CHAPTER 10

Equipment Selection, Specification and Design

10.1. INTRODUCTION

The first chapters of this book covered process design: the synthesis of the complete process as an assembly of units; each carrying out a specific process operation. In this and the following chapters, the selection, specification and design of the equipment required to carry out the function of these process units (unit operations) is considered in more detail. The equipment used in the chemical processes industries can be divided into two classes: proprietary and non-proprietary. Proprietary equipment, such as pumps, compressors, filters, centrifuges and dryers, is designed and manufactured by specialist firms. Non-proprietary equipment is designed as special, one-off, items for particular processes; for example, reactors, distillation columns and heat exchangers.

Unless employed by one of the specialist equipment manufacturers, the chemical engineer is not normally involved in the detailed design of proprietary equipment. His job will be to select and specify the equipment needed for a particular duty; consulting with the vendors to ensure that the equipment supplied is suitable. He may be involved with the vendor's designers in modifying standard equipment for particular applications; for example, a standard tunnel dryer designed to handle particulate solids may be adapted to dry synthetic fibres.

As was pointed out in Chapter 1, the use of standard equipment, whenever possible, will reduce costs.

Reactors, columns and other vessels are usually designed as special items for a given project. In particular, reactor designs are usually unique, except where more or less standard equipment is used; such as an agitated, jacketed, vessel.

Distillation columns, vessels and tubular heat exchangers, though non-proprietary items, will be designed to conform to recognised standards and codes; this reduces the amount of design work involved.

The chemical engineer's part in the design of "non-proprietary" equipment is usually limited to selecting and "sizing" the equipment. For example, in the design of a distillation column his work will typically be to determine the number of plates; the type and design of plate; diameter of the column; and the position of the inlet, outlet and instrument nozzles. This information would then be transmitted, in the form of sketches and specification sheets, to the specialist mechanical design group, or the fabricator's design team, for detailed design.

In this chapter the emphasis is put on equipment selection, rather than equipment design; as most of the equipment described is proprietary equipment. Design methods

are given for some miscellaneous non-proprietary items. A brief discussion of reactor design is included. The design of two important classes of equipment, columns and heat exchangers, is covered separately in Chapters 11 and 12. A great variety of equipment is used in the process industries, and it is only possible to give very brief descriptions of the main types in this volume. Further details are given in Volume 2; and descriptions and illustrations of most of the equipment used can be found in various handbooks: Perry *et al*. (1997), Schweitzer (1988) and Walas (1990). Equipment manufacturers' advertisements in the technical press should also be studied. It is worthwhile building up a personal file of vendors' catalogues to supplement those that may be held in a firm's library. In the United Kingdom, a commercial organisation, Technical Indexes Ltd., publishes the Process Engineering Index; which contains on microfilm information from over 3000 manufacturers and suppliers of process equipment.

The scientific principles and theory that underlie the design of and operation of processing equipment is covered in Volume 2.

10.2. SEPARATION PROCESSES

As was discussed in Chapter 1, chemical processes consist essentially of reaction stages followed by separation stages in which the products are separated and purified.

The main techniques used to separate phases, and the components within phases, are listed in Table 10.1 and discussed in Sections 10.3 to 10.9.

10.3. SOLID-SOLID SEPARATIONS

Processes and equipment are required to separate valuable solids from unwanted material, and for size grading (classifying) solid raw materials and products.

The equipment used for solid-solid separation processes was developed primarily for the minerals processing and metallurgical industries for the benefication (up-grading) of ores. The techniques used depend on differences in physical, rather than chemical, properties, though chemical additives may be used to enhance separation. The principal techniques used are shown in Figure 10.1; which can be used to select the type of processes likely to be suitable for a particular material and size range.

Sorting material by appearance, by hand, is now rarely used due to the high cost of labour.

10.3.1. Screening (sieving)

The methods used for laboratory particle size analysis are discussed in detail in Volume 2, Chapter 1.

Screens separate particles on the basis of size. Their main application is in grading raw materials and products into size ranges, but they are also used for the removal of trash (over-and under-sized contaminants) and for dewatering. Industrial screening equipment is used over a wide range of particle sizes, from fine powders to large rocks. For small particles woven cloth or wire screens are used, and for larger sizes, perforated metal plates or grids.

Table 10.1. Separation processes

Numbers refer to the sections in this chapter. Processes in brackets are used for separating dissolved components (solutions). The terms major and minor component only apply where different phases are to be separated; i.e. not to those on the diagonal

		MINOR COMPONENT					
		SOLID		LIQUID		GAS/VAPOUR	
MAJOR COMPONENT	SOLID	Sorting	10.3	Pressing	10.4.5	Crushing	10.10
		Screening	10.3.1	Drying	10.4.6.	Heating	—
		Hydrocyclones	10.3.2				
		Classifiers	10.3.3				
		Jigs	10.3.4				
		Tables	10.3.5				
		Centrifuges	10.3.6				
		Dense media	10.3.7				
		Flotation	10.3.8				
		Magnetic	10.3.9				
		Electrostatic	10.3.10				
	LIQUID	Thickeners	10.4.1	Decanters	10.6.1	(Stripping)	Volume 2
		Clarifiers	10.4.1	Coalescers	10.6.3		
		Hydrocyclones	10.4.4	(Solvent extraction)	10.7.1		
		Filtration	10.4.2				
		Centrifuges	10.4.3	(Distillation)	Chapter 11		
		(Crystallisers)	10.5.2	(Adsorption)	Volume 2		
		(Evaporators)	10.5.1	(Ion exchange)	Volume 2		
	GAS/VAPOUR	Gravity settlers	10.8.1	Separating vessels	10.9	(Adsorption)	Volume 2
		Impingement settlers	10.8.2	Demisting pads	10.9	(Absorption)	Volume 2
		Cyclones	10.8.3	Cyclones	10.8.3		
		Filters	10.8.4	Wet scrubbers	10.8.5		
		Wet scrubbers	10.8.5	Electrostatic precipitators	10.8.6		
		Electrostatic precipitators	10.8.6				

Screen sizes are defined in two ways: by a mesh size number for small sizes and by the actual size of opening in the screen for the larger sizes. There are several different standards in use for mesh size, and it is important to quote the particular standard used when specifying particle size ranges by mesh size. In the UK the appropriate British Standards should be used; BS 410 and BS 1796. A comparison of the various international standard sieve mesh sizes is given in Volume 2, Chapter 1.

The simplest industrial screening equipment are stationary screens, over which the material to be screened flows. Typical of this type are "Grizzly" screens, which consist of rows of equally spaced parallel bars, and which are used to "scalp" off over-sized rocks in the feed to crushers.

Dynamic screening equipment can be categorised according to the type of motion used to shake-up and transport the material on the screen. The principal types used in the chemical process industries are described briefly below.

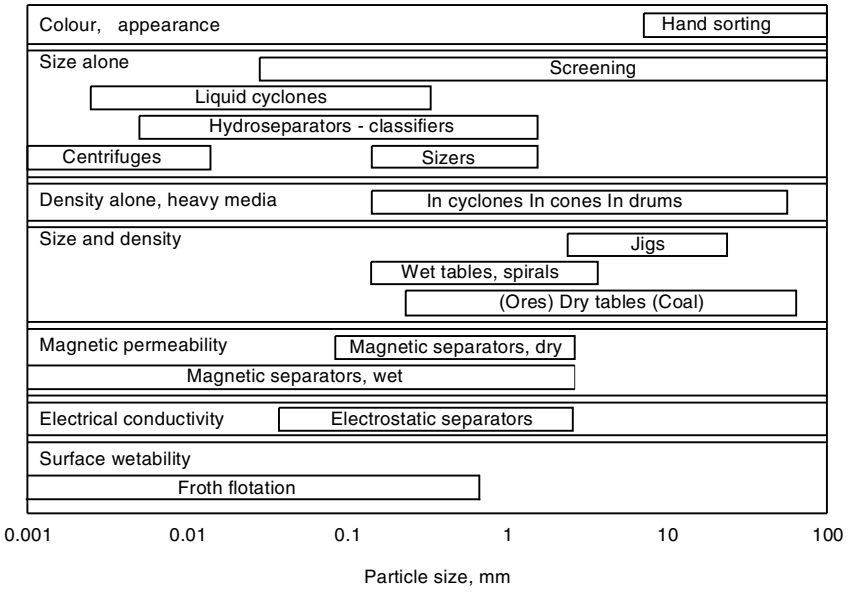


Figure 10.1. A particle size selection guide to solid-solid separation techniques and equipment (after Roberts *et al.* 1971)

Vibrating screens: horizontal and inclined screening surfaces vibrated at high frequencies (1000 to 7000 Hz). High capacity units, with good separating efficiency, which are used for a wide range of particle sizes.

Oscillating screens: operated at lower frequencies than vibrating screens (100–400 Hz) with a longer, more linear, stroke.

Reciprocating screens: operated with a shaking motion, a long stroke at low frequency (20–200 Hz). Used for conveying with size separation.

Shifting screens: operated with a circular motion in the plane of the screening surface. The actual motion may be circular, gyratory, or circularly vibrated. Used for the wet and dry screening of fine powders.

Revolving screens: inclined, cylindrical screens, rotated at low speeds (10–20 rpm). Used for the wet screening of relatively coarse material, but have now been largely replaced by vibrating screens.

Figure 10.2, which is based on a similar chart given by Matthews (1971), can be used to select the type of screening equipment likely to be suitable for a particular size range. Equipment selection will normally be based on laboratory and pilot scale screening tests, conducted with the co-operation of the equipment vendors. The main factors to be considered, and the information that would be required by the firms supplying proprietary screening equipment, are listed below:

1. Rate, throughput required.
2. Size range (test screen analysis).
3. Characteristics of the material: free-flowing or sticky, bulk density, abrasiveness.
4. Hazards: flammability, toxicity, dust explosion.
5. Wet or dry screening to be used.

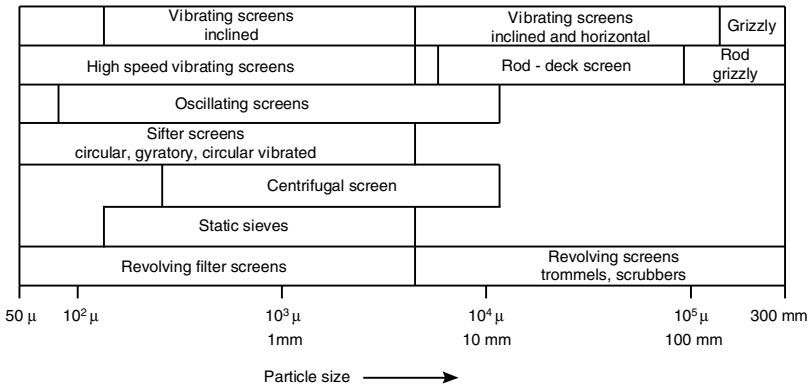


Figure 10.2. Screen selection by particle size range

10.3.2. Liquid-solid cyclones

Cyclones can be used for the classification of solids, as well as for liquid-solid, and liquid-liquid separations. The design and application of liquid cyclones (hydrocyclones) is discussed in Section 10.4.4. A typical unit is shown in Figure 10.3.

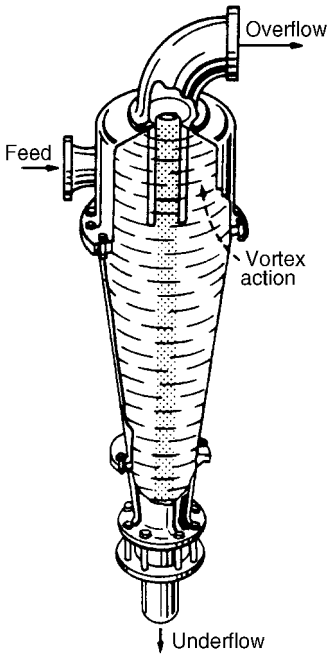


Figure 10.3. Liquid-solid cyclone (hydrocyclone)

Liquid cyclones can be used for the classification of solid particles over a size range from 5 to 100 μm. Commercial units are available in a wide range of materials of

construction and sizes; from as small as 10 mm to up to 30 m diameter. The separating efficiency of liquid cyclones depends on the particle size and density, and the density and viscosity of the liquid medium.

10.3.3. Hydroseparators and sizers (classifiers)

Classifiers that depend on the difference in the settling rates of different size particles in water are frequently used for separating fine particles, in the 50 to 300 μm range. Various designs are used. The principal ones used in the chemical process industries are described below.

Thickeners: thickeners are primarily used for liquid-solid separation (see Section 10.4). When used for classification, the feed rate is such that the overflow rate is greater than the settling rate of the slurry, and the finer particles remain in the overflow stream.

Rake classifiers: are inclined, shallow, rectangular troughs, fitted with mechanical rakes at the bottom to rake the deposited solids to the top of the incline (Figure 10.4). Several rake classifiers can be used in series to separate the feed into different size ranges.

Bowl classifiers: are shallow bowls with concave bottoms, fitted with rakes. Their operation is similar to that of thickeners.

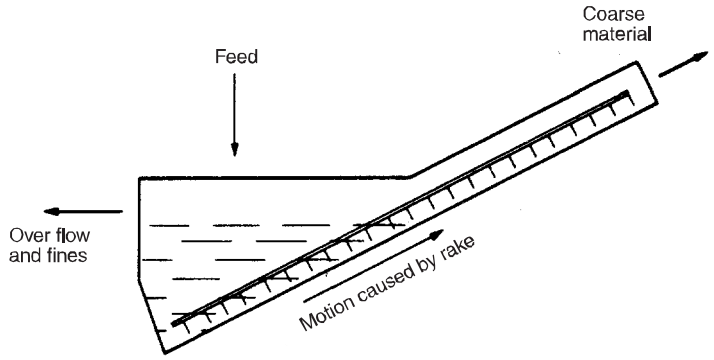


Figure 10.4. Rake classifier

10.3.4. Hydraulic jigs

Jigs separate solids by difference in density and size. The material is immersed in water, supported on a screen (Figure 10.5). Pulses of water are forced through the bed of material, either by moving the screen or by pulsating the water level. The flow of water fluidises the bed and causes the solids to stratify with the lighter material at the top and the heavier at the bottom.

10.3.5. Tables

Tables are used wet and dry. The separating action of a wet table resembles that of the traditional miner's pan. Riffled tables (Figure 10.6) are basically rectangular decks, inclined at a shallow angle to the horizontal (2 to 5°), with shallow slats (riffles) fitted to

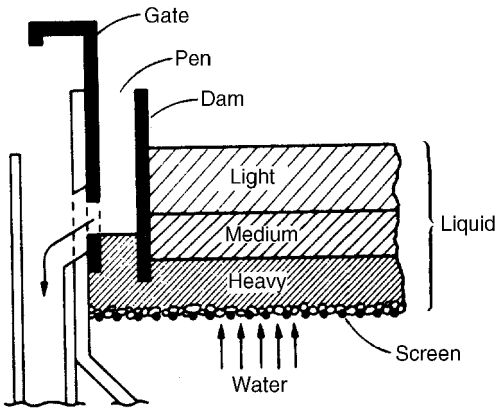


Figure 10.5. A hydraulic jig

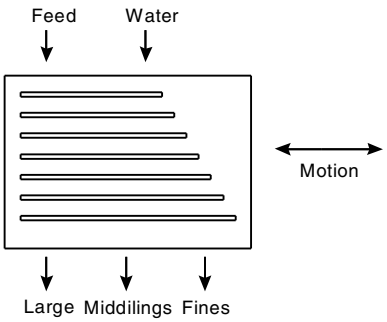


Figure 10.6. Wilfley riffled table

the surface. The table is mechanically shaken, with a slow stroke in the forward direction and a faster backward stroke. The particles are separated into different size ranges under the combined action of the vibration, water flow, and the resistance to flow over the riffles.

10.3.6. Classifying centrifuges

Centrifuges are used for the classification of particles in size ranges below 10 μm. Two types are used: solid bowl centrifuges, usually with a cylindrical, conical bowl, rotated about a horizontal axis; and "nozzle" bowl machines, fitted with discs.

These types are described in Section 10.4.3.

10.3.7. Dense-medium separators (sink and float processes)

Solids of different densities can be separated by immersing them in a fluid of intermediate density. The heavier solids sink to the bottom and the lighter float to the surface. Water suspensions of fine particles are often used as the dense liquid (heavy-medium). The technique is used extensively for the benefication (concentration) of mineral ores.

10.3.8. Flotation separators (froth-flotation)

Froth-flotation processes are used extensively for the separation of finely divided solids. Separation depends on differences in the surface properties of the materials. The particles are suspended in an aerated liquid (usually water), and air bubbles adhere preferentially to the particles of one component and bring them to the surface. Frothing agents are used so that the separated material is held on the surface as a froth and can be removed.

Froth-flotation is an extensively used separation technique, having a wide range of applications in the minerals processing industries and other industries. It can be used for particles in the size range from 50 to 400 μm.

10.3.9. Magnetic separators

Magnetic separators can be used for materials that are affected by magnetic fields; the principle is illustrated in Figure 10.7. Rotating-drum magnetic separators are used for a wide range of materials in the minerals processing industries. They can be designed to handle relatively high throughputs, up to 3000 kg/h per metre length of drum.

Simple magnetic separators are often used for the removal of iron from the feed to a crusher.

The various types of magnetic separators used and their applications are described by Bronkala (1988).

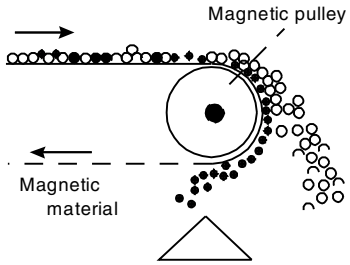


Figure 10.7. Magnetic separator

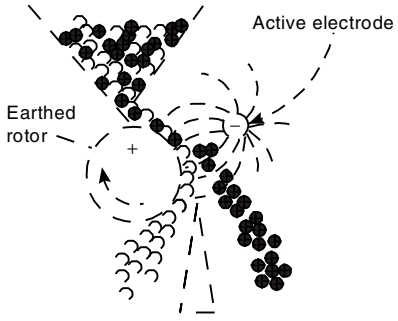


Figure 10.8. Electrostatic separator

10.3.10. Electrostatic separators

Electrostatic separation depends on differences in the electrical properties (conductivity) of the materials to be treated. In a typical process the material particles pass through a high-voltage electric field as it is fed on to a revolving drum, which is at earth potential (Figure 10.8). Those particles that acquire a charge adhere to the drum surface and are carried further around the drum before being discharged.

10.4. LIQUID-SOLID (SOLID-LIQUID) SEPARATORS

The need to separate solid and liquid phases is probably the most common phase separation requirement in the process industries, and a variety of techniques is used (Figure 10.9). Separation is effected by either the difference in density between the liquid and solids, using either gravity or centrifugal force, or, for filtration, depends on the particle size and shape. The most suitable technique to use will depend on the solids concentration and feed rate, as well as the size and nature of the solid particles. The range of application of various techniques and equipment, as a function of slurry concentration and particle size, is shown in Figure 10.10.

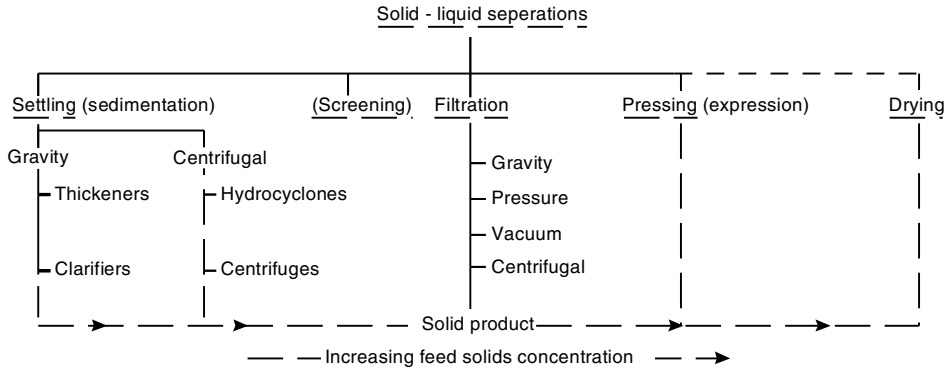


Figure 10.9. Solid-liquid separation techniques

The choice of equipment will also depend on whether the prime objective is to obtain a clear liquid or a solid product, and on the degree of dryness of the solid required.

The design, construction and application of thickeners, centrifuges and filters is a specialised subject, and firms who have expertise in these fields should be consulted when selecting and specifying equipment for new applications. Several specialist texts on the subject are available: Svarovsky (2001), Ward (2000) and Wakeman and Tarleton (1998). The theory of sedimentation processes is covered in Volume 2, Chapter 5 and filtration in Chapter 7.

10.4.1. Thickeners and clarifiers

Thickening and clarification are sedimentation processes, and the equipment used for the two techniques are similar. The primary purpose of thickening is to increase the concentration of a relatively large quantity of suspended solids; whereas that of clarifying,

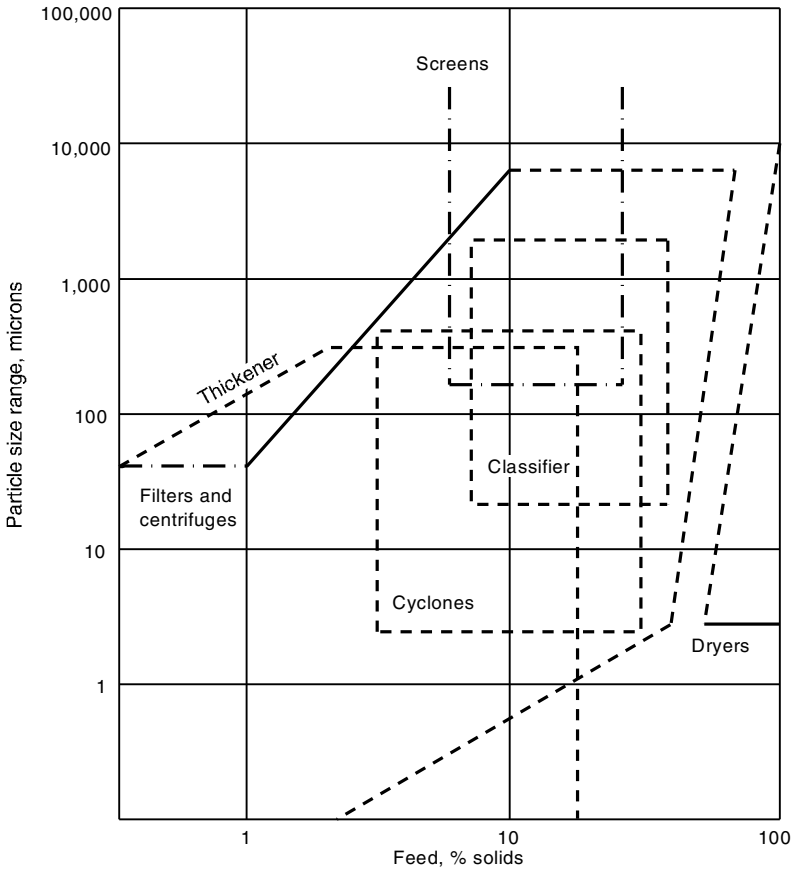


Figure 10.10. Solid-liquid separation techniques (after Dahlstrom and Cornell, 1971)

as the name implies, is to remove a small quantity of fine solids to produce a clear liquid effluent. Thickening and clarification are relatively cheap processes when used for the treatment of large volumes of liquid.

A thickener, or clarifier, consists essentially of a large circular tank with a rotating rake at the base. Rectangular tanks are also used, but the circular design is preferred. They can be classified according to the way the rake is supported and driven. The three basic designs are shown in Figure 10.11 (see p. 410). Various designs of rake are used, depending on the nature of the solids.

The design and construction of thickeners and clarifiers is described by Dahlstrom and Cornell (1971).

Flocculating agents are often added to promote the separating performance of thickeners.

10.4.2. Filtration

In filtration processes the solids are separated from the liquid by passing (filtering) the slurry through some form of porous filter medium. Filtration is a widely used separation

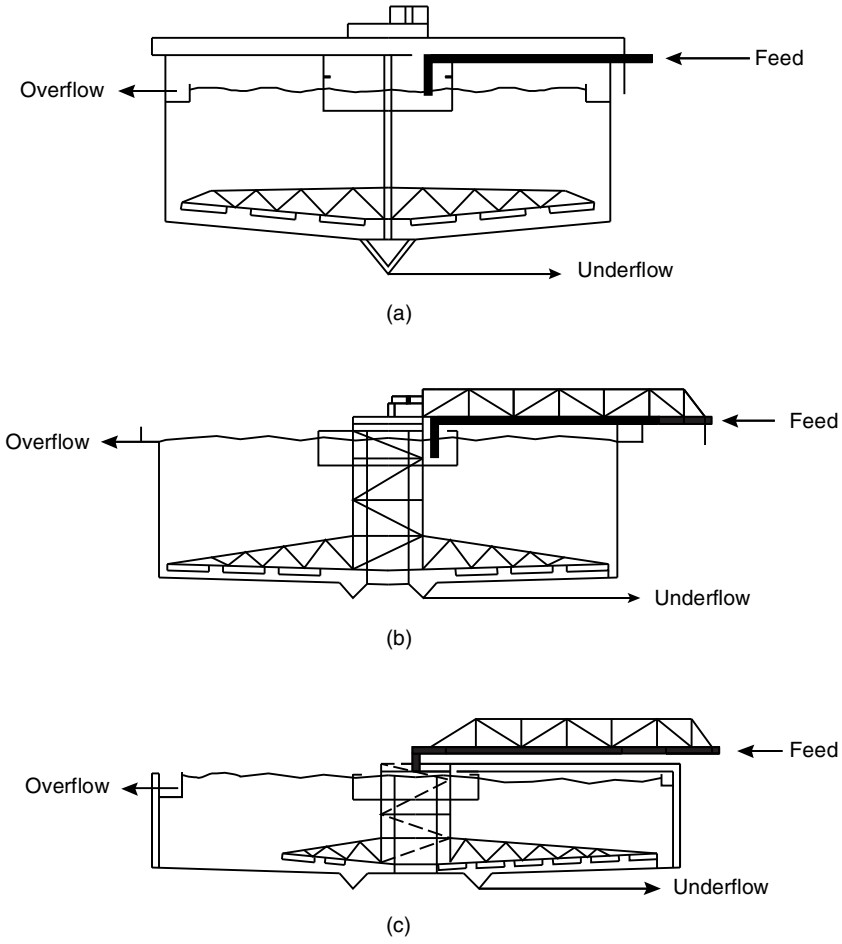


Figure 10.11. Types of thickener and clarifier (*a*) Bridge supported (up to <40 m dia.) (*b*) Centre column supported (<30 m dia.) (*c*) Traction driven (<60 m dia.)

process in the chemical and other process industries. Many types of equipment and filter media are used; designed to meet the needs of particular applications. Descriptions of the filtration equipment used in the process industries and their fields of application can be found in various handbooks: Perry *et al.* (1997), Dickenson (1997), Schweitzer (1997), and in several specialist texts on the subject: Cheremisnoff (1998), Orr (1977). A short discussion of filtration theory and descriptions of the principal types of equipment is given in Volume 2, Chapter 7.

The most commonly used filter medium is woven cloth, but a great variety of other media is also used. The main types are listed in Table 10.2. A comprehensive discussion of the factors to be considered when selecting filter media is given by Purchas (1971) and Mais (1971); see also Purchas and Sutherland (2001). Filter aids are often used to increase the rate of filtration of difficult slurries. They are either applied as a precoat

to the filter cloth or added to the slurry, and deposited with the solids, assisting in the formation of a porous cake.

Table 10.2. Filter media

Type	Examples	Minimum size particle trapped (μm)
1. Solid fabrications	Scalloped washers	
	Wire-wound tubes	5
2. Rigid porous media	Ceramics, stoneware	1
	Sintered metal	3
3. Metal	Perforated sheets	100
	Woven wire	5
4. Porous plastics	Pads, sheets	3
	Membranes	0.005
5. Woven fabrics	Natural and synthetic fibre cloths	10
6. Non-woven sheets	Felts, lap	10
	Paper, cellulose	5
7. Cartridges	Yarn-wound spools, graded fibres	2
8. Loose solids	Fibres, asbestos, cellulose	sub-micron

Industrial filters use vacuum, pressure, or centrifugal force to drive the liquid (filtrate) through the deposited cake of solids. Filtration is essentially a discontinuous process. With batch filters, such as plate and frame presses, the equipment has to be shut down to discharge the cake; and even with those filters designed for continuous operation, such as rotating-drum filters, periodic stoppages are necessary to change the filter cloths. Batch filters can be coupled to continuous plant by using several units in parallel, or by providing buffer storage capacity for the feed and product.

The principal factors to be considered when selecting filtration equipment are:

1. The nature of the slurry and the cake formed.
2. The solids concentration in the feed.
3. The throughput required.
4. The nature and physical properties of the liquid: viscosity, flammability, toxicity, corrosiveness.
5. Whether cake washing is required.
6. The cake dryness required.
7. Whether contamination of the solid by a filter aid is acceptable.
8. Whether the valuable product is the solid or the liquid, or both.

The overriding factor will be the filtration characteristics of the slurry; whether it is fast filtering (low specific cake resistance) or slow filtering (high specific cake resistance). The filtration characteristics can be determined by laboratory or pilot plant tests. A guide to filter selection by the slurry characteristics is given in Table 10.3; which is based on a similar selection chart given by Porter *et al.* (1971).

Table 10.3. Guide to filter selection

Slurry characteristics	Fast filtering	Medium filtering	Slow filtering	Dilute	Very dilute
Cake formation rate	cm/s	mm/s	0.02–0.12 mm/s	0.02 mm/s	No cake
Normal concentration	>20%	10–20%	1–10%	<5%	<0.1%
Settling rate	Very fast	Fast	Slow	Slow	—
Leaf test rate, kg/h m^2	>2500	250–2500	25–250	<25	—
Filtrate rate, m^3/h m^2	>10	5–10	0.02–0.05	0.02–5	0.02–5
Filter application					
Continuous vacuum filters					
Multicompartment drum	▬	▬	▬		
Single compartment drum	▬				
Top feed drum	▬				
Scroll discharge drum	▬	▬			
Tilting pan	▬	▬			
Belt	▬	▬			
Disc		▬	▬		
Batch vacuum leaf		▬	▬	▬	▬
Batch nutsche	▬	▬	▬	▬	▬
Batch pressure filters					
Plate and frame		▬	▬	▬	▬
Vertical leaf		▬	▬	▬	▬
Horizontal plate	▬	▬	▬	▬	▬
Cartridge edge					▬

The principal types of industrial scale filter used are described briefly below.

Nutsche (gravity and vacuum operation)

This is the simplest type of batch filter. It consists of a tank with a perforated base, which supports the filter medium.

Plate and frame press (pressure operation) (Figure 10.12)

The oldest and most commonly used batch filter. Versatile equipment, made in a variety of materials, and capable of handling viscous liquids and cakes with a high specific resistance.

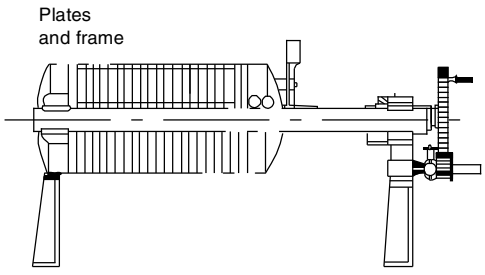


Figure 10.12. Plate and frame filter press

Leaf filters (pressure and vacuum operation)

Various types of leaf filter are used, with the leaves arranged in horizontal or vertical rows. The leaves consist of metal frames over which filter cloths are draped. The cake is

removed either mechanically or by sluicing it off with jets of water. Leaf filters are used for similar applications as plate and frame presses, but generally have lower operating costs.

Rotary drum filters (usually vacuum operation) (Figure 10.13)

A drum filter consists essentially of a large hollow drum round which the filter medium is fitted. The drum is partially submerged in a trough of slurry, and the filtrate sucked through the filter medium by vacuum inside the drum. Wash water can be sprayed on to the drum surface and multicompartment drums are used so that the wash water can be kept separate from the filtrate. A variety of methods is used to remove the cake from the drum: knives, strings, air jets and wires. Rotating drum filters are essentially continuous in operation. They can handle large throughputs, and are widely used for free filtering slurries.

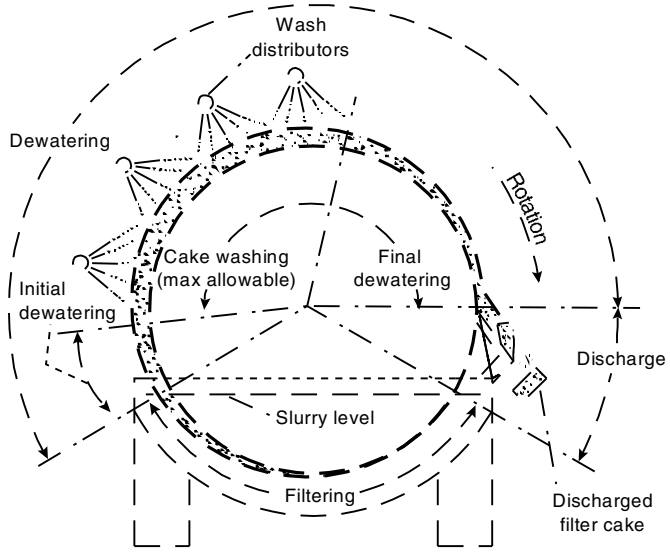


Figure 10.13. Drum filter

Disc filters (pressure and vacuum operation)

Disc filters are similar in principle to rotary filters, but consist of several thin discs mounted on a shaft, in place of the drum. This gives a larger effective filtering area on a given floor area, and vacuum disc filters are used in preference to drum filters where space is restricted. At sizes above approximately 25 m^2 filtration area, disc filters are cheaper; but their applications are more restricted, as they are not as suitable for the application of wash water, or precoating.

Belt filters (vacuum operation) (Figure 10.14)

A belt filter consists of an endless reinforced rubber belt, with drainage hole along its centre, which supports the filter medium. The belt passes over a stationary suction box, into which the filtrate is sucked. Slurry and wash water are sprayed on to the top of the belt.

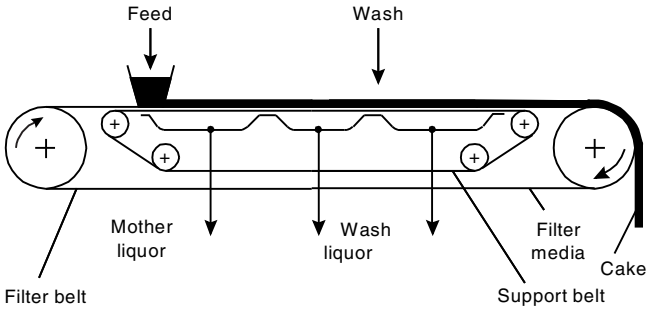


Figure 10.14. Belt filter

Horizontal pan filters (vacuum operation) (Figure 10.15)

This type is similar in operation to a vacuum Nutsche filter. It consists of shallow pans with perforated bases, which support the filter medium. By arranging a series of pans around the circumference of a rotating wheel, the operation of filtering, washing, drying and discharging can be made automatic.

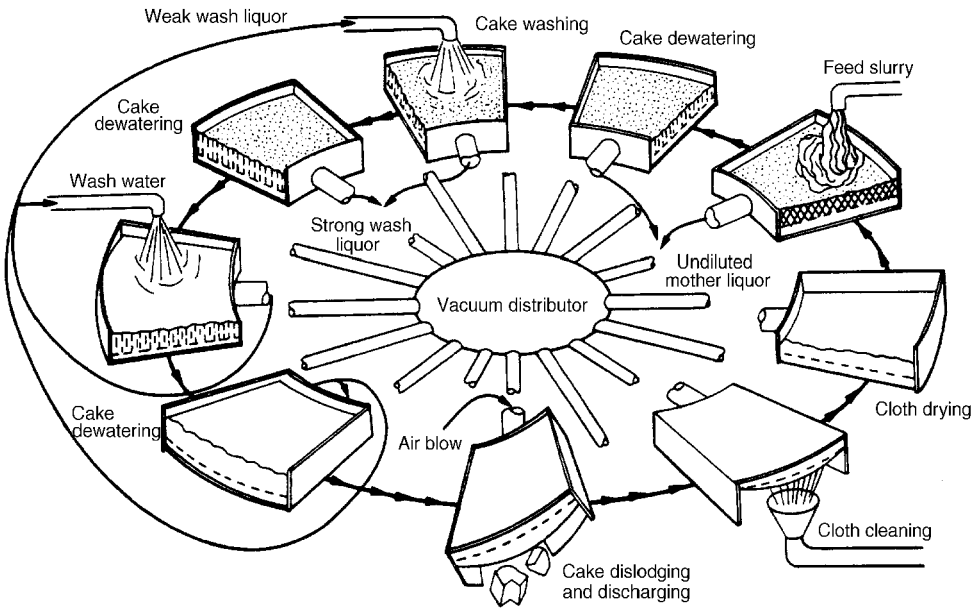


Figure 10.15. Pan filters

Centrifugal filters

Centrifugal filters use centrifugal force to drive the filtrate through the filter cake. The equipment used is described in the next section.

10.4.3. Centrifuges

Centrifuges are classified according to the mechanism used for solids separation:

(a) Sedimentation centrifuges: in which the separation is dependent on a difference in density between the solid and liquid phases (solid heavier).
(b) Filtration centrifuges: which separate the phases by filtration. The walls of the centrifuge basket are porous, and the liquid filters through the deposited cake of solids and is removed.

The choice between a sedimentation or filtration centrifuge for a particular application will depend on the nature of the feed and the product requirements.

The main factors to be considered are summarised in Table 10.4. As a general rule, sedimentation centrifuges are used when it is required to produce a clarified liquid, and filtration centrifuges to produce a pure, dry, solid.

Table 10.4. Selection of sedimentation or filter centrifuge

Factor	Sedimentation	Filtration
Solids size, fine		x
Solids size, >150 μm	x	
Compressible cakes	x	
Open cakes		x
Dry cake required		x
High filtrate clarity	x	
Crystal breakage problems		x
Pressure operation		
High-temperature operation	will depend on the type of centrifuge used	

A variety of centrifugal filter and sedimenter designs is used. The main types are listed in Table 10.5. They can be classified by a number of design and operating features, such as:

1. Mode of operation — batch or continuous.
2. Orientation of the bowl/basket — horizontal or vertical.
3. Position of the suspension and drive — overhung or underhung.
4. Type of bowl — solid, perforated basket, disc bowl.
5. Method of solids cake removal.
6. Method of liquid removal.

Descriptions of the various types of centrifuges and their fields of application can be found in various handbooks, in a book by Leung (1998) and articles by Ambler (1971) and Linley (1984).

The fields of application of each type, classified by the size range of the solid particles separated, are given in Figure 10.16. A similar selection chart is given by Schroeder (1998).

Sedimentation centrifuges

There are four main types of sedimentation centrifuge:

Table 10.5. Centrifuge types (after Sutherland, 1970)

Sedimentation	Filtration-fixed bed
Laboratory	Vertical basket
Bottle	Manual discharge
Ultra	Bag discharge
	Knife discharge
Tubular bowl	Horizontal basket
	Inclined basket
Disc	
Batch bowl	
Nozzle discharge	
Valve discharge	Filtration-moving bed
Opening bowl	
Imperforate basket	Conical bowl
Manual discharge	Wide angle
Skimmer discharge	Vibrating
	Torsional
	Tumbling
Scroll discharge	Scroll discharge
Horizontal	
Cantilevered	Cylindrical bowl
Vertical	Scroll discharge
Screen bowl	Pusher

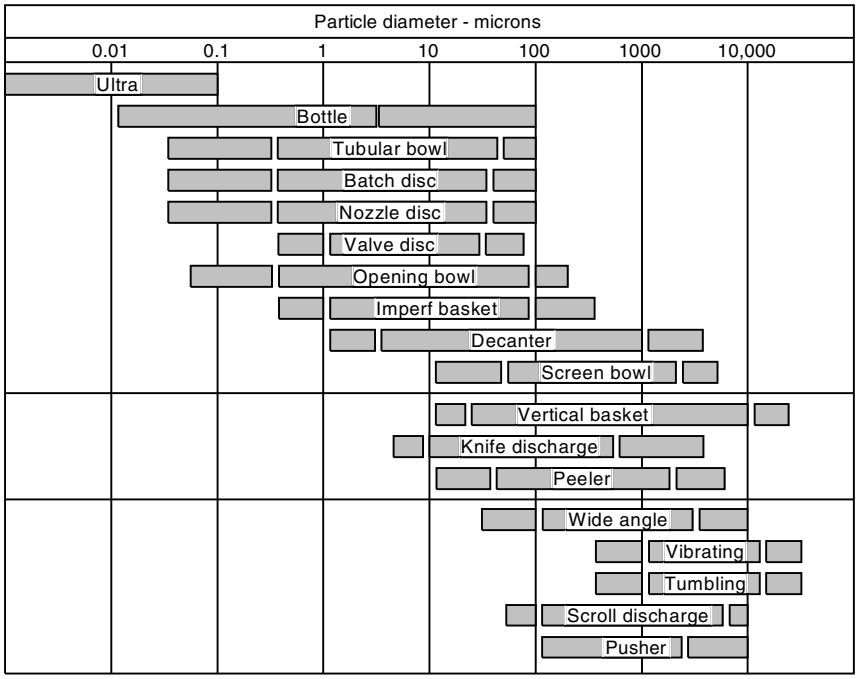


Figure 10.16. Classification of centrifuges by particle size (after Sutherland, 1970)

1. Tubular bowl (Figure 10.17)

High-speed, vertical axis, tubular bowl centrifuges are used for the separation of immiscible liquids, such as water and oil, and for the separation of fine solids. The bowl is driven at speeds of around 15,000 rpm (250 Hz) and the centrifugal force generated exceeds 130,000 N.

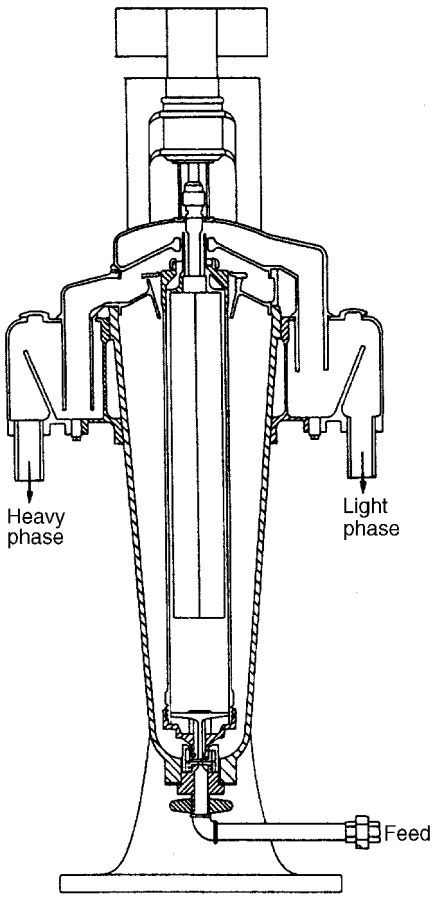


Figure 10.17. Tubular Bowl centrifuge

2. Disc bowl (Figure 10.18)

The conical discs in a disc bowl centrifuge split the liquid flow into a number of very thin layers, which greatly increases the separating efficiency. Disc bowl centrifuges are used for separating liquids and fine solids, and for solids classification.

3. Scroll discharge

In this type of machine the solids deposited on the wall of the bowl are removed by a scroll (a helical screw conveyer) which revolves at a slightly different speed from the

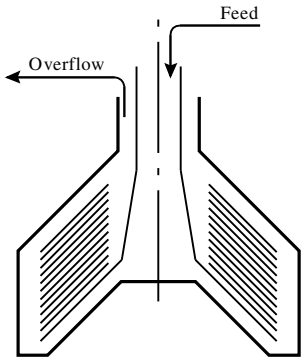


Figure 10.18. Disc bowl centrifuge

bowl. Scroll discharge centrifuges can be designed so that solids can be washed and relatively dry solids be discharged.

4. Solid bowl batch centrifuge

The simplest type; similar to the tubular bowl machine type but with a smaller bowl length to diameter ratio (less than 0.75). The tubular bowl type is rarely used for solids concentrations above 1 per cent by volume. For concentrations between 1 to 15 per cent, any of the other three types can be used. Above 15 per cent, either the scroll discharge type or the batch type may be used, depending on whether continuous or intermittent operation is required.

Sigma theory for sedimentation centrifuges

The basic equations describing sedimentation in a centrifugal field have been developed in Volume 2, Chapter 9. In that discussion the term *sigma* (Σ) is introduced, which can be used to define the performance of a centrifuge independently of the physical properties of the solid-fluid system. The sigma value of a centrifuge, normally expressed in cm^2, is equal to the cross-sectional area of a gravity settling tank having the same clarifying capacity.

This approach to describing centrifuge performance has become known as the "sigma theory". It provides a means for comparing the performance of sedimentation centrifuges and for scaling up from laboratory and pilot scale tests; see Ambler (1952) and Trowbridge (1962).

In the general case, it can be shown that:

$$Q = 2u_g\Sigma \tag{10.1}$$

and (where Stokes' law applies)

$$u_g = \frac{\Delta\rho d_s^2 g}{18\mu} \tag{10.2}$$

Note: The factor of 2 is included in equation 10.1 as d_s is the *cut-off* size, 50 per cent of particles of this size will be removed in passage through the centrifuge.

where Q = volumetric flow of liquid through the centrifuge, m^3/s,
u_g = terminal velocity of the solid particle settling under gravity through the liquid, m/s,
Σ = sigma value of the centrifuge, m^2,
$\Delta\rho$ = density difference between solid and liquid, kg/m^3
d_s = the diameter of the solid particle, *the cut-off* size, m ($\mu m \times 10^{-6}$),
μ = viscosity of the liquid, $Nm^{-2}s$.
g = gravitational acceleration, 9.81 m/s^2,

Morris (1966) gives a method for the selection of the appropriate type of sedimentation centrifuge for a particular application based on the ratio of the liquid overflow to sigma value (Q/Σ). His values for the operating range of each type, and their approximate efficiency rating, are given in Table 10.6. The efficiency term is used to account for the different amounts by which the various designs differ from the theoretical sigma values given by equation 10.1. Sigma values depend solely on the geometrical configuration and speed of the centrifuge. Details of the calculation for various types are given by Ambler (1952). To use Table 10.6, it is necessary to know the feed rate of slurry (and hence the liquid overflow Q), the density of the liquid and solid, the liquid viscosity; and the diameter of the particle for, say, a 98 per cent size removal. The use of Table 10.6 is illustrated in Example 10.1.

Table 10.6. Selection of sedimentation centrifuges

Type	Approximate efficiency (%)	Normal operating range Q, m^3/h at Q/Σ m/s
Tubular bowl	90	0.4 at 5×10^{-8} to 4 at 3.5×10^{-7}
Disc	45	0.1 at 7×10^{-8} to 110 at 4.5×10^{-7}
Solid bowl (scroll discharge)	60	0.7 at 1.5×10^{-6} to 15 at 1.5×10^{-5}
Solid bowl (basket)	75	0.4 at 5×10^{-6} to 4 at 1.5×10^{-4}

A selection guide for sedimentation centrifuges by Lavanchy *et al.* (1964), which includes other types of solid-liquid separators, is shown in Figure 10.19, adapted to SI units.

Example 10.1

A precipitate is to be continuously separated from a slurry. The solids concentration is 5 per cent and the slurry feed rate 5.5 m^3/h. The relevant physical properties at the system operating temperature are:

liquid density 1050 kg/m^3, viscosity 4 cp ($mNm^{-2}s$),
solid density 2300 kg/m^3, "cut-off" particle size 10 $\mu m = 10 \times 10^{-6}$ m.

Solution

$$\text{Overflow rate, } Q = 0.95 \times 5.5 = 5.23 \text{ m}^3/\text{h}$$
$$= \frac{5.13}{3600} = 1.45 \times 10^{-3} \text{ m}^3/\text{s}$$
$$\Delta\rho = 2300 - 1050 = 1250 \text{ kg/m}^3$$

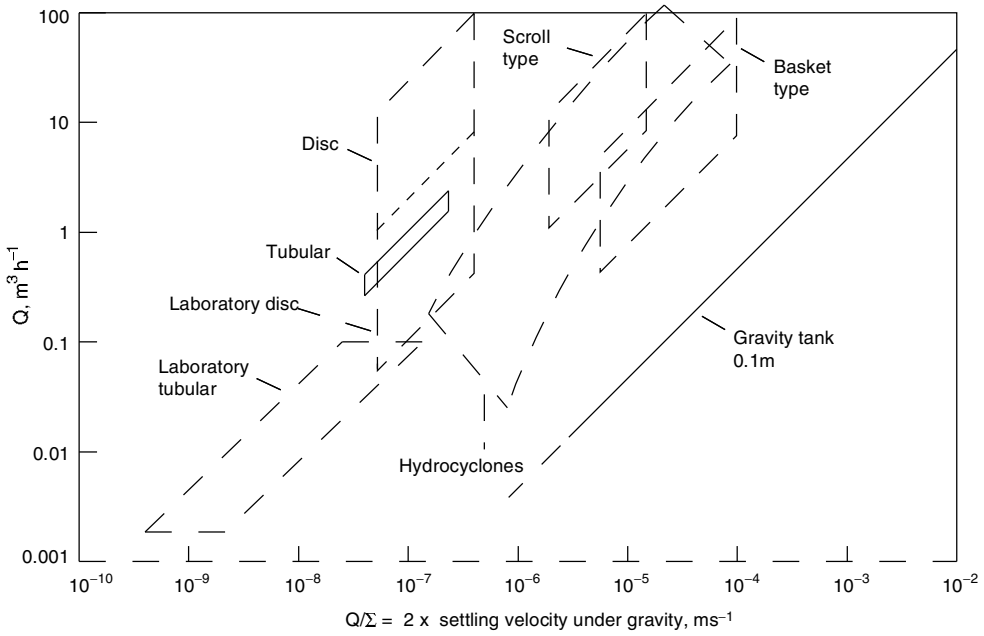


Figure 10.19. Performance of sedimentation equipment (after Lavanchy *et al.*, 1964)

From equations 10.1 and 10.2

$$\frac{Q}{\Sigma} = 2 \times \frac{1250(10 \times 10^{-6})^2}{18 \times 4 \times 10^{-3}} \times 9.81 = 3.4 \times 10^{-5}$$

From Table 10.6 for a Q of 5.23 m^3/h at a Q/Σ of 3.4×10^{-5} a solid bowl basket type should be used.

To obtain an idea of the size of the machine needed the sigma value can be calculated using the efficiency value from Table 10.6.

From equation 10.1:

$$\Sigma = \frac{Q}{eff. \times 2u_g} = \frac{1.45 \times 10^{-3}}{0.75 \times 3.4 \times 10^{-5}}$$

$$= \underline{\underline{56.9 \text{ m}^2}}$$

The sigma value is the equivalent area of a gravity settler that would perform the same separation as the centrifuge.

Filtration centrifuges (centrifugal filters)

It is convenient to classify centrifugal filters into two broad classes, depending on how the solids are removed: fixed bed or moving bed.

In the fixed-bed type, the cake of solids remains on the walls of the bowl until removed manually, or automatically by means of a knife mechanism. It is essentially cyclic in operation. In the moving-bed type, the mass of solids is moved along the bowl by the

action of a scroll (similar to the solid-bowl sedimentation type); or by a ram (pusher type); or by a vibration mechanism; or by the bowl angle. Washing and drying zones can be incorporated into the moving bed type.

Bradley (1965) has grouped the various types into the family tree shown in Figure 10.20.

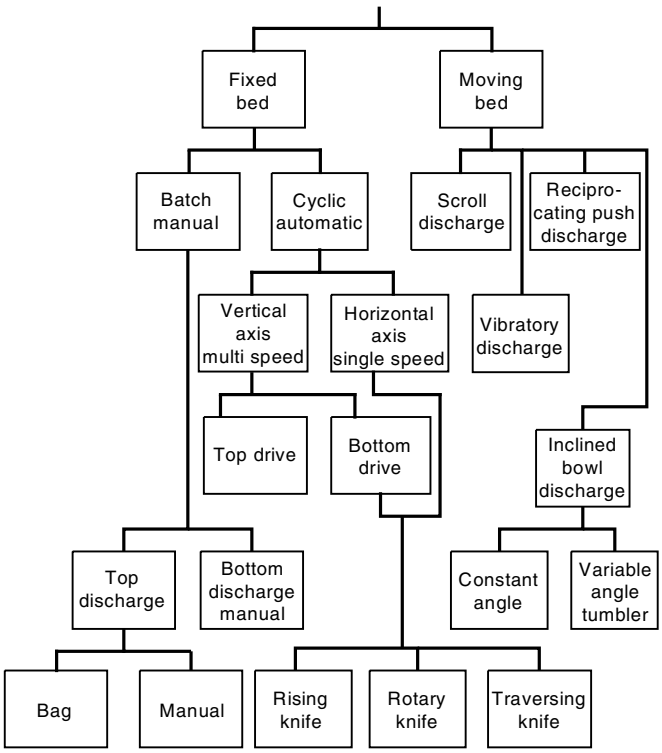


Figure 10.20. Filtration centrifuge family tree (after Bradley, 1965a)

Schematic diagrams of the various types are shown in Figure 10.21. The simplest machines are the basket types (Figures 10.21*a*, *b*, *c*), and these form the basic design from which the other types have been developed (Figures 10.21*d* to *o*).

The various arrangements of knife mechanisms used for automatic removal of the cake are shown in Figures 10.21*d* to *h*. The bottom discharge-type machines (Figures 10.21*d*, *e*) can be designed for variable speed, automatic discharge; and are suitable for use with fragile, or plate or needle-shaped crystals, where it is desirable to avoid breakage or compaction of the bed. They can be loaded and discharged at low speeds, which reduces breakage and compaction of the cake. The single-speed machines (Figures 10.21*f*, *g*, *h*) are used where cakes are thin, and short cycle times are required. They can be designed for high-temperature and pressure operation. When continuous operation is required, the scroll, pusher, or other self-discharge types are used (Figures 10.21*i* to *o*). The scroll discharge centrifuge is a low-cost, flexible machine, capable of a wide range of applications; but is not suitable for handling fragile materials.

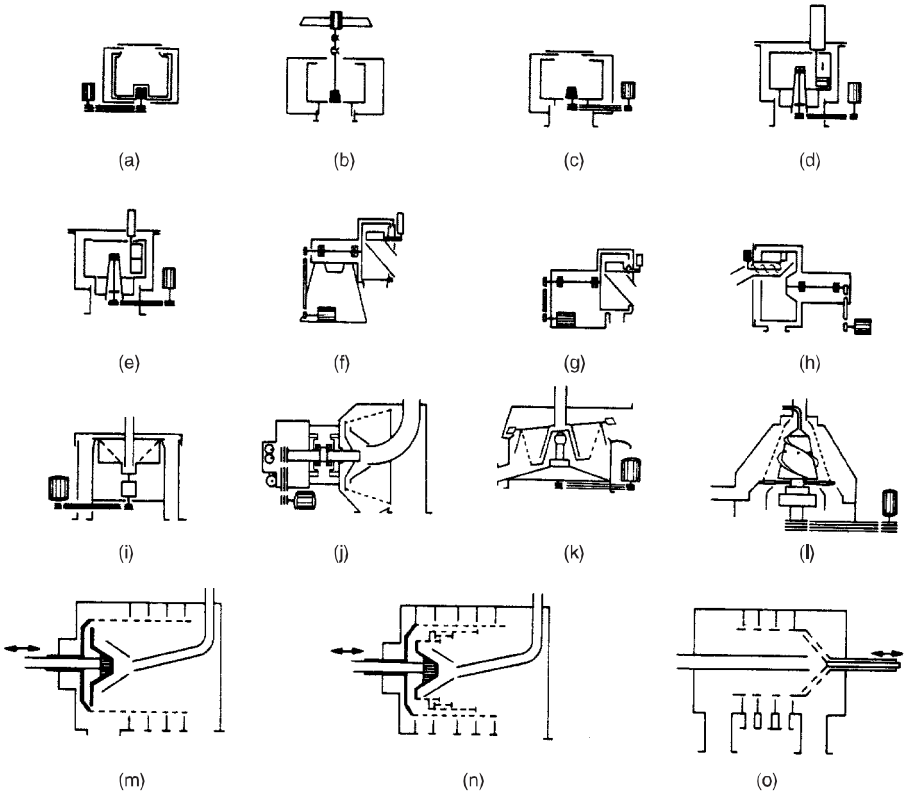


Figure 10.21. Schematic diagrams of filtration centrifuge types (Bradley, 1965) (*a*) Bottom drive batch basket with bag (*b*) Top drive bottom discharge batch basket (*c*) Bottom drive bottom discharge batch basket (*d*) Bottom drive automatic basket, rising knife (*e*) Bottom drive automatic basket, rotary knife (*f*) Single-reversing knife rising knife (*g*) Single-speed automatic rotary knife (*h*) Single-speed automatic traversing knife (*i*) Inclined wall self-discharge (*j*) Inclined vibrating wall self-discharge (*k*) Inclined "tumbling" wall self-discharge (*l*) Inclined wall scroll discharge (*m*) Traditional single-stage pusher (*n*) Traditional multi-stage pusher (*o*) Conical pusher with de-watering cone

It is normally used for coarse particles, where some contamination of the filtrate with fines can be tolerated.

The capacity of filtration centrifuges is very dependent on the solids concentration in the feed. For example, at 10 per cent feed slurry concentration 9 kg of liquid will be centrifuged for every 1 kg of solids separated; whereas with a 50 per cent solids concentration the quantity will be less than 1 kg. For dilute slurries it is well worth considering using some form of pre-concentration; such as gravity sedimentation or a hydrocyclone.

10.4.4. Hydrocyclones (liquid-cyclones)

Hydrocyclones are used for solid-liquid separations; as well as for solids classification, and liquid-liquid separation. It is a centrifugal device with a stationary wall, the centrifugal force being generated by the liquid motion. The operating principle is basically the same as that

of the gas cyclone described in Section 10.8.3, and in Volume 2, Chapter 8. Hydrocyclones are simple, robust, separating devices, which can be used over the particle size range from 4 to 500 μm. They are often used in groups, as illustrated in Figure 10.24*b*. The design and application of hydrocyclones is discussed fully in books by Abulnaga (2002) and Svarovsky and Thew (1992). Design methods and charts are also given by Zanker (1977), Day *et al*. (1997) and Moir (1985).

The nomographs by Zanker can be used to make a preliminary estimate of the size of cyclone needed. The specialist manufacturers of hydrocyclone equipment should be consulted to determine the best arrangements and design for a particular application.

Zanker's method is outlined below and illustrated in Example 10.2. Figure 10.23 is based on an empirical equation by Bradley (1960):

$$d_{50} = 4.5 \left[\frac{D_c^3 \mu}{L^{1.2}(\rho_s - \rho_L)} \right] \tag{10.3}$$

where d_{50} = the particle diameter for which the cyclone is 50 per cent efficient, μm,
D_c = diameter of the cyclone chamber, cm,

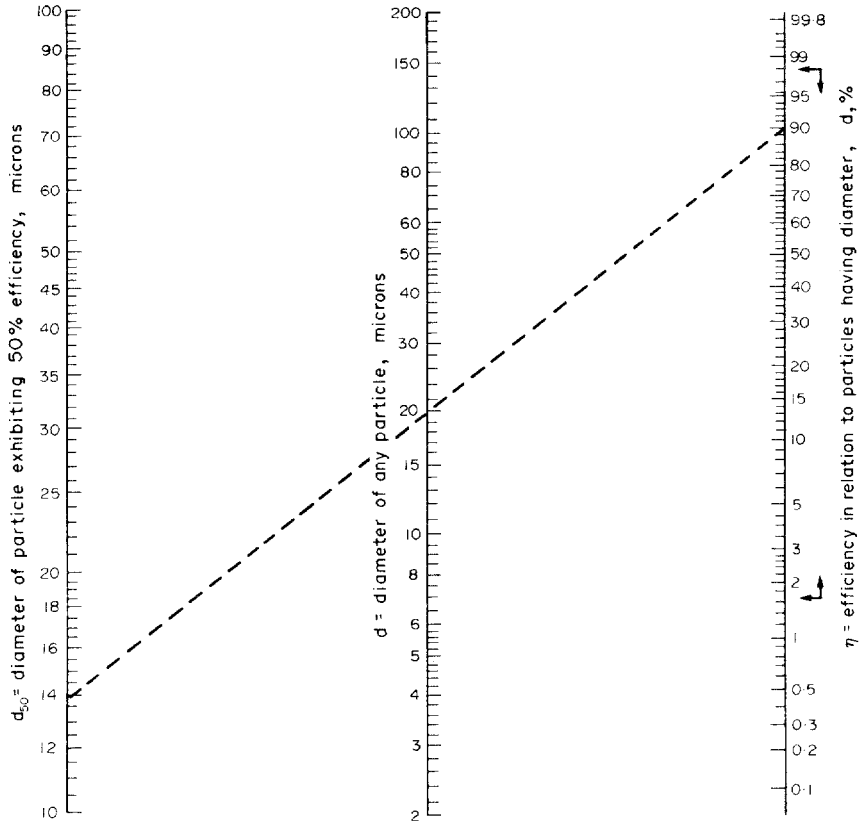


Figure 10.22. Determination of d_{50} from the desired particle separation (Equation 10.3, Zanker, 1977) (Example 10.2)

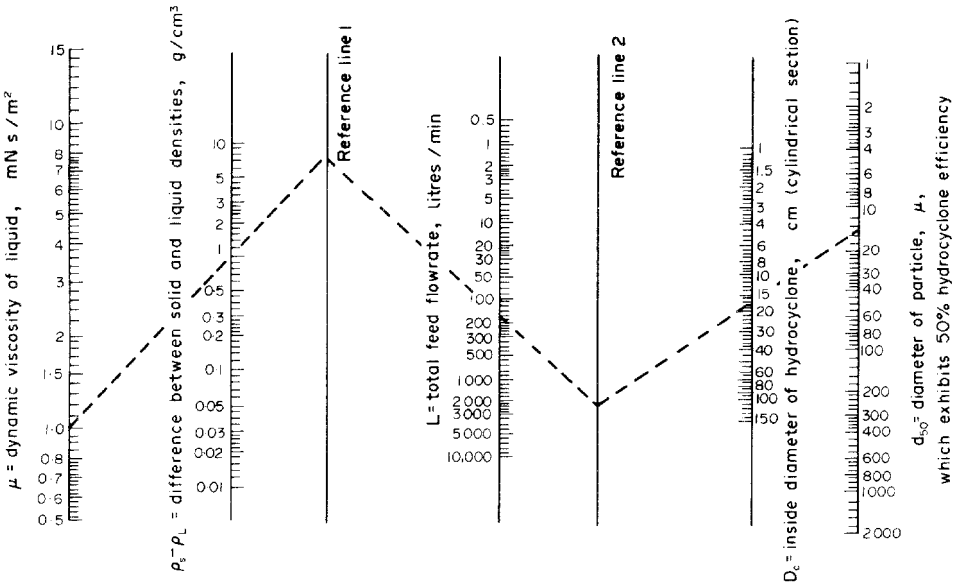
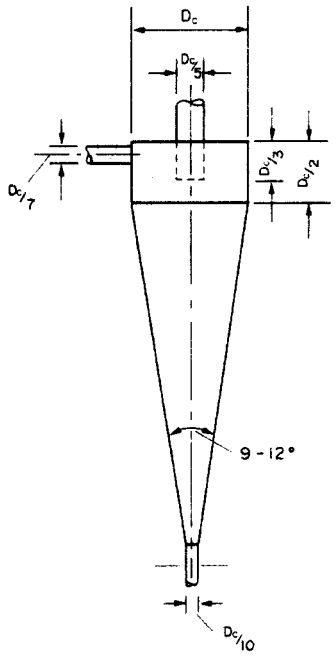


Figure 10.23. Chamber dia. D_c from flow-rate, physical properties, and d_{50} particle size (Equation 10.4, Zanker, 1977) (Example 10.2)



(a)

Figure 10.24. (*a*) Hydrocyclone-typical proportions

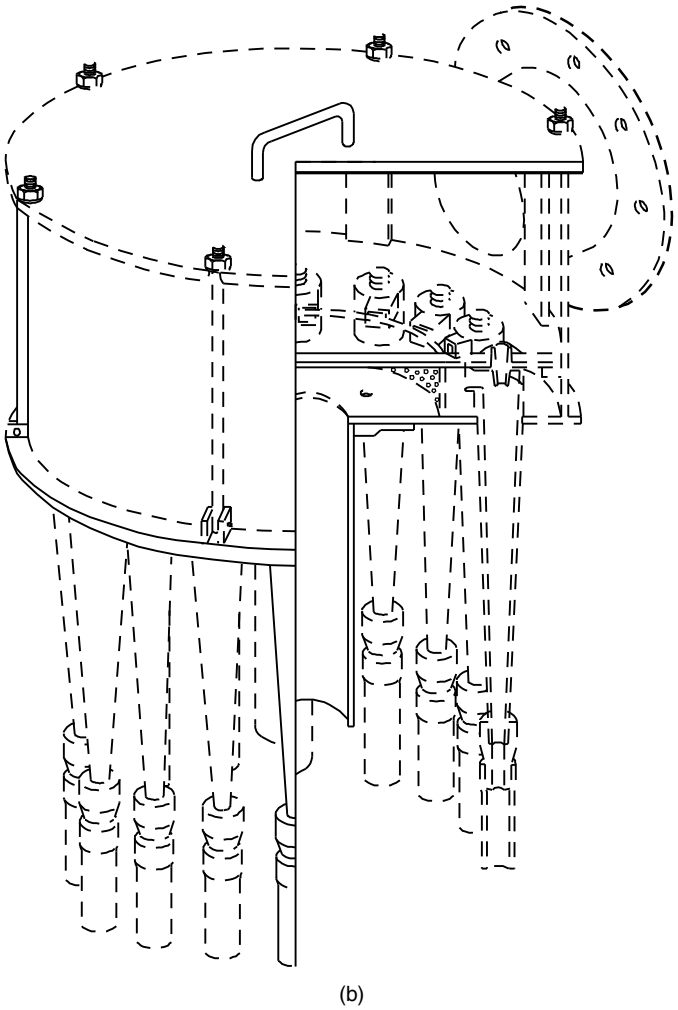


Figure 10.24. (*b*) A "Clog" assembly of 16 × 2 in (50 mm) diameter hydrocyclone. (Courtesy of Richard Mozley Ltd.)

μ = liquid viscosity, centipoise (mN s/m^2),
L = feed flow rate, l/min,
ρ_L = density of the liquid, g/cm^3,
ρ_s = density of the solid, g/cm^3.

The equation gives the chamber diameter required to separate the so-called d_{50} particle diameter, as a function of the slurry flow rate and the liquid and solid physical properties. The d_{50} particle diameter is the diameter of the particle, 50 per cent of which will appear in the overflow, and 50 per cent in the underflow. The separating efficiency for other particles is related to the d_{50} diameter by Figure 10.22, which is based on a formula by Bennett (1936).

$$\eta = 100\left[1 - e^{-(d/d_{50}-0.115)^3}\right] \tag{10.4}$$

where η = the efficiency of the cyclone in separating any particle of diameter d, per cent,
d = the selected particle diameter, μm.
The method applies to hydrocyclones with the proportions shown in Figure 10.24.

Example 10.2

Estimate the size of hydrocyclone needed to separate 90 per cent of particles with a diameter greater than 20 μm, from 10 m^3/h of a dilute slurry.

Physical properties: solid density 2000 kg/m^3, liquid density 1000 kg/m^3, viscosity 1 mN s/m^2

Solution

$$\text{Flow-rate} = \frac{10 \times 10^3}{60} = 166.71/\text{min}$$
$$(\rho_s - \rho_L) = 2.0 - 1.0 = 1.0 \text{ g/cm}^3$$

From Figure 10.22, for 90 per cent removal of particles above 20 μm

$$d_{50} = 14 \ \mu\text{m}$$

From Figure 10.23, for $\mu = 1$ mN s/m^2, $(\rho_s - \rho_L)$ = 1.0 g/cm^3, L = 167/min

$$D_c = \underline{\underline{16 \text{ cm}}}$$

10.4.5. Pressing (expression)

Pressing, in which the liquid is squeezed (expressed) from a mass of solids by compression, is used for certain specialised applications. Pressing consumes a great deal of energy, and should not be used unless no other separating technique is suitable. However, in some applications dewatering by pressing can be competitive with drying.

Presses are of two basic types: hydraulic batch presses and screw presses. Hydraulic presses are used for extracting fruit juices, and screw presses for dewatering materials; such as paper pulp, rubbish and manure. The equipment used is described in the handbooks; Perry *et al.* (1997).

10.4.6. Solids drying

Drying is the removal of water, or other volatile liquids, by evaporation. Most solid materials require drying at some stage in their production. The choice of suitable drying equipment cannot be separated from the selection of the upstream equipment feeding the drying stage.

Table 10.7. Dryer selection

Mode of operation	Generic type	Feed condition 1	2	3	Specific dryer types	Jacketed	Suitable for heat-sensitive materials	Suitable for vacuum service	Retention or cycle time	Heat transfer method	Capacity	Typical evaporation capacity
Batch	Stationary	←	→	→	1. Shelf 2. Cabinet 3. Compartment	Yes	Yes	Yes	6.48 h	Radiant and conduction	Limited	0.15–1.0 kg/hm² heat transfer area
			→	→	Truck	No	Yes	No	6.48 h	Convection	Limited	0.15–1.0
		→	→		1. Kettle 2. Pan	Yes	No	Yes	3.12 h	Conduction	Limited	1.5–15
			→	→	Rotary shell	Yes	Yes	Yes	4.48 h	Conduction	Limited	0.5–12
			→	→	Rotary internal	Yes	Yes	Yes	4.48 h	Conduction	Limited	0.5–12
			→	→	Double cone	Yes	Yes	Yes	3.12 h	Conduction	Limited	0.5–12
Continuous	Drum	→			1. Single drum 2. Double drum 3. Twin drum	No	Yes	Yes	Very short	Conduction	Medium	5–50
	Rotary		→	→	Rotary direct heat	No	No	No	Long	Convection	High	3–110 kg/hm³ dryer volume
			→	→	Rotary, indirect heat	No	No	No	Long	Conduction	Medium	15–200
			→	→	Rotary, steam tube	No	Depends on material	No	Long	Conduction	High	15–200
			→	→	Rotary, direct-indirect heat	No	No	No	Long	Conduction Convection	High	50–150
			→	→	Louver	No	Depends on material	No	Long	Convection	High	5–240
			→	→	Tunnel belt, screen	No	Yes	No	Long	Convection	Medium	1.5–35
	Conveyor		→	→	Rotary shelf	Yes	Depends on material	No	Medium	Conduction Convection	Medium	0.5–10 kg/hm²
			→	→	Trough	Yes	Depends on material	Yes	Varies	Conduction	Medium	0.5–15
			→	→	Vibrating	Yes	Depends on material	No	Medium	Convection Conduction	Medium	0.5–100
			→	→	Turbo	No	Depends on material	No	Medium	Convection	Medium	1–10
	Suspended particle	→			Spray	No	Yes	No	Short	Convection	High	1.5–50 kg/hm³
			→	→	Flash	No	Yes	No	Short	Convection	High	–
			→	→	Fluid bed	No	Yes	No	Short	Convection	Medium	–

←——→ = applicable to feed conditions noted

Key to feed conditions:

1. Solutions, colloidal suspensions and emulsions, pumpable solids suspensions, pastes and sludges.
2. Free-flowing powders, granular, crystalline or fibrous solids that can withstand mechanical handling.
3. Solids incapable of withstanding mechanical handling.

The overriding consideration in the selection of drying equipment is the nature and concentration of the feed. Drying is an energy-intensive process, and the removal of liquid by thermal drying will be more costly than by mechanical separation techniques.

Drying equipment can be classified according to the following design and operating features:

1. Batch or continuous.
2. Physical state of the feed: liquid, slurry, wet solid.
3. Method of conveyance of the solid: belt, rotary, fluidised.
4. Heating system: conduction, convection, radiation.

Except for a few specialised applications, hot air is used as the heating and mass transfer medium in industrial dryers. The air may be directly heated by the products of combustion of the fuel used (oil, gas or coal) or indirectly heated, usually by banks of steam-heated finned tubes. The heated air is usually propelled through the dryer by electrically driven fans.

Table 10.7, adapted from a similar selection guide by Parker (1963a), shows the basic features of the various types of solids dryer used in the process industries; and Table 10.8, by Williams-Gardner (1965), shows typical applications.

Batch dryers are normally used for small-scale production and where the drying cycle is likely to be long. Continuous dryers require less labour, less floor space; and produce a more uniform quality product.

When the feed is solids, it is important to present the material to the dryer in a form that will produce a bed of solids with an open, porous, structure.

For pastes and slurries, some form of pretreatment equipment will normally be needed, such as extrusion or granulation.

The main factors to be considered when selecting a dryer are:

1. Feed condition: solid, liquid, paste, powder, crystals.
2. Feed concentration, the initial liquid content.
3. Product specification: dryness required, physical form.
4. Throughput required.
5. Heat sensitivity of the product.
6. Nature of the vapour: toxicity, flammability.
7. Nature of the solid: flammability (dust explosion hazard), toxicity.

The drying characteristics of the material can be investigated by laboratory and pilot plant tests; which are best carried out in consultation with the equipment vendors.

The theory of drying processes is discussed in Volume 2, Chapter 16. Full descriptions of the various types of dryer and their applications are given in that chapter and in Perry *et al.* (1997) and Walas (1990). Only brief descriptions of the principal types will be given in this section.

The basic types used in the chemical process industries are: tray, band, rotary, fluidised, pneumatic, drum and spray dryers.

Tray dryers (Figure 10.25)

Batch tray dryers are used for drying small quantities of solids, and are used for a wide range of materials.

Table 10.8. Dryer applications

Dryer type	*System*	*Feed form*	*Typical products*
Batch ovens	Forced convection	Paste, granules, extrude cake	Pigment dyestuffs, pharmaceuticals, fibres
	Vacuum	Extrude cake	Pharmaceuticals
,, pan (agitated)	Atmospheric and vacuum	Crystals, granules, powders	Fine chemicals, food products
,, rotary	Vacuum	Crystals, granules solvent recovery	Pharmaceuticals
,, fluid bed	Forced convection	Granular, crystals	Fine chemicals, pharmaceuticals, plastics
,, infra-red	Radiant	Components sheets	Metal products, plastics
Continuous rotary	Convection Direct/indirect Direct Indirect Conduction	Crystals, coarse powders, extrudes, preformed cake lumps, granular paste and fillers, cakes back-mixed with dry product	Chemical ores, food products, clays, pigments, chemicals Carbon black
,, film drum	Conduction	Liquids, suspensions	Foodstuffs, pigment
,, trough	Conduction		Ceramics, adhesives
,, spray	Convection	Liquids, suspensions	Foodstuffs, pharmaceuticals, ceramics, fine chemicals, detergents, organic extracts
,, band	Convection	Preformed solids	Foodstuffs, pigments, chemicals, rubber, clays, ores, textiles
,, fluid bed	Convection	Preformed solids granules, crystals	Ores, coal, clays, chemicals
,, pneumatic	Convection	Preformed pastes, granules, crystals, coarse products	Chemicals, starch, flour, resins, wood-products, food products
,, infra-red	Radiant	Components sheets	Metal products, moulded fibre articles, painted surfaces

The material to be dried is placed in solid bottomed trays over which hot air is blown; or perforated bottom trays through which the air passes.

Batch dryers have high labour requirements, but close control can be maintained over the drying conditions and the product inventory, and they are suitable for drying valuable products.

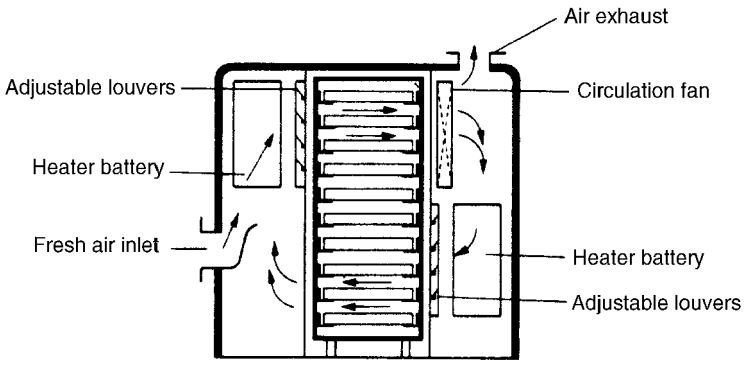


Figure 10.25. Tray dryer

Conveyor dryers (continuous circulation band dryers) (Figure 10.26)

In this type, the solids are fed on to an endless, perforated, conveyor belt, through which hot air is forced. The belt is housed in a long rectangular cabinet, which is divided up into zones, so that the flow pattern and temperature of the drying air can be controlled. The relative movement through the dryer of the solids and drying air can be parallel or, more usually, counter-current.

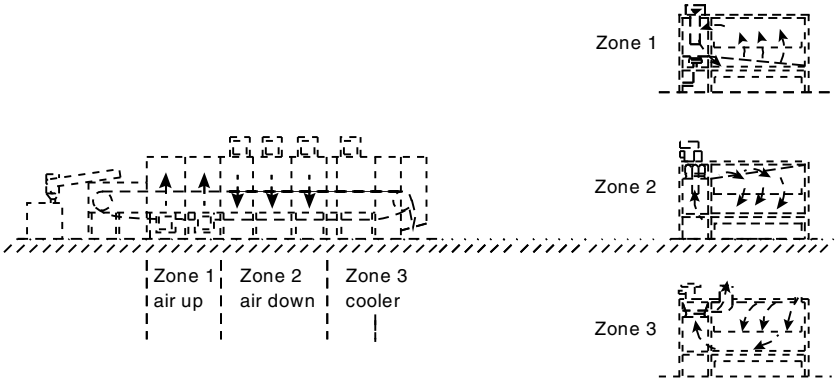


Figure 10.26. Conveyor dryer

This type of dryer is clearly only suitable for materials that form a bed with an open structure. High drying rates can be achieved, with good product-quality control. Thermal efficiencies are high and, with steam heating, steam usage can be as low as 1.5 kg per kg of water evaporated. The disadvantages of this type of dryer are high initial cost and, due to the mechanical belt, high maintenance costs.

Rotary dryer (Figure 10.27)

In rotary dryers the solids are conveyed along the inside of a rotating, inclined, cylinder and are heated and dried by direct contact with hot air gases flowing through the cylinder. In some, the cylinders are indirectly heated.

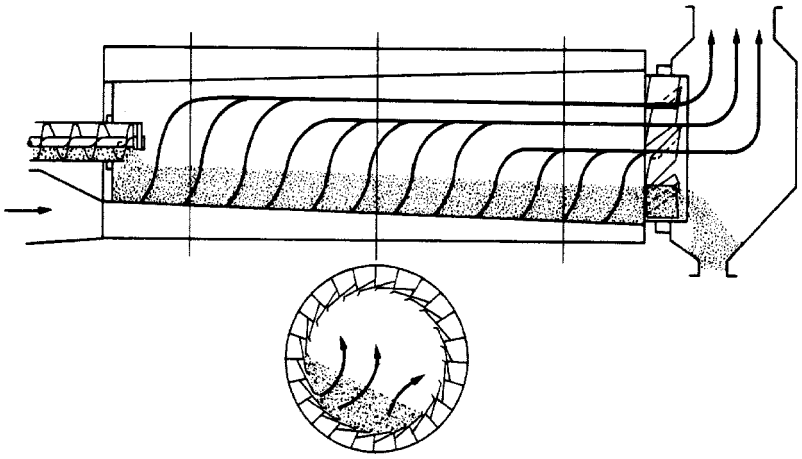


Figure 10.27. Rotary dryer

Rotating dryers are suitable for drying free-flowing granular materials. They are suitable for continuous operation at high throughputs; have a high thermal efficiency and relatively low capital cost and labour costs. Some disadvantages of this type are: a non-uniform residence time, dust generation and high noise levels.

Fluidised bed dryers (Figure 10.28)

In this type of dryer, the drying gas is passed through the bed of solids at a velocity sufficient to keep the bed in a fluidised state; which promotes high heat transfer and drying rates.

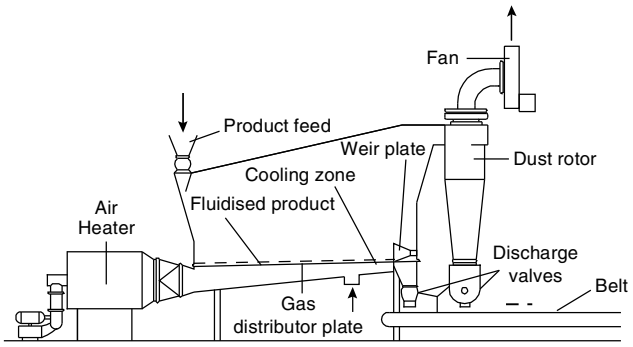


Figure 10.28. Fluidised bed dryer

Fluidised bed dryers are suitable for granular and crystalline materials within the particle size range 1 to 3 mm. They are designed for continuous and batch operation.

The main advantages of fluidised dryers are: rapid and uniform heat transfer; short drying times, with good control of the drying conditions; and low floor area requirements. The power requirements are high compared with other types.

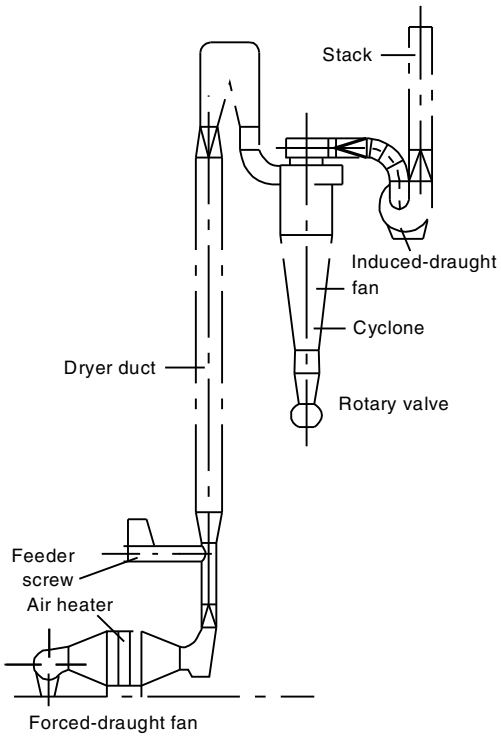


Figure 10.29. Pneumatic dryer

Pneumatic dryers (Figure 10.29)

Pneumatic dryers, also called flash dryers, are similar in their operating principle to spray dryers. The product to be dried is dispersed into an upward-flowing stream of hot gas by a suitable feeder. The equipment acts as a pneumatic conveyor and dryer. Contact times are short, and this limits the size of particle that can be dried. Pneumatic dryers are suitable for materials that are too fine to be dried in a fluidised bed dryer but which are heat sensitive and must be dried rapidly. The thermal efficiency of this type is generally low.

Spray dryers (Figure 10.30)

Spray dryers are normally used for liquid and dilute slurry feeds, but can be designed to handle any material that can be pumped. The material to be dried is atomised in a nozzle, or by a disc-type atomiser, positioned at the top of a vertical cylindrical vessel. Hot air flows up the vessel (in some designs downward) and conveys and dries the droplets. The liquid vaporises rapidly from the droplet surface and open, porous particles are formed. The dried particles are removed in a cyclone separator or bag filter.

The main advantages of spray drying are the short contact time, making it suitable for drying heat-sensitive materials, and good control of the product particle size, bulk density, and form. Because the solids concentration in the feed is low the heating requirements will be high. Spray drying is discussed in a book by Masters (1991).

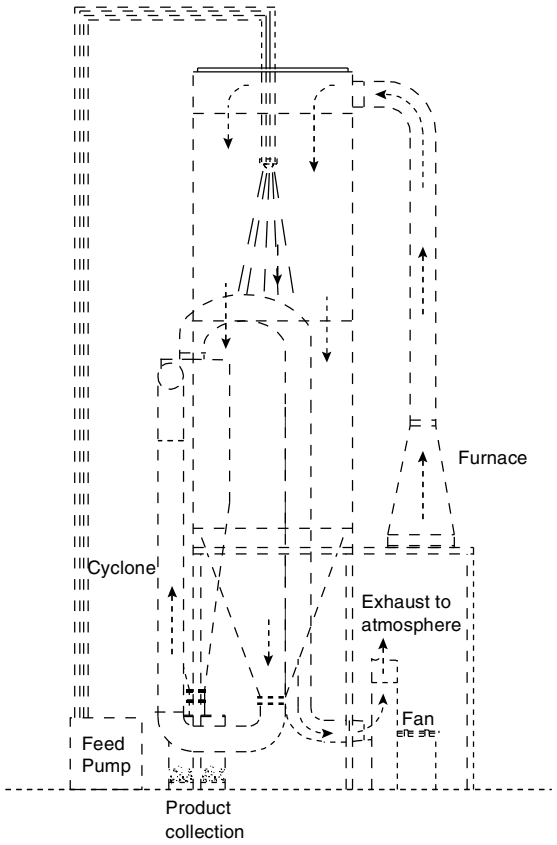


Figure 10.30. Spray dryer

Rotary drum dryers (Figure 10.31)

Drum dryers are used for liquid and dilute slurry feeds. They are an alternative choice to spray dryers when the material to be dried will form a film on a heated surface, and is not heat sensitive.

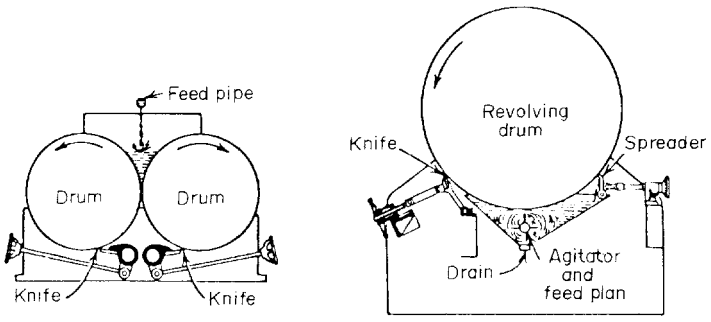


Figure 10.31. Rotary drum dryers

They consist essentially of a revolving, internally heated, drum, on which a film of the solids is deposited and dried. The film is formed either by immersing part of the drum in a trough of the liquid or by spraying, or splashing, the feed on to the drum surface; double drums are also used in which the feed is fed into the "nip" formed between the drums.

The drums are usually heated with steam, and steam economies of 1.3 kg steam per kg of water evaporated are typically achieved.

10.5. SEPARATION OF DISSOLVED SOLIDS

On an industrial scale, evaporation and crystallisation are the main processes used for the recovery of dissolved solids from solutions.

Membrane filtration processes, such as reverse osmosis, and micro and ultra filtration, are used to "filter out" dissolved solids in certain applications; see Table 10.9. These specialised processes will not be discussed in this book. A comprehensive description of the techniques used and their applications is given in Volume 2, Chapter 8; see also: Scott and Hughes (1995), Cheryan (1986), McGregor (1986) and Porter (1997).

Table 10.9. Membrane filtration process

Process	Approximate size range (m)	Applications
Microfiltration	10^{-8} to 10^{-4}	pollen, bacteria, blood cells
Ultrafiltration	10^{-9} to 10^{-8}	proteins and virus
Nanofiltration	5×10^{-9} to 15×10^{-9}	water softening
Reverse osmosis	10^{-10} to 10^{-9}	desalination
Dialysis	10^{-9} to molecules	blood purification
Electrodialysis	10^{-9} to molecules	separation of electrolytes
Pervaporation	10^{-9} to molecules	dehydration of ethanol
Gas permeation	10^{-9} to molecules	hydrogen recovery, dehydration

10.5.1. Evaporators

Evaporation is the removal of a solvent by vaporisation, from solids that are not volatile. It is normally used to produce a concentrated liquid, often prior to crystallisation, but a dry solid product can be obtained with some specialised designs. The general subject of evaporation is covered in Volume 2, Chapter 14. That chapter includes a discussion of heat transfer in evaporators, multiple-effect evaporators, and a description of the principal types of equipment. The selection of the appropriate type of evaporator is discussed by Cole (1984). Evaporation is the subject of a book by Billet (1989).

A great variety of evaporator designs have been developed for specialised applications in particular industries. The designs can be grouped into the following basic types.

Direct-heated evaporators

This type includes solar pans and submerged combustion units. Submerged combustion evaporators can be used for applications where contamination of the solution by the products of combustion is acceptable.

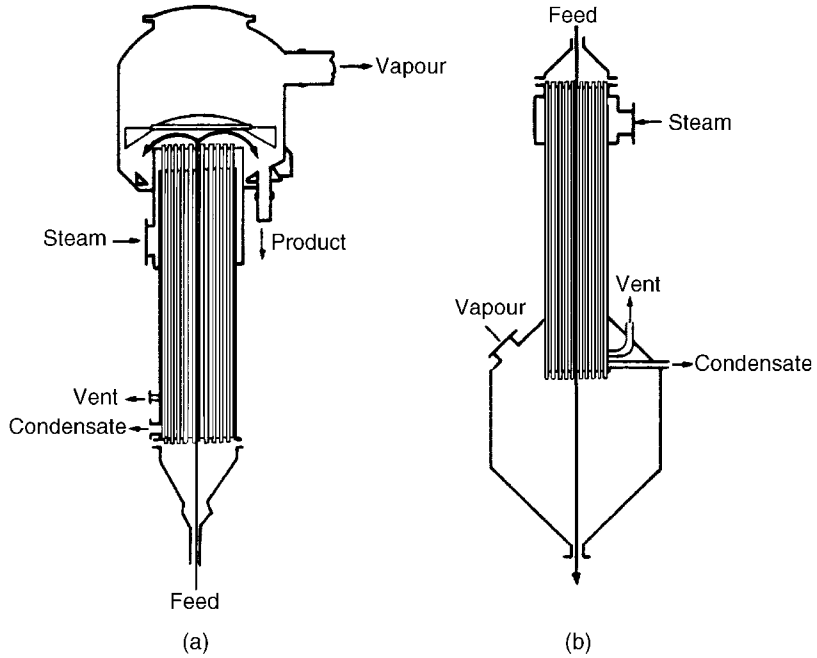


Figure 10.32. Long-tube evaporators (*a*) Rising film (*b*) Falling film

Long-tube evaporators (Figure 10.32)

In this type the liquid flows as a thin film on the walls of a long, vertical, heated, tube. Both falling film and rising film types are used. They are high capacity units; suitable for low viscosity solutions.

Forced-circulation evaporators (Figure 10.33)

In forced circulation evaporators the liquid is pumped through the tubes. They are suitable for use with materials which tend to foul the heat transfer surfaces, and where crystallisation can occur in the evaporator.

Agitated thin-film evaporators (Figure 10.34)

In this design a thin layer of solution is spread on the heating surface by mechanical means. Wiped-film evaporators are used for very viscous materials and for producing solid products. The design and applications of this type of evaporator are discussed by Mutzenburg (1965), Parker (1965) and Fischer (1965).

Short-tube evaporators

Short-tube evaporators, also called callandria evaporators, are used in the sugar industry; see Volume 2.

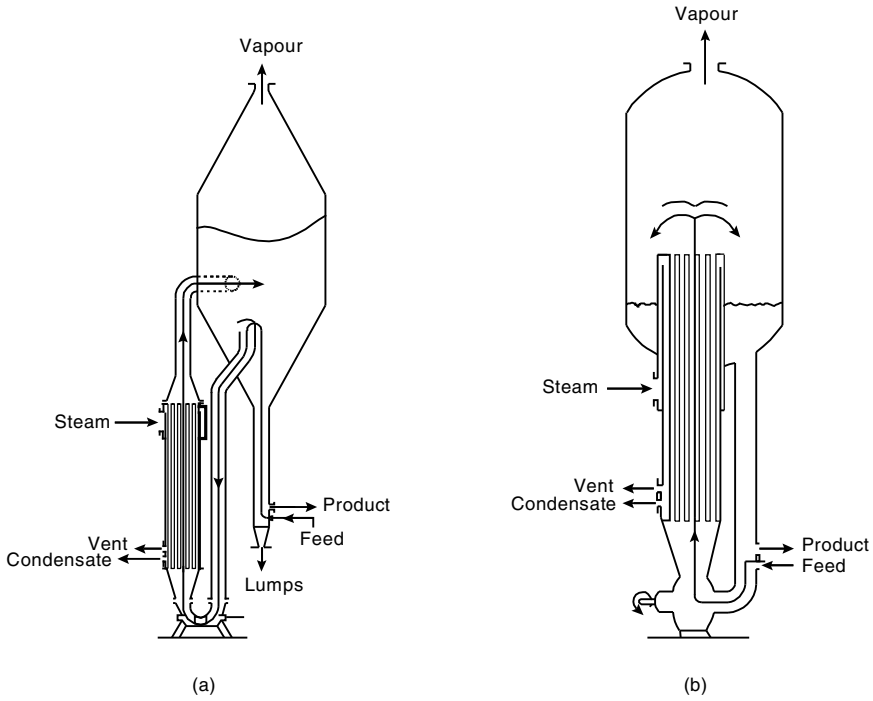


Figure 10.33. Forced-circulation evaporators (*a*) Submerged tube (*b*) Boiling tube

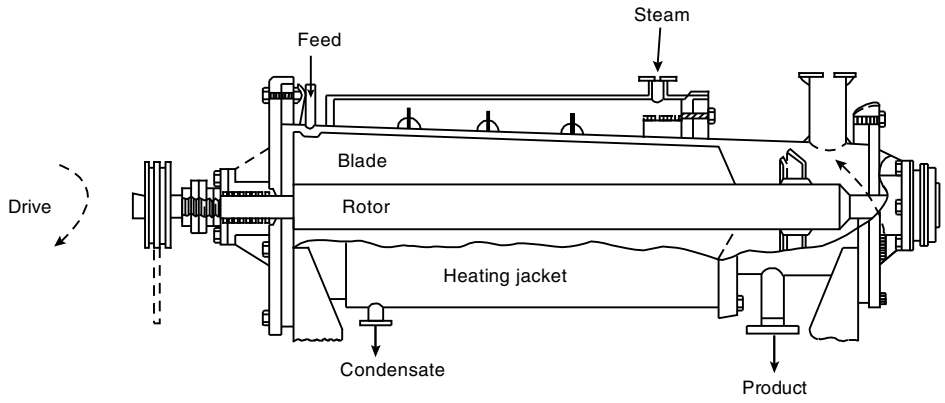


Figure 10.34. Horizontal wiped-film evaporator

Evaporator selection

The selection of the most suitable evaporator type for a particular application will depend on the following factors:

1. The throughput required.
2. The viscosity of the feed and the increase in viscosity during evaporation.

3. The nature of the product required; solid, slurry, or concentrated solution.
4. The heat sensitivity of the product.
5. Whether the materials are fouling or non-fouling.
6. Whether the solution is likely to foam.
7. Whether direct heating can be used.

A selection guide based on these factors is given in Figure 10.35; see also Parker (1963*b*).

	Feed conditions							Suitable for heat-sensitive materials
	Viscosity, mN s/m^2							
Evaporator type	Very viscous > 1000	Medium viscosity < 1000 max	Low viscosity < 100	Foaming	Scaling or fouling	Crystals produced	Solids in suspension	
Recirculating Calandria (short vertical tube)		←	—	—	—	—	→	No
Forced circulation		←	—	—	—	→		Yes
Falling film			↔					No
Natural circulation			←	→				No
Single pass wiped film	←	—	—	—	—	—	→	Yes
Tubular (long tube) Falling film			↔					Yes
Rising film			←	→				Yes

Figure 10.35. Evaporator selection guide

Auxilliary equipment

Condensers and vacuum pumps will be needed for evaporators operated under vacuum. For aqueous solutions, steam ejectors and jet condensers are normally used. Jet condensers are direct-contact condensers, where the vapour is condensed by contact with jets of cooling water. Indirect, surface condensers, are used where it is necessary to keep the condensed vapour and cooling water effluent separate.

10.5.2. Crystallisation

Crystallisation is used for the production, purification and recovery of solids. Crystalline products have an attractive appearance, are free flowing, and easily handled and packaged. The process is used in a wide range of industries: from the small-scale production of specialised chemicals, such as pharmaceutical products, to the tonnage production of products such as sugar, common salt and fertilisers.

Crystallisation theory is covered in Volume 2, Chapter 15 and in other texts: Mullin (2001) and Jones (2002). Descriptions of the various crystallisers used commercially can be found in these texts and in handbooks: Mersmann (2001), Perry *et al*. (1997) and

Schweitzer (1997). Procedures for the scale-up and design of crystallisers are given by Mersmann (2001), and Mersham (1988), (1984).

Precipitation, which can be considered as a branch of crystallisation, is covered by Sohnel and Garside (1992).

Crystallisation equipment can be classified by the method used to obtain supersaturation of the liquor, and also by the method used to suspend the growing crystals. Supersaturation is obtained by cooling or evaporation. There are four basic types of crystalliser; these are described briefly below.

Tank crystallisers

This is the simplest type of industrial crystallising equipment. Crystallisation is induced by cooling the mother liquor in tanks; which may be agitated and equipped with cooling coils or jackets. Tank crystallisers are operated batchwise, and are generally used for small-scale production.

Scraped-surface crystallisers

This type is similar in principle to the tank type, but the cooling surfaces are continually scraped or agitated to prevent the fouling by deposited crystals and to promote heat transfer. They are suitable for processing high-viscosity liquors. Scraped-surface

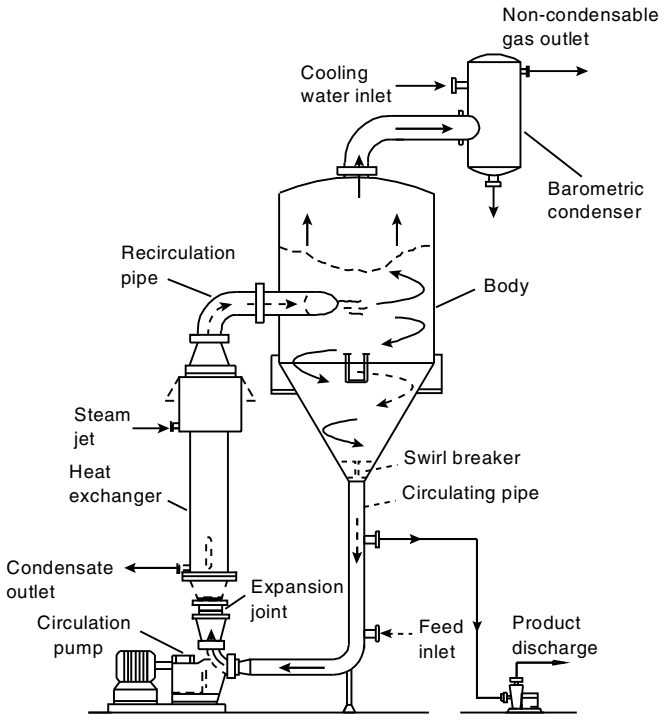


Figure 10.36. Circulating magma crystalliser (evaporative type)

crystallisers can be operated batchwise, with recirculation of the mother liquor, or continuously. A disadvantage of this type is that they tend to produce very small crystals.

Circulating magma crystallisers (Figure 10.36)

In this type both the liquor and growing crystals are circulated through the zone in which supersaturation occurs. Circulating magma crystallisers are probably the most important type of large-scale crystallisers used in the chemical process industry. Designs are available in which supersaturation is achieved by direct cooling, evaporation or evaporative cooling under vacuum.

Circulating liquor crystallisers (Figure 10.37)

In this type only the liquor is circulated through the heating or cooling equipment; the crystals are retained in suspension in the crystallising zone by the up-flow of liquor. Circulating liquor crystallisers produce crystals of regular size. The basic design consists of three components: a vessel in which the crystals are suspended and grow and are removed; a means of producing supersaturation, by cooling or evaporation; and a means of circulating the liquor. The Oslo crystalliser (Figure 10.37) is the archetypical design for this type of crystallising equipment.

Circulating liquor crystallisers and circulating magma crystallisers are used for the large-scale production of a wide range of crystal products.

Typical applications of the main types of crystalliser are summarised in Table 10.10 (see page 440); see also Larson (1978).

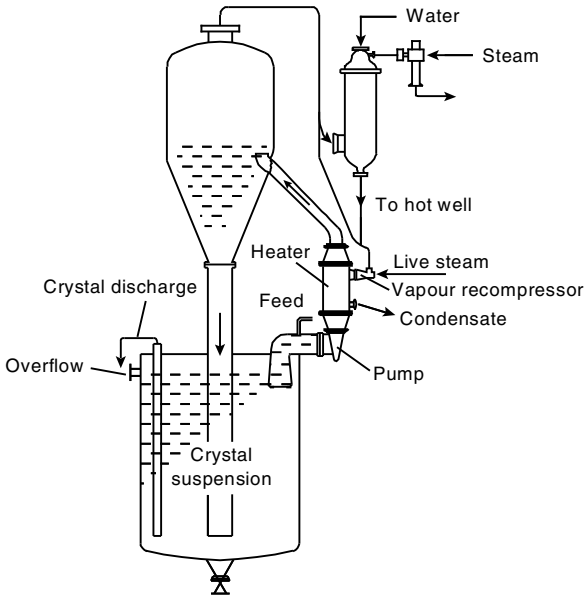


Figure 10.37. Oslo evaporative crystalliser

Table 10.10. Selection of crystallisers

Crystalliser type	Applications	Typical uses
Tank	Batch operation, small-scale production	Fatty acids, vegetable oils, sugars
Scraped surface	Organic compounds, where fouling is a problem, viscous materials	Chlorobenzenes, organic acids, paraffin waxes, naphthalene, urea
Circulating magma	Production of large-sized crystals. High throughputs	Ammonium and other inorganic salts, sodium and potassium chlorides
Circulating liquor	Production of uniform crystals (smaller size than circulating magma). High throughputs.	Gypsum, inorganic salts, sodium and potassium nitrates, silver nitrates

10.6. LIQUID-LIQUID SEPARATION

Separation of two liquid phases, immiscible or partially miscible liquids, is a common requirement in the process industries. For example, in the unit operation of liquid-liquid extraction the liquid contacting step must be followed by a separation stage (Chapter 11, Section 11.16). It is also frequently necessary to separate small quantities of entrained water from process streams. The simplest form of equipment used to separate liquid phases is the gravity settling tank, the decanter. Various proprietary equipment is also used to promote coalescence and improve separation in difficult systems, or where emulsions are likely to form. Centrifugal separators are also used.

10.6.1. Decanters (settlers)

Decanters are used to separate liquids where there is a sufficient difference in density between the liquids for the droplets to settle readily. Decanters are essentially tanks which give sufficient residence time for the droplets of the dispersed phase to rise (or settle) to the interface between the phases and coalesce. In an operating decanter there will be three distinct zones or bands: clear heavy liquid; separating dispersed liquid (the dispersion zone); and clear light liquid.

Decanters are normally designed for continuous operation, but the same design principles will apply to batch operated units. A great variety of vessel shapes is used for decanters, but for most applications a cylindrical vessel will be suitable, and will be the cheapest shape. Typical designs are shown in Figures 10.38 and 10.39. The position of the interface can be controlled, with or without the use of instruments, by use of a syphon take-off for the heavy liquid, Figure 10.38.

The height of the take-off can be determined by making a pressure balance. Neglecting friction loss in the pipes, the pressure exerted by the combined height of the heavy and light liquid in the vessel must be balanced by the height of the heavy liquid in the take-off leg, Figure 10.38.

$$(z_1 - z_3)\rho_1 g + z_3\rho_2 g = z_2\rho_2 g$$

hence

$$z_2 = \frac{(z_1 - z_3)\rho_1}{\rho_2} + z_3 \tag{10.5}$$

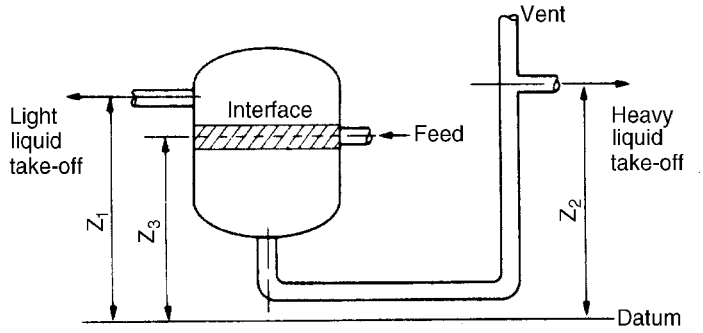


Figure 10.38. Vertical decanter

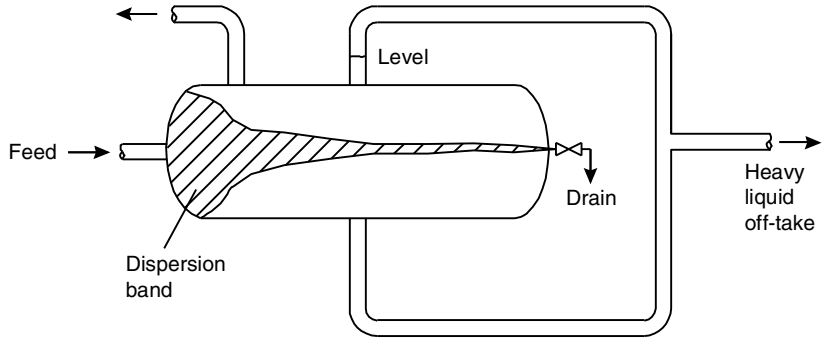


Figure 10.39. Horizontal decanter

where ρ_1 = density of the light liquid, kg/m^3,
ρ_2 = density of the heavy liquid, kg/m^3,
z_1 = height from datum to light liquid overflow, m,
z_2 = height from datum to heavy liquid overflow, m,
z_3 = height from datum to the interface, m.

The height of the liquid interface should be measured accurately when the liquid densities are close, when one component is present only in small quantities, or when the throughput is very small. A typical scheme for the automatic control of the interface, using a level instrument that can detect the position of the interface, is shown in Figure 10.40. Where one phase is present only in small amounts it is often recycled to the decanter feed to give more stable operation.

Decanter design

A rough estimate of the decanter volume required can be made by taking a hold-up time of 5 to 10 min, which is usually sufficient where emulsions are not likely to form. Methods

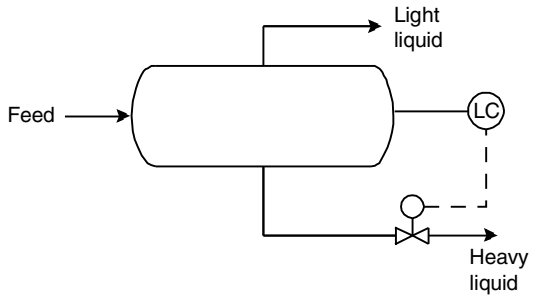


Figure 10.40. Automatic control, level controller detecting interface

for the design of decanters are given by Hooper (1997) and Signales (1975). The general approach taken is outlined below and illustrated by Example 10.3.

The decanter vessel is sized on the basis that the velocity of the continuous phase must be less than settling velocity of the droplets of the dispersed phase. Plug flow is assumed, and the velocity of the continuous phase calculated using the area of the interface:

$$u_c = \frac{L_c}{A_i} < u_d \tag{10.6}$$

where u_d = settling velocity of the dispersed phase droplets, m/s,
u_c = velocity of the continuous phase, m/s,
L_c = continuous phase volumetric flow rate, m^3/s,
A_i = area of the interface, m^2.

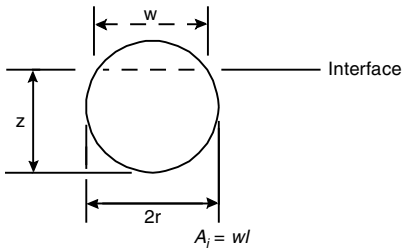
Stokes' law (see Volume 2, Chapter 3) is used to determine the settling velocity of the droplets:

$$u_d = \frac{d_d^2 g(\rho_d - \rho_c)}{18\mu_c} \tag{10.7}$$

where d_d = droplet diameter, m,
u_d = settling (terminal) velocity of the dispersed phase droplets with diameter d, m/s,
ρ_c = density of the continuous phase, kg/m^3,
ρ_d = density of the dispersed phase, kg/m^3,
μ_c = viscosity of the continuous phase, N s/m^2,
g = gravitational acceleration, 9.81 m/s^2.

Equation 10.7 is used to calculate the settling velocity with an assumed droplet size of 150 μm, which is well below the droplet sizes normally found in decanter feeds. If the calculated settling velocity is greater than 4×10^{-3} m/s, then a figure of 4×10^{-3} m/s is used.

For a horizontal, cylindrical, decanter vessel, the interfacial area will depend on the position of the interface.



and

$$w = 2(2rz - z^2)^{1/2}$$

where w = width of the interface, m,
z = height of the interface from the base of the vessel, m,
l = length of the cylinder, m,
r = radius of the cylinder, m.

For a vertical, cylindrical decanter:

$$A_i = \pi r^2$$

The position of the interface should be such that the band of droplets that collect at the interface waiting to coalesce and cross the interface does not extend to the bottom (or top) of the vessel. Ryon *et al.* (1959) and Mizrahi and Barnea (1973) have shown that the depth of the dispersion band is a function of the liquid flow rate and the interfacial area. A value of 10 per cent of the decanter height is usually taken for design purposes. If the performance of the decanter is likely to be critical the design can be investigated using scale models. The model should be scaled to operate at the same Reynolds number as the proposed design, so that the effect of turbulence can be investigated; see Hooper (1975).

Example 10.3

Design a decanter to separate a light oil from water.

The oil is the dispersed phase.

Oil, flow rate 1000 kg/h, density 900 kg/m^3, viscosity 3 mN s/m^2.

Water, flow rate 5000 kg/h, density 1000 kg/m^3, viscosity 1 mN s/m^2.

Solution

Take $d_d = 150\ \mu$m

$$u_d = \frac{(150 \times 10^{-6})^2\, 9.81(900 - 1000)}{18 \times 1 \times 10^{-3}} \tag{10.7}$$

$$= -0.0012 \text{ m/s}, -1.2 \text{ mm/s (rising)}$$

As the flow rate is small, use a vertical, cylindrical vessel.

$$L_c = \frac{5000}{1000} \times \frac{1}{3600} = 1.39 \times 10^{-3} \text{ m}^3/\text{s}$$

$$u_c \not> u_d, \text{ and } u_c = \frac{L_c}{A_i}$$

hence
$$A_i = \frac{1.39 \times 10^{-3}}{0.0012} = 1.16 \text{ m}^2$$
$$r = \sqrt{\frac{1.16}{\pi}} = 0.61 \text{ m}$$
$$\text{diameter} = \underline{\underline{1.2 \text{ m}}}$$

Take the height as twice the diameter, a reasonable value for a cylinder:

$$\text{height} = \underline{\underline{2.4 \text{ m}}}$$

Take the dispersion band as 10 per cent of the height = $\underline{\underline{0.24 \text{ m}}}$

Check the residence time of the droplets in the dispersion band

$$= \frac{0.24}{u_d} = \frac{0.24}{0.0012} = 200 \text{ s } (\sim 3 \text{ min})$$

This is satisfactory, a time of 2 to 5 min is normally recommended. Check the size of the water (continuous, heavy phase) droplets that could be entrained with the oil (light phase).

$$\text{Velocity of oil phase} = \frac{1000}{900} \times \frac{1}{3600} \times \frac{1}{1.16}$$
$$= 2.7 \times 10^{-4} \text{ m/s } (0.27 \text{ mm/s})$$

From equation 10.7

$$d_d = \left[\frac{u_d 18 \mu_c}{g(\rho_d - \rho_c)}\right]^{1/2}$$

so the entrained droplet size will

$$= \left[\frac{2.7 \times 10^{-4} \times 18 \times 3 \times 10^{-3}}{9.81(1000 - 900)}\right]^{1/2}$$
$$= \underline{\underline{1.2 \times 10^{-4} \text{ m}}} = 120 \ \mu\text{m}$$

which is satisfactory; below 150 μm.

Piping arrangement

To minimise entrainment by the jet of liquid entering the vessel, the inlet velocity for a decanter should keep below 1 m/s.

$$\text{Flow-rate} = \left[\frac{1000}{900} + \frac{5000}{1000}\right] \frac{1}{3600} = 1.7 \times 10^{-3} \text{ m}^3/\text{s}$$

$$\text{Area of pipe} = \frac{1.7 \times 10^{-3}}{1} = 1.7 \times 10^{-3} \text{ m}^2$$

$$\text{Pipe diameter} = \sqrt{\frac{1.7 \times 10^{-3} \times 4}{\pi}} = 0.047 \text{ m, say } \underline{\underline{50 \text{ mm}}}$$

Take the position of the interface as half-way up the vessel and the light liquid off-take as at 90 per cent of the vessel height, then

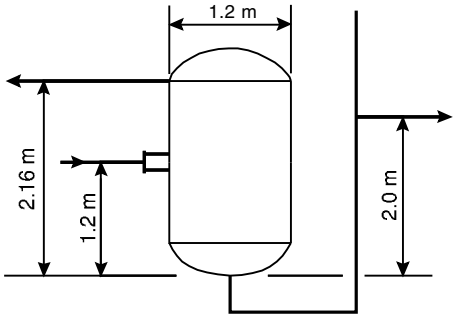
$$z_1 = 0.9 \times 2.4 = 2.16 \text{ m}$$

$$z_3 = 0.5 \times 2.4 = 1.2 \text{ m}$$

$$z_2 = \frac{(2.16 - 1.2)}{1000} \times 900 + 1.2 = \underline{2.06 \text{ m}} \tag{10.5}$$

say $\underline{2.0 \text{ m}}$

Proposed design



Drain valves should be fitted at the interface so that any tendency for an emulsion to form can be checked; and the emulsion accumulating at the interface drained off periodically as necessary.

10.6.2. Plate separators

Stacks of horizontal, parallel, plates are used in some proprietary decanter designs to increase the interfacial area per unit volume and to reduce turbulence. They, in effect, convert the decanter volume into several smaller separators connected in parallel.

10.6.3. Coalescers

Proprietary equipment, in which the dispersion is forced through some form of coalescing medium, is often used for the coalescence and separation of finely dispersed droplets. A medium is chosen that is preferentially wetted by the dispersed phase; knitted wire or plastic mesh, beds of fibrous material, or special membranes are used. The coalescing medium works by holding up the dispersed droplets long enough for them to form globlets of sufficient size to settle. A typical unit is shown in Figure 10.41; see Redmon (1963). Coalescing filters are suitable for separating small quantities of dispersed liquids from large throughputs.

Electrical coalescers, in which a high voltage field is used to break down the stabilising film surrounding the suspended droplets, are used for desalting crude oils and for similar applications; see Waterman (1965).

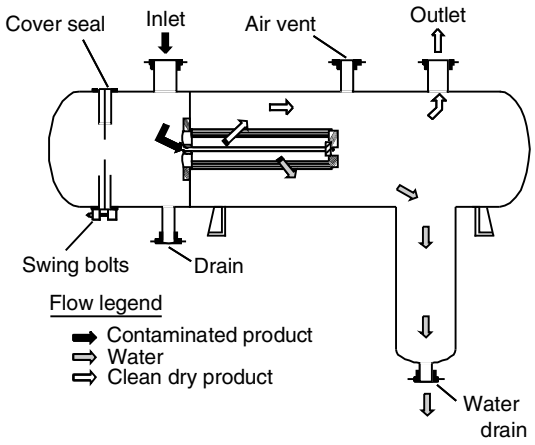


Figure 10.41. Typical coalescer design

10.6.4. Centrifugal separators

Sedimentation centrifuges

For difficult separations, where simple gravity settling is not satisfactory, sedimentation centrifuges should be considered. Centrifuging will give a cleaner separation than that obtainable by gravity settling. Centrifuges can be used where the difference in gravity between the liquids is very small, as low as 100 kg/m^3, and they can handle high throughputs, up to around 100 m^3/h. Also, centrifuging will usually break any emulsion that may form. Bowl or disc centrifuges are normally used (see Section 10.4.3).

Hydrocyclones

Hydrocyclones are used for some liquid-liquid separations, but are not so effective in this application as in separating solids from liquids.

10.7. SEPARATION OF DISSOLVED LIQUIDS

The most commonly used techniques for the separation and purification of miscible liquids are distillation and solvent extraction. In recent years, adsorption, ion exchange and chromatography have become practical alternatives to distillation or solvent extraction in many special applications.

Distillation is probably the most widely used separation technique in the chemical process industries, and is covered in Chapter 11 of this volume, and Chapter 11 of Volume 2. Solvent extraction and the associated technique, leaching (solid-liquid extraction) are covered in Volume 2, Chapters 13 and 10. Adsorption, which can be used for the separation of liquid and gases mixtures, is covered in Chapter 17 of Volume 2. Adsorption is also covered in the books by Suziki (1990) and Crittenden and Thomas (1998).