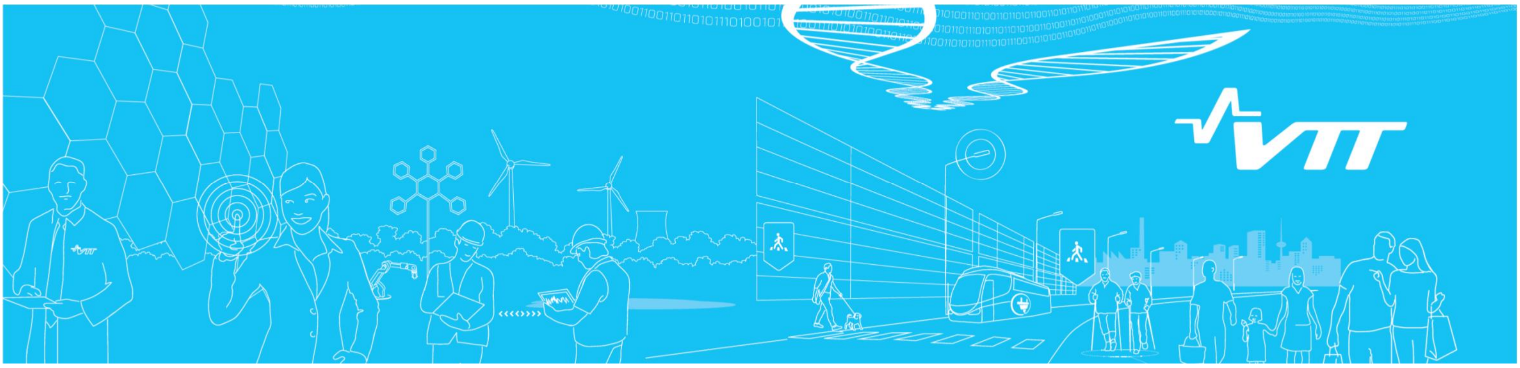


RESEARCH REPORT

VTT-R-06143-15

Design and selection of separation processes

Authors:	Lotta Sorsamäki, Marja Nappa
Confidentiality:	Public

Report's title

Design and selection of separation processes

Customer, contact person, address	**Order reference**
Project name ChemSep	**Project number/Short name** 104341
Author(s) Lotta Sorsamäki, Marja Nappa	**Pages** 68/
Keywords separation processes, economic evaluation, equipment cost estimation, modelling, Balas	**Report identification code** VTT-R-06143-15

Summary

Product separation and purification is costly but crucial step in chemical production concepts. Lots of information is needed in designing these systems, this is not however always available for first conceptual design. The main focus in this project was on developing competence and readiness to conduct reliably but rapidly conceptual techno-economic assessments of (bio)chemical production processes. The idea was to develop a rapid method and a generic "short-cut" model for evaluation of different alternative separation systems mainly for solid-liquid and liquid-liquid systems, but also for gas-liquid and gas-solid systems. The model was created with Balas® simulation software linked with MS Excel. The description of the model is presented in PowerPoint slides, *ChemSep – report slides*. This document, instead, acts as a theory source for the use of the model. In this document, the classification of separation processes into heterogeneous and homogeneous separation processes is presented, different unit operations of separation are shortly described and the possible application examples are listed. Heterogeneous separation processes that are described in detail include decantation, hydrocyclones, settling, sedimentation, flotation, centrifugal separation, filtration, wet scrubbers, leaching/washing and solid drying. Homogeneous separation processes that are described in detail include flash vaporization, distillation, drying of solutions, evaporation, crystallization and membrane technology. The operational principles, equipment types, selection of appropriate equipment and applications for each above mentioned separation process are described. The theory of the calculation of mass and energy balance in each unit operation are excluded. It can easily be found in the literature. Also, a short chapter of criteria affecting the selection of a suitable separation process for the mixture in question is presented.

Confidentiality	Public

Jyväskylä/Espoo 18.12.2015

Written by	**Reviewed by**	**Accepted by**
Lotta Sorsamäki, Marja Nappa	Eemeli Hytönen	Tuulamari Helaja
Research Scientists	Research Team Leader	Head of Research Area

VTT's contact address

P.O. Box 1000, 02044 VTT, Finland

Distribution (customer and VTT)

VTT

Preface

This report has been written in the own founded project *ChemSep - Rapid process design method for (bio)chemicals separation* during autumn 2015. The main focus in this project was on developing competence and readiness to conduct reliably but rapidly conceptual techno-economic assessments of novel biochemical production processes and waste water treatment systems. The results of the project include; (i) a model created with Balas® simulation software linked with MS Excel, (ii) this report, (iii) report slides containing description of the project and description and instructions of the model and also (iv) an MS Excel file where among the others data obtained from discussions with team members and from earlier models are collected. This document, *Design and selection of separation processes*, acts as a theory source for the use of the model. In this document, the classification of separation processes into heterogeneous and homogeneous separation processes is presented, different unit operation of separation are shortly described and the possible application examples are listed. The document is written by Lotta Sorsamäki and Marja Nappa. Writers thank Eemeli Hytönen who provided insight and expertise that greatly assisted the research, and also the (team) colleagues from discussions and sharing their competence during project.

Jyväskylä/Espoo 18.12.2015

Lotta Sorsamäki and Marja Nappa

Contents

1. Introduction

Product separation and purification is costly but crucial step in chemical production concepts. Many technologies for separating different liquid-phase (bio)chemical compounds from their reaction media or waste water exist (e.g. solvent extraction, crystallization, evaporation, distillation, membranes, phase separations, adsorption, chromatographic separation). Lots of information is needed in designing these systems, this is not however always available for first conceptual design.

This report is a result from the project *ChemSep - Rapid process design method for (bio)chemicals separation*. The main focus in this project was on developing competence and readiness to conduct reliably but rapidly conceptual techno-economic assessments of novel biochemical production processes and waste water treatment systems. The idea of this study was to develop a rapid method and a generic “short-cut” model for evaluation of different alternative separation systems mainly for solid-liquid and liquid-liquid systems, but also for gas-liquid and gas-solid systems. The created model will give technical specifications and economics of the systems for comparative and order-of-magnitude feasibility evaluations. It will also guide the experimental research to measure critical parameters needed in designing the product purification step. The method will give a systematic approach and enable faster assessment with reliable ranking of separation alternatives already at early technology development stages.

The model was created with Balas® simulation software linked with MS Excel. The name of the model-file is *ChemSepModelTool*. The description and instructions of the model are presented in PowerPoint slides, *ChemSep – report slides*. This document, instead, acts as a theory source for the use of the model. In this document, the classification of separation processes into heterogeneous and homogeneous separation processes is presented, different unit operation of separations are shortly described and the possible application examples are listed.

The separation methods are divided according the nature of the mixture to be treated. There are two kinds of mixtures: heterogeneous and homogeneous. In heterogeneous mixtures, two or more phases mix, but remain physically separate. Heterogeneous mixtures can include mixtures consisting of (i) two liquids, (ii) a solid and a liquid, (iii) a liquid and a gas, or even (iv) a gas and a solid. In a homogeneous mixture, the constituent phases are indistinguishable, having merged into a single uniform phase, with its original parts undetectable even at very high magnification. Solutions have only one phase and the most common solutions are liquids, although there can be solutions of gases (air) and solids (alloys).

In Chapter 3, separation technologies for heterogeneous mixtures are described. Only the basic principles of the process, equipment types and the selection of equipment are included. The theory of solving the mass and energy balances or dimensioning the equipment is excluded and can be found in literature. Good sources are for example Towler and Sinnott 2013 and Seader et al. 2011. The separation of heterogeneous mixtures is accomplished using mechanical-physical forces including gravitational and centrifugal. Principal method for the separation of heterogeneous liquid-liquid emulsions is decantation or hydrocyclones. For gas-solid and gas-liquid separation (i.e. gas cleaning), gravity settling, centrifugation, filtering, washing, and electrostatic precipitation may be used. The need to separate solid and liquid phases (i.e. suspension) is probably the most common separation requirement in the process industries, and many techniques are used (sedimentation, centrifugation, filtration). If the dry content of the solid-liquid heterogeneous mixture is remarkable high (the mixture is not anymore pumpable), it is justified to name it as wet solids rather than a suspension. In this case, leaching, washing and drying are the most used separation processes.

In Chapter 4 , separation of homogeneous fluid mixtures is described. There are five basic separation methods for separating homogeneous mixtures. The most common technic creates a second phase, immiscible with the feed phase (examples distillation, crystallization). A second technique adds another fluid phase, which selectively absorbs, extracts, or strips certain species from the feed (examples liquid-liquid extraction, absorption). Less common is the use of a barrier, usually a polymer membrane, which involves a gas or liquid feed and exploits differences in species permeabilities through the barrier (examples ultrafiltration, reverse osmosis). Techniques which involve contacting a vapour or liquid feed with a solid agent are also of growing importance (examples adsorption). Finally, external field (centrifugal, thermal, electrical, flow, etc.), are applied in specialized cases to gas or liquid feeds (examples electrophoresis). In most cases, the separation in one step is not perfect and if the feed contains more than two species, two or more separation operations may be required.

Since there is a wide range of different separation technologies for treating homogeneous mixtures, only a few were possible to focus in the ChemSep -project. Those technologies are described in detail in this document. In Chapter 5, flash, distillation, evaporation, drying of solutions, extraction, crystallization and membrane processes are described. Only the basic principles of the process, equipment types and the selection of equipment are included. The theory of solving the mass and energy balances or dimensioning the equipment is excluded and can be found in literature. Good sources are for example Towler and Sinnott 2013 and Seader et al. 2011.

In Chapter 6, some guidelines for choosing an appropriate separation method are given based on criteria such as phase and stream conditions, molecular, thermodynamic and transport properties, technological maturity and economics. Ultimately, the process having the lowest operating, maintenance, and capital costs is selected, provided it is controllable, safe, non-polluting, and capable of producing products that meet specifications.

In Chapter 7, some aspects of separation of bio-components are listed. In Chapter 8, two manuals of commercial simulation software, namely SuperProDesigner and Aspen Plus, have been gone through in order to get the idea how different separations processes are modelled in them.

2. Basic separation techniques

The creation of a mixture of chemical species from the separate species is a spontaneous process that requires no energy input. The inverse process, separation of a chemical mixture into pure components, is not a spontaneous process and thus requires energy. (Seader et al., 2011).

There are two kinds of mixtures: heterogeneous and homogeneous. In heterogeneous mixtures, two or more phases intermingle, but remain physically separate. Heterogeneous mixtures can include mixtures consisting of (i) two liquids, (ii) a solid and a liquid, (iii) a liquid and a gas, or even (iv) a gas and a solid. Liquid/liquid mixtures are referred to as emulsions, solid/liquid mixtures are suspensions, gas/liquids are aerosols, and gas/solids are referred to as smoke.

In a homogeneous mixture, the constituent phases are indistinguishable, having merged into a single uniform phase, with its original parts undetectable even at very high magnification. Solutions have only one phase and the most common solutions are liquids, although there can be solutions of gases (air) and solids (alloys). All solutions are classified as homogeneous mixtures.

If a mixture to be separated is heterogeneous or a multiphase mixture, then separation can be done physically by exploting differences in density between the phases. The separation is not accomplished on a molecular scale nor is it due to the differences among the various molecules. Instead, the separation will be accomplished using mechanical-physical forces and not molecular or chemical forces and diffusion. (Geankoplis, 1993).

Separation of the different phases of a heterogeneous mixture should be carried out before homogeneous separation, taking advantage of what already exists. Phase separation tends to be easier and should be done first. (Smith, 1995).

If a mixture to be separated is homogeneous, a separation can only be performed by the addition or creation of another phase within the system (Smith, 1995). The two phases are brought into more or less intimate contact with each other so that a solute or solutes can diffuse from one to the other. In gas-liquid and vapour-liquid separation processes (e.g. absorption, distillation) the separation depends on molecules diffusing or vaporizing from one distinct phase to another phase, i.e. on mass transfer of the molecules. In liquid-liquid separation processes (e.g. extraction), the two phases are quite different chemically, which leads to a separation on a molecular scale according to physical-chemical properties. In adsorption and membrane separation processes, the differences in the physical-chemical properties of the molecules lead to separation on a molecular scale. (Geankoplis, 1993).

3. Separation of heterogeneous mixtures

3.1 Introduction

In heterogeneous mixtures, two or more phases intermingle, but remain physically separate. Heterogeneous mixtures most often include mixtures consisting of (i) two liquids (i.e. emulsions) or (ii) a solid and a liquid (suspensions). Also liquid-gas mixtures and gas-solid mixtures exists, i.e. in gas cleaning the dispersed finely divided solids (dust) and liquid mists are removed from gas streams (Towler and Sinnott, 2013).

The separation of heterogeneous mixtures is accomplished using mechanical-physical forces; these forces will be acting on particles, liquids, or mixtures of particles and liquids themselves and not necessarily on the individual molecules. The mechanical-physical forces include gravitational and centrifugal, actual mechanical and kinetic forces arising from flow.

Particles and/or fluid streams are separated because of different effects produced on them by these forces. (Geankoplis, 1993).

Principal method for the separation of heterogeneous liquid-liquid emulsions is decantation or hydrocyclones.

The principal types of equipment for gas-solid and gas-liquid separation (i.e. gas cleaning) can be classified according to the mechanism employed to separate the solid particles or liquid mists: gravity settling, impingement, centrifugal force, filtering, washing, and electrostatic precipitation. (Towler and Sinnott, 2013). Only gravity settling, centrifugal separation and washing with wet scrubbers are described in detail below.

The need to separate solid and liquid phases is probably the most common separation requirement in the process industries, and many techniques are used (Figure 1). The most suitable technique will depend on the solids concentration and the 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 2. The choice of equipment also depends 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. (Towler and Sinnott, 2013).

If the dry content of the solid-liquid heterogeneous mixture is remarkable high (the mixture is not anymore pumpable), it is justified to name it as wet solids rather than a suspension. In this case, leaching, washing and drying are the most used separation processes. Leaching and washing are used to separate one solid from another or to separate the liquid bond to the solid. The processes differ from each other in defining the end product. In solid-liquid leaching, the soluble solid is the product, whereas in washing, the aim is the remove impurities from the solid leaving the solid as the main product. In drying, the aim is to remove liquid from the wet solid.

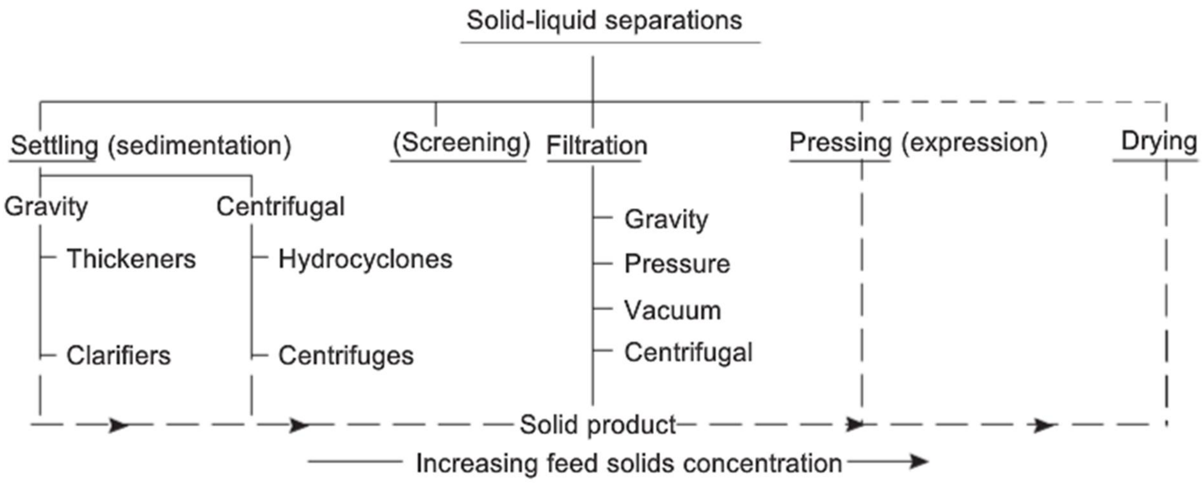


Figure 1. Solid-liquid separation technigues (Towler and Sinnott, 2013).

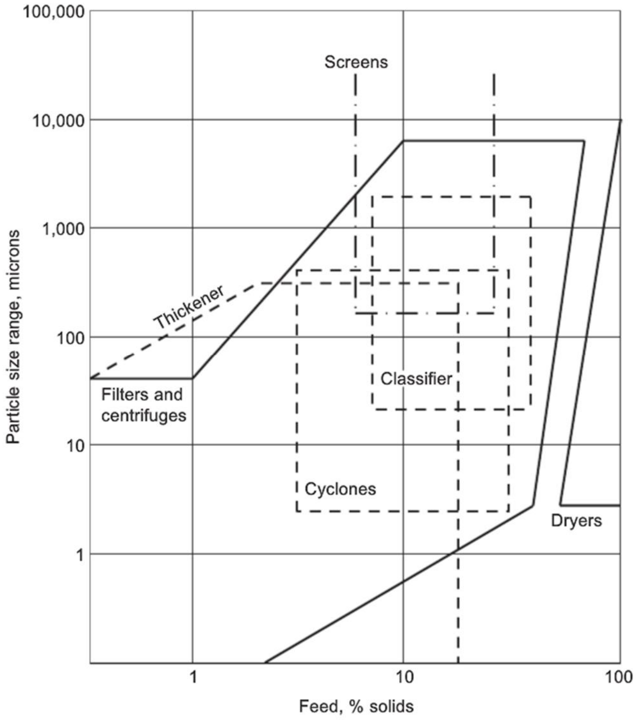
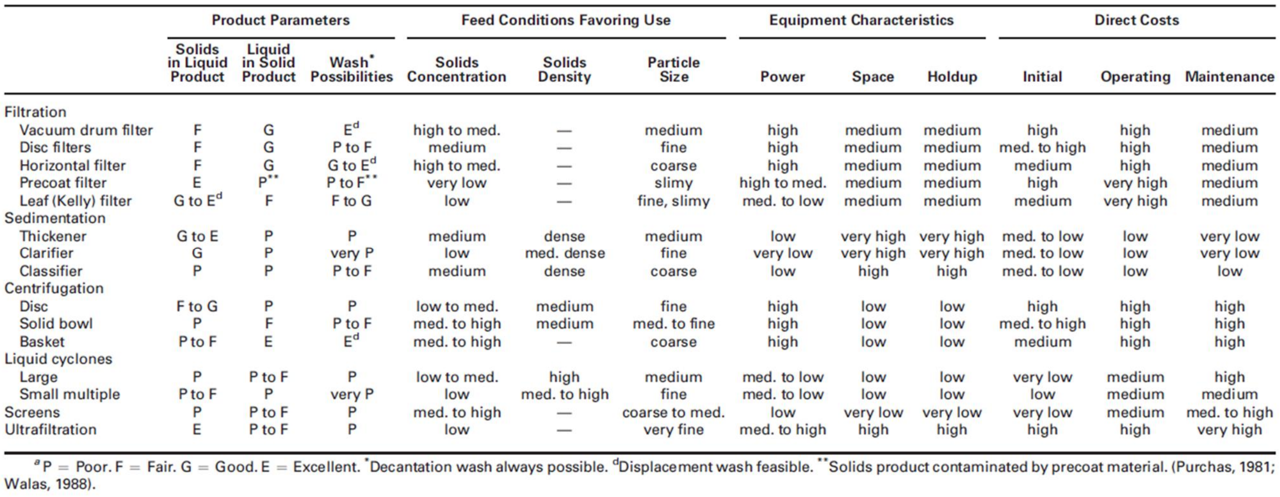


Figure 2. Solid-liquid separation techniques as a function of slurry concentration and particle size (Towler and Sinnott, 2013).

Characteristics of the performance of the main types of commercial solid-liquid separation equipment are summarized in Table 1

Table 1. Comparative performance of solid-liquid separation equipment (Couper et al., 2010).



	Product Parameters			Feed Conditions Favoring Use			Equipment Characteristics			Direct Costs		
	Solids in Liquid Product	Liquid in Solid Product	Wash[*] Possibilities	Solids Concentration	Solids Density	Particle Size	Power	Space	Holdup	Initial	Operating	Maintenance
Filtration												
Vacuum drum filter	F	G	E[d]	high to med.	—	medium	high	medium	medium	high	high	medium
Disc filters	F	G	P to F	medium	—	fine	high	medium	medium	med. to high	high	medium
Horizontal filter	F	G	G to E[d]	high to med.	—	coarse	high	medium	medium	medium	high	medium
Precoat filter	E	P[**]	P to F[**]	very low	—	slimy	high to med.	medium	medium	high	very high	medium
Leaf (Kelly) filter	G to E[d]	F	F to G	low	—	fine, slimy	med. to low	medium	medium	medium	very high	medium
Sedimentation												
Thickener	G to E	P	P	medium	dense	medium	low	very high	very high	med. to low	low	very low
Clarifier	G	P	very P	low	med. dense	fine	very low	very high	very high	med. to low	low	very low
Classifier	P	P	P to F	medium	dense	coarse	low	high	high	med. to low	low	low
Centrifugation												
Disc	F to G	P	P	low to med.	medium	fine	high	low	low	high	high	high
Solid bowl	P	F	P to F	med. to high	medium	med. to fine	high	low	low	med. to high	high	high
Basket	P to F	E	E[d]	med. to high	—	coarse	high	low	low	medium	high	high
Liquid cyclones												
Large	P	P to F	P	low to med.	high	medium	med. to low	low	low	very low	medium	high
Small multiple	P to F	P	very P	low	med. to high	fine	med. to low	low	low	low	medium	medium
Screens	P	P to F	P	med. to high	—	coarse to med.	low	very low	very low	very low	medium	med. to high
Ultrafiltration	E	P to F	P	low	—	very fine	med. to high	high	high	high	high	very high

[a] P = Poor. F = Fair. G = Good. E = Excellent. [*]Decantation wash always possible. [d]Displacement wash feasible. [**]Solids product contaminated by precoat material. (Purchas, 1981; Walas, 1988).

Separation-device selection of mechanical separation is based largely on the size of the particles carried by the fluid. Other considerations such as density, viscosity, particle concentration, and flow rate also affect the selection process, as do particle and fluid financial value and the device particle-capture efficacy and cost, but they are secondary. Typical particle sizes of possible feed streams and also equipment with particle size ranges are listed in Table 2. However these values may differ considerably and can hold only as preliminary guidelines (Seader et al., 2011).

Table 2. Typical particle sizes and particle size ranges for mechanical separation equipment (Seader et al., 2011).

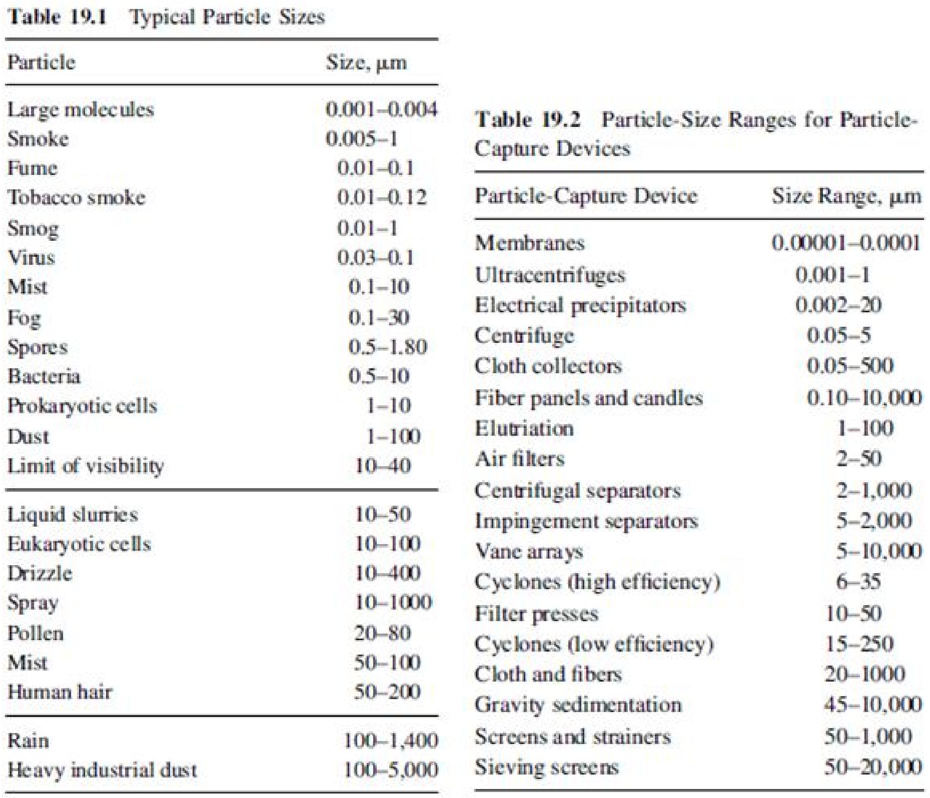


Table 19.1 Typical Particle Sizes

Particle	Size, µm
Large molecules	0.001–0.004
Smoke	0.005–1
Fume	0.01–0.1
Tobacco smoke	0.01–0.12
Smog	0.01–1
Virus	0.03–0.1
Mist	0.1–10
Fog	0.1–30
Spores	0.5–1.80
Bacteria	0.5–10
Prokaryotic cells	1–10
Dust	1–100
Limit of visibility	10–40
Liquid slurries	10–50
Eukaryotic cells	10–100
Drizzle	10–400
Spray	10–1000
Pollen	20–80
Mist	50–100
Human hair	50–200
Rain	100–1,400
Heavy industrial dust	100–5,000

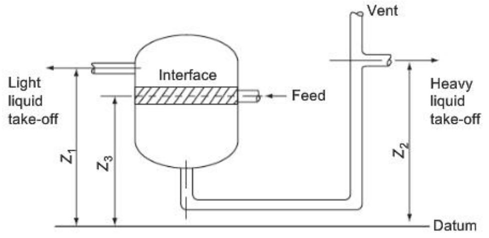
Table 19.2 Particle-Size Ranges for Particle-Capture Devices

Particle-Capture Device	Size Range, µm
Membranes	0.00001–0.0001
Ultracentrifuges	0.001–1
Electrical precipitators	0.002–20
Centrifuge	0.05–5
Cloth collectors	0.05–500
Fiber panels and candles	0.10–10,000
Elutriation	1–100
Air filters	2–50
Centrifugal separators	2–1,000
Impingement separators	5–2,000
Vane arrays	5–10,000
Cyclones (high efficiency)	6–35
Filter presses	10–50
Cyclones (low efficiency)	15–250
Cloth and fibers	20–1000
Gravity sedimentation	45–10,000
Screens and strainers	50–1,000
Sieving screens	50–20,000

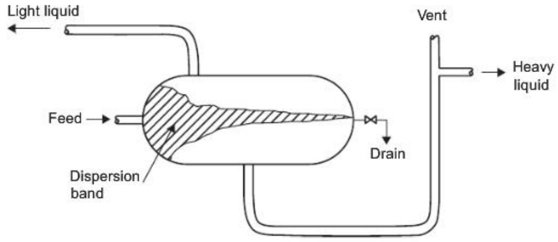
3.2 Decantation

The simplest form of equipment used to separate two immiscible liquid phases is the gravity settling tank, the decanter. 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 that 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: clear heavy liquid, separating dispersed liquid (the dispersion zone), and clear light liquid. (Towler and Sinnott, 2013). The purpose may be either to produce a clean decant, or to remove undesired liquid from other layers. If the aim is to produce a clean solution, a small amount of solution must generally be left in the container.

Decanters are normally designed for continuous operation. Many vessel shapes are used for decanters, but for most applications a cylindrical vessel will be suitable, and will be the cheapest shape. Typical designs are shown in Figure 3. 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 effectively convert the decanter volume into several smaller separators connected in parallel. (Towler and Sinnott, 2013).



Vertical decanter.



Horizontal decanter.

Figure 3. Typical design of decanters; a vertical decanter and a horizontal decanter (Towler and Sinnott, 2013).

3.3 Hydrocyclones

Liquid cyclonens (hydrocyclones) can be used for liquid-solid and liquid-liquid separation. Liquid cyclones can also be used for the classification of solid particles over a size range from 5 µm to100 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. (Towler and Sinnott, 2013).

Hydrocyclones for liquid-liquid separations are not as effective as in separating solids from liquids, as the high shear in a hydrocyclone can cause re-entrainment of droplets (Towler and Sinnott, 2013).

A hydrocyclone 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. They are often used in groups. A typical unit is shown in Figure 4. (Towler and Sinnott, 2013).

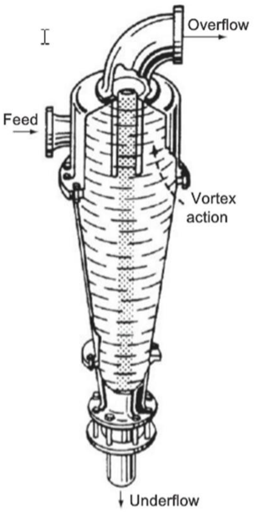


Figure 4. Liquid-solid cyclone (hydrocyclone) (Towler and Sinnott, 2013).

3.4 Settling

3.4.1 Introduction

In settling processes, particles are separated from a fluid by gravitational forces acting on the particles. The particles can be liquid drops or solid particles and the fluid can be a liquid or a gas, in other words settling may be used for emulsions (L-L), aerosols (L-G), suspensions (S-L) and smoke(S-G). (Smith, 1995). In some processes of settling the purpose is to remove the particles from the fluid stream so that the fluid is free of particle contaminants. In other processes the particles are recovered as the product. (Geankoplis, 1993).

3.4.2 Liquid-liquid settling

The simplest form of equipment used to separate two immiscible liquid phases is the gravity settling tank, the decanter. Decantation is described in Chapter 3.2.

3.4.3 Gas-solid settling

The primary need for gas-solids separation processes is for gas cleaning: the removal of dispersed finely divided solids from gas streams. Settling chambers are the simplest form of industrial gas-cleaning equipment, but have only a limited use; they are suitable for coarse dusts, particles larger than 50 µm. Settling chambers are essentially long, horizontal, rectangular chambers through which the gas flows. The solids settle under gravity and are removed from the bottom of the chamber. Horizontal plates or vertical baffles are used in some designs to improve the separation. Settling chambers offer little resistance to the gas flow, and can be designed for operation at high temperature and high pressure, and for use in corrosive atmospheres. (Towler and Sinnott, 2013).

3.4.4 Vapour-liquid settling

Settling by “flash” drum is used to separate a vapour-liquid mixture (aerosol) by gravity. (Smith, 1995).

3.4.5 Solid-liquid settling

Solid-liquid settling is referred as sedimentation, and described in Chapter 3.5.

3.5 Sedimentation

Sedimentation is the separation of suspended solid particles from a liquid mixture by gravity settling into a clear fluid and a slurry of higher solids content. Sedimentation can be done in a thickener, in a clarifier or in a classifier. (Smith, 1995). Since thickening and clarification are relatively cheap processes when used for the treatment of large volumes of liquid, they are used for pre-concentration of feeds to filtering.

The prime function in a thickener (Figure 5), is to produce a more concentrated slurry. Sedimentation rates are often assisted by addition of flocculating agents that neutralizes electric charges of the particles causing them to repel each other and remain dispersed. The prime function in a clarifier is to remove solids from a liquid to produce a clear liquid effluent rather than to produce a more concentrated solid-liquid mixture. In a classifier, the liquid-solid feed is divided into fraction according the particles sizes. (Smith, 1995).

A thickener, or clarifier, consists essentially of a large circular tank with a rotating rake at the base (Towler and Sinnott, 2013). In thickeners the slurry is introduced at the top centre, clear liquid overflows the top edge, whereas the solids settle out and are worked gradually towards the centre with slowly rotating rakes towards the discharge port at the bottom centre. The concentrated slurry then is suitable for filtering. Clarifiers are similar devices, primarily for recovering clear liquids from dilute suspensions. (Couper et al., 2010).

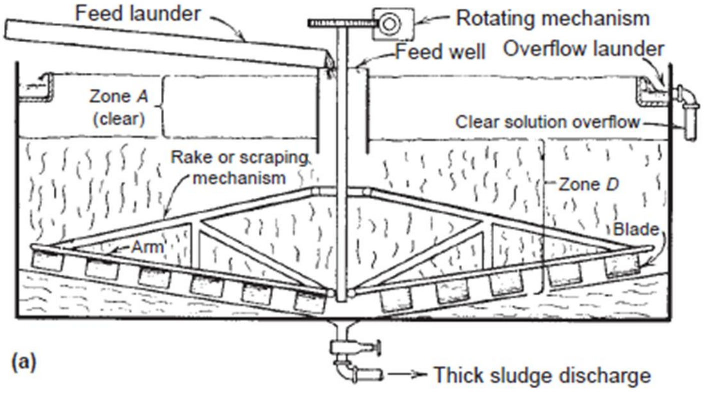


Figure 5. Thickeners for pre concentration of feed to filters (Couper et al., 2010).

3.6 Flotation

Flotation is a gravity separation process which exploits differences in the surface properties of particles. Gas bubbles are generated in a liquid. The bubbles become attached to solid particles or immiscible liquid droplets causing the particles or droplets to rise to the surface where they accumulate as a floating sludge. Flotation is used to separate mixtures of solid-solid particles and liquid-liquid mixture of finely divided immiscible droplets. (Smith, 1995).

Attachment of the bubbles to solid particles relies either on the surface properties of the solid particle or the nature of agglomerates or flocs. Bubble-particle attachment may be spontaneous or may require some form of chemical addition (chemical additives). (Purchas and Wakeman, 1986).

There are two dominant flotation techniques; dissolved air flotation and electrolytic flotation. Dissolved air flotation may be used for clarification of water or for thickening or concentration of solids suspensions. The central idea of the process is the generation of fine bubbles in the presence of suspended matter under conditions which enable the bubbles to intercept the suspended matter and carry it to the surface as a floating sludge carpet. In some cases the solids may already be capable of being captured, in other cases coagulation and flocculation may be needed. The quantity of air required for flotation depends upon the solids loading; normally air is dissolved to about 90 % saturation at the selected working pressure. (Purchas and Wakeman, 1986).

In electro-flotation, the bubbles of gas capture flocculated or destabilised particles in suspension. The gas is generated within the flotation chamber by direct electrolysis of the aqueous stream being treated. Electro-flotation appears to be used mainly in effluent treatment without a pre-flocculation stage. Air flotation process is a co-current contact process whereas electro-flotation is usually counter-current. (Purchas and Wakeman, 1986).

Comparing electrolyte flotation with dissolved air flotation, the former has the advantage that no pressurized recycle is required: the flow treated by the electro-flotation unit comprises the waste-water feed only. The recycle system employed for the dissolved air flotation increases the flow into the unit somewhat increasing the unit area. In electrolytic flotation there is no need for the recycle pumps, compressors or saturators as in dissolved air systems, however, the electro-flotation process requires expensive transformer-rectifier systems which may exceed the cost of the flotation unit itself. (Purchas and Wakeman, 1986).

3.7 Centrifugal separation

3.7.1 Introduction

Centrifugal separation is used when gravity separation (settling, sedimentation or flotation) is too slow and the particles do not settle readily or at all (Smith, 1995). Gravity separation may be too slow because of the closeness of the densities of the particle and the fluid, or because of association forces holding the components together, as in emulsions (Geankoplis, 1993).

In centrifugal separation processes, the separation of particles from a fluid is accomplished by centrifugal forces acting on the particles. Centrifugal separators make use of the common principle that an object whirled about an axis at a constant radial distance from the point is acted on by a force. (Geankoplis, 1993).

3.7.2 Gas-solid separation

Cyclones are the principal type of gas-solids separator employing centrifugal force, and are widely used. They are simple constructions, can be made from a wide range of materials, and can be designed for high temperature and pressure operation. Cyclones are suitable for separating particles above about 5 µm diameter; smaller particles, down to about 0.5 µm, can be separated when agglomeration occurs. The most commonly used design is the reverse-flow cyclone (Figure 6). In a reverse-flow cyclone the gas enters the top chamber tangentially and spirals down to the apex of the conical section; it then moves upward in a second, smaller diameter spiral, and exits at the top through a central vertical pipe. The solids move radially to the walls, slide down the walls, and are collected at the bottom. (Towler and Sinnott, 2013).

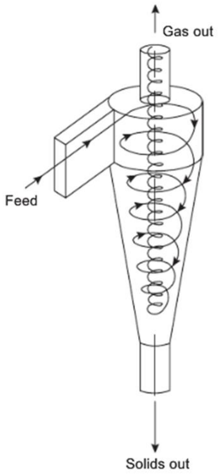


Figure 6. Reverse-flow cyclone (Towler and Sinnott, 2013).

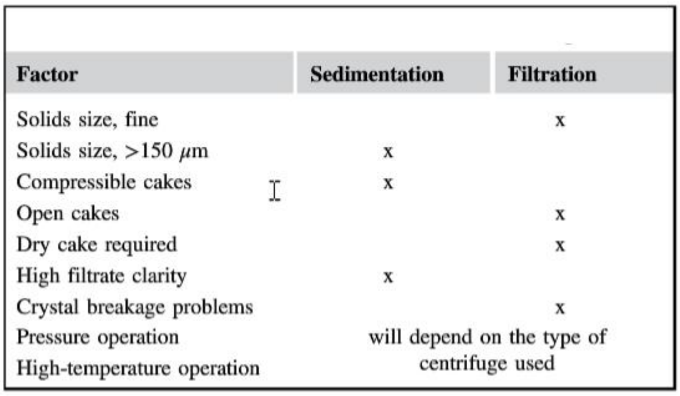
3.7.3 Liquid-solid separation

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

1. Sedimentation centrifuges: separation is dependent on a difference in density between the solid and liquid phases (solid heavier).
2. Filtration centrifuges: 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 summarized in Table 3. As a general rule, sedimentation centrifuges are used when a clarified liquid product is required, and filtration centrifuges to produce a pure, dry, solid product. (Towler and Sinnott, 2013).

Table 3. Selection of sedimentation or filter centrifuge (Towler and Sinnott, 2013).



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	will depend on the type of centrifuge used	
High-temperature operation		

Filtration centrifuges (or centrifugal filters) use centrifugal force to collect solids as a cake on a screen or cloth (Couper et al., 2010). They can be classified into two classes: 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. (Towler and Sinnott, 2013). Examples of filtration centrifuge units are basket, peeler, and pusher types as shown in Figure 7a and c (Couper et al., 2010). The simplest machines are the basket types (Towler and Sinnott, 2013).

The capacity of filtration centrifuges is very dependent on the solids concentration in the feed. For example, at 10% feed slurry concentration 9 kg of liquid will be centrifuged for every 1 kg of solids separated; whereas with a 50% 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, hydrocyclones, or cross-flow filtration. (Towler and Sinnott, 2013).

There are four main types of sedimentation centrifuge: tubular bowl, disc bowl, scroll discharge and solid bowl batch centrifuge. Tubular bowl centrifuges are used for the separation of immiscible liquids, such as water and oil, and for the separation of fine solids. Disc bowl centrifuges are used for separating liquids and fine solids, and for solids classification. Scroll dis-charge centrifuges can be designed so that solids can be washed and relatively dry solids can be discharged. The solid bowl batch centrifuge (Figure 7b) is the simplest type and similar to the tubular bowl machine. The tubular bowl type is rarely used for solids concentrations above 1% by volume. For concentrations from 1% to 15%, any of the other three types can be used. Above 15%, either the scroll discharge type or the batch type may be used, depending on whether continuous or intermittent operation is required. (Towler and Sinnott, 2013).

Centrifugal sedimentation in employed in many food industries, such as breweries, vegetable oil processing, fruit juice processing to remove cellular materials, and so on (Geankoplis, 1993).

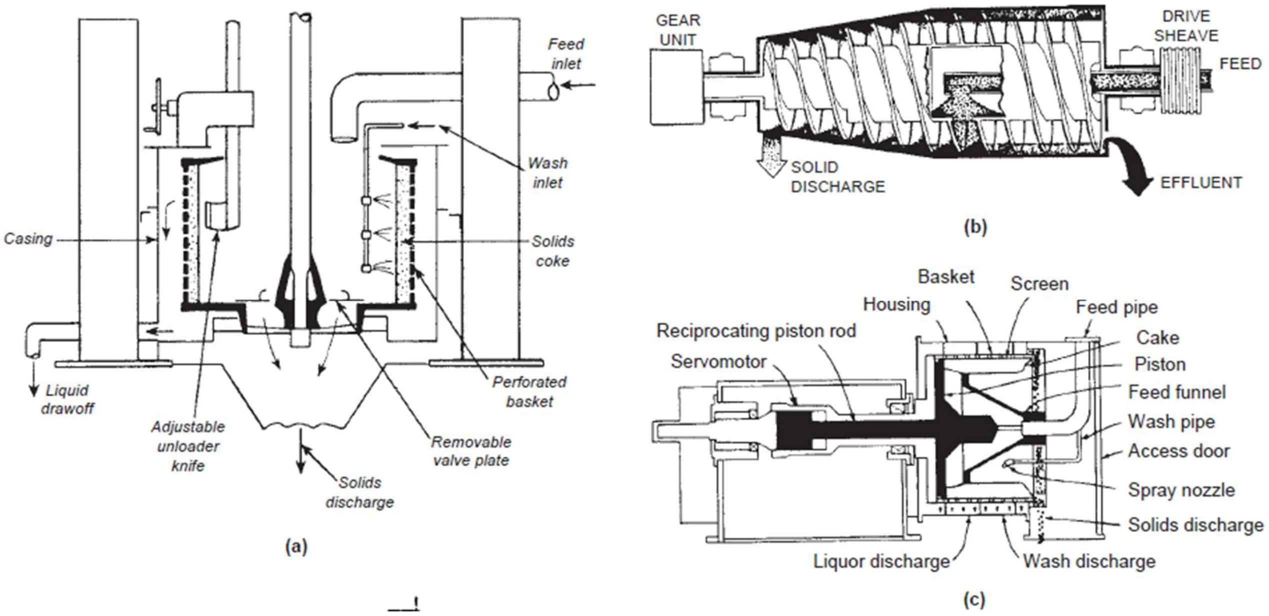


Figure 7. Centrifuges (a) top suspended batch centrifugal filter, (b) a solid bowl sedimentation centrifuge, (c) a pusher type centrifugal filter (Couper et al., 2010).

3.7.4 Liquid-liquid separation

Liquid-liquid immiscible mixtures can also be separated in the same centrifugal devices that are used for liquid-solid separations. For difficult separations, where simple gravity settling is not satisfactory, sedimentation centrifuges can be considered. Centrifuging will give a cleaner separation than that obtained by decanters. Centrifuges can be used where the difference in specific gravity between the liquids is very small, as low as 100 kg/m^3. Bowl or disc centrifuges are normally used. (Towler and Sinnott, 2013).

3.8 Filtration

In filtration, suspended solid particles in a liquid or gas are removed by passing the mixture through a porous medium that retains particles and passes the clear filtrate (Geankoplis, 1993). Filtration is performed on screens by gravity or on filters by vacuum, pressure or centrifugation (Couper et al., 2010).

The valuable product may be the clear filtrate from the filtration or the solid cake. (Geankoplis, 1993). The solid can be retained on the surface of the filter medium, which is cake filtration, or captured within the filter medium, which is depth filtration. In cake filtration, the filter medium can be a cloth of natural or artificial fibres or metal. (Smith, 1995). After filtering, the washing of the cake takes place by displacement of the filtrate and by diffusion (Geankoplis, 1993). In depth filtration, a granular medium consisting of layers of particulate solids causes the solid particles to be captured within the medium. To release the solid particles captured within the bed, the flow is periodically reversed. (Smith, 1995).

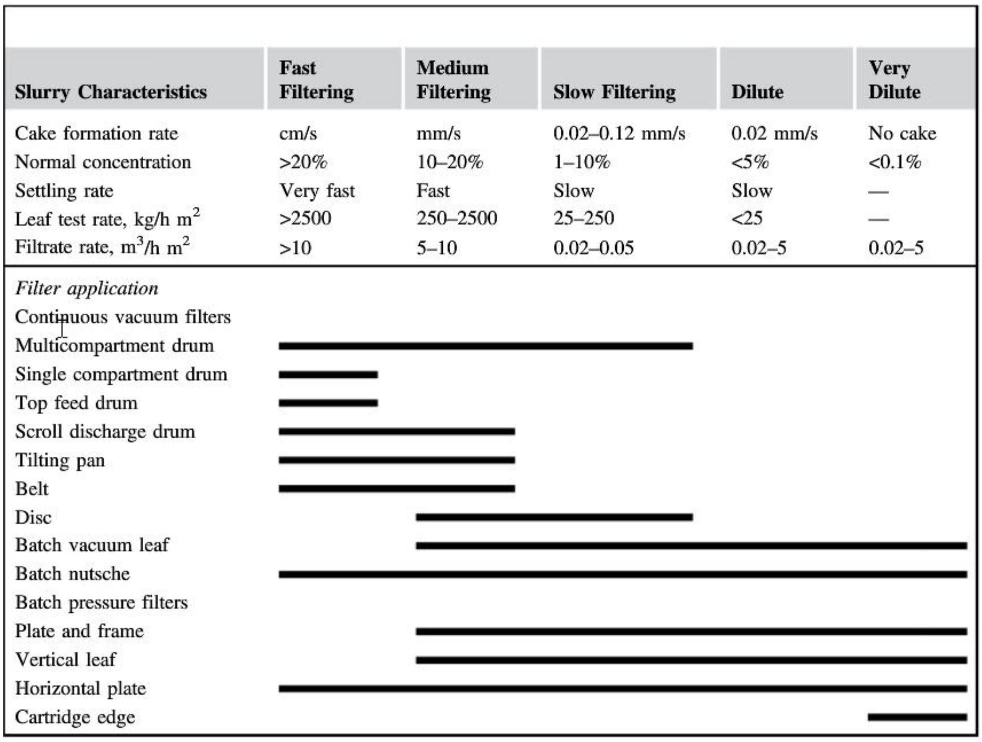
The most commonly used filter medium is woven cloth, but many other media are also used. Filter aids are often used to increase the rate of filtration of difficult slurries. They are either applied as a precoat to the filter cloth or added to the slurry and deposited with the solids, assisting in the formation of a porous cake. 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 and cross-flow 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. (Towler and Sinnott, 2013).

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 4. (Towler and Sinnott, 2013).

Table 4. Guide to filter selection (Towler and Sinnott, 2013).



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.

1. 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.
2. Plate and Frame Press (Pressure Operation) (Figure 8)
   These are the oldest and most commonly used batch filters. The equipment is versatile, made in a variety of materials, and capable of handling viscous liquids and cakes with a high specific resistance.
3. 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.

4. Rotary Drum Filters (Usually Vacuum Operation) (Figure 9)

   A drum filter consists essentially of a large hollow drum around which the filter medium is fitted. The drum is partially submerged in a trough of slurry, and the filtrate is sucked through the filter medium by vacuum inside the drum. Wash water can be sprayed on to the drum surface and multi-compartment 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.
5. 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.
6. Belt Filters (Vacuum Operation) (Figure 10)

   A belt filter consists of an endless reinforced rubber belt, with drainage holes along its center, 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.
7. Horizontal Pan Filters (Vacuum Operation)

   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.
8. Centrifugal Filters

   Centrifugal filters use centrifugal force to drive the filtrate through the filter cake.
9. Cross-Flow Filters

   Cross-flow filters are used to reject liquid from a slurry, similar to thickeners. The filter is arranged as tubular modules, and is often a porous membrane. The slurry is usually fed through the tubes and clear liquid is withdrawn through the tube wall as filtrate. The flow on the inside of the tube prevents the accumulation of solids and the concentrated slurry can then be sent to further processing. (Towler and Sinnott, 2013).

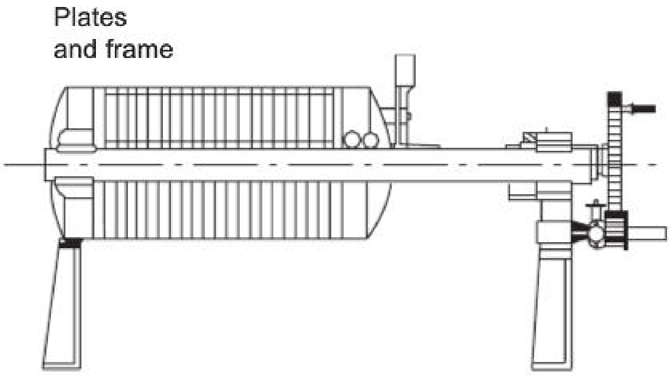


Figure 8. Plate and frame filter press (Towler and Sinnott, 2013).

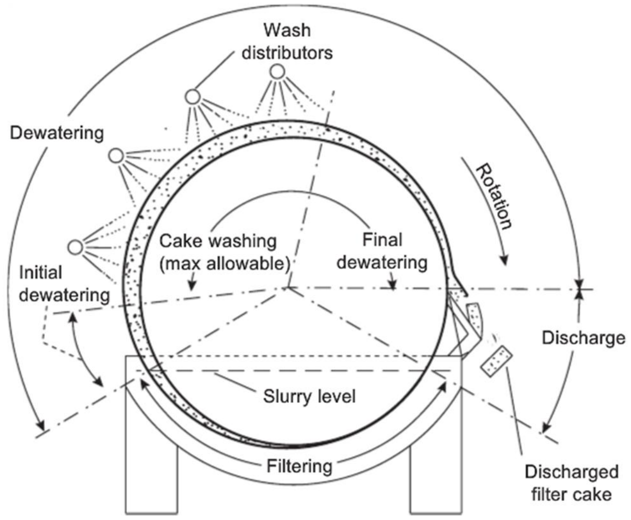


Figure 9. Drum filter (Towler and Sinnott, 2013).

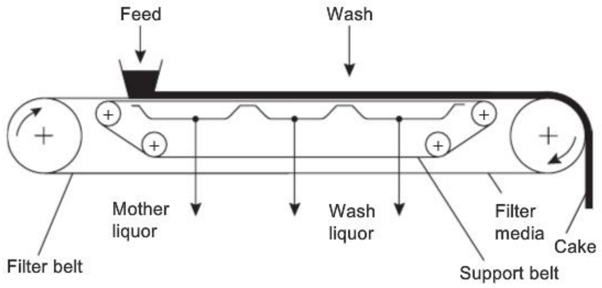


Figure 10. Belt filter (Towler and Sinnott, 2013).

3.9 Wet scrubbers

In wet scrubbing, the dust is removed from the gas stream by counter current washing with a liquid, usually water, and the solids are removed as a slurry. The principal mechanism involved is the impact (impingement) of the dust particles and the water droplets. Particle sizes down to 0.5 µm can be removed in suitably designed scrubbers. In addition to removing solids, wet scrubbers can be used to simultaneously cool the gas and neutralize any corrosive constituents. Spray towers and plate and packed columns are used. Spray towers have a low pressure drop but are not suitable for removing very fine particles, below 10 µm. The collecting efficiency can be improved by the use of plates or packing, but at the expense of a higher pressure drop. A packed or plate column for wet scrubbing would typically be designed with three to five plates or height equivalent to three theoretical stages of packing. Venturi and orifice scrubbers are simple forms of wet scrubbers. The turbulence created by the venturi or orifice is used to atomize water sprays and promote contact between the liquid droplets and dust particles. The agglomerated particles of dust and liquid are then collected in a centrifugal separator, usually a cyclone. (Towler and Sinnott, 2013).

3.10 Leaching/Washing

3.10.1 Introduction

Leaching, sometimes called solid–liquid (or liquid–solid) extraction, involves the removal of a soluble fraction (the solute or leachant) of a solid material by a liquid solvent. The solute diffuses from inside the solid into the surrounding solvent. Either the extracted solid fraction or the insoluble solids, or both, may be valuable products. Leaching is widely used in the metallurgical, natural product, and food industries. (Seader et al., 2011). In leaching, when an undesirable component is removed from a solid with water, the process is called washing. (Geankoplis, 1993).

Leachable solids generally undergo pre-treatment before being fed to leaching equipment so that reasonable leaching times are obtained. The particle size of the solid is usually reduced by hulling, cracking, flaking or cutting into slices, to promote rapid solute diffusion out of the solid and into the liquid solvent. The major difference between solid-liquid and liquid-liquid systems is the difficulty of transporting the solid (often as slurry or wet cake) from stage to stage. (Seader et al., 2011).

3.10.2 Equipment

3.10.2.1 Introduction

Industrial equipment for solid–liquid extraction is designed for batch wise, semi continuous or continuous processing. The method of contacting solids with solvent is either by percolation of solvent through a bed of solids or by immersion of the solid in the solvent followed by agitation of the mixture. When immersion is used, counter current, multistage operation is common. With percolation, either a stage wise or a differential contacting device is appropriate. An extractor must be efficient to minimize the need for solvent because of the high cost of solvent recovery. (Seader et al., 2011).

Effluents from a leaching stage are essentially solids-free liquid, called the overflow, and wet solids, the underflow. To reduce the concentration of solute in the liquid portion of the underflow, leaching is often accompanied by counter current flow washing stages. The combined process produces a final overflow, referred to as extract, which contains some of the solvent and most of the solute; and a final underflow, the extracted or leached solids, which are wet with almost pure solvent. Ideally, the soluble solids are perfectly separated from the insoluble solids, but solvent is distributed to both products. Therefore, additional processing of the extract and the leached solids is necessary to recover solvent for recycle. (Seader et al., 2011).

3.10.2.2 Batch extractors

When the solids to be leached are in the form of fine particles, perhaps smaller than 0.1 mm in diameter, batch leaching is conveniently conducted in an agitated vessel. The tank is a tall, cylindrical vessel constructed of wood, concrete, or metal that can be lined with an inert, noncorrosive, nontoxic material. Solvent and solids are placed in the tank and agitation is achieved by an air lift, whereby air bubbles entering at the bottom of a circular tube, concentric with the tank, cause upward flow and subsequent circulation of the solid–liquid suspension. During agitation, air continuously enters and leaves the vessel. When the desired degree of leaching is accomplished, agitation stops and solids are allowed to settle into a sludge at the bottom, where it is removed with the assistance of air. The supernatant extract is removed by siphoning from the top of the tank. (Seader et al., 2011).

When the solids are too coarse to be easily suspended by immersion in a stirred solvent, percolation techniques can be used. Again a tall, cylindrical vessel is employed. Solids to be leached are dumped into the vessel, followed by percolation of solvent down through the bed of solids. To achieve a high concentration of solute in the solvent, a series of vessels is arranged in a multibatch, counter current leaching technique. This technique can be used for such applications as batch removal of tannin from wood or bark, sugar from sugar beets, and water-soluble substances from coffee, tea, and spices. A typical vessel arrangement is shown in Figure 11, where Vessel 1 is off-line for emptying and refilling of solids. Solvent enters and percolates down through the solids in Vessel 2, and then percolates through Vessels 3 and 4, leaving as final extract from Vessel 4. The extraction of solids in Vessel 2 is completed first. When that occurs, Vessel 2 is taken off-line for emptying and refilling of solids and Vessel 1 is placed on-line. Fresh solvent first enters Vessel 3, followed by Vessels 4 and 1. In this manner, fresh solvent always contacts solids that have been leached for the longest time, thus realizing the benefits of counter current contacting. Heat exchangers are provided between vessels to adjust the liquid temperature, and pumps can be used to move liquid from vessel to vessel. Any number of vessels can be included in a battery. Note that although the system is batch wise with respect to solids, it is continuous with respect to solvent and extract. (Seader et al., 2011).

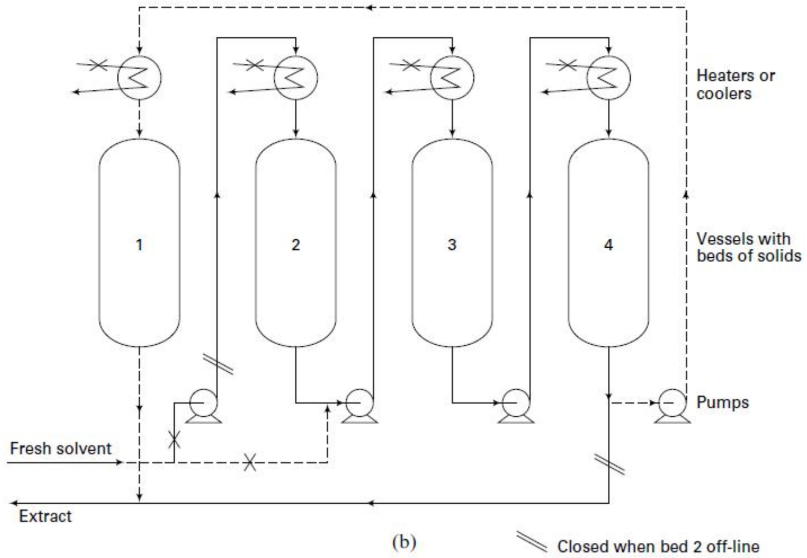
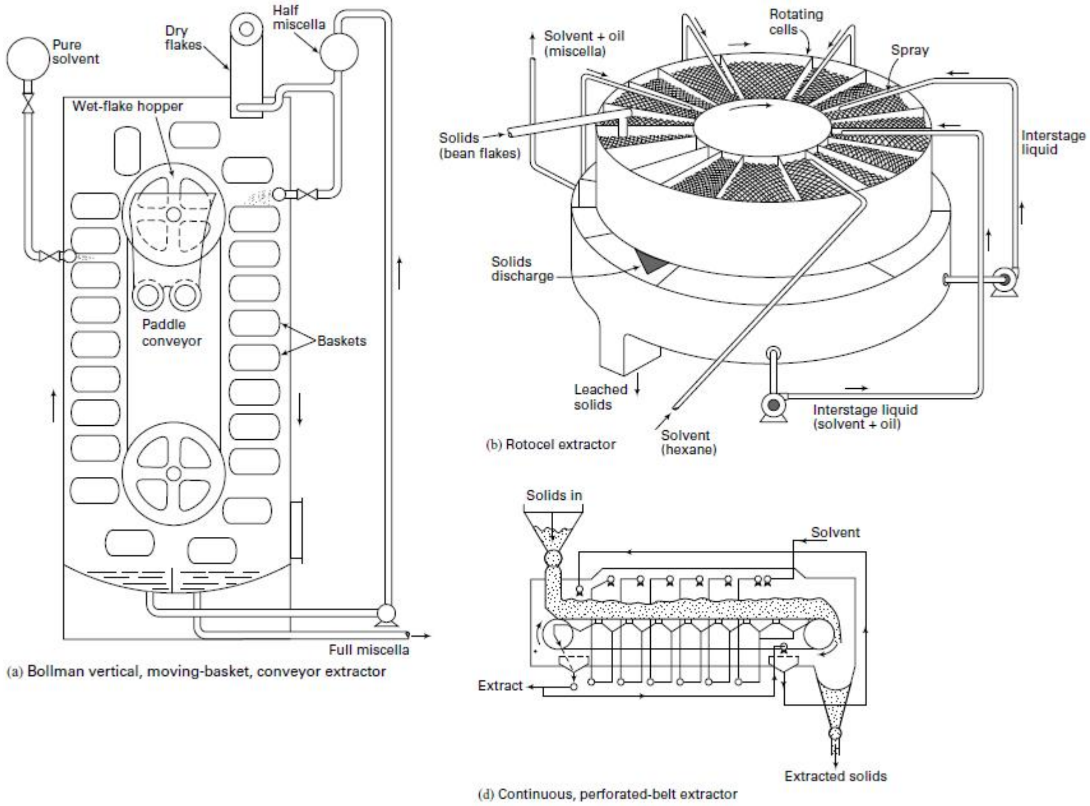


Figure 11. Counter current, multibatch battery system for leaching of large particles by percolation (Seader et al., 2011)

3.10.2.3 Continuous extractors

When leaching is carried out on a large scale, it is preferable to use an extraction device that operates with continuous flow of both solids and liquid. Many such patented devices are available, especially for the food industry. Some of the widely discussed extractors are shown schematically in Figure 12. These differ mainly with respect to the manner in which solids are transported and the degree to which agitation of solid–liquid mixtures is provided. (Seader et al., 2011).



(a) Bollman vertical, moving-basket, conveyor extractor

(b) Rotocel extractor

(d) Continuous, perforated-belt extractor

Figure 12. Equipment for continuous leaching (Seader et al., 2011).

3.10.2.4 Continuous, Counter current Washing

When leaching is very rapid, as with small particles containing very soluble solutes or when leaching has already been completed or when solids are formed by chemical reactions in a solution, it is common to counter currently wash the solids to reduce the solute concentration in the liquid adhering to the solids. This can be accomplished in a series of gravity thickeners (Figure 5) or centrifugal thickeners, called hydroclones (Figure 6), arranged for counter current flow of the underflows and overflows.

Residence times of solids and liquids in a gravity thickener are often large (minutes or hours) and, as such, are sufficient to provide adequate residence time for mass transfer and mixing when small particles are involved. When long residence times are not needed and the overflow need not be perfectly clear of solids, the hydroclone may be appropriate. (Seader et al., 2011).

3.11 Solid drying

3.11.1 Introduction

Most solid materials require drying at some stage in their production. Drying involves a combination of heat transfer and mass transfer. Heat is transferred to the surface of the solid to provide the heat of vaporization. The liquid evaporates and diffuses away from the surface into the bulk of the drying gas. Most dryers use a flowing gas to ensure that there is always an adequate partial pressure driving force for evaporation. Convective heat transfer is important in all dryers, although some designs also use conduction and radiation to increase the heating rate. (Towler and Sinnott, 2013).

Much of the terminology used in drying refers to air-water systems, as these are widely encountered in the food, paper, textile, and minerals-processing industries. The same concepts can be extended to other liquids or solvents and to other drying gases. The amount of drying that can be achieved depends on the nature of the solid and the moisture (or solvent) content of the inlet air (or gas). Nonporous solids such as sand can be dried to near zero moisture regardless of the inlet gas condition. Porous solids, cellular and fibrous materials, and hygroscopic solids will reach a moisture content that is in equilibrium with the humidity of the entering gas. Consequently, to reach the desired moisture content, the correct combination of inlet gas humidity, inlet gas temperature, and dryer residence time have to be carefully selected. The residual moisture remaining when the solids reach equilibrium with the enter-ng gas is referred to as the equilibrium moisture content. (Towler and Sinnott, 2013).

3.11.2 Equipment

The basic types of dryers used in the chemical process industry are tray, band, rotary, fluidized bed, pneumatic, drum, and spray dryers (Towler and Sinnott, 2013).

Batch tray dryers

Batch tray dryers (Figure 13a) are used for drying small quantities of solids, and are used for a wide range of materials. 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, and they are suitable for drying fine powders and valuable products. (Towler and Sinnott, 2013).

Conveyor dryer

In conveyor dryers i.e. continuous circulation band dryer (Figure 13b), the solids are fed on to an endless, perforated conveyor belt. Hot air or other drying gas is either passed over the solids on the belt or forced through the belt. The belt is housed in a long rectangular cabinet, which is divided up into zones. The relative movement through the dryer of the solids and drying air can be cocurrent, cross-flow, or, more usually, counter current. 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. (Towler and Sinnott, 2013).

Rotary dryer

In rotary dryers (Figure 13c), the solids are conveyed along the inside of a rotating, inclined cylinder and are heated and dried by direct contact with hot air or gases flowing through the cylinder. In some, the cylinders are indirectly heated. Most commonly, the drying gas is heated by a steam heater at the dryer inlet or is direct-fired with a fuel. Rotating dryers are suitable for drying free-flowing granular materials. They are suitable for continuous operation at high throughputs, and have a high thermal efficiency and relatively low capital cost and labour costs. Some disadvantages of this type are non-uniform residence time, dust generation, and high noise levels. The main cost of a rotary dryer is usually the heater for the air, as the shell is not usually pressure-retaining and so is not a high cost item. (Towler and Sinnott, 2013).

Fluidized-bed dryer

In fluidized-bed dryer (Figure 13d), the drying gas is passed through the bed of solids at a velocity sufficient to keep the bed in a fluidized state, which promotes high heat transfer and drying rates. Fluidized-bed dryers are suitable for granular and crystalline materials within the particle size range 0.5 mm to 3 mm. They are designed for continuous and batch operation. The main advantages of fluidized 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. Cyclones are usually incorporated on the exit gas to prevent formation of dust from fine particles that are elutriated by the drying gas. (Towler and Sinnott, 2013).

Pneumatic dryer

Pneumatic dryers (Figure 13e), 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 fluidized-bed dryer but which are heat sensitive and must be dried rapidly. The thermal efficiency of this type is generally low. (Towler and Sinnott, 2013).

Spray dryer

Spray dryers (Figure 13f) 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 atomized in a nozzle, or by a disc-type atomizer, positioned at the top of a vertical cylindrical vessel. Hot air flows up the vessel and conveys and dries the droplets. The liquid vaporizes 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. (Towler and Sinnott, 2013).

Rotary drum dryer

Rotary drum dryers (Figure 13g) 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. A drum dryer consists 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. (Towler and Sinnott, 2013).

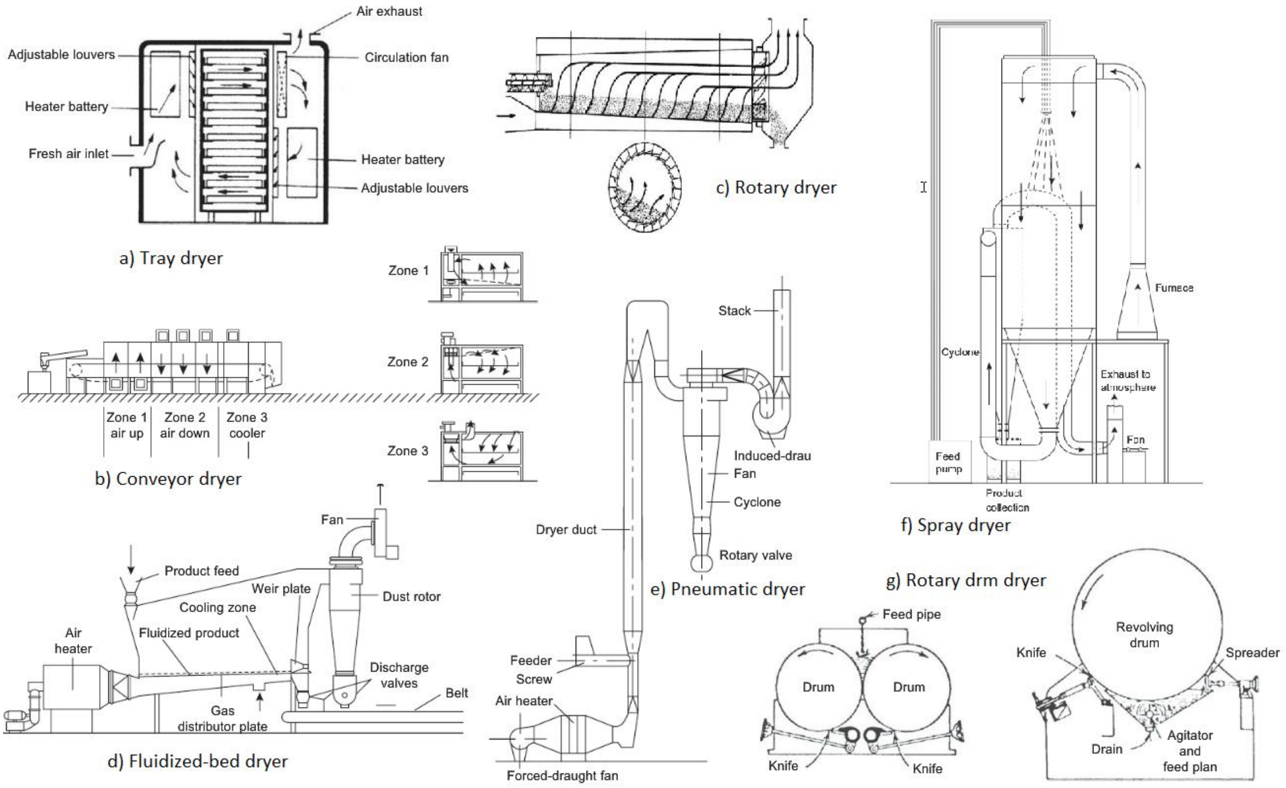


Figure 13. Solid drying equipment (adopted from Towler and Sinnott, 2013).

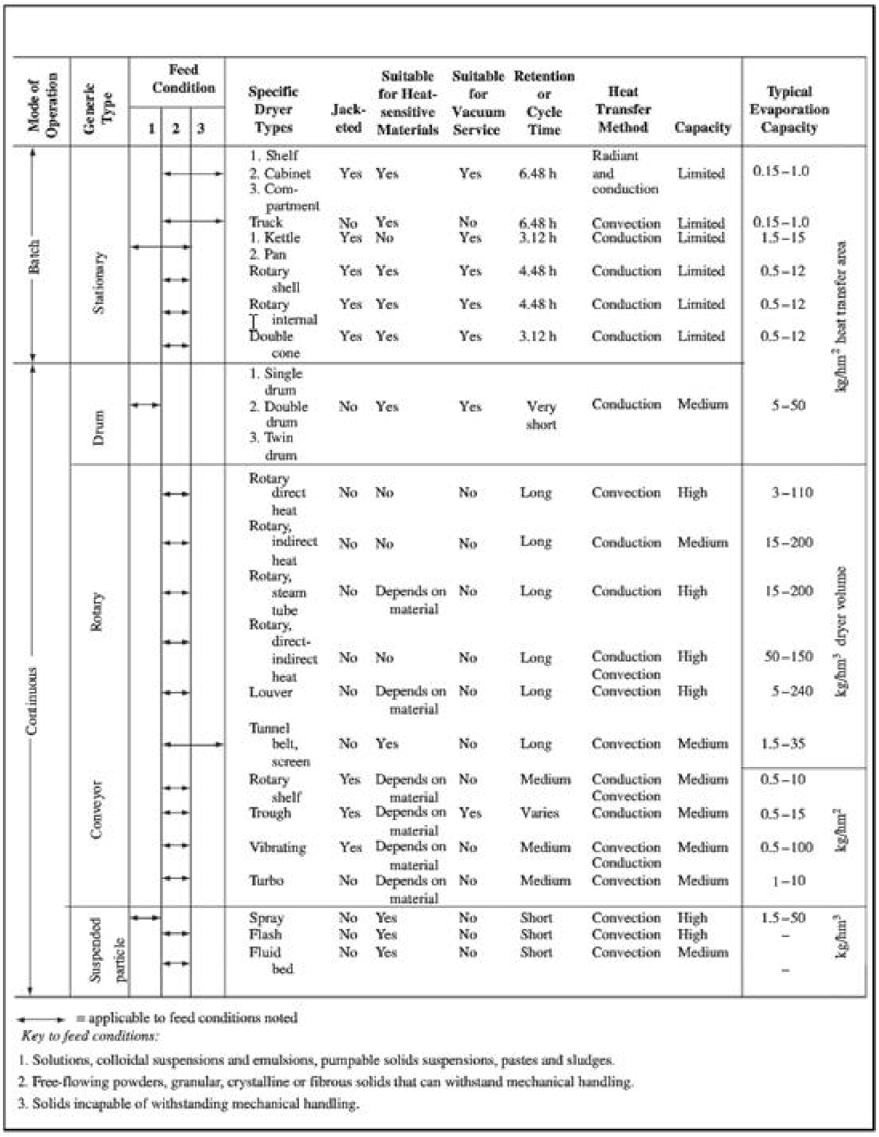
3.11.3 Choosing of drying equipment

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, fluidized
4. Heating system: conduction, convection, radiation.

Hot air is usually used as the heating and mass transfer medium in industrial dryers unless there are concerns about solvent flammability, in which case nitrogen, depleted air, or recirculating flue gas are used. The air may be directly heated by firing a burner into the air stream (using oil, gas, or coal as fuel) 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 5 shows the basic features of the various types of solids dryer used in the process industries. Batch dryers are normally used for small-scale production and when the drying cycle is likely to be long. Continuous dryers require less labour, less floor space, and produce a more uniform quality product. 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 pre-treatment equipment will normally be needed, such as extrusion or granulation. (Towler and Sinnott, 2013).

Table 5. Dryer selection (Towler and Sinnott, 2013)



Mode of Operation	Generic Type	Feed Condition 1	Feed Condition 2	Feed Condition 3	Specific Dryer Types	Jack-eted	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. Com-partment	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	
	Conveyor		↔	↔	Tunnel belt, screen	No	Yes	No	Long	Convection	Medium	1.5–35	
			↔		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.

4. Separation of homogeneous fluid mixtures

4.1 Introduction

In a homogeneous mixture, the constituent phases are indistinguishable, having merged into a single uniform phase. The Figure 14 below shows the five basic separation methods for separating of homogeneous mixtures. In most cases, the separation is not perfect and if the feed contains more than two species, two or more separation operations may be required. (Seader et al., 2011).

The most common separation technic, shown in the Figure 14a, creates a second phase, immiscible with the feed phase, by energy transfer or by pressure reduction (examples distillation, crystallization). A second technique, shown in Figure 14b, adds another fluid phase, which selectively absorbs, extracts, or strips certain species from the feed (examples liquid-liquid extraction, absorption). (Seader et al., 2011).

Less common, but of growing importance, is the use of a barrier (shown in Figure 14c), usually a polymer membrane, which involves a gas or liquid feed and exploits differences in species permeabilities through the barrier (examples ultrafiltration, reverse osmosis).

Techniques that involve contacting a vapour or liquid feed with a solid agent (shown in Figure 14d), are also of growing importance (examples adsorption). Most commonly, the solid agent consists of particles that are porous to achieve a high surface area, and differences in species adsorption ability are exploited. Finally, external field (centrifugal, thermal, electrical, flow, etc.), shown in Figure 14e, are applied in specialized cases to gas or liquid feeds (examples electrophoresis). (Seader et al., 2011).

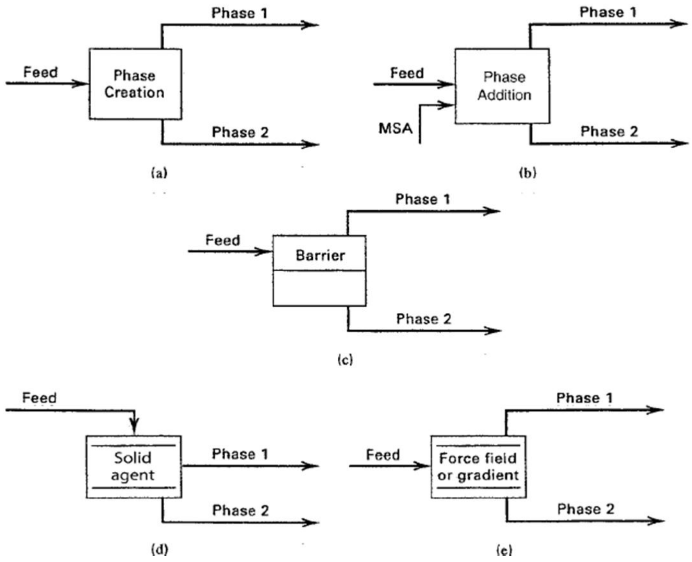


Figure 14 . Basic separation techniques for separating homogeneous mixtures: (a) separation by phase creation; (b) separation by phase addition; (c) separation by barriers; (d) separation by solid agent; (e) separation by force or gradient (Seader et al., 2011).

4.2 Separation by phase addition or creation

4.2.1 Introduction

If the feed is single-phase homogeneous solution, a second separable phase must be developed before separation of the species can be achieved. The second phase is created by an energy-separating agent (ESA) (Figure 14a) and/or added as a mass-separating agent (MSA) (Figure 14b). An ESA involves heat transfer or transfer of shaft work to or from the mixture. An MSA may be partially immiscible with one or more mixture components and frequently is the constituent of highest concentration in the added phase. Alternatively, the MSA may be miscible with a liquid feed mixture, but may selectively alter partitioning of species between liquid and vapour phases. This facilitates a separation when used in conjunction with an ESA. (Seader et al. 2011).

Disadvantages of using an MSA are (1) need for an additional separator to recover the MSA for recycle, (2) need for MSA makeup, (3) possible MSA product contamination, and (4) more difficult design procedures. Table 6 includes the more common separation operations based on interphase mass transfer between two phases, one of which is created by an ESA or added as an MSA. (Seader et al., 2011).

4.2.2 Partial condensation or partial vaporization

Partial condensation (#1 in Table 6) or partial vaporization (#1 in Table 6) is used when the feed mixture includes species that differ widely in volatility. Two phases are created when a vapour feed is partially condensed by removing heat, and a liquid feed a partially vaporized by adding heat. After partitioning of species has occurred by interphase mass transfer, the resulting vapour phase is enriched with respect to the species that are more easily vaporized, while the liquid phase is enriched with respect to the less-volatile species. The two phases are then separated by gravity. (Seader et al., 2011). Partial condensation is described in more detail in Chapter 5.1.

4.2.3 Flash vaporization

Partial vaporization can be initiated by flash vaporization (#2 in Table 6) by reducing the feed pressure with a valve or turbine. After partitioning of species has occurred by interphase mass transfer, the resulting vapour phase is enriched with respect to the species that are more easily vaporized, while the liquid phase is enriched with respect to the less-volatile species. The two phases are then separated by gravity. (Seader et al., 2011). Flash vaporization is described in more detail in Chapter 5.1.

4.2.4 Distillation

If the degree of separation achieved by a single contact of two phases is inadequate, distillation (#3 in Table 6), the most widely utilized industrial separation method, should be considered. Distillation involves multiple contacts between counter-currently flowing liquid and vapour phases. Vapour, flowing up the column, is increasingly enriched with respect to the more-volatile species, and liquid flowing down the column is increasingly enriched with respect to the less-volatile species. (Seader et al., 2011). Distillation is described in more detail in Chapter 5.2.

4.2.5 Extractive distillation

When the volatility difference between to species to be separated is so small as to necessitate more than about 100 distillation trays, extractive distillation (#4 in Table 6) is considered. A miscible MSA, acting as a solvent, increases the volatility difference among species in the feed, thereby reducing the number of trays. A subsequent operation, usually distillation, is used to recover the MSA for recycling. (Seader et al., 2011).

4.2.6 Reboiled absorption

If it is difficult to condense the vapour leaving the top of a distillation column, a liquid MSA called an absorbent may be fed to the top tray in place of reflux. The separation process is called reboiled absorption (#5 in Table 6). (Seader et al., 2011).

4.2.7 Absorption

The reboiled absorption in simplified to absorption (#6 in Table 6), if the feed is vapour ant the stripping section of the column i.e. the portion of the column below the feed entry, is not needed. Absorbers generally do not require an ESA (i.e. reboiler) and are frequently conducted at ambient temperature and elevated pressure. The vapour feed is fed at the top of the column. A subsequent operation, usually stripping, is used to recover the liquid MSA (absorbent) for recycling. (Seader et al., 2011).

4.2.8 Stripping

The inverse of absorption is stripping (#7 in Table 6) where liquid mixtures are separated at elevated temperature and ambient pressure by contacting the feed with a vapour stripping agent. This MSA eliminates the need to reboil the liquid at the bottom of the column, which may be important if the liquid is not thermally stable. The liquid feed is fed at the bottom of the column. Additional separation operations may be required to recover MSAs for recycling. (Seader et al., 2011).

4.2.9 Refluxed stripping

If in the stripping column trays are needed above the feed tray to achieve the separation, a refluxed stripper (#8 in Table 6) may be employed. Additional separation operations may be required to recover MSAs for recycling. (Seader et al., 2011).

4.2.10 Reboiled stripping

If the bottoms product from a stripper is thermally stable, it may be reboiled without using an MSA. In that case, the column is a reboiled stripper (#9 in Table 6). Additional separation operations may be required to recover MSAs for recycling. (Seader et al., 2011).

4.2.11 Azeotropic distillation

In azeotropic distillation (#10 in Table 6), an MSA is used to break an azeotrope that the species to be separated forms with the liquid phase. The MSA generates a two-liquid heterogeneous minimum-boiling azeotrope with the liquid phase, and is used as an entrainer in the separation process. The azeotrope is taken overhead, condensed, and separated. The MSA is recirculated. (Seader et al., 2011).

4.2.12 Liquid-liquid extraction

Liquid-liquid extraction (#11 and 12 in Table 6) with one or two solvents (MSAs) can be used when distillation is impractical, especially when the mixture to be separated is temperature-sensitive. A solvent (MSA) selectively dissolves only one or a fraction of the components in the feed. Is a two-solvent extraction, each solvent has its specific selectivity for the feed components. Additional operations are required to recover solvent from the streams leaving the extraction operation. (Seader et al., 2011). Liquid-liquid is described in more detail in Chapter 5.5.

4.2.13 Drying

Although, in drying (#13 in Table 6), the only requirement is that the vapour pressure of the liquid to be evaporated from the solid is higher than its partial pressure in the gas stream, the dryer design and operation represents a complex problem (Seader et al., 2011). Drying is described in more detail in Chapter 5.4.

4.2.14 Evaporation

Evaporation (#14 in Table 6) is defined as the transfer of volatile components of a liquid into a gas by heat transfer. Applications include humification, air conditioning, and concentration of aqueous solutions. (Seader et al., 2011). Evaporation is described in more detail in Chapter 5.3.

4.2.15 Crystallization

Crystallization (#15 in Table 6) is carried out where the desired product is a finely divided solid. Crystallization is a purification step, so the conditions must be such that impurities do not precipitate with the product. In solution crystallization, the mixture, which includes a solvent, is cooled and/or the solvent is evaporated. In melt crystallization, two or more soluble species are separated by partial freezing. (Seader et al., 2011). Crystallization is described in more detail in Chapter 5.6.

4.2.16 Desublimation

Desublimation (#16 in Table 6) is the transfer of a species from the gas to solid without formation of an intermediate liquid phase (Seader et al., 2011).

4.2.17 Leaching

Solid-liquid extraction, leaching (#17 in Table 6) is used in the metallurgical, natural product, and food industries. To promote rapid solute diffusion out of the solid and into the liquid solvent, particle size of the solid is usually reduced. The major difference between solid–liquid and liquid–liquid systems is the difficulty of transporting the solid (often as slurry or a wet cake) from stage to stage. (Seader et al., 2011). Leaching is described more detailed in Chapter 3.10.

4.2.18 Foam fractionation

In foam fractionation (#18 in Table 6), a natural or chelate induced surface activity causes a solute to migrate to rising bubbles and is thus removed as foam (Seader et al., 2011).

Table 6. Separation operations based on phase creation or addition (Seader et al., 2011).

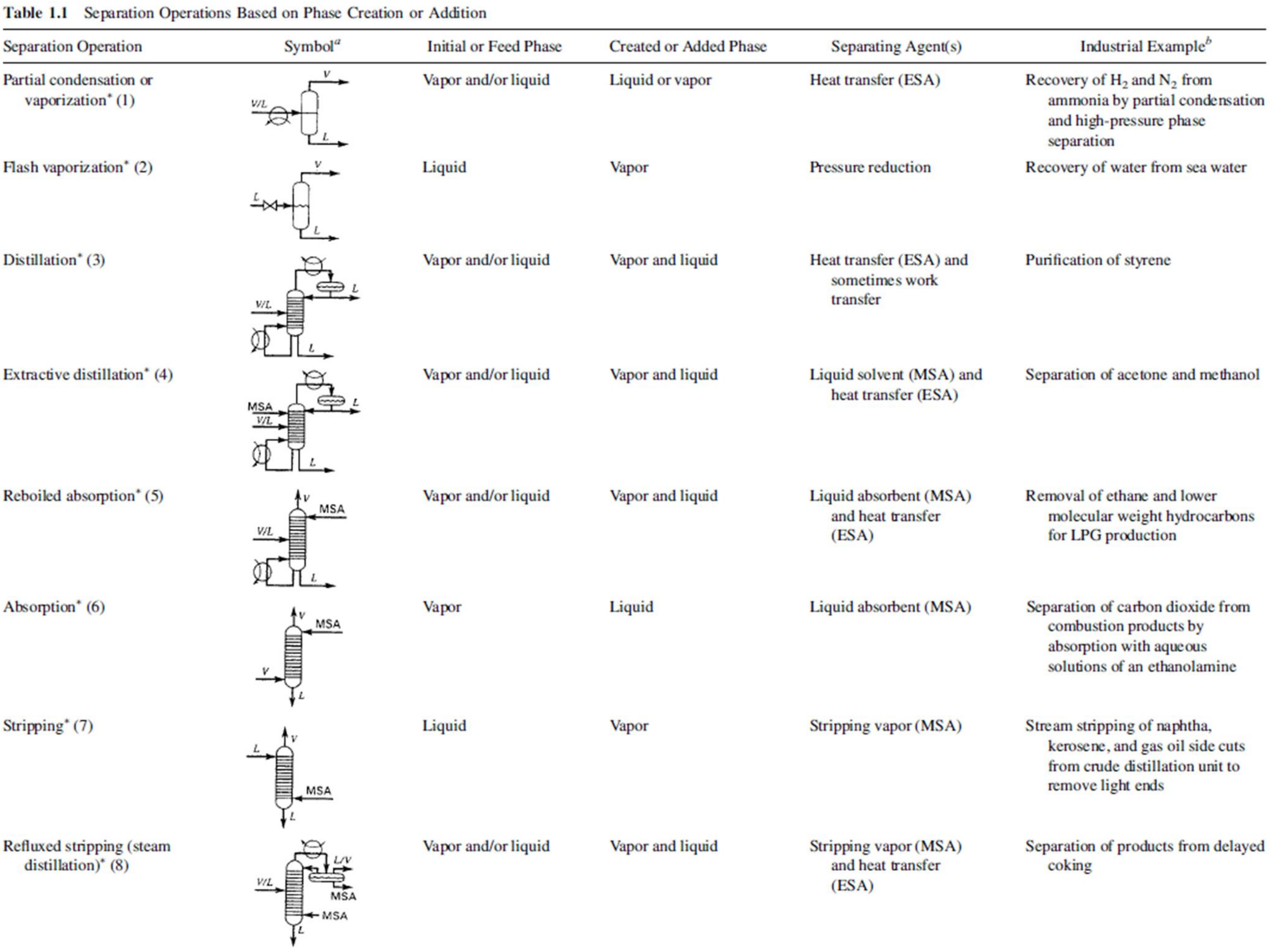
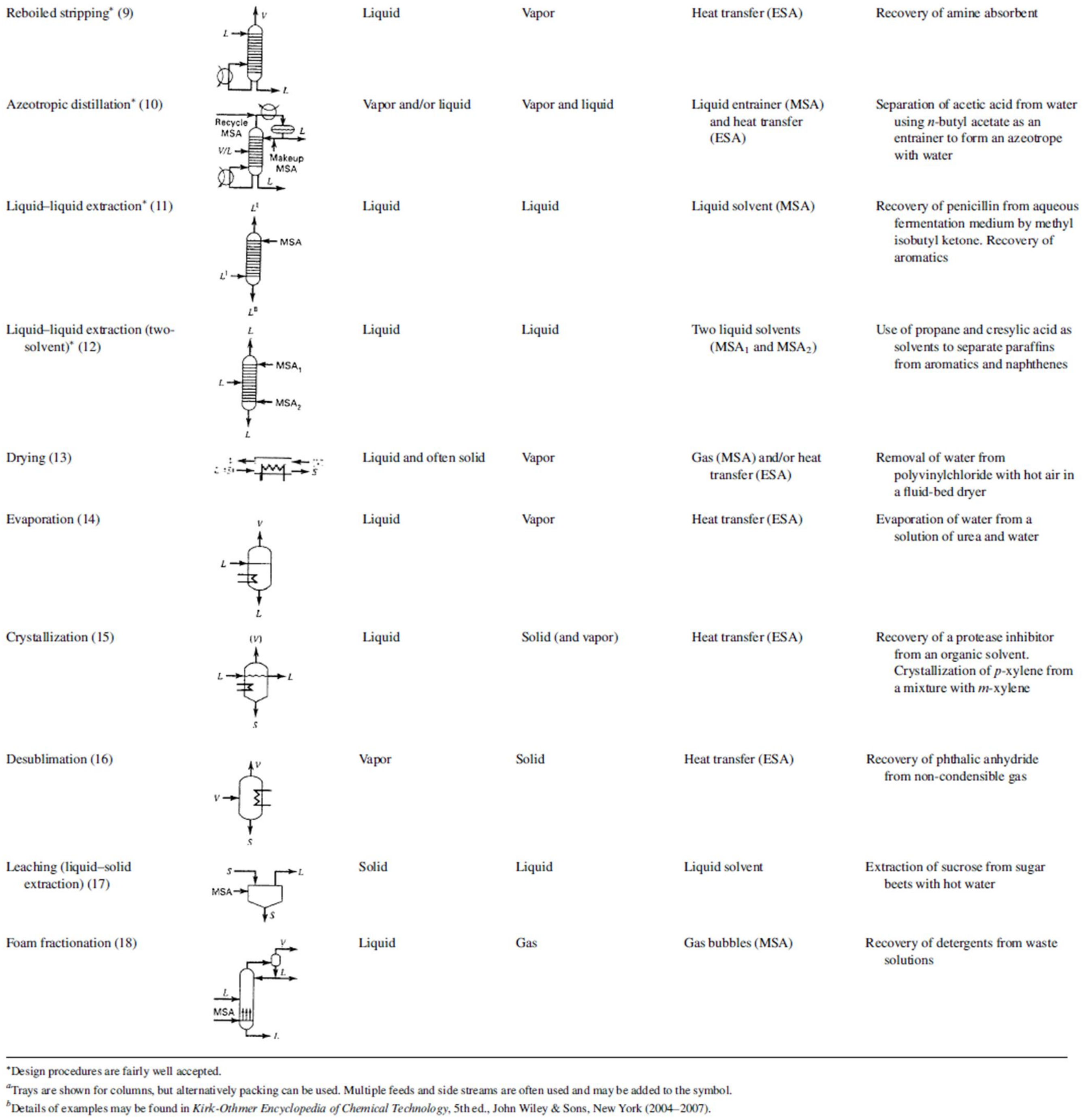


Table 1.1 Separation Operations Based on Phase Creation or Addition

Separation Operation	Symbol[a]	Initial or Feed Phase	Created or Added Phase	Separating Agent(s)	Industrial Example[b]
Partial condensation or vaporization* (1)	V/L, V, L	Vapor and/or liquid	Liquid or vapor	Heat transfer (ESA)	Recovery of H_2 and N_2 from ammonia by partial condensation and high-pressure phase separation
Flash vaporization* (2)	L, V, L	Liquid	Vapor	Pressure reduction	Recovery of water from sea water
Distillation* (3)	V/L, L, L	Vapor and/or liquid	Vapor and liquid	Heat transfer (ESA) and sometimes work transfer	Purification of styrene
Extractive distillation* (4)	MSA, V/L, L, L	Vapor and/or liquid	Vapor and liquid	Liquid solvent (MSA) and heat transfer (ESA)	Separation of acetone and methanol
Reboiled absorption* (5)	V, MSA, V/L, L	Vapor and/or liquid	Vapor and liquid	Liquid absorbent (MSA) and heat transfer (ESA)	Removal of ethane and lower molecular weight hydrocarbons for LPG production
Absorption* (6)	V, MSA, V, L	Vapor	Liquid	Liquid absorbent (MSA)	Separation of carbon dioxide from combustion products by absorption with aqueous solutions of an ethanolamine
Stripping* (7)	L, V, MSA, L	Liquid	Vapor	Stripping vapor (MSA)	Stream stripping of naphtha, kerosene, and gas oil side cuts from crude distillation unit to remove light ends
Refluxed stripping (steam distillation)* (8)	L/V, V/L, MSA, MSA, L	Vapor and/or liquid	Vapor and liquid	Stripping vapor (MSA) and heat transfer (ESA)	Separation of products from delayed coking



Reboiled stripping[*] (9)		Liquid	Vapor	Heat transfer (ESA)	Recovery of amine absorbent
Azeotropic distillation[*] (10)		Vapor and/or liquid	Vapor and liquid	Liquid entrainer (MSA) and heat transfer (ESA)	Separation of acetic acid from water using *n*-butyl acetate as an entrainer to form an azeotrope with water
Liquid–liquid extraction[*] (11)		Liquid	Liquid	Liquid solvent (MSA)	Recovery of penicillin from aqueous fermentation medium by methyl isobutyl ketone. Recovery of aromatics
Liquid–liquid extraction (two-solvent)[*] (12)		Liquid	Liquid	Two liquid solvents (MSA_1 and MSA_2)	Use of propane and cresylic acid as solvents to separate paraffins from aromatics and naphthenes
Drying (13)		Liquid and often solid	Vapor	Gas (MSA) and/or heat transfer (ESA)	Removal of water from polyvinylchloride with hot air in a fluid-bed dryer
Evaporation (14)		Liquid	Vapor	Heat transfer (ESA)	Evaporation of water from a solution of urea and water
Crystallization (15)		Liquid	Solid (and vapor)	Heat transfer (ESA)	Recovery of a protease inhibitor from an organic solvent. Crystallization of *p*-xylene from a mixture with *m*-xylene
Desublimation (16)		Vapor	Solid	Heat transfer (ESA)	Recovery of phthalic anhydride from non-condensible gas
Leaching (liquid–solid extraction) (17)		Solid	Liquid	Liquid solvent	Extraction of sucrose from sugar beets with hot water
Foam fractionation (18)		Liquid	Gas	Gas bubbles (MSA)	Recovery of detergents from waste solutions

---

[*] Design procedures are fairly well accepted.

[a] Trays are shown for columns, but alternatively packing can be used. Multiple feeds and side streams are often used and may be added to the symbol.

[b] Details of examples may be found in *Kirk-Othmer Encyclopedia of Chemical Technology*, 5th ed., John Wiley & Sons, New York (2004–2007).

4.3 Separations by barriers

4.3.1 Introduction

Use of microporous and nonporous membranes as semipermeable barriers for selective separations is gaining ground. Membranes are fabricated mainly from natural fibres and synthetic polymers, but also from metals and ceramics. Membranes are fabricated into tubes, flat sheets, hollow fibres, or spiral-wound sheets, and incorporated into commercial modules or cartridges. For microporous membranes, separation is effected by rate of species diffusion through the pores; for nonporous membranes, separation is controlled by differences in solubility in the membrane and rate of species diffusion. (Seader et al., 2011). Table 7 lists membrane-separation operations.

4.3.2 Osmosis

Osmosis (#1 in Table 7) involves transfer, by a concentration gradient, of a solvent through a membrane into a mixture of solute and solvent. The membrane is almost impermeable to the solute. (Seader et al., 2011).

4.3.3 Reverse osmosis

In reverse osmosis (#2 in Table 7), transport of solvent in the opposite direction is effected by importing a pressure, higher than the osmotic pressure, on the feed side (Seader et al., 2011).

4.3.4 Dialysis

Dialysis (#3 in Table 7) is the transport by a concentration gradient of small solute molecules through a porous membrane. The molecules unable to pass through the membrane are small, insoluble, non-diffusible particles. (Seader et al.,2011).

4.3.5 Microfiltration and ultrafiltration

Microporous membranes selectively allow small solute molecules and/or solvents to pass through membrane, while preventing large dissolved molecules and suspended solids from passing through. Microfiltration (#4 in Table 7) refers to the retention of molecules from 0,02 to 10 µm. Ultrafiltration (#5 in Table 7) refers to retention of molecules that range from 1 to 20 nm. (Seader et al., 2011). Micro- ultra- and nanofiltration is described more detailed in Chapter 5.7.

4.3.6 Pervaporation

To achieve high purities, pervaporation (#6 in Table 7) can be used. In pervaporation the species transported through the nonporous membrane is evaporated and the heat of vaporization must be supplied. Pervaporation is used to separate azeotropic mixtures. (Seader et al., 2011).

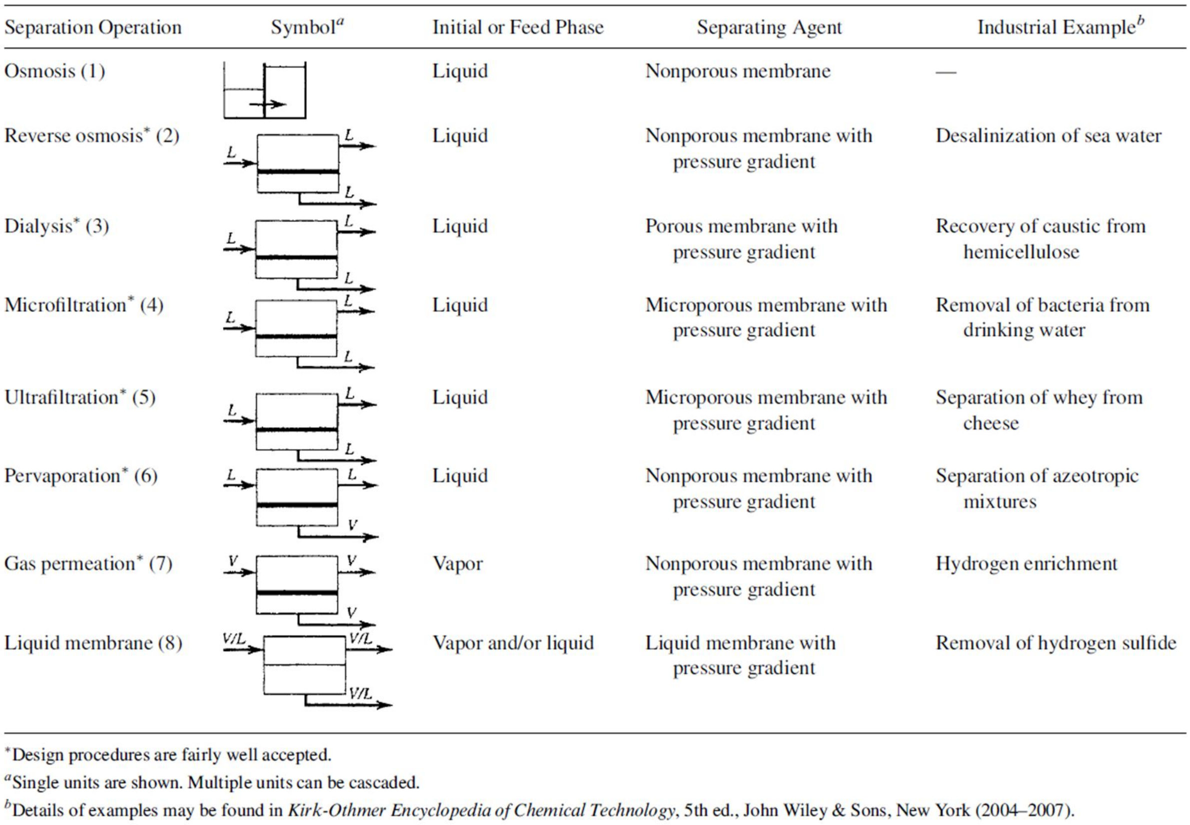
4.3.7 Gas permeation

Selective gas permeation (#7 in Table 7) using a pressure driving force is used for separation of gases. Gas permeation is performed with centrifuges (Seader et al., 2011).

4.3.8 Liquid membrane

Liquid membranes (#8 in Table 7), only a few molecules thick, can be formed from surfactant-containing mixtures at the interface between two fluid phases or alternatively by imbibing the micropores with liquids doped with additives to facilitate transport of solutes (Seader et al., 2011).

Table 7. Separation operations based on a barrier (Seader et al., 2011).



Separation Operation	Symbol[a]	Initial or Feed Phase	Separating Agent	Industrial Example[b]
Osmosis (1)		Liquid	Nonporous membrane	—
Reverse osmosis* (2)	L L L	Liquid	Nonporous membrane with pressure gradient	Desalinization of sea water
Dialysis* (3)	L L L	Liquid	Porous membrane with pressure gradient	Recovery of caustic from hemicellulose
Microfiltration* (4)	L L L	Liquid	Microporous membrane with pressure gradient	Removal of bacteria from drinking water
Ultrafiltration* (5)	L L L	Liquid	Microporous membrane with pressure gradient	Separation of whey from cheese
Pervaporation* (6)	L L V	Liquid	Nonporous membrane with pressure gradient	Separation of azeotropic mixtures
Gas permeation* (7)	V V V	Vapor	Nonporous membrane with pressure gradient	Hydrogen enrichment
Liquid membrane (8)	V/L V/L V/L	Vapor and/or liquid	Liquid membrane with pressure gradient	Removal of hydrogen sulfide

*Design procedures are fairly well accepted.

[a] Single units are shown. Multiple units can be cascaded.

[b] Details of examples may be found in *Kirk-Othmer Encyclopedia of Chemical Technology*, 5th ed., John Wiley & Sons, New York (2004–2007).

4.4 Separations by solid agents

4.4.1 Introduction

Separations that use solid agents are listed in Table 8. The solid, in the form of packing or granular material, is the adsorbent itself, or it acts as an inert support for a thin layer of adsorbent by selective adsorption or chemical reaction with species in the feed. Adsorption is confined to the surface of the solid adsorbent. The active separating agent eventually becomes saturated with solute and must be regenerated or replaced. (Seader et al., 2011).

4.4.2 Adsorption

Adsorption (#1 in Table 8) is used to remove species in low concentrations and is followed by desorption to regenerate the adsorbents. Commonly used adsorbents are activated carbon, aluminium oxide, silica gel, and synthetic sodium or calcium aluminosilicate zeolites. Adsorbers consist of a cylindrical vessel packed with a bed of solid adsorbent particles through which the gas or liquid flows. Because the adsorbent must be regenerated, two or more vessels are used. Regeneration occurs by one of four methods: (1) vaporization of the adsorbate with a hot purge gas, (2) reduction of pressure to vaporize the adsorbate, (3) inert purge stripping without change in temperature or pressure, and (4) displacement desorption by a fluid containing a more strongly adsorbed species. (Seader et al., 2011).

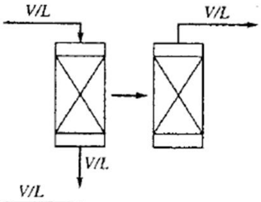
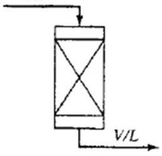
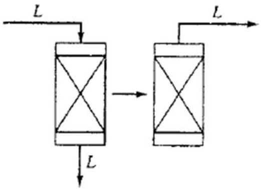
4.4.3 Chromatography

Chromatography (#2 in Table 8) separated gas or liquid mixtures by passing them through a packed bed. The bed may be solid particles (gas-solid chromatography) or a solid-inert support coated with a viscous liquid (gas-liquid chromatography). Because of selective adsorption on the solid surface, or absorption into liquid adsorbents followed by desorption, components move through the bed at different rates, thus effecting the separation. (Seader et al., 2011).

4.4.4 Ion exchange

In ion exchange (#3 in Table 8), as well as adsorption, solid particles are used and regenerated. Unlike in adsorption, a chemical reaction is involved (Seader et al., 2011).

Table 8. Separation operations based on a solid agent (Seader et al., 2011).

Separation Operation	Symbol[a]	Initial or Feed Phase	Separating Agent	Industrial Example[b]
Adsorption* (1)		Vapor or liquid	Solid adsorbent	Purification of *p*-xylene
Chromatography* (2)		Vapor or liquid	Solid adsorbent or liquid adsorbent on a solid support	Separation and purification of proteins from complex mixtures. Separation of xylene isomers and ethylbenzene
Ion exchange* (3)		Liquid	Resin with ion-active sites	Demineralization of water

*Design procedures are fairly well accepted.

[a] Single units are shown. Multiple units can be cascaded.

[b] Details of examples may be found in *Kirk-Othmer Encyclopedia of Chemical Technology*, 5th ed., John Wiley & Sons, New York (2004–2007).

4.5 Separations by external field or gradient

4.5.1 Introduction

External fields can take advantage of differing degrees of response of molecules and ions to force fields. Table 9 lists separation operations by applied field or gradient. Centrifugation (#1 in Table 9) establishes a pressure field that separates fluid mixtures according to molecular weight. In thermal diffusion (#2 in Table 9), a temperature gradient is applied to a homogeneous solution and concentration gradients are established. In electrolysis (#3 in Table 9), molecules are decomposed into atoms. In electrodialysis (#4 in Table 9), cation- and anion-permeable membranes carry a fixed charge, thus preventing migration of species of like charge. Electrophoresis (#5 in Table 9) exploits the different migration velocities of charged colloidal or suspended species in an electric field. Positively charged species migrate to the cathode, while negatively charged particles go to the anode. In field-flow fractionating (#6 in Table 9), an electrical or magnetic field or thermal gradient is established perpendicular to a laminar-flow field. Components of the mixture travel in the flow direction at different velocities, so a separation is achieved. (Seader et al., 2011).

Table 9. Separation operations by applied field or gradient (Seader et al., 2011).

Separation Operation	Initial or Feed Phase	Force Field or Gradient	Industrial Example[a]
Centrifugation (1)	Vapor or liquid	Centrifugal force field	Separation of uranium isotopes
Thermal diffusion (2)	Vapor or liquid	Thermal gradient	Separation of chlorine isotopes
Electrolysis (3)	Liquid	Electrical force field	Concentration of heavy water
Electrodialysis (4)	Liquid	Electrical force field and membrane	Desalinization of sea water
Electrophoresis (5)	Liquid	Electrical force field	Recovery of hemicelluloses
Field-flow fractionation (6)	Liquid	Laminar flow in force field	—

[a] Details of examples may be found in *Kirk-Othmer Encyclopedia of Chemical Technology*, 5th ed., John Wiley & Sons, New York (2004–2007).

5. Selected separation processes for homogeneous solutions

5.1 Flash vaporization and partial condensation

A flash is a single-equilibrium-stage distillation in which a feed is partially vaporized to give a vapour richer than the feed in the more volatile components. In Figure 15a, (1) a pressurized liquid feed is heated and flashed adiabatically across a valve to a lower pressure, resulting in creation of a vapour phase that is separated from the remaining liquid in a flash drum, or in Figure 15b, (2) if the valve is omitted, a liquid can be partially vaporized in a heater and then separated into two phases (isothermal flash). Alternatively, a vapour feed can be cooled and partially condensed, as in Figure 15c, to give, after phase separation, a liquid richer in the less-volatile components. For properly designed systems, the streams leaving the drum will be in phase equilibrium. Unless the relative volatility is very large, flashing (partial vaporization) or partial condensation is not a replacement for distillation, but an auxiliary operation used to prepare streams for further processing. (Seader et al., 2011).

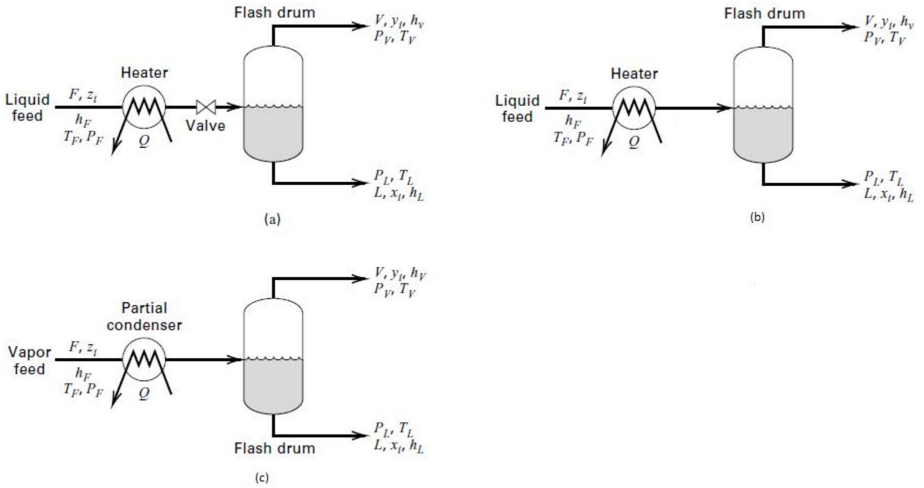


Figure 15. Continuous, single-stage equilibrium separations: (a) flash vaporization and (b) partial condensation (Seader et al., 2011).

5.2 Distillation

Distillation is widely used separation method for homogenous mixtures. It has many advantages including low capital costs, easy scalability and simple flowsheet (Peters et al. 2003). The separation of liquid mixtures by distillation depends on differences in volatility between the components. Peters et al., (2003) advice to use distillation, if the relative volatility of key components is greater than 1.2, to separate components in homogenous mixture.

5.2.1 Description

Flash vaporization described in 4.2.3 can be called also flash distillation. There vapor is produced by boiling the liquid mixture and vapor and liquid is separated without letting any condensing vapor to return to the still. In flash distillation there is no reflux. The distillation with reflux is based on the return of part of the condensate to the still. (McCabe et al., 1993) In distillation vapor flows up the column and liquid flows counter currently down the column. The vapor and liquid are brought into contact on plates or packing. Part of the condensate from the condenser is returned to the top of the column to provide liquid flow above the feed point (reflux), and part of the liquid from the base of the column is vaporized in the reboiler and returned to provide the vapor flow. (Towler and Sinnott, 2013). Figure 16 shows continuous distillation column with rectifying and stripping sections.

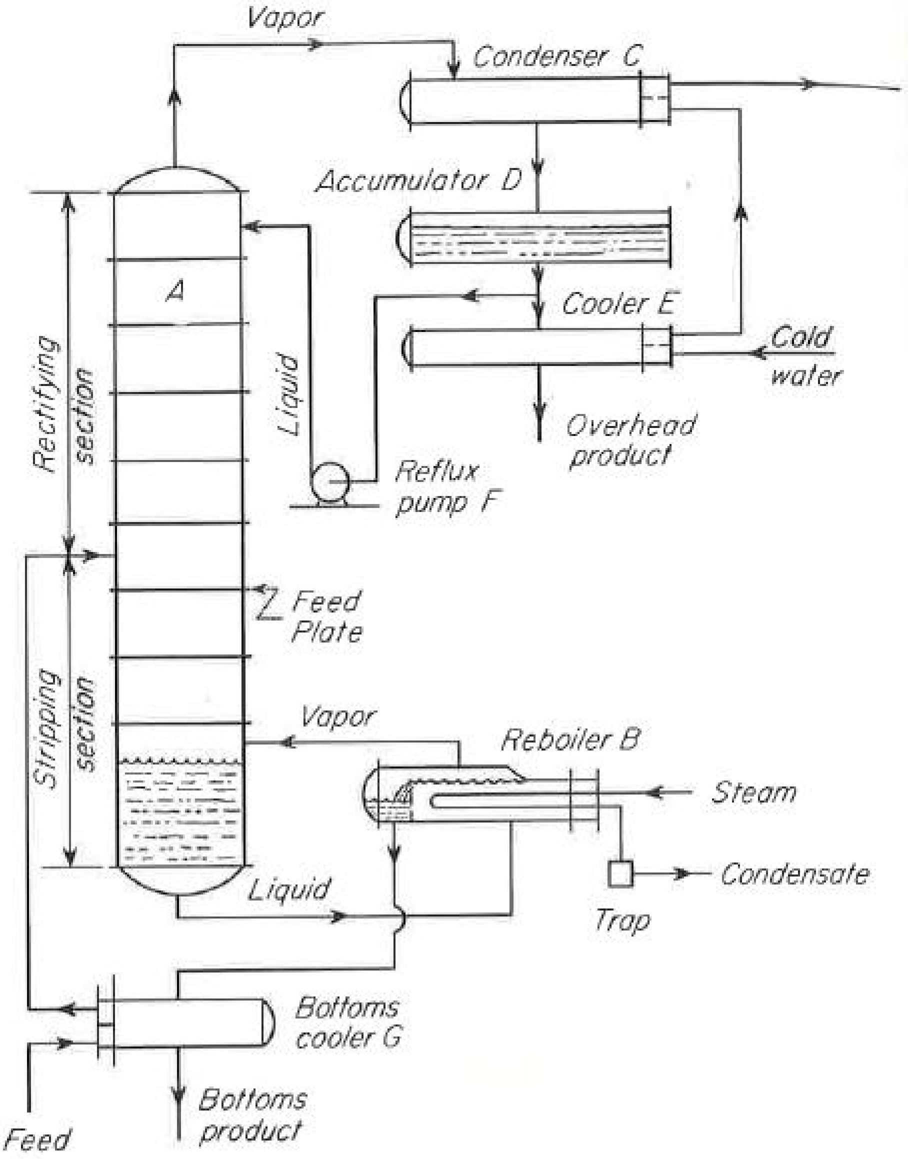


Figure 16. Continuous distillation column with rectifying and stripping sections (McCabe et al., 1993)

5.2.2 Distillation columns

Equipment needed for distillation is a column, a reboiler and a condenser and some relative small ancillary equipment. Column internals can be tray, random packing or structural packing. Typically, trays are favored for high liquid flow rate and operating pressure of distillation and when column diameter is large. Random packing is more often used for small column diameters or when corrosion and foaming are present. Structural packing is considered for low-pressure and vacuum operation. (Peters et al., 2003)

Schematic views of a tray and packed column are shown Figure 17. Comparison of packing and tray types are shown in Table 10 and Table 11.

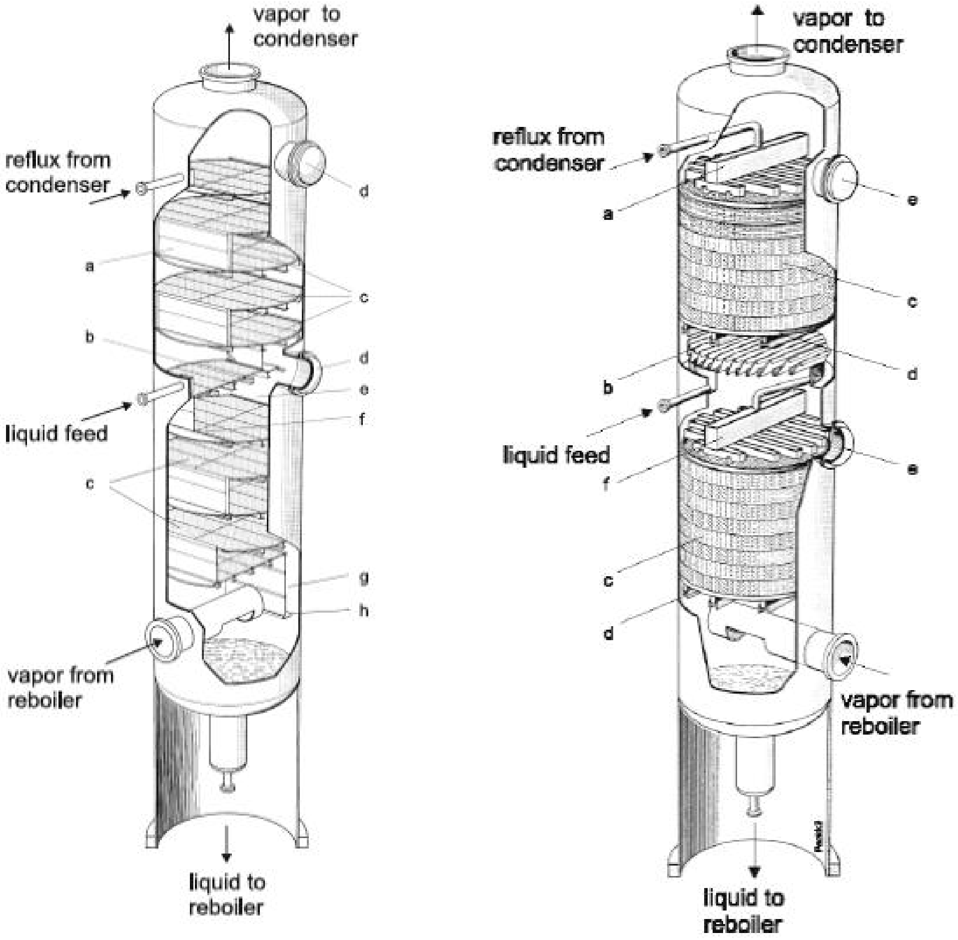
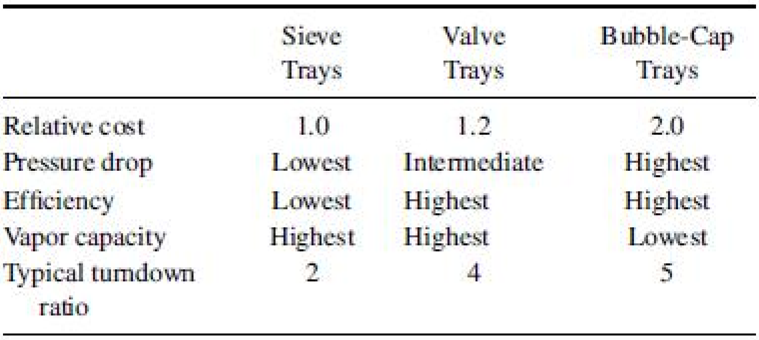


Figure 17. Perspective view of a tray column (left) and packed column (right). Tray column: (a) downcomer, (b) tray support, (c) sieve trays, (d) man way, (e) outlet weir, (f) inlet weir, (g) side wall of downcomer, and (h) liquid seal. Packed column: (a) liquid distributor, b) liquid collector, (c) structured packing, (d) support grid, (e) man way, and (f) liquid re-distributor (Modified from Mersmann et al., 2011)

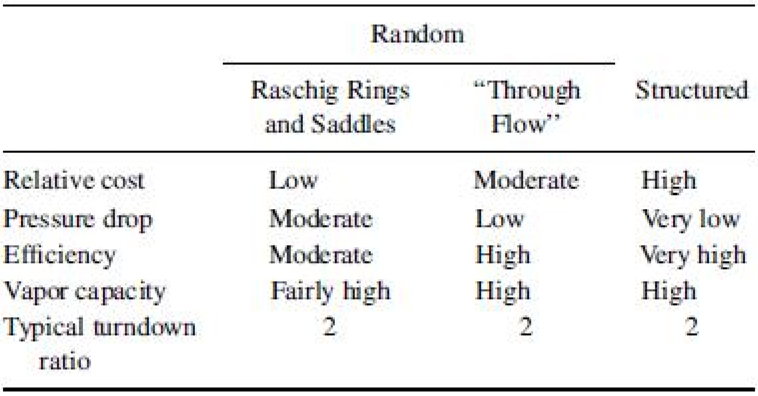
Distillation operations can be described as a cascade of equilibrium trays with each one solved as a flash unit. The feed stream enters to intermediate tray. At the bottom liquid product is removed and a reboiler vaporizes the liquid stream on the lowest stage. Vapor leaving the top tray is condensed and overhead product is removed with the remaining liquid returned to the top tray.

Table 10. Comparison of tray types for separation columns (Seader et al., 2011)



	Sieve Trays	Valve Trays	Bubble-Cap Trays
Relative cost	1.0	1.2	2.0
Pressure drop	Lowest	Intermediate	Highest
Efficiency	Lowest	Highest	Highest
Vapor capacity	Highest	Highest	Lowest
Typical turndown ratio	2	4	5

Table 11. Comparison of packing types for separation columns (Seader et al., 2011)



	Random		
	Raschig Rings and Saddles	“Through Flow”	Structured
Relative cost	Low	Moderate	High
Pressure drop	Moderate	Low	Very low
Efficiency	Moderate	High	Very high
Vapor capacity	Fairly high	High	High
Typical turndown ratio	2	2	2

5.2.3 Distillation Column Design

The design of a distillation column can be divided into the following steps (Towler and Sinnott, 2013):

1. Specify the degree of separation required: set product specifications.
2. Select the operating conditions: batch or continuous, operating pressure.
3. Select the type of contacting device: plates or packing.
4. Determine the stage and reflux requirements: the number of equilibrium stages.
5. Size the column: diameter, number of real stages.
6. Design the column internals: plates, distributors, packing supports.
7. Mechanical design: vessel and internal fittings.

The principal step is to determine the stage and reflux requirements. This is a relatively simple procedure when the feed is a binary mixture, but can be complex when the feed contains more than two components (multicomponent systems). Almost all distillation design is carried out using commercial process simulation software. (Towler and Sinnott, 2013)

A distillation column operates between the limiting conditions of minimum and total reflux. As reflux ratio increases, number of stages (N) decreases, column diameter increases, and reboiler steam and condenser cooling-water requirements increase. These have an effect on the operating costs. Based on fixed and variable costs related to needed equipment sizes and duties and utility needs the optimal reflux ratio can be obtained as illustrated in Figure 18. (Seader et al., 2011)

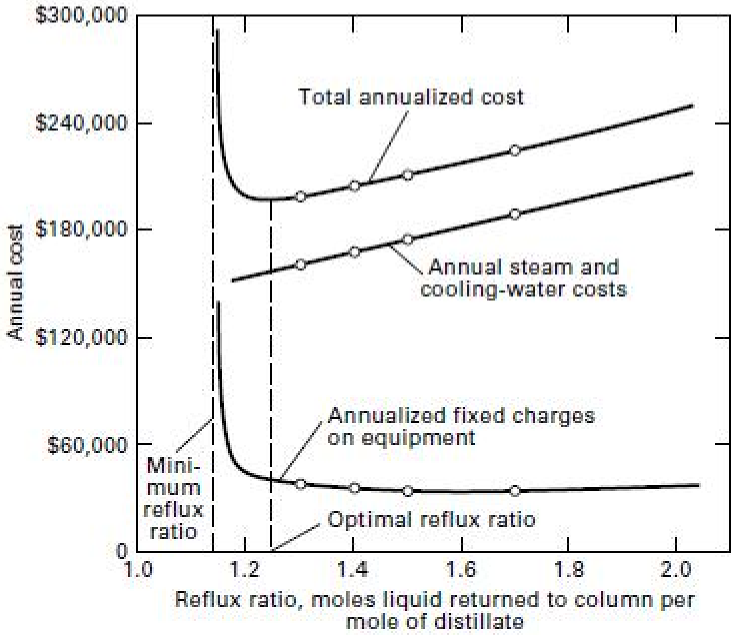


Figure 18. Annual operating costs as a function of reflux ratio to obtain optimal reflux ratio for a representative distillation operation. (Seader et al., 2011)

5.2.4 Multicomponent distillation

A problem of determining the stage and reflux requirements for multicomponent distillations is much more complex than for binary mixtures. With a multicomponent mixture, fixing one component composition does not uniquely determine the other component compositions and the stage temperature. Also when the feed contains more than two components it is not possible to specify the complete composition of the top and bottom products independently. The separation between the top and bottom products is usually specified by setting limits on two “key” components, between which it is desired to make the separation. Clearly, the greater the number of components considered, the more difficult the problem. (Towler and Sinnott, 2013).

5.2.5 Enhanced distillation

When relative volatility < 1.10, separation by ordinary distillation may be uneconomical, and even impossible if an azeotrope forms. In that case, one of the following techniques, described by Seader et al., 2011, may be an option.

1. Extractive Distillation: Uses large amounts of a relatively high-boiling solvent to alter the liquid-phase activity coefficients so that the relative volatility of key components becomes more favourable. Solvent enters the column a few trays below the top, and exits from the bottom without forming any azeotropes. If the column feed is an azeotrope, the solvent breaks it. It may also reverse key-component volatilities.

2. Salt Distillation: A variation of extractive distillation in which a of the key components is altered by adding to the top reflux a soluble, non-volatile ionic salt, which stays in the liquid phase as it passes down the column.

3. Pressure-Swing Distillation: Separates a mixture that forms a pressure-sensitive azeotrope by utilizing two columns in sequence at different pressures.

4. Homogeneous Azeotropic Distillation: A method of separating a mixture by adding an entrainer that forms a homogeneous minimum- or maximum-boiling azeotrope with feed component(s). Where the entrainer is added depends on whether the azeotrope is removed from the top or the bottom of the column.

5. Heterogeneous Azeotropic Distillation: A minimum boiling heterogeneous azeotrope is formed by the entrainer. The azeotrope splits into two liquid phases in the overhead condenser. One liquid phase is sent back as reflux; the other is sent to another separation step or is a product.

6. Reactive Distillation: A chemical that reacts selectively and reversibly with one or more feed constituents is added, and the reaction product is then distilled from the non-reacting components. The reaction is later reversed to recover the separating agent and reacting component. This operation, referred to as catalytic distillation if a catalyst is used, is suited to reactions limited by equilibrium constraints, since the product is continuously separated. Reactive distillation also refers to chemical reaction and distillation conducted simultaneously in the same apparatus.

5.3 Evaporation

5.3.1 Description

On an industrial scale, evaporation and crystallization are the main processes used for the recovery of dissolved solids from solutions. Evaporation is the removal of a solvent by vaporization, from solids that are not volatile. It is normally used to produce a concentrated liquid, often prior to crystallization, but a dry solid product can be obtained with some specialized designs. (Towler and Sinnott, 2013).

Most evaporators are heated with steam condensing on metal tubes. Usually the material to be evaporated flows inside the tubes. Usually steam is below 3 atm and boiling liquid under moderate vacuum. Reducing the boiling point temperature of the liquid increase the temperature difference between steam and boiling liquid and thus increases the heat-transfer rate in the evaporator. (McCabe et al., 1993).

Figure 19 is a schematic diagram for the model used to make mass-balance, energy-balance, and heat-transfer calculations to size evaporators operating under continuous-flow, steady-state conditions. So-called thin liquor at temperature T_f with weight-fraction solute w_f, is fed to the evaporator at mass flow rate m_f. A heating medium, e.g., saturated steam, is fed to the heat-exchanger tubes at T_s, P_s, and mass flow rate m_S. Saturated condensate leaves the heat exchanger at the same pressure and temperature. Heat transfer rate Q to the solution in the evaporator in the temperature T_e causes the solution partially evaporate to produce vapour at temperature T_V, with flow rate mV. The thick-liquor concentrate leaves at temperature T_P, with weight fraction solute w_p at mass flow rate m_P. The heat exchanger has a heat-transfer area A and overall heat-transfer coefficient U. (Seader et al., 2011). Since the solution in the evaporator is assumed to be completely mixed, the concentrated product and the remaining solution in the evaporator have the same composition and temperature, which is the boiling point of the solution i.e. T_e=T_p. The temperature of the evaporated vapour is also same, since it is in equilibrium with the boiling solution, i.e. T_e=T_p=T_v. The pressure of the system is the vapour pressure of the solution at the present temperature. (Geankoplis 1993).

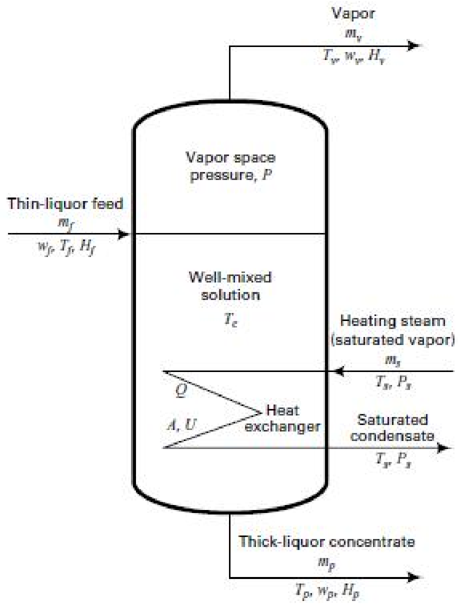
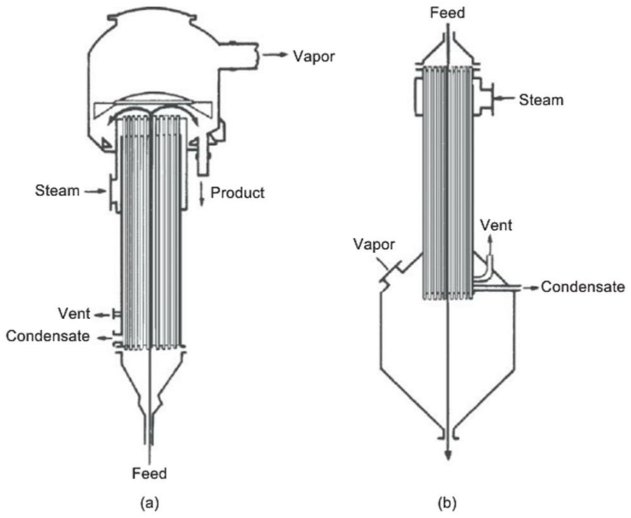


Figure 19. Continuous-flow, steady-state model for an evaporator (Seader et al., 2011).

5.3.2 Evaporator types

Evaporator designs can be grouped into the following four basic types: (1) direct-heated evaporators, (2) long-tube evaporators, (3) forced-circulation evaporators, and (4) wiped-film evaporators (Towler and Sinnott, 2013).

In long-tube evaporators (Figure 20) the liquid flows as a thin film on the walls of long, vertical heated tubes. Both falling film and rising film types are used. They are suitable for high capacities and low viscosity solutions. (Towler and Sinnott, 2013).



(a) (b)

Figure 20. Long-tube evaporators: (a) rising film; (b) falling film (Towler and Sinnott, 2013).

In forced-circulation evaporators (Figure 21) the liquid is pumped through the tubes. They are suitable for use with materials that tend to foul the heat transfer surfaces, and where crystallization can occur in the evaporator. (Towler and Sinnott, 2013).

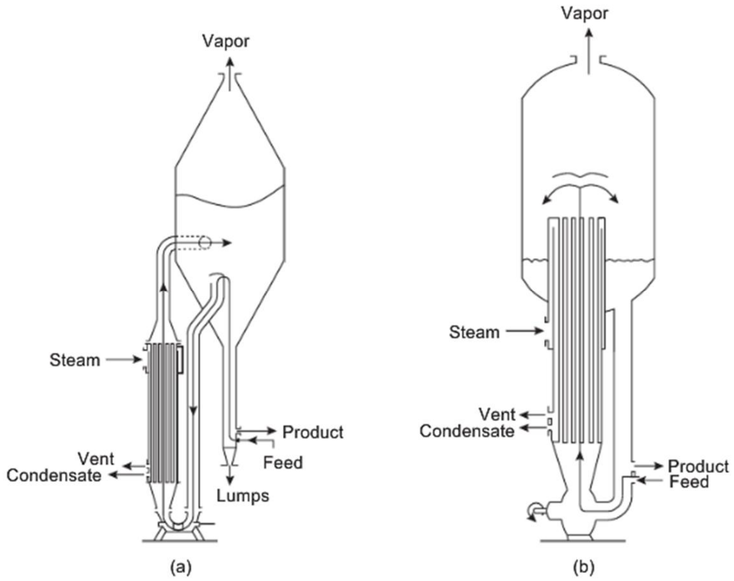


Figure 21. Forced-circulation evaporators: (a) submerged tube; (b) boiling tube (Towler and Sinnott, 2013).

In wiped-film evaporators (Figure 22) a thin layer of solution is spread on the heating surface by mechanical means. They are used for very viscous materials and for producing solid products. (Towler and Sinnott, 2013).

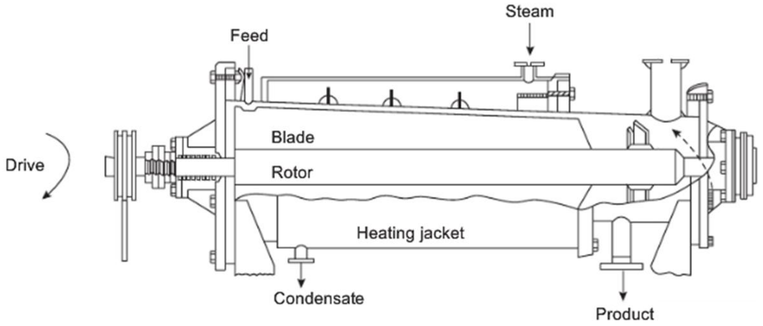
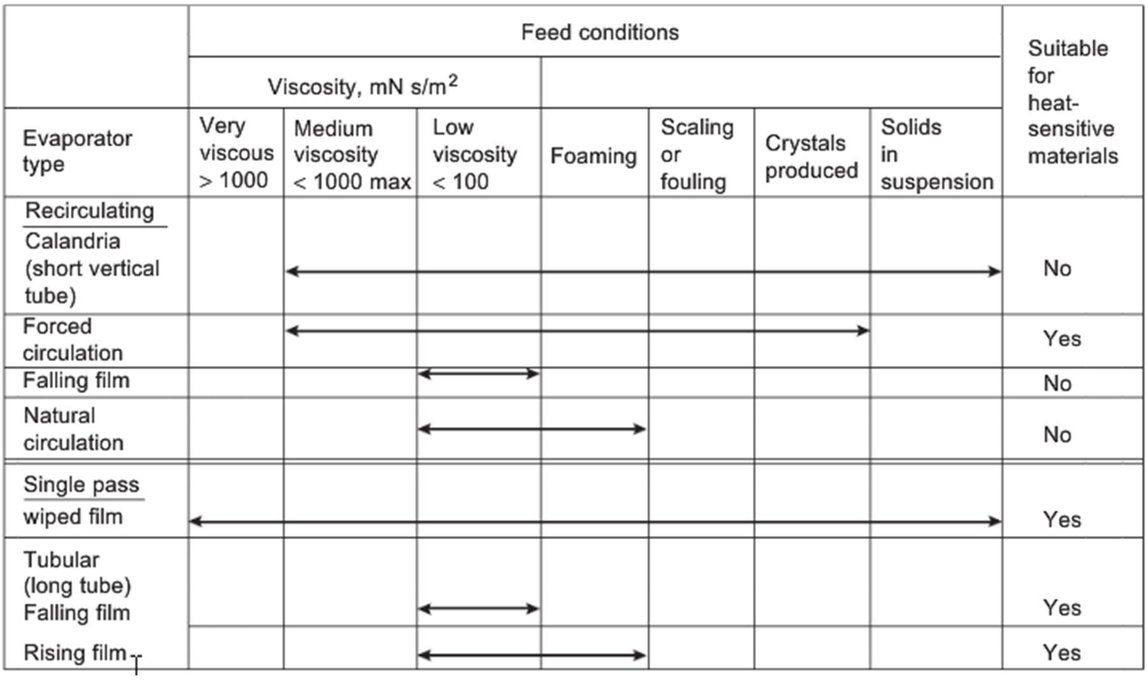


Figure 22. Horizontal wiped-film evaporator (Towler and Sinnott, 2013).

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, and (7) whether direct heating can be used. A selection guide based on these factors is given in Figure 23. (Towler and Sinnott, 2013).



	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 23. Evaporator selection guide (Towler and Sinnott, 2013).

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. (Towler and Sinnott, 2013).

5.3.3 Operation modes

Single-effect evaporators are only used when the capacity needed is small. Multi-effect systems recover and reuse the latent heat of the vaporized material which is lost in a single-effect evaporation. In multi-effect evaporator, each effect in itself acts as a single effect evaporator. (Geankoplis, 1993). Multi-effect evaporation systems may be forward-feed operated, backward-feed operated or parallel-feed operated (Smith, 1995).

In forward-feed operation (Figure 24), the fresh feed is added to the first stage and flows to the next effect in the same direction as the vapour flow. The boiling temperature decreases from effect to effect, which means that if the first effect is at ambient pressure, the last effect will be under vacuum. This arrangement is used when the concentrated product is subjected to decomposition at higher temperatures or the feed is hot. (Geankoplis ,1993).

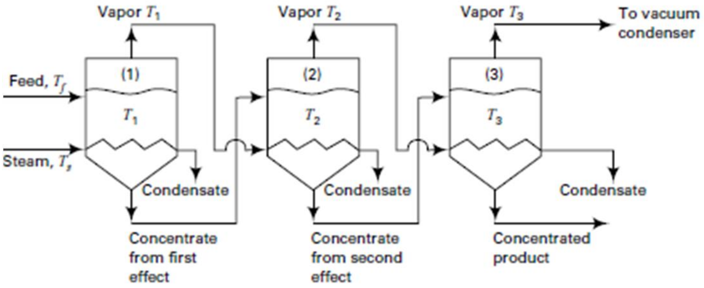


Figure 24. Forward-feed, triple-effect evaporator (Seader, Henley, Roper 2011).

In backward-feed operation (Figure 25), the fresh feed enters the last and coldest effect and leaves the first effect as concentrated product. This method is used when the concentrated product is highly viscous or when the fresh feed is cold. Since the solutions flow against the pressure gradient between stages, pumps must be used to transfer solutions between stages. (Geankoplis, 1993).

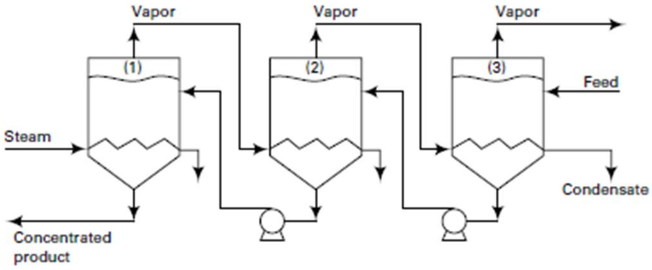


Figure 25. Backward-feed, triple-effect evaporator (Seader, Henley, Roper 2011).

In parallel-feed operation, fresh feed is added to each stage, and product withdrawn from each stage. The vapour from each stage is used to heat the next stage. This operation style is used mainly when the feed is almost saturated, particularly when solid crystals are the product. (Smith, 1995).

5.3.4 Designing

The designing of a multistage evaporation system starts with making a reasonable assessment of the number of stages. Having made this decision, the heat flow through the system is temporarily fixed. The maximum temperature in evaporators is set by the product decomposition and fouling. Therefore, the highest-pressure stage is operated at a pressure low enough to be below this maximum temperature. The pressure of the lowest-pressure stage is normally chosen to allow heat rejection to cooling water or air cooling. If fouling and decomposition are not a problem, then the stage pressures should be chosen such that the highest-pressure stage is below steam temperature and the lowest-pressure stage above cooling water or air cooling temperature. (Smith, 1995).

The chemical and physical properties of the solution being concentrated and of the vapour being removed have a great effect on the type of evaporator and on the pressure and temperature of the process. The heat-transfer coefficient drop markedly as evaporation proceeds since the concentration and viscosity of the concentrated solute increase. As solutions are heated and concentration of the solute or salt increases, the solubility limit of the material in solution may be exceeded and crystals may form. This may limit the maximum concentration in solution which can be obtained by evaporation. (Geankoplis, 1993).

If the solution to be evaporated is dilute, it is justified to assume that the boiling point of the solution is the same as for water. However, in the majority of cases in evaporation the solutions are not such dilute solutions. In most cases, the concentrations of the solutions are high enough so that the heat capacity and boiling point will be greater than that of pure water. This phenomenon is called boiling-point rise. (Geankoplis, 1993).

The inlet temperature of the feed has a large effect on the operation of the evaporator. If the feed entering the evaporator is at a cold temperature compared to the boiling point, a lot of steam is lost for heating the feed. Preheating the feed can reduce the size of evaporator heat-transfer area needed. (Geankoplis, 1993).

In many cases, a large temperature difference between the steam and the vapour space in the evaporator is desired since as the ΔT increases, the heating surface area A and cost of evaporator decrease. Decreasing the pressure in the vapour space of the evaporator, decreases the boiling point temperature, increasing the ΔT and decreasing the heating-surface are. (Geankoplis, 1993).

According to Figure 26 the investment cost is rising and the energy cost is decreasing with an increasing number of effects. The most economical number is given where the total cost is passing a minimum. (Mersmann et al., 2011).

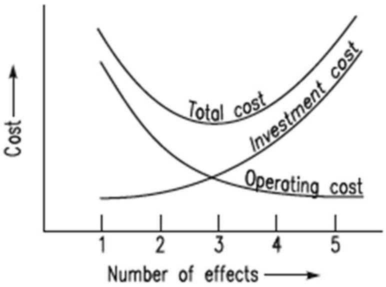


Figure 26. Investment, operating and total costs as a function of the number of effects (Mersmann et al., 2011).

Using higher pressure, saturated steam increases the T, which decreases the cost and size of the evaporator. However, high-pressure steam is more costly and also is often valuable elsewhere. (Geankoplis, 1993).

For a given number of stages, if (1) all heat transfer coefficients are equal, (2) all evaporation and condensation duties are at constant temperature, (3) boiling point rise of the evaporating mixture is negligible, and (4) latent heat is constant through the system, then minimum capital cost is given when all temperature differences are equal. When evaporator pressure is not limited by the steam temperature but by product decomposition and fouling, then the temperature differences should be spread out equally between the upper practical temperature limit and the cold utility. If the feed is cold, preheating the feed by heat integration with the rest of the process might be possible. (Smith, 1995).

5.3.5 Economy

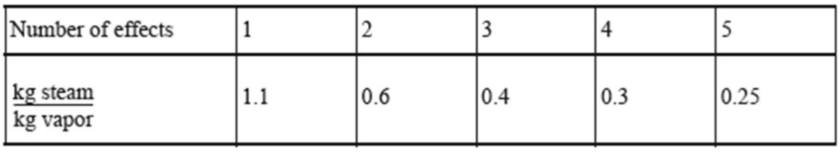
Economy is the number of kilograms vaporized per kilogram of steam fed to the unit. For single effect evaporator economy is almost always less than 1. For multiple-effect evaporator it can be considerable greater. Simple estimation can be done from equation:

$$E = 0.85*N$$

where E is economy and N is number of effects. (Saravacos and Kostaropoulos, 2002).

In industrial plants the specific steam consumptions according Table 12 are obtained (Mersmann et al., 2011).

Table 12. Specific steam consumption in industrial plants (Mersmann et al.,2011).



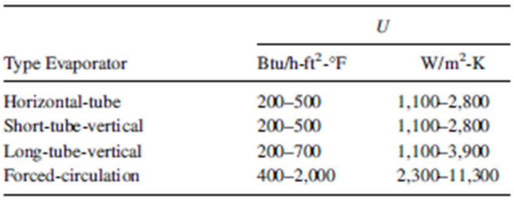
Number of effects	1	2	3	4	5
$\frac{\text{kg steam}}{\text{kg vapor}}$	1.1	0.6	0.4	0.3	0.25

5.3.6 Overall heat transfer coefficient

Approximate design values for overall heat transfer coefficient U can be found from literature. Peters et al. (2003) gives U values for heaters heated by steam for aqueous solutions varies from 500-2500 W/m^2K ($\mu > 2 * 10^{-3}$ Pas) and 1000-3500 W/m^2K ($\mu < 2*10^{-3}$ Pas). Upper range of values given for heaters can be used for evaporators. The type of evaporator also affects the overall heat transfer coefficient, being higher with forced circulation than with natural circulation. (McCabe et al., 1993).

Table 13 lists ranges of overall heat-transfer coefficients for different types of evaporators (Seader et al., 2011).

Table 13. Typical heat-transfer coefficients in evaporators (Seader et al., 2011).



	U	
Type Evaporator	Btu/h-ft^2-°F	W/m^2-K
Horizontal-tube	200–500	1,100–2,800
Short-tube-vertical	200–500	1,100–2,800
Long-tube-vertical	200–700	1,100–3,900
Forced-circulation	400–2,000	2,300–11,300

5.4 Drying of solutions, slurries and pastes

5.4.1 Introduction

Drying is used to separate volatile components (i.e. moisture) from a carrier. In many cases this implies the separation of water. The carrier may be solid, liquid, or gas. Drying of fluids denotes the removal of small amount of water from gases or organic liquids. Adsorption as well as absorption and rectification processes are used for this. (Mersmann et al., 2011). Drying of solid materials is discussed in Chapter 3.11

For drying of solutions, slurries and pastes, spray dryers, drum dryers, vacuum drum dryers, and sometimes fluidized bed dryers are used.

5.4.2 Drum dryers

Solutions, slurries, and pastes may be spread as thin films and dried on steam-heated rotating drums. Drum dryers (Figure 13g) are described in Chapter 3.11.2. For mechanical reasons, drums larger than 5 ft diameter by 12 ft long are impractical. Competitors for drum dryers are solid belt conveyors that can handle greater thicknesses of pasty materials, and also spray dryers that have largely taken over the field. (Couper et al., 2010).

Drum dryers are used for drying of slurries and solutions like buttermilk, skim milk, dextrins, polyacylamides, sodium benzoate, propionates, acetates, phosphates, chelates, aluminum oxide, m-disulfuric acid, barium sulfate, calcium acetate–arsenatecarbonate– hydrate-phosphate, caustic, ferrous sulfate, glue, lead arsenate, sodium benzene sulfonate, and sodium chloride. Vacuum drum dryers are used to dry solutions and slurries like syrups, malted milk, skim milk, coffee, malt extract, and glue. (Couper et al., 2010).

5.4.3 Spray dryers

Spray dryers (Figure 13f) are described in Chapter 3.11.2. Spray dryers are capable of large evaporation rates, 12,000–15,000 lb/hr or so, whereas a 300 sqft drum dryer for instance may have a capacity of only 3000 lb/hr. The completely enclosed operation of spray dryers also is an advantage when toxic or noxious materials are handled. (Couper et al., 2010).

Spray dryers are used to dry solutions and slurries like sulfonates, inorganic phosphates, coffee, detergents, pharmaceuticals, pigments, inks, melamine and urea formaldehyde resins, polyvinyl chloride, microspheres, skim milk, eggs, starch, yeast, silica gel and urea. (Couper et al., 2010).

5.5 Extraction

5.5.1 Introduction

Solvent extraction, also called liquid-liquid extraction, can be used to separate a solute from a solution by extraction into another solvent. The two solvents must be essentially immiscible. Solvent extraction can be used either to recover a valuable substance from the original solution, or to purify the original solvent by removing an unwanted component. (Towler and Sinnott, 2013).

The simplest extraction system is made up of three components: the solute (material to be extracted); the carrier, or nonsolute portion of the feed; and the solvent, which should have a low solubility in the carrier. Figure 27 illustrates a counter current extraction with a light-phase solvent. The diagram can be inverted for a heavy-phase solvent. The carrier-rich liquid leaving the extractor is referred to as the raffinate phase and the solvent-rich liquid leaving the extractor is the extract phase. The solvent may be the dispersed phase or it may be the continuous phase; the type of equipment used may determine which phase is to be dispersed in the other phase. Customarily the phase with the highest volumetric rate is dispersed since a larger interfacial area results in this way with a given droplet size. In equipment that is subject to back mixing, such as spray and packed towers but not sieve tray towers, the disperse phase is made the one with the smaller volumetric rate. (Couper et al. 2010).

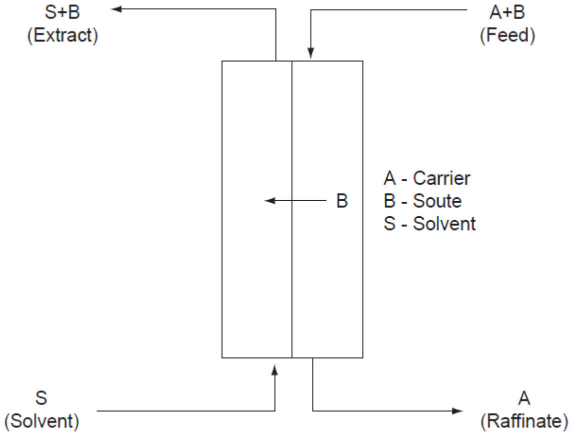


Figure 27. Solvent extraction process (Couper et al., 2010).

The principal scheme of an extraction process is depicted in Figure 28. The feed is brought into intimate contact with the solvent to enhance mass transfer between the two phases. Then the phases are separated. The raffinate is withdrawn from the unit. The loaded solvent (extract) is fed in to a regenerator for further processing. The regenerated solvent is recycled to the extractor. (Mersmann et al., 2011).

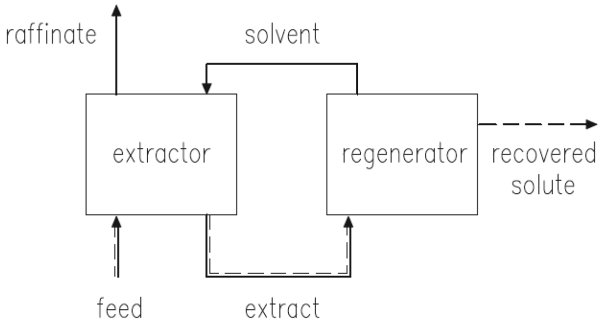


Figure 28. The principal scheme of an extraction process (Mersmann et al., 2011).

The extraction process may have energy saving possibilities, when solvent recovery is easy as the process can be operated in moderate temperatures. However, the solvent must be recovered for reuse and this is usually done by distillation, which complicates the process. (Peters et al., 2003).

The prerequisite of all types of extraction processes is the existence of a large miscibility gap between raffinate and extract. The decisive quantity for phase splitting is the density difference between the phases. As the density of the raffinate is given by the system at hand the solvent has to be selected with respect to density. The density difference must not fall below values of approximately 30–50 kg/m^3. In industrial extraction processes the regeneration of the solvent is often established by distillation. Consequently, a further aspect for solvent selection is an easy separability of transfer component and solvent by distillation. Furthermore, the solvent must not form azeotropes with the transfer component. Important aspects are also costs, availability, chemical stability, and nontoxicity of the solvent. (Mersmann et al., 2011)

5.5.2 Equipment

Extraction equipment can be divided into two broad groups:

1. Stage-wise extractors, in which the liquids are alternately contacted (mixed) and then separated, in a series of stages. The simplest form of extractor, the “mixer-settler” contactor is an example of this type. Several mixer-settlers are often used in series to increase the effectiveness of the extraction.
2. Differential extractors, in which the phases are continuously in contact in the extractor and are only separated at the exits; for example, in packed-column extractors. (Towler and Sinnott, 2013).

In the process industry a great variety of different equipment designs is used in extraction processes. This is due to the fact that the density difference between two liquid phases is very small, typically smaller than $\Delta\rho < 100$ kg/m^3. This small density difference restricts the velocities of the phases to very small values and, in turn, reduces the interfacial mass transfer rates. This problem is solved by putting external motions into the system, for instance, by pulsation or agitation. These externally induced motions are meant to enhance the interfacial mass transfer rates. Three types of solvent extractors can be distinguished: static devices, pulsed devices, and agitated devices. (Mersmann et al., 2011).

5.5.2.1 Static columns

Three examples of static columns operated counter currently are depicted in Figure 29. The spray column does not have any internals. The dispersed phase (droplet phase) is moving counter currently to the continuous phase. Spray columns can only be used if the density difference between the two phases is higher than 150 kg/m^3 and if the required separation efficiency is low. Hence, spray columns are seldom used in extraction. The static packed column is more efficient with respect to mass transfer. Its design is similar to that of gas/liquid systems (e.g., absorption and distillation). In contrast, the design of a static tray column, used for solvent extraction, is completely different from the corresponding column for gas/liquid service. The sieve trays have very small hole diameters (2–4 mm). (Mersmann et al., 2011).

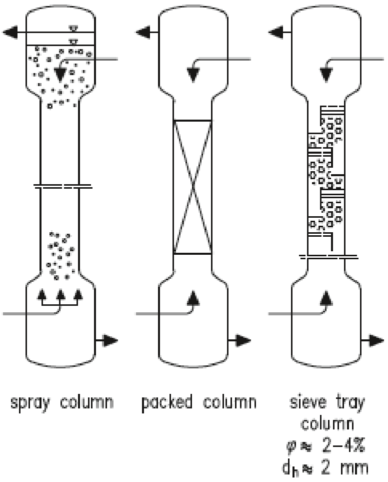


Figure 29. Scheme of some static extraction columns (Mersmann et al., 2011).

5.5.2.2 Pulsed columns

Pulsed columns, depicted in Figure 30, are frequently used in solvent extraction. The design of pulsed packed columns is identical with the design of static packed columns. Just the two-phase liquid hold-up is periodically vertically moved with the frequency f. The pulsation effects a decrease of droplet size and, in turn, an increase of interfacial mass transfer rates. Pulsed sieve tray columns differ significantly from static sieve tray columns as they do not have any down comers. The light and the heavy phase pass periodically through the same holes. Furthermore, pulsed tray columns have a much larger relative free area of up to with hole diameters of 2–4 mm. (Mersmann et al., 2011).

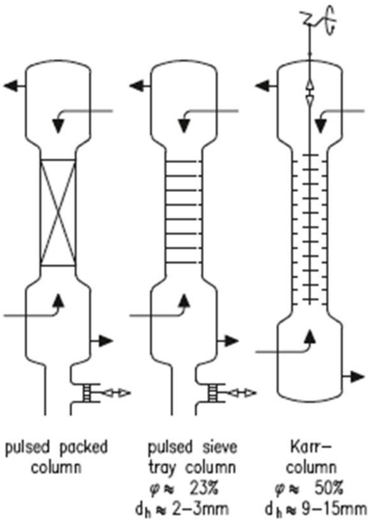


Figure 30. Scheme of some pulsed extraction columns (Mersmann et al., 2011).

5.5.2.3 Agitated extractors

Some examples of agitated extractors are depicted in Figure 31 . The rotating disc contactor (RDC) uses flat disc agitators. A modification of this design is the asymmetrical rotating disc contactor (ARD). Conventional agitator elements are used in the Kühni and the RZE extractors. All agitated extraction columns are equipped with stator rings, arranged between the agitators, to suppress the vertical back mixing of the phases. (Mersmann et al., 2011).

The oldest and most important agitated extractor is, however, the mixer settler. Its design is very simple (agitation and decantation zones). Mixer settler can be built in large units and with many stages. In each stage, the two phases are firstly mixed to form a two-phase system with large interfacial area for good mass transfer. The attached decantation zone is very large and provides a very effective phase splitting. In each stage the two phases move co currently. In a cascade (vertically or horizontally arranged) the two phases move counter currently. (Mersmann et al., 2011).

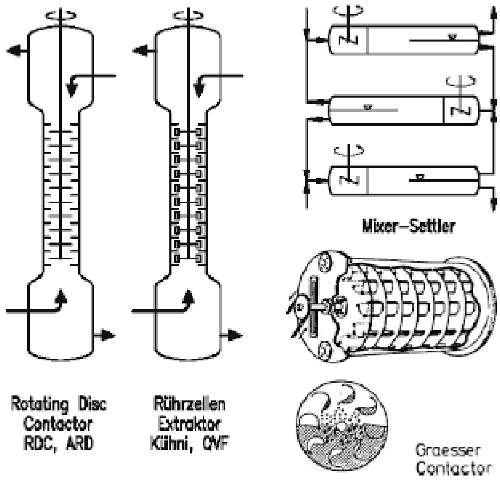


Figure 31. Scheme of some agitated extractors (Mersmann et al., 2011).

5.5.2.4 Selection of equipment

Extraction should be considered as an alternative to distillation in the following situations:

1. When the components in the feed have close boiling points. Extraction in a suitable solvent maybe more economic if the relative volatility is below 1.2.
2. If the feed components form an azeotrope.
3. If the solute is heat-sensitive, and can be extracted into a lower-boiling solvent, to reduce the thermal history during recovery. (Towler and Sinnott, 2013).

The following factors should be taken into consideration when selecting an extractor for a particular application: (1) the number of stages required, (2) The throughputs, (3) the settling characteristics of the phases, and (4) the available floor area and head room. Figure 32 gives a selection guide, which can be used to select the type of equipment most likely to be suitable. (Towler and Sinnott, 2013).

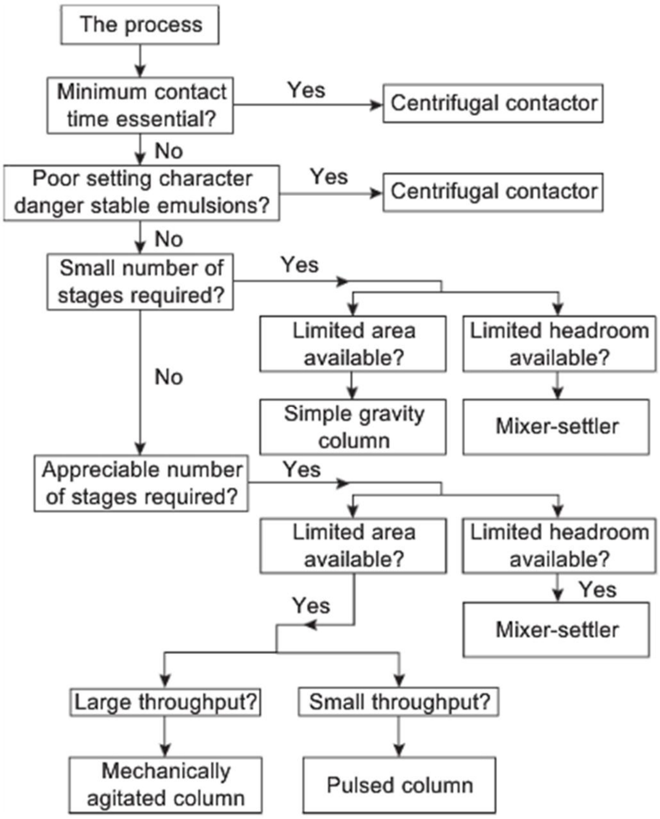


Figure 32 Selection guide for liquid-liquid contactors (Towler and Sinnott, 2013).

5.6 Crystallization/Precipitation

5.6.1 Introduction

Crystallization is used for the production, purification, and recovery of dissolved solids from solutions. Crystalline products have an attractive appearance, are free flowing, and are easily handled and packaged. The process is used in a wide range of industries, from the small-scale production of specialized chemicals, such as pharmaceutical products, to the high tonnage production of commodity products such as sugar, common salt, and fertilizers. (Towler and Sinnott, 2013). Crystallization is an important unit operation because the process of crystallization is capable of producing very high purity products from solutions containing significant amounts of impurities with very low energy input compared to other unit operations such as distillation (Couper et al., 2010).

Crystallization is a solid-liquid separation process in which mass transfer occurs of a solute from the liquid solution to a pure solid crystalline phase. In crystallization solid particles are formed from a homogeneous phase and the solution is concentrated and usually cooled until the solute concentration becomes greater than its solubility at that temperature. Then the solute comes out of the solution forming crystals of approximately pure solute. (Geankoplis, 1993).

In most of the industrial crystallization processes, the solution (mother liquor) and the solid crystals are in contact for a long enough time to reach equilibrium. Hence, the mother liquor is saturated at the final temperature of the process, and the final concentration of the solute in the solution can be obtained from the solubility curve. Solubility is dependent mainly upon temperature, not pressure. The yield of crystals can then be calculated knowing the initial concentration of solute, the final temperature, and the solubility at this temperature. (Geankoplis, 1993).

Crystallization cannot occur without the supersaturation of the mother liquor. Supersaturation can be achieved by cooling a solution or by evaporation of the solvent. This is called cooling and evaporative crystallization. For vacuum crystallization flash evaporation is used to create supersaturation. In this case cooling and evaporation superimpose. (Mersmann et al., 2011).

Sometimes a drowning-out medium is added to a solution. It reduces the solubility of the solute and hence leads to supersaturation. This is called drowning-out crystallization. The solubility of many aqueous solutions of inorganic salts can be reduced by addition of organic solvents (e.g., acetone, methanol). In reactive crystallization two or more reactants form a product which is less soluble and therefore crystallizes. This is called precipitation crystallization. (Mersmann et al., 2011).

5.6.2 Crystallizer types

There are four basic types of crystallizer: (1) tank crystallizers, (2) scraped-surface crystallizers, (3) circulating magma crystallizers, and (4) circulating liquor crystallizers. (Towler and Sinnott, 2013).

Tank crystallizers are the simplest type of industrial crystallizing equipment. Crystallization is induced by cooling the mother liquor in tanks, which may be agitated and equipped with cooling coils or jackets. Tank crystallizers are operated batch-wise, and are generally used for small-scale production. (Towler and Sinnott, 2013).

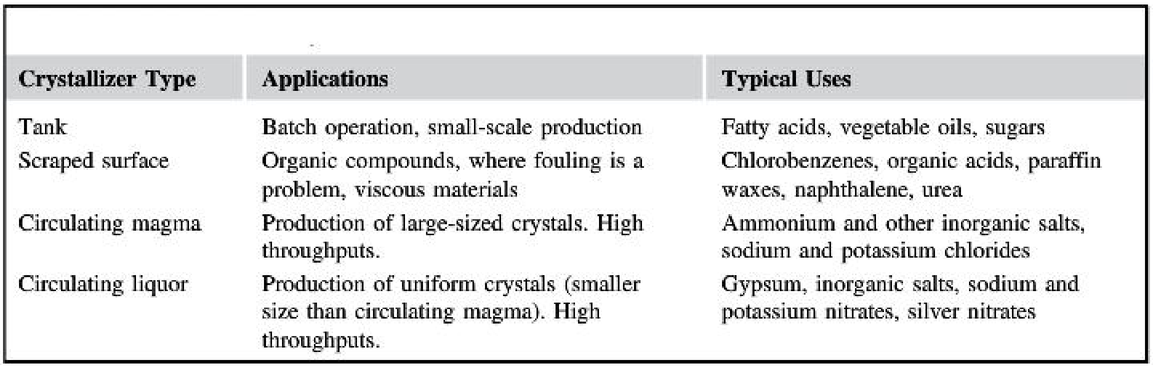
Scraped-surface crystallizers are similar in principle to the tank type, but the cooling surfaces are continually scraped or agitated to prevent fouling by deposited crystals and to promote heat transfer. They are suitable for processing high-viscosity liquors. Scraped-surface crystallizers can be operated batch-wise, with recirculation of the mother liquor, or continuously. A disadvantage of this type is that they tend to produce very small crystals. (Towler and Sinnott, 2013).

In circulating magma crystallizers, both the liquor and growing crystals are circulated through the zone in which supersaturation occurs. They are probably the most important type of large-scale crystallizers used in the chemical process industry. Designs are available in which supersaturation is achieved by direct cooling, evaporation, or evaporative cooling under vacuum. (Towler and Sinnott, 2013).

In a circulating liquor crystallizer only the liquor is circulated through the heating or cooling equipment; the crystals are retained in suspension in the crystallizing zone by the upflow of liquor. Circulating liquor crystallizers 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. (Towler and Sinnott, 2013).

Typical applications of the main types of crystallizer are summarized in Table 14.

Table 14. Selection of crystallizers (Towler and Sinnott, 2013).



Crystallizer 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

5.6.3 Cooling crystallization

In crystallizers producing supersaturation by cooling, the substances must have a solubility curve that decreases markedly with temperature. This occurs for many substances, and this method is commonly used. (Geankoplis, 1993). Aqueous solutions of potassium, sodium, and ammonium nitrate as well as copper sulfate are typical examples. The hot, undersaturated solution is fed into the crystallizer and then cooled. (Mersmann et al., 2011).

It is advantageous to adjust the cooling rate such that the supersaturation is kept roughly constant during the cooling process, i.e. cooling effect must be increased as the crystallization proceeds (Mersmann et al., 2011). Energy consumption in cooling crystallization is smaller than in evaporative crystallization.

Typical crystallizing equipment are tank and batch type crystallizers (Geankoplis, 1993). Batch crystallizers are used primarily for production of fine chemicals and pharmaceuticals at the rate of 1-100 tons/week. The one exception is the sugar industry that still employs batch vacuum crystallization on a very large scale. (Couper et al., 2010). Although batch crystallizers can be operated without agitation or circulation, it is more preferable that they are equipped with circulation through an external, cooling heat exchanger (Couper et al., 2010).

5.6.4 Evaporative crystallization

When the solubility curve changes little with temperature, evaporation of the solvent to produce supersaturation is often used (Geankoplis, 1993). Typical systems for this are aqueous solutions of sodium chloride, ammonium sulphate, and potassium sulphate. The undersaturated solution is fed into the crystallizer and heated up to the boiling point, so that the solvent evaporates (Mersmann et al., 2011). Typical crystallizing equipment are evaporator-crystallizers and multistage crystallizing evaporators (Geankoplis, 1993). Natural circulation evaporators equipped with continuous salt removal are adapted to crystallization service. For large production rates, however, forced circulation evaporators are the most often used. The lower limit for economic continuous operation is 1-4 tons/day of crystals, and the upper limit in a single vessel is 100-300 tons/day. (Couper et el., 2010).

5.6.5 Precipitation

Precipitation can be considered as a branch of crystallization, although the solid phase that is formed need not be crystalline. The solubility of organic solutes can be influenced by temperature, composition, pH, solvent polarity, and ionic strength. If something is added to the solvent to change one or more of these properties, the solute can sometimes be precipitated out of the solution. Some of the common techniques that are used include: (Towler and Sinnott, 2013)

(1) Salting out, in which a salt is added to an aqueous solution to raise the ionic strength and cause precipitation.
(2) Changing solution polarity by adding a suitable solvent.
(3) Changing the pH by adding acid or base.
(4) Heat treatment (“cooking”) to thermally degrade an unwanted solute that is then precipitated.
(5) Adsorption precipitation by adding large particles (casein, gelatin) that can adsorb the organic species and then settle as precipitate.

Precipitation operations usually do not require evaporation of solvent or cooling of a saturated solution, and so can be carried out in simpler equipment than crystallization. The process usually consists of a mixing tank or inline mixer, followed by a solid-liquid separation device such as a hydrocyclone or centrifuge. (Towler and Sinnott, 2013).

5.7 Membranes

5.7.1 Introduction

To define a membrane is quite challenging. In the most general sense, a synthetic membrane is a barrier which separates two phases and restricts the transport of various chemical species in a rather specific manner. (Porter, 1990). The membrane has the ability to transport one component more readily than other because of differences in physical and/or chemical properties between the membrane and the permeating components (Mulder, 1996). A membrane may be homogeneous or heterogeneous, symmetric or asymmetric in structure; it may be solid or liquid; it may be neutral, or carry charges, or be bipolar. Its thickness varies between less than 100 nm to more than a centimetre. (Porter, 1990).

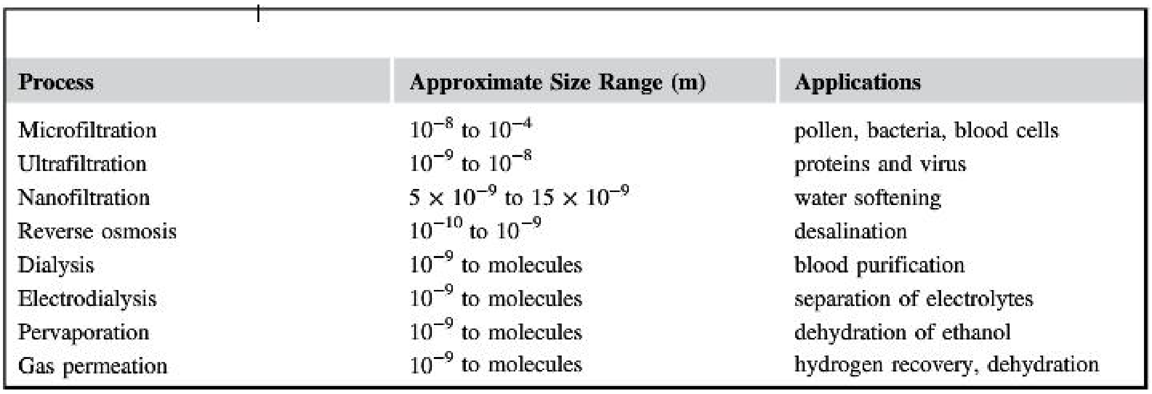
Membranes are widely used for concentration of solutions of dissolved solids, as well as suspensions of particulates. The use of membrane for solvent removal is usually preferred over solvent evaporation if the solute is sensitive to high temperatures. This is often the case for biologically active large molecules such as proteins and enzymes and for flavour compounds found in foods and beverages. (Towler and Sinnott, 2013).

In membrane processes the feed stream is divided into two streams, i.e. into retentate or concentrate stream and the permeate stream with either the concentrate or permeate stream is the product. If the aim is concentration, the retentate will usually be the product stream. In the case of purification, both the retentate or the permeate can yield the desired product depending on the impurities that have to be removed. (Mulder, 1996).

Mass transport through a membrane may be caused by convection or by diffusion of individual molecules induced by an electric field, or a concentration, pressure or temperature gradient. The transport rate is determined by the driving force or forces acting on the individual components and their mobility and concentration within the interphase. The mobility is primarily determined by the solute’s molecular size and the physical structure of the interphase material, while the concentration of the solute in the interphase is determined by chemical compatibility of the solute and the interphase material, the solute’s size, and the membrane structure. The mobility and concentration of the solute within the interphase determine how large a flux is produced by a given driving force. (Porter, 1990).

Membrane processes can be divided into first generation membrane processes such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO), electrodialysis (ED), membrane electrolysis (ME), diffusion dialysis (DD), and dialysis and into second generation membrane processes such as gas separation (GS), vapour permeation (VP), pervaporation (PV), membrane distillation (MD), membrane contactors (MC) and carrier mediated processes. (Mulder, 1996). Table 15 lists some membrane filtration processes (Towler and Sinnott, 2013).

Table 15. Membrane filtration processes (Towler and Sinnott, 2013).



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

In most the membrane processes the driving force is a pressure difference or a concentration difference across the membrane. Driving forces through the membrane may also be temperature gradient or electrical potential difference. An overview of various membrane processes and driving forces are listed in Table 16. (Mulder, 1996).

Table 16. Some membrane processes and driving forces (Mulder, 1996).

Membrane process	phase 1	phase 2	driving force
microfiltration	L	L	Δp
ultrafiltration	L	L	Δp
nanofiltration	L	L	Δp
reverse osmosis	L	L	Δp
piezodialysis	L	L	Δp
gas separation	G	G	Δc/ Δp
vapour permeation	G	G	Δc/ Δp
pervaporation	L	G	Δc/ Δp
electrodialysis	L	L	ΔE
membrane dialysis	L	L	ΔE
dialysis	L	L	Δc/ Δp
diffusion dialysis	L	L	Δc/ Δp
membrane contactors	L	L	Δc
	G	L	Δc/ Δp
	L	G	Δc/ Δp
thermos-osmosis	L	L	ΔT/ Δp
membrane distillation	L	L	ΔT/ Δp

5.7.2 Membrane modules

There are three kinds of membrane shapes; flat sheets (Figure 33a), tubular membranes (Figure 33b) and hollow fibres(Figure 33c) (Seader et al., 2011).

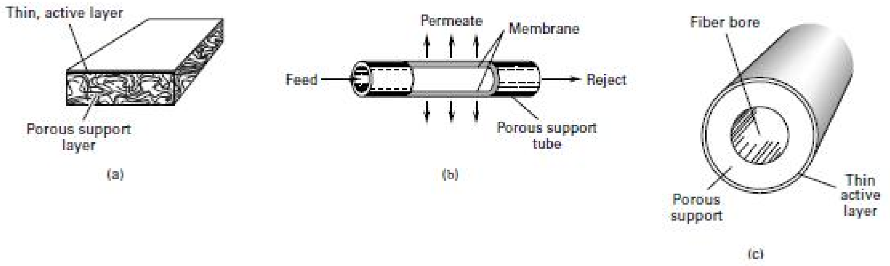


Figure 33. Common membrane shapes: (a) flat, asymmetric or thin-film composite sheet; (b) tubular; (c) hollow-fibre (Seader et al., 2011).

The shapes in Figure 33 are incorporated into modules and cartridges, some of which are shown in Figure 34. Flat sheets used in plate-and-frame modules are circular, square, or rectangular in cross section. The sheets are separated by support plates that channel the permeate. Flat sheets are also fabricated into spiral-wound modules, as in Figure 34b. A laminate, consisting of two membrane sheets separated by spacers for the flow of the feed and permeate, is wound around a central, perforated collection tube to form a module that is inserted into a pressure vessel. Feed flows axially in the channels created between the membranes by porous spacers. Permeate passes through the membrane, traveling inward in a spiral path to the central collection tube. From there, the permeate flows in either axial direction through and out of the tube. The four-leaf modification in Figure 34c minimizes the permeate pressure drop because the permeate travel is less for the same membrane area. The hollow-fibre module in Figure 34d, for a gas-permeation application, resembles a shell-and-tube heat exchanger. The pressurized feed enters the shell side at one end. While flowing over the fibres toward the other end, permeate passes through the fibre walls into the central fibre channels. Typically, the fibres are sealed at one end and embedded into a tube sheet with epoxy resin at the other end. The tubular module in Figure 34e also resembles a heat exchanger, but the feed flows through the tubes. Permeate passes through the tube wall into the shell side of the module. Tubular modules contain up to 30 tubes. (Seader et al., 2011)

Table 17 is a comparison of the characteristics of four of the modules shown in Figure 34. The packing density is the membrane surface area per unit volume of module, for which hollow-fibre membrane modules are clearly superior. Although the plate-and-frame module has a high cost and a moderate packing density, it finds use in all membrane applications except gas permeation. It is the only module widely used for pervaporation. The spiral-wound module is very popular for most applications because of its low cost and reasonable resistance to fouling. Tubular modules are used only for low-flow applications or when resistance to fouling and/or ease of cleaning is essential. Hollow-fibre modules, with their very high packing density and low cost, are popular where fouling does not occur and cleaning is not necessary. (Seader et al., 2011).

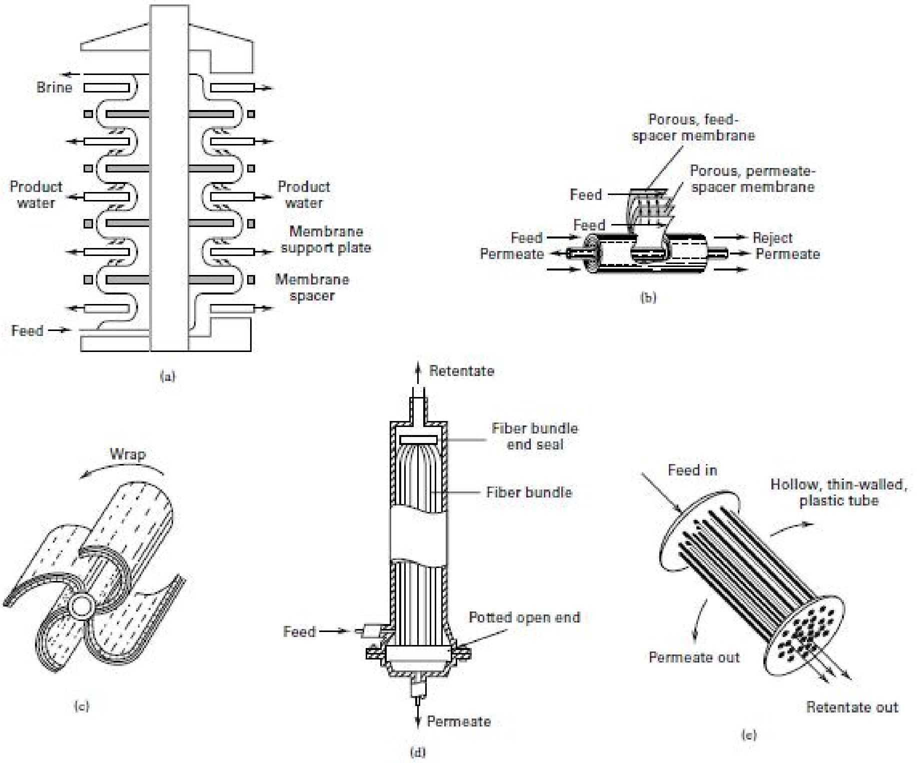


Figure 34. Common membrane modules: (a) plate-and-frame, (b) spiral-wound, (c) four-leaf spiral-wound, (d) hollow-fibre, (e) tubular (Seader et al., 2011).

Table 17. Typical Characteristics of Membrane Modules (Seader et al., 2011).

	Plate-and-Frame	Spiral-Wound	Tubular	Hollow-Fiber
Packing density, m^2/m^3	30 to 500	200 to 800	30 to 200	500 to 9,000
Resistance to fouling	Good	Moderate	Very good	Poor
Ease of cleaning	Good	Fair	Excellent	Poor
Relative cost	High	Low	High	Low
Main applications	D, RO, PV, UF, MF	D, RO, GP, UF, MF	RO, UF	D, RO, GP, UF

Note: D, dialysis; RO, reverse osmosis; GP, gas permeation; PV, pervaporation; UF, ultrafiltration; MF, microfiltration.

6. Selecting the appropriate separation method

6.1 Phase conditions

Almost all chemical processes include separation of mixtures of components. Separation can be involved in almost any part of process. A separation system may be needed to purify feed stream or product stream. Also an effluent separation system may be needed.

When selecting the separation method phase condition is to be considered first. In

Table 18, a guide for selection of a suitable separation process is given. In the guide, the separation processes have been grouped based on the phases that are separated.

Table 18. A guide for selection of separation process based on the phases that are separated (Towler and Sinnott, 2013).

		MINOR COMPONENT		
		Solid	Liquid	Gas/Vapour
MAJOR COMPONENT	Solid	Sorting Screening Hydrocyclones Classifiers Jigs Tables Centrifuges Dense media Flotation Magnetic Electrostatic	Pressing Drying	
	Liquid	Thickeners Clarifiers Hydrocyclones Filtration Centrifuges Crystallizers Evaporators Precipitation Membranes Reverse osmosis Ion exchange Adsorption	Decanters Coalescers Solvent extraction Leaching Chromatography Distillation	Stripping
	Gas/Vapour	Gravity settlers Impingement separators Cyclones Filters Wet scrubbers Electrostatic precipitators	Separating vessels Demisting pads Cyclones Wet scrubbers Electrostatic precipitators	Adsorption Absorption Membranes Cryogenic distillation Condensation
The terms major and minor component only apply where different phases are to be separated, i.e., not to those on the diagonal.				

6.2 Stream conditions, technological maturity and economics

Key factors affecting the selection of feasible separation method are listed in Figure 35. These deal with feed and product conditions, property differences, characteristics of the candidate separation operations, and economics. (Seader et al. 2011).

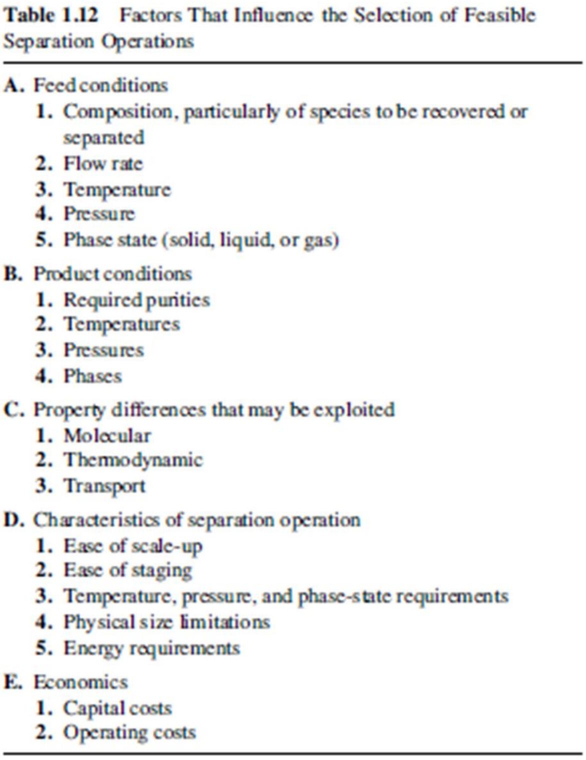


Table 1.12 Factors That Influence the Selection of Feasible Separation Operations

A. Feed conditions
  1. Composition, particularly of species to be recovered or separated
  2. Flow rate
  3. Temperature
  4. Pressure
  5. Phase state (solid, liquid, or gas)

B. Product conditions
  1. Required purities
  2. Temperatures
  3. Pressures
  4. Phases

C. Property differences that may be exploited
  1. Molecular
  2. Thermodynamic
  3. Transport

D. Characteristics of separation operation
  1. Ease of scale-up
  2. Ease of staging
  3. Temperature, pressure, and phase-state requirements
  4. Physical size limitations
  5. Energy requirements

E. Economics
  1. Capital costs
  2. Operating costs

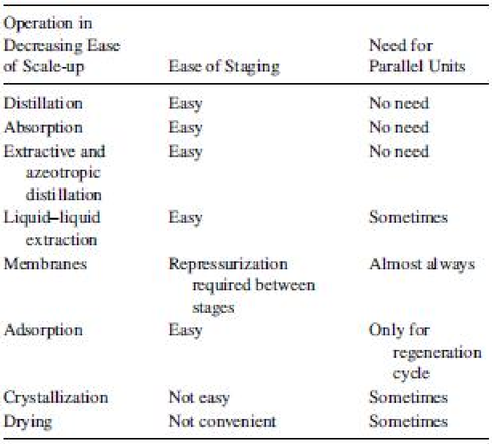
Figure 35. Key factor for selecting of feasible separation method(s). Adopted from Seader et al., 2011.

The most important feed conditions are composition and flow rate, because the other conditions (temperature, pressure, and phase) can be altered to fit a particular operation. However, feed vaporization, condensation of a vapour feed, or compression of a vapor feed can add significant energy costs to chemical processes. Some separations, such as those based on the use of barriers or solid agents (Chapters 4.3 and 4.4), perform best on dilute feeds. (Seader et al., 2011).

The most important product conditions are purities because the other conditions listed can be altered by energy transfer after the separation is achieved. When a very pure product is required, large differences in volatility or solubility or significant numbers of stages are needed for chemicals in commerce. For biochemicals, especially proteins, very expensive separation methods may be required. Accurate molecular and bulk thermodynamic and transport properties are also required. (Seader et al., 2011).

Operations based on barriers (Chapter 4.3) are more expensive than operations based on the use of a solid agent (Chapter 4.4) or the creation or addition of a phase (Chapter 4.2). All separation equipment is limited to a maximum size. For capacities requiring a larger size, parallel units must be provided. Except for size constraints or fabrication problems, capacity of a single unit can be doubled, for an additional investment cost of about 60%. If two parallel units are installed, the additional investment is 100%. In Table 19, there is an indication of the ease of providing multiple stages and whether parallel units may be required. Maximum equipment size is determined by height limitations, and shipping constraints unless field fabrication is possible and economical. (Seader et al., 2011).

Table 19. Ease of Scale-up of the Most Common Separation Operations (Seader et al., 2011).



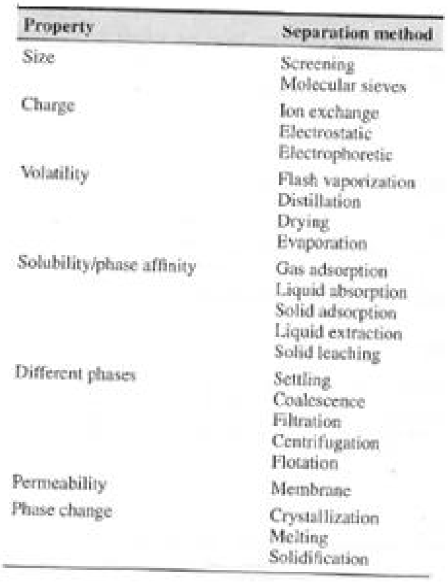
Operation in Decreasing Ease of Scale-up	Ease of Staging	Need for Parallel Units
Distillation	Easy	No need
Absorption	Easy	No need
Extractive and azeotropic distillation	Easy	No need
Liquid–liquid extraction	Easy	Sometimes
Membranes	Repressurization required between stages	Almost always
Adsorption	Easy	Only for regeneration cycle
Crystallization	Not easy	Sometimes
Drying	Not convenient	Sometimes

6.3 Molecular, thermodynamic and transport properties

The extent of separation possible depends on the exploitation of differences in molecular, thermodynamic, and transport properties of the species. Molecular properties include molecular weight, Van der Waals volume, Van der Waals area, molecular shape (acentric factor), dipole moment, polarizability, dielectric constant, electric charge and radius of gyration. Thermodynamic and transport properties include vapour pressure, volatility, solubility, adsorptivity and diffusivity. Values of these properties appear in reference books, handbooks, and journals. Many can be estimated using process simulation programs. When property values are not available, they must be estimated or determined experimentally if a successful application of the separation operation is to be achieved. (Seader et al. 2011).

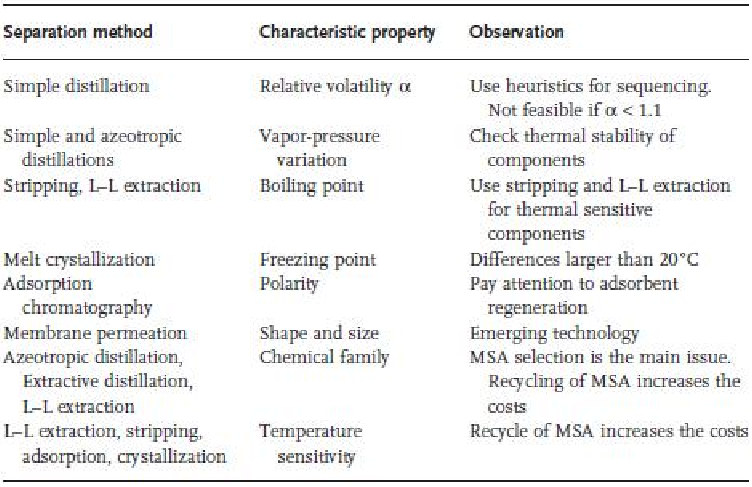
Peters et al., (2003) and Dimian and Bildea (2008) have listed some characteristic physical properties and corresponding separation methods in Table 20 and in Table 21.

Table 20. Selecting a separation method based on physical properties (Peters et al., 2003).



Property	Separation method
Size	Screening Molecular sieves
Charge	Ion exchange Electrostatic Electrophoretic
Volatility	Flash vaporization Distillation Drying Evaporation
Solubility/phase affinity	Gas adsorption Liquid absorption Solid adsorption Liquid extraction Solid leaching
Different phases	Settling Coalescence Filtration Centrifugation Flotation
Permeability	Membrane
Phase change	Crystallization Melting Solidification

Table 21. Selecting a separation method based on physical properties (Dimian and Bildea, 2008).



Separation method	Characteristic property	Observation
Simple distillation	Relative volatility α	Use heuristics for sequencing. Not feasible if α < 1.1
Simple and azeotropic distillations	Vapor-pressure variation	Check thermal stability of components
Stripping, L–L extraction	Boiling point	Use stripping and L–L extraction for thermal sensitive components
Melt crystallization	Freezing point	Differences larger than 20°C
Adsorption chromatography	Polarity	Pay attention to adsorbent regeneration
Membrane permeation	Shape and size	Emerging technology
Azeotropic distillation, Extractive distillation, L–L extraction	Chemical family	MSA selection is the main issue. Recycling of MSA increases the costs
L–L extraction, stripping, adsorption, crystallization	Temperature sensitivity	Recycle of MSA increases the costs

6.4 Rules of thumb

Peters et al., (2003) have also listed some guidelines for selection separation technology. These include:

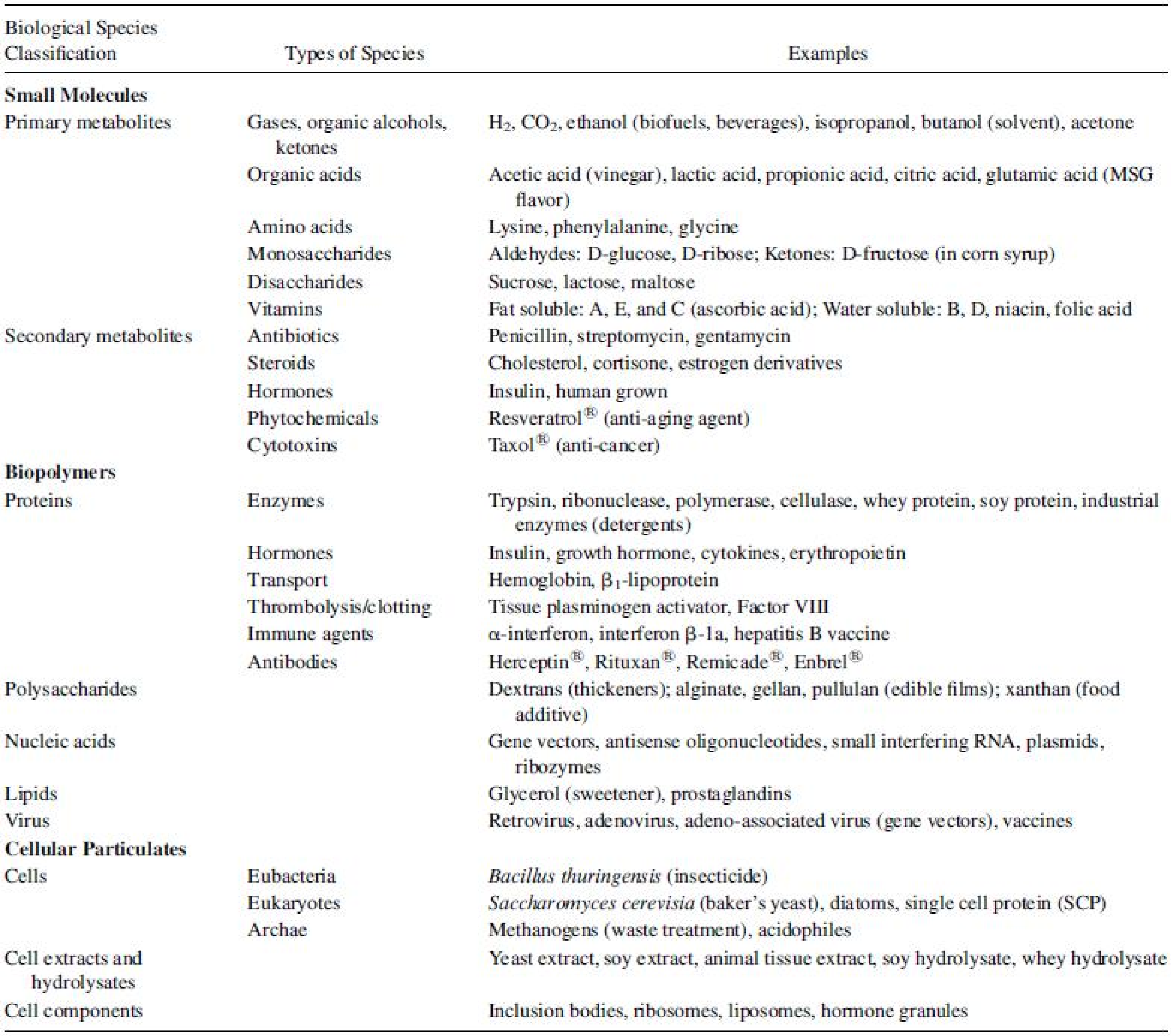
1. Use distillation if the relative volatility of key components is greater than 1.2. Avoid vacuum distillations. Distillation suits for materials whose boiling points are not too low or high or too closed to each other.
2. Favor known techniques, such as distillation, filtration, and extraction.
3. Avoid introducing foreign species; but if they are used remove them immediately after use.
4. Favor energy over mass separation technologies.
5. Don’t perform unnecessary separations; there are no use to separate components that are wanted to the same stream.
6. Separate corrosive and reactive components first.
7. Perform challenging separations last; they are easier to perform when other disturbing components are removed and the flows are smaller.
8. Separate the component with highest content first; stream flows to be handled are smaller.
9. Favor separations where product flows are equal (e.g. 50/50).

The selection of separation techniques for both homogeneous and heterogeneous phases, with many examples, is given by Woods (1995). Couper et al. (2010) have listed rules of thumb for 27 most common separation processes. Such rules are of value for approximate design and preliminary cost estimation. Simian and Bildea (2008) have designed a procedure for selecting the appropriate separation sequence.

Ultimately, the process having the lowest operating, maintenance, and capital costs is selected, provided it is controllable, safe, nonpolluting, and capable of producing products that meet specifications (Seader et al., 2011).

7. Separation of biocomponents

Bioproducts are products extracted from plants, animals, and microorganisms to sustain life and promote health, support agriculture and chemical enterprises, and diagnose and remedy disease (Seader et al., 2011). In Figure 36, products from bioseparation are listed.



Biological Species Classification	Types of Species	Examples
Small Molecules		
Primary metabolites	Gases, organic alcohols, ketones	H_2, CO_2, ethanol (biofuels, beverages), isopropanol, butanol (solvent), acetone
	Organic acids	Acetic acid (vinegar), lactic acid, propionic acid, citric acid, glutamic acid (MSG flavor)
	Amino acids	Lysine, phenylalanine, glycine
	Monosaccharides	Aldehydes: D-glucose, D-ribose; Ketones: D-fructose (in corn syrup)
	Disaccharides	Sucrose, lactose, maltose
	Vitamins	Fat soluble: A, E, and C (ascorbic acid); Water soluble: B, D, niacin, folic acid
Secondary metabolites	Antibiotics	Penicillin, streptomycin, gentamycin
	Steroids	Cholesterol, cortisone, estrogen derivatives
	Hormones	Insulin, human grown
	Phytochemicals	Resveratrol® (anti-aging agent)
	Cytotoxins	Taxol® (anti-cancer)
Biopolymers		
Proteins	Enzymes	Trypsin, ribonuclease, polymerase, cellulase, whey protein, soy protein, industrial enzymes (detergents)
	Hormones	Insulin, growth hormone, cytokines, erythropoietin
	Transport	Hemoglobin, β_1-lipoprotein
	Thrombolysis/clotting	Tissue plasminogen activator, Factor VIII
	Immune agents	α-interferon, interferon β-1a, hepatitis B vaccine
	Antibodies	Herceptin®, Rituxan®, Remicade®, Enbrel®
Polysaccharides		Dextrans (thickeners); alginate, gellan, pullulan (edible films); xanthan (food additive)
Nucleic acids		Gene vectors, antisense oligonucleotides, small interfering RNA, plasmids, ribozymes
Lipids		Glycerol (sweetener), prostaglandins
Virus		Retrovirus, adenovirus, adeno-associated virus (gene vectors), vaccines
Cellular Particulates		
Cells	Eubacteria	*Bacillus thuringensis* (insecticide)
	Eukaryotes	*Saccharomyces cerevisia* (baker's yeast), diatoms, single cell protein (SCP)
	Archae	Methanogens (waste treatment), acidophiles
Cell extracts and hydrolysates		Yeast extract, soy extract, animal tissue extract, soy hydrolysate, whey hydrolysate
Cell components		Inclusion bodies, ribosomes, liposomes, hormone granules

Figure 36. Some products from bioseparation. (adobted from Seader et al., 2011)

Many industrial chemical separation processes have been adapted for use in bioproduct separation and purification. Specialized needs for adaptation arise from features unique to recovery of biological species. Complex biological feedstocks must often be processed at *moderate temperatures*, with *low shear* and *minimal gas–liquid interface* creation, in order to maintain activity of labile biological species. Steps in a recovery sequence to remove biological species from the feed, which complexity and size has broad range, are often determined by unique cell characteristics. For example, extracellular secretion of product may eliminate the need for cell disruption. The value per gram of some bioproducts, for example biopharmaceutical products relative to commodity chemicals is higher due to the higher specific activity of the former in vivo. This permits cost-effective use of highresolution chromatography or other specialized operations that may be cost-prohibitive for commodity chemical processes. (Seader et al. 2011)

The structure of bioproducts are often complex. For example, the structure of food is by no means simple and for the most foods principal component is water, which plays an important role in the overall behaviour of the food. One of the most important branches of separation in food industry is the removal of water, to save transportation costs and improve microbial stability. The other components can be classified into major components, such as protein, fat or lipid, sugars, starch and fibre. The minor components include the minerals, which are known collectively as ash, vitamins and organic acids. (Grandison and Lewis, 1996).

Some examples what is removed from milk is listed below (Grandison and Lewis, 1996).

- water removal to produce evaporated or dried products
- fat separation to produce creams and butter
- protein separation to produce cheese or protein concentrates
- calcium removal to improve stability
- lactose removal, as a specialized ingredient or for low-lactose products
- removal of components responsible for tainting raw milk or the cooked flavor of
- heat-treated milk products
- removal of radionuclides from milk

There are limitations with biological materials: higher temperatures increase degradation reactions, causing colour and flavour changes, enzyme inactivation, protein denaturation, loss of, functionality and a reduction in nutritional value. Safety issues, such as microbial growth and contamination may also need to be considered. Figure 37 gives examples of physical properties and separation processes exploiting these properties. (Grandison and Lewis, 1996).

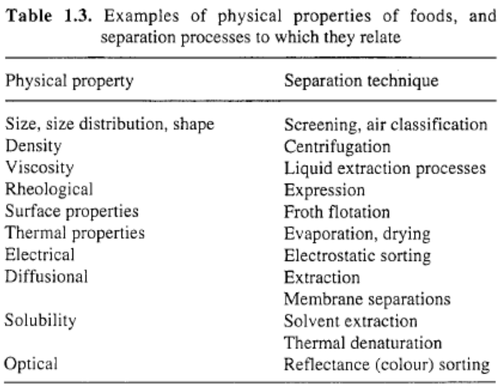


Table 1.3. Examples of physical properties of foods, and separation processes to which they relate

Physical property	Separation technique
Size, size distribution, shape	Screening, air classification
Density	Centrifugation
Viscosity	Liquid extraction processes
Rheological	Expression
Surface properties	Froth flotation
Thermal properties	Evaporation, drying
Electrical	Electrostatic sorting
Diffusional	Extraction Membrane separations
Solubility	Solvent extraction Thermal denaturation
Optical	Reflectance (colour) sorting

Figure 37. Examples of physical properties of foods and separation processes exploiting these properties. (Adobted from Grandison and Lewis, 1996)

8. Modelling of separation processes with simulation softwares

8.1 SuperProDesigner

1.1.1 Unit processes for filtration include

- Microfiltration / Ultrafiltration / Reverse Osmosis / Diafiltration Batch Procedure
- Microfiltration / Ultrafiltration / Reverse Osmosis Continues Procedure
- Cake Drying (in a Nutsche Filter)
- Rotary Vacuum Filtration
- Air Filtration
- Belt Filtration
- Granular Media Filtration

- Baghouse Filtration
- Electrostatic Precipitation

1.1.2 Unit operations for drying include

- Tray Drying
- Freeze Drying
- Discrete Freeze Drying
- Drying
- Granulation
- Spray Drying
- Fluid Bed Drying
- Drum Drying

Rotary Drying

8.2 Aspen

8.2.1 Mixers/Splitters

- Mixer
    - Stream mixer
    - Combines material/heat/work streams into one outlet stream.
    - Specification
        - Optional water decant stream to decant free water from the outlet
        - An outlet pressure
        - Pressure drop
    - Performs an adiabatic phase equilibrium flash calculation on the composite feed streams.
- FSplit
    - Stream splitter
    - Combines material/heat/work feed streams and then divides the resulting stream into two or more outlet streams based on user's specifications.
    - All the outlets have the same composition and properties.
    - Specification
        - Specify all but one outlet streams.
- Separators
    - Flash2
        - Two-outlet flash
        - Determines the thermal and phase conditions of a mixture with one or more inlet streams.
        - Performs adiabatic, isothermal and isobaric phase equilibrium flash calculations, and dew or bubble points.
        - Performs rigorous 2 (V-L) or 3 (V-L-L) phase equilibrium calculations.
        - Produces one V outlet stream, one L outlet stream, and an optional water decant stream.
        - Use to model flashes, evaporators, knock-out drums, and any other single-stage separators, with sufficient vapour disengagement space.
        - Specifications
            - A combination of any two of
                - Temperature
                - Pressure

      - Heat duty
      - Molar vapour fraction
      - The combination of heat duty and molar vapour fraction is not allowed in the flash model.
    - A percentage of the liquid phase to be entrained in the vapour stream.
- Flash3
  - Three-outlet flash
  - Determines the thermal and phase conditions of a mixture with one or more inlet streams.
  - Performs adiabatic, isothermal and isobaric phase equilibrium flash calculations, and dew or bubble points.
  - Performs rigorous 3 (V-L-L) phase equilibrium calculations.
  - Produces one V outlet stream and two L outlet stream.
  - Use to model any single-stage separator with sufficient vapour-liquid disengagement space as well as two liquid phase settling space.
  - Specifications
    - A combination of any two of
      - Temperature
      - Pressure
      - Heat duty
      - Molar vapour fraction
      - The combination of heat duty and molar vapour fraction is not allowed in the flash model.
      -
    - Entrainment of each liquid phase in the vapour stream.
  - Use instead of Decanter model, if you don´t know whether there is a vapour phase.
- Decanter
  - Liquid-liquid decanter
  - Use to model knock-out drums, decanters, and other single-stage separators with sufficient residence time for separation of two liquid phases but without a vapour phase.
  - Determines the thermal and phase conditions of a mixture with one or more inlet streams, at the specified temperature or heat duty.
  - Model assumes implicitly that there is no vapour phase formation; if there is use Flash3 model.
  - Specifications
    - Temperature
    - Heat duty
- Sep
  - Multi outlet component separator
  - Combines feed streams and then separates the resulting stream into two or more streams, according to splits user specify for each substream.
  - Used for component separation operations such as distillation column or absorption when fractionating achieved or desired by the column is known but the details of the column energy balance are unknown or unimportant.
  - Specification
    - The splits for each component in each substream.

- Sep2
    - Two-outlet component separator
    - Combines feed streams and then separates the resulting stream into two outlet streams based on user´s specifications.
    - Similar to Sep, but offers a wider variety of specifications, such as component purity or recovery.
    - Used for component separation operations such as distillation column or absorption when fractionating achieved or desired by the column is known but the details of the column energy balance are unknown or unimportant.
    - Specifications
        - The splits for each component in each substream.
        - Component purity in each substream.
        - Component recovery in each substream.

8.2.2 Columns

- DSTWU
    - shortcut distillation design model
    - Performs a Winn-Underwood-Gilliand shortcut design calculation for a single-feed, two-product distillation column, with a partial or total condenser.
    - For single columns
    - Perform free-water calculations in the condenser
    - Allows the use of water decant streams to decant free-water from the condenser.
    - Estimates the minimum of either
        - Reflux ratio
        - Number of theoretical stages
    - Estimates either
        - Reflux ratio given the number of theoretical stages or
        - Number of theoretical stages given the reflux ratio.
    - Estimates
        - Optimum feed stage location
        - Condenser and reboiler duties.
    - Specifications
        - Recovery of the light and heavy key components in the product streams.
- Distl
    - Shortcut multicomponent distillation rating model.
    - Uses the Edminster approach to separate an inlet stream into two products
    - For single columns
    - Performs free-water calculations in the condenser.
    - Allows the use of water decant streams to decant free-water from the condenser.
    - Estimates
    - The condenser and reboiler duties.
    - Specifications
        - Number of theoretical stages
        - Reflux ratio
        - Overhead product rate
        - Either a total or partial condenser.
- RadFrac
    - Rigorous multi-stage distillation
    - Choose from several common tray types, random or structured packings.

    - For all types of multistage vapour-liquid fractionation operations.
    - Simulates
        - Absorption
        - Reboiled absorption
        - Stripping
        - Reboiled stripping
        - Extractive and azeotropic distillation
    - Suitable for
        - Three-phase systems
        - Narrow-boiling and wide-boiling systems
        - Systems exhibiting strong liquid phase nonideality
    - Handles a free-water phase or other second liquid phase anywhere in the column.
    - Decants free water from the condenser.
    - Handles solids on every stage.
    - Models columns where chemical reactions are occurring.
    - Can model salt precipitation.
    - Operates in
        - rating mode
            - Rating mode calculates
                - Temperature
                - Flow rate
                - Mole fraction profiles
            - Specifications
                - Reflux ration
                - Product rates
                - Heat duties
                - Component or stage efficiencies
        - Design mode
            - Specifications
                - Temperatures
                - Flow rates
                - Purities
                - Recoveries
                - Stream properties (volume flow, viscosity, etc)
- Extract
    - Rigorous model for simulating liquid-liquid extractors.
    - Only for rating calculations.
    - Can have multiple feeds, heaters/coolers, and sidestreams.
    - Specifications
        - Component efficiencies
        - Stage efficiencies
- MultiFrac
    - Rigorous multi-stage distillation for complex columns
    - Models a complex configuration of
        - Any number of columns, each with any number of stages
        - Any number of connections between columns or within columns
        - Arbitrary flow splitting and mixing of connecting streams.
    - Handles
        - Side strippers

        - Pumparounds
        - Bypasses
        - External heat exchangers
        - Single-stage flashes
        - Feed furnaces
    - Typical applications
        - Heat-integrated columns
        - Air separation column systems
        - Absorber/stripper combinations
    - Assumes equilibrium stage calculations; however user can specify efficiencies
    - Use for sizing and rating trays and packings
- RateFrac
    - Rate-based rigorous model for non-equilibrium multi-stage distillation
    - Simulates actual tray and packed columns, rather than idealized representations
    - Requires a separate license
- BatchFrac
    - Rigorous model for simulating multistage batch distillation columns
    - Requires a separate license

8.2.3 Solids

- Crystallizer
    - Mixed suspension, mixed product removal (MSMPR) crystallizer
    - Feed mixes with recirculated product magma and passes through a heat exchanger before it enters the crystallizer. Product stream contains liquid and solids. The product stream can be passed through a hydrocyclone, filter, or other fluid-solid separator to separate the phases.
    - Can have an outlet vapour phase.
- Screen
    - Simulates the separation of various sizes of solid particles in a mixture in a screen.
    - Calculates the separation efficiency of the screen.
    - Specifications
        - Sizes of screen openings.
- FabFl
    - Simulates baghouse fabric filter units.
    - Solids separation from a gas stream
- Cyclone
    - Simulates cyclone separators
    - Solids separation from a gas stream using the centrifugal force of a gas vortex.
- VScrub
    - Simulates venturi scrubbers
    - Solids separation from a gas stream by direct contact with an atomized liquid stream.
- ESP
    - Simulates dry electrostatic precipitators
    - Solids separation from a gas stream
- HyCyc
    - Simulates hydrocyclones

  - Separation of solids from the inlet liquid stream by the centrifugal force of a liquid vortex.
- CFuge
  - Simulates centrifuge filters
  - For separation of liquids from solids by the centrifugal force of a rotating basket.
  - Assumes that the separation efficiency of the solids equals 1, so the outlet filtrate stream contains no residual solids.
- Filter
  - Simulates continuous rotary vacuum filters.
  - Assumes that the separation efficiency of the solids equals 1, so the outlet filtrate stream contains no residual solids.
  - Separation of liquids from solids.
- Swash
  - Single-stage solids washer
  - Solids washers that recover dissolved components from an entrained liquid of a solids stream.
  - Does not consider a vapour phase.
- CCD
  - Simulates a counter-current decanter or a multistage washer
  - Multistage solids washer that recovers dissolved components from an entrained liquid of a solids stream.
  - Does not consider a vapour phase.
  - Calculates the outlet flow rates and compositions
  - Specifications
    - Pressure
    - Mixing efficiency
    - Number of stages
    - The liquid-to-solid mass ratio of each stage.

References

Biegler, L.T., Grosmann, I.E., Westerberg, A.W., *Systematic Methods of chemical process design*, 1997.

Couper J. R., Penney, W. R., Fair, J. R., Walas, S.M., *Chemical Process Equipment, Selection and Design*, 2nd ed., Elsevier, 2010.

Dimian, A.C., Bildea, C.S., *Chemical Process Design, Computer-Aided Case Studies*, Wiley, Weinheim, 2008.

Geankplis, C.J., *Transport Processes and Unit Operations*, Prentice-Hall, New Jersey, 3rd ed., 1993.

McCabe, W.L., Smith, J.C., Harriott, P., *Unit Operations of Chemical Engineering*, 5th ed., 1993.

Mersmann, A., Kind, M., Stichlmair, J., *Thermal Separation Technology*, Springer, Berlin, 2011.

Mulder, M., *Basic Principles of Membrane Technology*, Kluwer, Dordrecht, 1996.

Perry, R.H., Green, D., *Perry's Chemical Engineers' Handbook*, Mc-Graw-Hill, New York, 1984.

Peters, M.S., Timmerhaus, K.D., West, R.E., *Plant Design and Economics for Chemical Engineers*, 5th ed., 2003.

Porter, M.C., *Handbook of Industrial Membrane Technology*, Noyes Publications, New Jersey, 1990.

Purchas, D.B., and Wakeman, R.J., *Solid/Liquid Separation Equipment Scale-up*, Uplands Press Ltd, London, 1986.

Saravacos, G. D. and Kostaropoulos, A. E., *Handbook of food processing equipment*, Springer Science+Business Media, New York, 2002.

Seader, J.D., Henley, E.J., Roper, D.K., *Separation Process Principles*, Wiley, New York, 2011.

Smith, R., *Chemical Process Design*, McGraw-Hill, New York, 1995.

Towler, G., Sinnott, R. K., *Chemical Engineering Design - Principles, Practice and Economics of Plant and Process Design* (2nd Edition), Elsevier, 2013. Online version available at: http://app.knovel.com/hotlink/toc/id:kpCEDPPEP4/chemical-engineering/chemical-engineering

Woods, D.R., *Process Design and Engineering Practice*, Prentice Hall, New Jersey, 1995.