec. weight than the surrounding liquid phase and thus are rising up against the direction of earth gravity or a centrifugal field. If the particles show a larger spec. weight than the surrounding liquid phase, they nevertheless can rise up against gravity to the top of a so called „flotation cell“ as a froth by adhering gas bubbles. Figure 2.2 illustrates the general principle of froth flotation. Industrially above all flotation in aqueous suspensions is used for sorting particles of different material. Flotation is used in particular in the ore, mineral and coal processing to separate fine grained particle mixtures of liberated minerals and tailings. In the field of paper recycling flotation is used for deinking of the pulp. However, flotation is also applied as energetic favourable and low-cost process for total separation of organic particles, which have only small difference in spec. weight to the surrounding liquid. This is used increasingly in the sewage water treatment as an alternative to sedimentation

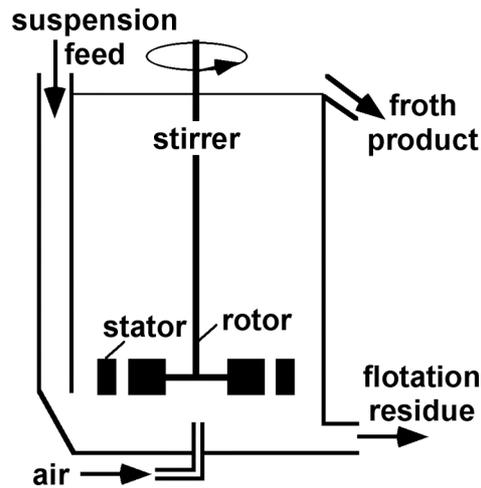
processes. The required clarification area can be reduced in that way under circumstances remarkably.



**Fig. 2.2** Principle of the froth flotation process

In order to enable the attachment of gas bubbles to the particle surfaces, these must be hydrophobic. If they are not hydrophobic by nature, they can be made hydrophobic by adding special surfactants, which are called „collector“. Surfactants or tensides are boundary surface active molecules, which consist of a hydrophobic nonpolar hydrocarbon chain and a hydrophilic polar group. According to the kind of polarity one differentiates between anionic, cationic and nonionic surfactants. Anionic collectors are among others xanthanates, carboxylates, alkylsulfates or mercaptanes. Cationic collectors are alkylamines. The particles not to be discharged with the froth must show hydrophilic surface properties and are settling in the direction of gravity to the bottom of the flotation cell. If they are not hydrophilic by nature, they can be made hydrophilic by special surfactants, which are called „depressants“. As depressants alkali cyanide, lime hydrate, zinc sulphate or water glass are used among other substances. The froth itself can be stabilized against coalescence by so called „frothers“. As frothers are used polypropylene glykol or aliphatic alcohols among other substances. The optimally floatable particle diameters are in the range between 40  $\mu\text{m}$  and 150  $\mu\text{m}$  for solids spec. weights of more than 3000  $\text{kg m}^{-3}$ . Upwards the flotation process is limited by the particle weight and downwards by the reducing selectivity. Due to the ascending particle loaded bubbles three-phase froths are originating in a flotation process. Into the froth product not only the particles adhering at the bubbles by heteroagglomeration are present but also a small amount of finest hydrophilic particles. This phenomenon limits the selectivity of the process. Generally several process steps like basic flotation, secondary flotation and purification flotation are necessary to produce the final solids concentrate. From the apparative point of view one differentiates between the mechanical and the pneumatic procedures. Furthermore special constructions like pressure release flotation and electro-flotation (see chapter 2.4) are existing. Flotation apparatuses in general are working continuously.

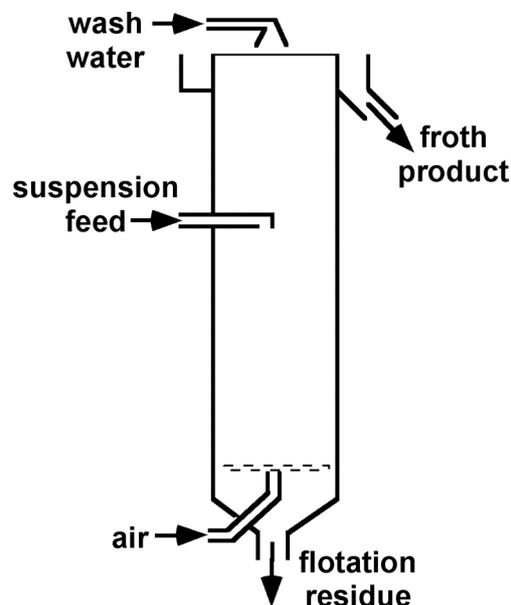
With the mechanical flotation apparatuses the necessary energy for mixing and dispersing is supplied according to Fig. 2.3 by means of rotor/stator systems to the aerated suspension.



**Fig. 2.3** Mechanical flotation apparatus

The air is sucked into the liquid through the hollow shaft of the mixer itself or is supplied from outside by means of a nozzle, then dispersed in the rotor/stator system into small bubbles and mixed with the suspension under highly turbulent flow conditions. After turbulent dispersion and mixing the froth must have the possibility to rise up under calm conditions.

With the pneumatic flotation apparatuses no rotor/stator system exists. The required air is supplied here in every case from the outside under pressure and dispersed into bubbles by means of a suitable aeration system. In Fig. 2.4 such a flotation column is shown. The height of flotation columns can vary from 5 m to 15 m with a diameter of up to 4 m. Here gas and suspension are fed in the countercurrent. This leads to an improved selectivity compared with the mechanical flotation systems, which show a cross-flow principle.



**Fig. 2.4** Pneumatic flotation apparatus

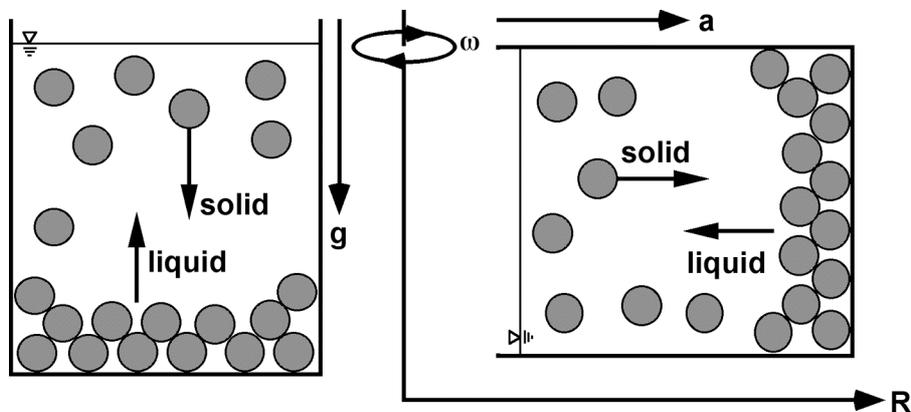
With the modern process of pressure release flotation the pressure dependence of the gas solubility in water is utilized for the bubble generation. The water is saturated under increased pressure with air and afterwards released to the atmospheric pressure. In comparison to mechanical and pneumatic flotation with bubble diameters of more than 100  $\mu\text{m}$  here particularly small bubbles from approximately 50  $\mu\text{m}$  to 80  $\mu\text{m}$  diameter can be produced for relatively high pressures from

approximately 450 kPa to 700 kPa. The bubbles originate partly direct at the particle surfaces, where the nucleus formation work is lowest.

Further and more detailed informations about froth flotation can be found in Rao (2004) and Fuerstenau, et. al. (2007)

### 2.2.2 Sedimentation

The setting of tasks for sedimentation procedures to separate solids from liquid range from concentration or clarification of dilute suspensions to extensive dehumidification of the separated solids. Also classification and sorting can be realized. A specific feature of sedimentation procedures exists in the possibility to separate suspensions into their components, which contain beside the solid particles two molecularly not miscible liquids of different spec. weight (water/oil). With the sedimentation procedures the solid particles are separated according to Fig. 2.5 in contrast to the flotation in the direction of gravity  $g$  or a centrifugal acceleration  $a$  towards a solid and impermeable wall.



**Fig. 2.5** Principle of sedimentation

While the acceleration due to gravity is handled as constant, the centrifugal acceleration depends on the rotor radius  $R$  and the angular velocity of the rotor  $\omega$ :

$$a = R \cdot \omega^2 \quad (2.1)$$

Although the angular velocity of a centrifuge may remain constant the acceleration of a settling particle increases due to the increasing radius. Often can be calculated exactly enough with a constant mean centrifugal acceleration, if the centrifuge radius is large in relation to the thickness of the liquid layer in the rotor („long arm approximation“). A dimensionless centrifugal factor  $C$  indicates the multiple of acceleration due to gravity, which can be realized in the respective centrifuge and serves to compare the efficiency of centrifuges.

$$C = \frac{a}{g} = Fr \quad (2.2)$$

This characteristic number is also known as Froude number and is used in mixing, agglomeration and comminution technology for rotating apparatuses.

In the case of sedimentation the spec. weight of the particles  $\rho_s$  is always larger than the spec. weight of the surrounding liquid phase  $\rho_L$ . The liquid displaced by the downwards settling particles must move upwards. Apart from the density difference between solids and liquid  $\Delta\rho$  and the acceleration  $g$  or  $a$  the settling velocity  $v$  is

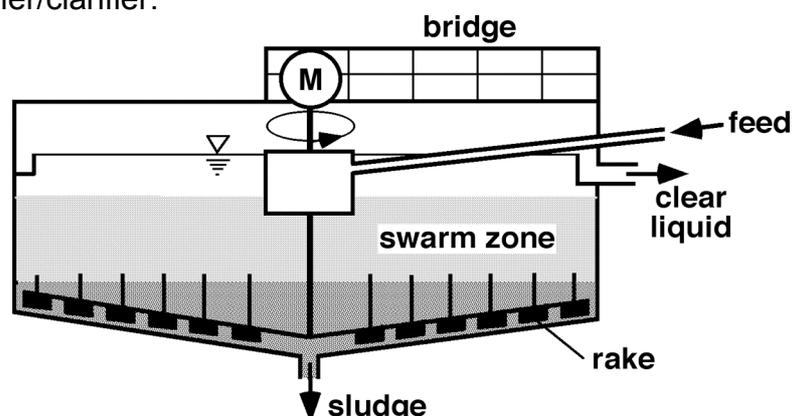
influenced by the dynamic viscosity of the liquid  $\mu_L$ , the particle diameter  $d$  and the solids volume concentration of the suspension. For the settling velocity  $v_{St}$  of single (highly diluted suspension) spherical particles, laminar flow and Newtonian fluids the Stokes-law is valid:

$$v_{St} = \frac{\Delta\rho \cdot g \cdot C \cdot d^2}{18 \cdot \mu_L} \quad (2.3)$$

If one assumes simplifying constant acceleration for a centrifuge, the centrifugal settling velocity can be calculated easily by multiplication of the settling velocity in the earth field with the C-value. If the solids concentration increases, the particles are hindering each other during sedimentation more and more and are moving finally independently of their individual characteristics with the same velocity. A sharply distinctive sedimentation front is forming with no particles in the clear liquid zone above. This phenomenon is called „swarm sedimentation“. The settling velocity of the swarm is sensitively dependent on the suspension concentration. When a sediment has formed by the settling procedure, a further displacement of liquid can take place only by compression (consolidation). This consolidation results from the dead weight of the particle layer, which is exposed either to gravity or a centrifugal field. The pores of the sediment however remain completely filled with liquid. The first layers of the sediment are most strongly consolidated. At the surface of the sediment no further compression takes place. Thus a nonlinear concentration (porosity) gradient is forming over the sediment height.

A physical limit for sedimentation in the earth field is set for a particle size of approximately  $1 \mu\text{m}$ , because thermal convection and Brownian motion are keeping smaller particles normally stable in suspension. Nevertheless by means of agglomeration (see chapter 2.6.3) one can make however also smallest particles accessible to gravity sedimentation. If a change of suspension properties is not permitted, the mass forces must be increased by changing from earth to centrifugal field.

Technically for gravity sedimentation predominantly continuously operated circular and rectangular basins as well as lamella clarifiers are used. Figure 2.6 shows a circular thickener/clarifier.

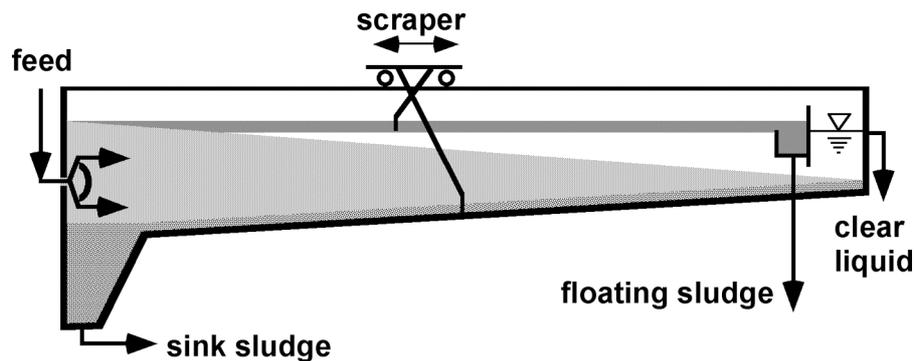


**Fig. 2.6** Circular sedimentation basin

The suspension is supplied centrally by means of a feed pipe and then spreads radially in the basin. Swarm sedimentation behavior of the suspension is aimed for to realize a sharp sedimentation front, which separates the clear liquid and the sedimentation zone. If this behavior is not given by the feed conditions, flocculation helps to enforce swarm sedimentation. In addition the smallest particles are bound in

the floc structure and the overall sedimentation velocity increases. With modern high efficiency basins the suspension is lead into the swarm zone, which then works like an dynamic filter for the ascending clear liquid. The sludge collected at the bottom of the basin is transported by means of a slowly rotating rake machanism towards the central discharge opening. To increase the efficiency of the apparatus thin sticks can be installed on top of the rabble arms, which are cutting drainage channels into the sediment. Thus it consolidates faster and the sludge concentration can be increased. Large circular sedimentation basins are built in concrete construction up to diameters of approximately 200 m.

Figure 2.7 shows a rectangular sedimentation basin. In this case suspension flow and settling direction are perpendicular to each other.

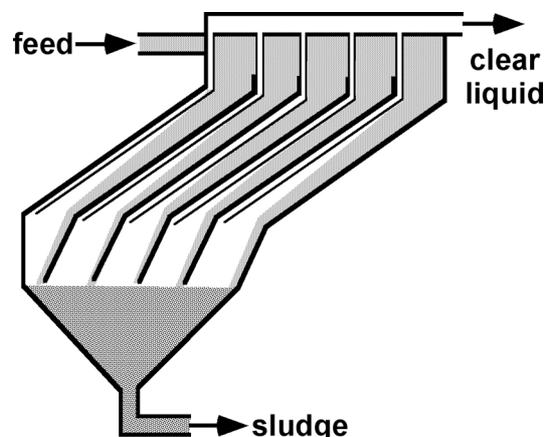


**Fig. 2.7** Rectangular sedimentation basin

In Fig. 2.7 is to be recognized, that density separation processes are well suited for multiphase separation. Also floting substances can be separated here by a special discharge construction, which separates floting sludge and clarified liquid.

With given feed stream the separation condition for the particles can be adjusted by the so called „clarification area“ of the basin. The feed volume flow rate divided by the clarification area determines the up-flow velocity of the liquid, which must be smaller than the sedimentation velocity of the particles. The continuously operated apparatuses for gravity sedimentation need realtively large clarification areas because of the usually low settling velocity of the particles.

Very much clarification area on a small footprint area can be made available by a lamella clarifier like demonstrated schematically in Fig. 2.8.



**Fig. 2.8** Lamella clarifier

Angular arranged plates in the settling chamber lead to a drastic shortening of the necessary settling distance for the particles until the are separated. A precondition for

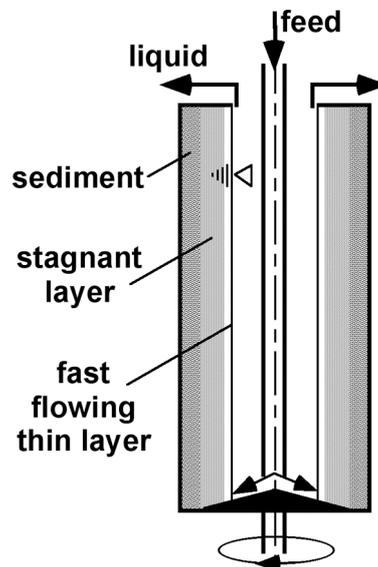
the operability of this apparatus is, that the separated particles are able to slide down the plates and are not sticking on their surface. This can be ensured within certain limits by adjustment of the plate inclination.

Alternatively to the gravity sedimentation processes sedimentation centrifuges can be chosen. For this purpose the following basic types of centrifuges are available:

- Beaker centrifuges
- Tube and overflow centrifuges
- Hydrocyclones
- Decanter centrifuges
- Disc stack separators

In the laboratory for analytic purposes and preparation of small product samples discontinuously operating beaker centrifuges can be used.

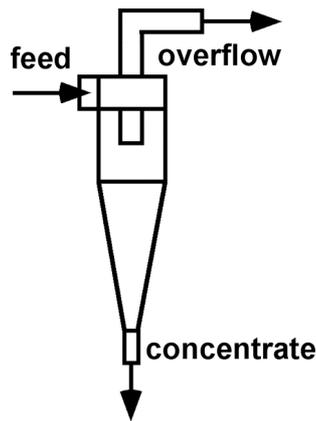
For extremely difficult separation conditions tube centrifuges with centrifugal values  $C$  of up to ca. 50.000 are applied. Figure 2.9 shows the principle of such a centrifuge.



**Fig. 2.9** Tube centrifuge

This type of centrifuge operates quasi-continuously but discontinuously in reality. During permanent feeding through a central feed pipe a sediment is growing up in the rotor. On top of the sediment a stagnant zone of liquid is standing, which is overflowed by a thin and fast moving liquid layer. As soon as the particles are entering the stagnant zone they are to be considered as separated and are settling down to the surface of the sediment. If the sediment has achieved a certain critical height, the centrifuge must be shut down in most cases, dismantled and released manually from the solids. As an extreme case for the pharmaceutical industry a tube centrifuge for only single use is known. On the other hand some types of tube centrifuges are equipped with an automated mechanical sediment discharge. Similar types of discontinuously operating centrifuges for much lower  $C$ -values with automated or manual solids discharge are known from the cleaning of cooling lubricants and similar slurries

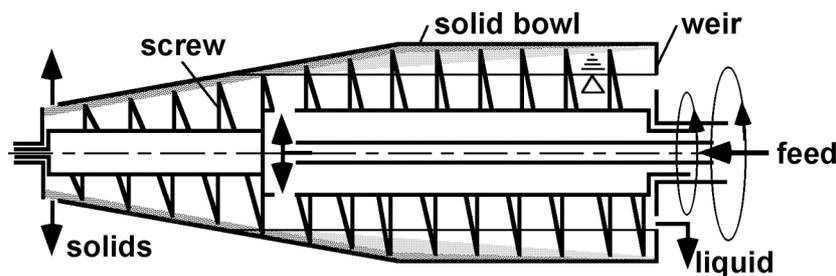
As first representative for continuously operating centrifugal separators the hydrocyclone should be named. According to Fig. 2.10 hydrocyclones are very simple designed apparatuses.



**Fig. 2.10** Hydrocyclone

The suspension to be separated is accelerated here not due to the rotation of a solid bowl but by tangential injection under pressure. The liquid is forced by the tangential inlet into the cylindrical/conical process room on spiral circular paths. Thus particles up to a certain cut size are settling outwards through the appearing potential vortex, separated at the solid wall of the cylinder and discharged as a concentrate through the underflow apex orifice. The smaller particles are following the streamlines and are removed together with the main part of the liquid through a cylindrical tube fixed in the center of the top and projecting some distance into the cyclone. This called the overflow pipe is called „vortex finder“. In this way both a separation and in particular a classifying of the particles can be realized. The centrifugal acceleration is determined by the injection pressure and the cyclone diameter. Maximum pressure losses of ca. 400 kPa and minimum cyclone diameters of ca. 10 mm limit the cut size to ca. 5µm. Therefore, for large amounts of liquid and small cut size several small cyclones must be arranged in parallel. An agglomeration to improve separation is not meaningful in hydrocyclones. The strong shear stresses due to the vortex flow destroy the previously formed agglomerates. Because of the simple construction hydrocyclones can be built of very different materials like metal, polymers or ceramic and thus optimally adapted to the conditions of a process.

Figure 2.11 shows the principle of a decanter centrifuge in standard design.

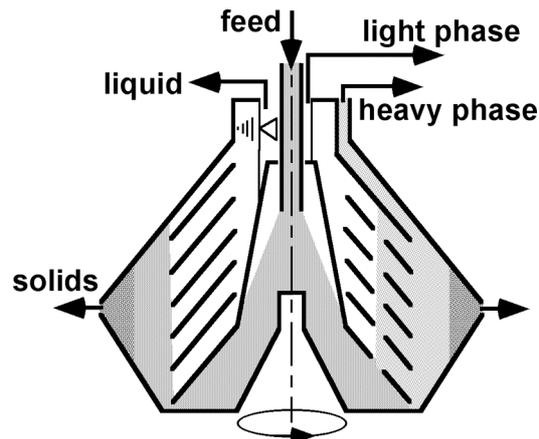


**Fig. 2.11** Decanter centrifuge

In contrast to the hydrocyclone the suspension is fed here into a rotating cylindrical/conical solid bowl by an axially arranged inlet pipe. A liquid layer („pond“) is formed, which can be adjusted in its height by a weir at the end of the cylindrical part of the drum. In modern centrifuges the weir height can be adjusted directly during operation by different technical systems. Over the weir the clarified liquid is removed from the centrifuge. The settled solid particles must be transported along the solid wall of the bowl to the opposite side of the centrifuge by a screw. This requires a differential speed of screw and solid bowl. The differential speed can be adjusted by different types of special gearings. During the transport from the cylindrical to the conical part of the bowl the solids are leaving the liquid level

(„beach“) and thus some liquid is able to drain back to the pond. In decanter centrifuges the separation can be strongly improved by agglomeration of the particles to be separated, because they can settle with comparatively low stress. Decanter centrifuges can be used by constructive adaption very flexibly for the three-phase separation, extraction, sorting and partly for classifying (Anlauf, H., 2007b). Decanter centrifuges can reach centrifugal values  $C$  up to ca. 5000 and are built to drum up to 1800 mm.

If the decanter centrifuge represents the translation of the gravity settling basin into the centrifugal field, then the principle of the disc stack separator represented in fig. 2.12 corresponds to the gravity lamella clarifier.



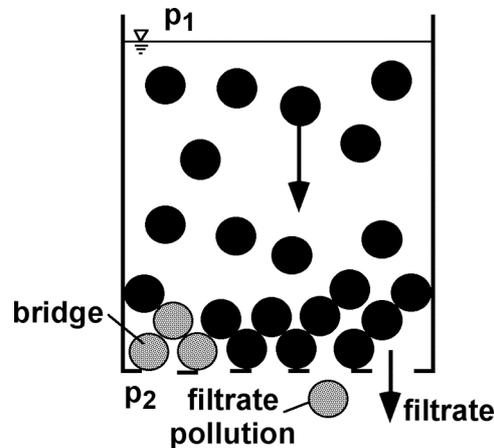
**Fig. 2.12** Disc stack separator for solid-liquid separation (left side) and Solid-liquid-liquid separation (right side)

The clarification area is increased in this centrifuge remarkably by a plate package. The centrally by a feed pipe supplied suspension flows in the case of solid-liquid separation (Fig. 2.12, left side) from the outer radius through the plate package to the inner radius of the centrifuge, where the clarified liquid is discharged. The solid particles are settling to the outer radius of the double conical drum and collected there. The consolidated however still flowable sludge is discharged from there depending on its amount either by permanently open or periodically opened nozzles. If beside a solid material a second liquid has to be separated (Fig. 2.12, right side) then the plate package shows some rising channels, which are distributed equally on the circumference. Their radial position corresponds to the volume ratio of the two liquids to be separated. The supplied suspension penetrates into the rising channels and the lighter liquid is led inwards. The heavier liquid and the solids are moving outwards. Light and heavy liquid are discharged by separate openings. Disc stack separators can realize centrifugal values  $C$  of up to ca. 15000. Due to the large clarification area in connection with high centrifugal values a disc stack separator can replace gravity clarification surfaces of up to 300.000 m<sup>2</sup> on very small foot print area and separate even very small particles of less than 1  $\mu\text{m}$ . Disc stack separators are used to separate in the biotechnological and pharmaceutical sector often not only very small but also very sensitive particles. Modern disc stack separators are equipped with special suspension feeding and solids discharge systems for gentle particle treatment. The suspension is not given on the surface of the fast rotating liquid in the centrifuge but directly into the liquid in order to realize a smooth acceleration. The solids are discharged through self-regulating nozzles near the axis of rotation to avoid the high pressures up to 20 MPa for nozzle discharge at the outer periphery. The self-regulation of the nozzles are furthermore compensating changing feed conditions and guarantee constant separation results (Anlauf, 2007 b).

## 2.3 Filtration

### 2.3.1 Cake filtration

With cake filtration the particles are separated according to Fig. 2.13 on the surface of a porous filter medium. The liquid flows as filtrate through the pores of the cake and the filter medium.



**Fig. 2.13** Principle of cake filtration

Particles and liquid are moving due to a pressure difference  $\Delta P$  between the absolute pressure at the suspension surface  $p_1$  and the pressure underneath the filter medium  $p_2$  ( $p_1 > p_2$ ) in the same direction. This pressure difference can be generated pneumatically, hydraulically, mechanically or by a centrifugal field. In order to avoid a blockage of the filter medium its pores usually are chosen so open, that during a very short time at the very first moment of the filtration single particles are able to get into the filtrate before particle bridges are forming across the meshes and sealing them. On top of these bridges then the filter cake grows up without further filtrate pollution. Cake filtration procedures are applied for moderate to higher concentrated suspensions within the particle size range of ca.  $1 \mu\text{m}$  to  $1000 \mu\text{m}$ . Smaller particles cannot be separated meaningful by cake filtration due to the arising extremely high pressure losses. However the range of application for cake filtration processes can be shifted by particle agglomeration to even smaller particles (see chapter 2.6.3). Larger particles separate spontaneously by gravity on screens and are dehumidified on a dump.

The calculation of filtration processes is generally based on the law of Darcy, which describes laminar flow of liquids through porous layers:

$$v = \frac{\Delta P}{\alpha \cdot H \cdot \mu_L} \quad (2.4)$$

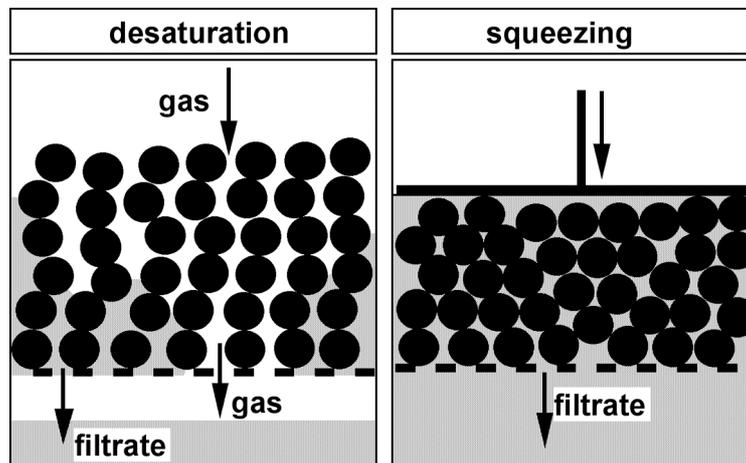
The flow velocity  $v$  (empty pipe) is depending on the effective pressure difference  $\Delta P$ , the layer thickness referring spec. flow resistance  $\alpha$ , the layer thickness  $H$  itself and the dynamic viscosity of the liquid  $\mu_L$ . This generally formulated law has to be adapted and modified for the special conditions of each filtration process. In that way the law of Darcy also can be adapted to the conditions of compressible gases and the two phase flow of liquid and gas during cake desaturation or the liquid permeation during cake washing. During the cake formation phase the porous filter medium as well as the increasing filter cake are permeated by the liquid. From the law of Darcy the time  $t$  necessary to form a certain cake height  $H$  can be derived:

$$t = \frac{H^2 \cdot \alpha \cdot \mu_L}{2 \cdot \kappa \cdot \Delta P} + \frac{H \cdot \beta \cdot \mu_L}{\kappa \cdot \Delta P} \quad (2.5)$$

Here the flow resistance of the filter medium  $\beta$  is taken into consideration. The concentration parameter  $\kappa$  is calculated from the data of the solids volume concentration of the suspension  $c_V$  and the cake porosity  $\varepsilon$ :

$$\kappa = \frac{c_V}{1 - \varepsilon - c_V} \quad (2.6)$$

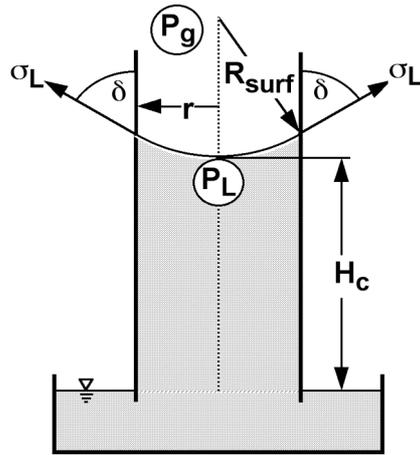
Cake and cloth resistance have to be determined experimentally. An appropriate measuring regulation gives the VDI guideline No. 2762. After the step of the filter cake formation follows, if necessary, a cake washing procedure in order to remove soluble components of the suspension liquid from the filter cake. For this purpose the filter cake is either resuspended in washing liquid and filtered again or is permeated by the washing liquid (see chapter 2.1). After the washing or directly after the formation follows a mechanical dehumidification of the filter cake follows. As can be seen in Fig. 2.14 two principally different possibilities of cake dehumidification can be distinguished.



**Fig. 2.14** Mechanisms of filter cake dehumidifying

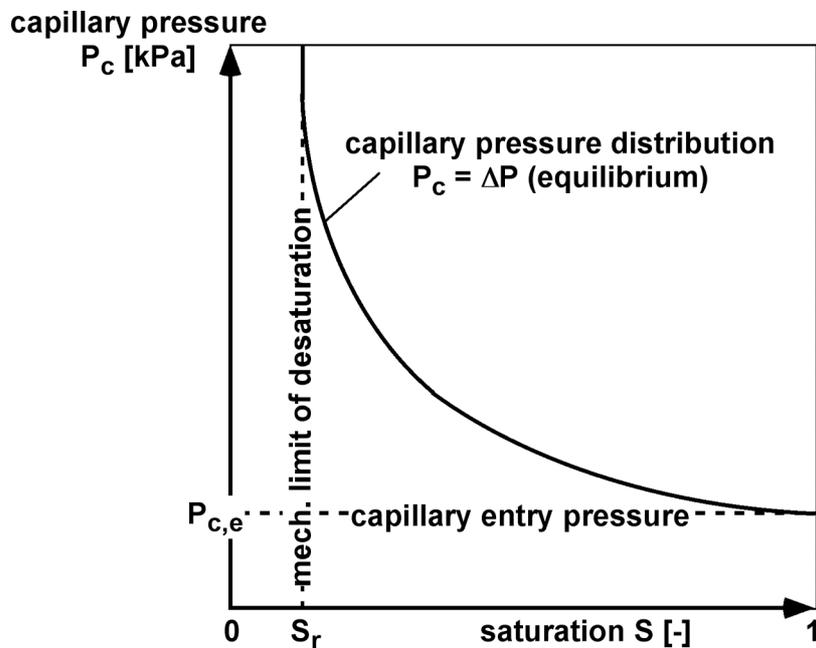
These are desaturation (Fig. 2.14, left side) and squeezing (Fig. 2.14, right side). For particles with diameters of more than ca. 10  $\mu\text{m}$  or if particle adhesion forces do not matter nearly incompressible filter cake structures are formed. In this case the pore liquid can be displaced by gas (Fig. 2.14, left side). From the origin of a boundary surface between liquid and gas capillary forces are resulting, which hold the liquid back in the pores of the cake. If in the case of pneumatic pressure the external pressure difference is larger than the capillary pressure, gas penetrates into the pores and displaces liquid until an pressure equilibrium is reached. The filter cake becomes desaturated and a two phase flow of gas and liquid develops. At the contact points of the particles, on their surfaces and in eventually existing inner pores some liquid remains, which can not be further removed mechanically. The capillary pressure  $P_c$  of a circular pore depends on the surface tension of the liquid  $\sigma_L$  and the radius of the boundary surface curvature  $R_{\text{surf}}$ , which is correlated with the wetting angle  $\delta$  and the capillary radius  $r$ , as is to be seen in Fig. 2.15. The force balance at the boundary surface can be formulated by the Laplace-equation:

$$P_c = \frac{2 \cdot \sigma_L \cdot \cos \delta}{r} \quad (2.7)$$



**Fig. 2.15** Capillary pressure in a circular pore

In the equilibrium state the liquid rises up to the capillary height  $H_c$ . The distribution of pore sizes in a filter cake entails a capillary pressure distribution. It is characterized by the capillary pressure curve shown in Fig. 2.16.



**Fig. 2.16** Capillary pressure curve

This function has to be determined experimentally and provides the information, which minimum saturation degree  $S$  appears in the filter cake for the respective applied pressure difference  $\Delta P$ . The saturation degree  $S$  is determined from the relationship of liquid volume  $V_L$  and void volume  $V_V$  in the filter cake:

$$S = \frac{V_L}{V_V} \quad (2.8)$$

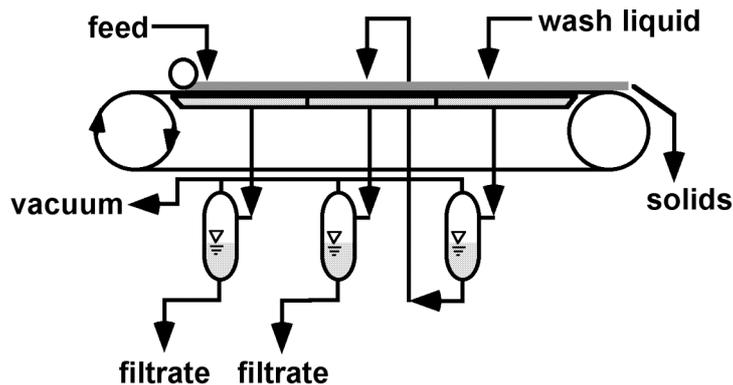
The capillary pressure curve is limited downward by the capillary entry pressure  $P_{c,e}$ , which must be overcome at least to initiate a first desaturation. The mechanical limit of desaturation  $S_r$  cannot be fallen below with any increase of the pressure difference. If in particular with small particles with diameters of less than ca.  $10\mu\text{m}$  adhesion forces are acting between the particles, a noticeably compressible filter cake structure is formed. In this case the cake can be squeezed (Fig. 2.14, right

side). Thus liquid from the pores of the filter cake is displaced although it remains completely saturated. The pressure for cake consolidation can be generated

- hydraulically by pressing additional suspension into an already with cake filled filter chamber,
- mechanically by a diaphragm or a piston or by
- mass forces in a centrifugal field.

The procedures of cake filtration can be distinguished systematically according to the kind of the filtration pressure.

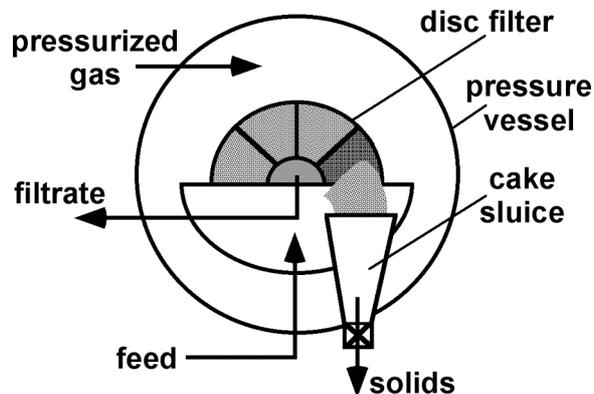
Gas differential pressure can be generated both by creation of a vacuum behind the filter medium or by application of an overpressure above the suspension. While vacuum filters are limited by the vapour pressure of the liquid on pressure differences below 0.1 MPa, gas overpressure in technical efficient limits can be chosen freely. The technical upper limit for encased gas overpressure filters is given at ca. 1 MPa. The pressure difference remains usually constant during filtration. Modern vacuum filters today are only built for continuous mode of operation. Discontinuous vacuum filters like the Buchner-funnel are used only in the lab. Drum filters, disc filters, belt filters and pan filters are the main types of technically used continuous vacuum filters. Figure 2.17 shows exemplarily a vacuum belt filter.



**Fig. 2.17** Vacuum belt filter

This type of filter is particularly well suited for filter cake washing, however it needs comparatively much floor space for installation.

Drum and disc filters are not only used as vacuum filters but are operating encased as overpressure filters (hyperbar filters). Figure 2.18 shows a disc filter installed completely in a pressure vessel.



**Fig. 2.18** Hyperbar disc filter

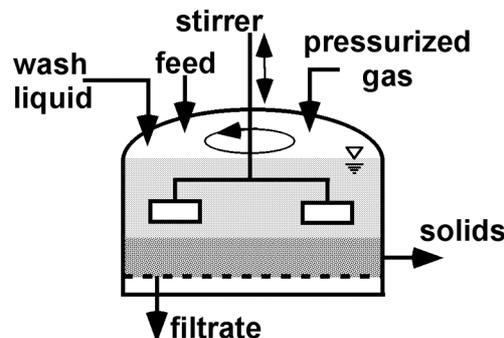
The filter disc consists for modern filters of up to 40 filter segments, which are filtering on both sides. The slender segment shape leads to a very homogeneous filter cake. On the central filter shaft several filter discs can be mounted one behind the other to increase the capacity of the filter unit.

In that way up to 200 m<sup>2</sup> of filter area can be installed in one pressure vessel. The special advantage of disc filters is the high capacity whereby drum filters are exceptionally flexible to adjust to different slurry and cake behavior.

On a drum filter rectangular filter cells are arranged axially on the surface of the horizontally mounted filter drum. To react to different cake consistency various possibilities for a safe cake discharge are available. This is unique for drum filters.

Pan filters are used for fast settling coarse particles and correspond in their construction to a 90 degree turned and horizontally arranged disc filter with one single disc.

In comparison to vacuum filtration overpressure filters are operating continuously and batchwise. The stirred pressure nutsch filter represented in Fig. 2.19 is an example of batchwise operating gas overpressure filters for the separation of relatively easy to filter suspensions.



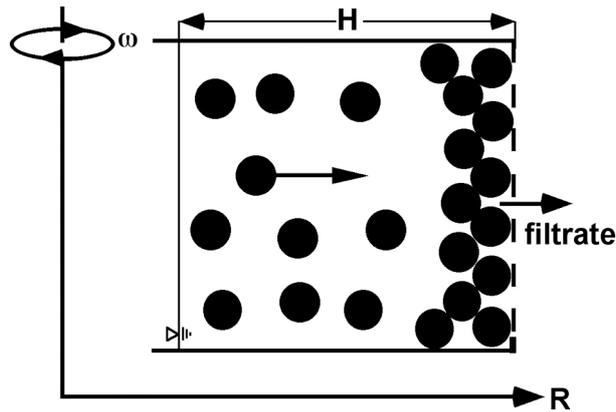
**Fig. 2.19** Stirred pressure nutsch filter

This apparatus with filter areas up to 15 m<sup>2</sup> is very flexibly adaptable to changing product properties. Thus it is used frequently in pharmacy or fine chemistry for smaller product quantities of often varying compositions. The stirred pressure nutsch filter provides relatively small filter area in relation to the process room. For relatively hard to filter suspensions several filter discs, cylindrical filter candles, flat rectangular filter leaves or filter bags are installed in the pressure vessel to increase the active filter area.

Alternatively to the gas differential pressure mass forces can be used for filtration. For coarse particles with diameters of more than ca. 1mm the force of gravity is used. Such particles are separated by bended or vibrating screens. With vibrating screens even C-values up to ca. 5 can be realized. A further dehumidifying then usually takes place via drainage in the earth field. In order to get larger accelerations, centrifuges have to be used. Figure 2.20 clarifies the principle. The centrifugal pressure is to be calculated as hydrostatic pressure difference  $\Delta P$  of a liquid column of the height H in the centrifugal field:

$$\Delta P = \rho_L \cdot g \cdot C \cdot H = \rho_L \cdot R \cdot \omega^2 \cdot H \quad (2.9)$$

The centrifugal pressure changes with the level height H of the liquid layer in the centrifuge bowl. This is a remarkable difference in comparison to the gas differential pressure, which can be held constant.

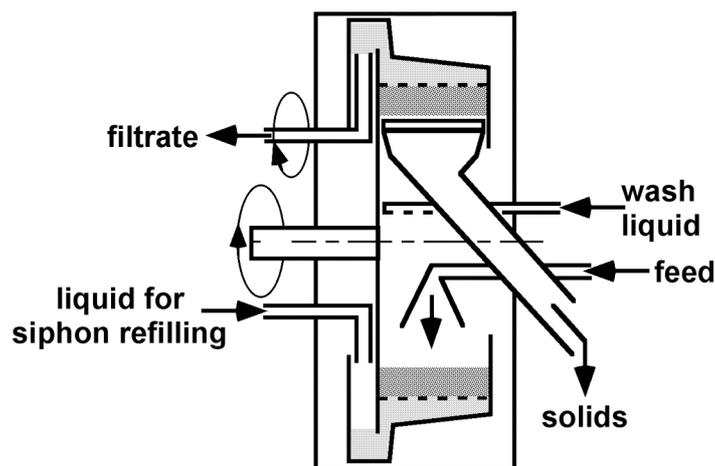


**Fig. 2.20** Principle of centrifugal cake filtration

Like in the case of gas overpressure filters centrifugal pressures up to ca. 1 MPa are realized technically. Due to the fast settling of particles in the centrifugal field filter cakes in centrifuges mostly are formed by quick sedimentation and subsequent drainage of the clear liquid layer above the sediment. During desaturation a moisture gradient is appearing along the cake height, because the centrifugal pressure becomes smaller and smaller in the emptying pore. The capillary pressure in a pore of constant diameter remains constant. If both pressures become equal in the equilibrium state, a capillary liquid height  $H_c$  remains in the cake, which cannot be further removed with constant rotational speed of the centrifuge:

$$H_c = \frac{P_c}{\rho_L \cdot g \cdot C} = \frac{2 \cdot \sigma_L \cdot \cos \delta}{r \cdot \rho_L \cdot g \cdot C} \quad (2.10)$$

For centrifugal filtration a large number of discontinuously and continuously operating machines are available. To the family of discontinuous filter centrifuges essentially belong the horizontal and vertical peeler centrifuges, the inverting filter centrifuge and centrifuge dryers (see chapter 2.5). Discontinuous filter centrifuges can realize centrifugal pressures of up to 1 MPa and are built with drum diameters of up to ca. 1500mm. Exemplary for these centrifuges a horizontal siphon peeler centrifuge is represented in Fig. 2.21.



**Fig. 2.21** Horizontal siphon peeler centrifuge

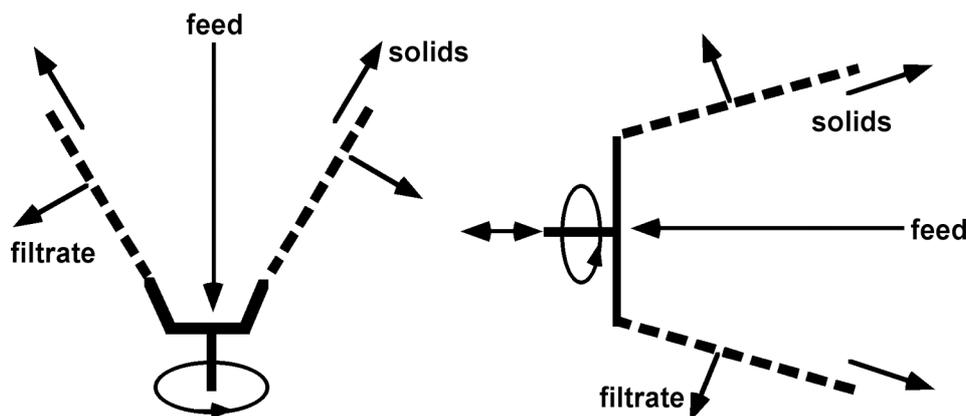
The suspension to be separated is supplied to the continuously rotating filter drum. After cake formation the filter cake can be washed and afterwards desaturated. At the end the filter cake is removed from the rotating drum by an advancing knife. Necessarily a thin product layer remains on the filter cloth. This heel must be

removed or regenerated periodically. While with conventional peeler centrifuges the filtrate is getting directly into the housing and removed from there by a fixed pipe, it is collected in the case of a siphon peeler centrifuge in a solid filtrate collecting room and then removed from a ring cup by a pivoting pipe. This leads to two advantages. If a part of the previously produced filtrate is returned again into the ring cup, the filter cloth can be cleaned by backflushing and the clogged heel becomes resuspended. If with the next load the pivoting pipe is immersed below the level of the filter medium into the ring cup, the liquid column behind the filter medium generates a vacuum, which supports the filtration process additionally.

In contrast to a peeler centrifuge a heel can be avoided totally in an inverting filter centrifuge, where the filter medium is pushed for cake discharge axially out of the centrifuge drum by a special installation. The filter cloth is turned and the solids are becoming detached completely.

In a centrifuge dryer follows a thermal drying process by hot gas after the mechanical dehumidifying and finally a dry powder is produced (see chapter 2.5).

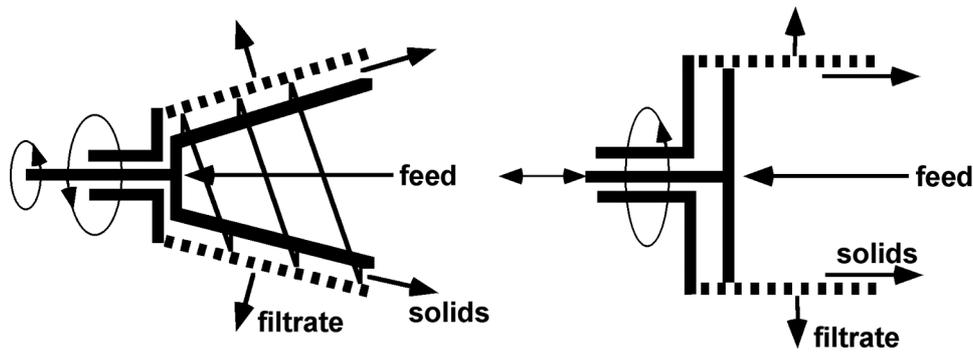
With the continuously operating filter centrifuges the separated solids are transported axially along the filter medium to the open end of the rotating perforated drum. To realize safe transport for sufficient life time wear-resistant metallic wedge wire screens are necessary. In these centrifuges the residence time of the product in the process room amounts to clearly less than 1min for filtration pressures of ca. 0.1 MPa. Therefore these centrifuges are used for large quantities of easy to filter and highly concentrated suspensions. The particle diameters in most cases are above 100  $\mu\text{m}$ . Sliding discharge centrifuges and vibrating screen centrifuges possess a conical rotor and the solids are sliding as a result of the centrifugal force towards the greater drum diameter and its open end. For the better traction control of the solids the static friction between product and drum can be overcome with vibrating screen centrifuges by an overlay of drum rotation and axial oscillation. In Fig. 2.22 the principle of these centrifuges is outlined.



**Fig. 2.22** Principle of sliding discharge centrifuge (left side) and vibrating screen centrifuge (right side)

Worm screen centrifuges possess a screw similar to the decanter centrifuge. Pusher centrifuges are equipped with an axially oscillating pusher plate for discharging the filter cake. In Fig. 2.23 the principle of these centrifuges is sketched.

Continuously operating filter centrifuges are often combined with settling basins to guarantee enough concentrated feed for a safe function. The filtrate of these machines contains in many cases too much particles and is given back into the thickening device (see chapter 2.6.2).

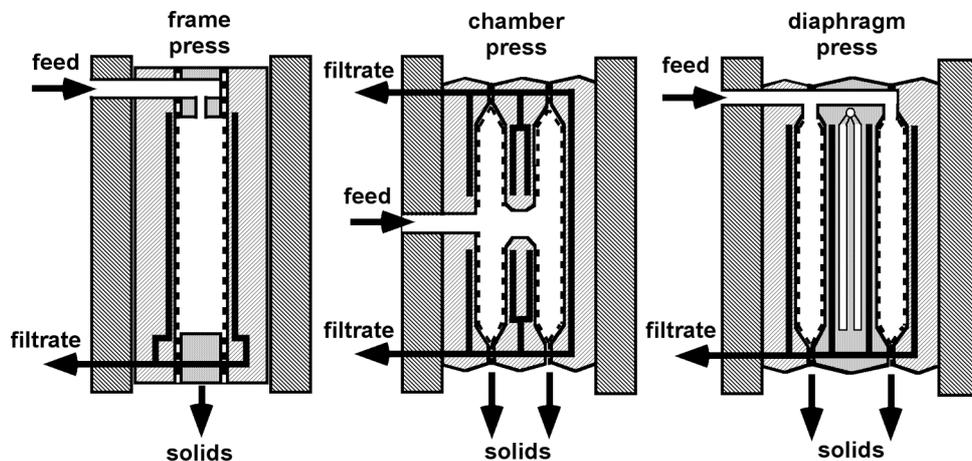


**Fig. 2.23** Principle of worm screen centrifuge (left side) and pusher centrifuge (right side)

Continuous filter centrifuges can be also combined with an internal machine part in which the slurry is thickened by pure sedimentation first. The screen bowl decanter centrifuge would be an example for these machines.

Last but not least a hydraulic or mechanical pressure can be utilized for press filtration of suspensions, which are forming a compressible filter cake. Depending on the flow resistance of the originating filter cakes maximal filtration pressures of approximately 10 MPa are used in the relative rare case of discontinuous piston and tube presses. Up to 2 MPa are used for discontinuous frame, chamber and diaphragm presses and relatively low pressure of about 0.1 MPa is applied to continuous screw and belt presses.

One of the most frequent used filter apparatus for solid-liquid separation of relatively difficult to filter suspensions is the discontinuous filter press. Figure 2.24 illustrates the three basic constructions of these apparatuses.

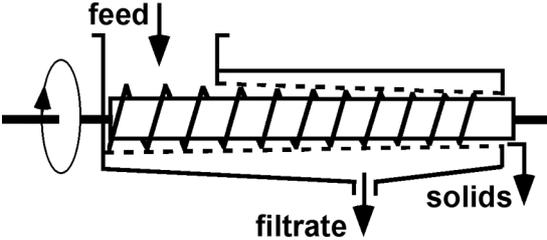


**Fig. 2.24** Frame, chamber and diaphragm filter press

In the case of the historically oldest frame presses the filter chamber is formed by a frame between two flat filter plates. The filter plates are covered on both sides with a filter cloth. This applies to all types of filter presses. The filter chambers of a chamber filter press are formed by the geometry of the filter plates themselves. While the filter cake in frame and chamber filter presses is consolidated hydraulically by additional pressing of suspension into the already cake filled chambers, the filter cake in modern diaphragm filter presses can be squeezed mechanically by means of a flat diaphragm very efficient and homogeneous. With all filter presses the filter plates must be pressed together by hydraulic or mechanical means in order to avoid leakages. For the cake discharge the plates are separated from each other and the cake is removed downwards from the chambers. The cake discharge turn out to be

most difficult in the case of frame presses, because the cake sticks in the frame. In chamber and diaphragm presses gravity helps but in many cases an additional mechanical support is necessary to detach the cake from the filter cloth. The latest development are fully automated diaphragm filter presses with horizontally arranged filter plates. The automated cake discharge is guaranteed here by an endless filter belt around all plates, which can be moved. The moving filter belt transportes the cake out of the filter chamber and detaches the solids by sharp redirection.

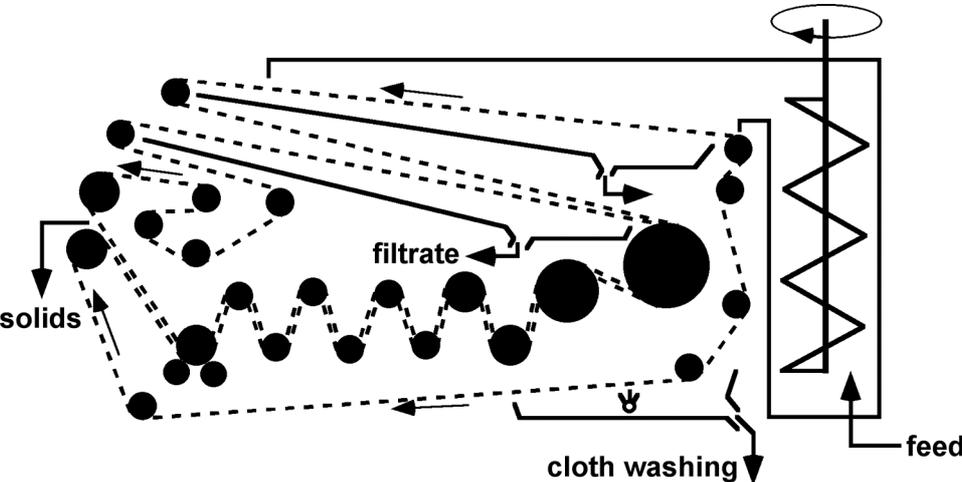
Continuously operating press filters are built mainly as screw or belt presses and are used for relatively easy filtrable fiber containing or strongly flocculated suspensions. In Fig. 2.25 the principle of a screw press is depicted.



**Fig. 2.25** Screw press

The suspension to be separated is supplied to a transport and press screw, which is slowly rotating in a sieve basket. As filter medium metallic wedge wire screens are used. The diameter of the conical sieve basket in transport direction and the distance between the screw blades becomes smaller. Thus the volume available for the solids is reduced continuously and they are squeezed. The filtrate penetrates the filter medium, gets into the housing of the screw press and is led away from this. The squeezed and dewatered solids are discharged at the end of the press channel from the sieve basket. The screen has to be cleaned periodically from outside by means of a strong water spray to avoid pore blockage.

In contrast to the screw press Fig. 2.26 shows a double belt press filter. The suspension is given continuously between two filter belts and is squeezed by press rollers. The smaller the roller diameter becomes, the greater becomes the squeezing pressure. Due to the enlacement of the rollers by the filter belts the cake is not only pressed uniaxially but sheared additionally. This leads to a further cake consolidation and therefore to lower residual cake moisture.



**Fig. 2.26** Double belt press filter

According to the described variety of filter apparatuses for cake filtration very different demands are made on the filter media. From filter fabrics with various weave construction, fleeces and felts, porous sinter materials and metallic wedge wire screens to microporous membranes most different filter media are used (Purchas, 1996). The filter medium represents the interface between filter apparatus and suspension. Only for the right adjustment between filter medium, filter apparatus, operating conditions and suspension properties a separation process can be realized successfully (Anlauf, 2004). The application of recently developed new micro-porous membrane filter media can lead in the case of vacuum filtration to perfect particle retention and in addition to an avoidance of the undesired gas flow during the desaturation phase of the filter cake. If the capillary pressure in the filter mediums pores is larger than in the pores of the filter cake, the cake can be desaturated by means of a gas differential pressure however the gas cannot penetrate into the fully saturated pores of the filter medium. A semipermeable behavior of the filter medium regarding liquid and gas is generated. This could save energy for the operation of the vacuum pump (Anlauf, 2008).

### 2.3.2 Sieving and blocking filtration

If liquids may not contain particles with diameters of more than ca. 5  $\mu\text{m}$  and these particles are present only in very low concentration, surface filters like sieve or blocking filters are applied. As „police filters“ they are protecting e.g. hydrocyclones or disc stack separators (see chapter 2.2.2) against oversized particles, which could block the solids discharge nozzles. In water or oil cycles impurities like rust particles or particles from abrasion in a motor or gearing are removed from the liquid by sieve filters. Each separated particle blocks a pore of the screen and the pressure loss of the filter rises with time. If it exceeds a critical value, the filter must be regenerated. This takes place mechanically via rotating brushes or other devices or via back flushing with own or external liquid. As filter media serve sieve fabrics or wedge wire screens. Figure 2.27 shows exemplarily for this filter family the principle of a quasicontinuously operating back flushing sieve filter in the filtration and back flushing phase.

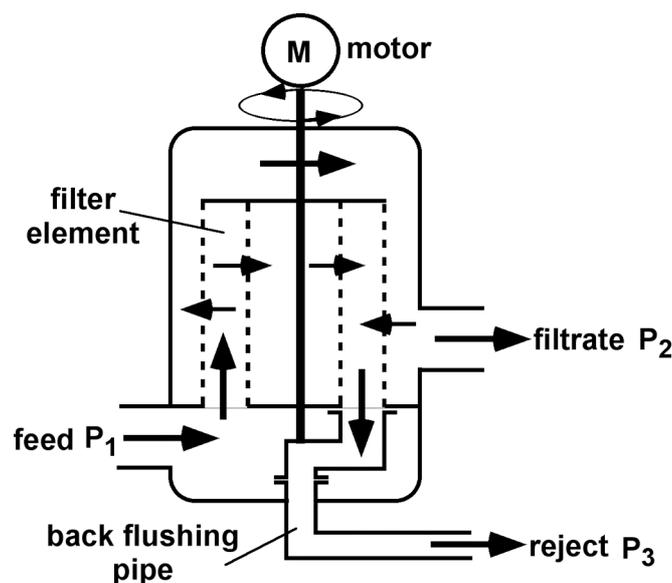
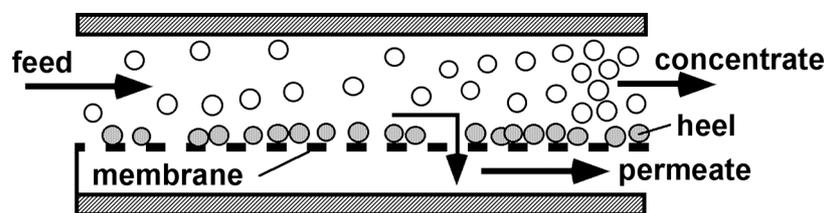


Fig. 2.27 Back flushing filter

The liquid to be purified enters the lower part of the filter housing with the pressure  $P_1$  and flows into several parallel and in a circle arranged filter candles. The filtrate passes the filter medium, flows into the upper part of the filter housing and is discharged from there with the pressure  $P_2$ . Due to the low pressure loss of the sieve filter  $P_2$  is only slightly lower than  $P_1$ . The absolute pressure inside the filter is ca. 100 ÷ 200 kPa higher than the atmospheric pressure  $P_3$  outside the filter. If a filter candle is blocked on the inner surface by particles a back flushing tube is moved under the the candle. This tube connects the filtrate space with pressure  $P_2$  and the atmospheric pressure  $p_3$ . Now the filtrate flows from outside inwards into the candle and washes away the separated particles from the filter surface. After the end of a very short cleaning phase the back flushing tube moves to the next blocked candle.

### 2.3.3 Crossflow micro and ultra filtration

The crossflow filtration is according to Fig. 2.28 one basic modification of surface filtration. As filter media normally microporous membranes are used.



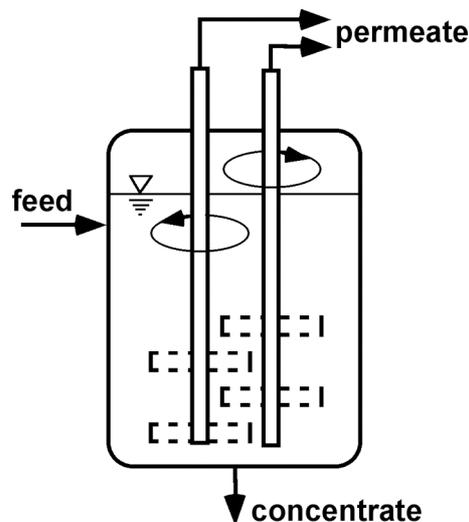
**Fig. 2.28** Principle of crossflow filtration

Crossflow filtration serves in particular the separation of very small particles in the range of less than 1  $\mu\text{m}$  diameter, colloids and dissolved macromolecules. Especially the mechanical separation of large molecules being in solution represents a direct competition for thermal evaporation. In the case of solid particles the crossflow filtration competes with high speed centrifuges like disc stack separators or tube centrifuges (see chapter 2.2.2) and depth or precoat filtration processes (see chapter 2.3.4). Compared with depth or precoat filtration and not regenerable filter layers the crossflow filtration is characterized by the separation of the particles in pure form. The crossflow filtration is called also shear stress, dynamic or delayed cake filtration. The solids (concentrate) as well as the liquid (permeate) can represent the value product. The suspension to be separated is pumped under pressure through the filter module. The liquid permeates the filter medium and is discharged as permeate. Particles try to follow the liquid and to build up a cake on the filter medium. However during the flow of the suspension tangentially to the filter medium particles are removed from the membranes surface mainly by shear forces except a very small layer and are consecutively concentrated. In this manner the transmembrane pressure loss can be kept low and the filter can be operated for longer time stationarily. With declining filtrate flow by successive blockage or fouling of the membranes pores and/or the heel the filter system must be periodically regenerated by back flushing and/or various washing procedures. There are also attempts known to prevent a heel by creation of an electrical field across the feed/concentrate channel to generate electrical repulsion of the charged particles from the filter medium (see chapter 2.6.3). Along the direction of flow a concentration of the suspension or solution takes place at most near to the yield point of the concentrate. Thus a crossflow filter is preferably suitable for the concentration of suspensions or solutions in a broad spectrum of possible degrees of enrichment. With shear thinning or thixotrope flow behaviour the concentrate can be kept still flowable by the creation

of high shear rates despite high thickening at the discharge valve of the filter apparatus. In such cases the sludge solidifies after the discharge under circumstances to a semisolid mass. The principle of crossflow filtration is limited on one hand by an often insufficient high degree of moisture content of the concentrate or under circumstances in spite of regeneration measures by an irreversible blockage of the filter medium after some time.

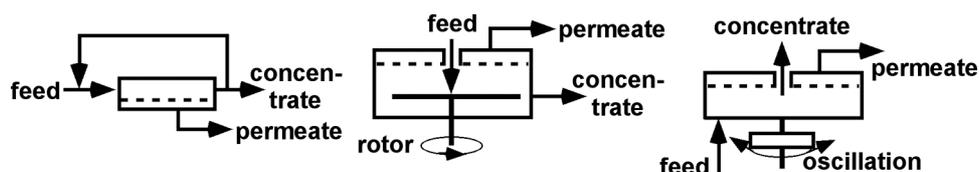
With discontinuous operating method liquid is withdrawn from the suspension to be separated by permanent filtration and concentrate recycling until the target concentration is reached. With the fed-batch procedure concentrate is fed back likewise, but the distant filtrate quantity is filled up with fresh suspension until the total volume of the feed vessel has reached the final concentration. With the full-continuous procedure no concentrate recycling takes place and the target concentration can be achieved usually only via a serial connection of several filter modules. Dia-filtration is a procedure of dilution washing with which in the suspension dissolved substances are rinsed through the filter medium. Similar to the fed-batch procedure the concentrate is recycled here but filled up again not with suspension but with fresh wash liquid. With continuous mode of operation and serial arrangement of modules (see chapter 2.6.2) the dia-filtration can be operated particularly effective by counter-current flow of the wash liquid.

The shear flow to prevent the particles from becoming deposited on the filter medium can be increased in dynamic filters by rotor/stator or rotor/rotor systems. Figure 2.29 depicts this for the example of overlapping counterrotating filter discs.



**Fig. 2.29** Dynamic crossflow filter

Furthermore the filter module can be mounted on a torsion rod and be oscillated with up to 50 Hz. Figure 2.30 concludes all three possibilities of creating relative motion between suspension and filter medium.



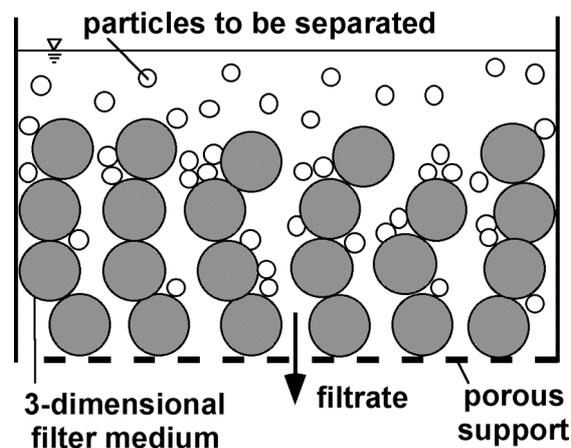
**Fig. 2.30** Modifications for crossflow generation

Because of the extremely small particle diameters microporous membranes are used as filter media for crossflow filtration processes. These can be manufactured tube-shaped as pipe or capillary modules or in flat form as cushion, spiral or sheet

modules. For the membrane production can be employed most different polymers, ceramics, glass and other materials. With the crossflow processes one distinguishes according to the particle sizes, which are to be separated, into the microfiltration with feed pressures of ca. 0.1 MPa and ultrafiltration with feed pressures of ca. 1.0 MPa. The pore dimensions of membranes for microfiltration lie in the range between ca. 1.0 and 0.05  $\mu\text{m}$ . Membranes for ultrafiltration are still pore membranes with convective mass flow and can be labeled as molecular sieves with pore sizes in the range between ca. 0.1 and 0.005  $\mu\text{m}$ . Pure diffusive mass transfer takes place by using poreless membrane structures for processes like reverse osmosis, which are applied e.g. to desalinate seawater.

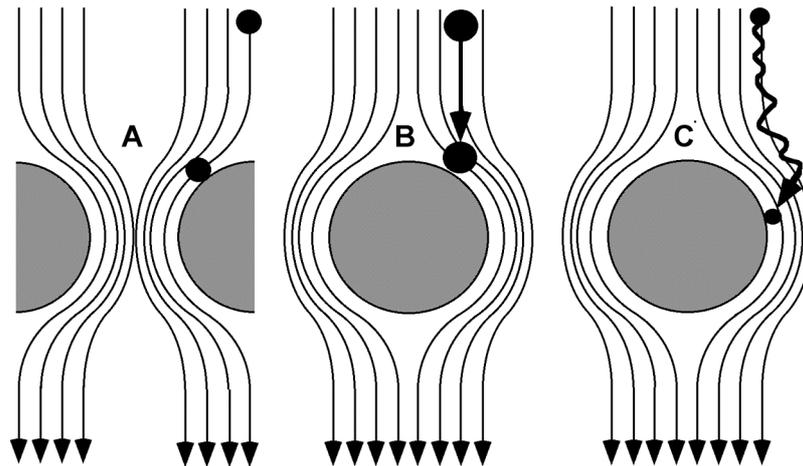
### 2.3.4 Depth and precoat filtration

The depth filtration is used preferably, when slightly turbid liquids have to be clarified. The liquid usually represents the value product. The solids concentration adds up here to some  $\text{g m}^{-3}$  or less and the particle diameters are below ca. 1  $\mu\text{m}$ . According to Fig. 2.31 the particles present in the suspension are separated inside a three-dimensional porous and highly permeable filter layer.



**Fig. 2.31** Principle of depth filtration

Figure 2.31 shows the standard case of a depth filtration, with which a porous filter layer is permeated by the liquid to be purified. The filter cycle has to be shut down, if the capacity limit of the filter layer is reached. This capacity limit is reached if either the pressure loss of the flow exceeds a critical value or if the turbidity of the filtrate reaches an unacceptable level. The solids concentration in the filter feed may not exceed a critical maximum value in order to avoid blockage of the filter surface. Such blockings originate, if in the case of higher concentrations several particles try to enter a filter pore at the same time and are hindering each other or if pores are gradually blocked bit by bit by separated particles. In the case of cake filtration (see chapter 2.3.1) this bridge formation by particles is necessary to initiate the cake formation. With the depth filtration such a particle bridging would prevent following particles from penetrating into the inside of the filter layer and lead to the failure of the procedure due to the drastic rise of the flow resistance. The particle separation in a depth filter requires a transport mechanism, that brings the particles to the surface of the filter medium (collector) and an adhesion mechanism, which holds the particles to the collector. The mechanisms of particle separation inside the filter layer are very complex like to be seen in Fig. 2.32 on three essential examples of the particle transport.



**Fig. 2.32** Mechanisms of particle deposition in depth filters

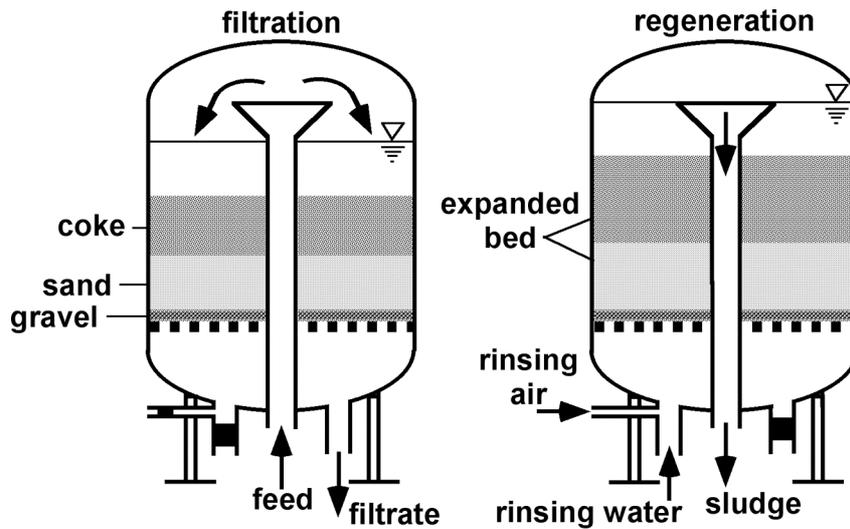
Case A is called „interception“. The particle follows a streamline of the liquid and touches the collector compulsorily due to its physical dimension. B marks the „inertia effect“, if a particle due to its mass cannot follow the streamline around the collector and settles to its surface. C represents the „diffusion effect“ and is relevant especially for very small particles, which are subject to diffusion and are coming into contact with the collector by stochastic movement. A further mechanical dehumidifying of the separated solids is not possible with depth filtration processes. The separated solids are removed either more or less efficient by back flushing of the filter layer with a small part of the previously clarified liquid or is discharged completely together with the filter layer from the filter apparatus. One distinguishes between different depth filtration processes like

- packed bed filters,
- cartridge and sheet filters and
- discontinuous and quasi-continuous precoat filters.

The packed bed filters consist of discrete disperse particle layers, which are regenerated after the filtration by back flushing or must be exchanged. This type of filter is used very frequently in the water purification (Gimbel 2006). As filter material gravel, sand, diatomaceous earth, perlite, filter coke, activated charcoal or others are used. Materials like activated charcoal or ion-exchange resins are able beyond the pure mechanical separation of particles to bind in addition dissolved substances by adsorption. Figure 2.33 shows exemplary an encapsulated packed bed filter during filtration and back flushing. Here an optimized two-layer filter is depicted. A coarse-grained coke layer is located on top of a fine-grained sand layer. Thus the pores of the filter top side downward become smaller and the total capacity of the filter is increased in comparison to a mono-layer filter. To avoid a mixture of coarse and fine particles during back flushing, the upper coarse particle fraction (coke) must have lower spec. weight in relation to the underneath located fine particle fraction (sand).

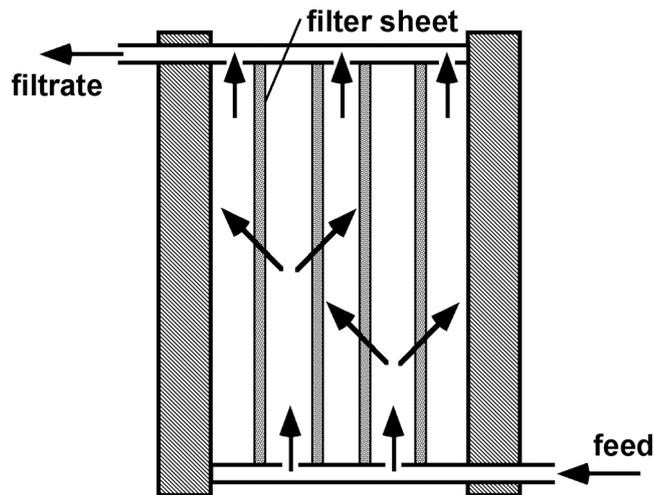
Cylindrical non-pleated or pleated cartridge filters or flat-shaped sheet filters use prefabricated filter media, which are not regenerable as a rule and must be exchanged after reaching their limit of capacity. In case of cartridge filters yarn wound candles, sintered or resin bound porous layers are normally used. Flat filter sheets mostly are composed of fibers (cellulose) to form a structure and particles (diatomaceous earth) for separation, which are mixed and bound together by resins. Recently also filter layers from pure cellulose are produced. The spec. surface of the cellulose fibers and thus the separation capability is increased by a special

defibrination process, which is called „fibrillation“. Cartridge and sheet filters are applied for very different settings of tasks and a very broad product spectrum.



**Fig. 2.33** Packed bed filter in filtration mode (left side) and back flushing mode (right side)

Exemplarily a sheet filter with rectangular sheets is represented in Fig. 2.34.

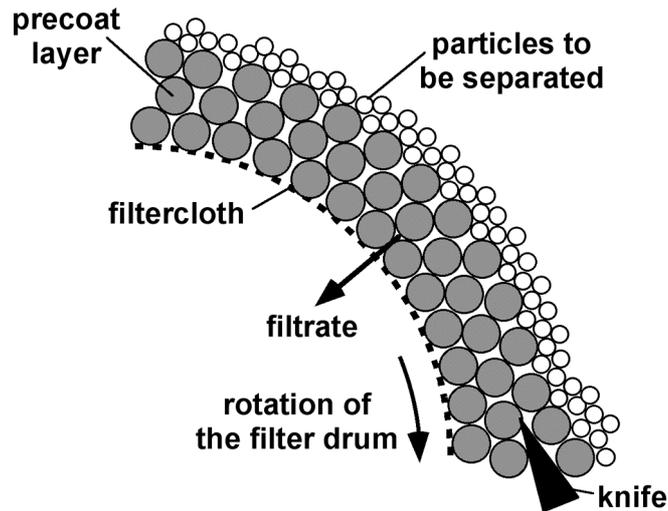


**Fig. 2.34** Sheet filter

The apparatus is designed similar to a frame filter press and consists of a package of filter plates and frames. The suspension is pumped into several in parallel arranged filter chambers. The liquid penetrates the filter media and is discharged as clarified filtrate through the filtrate channel. The particles are separated inside of the porous filter sheet structure. If their particle storage capacity is run out, they must be replaced by new filter sheets.

A further variant of the depth filtration can be realized on many different apparatuses for cake filtration like discontinuous candle or leaf filters (see chapter 2.3.1). Here a porous auxiliary particle layer, which serves as depth filter, is formed on a filter medium as a cake before the actual product filtration can take place. This kind of filtration is called precoat filtration. After reaching the limit of dirt holding capacity the entire precoat layer is discharged as filter cake and the cycle starts again with forming a new precoat layer.

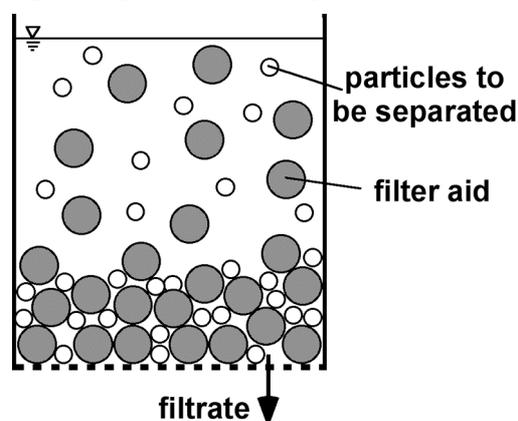
The precoat filtration on continuous vacuum drum filters offers the possibility of peeling off a very thin layer from the surface of the precoat, which is clogged by separated particles after one drum revolution. Thus a fresh and clean precoat surface immerses again into the suspension in the filter trough. This allows the filtration of very small particles of higher concentration, which leads to a transition from depth to cake filtration. Many applications for this technique are to be found in the purification of beverages or the production of bakers yeast. Both the liquid or the solids can represent the value product. Figure 2.35 demonstrates the principle of particle discharge from a continuous precoat drum filter.



**Fig. 2.35** Principle of particle discharge from precoat drum filters

In a first working step a filter aid layer of several centimeters thickness ( $H_c \approx 10 \text{ cm}$ ) is built up on the drum covered with a well suited filter fabric. In the second working step the suspension to be clarified is filtered. The particles are separated in the uppermost layers of the precoat material and a very thin filter cake ( $H_c < 1 \text{ mm}$ ) is formed on top of the clogged precoat surface. Then the layer containing the separated particles is scraped off continuously by a sharp knife and fresh precoat surface immerses again into the suspension. Most modern precoat drum filters realize today a knife feed motion of 50 to 100  $\mu\text{m}$  per drum revolution. Thus it is possible to filter several hours continuously without interruption to build up a new precoat layer.

With a special variant of precoat filtration, which is called „body feed filtration“ the filter aid is mixed according to Fig. 2.36 directly with the suspension to be clarified.



**Fig. 2.36** Principle of body feed filtration

The particles of the filter aid are forming a framework into which the target particles are incorporated. Precoatfiltration is often combined with body feed filtration in order to improve the filtration characteristics of the suspension and thus to increase the service life of the precoat layer. Filter aids for precoat filtration are either of mineral or organic origin. As particulate materials diatomaceous earth, perlite, coke, starch among others are used. As fibrous materials depending upon requirements to the process wood flour, extract-free cellulose or ultrapure cellulose among others are applied. With the selection of a suitable filter aid acquisition and disposal costs play an important role beside separation characteristics and purity requirements. In particular with regard to the waste disposal organic filter aids possess advantages in comparison to mineral materials like low bulk density, biological degradability and nearly ash-free combustibility. A general trend towards increased use of organic filter aids on the basis of cellulose in comparison to mineral filter aids can be observed.

## **2.4 Enhancement of separation processes by additional electric or magnetic forces**

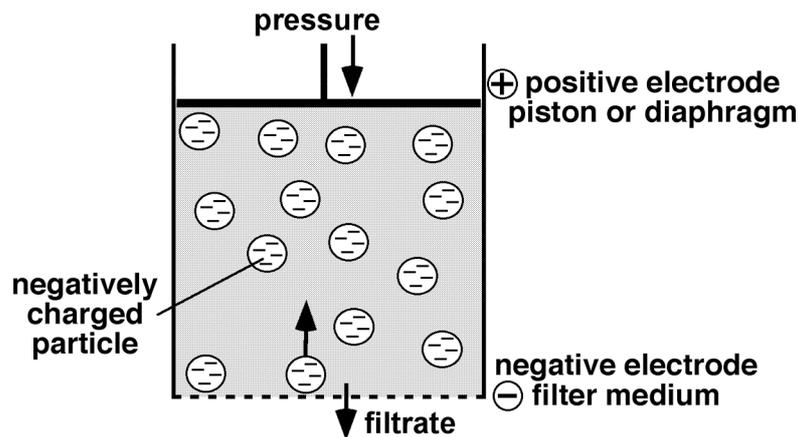
If particles dispersed in liquid carry an electrical charge or if they can be magnetized in a magnetic field, then two additional field forces are available to move particles and liquid relatively to each other. The use of electric and magnetic fields for technical solid-liquid separation is normally no standard procedure and thus limited to special applications or still object of basic research.

If one exposes a suspension to an electrical field, then charged particles are moving towards the electrode of opposite charge. This process is called electrophoresis. If hydrated ions are moving in an aqueous environment to the electrodes of opposite charge, they are transporting water molecules and one refers to electroosmosis. These effects can be used in technical solid-liquid separation processes. However it is to be noted that apart from the transport processes also electrolysis at the electrodes occurs and the temperature of the liquid rises up. Due to the electrolytic effects foreign ions from the electrodes get into the suspension or a decomposition of water into gaseous oxygen and hydrogen occurs (Delgado, et.al., 2007).

A rather special application in the area of water purification is represented by electrofloatation (Chen, et al., 2002), with which the gas bubble formation takes place via electrolytic decomposition of the water. The oxygen bubbles used for flotation (see chapter 2.2.1) are reducing simultaneously organic impurities in the water by oxidation. Cationic materials are floated very well by the hydrogen bubbles. With the choice of a suitable electrode material ions can become free, which are supporting the flocculation of the particles to be separated (see chapter 2.6.3).

Electrochromatography is used only for analytic purposes and has yet no technical relevance.

In the area of crossflow filtration current research work is known to position an electrode behind the microporous membrane, which possesses the same charge as the particles to be separated. Thus particles are prevented from adhering at the membrane surface by electrostatic repulsion (Huotari et. al., 1999). The application of electrical fields to support cake filtration of very hard to filter suspensions has been realized already occasionally in filter presses (see chapter 2.3.1). The principle of this process is explained in Fig. 2.37. Due to the arrangement and the polarity of the electrodes the particles are moved against the filtration direction. Thus a delayed formation of a filter cake with very high flow resistance on the filter medium results and the liquid can be removed much easier.



**Fig. 2.37** Principle of electro/press-filtration

In this way the necessary filtration time in many cases can be shortened drastically. As disturbing for the process accompanying effects like electrolysis have to be taken into account (Weber, Stahl, 2002).

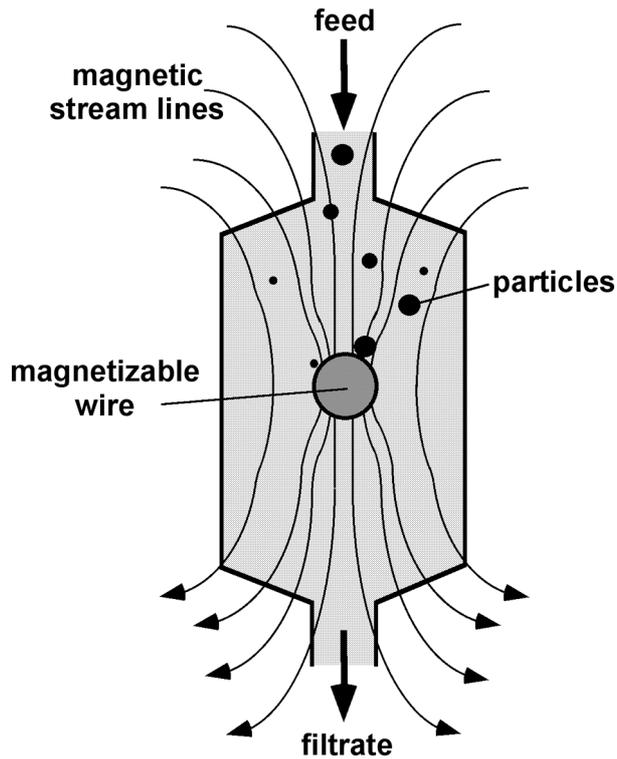
If particles are magnetizable they can be separated with weak and strong magnetic field separators from liquids (Svoboda, 1987). Such procedures find application in the classical mineral processing, where drum separators are preferred. A slowly rotating drum, which contains inside a magnet immerses into a trough filled with suspension and the magnetizable particles are adhering at the exterior surface of the drum. After emerging of the drum from the suspension and switching the magnetic field off the separated particles are removed from the drum.

Latest developments for mechanical separation in biotechnological production, which are still under academic research and technical development are summarized under the name „Magnetic Fishing“ (Eichholz, et. al., 2008). Ferromagnetic micro-particles are integrated into a polymeric matrix and then coated with specifically active ligands, which are able to bind selectively a target substance from a fermentation broth by the principle of key and lock. During the following separation process a magnetic field is overlaid to a conventional filter or sedimentation apparatus. The target substance bound to the magnetic beads will become held back and the not desired materials are lead away. Subsequently the isolated and concentrated target substance is purified and removed from the magnetic beads, which are recycled again to the process.

Magnetic separation can be realized in form of the High Gradient Magnetic Separation (HGMS) or the Open Gradient Magnetic Separation (OGMS). With the HGMS the slurry to be separated flows like in the case of a depth filtration (see chapter 2.3.4) through a comparatively very open wire mesh, which is located in a magnetic field. The streamlines of the magnetic field are distorted remarkably by the wires and strong field gradients  $\nabla H_{\text{mag}}$  originate, which directly influence the magnetic force  $F_{\text{mag}}$ . The magnetic force depends apart from the the permeability number  $\mu_0$  on the particle Volume  $V_p$ , the susceptibility  $\chi$ , the magnetic field strength  $H_{\text{mag}}$  and the field gradient  $\nabla H_{\text{mag}}$ .

$$F_{\text{mag}} = \mu_0 \cdot V_p \cdot \chi \cdot H_{\text{mag}} \cdot \nabla H_{\text{mag}} \quad (2.11)$$

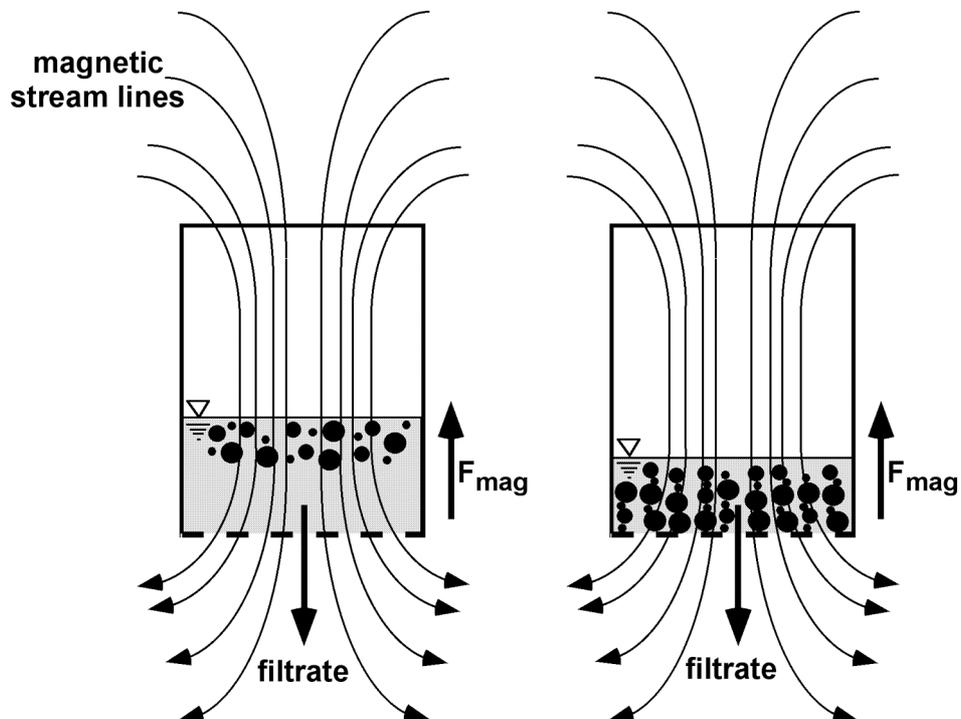
Figure 2.38 shows the distortion of the magnetic streamlines and the principle of separation in a HGMS filter for the example of one single wire.



**Fig. 2.38** Principle of HGMS

Using electro-magnets the filter can be released after switching off the magnetic field from the separated particles by rinsing out the wire mesh.

The OGMS procedures are working in accordance with Fig. 2.39 without a field-strengthening matrix and are using the gradient of an external magnetic field to influence the magnetizable particles.



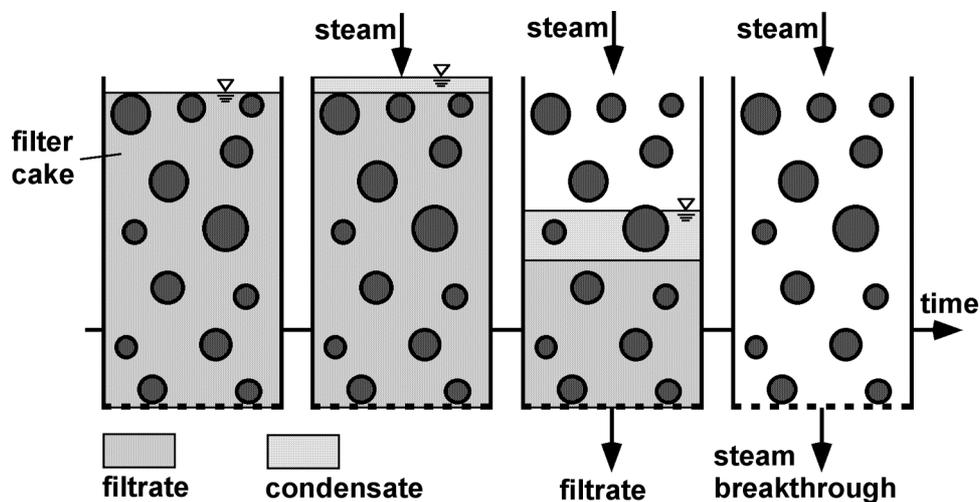
**Fig. 2.39** Principle of OGMS

By the positioning of the magnet can be determined, in which direction the particles should move. In a Nutsch-filter the particles can be kept away from the filter medium

analogously to the filtration in an electrical field (see Fig. 2.39 left side). The filtration time can be reduced strongly in that way. Favouring it is added that the particles in the cake are deposited chain-like along the stream lines of the magnetic field and thus forming a very open and permeable cake structure (see Fig. 2.39 right side and Fig. 2.37). If particles already are treated in the feed pipe of the separation apparatus they can agglomerate, what improves the following separation. In these agglomerates even non-magnetizable particles can be included. This phenomenon is called heterocoagulation.

## 2.5 Mechanical/thermal hybrid processes

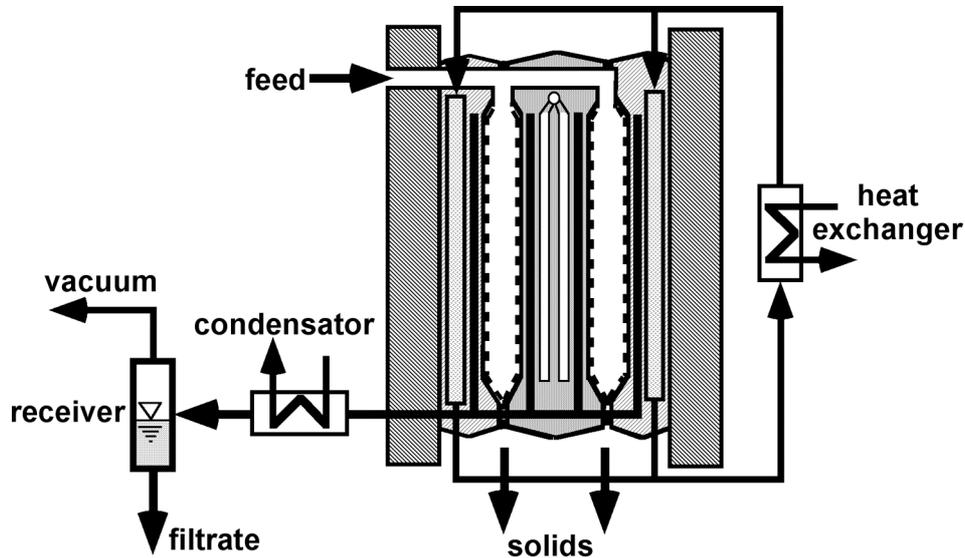
Apart from the pure mechanical liquid separation in various filter apparatuses the solids directly can be thermally dried. This is realized in discontinuous stirred Nutsch filters or filter centrifuge dryers by means of hot gas, which flows through the previously desaturated filter cake. In this way a separate thermal dryer can be saved. Real synergetic effects can be created with in the meantime industrially established steam pressure filtration processes (Peucker, Stahl, 2001). With this process pressurized steam serves as gaseous displacement medium for the pore liquid contained in the filter cake. During the conventional desaturation of a filter cake with air or nitrogen the larger pores are emptying faster than the smaller ones and it comes inevitably to an early and unwanted gas breakthrough at the filter medium. The gas flowing through these pores must be supplied later by the compressor in order to maintain the filtration pressure. This „fingering“ effect can be avoided completely with the steam pressure filtration and according to Fig. 2.40 the liquid is displaced almost ideally piston-like from the entire filter cake.



**Fig. 2.40** Course of cake dehumidification with steam pressure filtration

Pressurized steam possesses a high temperature according to its vapour pressure curve and condenses first at the cold filter cake surface. This is heated up and the steam standing under pressure penetrates into the cakes pores. If a coarse pore is emptying faster, the steam gets into the cold region of the cake. Thus the steam condenses, the pore is filled up with liquid again and an evenly advancing desaturation front develops. The produced condensate is an excellent washing medium, so that one can purify highly efficiently the particles directly combined with the dehumidification of the cake. After the steam breakthrough at the filter medium the hot filter cake is dehumidified to a great extent and an additive conventional contact or perfusion drying can be carried out.

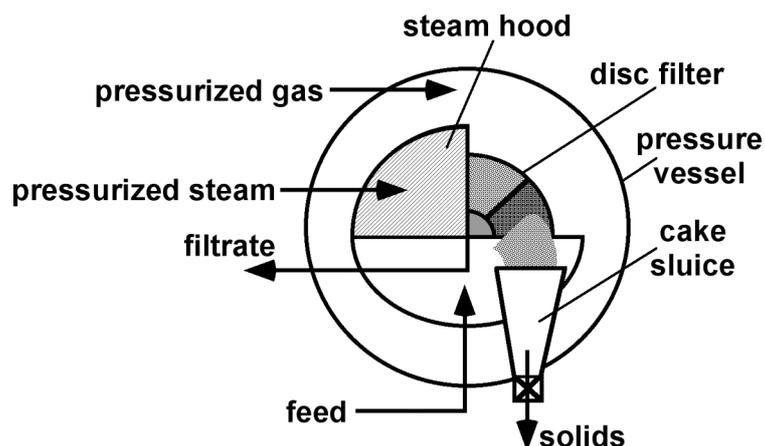
Industrially the steam pressure filtration is realized by means of discontinuous diaphragm filter presses and continuous hyperbaric filtration on drum or disc filter basis. Figure 2.41 shows the principle of a diaphragm filter press with heatable filter plates.



**Fig. 2.41** Steam pressure filtration with diaphragm filter presses

The filtrate outlet pipe is connected with a vacuum pump, in order to lower the boiling temperature of the liquid. After the steam breakthrough the filter cake can be further dehumidified by means of thermal contact drying. A shrinkage of the filter cake during drying is compensated by the diaphragm.

In the case of steam pressure filtration using continuously operating hyperbar filters the pressurized steam is according to Fig. 2.42 fed into a special steam hood (Bott, et. al., 2003).



**Fig. 2.42** Steam pressure filtration with hyperbar filters

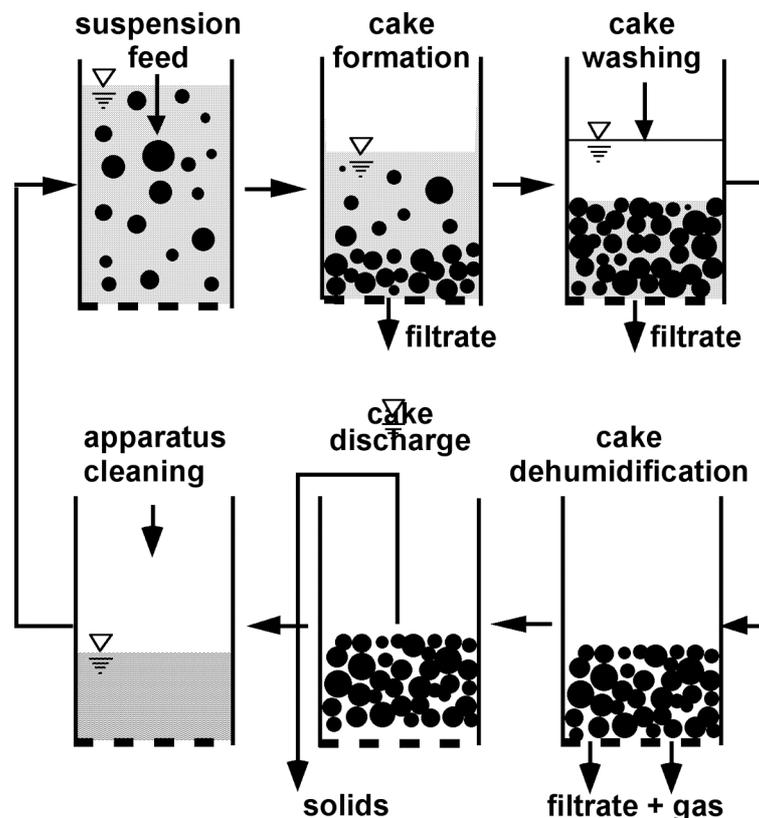
The filter cake emerges from the cold suspension and enters the hot steam hood. The pressure in the steam hood is equal to the gas pressure in the filter vessel. The process is dimensioned in the way, that the cake leaves the steam hood in the moment, during which in the filter medium the steam breakthrough occurs. After leaving the steam hood pressurized air flows through the hot and still moist cake, so that a thermal postdrying by perfusion takes place.

## 2.6 Superordinate aspects of efficient solid-liquid separation processes

### 2.6.1 Mode of apparatus operation

For almost any solid-liquid separation process discontinuously and continuously operating apparatus alternatives are existing. Due to the mode of operation they exhibit specific advantages and disadvantages.

The principle of the discontinuous mode of operation can be explained at the example of a simple vacuum nutsch filter for cake filtration depicted in Fig. 2.43. Each single process step like feeding, filter cake formation, cake washing, cake dehumidification, solids discharge and apparatus cleaning can be adjusted independently from each other in its temporal duration.

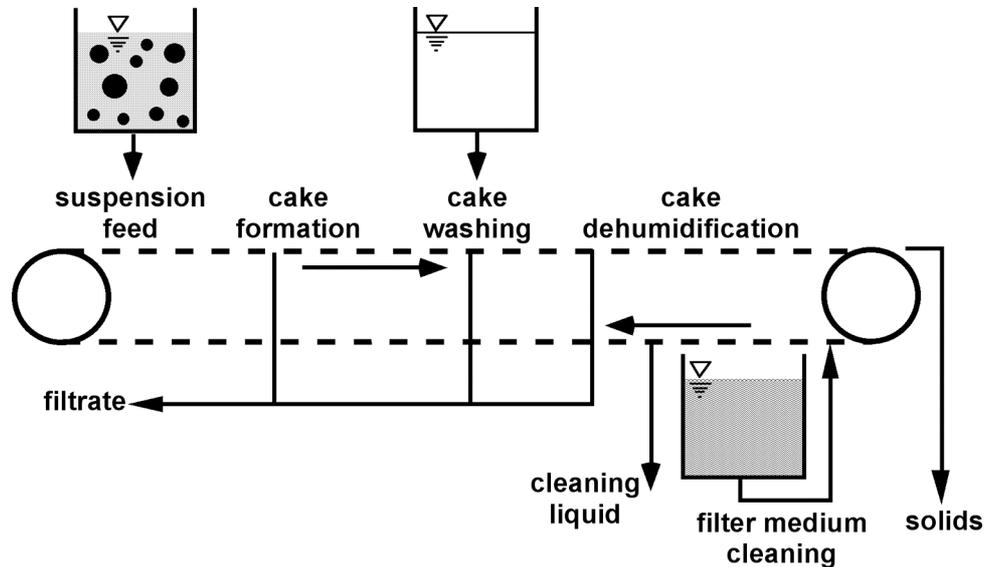


**Fig. 2.43** Discontinuous solid-liquid separation process

Thus the separation apparatus can be adapted with maximum flexibility to the requirements of the respective product. On the other hand, cake is produced only during the filtration time. However the solids throughput of the apparatus is specified by the cake mass formed during the entire time of the batch. The total batch time is subdivided into the active cake formation time and the so-called „dead time“. The dead time encloses all process steps, while no filter cake is formed. One can show for constant pressure filtration that the maximum throughput of a discontinuously operating apparatus is exactly reached in the case, that cake formation time and dead time are commensurate. If the flow rate is kept constant, the maximum throughput is reached if the maximum pressure given by the pump is reached. A quasi-continuous mode of operation of batchwise operating apparatuses can be realized by parallel connection and time shifted operation of several units. Another possibility for this exists in the installation of a suspension buffer tank. The continuous

feed, which flows into the buffer tank must correspond to the capacity of the discontinuous separation apparatus which is fed from that tank.

The full-continuous mode of operation of a solid-liquid separation apparatus can be described in contrast to the discontinuous principle like shown in Fig. 2.44 by the example of a vacuum belt filter.



**Fig. 2.44** Continuous solid-liquid separation process

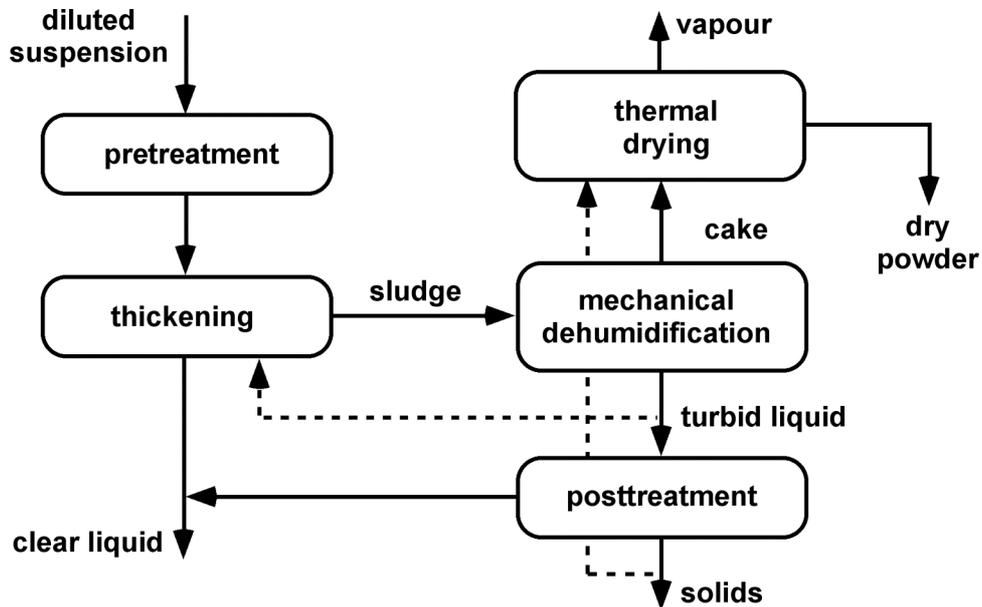
With continuous mode of operation the residence time of the product in each process step is fixed by the common transport velocity and the geometrical length of the process zone. All process steps are coupled with each other by the common transport velocity. A change of the time relationship for the individual process steps is only possible by adjusting the geometrical length of the zone. This can be realized constructionally only in relatively narrow limits. On the other hand the throughput of such a continuously operating apparatus is affected positively due to the omission of dead times. In a batchwise production process a continuously operating separation apparatus can be applied either by connection with a suspension storage tank or by periodical shut down of the separation apparatus.

### 2.6.2 Combination of separation apparatuses

A solid-liquid separation task is solved in most cases not via one single apparatus, but by means of a combination of several separation devices. During the separation process the mechanical properties of the solid-liquid mixture to be separated can change so strongly that the limits of application of one single apparatus are exceeded or its operation is no longer economical. Under circumstances it is also advantageous to split a suspension already at the beginning of the separation process and to supply each subset afterwards to an ideally suited separation apparatus. Depending upon setting of tasks different principles of apparatus combinations can be differentiated from each other into

- function separation,
- function integration,
- serial arrangement,
- parallel arrangement and
- cross arrangement.

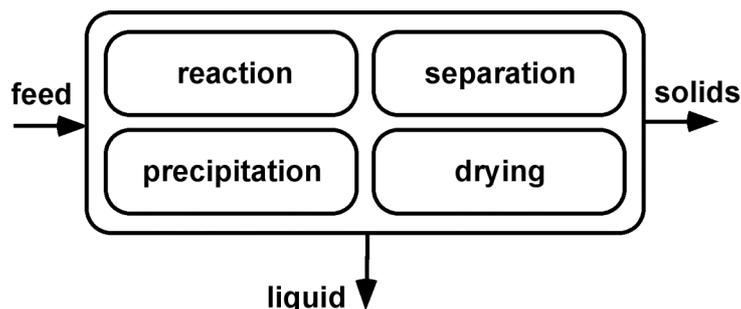
If for example a solid dispersed first in low concentration should be isolated at the end completely dry, then this task cannot be solved in general meaningfully in only one single separation apparatus, but a combination of several coordinated apparatuses must be chosen, like represented schematically in Fig. 2.45.



**Fig. 2.45** Function separation

The whole task of separation is divided into single steps and assigned to each step an optimally suited apparatus. At the beginning a low concentrated suspension of small particles is submitted in many cases at first to a pretreatment by agglomeration to facilitate the following separation (see chapter 2.2.2). In a concentration step a remarkable part of clear liquid is then withdrawn from the suspension in order to relieve the following dehumidification of the solids (see chapter 2.2.3). Now the concentrate taken off from thickening is supplied to a separation apparatus, which is specialized in the extensive mechanical separation of the liquid. Since the separated liquid is often not yet perfectly particle-free, it can be led back if necessary into the thickening stage or can be processed in a specialized post-clarification device. The mechanically dehumidified sediment or filter cake is given finally into a thermal dryer to evaporate the last adhering liquid and appears at the end of the process as a completely dry powder.

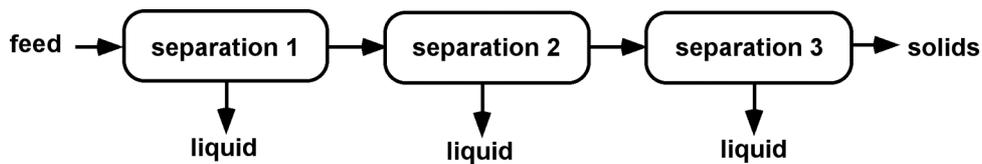
Contrary to the principle of function separation also a combination of several process steps in only one apparatus can represent the optimal solution of a solid-liquid separation problem. As follows from Fig. 2.46 steps for the generation of the particle system as well as the mechanical dehumidification and the final thermal drying can be integrated.



**Fig. 2.46** Function integration

Such configurations make sense if product losses as far as possible have to be avoided and if the operation must be realized under extremely sanitary conditions. This is for example the case for very expensive pharmaceutical active substances. If such a process would be realized in several individual apparatuses, then these apparatuses must be interconnected by pipings, flanges and armatures. This entails a large expenditure for cleaning. In addition product is lost in every process step. The greater the number of single steps become, the smaller becomes the product yield. Examples of such integrated apparatuses are modern filter reactors, filter dryers und centrifuge dryers.

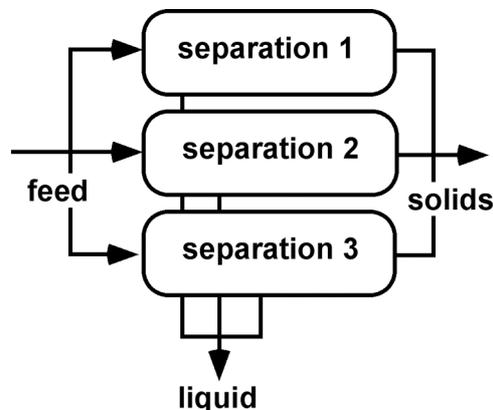
The serial arrangement of identical apparatuses is used for the intensification and thus improvement of the process result. Figure 2.47 represents this in a schematic manner.



**Fig. 2.47** Serial arrangement

An example for this would be the continuous cross-flow filtration. If after one passage of the suspension through a membrane module the required concentration is not yet reached, then one can further increase the concentration by additional modules.

A parallel arrangement of identical separation devices serves the capacity increase of a separation step by modular extension. Figure 2.48 shows the principle.

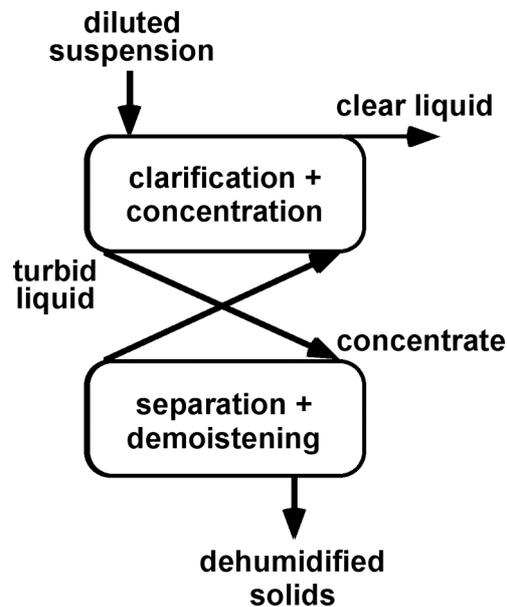


**Fig. 2.48** Parallel arrangement

An alternative for this would be the installation of one larger and more efficient apparatus. However this is not always possible or meaningful. Thus in the case of hydrocyclones the throughput rises with the cyclone diameter, but the cut-size rises likewise. If a small cut-size is required and a big amount of liquid has to be handled, then several hydrocyclones of small diameter must be arranged in parallel. As already mentioned in chapter 2.6.1, a quasi-continuously operating process can be made possible by parallel arrangement of discontinuously operating separation apparatuses. A further aspect of the parallel arrangement of several apparatuses is the larger operational reliability, if one apparatus would fail. This is similar to the installation of a parallel arranged and normally not used spare apparatus under the criterion of the redundancy.

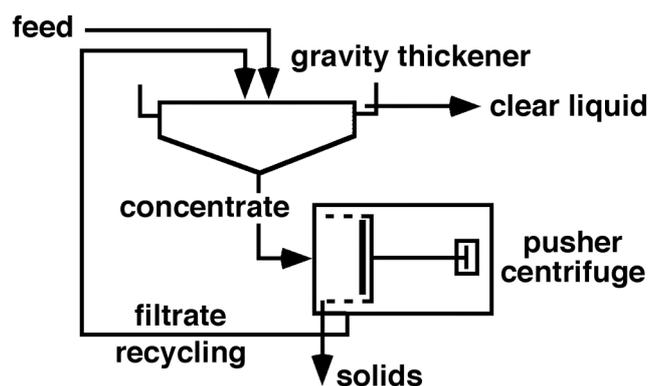
No separation apparatus works perfectly. In a cross arrangement two apparatuses are combined in the way that their respective strengths are of benefit for the solid and

liquid product whereby their weak points are compensating each other mutually. In Fig. 2.49 this is shown by the example of the combination of an apparatus for thickening and an apparatus for maximal dehumidification of the solids.



**Fig. 2.49** Cross arrangement

The apparatus for thickening of a low concentrated suspension can be optimized to produce a particularly particle-free liquid, which means it works as clarifier. The concentrate, which then still contains much liquid is supplied to an apparatus, which is suitable for the maximum solids dehumidification. As a consequence of these conditions the separated liquid contains often an unacceptable amount of particles. This turbid liquid is given back to the thickener to be clarified again. A practical example for this is given in Fig. 2.50 for the combination of a circular static sedimentation basin and a continuously operating filter centrifuge.



**Fig. 2.50** Cross arrangement by sedimentation tank and filter centrifuge

The here shown pusher centrifuge transports the separated solids with the help of an axially oscillating pusher plate continuously to the discharge at the open end of the rotating filter basket. The residence time of the product in the filter basket amounts approximately 10-20 sec. To guarantee filter cake formation, eventually washing and dehumidification within such a short time, the suspension must be highly concentrated and the filter medium must be as permeable as possible. The relatively open wedge wire screen produces a cloudy filtrate which is led back for post-clarification to the thickener/clarifier. This takes over the clarification and guarantees

the physical function of the centrifuge by delivering an enough concentrated suspension.

### **2.6.3 Suspension pre-treatment methods to improve separation conditions**

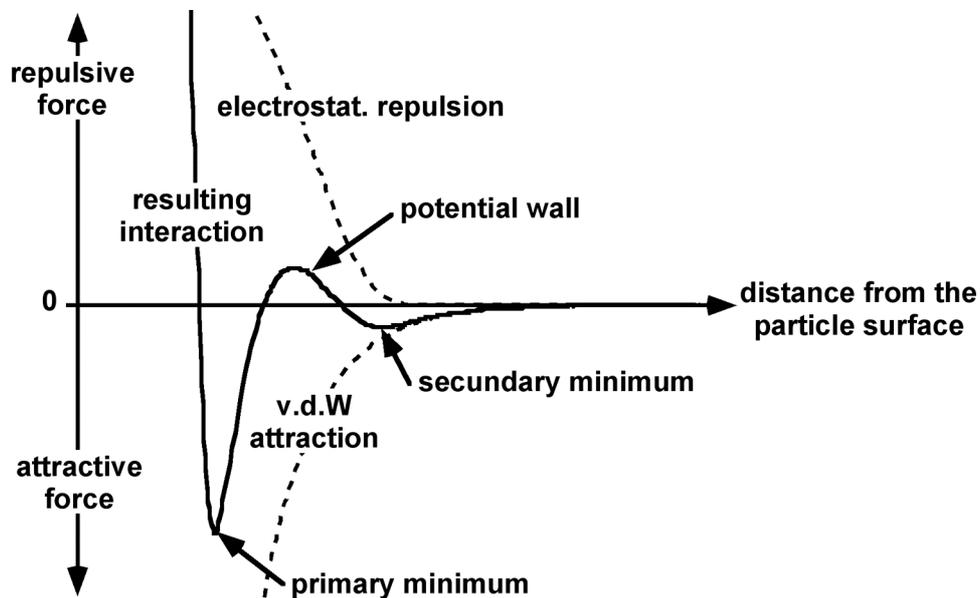
The particle separation from liquids can be made easier or possible at all by different methods of suspension pre-treatment. The most important procedures for the suspension pre-treatment are

- agglomeration of very small particles,
- preconcentration of the suspension,
- addition of filter aid material,
- suspension degritting or desliming and
- defoaming of suspensions.

The pre-concentration of highly diluted suspensions and classifying represent already independent steps of solid-liquid separation, however, serve often in a combination arrangement as necessary preliminary stage for an apparatus downstream for the most extensive mechanical liquid separation. The pre-concentration of a suspension serves above all to withdraw a remarkable part of clear liquid at minimal possible expenditure to relieve the following more effective and more costly separation equipment. For this operation in particular separation procedures are well suited, which deliver the solids anyway in still flowable form. Static and centrifugal sedimentation, cross-flow filtration and backflushing filters are normally used for this purpose. In particular the sedimentation procedures are frequently combined with particle agglomeration.

The separation of particles with diameters less than approximately 10  $\mu\text{m}$  is substantially facilitated by agglomeration. On one hand agglomerates are settling faster than single particles. On the other hand individual sedimentation of single particles usually unwanted for separation processes can be transferred into swarm sink behaviour. This is characterized by the fact that all particles independently of their size, shape or spec. weight are settling with nearly the same velocity and a sharp sedimentation front is forming. The integration of the whole particle size distribution into the agglomerates prevents an undesirable demixing of the particles according to their size. Moreover, the necessary clarification surface of a sedimentation apparatus must not be laid out any more according to the smallest single particle to ensure particle-free overflow. In filtration processes more permeable filter cakes are formed in the case of particle agglomeration. This leads to a comparable faster cake formation. Furthermore the filtrate pollution by particles, which are able to get through the filter medium in the very first moment of cake formation can be reduced or even totally prevented by agglomeration. In order to avoid pore blockage of the filter medium, its pore size often is chosen so large, that for a complete particle separation as a start particle bridges must be formed across the meshes. Finally also in filtration processes a particle demixing can be avoided by agglomeration, because gravity cannot be switched off and sedimentation takes place in the suspension in every case. To build up agglomerates mainly van-der-Waals forces in case of coagulation and polymer molecules of large molecular weight in case of bridging flocculation are used (Russel et. al., 1989). Yet less important today are processes present in development like electrocoagulation and magnetic flocculation (see chapter 2.4). Particles, which are dispersed in ion containing liquids,

in general carry an electric surface charge and are repelling each other. In order to enable a particle adhesion by van-der-Waals forces the electrostatic repulsion must be reduced to a level, which allows the particles to come together close enough. The balance between attractive and repulsive forces is described in the DLVO theory. Figure 2.51 shows this in simplified representation.



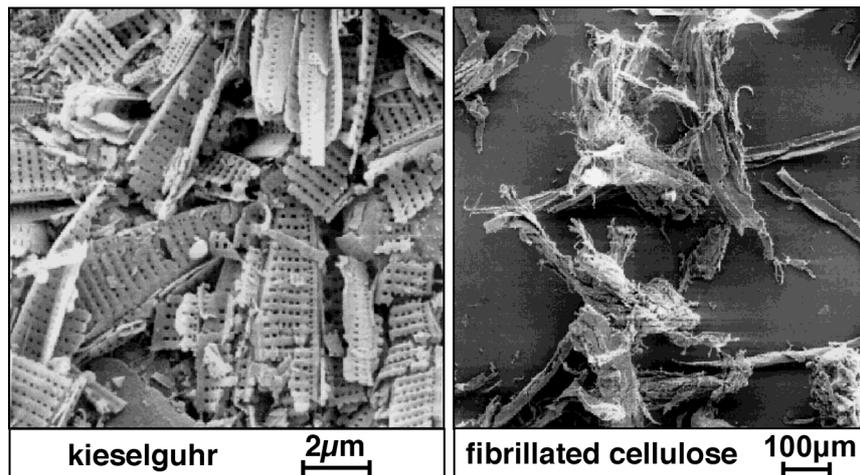
**Fig. 2.51** Attraction and repulsion as function of distance from the particle surface

In the case shown here a strong primary and a more weak secondary maximum of adhesion is forming as a result of balancing repulsive and adhesive forces. With accordingly large kinetic energy particles can overcome the remaining barrier of the potential wall and agglomerate very stable in the primary maximum of adhesion. The physico/chemical conditions favourable for coagulation can be adjusted by the pH value and/or the ion concentration of the suspension (Lyklema, 1991, 2001). As a measure for the electrostatic charge and derived from this the mutual repulsion of the particles serves the Zeta-potential, which can be determined by measurement. If the Zeta-potential becomes zero at the isoelectric point the particles show outwardly no electrostatic charge. In this condition they are able to adhere maximally.

Furthermore particles can be flocculated by addition of special soluble polymers. The adhesion between the particles is realized here by partial adsorption of the polymers and thus by direct interlinking of the particles. According to the electric charge one can distinguish between anionic, cationic and nonionic polymers. As polymeric chain frequently polyacrylamide is used. The molecular weight is in the order of magnitude of approx. 10 millions. The solids mass concentration of the solution ready for use usually is chosen between 0.03÷0.1%. The preparation of the flocculant solution, the dosage, the mixture of flocculant and suspension and the controlled floc formation represent an independent step in the entire solid-liquid separation process chain. The necessary amount of flocculant is analyzed by charge detectors and the analysis of the agglomeration result to find out the optimal flocculant can be done by modern analytic centrifuges (Sobisch, Lerche, 2000). Depending on the real technical separation process the agglomerates must withstand different kind of mechanical load. Flocs, which are separated in a centrifuge or in a pressure filter must be much more stable than those in a gravity settling tank. The strength of agglomerates can be adjusted in special flocculation reactors.

However, in many separation processes like often in the food or pharmaceutical industry the addition of foreign matter is forbidden. In such cases usually the driving forces must be increased to solve the separation problem.

The mixture of a hard to separate suspension with mineral or organic filter aid materials can lead to an increased permeability of filter cakes. This procedure is called „body feed filtration“. This method in particular is used, if the liquid represents the value product. The range of the materials used here is very large. Today mineral filter aids like diatomaceous earth (kieselguhr) or perlite are displaced increasingly by organic filter aids based on cellulose from renewable raw materials. Figure 2.52 shows microscopic pictures from diatomaceous earth and cellulose fibers.



**Fig. 2.52** Examples for filter aid materials

Depending upon the special requirements of the process can be selected by the natural finished wood flour over extract-free cellulose up to highly pure cellulose from many different products.

In the case of very broad particle size distributions the largest as well as the smallest particles can turn out to be disturbing the separation process. Separation apparatuses like disc stack separators or hydroclones with nozzle discharge but also cross-flow modules with very close flow channels must be protected against coarse particles, which can block the nozzle or flow channel. On the other side a small quantity of finest particles can already clog the pores of a filter cake and generate a very high pressure loss. Depending on particle size and flow rate most different apparatuses can be used to separate such disturbing particle fractions. Coarse particles can be removed for example by strainers or bended screens, fine particles by hydrocyclones or up-stream classifiers. As pre-filter for ultra-filtration modules for mechanical molecule separation depth or even micro-membrane filters can be applied.

With foaming suspensions it can be advantageous or necessary for a safe and troublefree operation of separation apparatuses to degrade the foam by chemical or mechanical means. Foams are developing in flotation processes and in particular in the presence of organic substances like for instance proteins. Foam condensation by chemical means in most cases is not preferred due to the necessary addition of substances. More advantageous is the mechanical foam suppression via water spray or special defoaming turbines.

## 2.7 Notation

### Latin letters

a	centrifugal acceleration	$\text{m s}^{-2}$
c	concentration	-
C	centrifugal number	-
d	diameter or size of particles	m
F	force	N
Fr	Froude number	-
g	acceleration due to gravity	$\text{m s}^{-2}$
H	height	m
H	field strength	$\text{A m}^{-1}$
P	pressure	Pa
R	radius	m
S	saturation	-
t	time	s
v	velocity	$\text{m s}^{-1}$
V	Volume	$\text{m}^3$

### Operators

$\nabla$	gradient operator
$\Delta$	difference operator

### Greek letters

$\alpha$	spec. filter cake resistance	$\text{m}^{-2}$
$\beta$	filter medium resistance	$\text{m}^{-1}$
$\chi$	volume based susceptibility	-
$\delta$	wetting angle	°
$\varepsilon$	porosity	-
$\kappa$	concentration parameter	-
$\mu$	dynamic viscosity	$\text{kg m}^{-1} \text{s}^{-1}$
$\mu$	permeability number	$\text{V s A}^{-1} \text{m}^{-1}$
$\rho$	density	$\text{kg m}^{-3}$
$\sigma$	surface tension	$\text{N m}^{-1}$
$\omega$	angular velocity	rad

### Subscripts

c	capillary
e	entry
g	gas
L	liquid
mag	magnetic
p	particle
r	remanent
s	solid
St	Stokes
surf	surface
v	void
V	volume based
0	vacuum

## 2.7 References

- Anlauf, H., 2004. The filter medium – crucial interface between apparatus and suspension. *Proceedings of the 9th World Filtration Congress*, New Orleans, USA, session 336.2
- Anlauf, H., 2006. Recent Research and Machinery of Solid Liquid Separation Processes. *Drying Technology*, **24**:1235-1241
- Anlauf, H., 2007a. Overview and recent developments in effective particle decontamination by washing processes. *Filtration*, **7**(1):20-25
- Anlauf, H., 2007b. Recent development in centrifuge technology. *Separation and Purification Technology*, **58**:242-246
- Anlauf, H., 2008. Solid-Liquid Separation by Cake Filtration – State of the Art and Future Expectations. *Proceedings of the 10th World Filtration Congress*, Leipzig, Germany. Volume I: 21-28 and *Global Guide of the Filtration and Separation Industry*, VDL, Rödermark, Germany: 108-114. ISBN 978-3-00-024080-5
- Bott, R., et. al., 2003. Recent Developments and Results in Continuous Pressure and Steam Pressure Filtration. *AT Mineral Processing*, **44** (5): 5-18
- Chen, X. et al., 2002. Novel Electrode System for Electroflotation of Wastewater, *Environ. Sci. Technol.*, **36** (4): 778-783
- Delgado, A.V. et. al., 2007. Measurement and interpretation of electrokinetic phenomena. *Journal of Colloid and Interface Science*, **309**: 194-224
- Dickenson, T.C., 1997. *Filters and Filtration Handbook*. Elsevier, Oxford, U.K.. ISBN:1 85617 322 4
- Eichholz, C., et. al., 2008. Magnetic field enhanced cake filtration of superparamagnetic PVAc-particles. *Chemical Engineering Science*, **63**: 3293-3300
- Fuerstenau, M.C., Yoon, R.-H., 2007. *Froth Flotation – A Century of Innovation*. SME, Sci-Tech Book News, Portland, USA
- Gimbel, R. et. al., 2006, *Recent Progress in Slow Sand and Alternative Biofiltration Processes*. IWA Publishing, London, U.K.. ISBN: 9781843391203
- Höflinger, W., 2008. Depth filtration with fibrous filter layers. *Global Guide of the Filtration and Separation Industry*, VDL, Rödermark, Germany: 136-138. ISBN 978-3-00-024080-5
- Huotari, H.M., et. al., 1999. Crossflow Membrane Filtration Enhanced by an External DC Electric Field: A Review. *Chemical Engineering Research and Design*, **77** (5): 461-468
- Jornitz, M., Meltzer, Th., 2001. *Sterile Filtration*. Marcel Decker, New York, USA. ISBN: 0-8247-0282-4
- Kopf, M.H. et. al., 2008. The centrifuges of today and tomorrow: larger, more efficient and more specific. *Global Guide of the Filtration and Separation Industry*, VDL, Rödermark, Germany: 125-130. ISBN 978-3-00-024080-5
- Leung, W.W.-F, 1998. *Industrial Centrifugation Technology*. McGraw-Hill, New York, USA. ISBN: 0-07-037191-1
- Leung, W.W.-F, 1998. *Centrifugal Separations in Biotechnology*. Elsevier, Oxford, UK. ISBN: 978-1-85-617477-0

- Lyklema, J., 1991. *Fundamentals of Interface and Colloid Science; Fundamentals*, Academic Press, London, U.K.. ISBN: 0-12-460525-7
- Lyklema, J., 2001. *Fundamentals of Interface and Colloid Science; Solid-Liquid Interfaces*, Academic Press, London, U.K.. ISBN: 0-12-460524-9
- Lyko, H., 2008. Membrane technology: Innovative separation techniques for many fields of application. *Global Guide of the Filtration and Separation Industry*, VDL, Rödermark, Germany: 144148. ISBN 978-3-00-024080-5
- Peuker, U., Stahl, W., 2001. Steam Pressure Filtration: Mechanical-Thermal Dewatering Process. *Drying Technology*, **19** (5): 807-848
- Purchas, D., 1996. *Handbook of Filter Media*. Elsevier, Oxford, UK. ISBN: 1-85617-278-3
- Rao, S.A., 2004. *Surface Chemistry of Froth Flotation*. Springer, Heidelberg, Germany. ISBN: 978-0-306-48180-2
- Ripperger, 2008. Filter media for cake filtration. *Global Guide of the Filtration and Separation Industry*, VDL, Rödermark, Germany: 115-124. ISBN 978-3-00-024080-5
- Rushton, A., Ward, A.S., Holdich, R.G., 1996. *Solid-Liquid Filtration and Separation Technology*. VCH, Weinheim, Germany. ISBN: 3-527-28613-6
- Russel, W.B., et. al., 1989. *Colloidal Dispersions*. Cambridge University Press, Cambridge, U.K. ISBN: 0-521-42600-6
- Sobisch, T., Lerche, D., 2000. Application of a new separation analyzer for characterization of dispersions stabilized with clay derivatives. *Colloid Polym. Sci*, **278**: 369-374
- Sorrentino, J. A., 2002. *Advances in correlating filter cake properties with particle collective characteristics*. Shaker, Aachen, Germany. ISBN: 3-8322-0972-7
- Sutherland, K. S., 2005. *Solid/Liquid Separation Equipment*. Wiley-VCH, Weinheim, Germany. ISBN: 3-527-29600
- Svarovsky, L., 2000. *Solid-Liquid Separation*. Butterworth Heinemann, Oxford, UK ISBN: 0750645687
- Svoboda, J., 1987. *Magnetic Methods for the Treatment of Minerals*. Elsevier, Amsterdam, Netherlands
- Wakeman, R.J., Tarleton, E.S., 2005. *Solid Liquid Separation - Principles of Industrial Filtration*. Elsevier, Oxford, UK. ISBN: 1856174190
- Wakeman, R.J., Tarleton, E.S., 2005. *Solid Liquid Separation – Scale-up of Industrial Equipment*. Elsevier, Oxford, UK. ISBN: 1856174204
- Wakeman, R.J., Tarleton, E.S., 2007. *Solid Liquid Separation – Equipment Selection and Process Design*. Elsevier, Oxford, UK. ISBN: 1856174212
- Weber, K, Stahl, W., 2002. Improvement of filtration kinetics by pressure electrofiltration. *Separation and Purification Technology*, **26**: 69-80

