

SOLID-LIQUID SEPARATIONS IN PILOT- AND COMMERCIAL- SCALE OPERATIONS

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ABSTRACT

Benchtop processes that are developed with the intent of being run at the demonstration scale present a multitude of technical challenges. Solid-liquid separations are among the most common process operations that become large and often unanticipated capital and operational costs due to insufficient development effort in the research phase. Often separation technologies from established industries can be applied to new processes to mitigate the risk involved with scale up. The presentation will focus on Hazen's experience with solid-liquid separations, including dissolved air flotation (DAF), counter current decantation (CCD), as well as various incarnations of filtration and centrifugation.

SOLID-LIQUID SEPARATIONS

Separation costs are difficult to isolate given the complexity of the chemical industry but have been estimated to account for between 40 and 70% of capital and operating costs in a commercial process.³ The principles and technologies are well-established and criteria for equipment selection are available, yet these unit operations continue to present difficulties in scale up and operations. Separation processes are often not given sufficient attention at the pilot scale; it is more common to adopt a trial-and-error method in development until a piece of equipment that offers promise is selected at the bench scale. Very often, a small variance in the process can render the specified equipment useless and cause a return to the design phase or worse, development at scale.

All mechanical separation equipment, with the possible exception of those used for impingement, are based on the principles of sedimentation or filtration,⁴ as shown in Figure 1.

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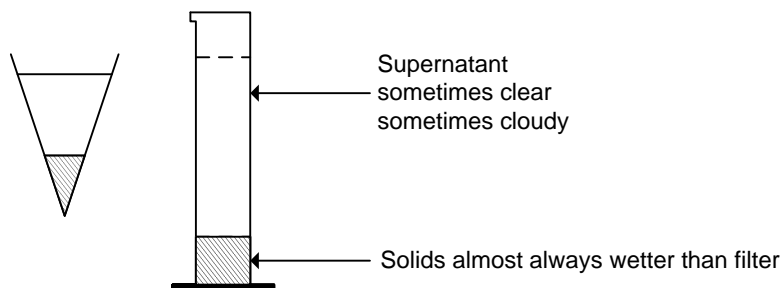
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³Spear, M., 2009. Separations in flux. <http://www.chemicalprocessing.com/articles/2006/025.html> (accessed September 27, 2010).

⁴Pierson, H.G.W., "The Selection of Solid-Liquid Separation Equipment", *Solid-Liquid Separation*, L. Svarovski, 2nd ed., 1981, p. 525.

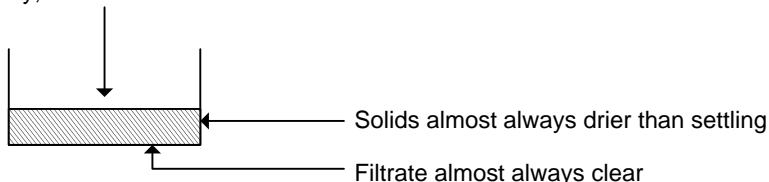
Figure 1. Sedimentation and filtration schematics⁵

Settling Systems



Filter systems

Gravity, Pressure or Vacuum



Each technique has numerous configurations and applications, along with well-established strengths and weaknesses, yet all are dependent on the nature of the two phases in the slurry. Often, separation unit operations that have not had sufficient attention in the research phase of a process will fall to centrifugation or belt filtration equipment. Both of these technologies allow for a measure of flexibility (which makes them a relatively safe bet to accomplish a separation), but the respective capital costs of these technologies are very high. Continuous vacuum filtration equipment costs generally range from \$1,400 to \$3,700/ft² FOB point of manufacture. Scroll-conveyor sedimentation centrifuges are dependent on the required bowl diameter, but can cost up to \$150,000 for simple carbon steel. Special materials of construction, high throughputs, and material handling issues that require a large unit such as a Sharples Super D-Canter can run as high as \$300,000.⁶ Installed costs will vary depending on the equipment requirements, but for commercial centrifuge units, infrastructure and ancillary equipment can generally be expected to be 1–3 times the purchase price.⁷

Savings are certainly realized when the development step is minimized, but short-cutting the generation of proper data to select and properly size the solid–

⁵Ibid., p. 526.

⁶Peters, Max S., Timmerhaus, Klaus D. and West, Ronald E., Plant Design and Economics for Chemical Engineers, 5th ed. McGraw-Hill, 2003, pp. 864-867, corrected by Annual Index (2003-2009) Chemical Engineering Plant Cost Index (CEPCI), Chemical Engineering, Volume 117, No. 7, July 2010, p. 60.

⁷Perry, Robert H., and Green, Don W., editors, Perry's Chemical Engineer's Handbook, 7th ed., McGraw-Hill, 1997, pp. 18-125.

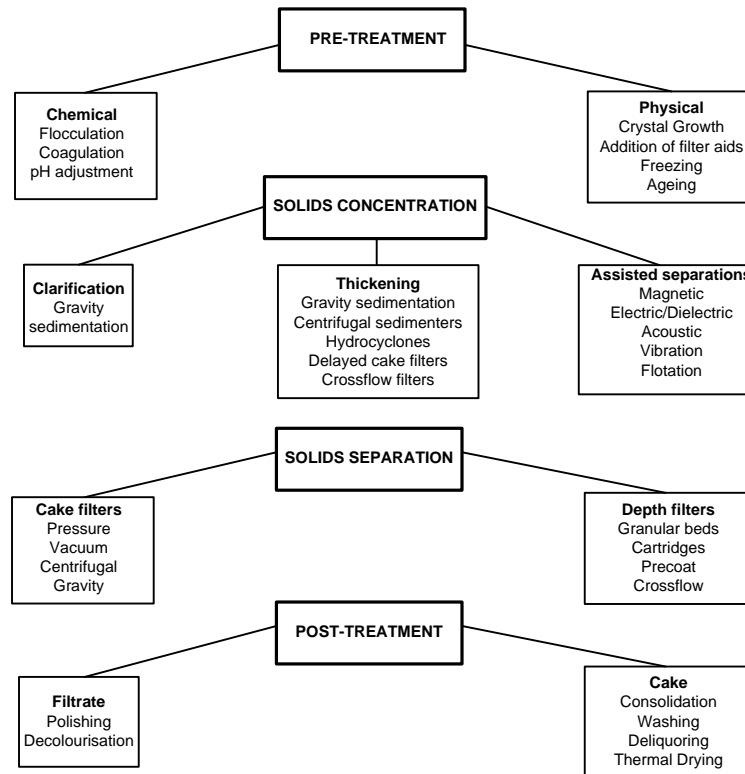
liquid separation equipment usually results in poor performance. Some of the most expensive capital equipment in a process requires larger-than-expected operating costs, and can potentially become the rate-limiting step in an entire process, necessitating an expensive retrofit.

A number of excellent publications have covered the topic of solid–liquid separation technologies and equipment selection criteria. The current paper will attempt to introduce the core principles found in these texts, comment on our experience with these technologies, and provide a brief overview of the process options available to the engineer. For further reference, a complete bibliography has been included in the last section of the document. In general, the examples follow the Duty Specification and Separation Characteristics classification first published by Purchas and Wakeman in 1986.⁶

The first step in tackling the solid–liquid separation needs for a process is to define the requirements of the separation. Aside from the general scale of the proposed operation, the product phases must be defined; if both the solids and liquids are to be collected, the choice of available technologies may be somewhat limited. Generally, a solid–liquid separation is used to recover 1) valuable solids such as crystals formed in a purification process, 2) a valuable liquid such as a plant extract, 3) both phases such as leachate and tails both containing mineral values, or 4) neither phase such as the removal of particulates of a wastewater stream prior to discharge. Other considerations are the acceptable yields, required throughput, the value of the product(s), potential feedstock variation, the ability to introduce reagents to aid in the separation, the requirement for batch integrity and cleanliness, and the acceptable liquid content of the solids, both before and after a potential wash step. Engineering concerns include the footprint of various technologies, as well as required infrastructure, capital costs, lead times, and maintenance costs and labor.

It quickly becomes clear that specifying a particular unit operation for solid–liquid separations is complex and presents a significant challenge in most cases. It is crucial that experimentation at the laboratory scale for a process include studies on slurry behavior and sedimentation/filtration tests. One advantage of a thorough assessment of available technologies is the revelation that multiple techniques may be employed to effect the required balance between efficiency and throughput. In this way, what normally appears as a single operation in the classical process flow diagram becomes a small process itself. This is shown in Figure 2.

Figure 2. Solid-liquid separation as a multi-component process⁸



If one considers the possibility of pretreating or concentrating a particularly large process stream, separation and post-treatment options may still be required, but the slurry stream will in theory be much smaller, opening the door to more capital-intensive technologies that would previously have been untenable. A number of the options listed in the chart are covered in this document, along with some general notes on predicting behaviors and laboratory programs that can test the suitability of a given system.

It should be noted that chemical treatment of a slurry is a common and often very effective practice that enables the use of many of the techniques described in this document. However, the subject is not within the scope of the current document. The principles and techniques for chemical treatment of slurries (addition of coagulants and flocculants, filter aids, etc.) is covered in depth in many of the sources listed in the bibliography.

SEDIMENTATION

Sedimentation is based on the behavior of solids suspended in a fluid under the influence of a number of forces, often the force of gravity, but also potentially centrifugal, buoyant, and compression forces. The particle flow through a fluid is

⁸Wakeman, R. and Tarleton, S., Solid/Liquid Separation: Principles of Industrial Filtration, Elsevier Advanced Technology, 2005, p. 2.

described by the Reynolds number,⁹ which is essentially a ratio of the inertial forces (in the simplest case, gravity or centrifugal force) to viscous forces (drag force and buoyancy).

$$Re = \rho u x / \mu$$

Where ρ and μ are the density and viscosity of the fluid, respectively, u is the particle-fluid relative velocity, and x is the particle size. In systems for which the particles are very small or nearly neutrally buoyant or for which the fluid is sufficiently viscous, the Reynolds number will be small; for most solid-liquid systems that present separation difficulties, the solids are present as fine particles and are difficult to separate, and $Re < 0.2$. This is important, as the flow around the particles as they settle is laminar and makes possible the use of the Navier-Stokes equations for calculating the drag force, and allows one to assume that the time to the terminal settling velocity can be assumed to be close to zero. The calculation for determining the terminal settling velocity then becomes:¹⁰

$$u_t = x^2 (\rho_s - \rho) g / 18\mu$$

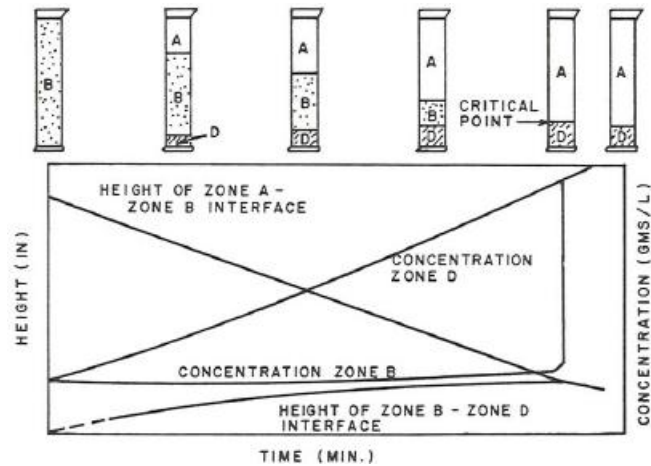
This parameter is key in evaluating potential technologies that could be used in the separation process. In Hazen's experience, values above 0.4–0.5 m/h warrant experimental verification and further study to size equipment, whereas values below this range indicate that the size of the equipment required to effect a gravity sedimentation will be prohibitively expensive and that gravity separation is not a feasible solution.

The calculated terminal settling velocity only represents a general idea of how a slurry will perform in sedimentation. As the solids settle out of solution, their behavior changes due to their increasing proximity to other particles in solution, to the point where several zones of concentration are formed (see Figure 3). Eventually, a point is reached at which the sedimentation presents a single interface between the solids and the clear liquid. This is known as the critical separation point; the sedimentation behavior of the solids is fundamentally changed, and the settling velocity no longer represents the movement of the particles; the sedimented pulp slowly compresses as liquid is forced upwards through channels in the deposit.

⁹Ibid., p. 20.

¹⁰Ibid., p. 22.

Figure 3. Plots of interface height and solids concentration versus time for batch settling¹¹



The calculated settling behavior of particles in a slurry can be verified by a simple Kynch test, in which a slurry of known volume is allowed to settle in a graduated cylinder. By measuring the height of the interface over time and sampling the final settled solids to determine their concentration, a clear picture of the slurry behavior will usually indicate if the expected settling rate is borne out and, more importantly, how appropriate gravity sedimentation may be for a particular process. Utilizing a plot of the data generated during the settling test, the Kynch method will also allow for a calculation of the required unit area to sediment a slurry in $\text{ft}^2/\text{short ton/day}$ (or $\text{m}^2/\text{metric ton/day}$), which will drive design decisions and the subsequent cost/benefit analysis. The equation used to generate this figure includes a correction for the depth of the settled solids based on empirical comparisons between full-scale thickeners and lab-scale cylinders.¹²

TYPES OF SEDIMENTATION

Sedimentation is generally divided into two types of processes. Thickening refers to the concentration of solids in a feed stream, while clarifying refers to the removal of solids from a relatively dilute feed stream.¹³ Thickeners/clarifiers operate on the same principles and are, in their most basic form, circular settling tanks with a rake to move the settled solids to a central underflow discharge. To aid in the separation, the bottom of the tank is generally sloped towards a discharge port in the center. These vessels are often large, as this allows for sufficient throughput given the typical terminal velocity of settled solids. The overflow and the underflow can be collected continuously, allowing for an

¹¹Figure from Chermisinoff, Nicholas, Pocket Handbook for Solid-Liquid Separations: Calculations and Guidelines for Process Engineering, Gulf Publishing Company, 1984, p. 89.

¹²For details, see Seidel, D. C. "Laboratory Procedures for Hydrometallurgical-Processing and Waste-Management Programs", United States Department of the Interior Bureau of Mines Information Circular 9431, 1995.

¹³Chermisinoff, p. 85.

extremely large process flow given the correct conditions. A cross section showing the various parts of a basic thickener is shown in Figure 4.

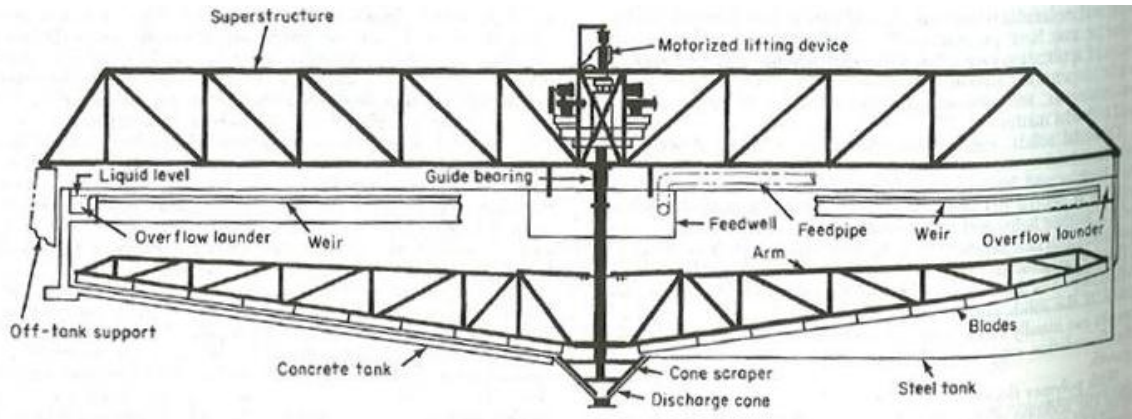
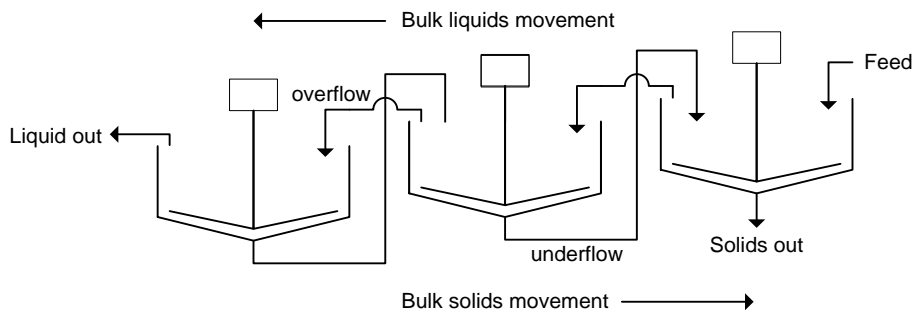


Figure 4. Unit thickener (EIMCO Process Equipment Co.)¹⁴

A very common use of sedimentation technology is with continuous countercurrent decantation (CCD). This application includes a number of thickeners arranged in series, where the feed enters the circuit in one direction and the underflow is continually pumped upstream, resulting in a bulk clarified liquid flow in one direction (the same as the feed), and the bulk flow of concentrated solids slurries moves in the other direction. A diagram of a CCD process is shown in Figure 5.

Figure 5. Schematic diagram of a CCD process



One of the advantages of this type of operation is the ability to tolerate large swings in the feed composition. Because the holdup volume of these types of systems is so large, short-term slurry concentration variances are absorbed without much difficulty.

FLOTATION

¹⁴Perry and Green, pp. 18-64.

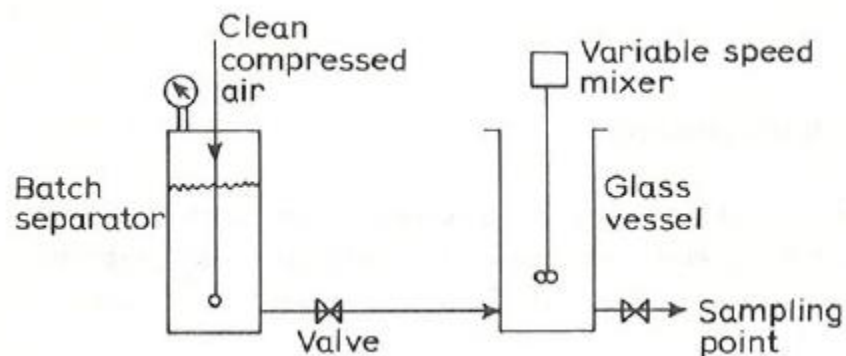
Flotation processes rely on the affinity of the solids for the solution in which they are contained and are generally applied to aqueous systems in which the introduction of a gas will alter the buoyancy of the solids through adhesion and subsequent gas–solid agglomeration. Many factors (nature of the solids, size of the gas bubbles formed, surface tension, etc.) affect the behavior of a flotation system, and extensive experimental programs are often needed to determine the optimal setup. Flotation is governed by the same equation as sedimentation, the difference being that now the relative density of the solids will be less than that of the liquid and will rise in solution, eventually at the terminal velocity described by:

$$u_t = x^2 (\rho_l - \rho_g)g / 18\mu$$

Where, in this case, x^2 is the diameter of the gas–solid agglomerate, and ρ_l and ρ_g are the respective densities of the liquid and the gas.¹⁵ This equation is applicable for solutions with low Reynolds numbers (< 1) and gas bubbles that are sufficiently small (~0.2 mm).

In general, the smaller the bubbles the better the flotation, because smaller particle sizes will also aid in the adhesion process. Generally, it is easier to alter the size of the bubbles in a separation system, as the particle sizes are often driven by preceding steps in the process that created the slurry. For systems in which high mixing rates are possible (the solid phase is not friable), dispersed air flotation can be used, where the gas is entrained in the solution by vigorous mixing. A more common technique is dissolved air flotation (DAF), where the gas and the slurry are mixed under pressure, which is then released, causing the formation of very small gas bubbles. An example of a test setup for this is shown in Figure 6.

Figure 6. A DAF test apparatus¹⁶

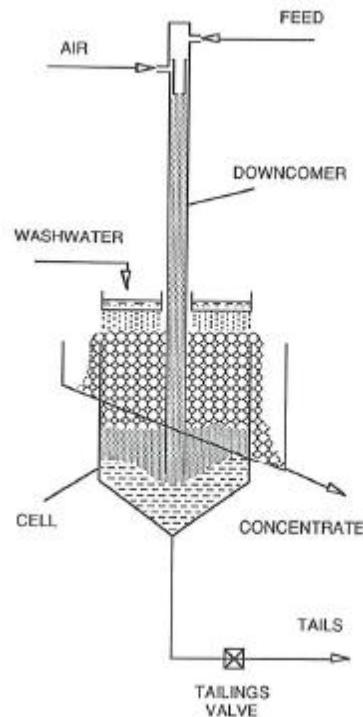


¹⁵Matis, K.A. and Zouboulis, A.I., "The Role of Bubble/Particle Size", *Flotation Science and Engineering*, Marcel Dekker, Inc., 1995, p. 79.

¹⁶Gochin, R.J., "Flotation", *Solid-Liquid Separation*, 2nd ed., edited by Svarovsky, Butterworths and Co., 1981, p. 517.

A number of different flotation technologies have been developed with varying geometries and applicability. Separation efficiency, sensitivity to system inputs (gas introduction rate location and methods, necessity for positive bias (air bubble/solids agglomerates retain their buoyancy) through the net downflow of wash water), selectivity, and capital requirements have led to the development of robust simple configurations, as well as very specialized and fully engineered flotation systems, such as the Jameson Cell, shown in Figure 7.¹⁷

Figure 7. Layout of a Jameson Cell



The Jameson Cell allows for significant contact time between the gas and the feed in the downcomer. Once the mixture moves into the cell, the reduction in the downward superficial velocity allows the particle/bubble agglomerates to rise to the surface as a froth, which then overflows the cell and is collected.¹⁸

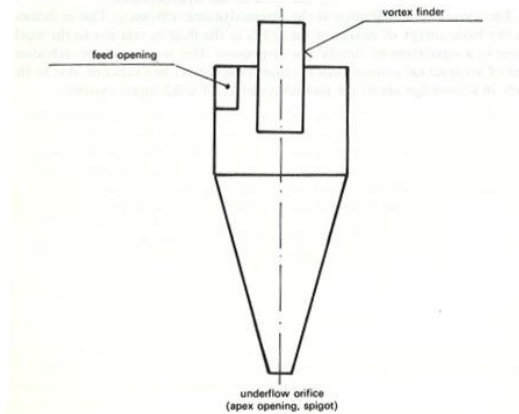
HYDROCYCLONES

The hydrocyclone is a cono-cylindrical vessel with an outlet at the bottom and at the top, fed by a tangential inlet as shown in Figure 8.

¹⁷Evans, Geoffrey M., Atkinson, Bruce W. and Jameson, Graeme J., "The Jameson Cell", *Flotation Science and Engineering*, Marcel Dekker, Inc., 1995, p. 334.

¹⁸Ibid, p. 333-334.

Figure 8. Basic hydrocyclone diagram¹⁹



The separation of solids in the hydrocyclone is typically size-dependent, and the terminal velocity of the particle can be described by a slightly more complicated expression of the Stokes equation:

$$u_t = (\Delta\rho/\rho)\tau^*a$$

Where $\Delta\rho$ is the density difference $\rho_s - \rho$, τ^* is the particle relaxation time $\rho_s x^2 / 18\mu$, and a is the field acceleration of particles, which changes with particle position.²⁰ It should be noted that the primary strength of a hydrocyclone separation is in its ability to make fine cuts in particle size, once properly sized. Rather than a wholesale solids separation, this technology is best utilized as a pretreatment/solids conditioning step, aiding the efficiency of a subsequent solids separation step. A good example of this would be fines removal using a hydrocyclone that would allow for better flow through a filter bed (see p. 13).

Key to the operation of the hydrocyclone is the sizing and geometric arrangement of the cone, as well as the outlet downcomer, commonly called the vortex finder. Once a stable vortex is formed in the hydrocyclone body, the presence of the vortex finder will create circulation eddies that generate areas of lower resistance where fine particles can flow from the high-pressure areas near the inner cyclone wall towards the low-pressure areas in the center of the unit. A central air core usually forms in the center of the hydrocyclone, an indication of a stable vortex and a place where dispersed and even dissolved gases will report during operation.²¹

Particle movement in a slurry flowing in a hydrocyclone can be described by the Reynolds number according to the following equation:

¹⁹Svarovsky, L., Hydrocyclones, Hold, Rinehart and Winston Ltd., 1984, p. 30.

²⁰Ibid., p. 7.

²¹Ibid., p. 33.

$$Re = (v \cdot D \cdot \rho) / \mu$$

Where v is the characteristic velocity (tangential component of the fluid velocity), D is the hydrocyclone diameter, and ρ and μ are again the density and the viscosity of the liquid, respectively. Using this general behavioral equation, one sees that an increase in diameter will result in a larger Reynolds number and hence a rougher cut; finer particles with a slight density difference from the liquid phase will be more inclined to report to the underflow. For this reason, hydrocyclone applications are often designed to include multiple units working in concert as opposed to a single large unit. Fortunately, this also aids in the hydrocyclone behavior, as very large units must account for gravity's influence, while smaller units can be installed in just about any orientation as needed.²²

CENTRIFUGAL SEDIMENTATION

Centrifugal sedimentation also relies on the density difference between liquids and solids subjected to a centrifugal force. Operation of the centrifuge allows for the application of much greater forces than are possible in a hydrocyclone, enabling more efficient separations. Assuming the concentration of solids in a slurry is sufficiently low that interaction between the particles can be neglected, the behavior of solids in a sedimenting centrifuge can be described by a further modification of Stokes' law:

$$dr/dt = (\Delta\rho x^2 r \omega^2) / 18\mu$$

Where r is the radial position of the particle of size x , t is the time interval that the particle is subjected to the centrifugal acceleration, $\Delta\rho$ is the density difference between the solid and the liquid, ω is the angular velocity, and μ is the fluid viscosity.²³

Due to the complex nature of the interactions between slurry components and the body of a centrifuge, numerous factors must be considered when scaling a sedimenting centrifuge, particularly when evaluating units that are not of similar geometry. One way of attempting to evaluate this is to consider the Sigma factor, which can normally be approximated as:

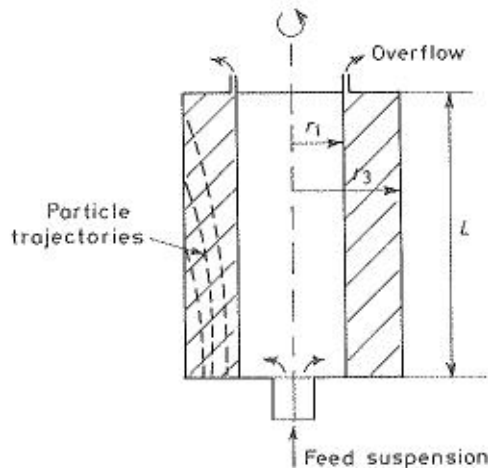
$$\Sigma \approx (\omega^2/g)\pi L(3/2 r_3^2 + 1/2 r_1^2)$$

Where ω is the angular velocity, L represents the full length of the settling zone, and r_3 and r_1 represent the total radius of the rotating portion of the centrifuge and the distance between the center axis and the start of the settling zone, respectively. This is shown in Figure 9 below.

²²Ibid., p. 83.

²³Svarovsky, L. "Separation by Centrifugal Sedimentation", Solid-Liquid Separation, 2nd ed., edited by Svarovsky, Butterworths and Co., 1981, p. 192.

Figure 9. Schematic diagram of a simple tubular centrifuge²⁴



The Sigma factor is a widely used criterion for the comparison of centrifuges of similar geometry, liquid flow patterns, and the same centrifugal gravity (G -force)²⁵ and allows for the approximation of unit performance at scale from experimental data taken with a smaller unit. The Sigma factor makes some assumptions that cause the correlation to be inexact; Stokes law is likely not entirely descriptive of the settling solids behavior over the entire range of G , as the solids in the liquid stream may contain entrained liquid once settled, eddies and flow disruptions probably occur at the entrance and the exit of the unit that are disregarded, etc. To offset these known weaknesses in the Sigma factor, efficiency factors are utilized, which are often well understood and constant (tubular centrifuges) or can vary widely (decanting centrifuges) depending on the nature of the settled solids removed and other considerations.

Great care must be taken in selecting a centrifuge for a commercial operation as the capital expense, as well as operational and maintenance costs for these unit operations, can be very large.

Sedimentation technologies are very useful in solid–liquid separations and are often powerful yet inexpensive techniques that can be used to concentrate solids before the final solid–liquid separation. Likewise, if a particular particle size in a slurry is the desired product, the use of sedimentation principles can often achieve the product separation. The major disadvantage of the majority of these technologies is the requirement for continuous operation, precluding any ability to maintain batch integrity, as well as the addition of large amounts of liquid in order to achieve the solids concentration desired. While this is not an issue for many industries, those that are more heavily regulated from a quality standpoint may find this problematic.

FILTRATION

²⁴Ibid., p. 191.

²⁵Perry and Green, pp. 18-116.

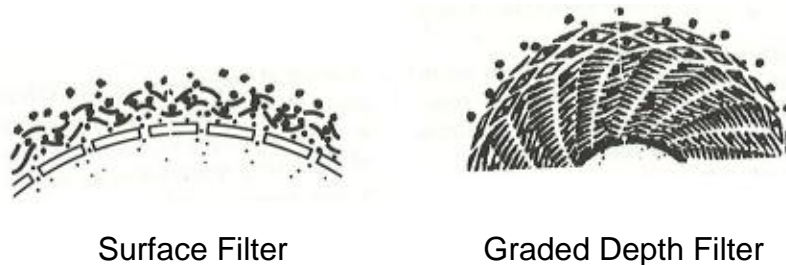
The fundamental difference between filtration and sedimentation is, of course, the existence of a barrier in filtration that is impermeable to one of the constituents of a slurry. The flow of liquid through the cake that builds on the barrier (filter media) can be described by Darcy's basic filtration equation:²⁶

$$Q = K(A\Delta p/\mu L)$$

Where Q is the flow through the bed of solids, K is the permeability of the bed, A is the surface area of the filtration, Δp is the driving pressure, μ is the fluid viscosity, and L is the thickness of the bed. Naturally, as a filtration progresses, many of these factors are changing and most of them are independent of the equipment used, save for designs that are utilized to benefit the flow (i.e., removing a cake from the filter surface during operations so that the bed thickness remains small). Typically, pretreatment or solids concentration of a slurry will greatly aid filtration, as particle behavior impacts flow to a significant degree. In sedimentation this is not such a concern, as the settling of solids displaces entrained liquid but no actual flow through the settled solids is required.

There are two general classes of mechanical filtration: surface filtration, which works largely by direct interception of the particles from the slurry as it passes through the surface of the filter, and depth filtration, which utilizes a medium that has increasingly dense layers from the upstream to the downstream side. The two types are shown in Figure 10.

Figure 10. A surface filter compared with a graded-depth-type filter.²⁷



Applications that utilize surface filters generally encounter a reduction in the effective pore size, as accumulating particles gradually close off pores. This phenomenon can be exploited to aid in a filtration (see below), but can also be the cause of a functioning filtration unit suddenly losing any effective flow through the bed as the pore sizes become so fine that they will not allow sufficient liquid flow. Softer particles that are more prone to deformation under the influence of force can also block pores in a surface filter, which is one of the reasons why filtering gels can present a significant problem. The filter shown in Figure 10

²⁶Svarovsky, L. "Filtration Fundamentals", Solid-Liquid Separation, 2nd ed., edited by Svarovsky, Butterworths and Co., 1981, p. 244.

²⁷Warring, R.H., Filters and Filtration Handbook, 1st ed., Gulf Publishing Company, 1981, pp. 17-18.

offers a potential advantage in that the fine particles that initially pass through the filter can be re-loaded on the unit after the cake has formed. The larger solids on the filter surface then act as a filtering aid for the finer particles. This is a common occurrence and is often utilized in commercial applications.

Solids that are removed by a depth filter, on the other hand, are subjected to a much longer and more complex path before they are removed entirely from the liquid flow. The depth filter is designed to allow (relatively) constant liquid flow by exposing the solids to a very large surface area that contains varying pore sizes. Because the surface area is so much larger and the effective smallest pore size is so much smaller, a greater pressure drop across the filter is usually required to obtain equivalent flow rates with a surface filter. The strength of the technology is in the depth filter's ability to remove much larger amounts of solids, particularly those that are difficult to remove by surface filtration, all while retaining the ability to collect very fine particles and maintaining reasonable flow rates.

FILTER SELECTION

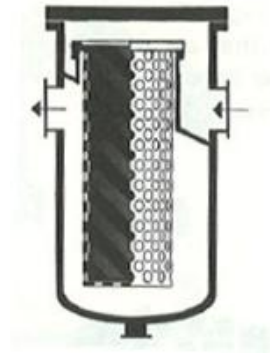
The selection of filter media is critical to the success of a filtration operation, such that the media warrants a brief overview. As always, the following represents a general overview of the options available to the engineer, and more complete lists can be found in the references utilized in writing this paper. Generally, the media types discussed here range from those that give the roughest form of separation to those that give the most precise. These different categories have significant overlap, in that one type of filter may include a subset of another.

STRAINERS

The simplest filter media is the strainer, which is normally used to offer protection of equipment or process streams from minute amounts of solid contaminants above a specified size. Strainers can take the form of a flat disc or a conical basket and may be constructed from simple perforated plates or supported wire mesh. Perforated plates typically provide coarse filtration for particles down to 150 μm , while wire-mesh strainers can accommodate solids down to about 40 μm , although with a requirement for additional support as the wire mesh can be very fine.²⁸ In larger pipelines, a basket-style strainer is usually preferred, both for its relative capacity to collect solids and for ease of removal and cleaning. A typical basket strainer is shown in Figure 11.

²⁸Ibid., pp. 67-71.

Figure 11. Diagram of a basket-style strainer.

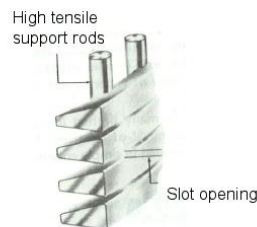


SCREENS

Screens are porous barriers that are designed to capture a particular particle size, while allowing smaller particle sizes to pass through. Strainers would be considered screens that are designed for incidental solid removal; most screens are utilized to accomplish a much more complete particle separation and oftentimes serve as support for other filter media. The type of screen and the size range of the feed to be treated will be the major factors determining the capacity of a screening operation.²⁹

Screens can be static (fixed) or moving (reciprocating) depending on the application. The simplest static screen for removing larger particles is the grizzly, which is a set of parallel bars held apart by spacers that create regular openings. A slightly more complex version of this same layout is the wedge wire deck, where the “bars” have a specific geometry that helps to prevent screen blinding (solids plugging all the apertures in the screen and preventing further flow through the screen). An illustration of a wedge wire screen is shown in Figure 12.

Figure 12. Illustration of a wedge wire screen³⁰



²⁹Osborne, D.G. “Screening”, *Solid-Liquid Separation*, 2nd ed., edited by Svarovsky, Butterworths and Co., 1981, pp. 215-217.

³⁰Warring, p. 73.

The geometry is crucial in this design, as any solids that manage to make it through the slot opening will then pass freely through the screen. Installing a wedge wire screen in the reverse fashion would almost certainly ensure that the screen will be blinded when separating solids with diameters close to that of the slot opening.

In general, screens are subjected to significant abrasion and must be constructed of sufficiently robust material to function with expected efficiency over time. This is especially true for reciprocating screens that are utilized in filters that introduce movement to aid in separations. Once the desired aperture size and surface area (percentage of open area) have been specified, it must then be determined whether the screen will be woven wire, round or wedged bars, “comb” style (round bars that are not fixed so their rotation will naturally aid in clearing the buildup of solids), or a simple perforated plate. Additionally, some screens are conical, and many screening applications utilize multiple screens with progressively smaller apertures to achieve a high throughput and efficiency.

CLOTH AND PRECOAT/FILTER MEDIA

Filters can utilize “cloth” in the form of woven wire, paper, adsorbent media, and synthetic fibers. The media are often inert solids such as diatomaceous earth that can be loaded onto a screen or other support to provide a porous surface on which solids accumulate; however, chemically active filter media can be used if required by the separation. Typically, the type of cloth or media utilized is that which allows the greatest flow through the bed without creating difficulties in cleaning the removed solids from the media.

MEMBRANES

Filter membranes represent a separate class of media, as they allow for the use of filters that are capable of separating extremely fine particles (removal of bacteria or even viruses) from liquid. Membranes vary in their configuration, but are commonly pleated cartridges, flat sheets, or hollow fibers.³¹ Microfiltration (pore size measured in microns) and ultrafiltration (pore size measured in Daltons) are made possible by the use of membranes, which are most commonly made of polysulphone, polyacrylamide, polyvinylidenedifluoride, polyethylene, and ceramics. As one would expect, the pore sizes are very small, so the pressure required to enable flow through the membranes during operation can be quite high.

The type of filter cloth or media required is usually determined in the development of processes that are to be scaled, or at least the nominal particle size of the solids to be separated is known. Often a filtration, whether it utilizes a cotton cloth on a belt filter, a woven wire screen, or a synthetic fiber membrane,

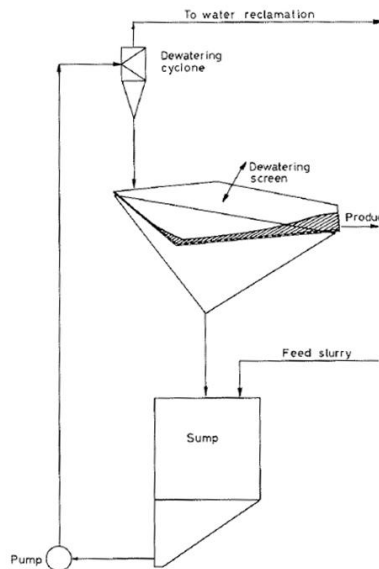
³¹Wakeman, R. and Tarleton, S., Solid/Liquid Separation: Scale-up of Industrial Equipment, Elsevier Advanced Technology, 2005, p. 140.

will perform better as a separation progresses, but experience with this serendipitous phenomenon is generally obtained through multiple runs of a separation at scale and cannot be estimated or counted on in the design phase.

TYPES OF FILTRATION

The simplest type of commercial filtration is a screening operation in which large particles are separated on a mesh that is large enough to catch the particles but provides essentially no resistance to the liquid flow. Typically, these have been used to dewater coarse materials, such as mineral ore and coal. The nature of the particles in these systems (whether the fines contain values or can be lost to the drain, as well as the overall particle size distribution) has a major influence on the screen material selection and design. The driving force for the separation is typically the flow of the liquid through the screen, though some screening operations utilize an additional pressure drop, such as a vacuum. A typical dewatering process is shown in Figure 13.

Figure 13. A dewatering process including a cyclone to pretreat the slurry³²



Screening operations are often subjected to abrasive conditions due to the material being separated. Dewatering applications can be quite disparate in their designs, and can utilize a solids concentration step prior to the screen, vibrating screens, deck inclination or curved screening surfaces, a spiraling vortex, vacuum to increase the pressure across the screen, and other advanced equipment, depending on the behavior of the solids and the desired product.

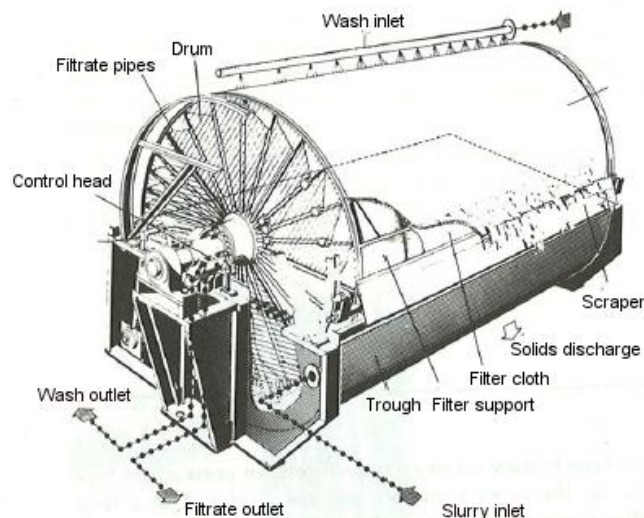
VACUUM FILTRATION

³²Warring, p. 233.

Solid liquid separations can be readily accomplished in many processes utilizing a vacuum filtration, however the major limitation is that the pressure driving the flow through the cake will be somewhat less than 1 atmosphere (14.7 psi). Additionally, the lower pressures involved tend to liberate volatile solvents in a slurry which can present significant challenges at scale. Despite these disadvantages, vacuum filtration is widespread and used in almost all industries that require bulk liquid/solid separations.

The rotary vacuum drum filter is a cloth-covered perforated drum suspended over a trough containing the slurry to be separated. The fundamental design of the rotary drum vacuum filter is relatively unchanged since it was patented by James and William Hart in 1872.³³ Anywhere from 25-75% of the filter area can be submerged in the slurry trough, but most commonly approximately 35% of the filter area is utilized. Slurry is pulled onto the surface of the drum that is submerged and the solids are retained on the cloth. They then rotate with the drum out of the vessel, often where they are washed and then removed from the cloth surface by means of a backflow of compressed air, a scraper, or both. A schematic of the rotary vacuum drum filter is shown in figure 14.

Figure 14. A schematic of a rotary vacuum drum filter³⁴



Because the slurry is subject to gravity, it must be mixed while the drum filter is utilized in order to ensure that the solids do not sand out in the trough instead of being removed on the filter. Additionally, only the area of the drum that is not submerged can effectively dewater the cake, so the mechanism used to feed the unit (amount of filter area submerged) is in competition for filter area with the washing and dewatering portions of the drum. Drum filters can be used in precoat applications, and can utilize cake compression to aid in removal of

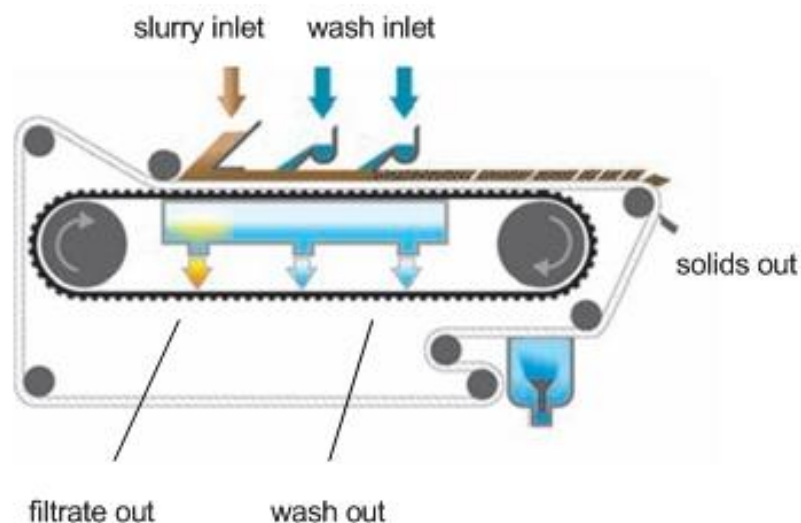
³³ Wakeman, Richard and Tarleton, Steve, Solid-Liquid Separation: Scale-up of Industrial Equipment, Elsevier Inc, 2005, p.244

³⁴ Warring, p.124.

mother liquor or wash. Top feed drum filters are also used, however the effective filter area of these units is limited by the geometry of the drum and by gravity, and thus not very common except in very specialized applications.

Another example of a commercial vacuum filtration unit is the horizontal vacuum belt filter (HVBF), which like the rotary drum filter allows the filter cloth to be washed and recycled during the operation. In the more complex configurations of this technology, the belt is advanced through the use of a continuously moving belt with a support tray that moves over horizontal vacuum chambers. Breaking the vacuum at the end of a cycle (when the tray must reset by sliding under the belt back to its original position), allows for constant feed of the slurry to the belt, limited only by the speed at which the cake dewateres.³⁵ A schematic of a HVBF is shown in figure 15.

Figure 15. A schematic of a Pannevis RT/RB horizontal vacuum belt filter



The horizontal vacuum belt filter generally has a very large footprint, and while it operates on similar principles with the drum filter, it does have the advantage that the belt speed can be adjusted to allow for sufficient dwell time to dewater the cake.³⁶ This capability is not possible on a drum filter, as the feed characteristics will change if the rotation of the drum is simply slowed. The belt filter can slow the filtration while keeping the feed constant, allowing for the exploitation of thicker beds if it suits the process. Additionally the filtrate and the wash can be collected separately as the unit can be run so the different streams report to separate collection vessels. Units like the HVBF have many moving parts, and while generally reliable, they do require regular maintenance.

³⁵ Wakeman, Richard and Tarleton, Steve, Solid-Liquid Separation: Scale-up of Industrial Equipment, Elsevier Inc, 2005, p.254

³⁶Ibid., p. 254.

PRESSURE FILTRATION

The pressure limitations of vacuum filtration can be overcome by using pressure filtration. The larger Δp provides a greater driving force, giving a greater throughput for a given filtration area. The solids cake that is collected is often compressible, such that the cake permeability changes during a pressure filtration, affecting the flow through the bed. To account for this, data that relate to the specific resistance of a cake (α) and the cake porosity (ε) to the pressure differential (Δp) and the effect of particle size are described by:

$$\alpha = (180/\rho_s x^2) \cdot (1 - \varepsilon/\varepsilon^3)^{37}$$

Cake resistance (R_c , as opposed to the resistance of the filter itself, R) is defined by the specific resistance (α), multiplied by the area of the filter (A), as:

$$R_c = \alpha A$$

From these, it is understood that the pressure filtration will be generally described by a simplified form of Darcy's Law:

$$Q = \Delta p / (\mu(R_c + R))^{38}$$

In general, the resistance of the cake, R_c , will increase as the cake compresses. This is usually a constant process, which is why most pressure filtration equipment is operated in batch mode, to allow for removal of the cake from the filter media and reduce the cake resistance so the operation can start again. It is inherently more expensive to operate than continuous equipment because of added labor requirements.

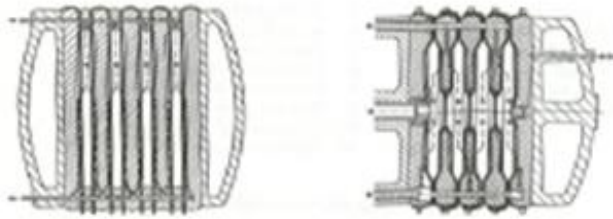
Filter presses, which are a series of vertical chambers produced by stacked plates of varying designs to create small regions between each plate, are commonly used to accomplish these separations at scale.³⁹ A simple diagram of two types of filter presses is shown in Figure 16.

³⁷Wakeman and Tarleton (Solid/Liquid Separation: Principles of Industrial Filtration), p. 110.

³⁸Ibid., p. 101.

³⁹Warring, p. 136.

Figure 16. Examples of a Frame filter press (left) and a Chamber filter press (right)

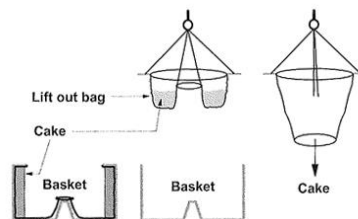


The slurry is pumped into the unit at elevated pressure, and the solids are retained as a cake on the filter cloth attached to the frames. Initially, the filter cloth acts as the filter, but as the cake builds up on the frames, the separated solids perform the bulk of the filtration. The supply pressure is gradually increased to compensate for the increasing resistance of the cake, until the flow of filtrate out of the unit has reached a point where the operation is effectively no longer functioning. At this point, the operation is stopped. In many instances, more filtrate is obtained by blowing air or inert gas through the filter cake and compressing the cake with built-in bladders. The filter cake can then be washed, the filter is disassembled to collect the filter cake, and the frames are cleaned of solids for reuse.

CENTRIFUGAL FILTRATION

These operations are performed in a manner similar to that of a sedimenting centrifuge, save for the fact that the forces at play in filtration, particularly compressible cakes, must be considered. The filtering centrifuge is designed to be operated in batch or semi-batch fashion, given the requirement that the solids must be periodically removed from the unit. Different technologies are available to accomplish this type of operation, the simplest type being the basket batch centrifuge, for which a filter bag is utilized to perform the filtration. However, these units require stopping the centrifuge in order to remove the bag to collect the filter cake. A schematic of the liftout bag discharge is shown in Figure 17.

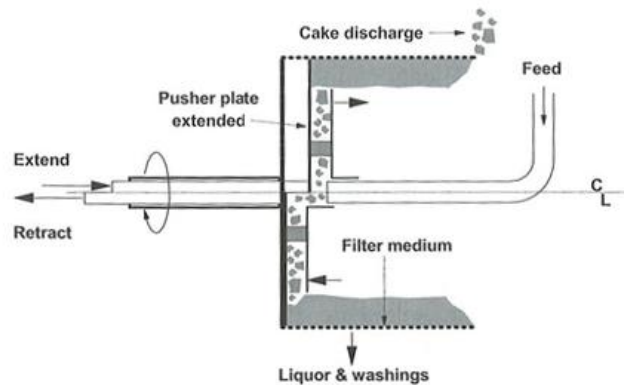
Figure 17. Liftout bag discharge of a filtering centrifuge⁴⁰



⁴⁰Wakeman and Tarleton (Solid/Liquid Separation: Scale-up of Industrial Equipment), p. 329.

There is a considerable operational cost advantage to clearing the filter cakes while maintaining the movement of the centrifuge body. Industry has recognized this need, which has yielded some very innovative designs, such as the peeler centrifuge (solids are scraped from the inside of the bowl once the cake is of sufficient thickness), the inverting bag centrifuge (the moving filter bag is pushed out of the moving basket and discharges the solids when inverted), and the pusher centrifuge (which pushes the solids out the top of the bowl periodically during operation). An example of this type of design is shown in Figure 18.

Figure 18. A filtering pusher centrifuge with solids discharge⁴¹



There are many other iterations of this same concept. It should be noted that these designs are superb for their intended use, but they normally come at a very high capital cost.

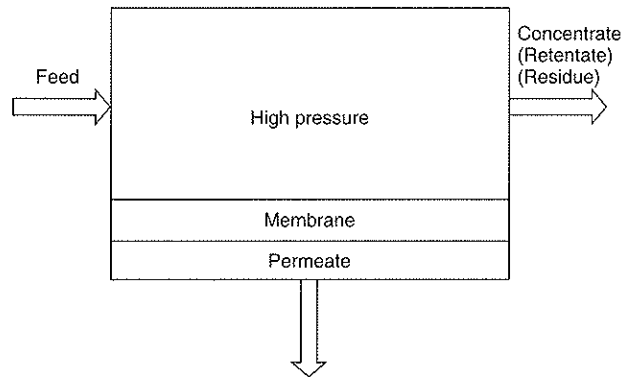
MICROFILTRATION AND ULTRAFILTRATION

When a solid–liquid separation requires removing extremely small particles, microfiltration or ultrafiltration is a good option. In general, the pore size used to describe these systems is typically related to the size of the material to be separated.⁴² The membranes are arranged in such a way as to maximize the filtrate (referred to as the permeate) rate for the smallest membrane surface area. This manner of operation allows for the removal of a portion of the filtrate while using the retained slurry both as feed for the membrane further downstream and also as a way of sweeping solids from the membrane in an active manner. A diagram of cross-flow filtration is shown in Figure 19.

⁴¹Ibid., p. 341.

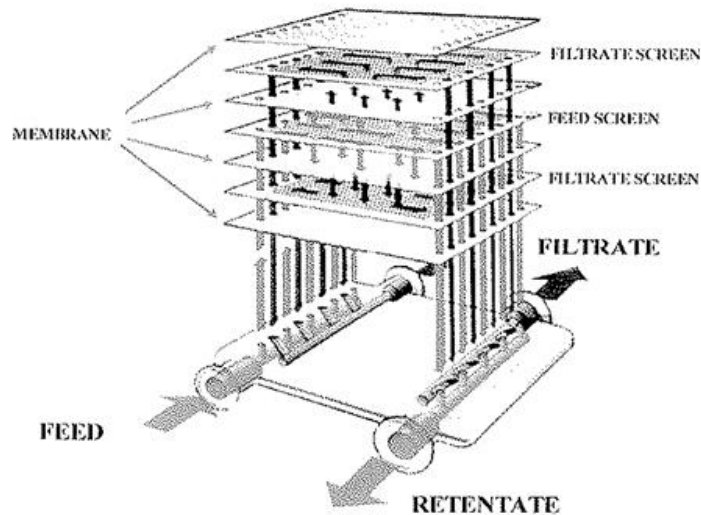
⁴²Ibid., p. 140.

Figure 19. Tangential flow (cross-flow) filtration⁴³



Membranes are arranged into varying geometries based on the needs of the process. A common arrangement is the cassette, which is shown in Figure 20.

Figure 20. A flat sheet membrane assembly used in a cassette⁴⁴

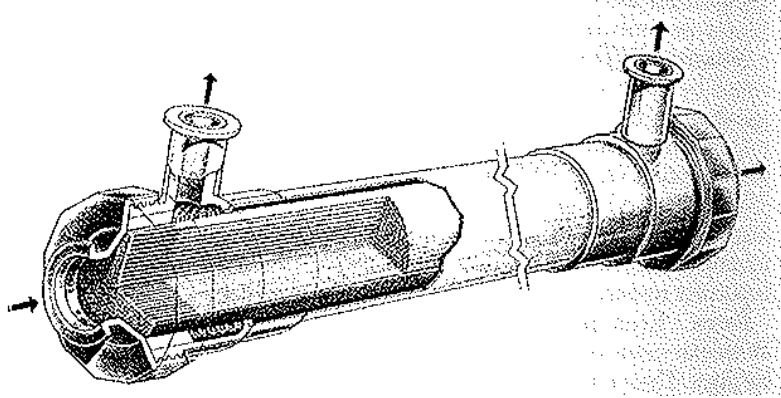


Another common practice is the use of hollow fiber membranes, which contain numerous hollow tubes. The tubular membranes are supported by a pressure vessel and are often run in series. An example of a simple hollow tube module is shown in Figure 20.

⁴³Perry, pp. 22-38.

⁴⁴Wakeman and Tarleton (Solid/Liquid Separation: Scale-up of Industrial Equipment), p. 158.

Figure 20. Cutaway view of a hollow tube membrane filter module⁴⁵



These types of membranes can also be constructed of ceramics, which are typically constructed as monoliths of tubular capillaries selected to serve a particular process separation need. Many of these configurations are plumbed so that a backflow of permeated liquid can be periodically used to push accumulated solids out of the membrane into the feed side to maintain filtration rates. By utilizing proper flows and an effective backflow, membrane filtrations can accommodate a large amount of slurry before a formal cleaning cycle is used to regain the flow through the membranes. It should be noted that although these systems are very effective at removing small amounts of small particles, a heavy solids load on the membranes will cause blinding, which may be irreversible.

CONCLUSIONS

All of the solid-liquid separation technologies in this paper are covered in much greater detail in the references. The theme that one finds with any description of commercial solid-liquid separation equipment is that every technology has its strengths and weaknesses, and often a solid-liquid separation must be evaluated at the pilot scale to verify any equipment selections made from laboratory scale experimentation. Also consistent in the literature is the suggestion that proper separation process design will allow the engineer to take advantage of the benefits of a technology while minimizing those factors that may detract from its perceived value.

The simplest approach to this, in the author's opinions, is to do everything possible to minimize the mass of slurry subject to the solids separation step. Recalling the diagram from Wakeman and Tarleton on p. 4, this requires a development effort in the pretreatment and solids concentration steps in the separation process. Significant benefits to process costs can usually be realized by the introduction of low cost/high throughput technologies prior to the use of effective yet expensive solid-liquid separation unit operations.

⁴⁵Perry, pp. 22-40.

FURTHER READING

Cheremisinoff, Nicholas, Pocket Handbook for Solid-Liquid Separations: Calculations and Guidelines for Process Engineering, Gulf Publishing Company, 1984.

Evans, Geoffrey M., Atkinson, Bruce W. and Jameson, Graeme J., "The Jameson Cell", Flotation Science and Engineering, Marcel Dekker, Inc., 1995.

Gochin, R.J., "Flotation", Solid-Liquid Separation, 2nd ed., edited by Svarovsky, Butterworths and Co., 1981.

Matis, K.A. and Zouboulis, A.I., Flotation Science and Engineering, Marcel Dekker, Inc., 1995.

Perry, Robert H., and Green, Don W., editors, Perry's Chemical Engineer's Handbook, 7th ed., Sections 18, 19 and 22.

Peters, Max S., Timmerhaus, Klaus D. and West, Ronald E., Plant Design and Economics for Chemical Engineers, 5th ed. McGraw-Hill, 2003.

Pierson, H.G.W., "The Selection of Solid-Liquid Separation Equipment", Solid-Liquid Separation, L. Svarovski, 2nd ed., 1981.

Seidel, D. C. "Laboratory Procedures for Hydrometallurgical-Processing and Waste-Management Programs", United States Department of the Interior Bureau of Mines Information Circular 9431, 1995.

Svarovsky, L., Hydrocyclones, Hold, Rinehart and Winston Ltd., 1984.

Wakeman, R. and Tarleton, S., Solid/Liquid Separation: Principles of Industrial Filtration, Elsevier Advanced Technology, 2005.

Wakeman, R. and Tarleton, S., Solid/Liquid Separation: Scale-up of Industrial Equipment, Elsevier Advanced Technology, 2005.

Warring, R.H., Filters and Filtration Handbook, 1st ed. Gulf Publishing Company, 1981.