

Mechanical separations

Mechanical separations comprise the operations in which different phases are parted from one another. There are five general situations, namely where the phases are

1. *liquid and liquid (immiscible or only slightly miscible in each other)*
2. *solid and solid*
3. *gas and liquid*
4. *gas and solid*
5. *liquid and solid.*

Mechanical separations are distinguished by the absence of component transfer between phases from molecular separations. The need for a mechanical separation may arise in dealing with mixtures of phases found in natural raw materials or in purifying process materials where extraneous phases have infiltrated. A mechanical separation also may be the final step in a molecular separation: two phases may have been brought into intimate contact in order to promote component molecular transfer between the phases; afterwards the phases have to be separated. In another situation a fluid (e.g., air) may have been used to convey solid pellets and must then, at the end of the travel, be separated from both the pellets and from any fines that were generated. A mechanical and a molecular separation may be used in sequence: in a familiar domestic operation such as drying a load of washing, the clothes are first wrung or spun to remove the bulk of the rinse water (mechanical operation), then hung or tumbled to diffuse out the rest of the water - molecular operation.

1. Liquid-Liquid Separations

There are many situations where a process ends up with a mixture of two (or more) liquid phases. The liquids may both be organics or, very often, one is organic and the other is largely water. Some situations are

- liquid-liquid extraction
- a chemical reaction that generates an immiscible product or by-product (e.g., water)
- a feed stream containing water
- cooled condensate from azeotropic distillation
- cooled condensate from steam stripping
- aqueous solution used to wash or otherwise treat an organic liquid.

The three common methods of separation all rely on a difference in density of the two phases.

I. Decantation or Settling

If the density difference is sufficient, the simplest method is to use gravity to pull the phases apart. The device or vessel is called a *decanter* if it operates partially full and a *settler* if it operates full. If operating continuously, the mixture enters at one end and soon separates into a light top layer and a heavy bottom layer. Part of the light phase remains in dispersed form in the bottom layer and part of the heavy phase remains in dispersed form in the top layer.

Typically, a droplet size of 150 microns (a micron being 1/1000th of a millimeter) is used for calculation purposes. In the rest of the operation the droplets rise or fall to join their appropriate phase and the vessel is sized to allow this to happen. Sigales recommends that, regardless of the calculated value of droplet fall or rise velocity, the velocity never be assumed greater than 10 inches per minute. Ludwig (1995) quotes one hour as a typical time for separation. Provision is made at the end of the vessel to draw off the two phases. This is done in a controlled manner, to regulate the level of the interface between phases and, in the case of a decanter, to regulate the overall height in the vessel.

II. Centrifugation

This is a mechanical process which involves the use of the centrifugal force to separate particles from a solution according to their size, shape, density, medium viscosity and rotor speed. The denser components of the mixture migrate away from the axis of the centrifuge, while the less dense components of the mixture migrate towards the axis. Chemists and biologists may increase the effective gravitational force of the test tube so that the precipitate (pellet) will travel quickly and fully to the bottom of the tube. The remaining liquid that lies above the precipitate is called a supernatant or supernate. There is a correlation between the size and density of a particle and the rate that the particle separates from a heterogeneous mixture, when the only force applied is that of gravity. The larger the size and the larger the density of the particles, the faster they separate from the mixture. By applying a larger effective gravitational force to the mixture, like a centrifuge does, the separation of the particles is accelerated. This is ideal in industrial and lab settings because particles that would naturally separate over a long period of time can be separated in much less time. The rate of centrifugation is specified by the angular velocity usually expressed as revolutions per minute (RPM), or acceleration expressed as *g*. The conversion factor between RPM and *g* depends on the radius of the centrifuge rotor. The particles' settling velocity in centrifugation is a function of their size and shape, centrifugal acceleration, the volume fraction of solids present, the density difference between the particle and the liquid, and the viscosity. The most common application is the separation of solid from highly concentrated suspensions, which is used in the treatment of sewage sludges for dewatering where less consistent sediment is produced. Centrifugation makes use of rotation to create large centrifugal forces, hundreds or thousands of times greater than gravity. Droplets that would move only very slowly under gravity can be greatly speeded up in a centrifuge.

III. coalescence

In chemistry, **coalescence** is a process in which two phase domains of the same composition come together and form a larger phase domain. In other words, the process by which two or more separate masses of miscible substances seem to "pull" each other together should they make the slightest contact. To *coalesce* is to bring together and unite. In the present case the coalescence is of small liquid droplets dispersed in another liquid. Typically, the droplets are in the size range 0.1 - 50 microns, which is too small to separate under the influence of gravity. (This is because the terminal velocity of small droplets varies as the square of

diameter.) In coalescence the two-phase mixture is passed through a bed of fibres or particles onto which the dispersed liquid attaches (possibly adsorbs) and builds up into larger droplets (e.g., 500-5000 microns), which can then be separated by gravity. The effectiveness of coalescence in liquid-liquid separation requires some degree of interfacial tension (IFT) between the two liquids.

2. Solid-Solid Separations

Solid particles or pellets of different types often occur as mixtures. The difference in type may be simply size or it may be the chemical nature of the particles. Some separation situations are

- remove over-sized particles that would ruin a surface finish or cause an electronic product to fail
- remove fine particles that would cause a dusting problem or that would get rubbed off in a molded or coated object
- sort out and recycle over-size particles in a comminution operation
- prepare feed for a mass-transfer operation or a chemical reaction, where small size processes faster
- remove 'tramp' metal from a solid product

Some separation mechanisms depend on differential behavior in a separation system for gases and solids or liquids and solids, examples being *centrifugation*, *cyclone flow*, *hydroclone flow*, *differential settling* and *flotation*. These operations are discussed subsequently in this lecture. It should be noted that, in these cases, the behavior of the solid particles may depend on a combination of their size, shape and density, so the separation with respect to the characteristic of interest may not be as distinct as desired. Three methods specific to solid-solid separation are *screening*, *air-classification* and *separation by magnetic or electrostatic forces*.

Elutriation, Air-Classification

A particle immersed in a stationary or moving fluid but with a velocity different from the fluid is acted upon by a drag force which tends to make the particle take the same velocity as the fluid. If an external force (e.g., gravitational or centrifugal) acts on the particle then the particle adopts a speed and/or direction different from the fluid. The extent to which this happens depends on the relative magnitudes of drag force and the external force. In general, the greater the size (and the greater the density) of the particle, the greater the deviation. This principle is used in centrifugation, cyclone separation, and settling, as well as in elutriation and air-classification. **Elutriation** itself is a crude separation tool, depending on only gravity as the external force. If particles are introduced into an upward-moving stream of gas, the lighter ones are carried with the gas and the heavier ones fall through the gas. The selectivity of this separation is poor but elutriation may be a good pre-separation step. Some commercial elutriators are in the form of a zig-zag channel.

Air-classification uses rotational motion of air to create centrifugal force. The rotation may come from tangentially introduced air, as in a cyclone, but the more effective and high-throughput classifiers use a mechanical rotor. Cyclones are effective down to particle size

around 45 microns; they do not give good 'sharpness of cut'. Mechanically-driven classifiers, incorporating also an elutriation section, operate in the range 5-250 microns. Special high-energy dispersion classifiers operate in the range 1-50 microns. Classifiers are often used in combination with comminution and sometimes with on-line laser measurement of particle average size.

Magnetic Separation, Electrostatic Precipitation

If particles of different composition are to be separated, then other properties besides size may be put to use. Differences in density or shape (or size) may be invoked. Magnetic susceptibility is another possibility. Certainly, if one component is a *ferromagnetic*, such as iron, cobalt or nickel, then, because of its very high susceptibility, it is relatively easy to pull free from a mixture of particles. Many other materials - other elements and compounds - are *paramagnetic* and have the ability (susceptibility) to be temporarily magnetized in a magnetic field. If the types of particles in a mixture have different susceptibilities, then they may be separable by magnetic means. Tables of susceptibility are available. Some substances are *diamagnetic* and are repelled by magnetic fields. The equipment whereby separation is carried out is equipped first to attract the susceptible particles and then to deposit them in a different location from the non (or lesser) attracted particles.

3. Gas-Liquid Separations

Two general situations are liquid-dispersed-in-gas and gas-dispersed-in-liquid. The first situation arises for instance when

- vapor from a boiling or sparged pool of liquid carries (entrains) liquid droplets
- vapor emerges from a flashing two-phase flow
- a gas-vapor mixture is cooled.

The second situation may arise when

- gas passes through, or vapor is generated within, a liquid, and is particularly pronounced if the liquid is viscous
- a liquid is agitated vigorously.

In general, these gas-liquid mixtures are undesired and require separation.

The liquid-in-gas situation is examined first. The method of separation depends on the size of the liquid droplets and also on the loading in the gas. We examine the methods starting with the largest droplets and the highest loadings.

Gravity Settling

Two-phase mixtures emerging from gas-liquid pipelines often have large liquid loadings and gross dispersion of liquid in the gas. A typical liquid concentration is 50 litres per cubic metre of gas. A simple 'knock-out' pot can do the initial separation of gas and liquid and should be installed ahead of any more sophisticated device. Gas leaves at the top and liquid leaves at the bottom, both streams probably containing some amount of the other phase.

Cyclone Flow

For liquid content in the range 1 to 50 litres per cubic metre a cyclone is effective in separating liquid from gas, especially if the liquid droplets are larger than 10-50 microns. However, smaller droplets are carried out with the gas stream in the process called the cyclone flow.

Inertial Precipitation: De-Misting, Scrubbing

For droplets greater than 5-50 microns, passage of the gas-liquid through a wire-mesh pad can remove most of the liquid. The gas passes around the wire elements but the liquid, having more inertia, collides with the wire and collects (precipitates) on it. The liquid runs together (i.e., coalesces) and forms droplets that are large enough to detach and fall back against the main flow

4. Gas-Solid Separations

Three possible reasons why gas stream bearing particulates may require separation are

- the particulates have value
- the equipment into which the gas is subsequently entering would be damaged by particulates (e.g., a compressor)
- the gas is being released to atmosphere and must be clean.

A common situation is in pneumatic conveying of pellets, where not only must the pellets be separated from the gas stream but so must any fines that were generated by attrition.

Methods for removing particulates (both solid and liquid) from gases are as follows

- gravity settling
- cyclone flow
- scrubbing
- filtration
- electrostatic precipitation

5. Liquid-Solid Separations

Some of the situations in which liquid and solid particles are mixed with each other are

- naturally occurring streams of water bearing silt
- liquid-phase reaction mixtures containing a catalyst in pellet form
- mother liquor and crystallized material
- waste streams containing sludge or other environmental contaminants.

The most often used separation techniques are centrifugation and filtration. They along with some lesser methods are discussed here.

Sedimentation Centrifugation

As explained previously, centrifugation is an extension of the technique of *settling*, where the force of gravity is replaced by a much greater *centrifugal* force. The smallest and simplest centrifuge is that found in the chemistry laboratory, where a circular rack of test tubes is whirled at high speed generally to drive a dispersed precipitate to the bottom of the tube.

The *tubular* (sometimes called *tubular bowl*) centrifuge is a device for separating immiscible liquids. This device is also used for liquid-solid mixtures. This is a more common usage of centrifuges in general and, when so designed and used, they are called ***sedimentation centrifuges***.

The *sedimentation centrifuges* styles.

The *solid-bowl basket (or solid-bowl batch)* centrifuge is similar to the tubular but is less elongated and is used at larger scales and can tolerate larger solid particles. Like the tubular it is a batch unit, requiring periodic shutdown to remove the solids that are deposited at the wall. Liquid is continuously discharged. Both centrifuges rotate about a vertical axis.

The *multi-chamber (or chamber bowl)* centrifuge is also a batch vessel rotating about a vertical axis. Internally it is fitted with concentric vertical partitions such that the slurry has to up and down successive annuli. The effect is to produce more surface area for solids to settle on.

Settling

If the difference in densities of liquid and solid is great enough then an adequate separation may be achieved simply by letting the particles settle to the bottom of a vessel. The operation may be batch or continuous. If the objective is to produce a clear liquid (e.g., water) then the settler is called a *clarifier*. If the objective is to recover a valuable solid product, then the settler is called a *thickener*. In any case the behavior of the descending solid phase is complicated by the interaction of particles with one another. One cannot simply extrapolate from the behavior of single particles. In continuous operation there will always be a gradient of solid concentration from top to bottom of the vessel.

Flotation

In settling, the more common case and the case usually considered is that of particles heavier than the liquid. Solids settle to the bottom. In the event that the solid material is less dense than the liquid, the particles float to the top where they may be skimmed off. However even denser particles can be made to float if bubbles attach to them. This is the principle of ***flotation***.

Flocculation

In settling, where terminal velocity varies as the square of particle diameter, and in any process where fluid drag forces are involved, it is advantageous to have bigger particles. A flocculating agent acts to bring small particles together into larger faster-settling entities. Hughes (1977) lists a number of flocculants, all of them organic, most of them polymeric, some non-ionic, some containing acidic or basic functional groups. To be economically

practical a flocculating agent must be effective in low concentrations. They are used in settling and also in decanting centrifuges.

Expression and Wicking

If the liquid-solid mixture is largely solid and if the liquid is loosely held, then it may be possible to squeeze out, or *express* a significant part of the liquid. Wringing a cloth or squeezing an orange are domestic examples and in fact expression is used commercially in the making of juices. Expression may be used to get rid of the bulk of the liquid before a more intense step to reach the final desired level. In paper-making, press-felts are brought into contact with the formed sheet to *wick away* some of the residual water left after the initial formation of the sheet. This step precedes the final drying over steam-heated rolls. Blotting paper is another example of a wicking material.

Table 1 Summary of Mechanical Separations

<u>METHOD</u>	<u>liquid-liquid</u>	<u>solid-solid</u>	<u>gas-liquid</u>	<u>gas-solid</u>	<u>liquid-solid</u>
1. Decantation	yes				
2. Coalescence	yes		yes		
3. Centrifugation	yes	yes			yes
4. Screening		yes			
5. Elutriation, Classification		yes			
6. Magnetic attraction		yes			
7. Cyclone flow		yes	yes	yes	
8. Settling, Differential settling		yes	yes	yes	yes
9. Flotation		yes			yes
10. Inertial precipitation: De-misting, Scrubbing			yes	yes	
11. Foam-breaking			yes		
12. Electrostatic precipitation			yes	yes	
13. Filtration				yes	yes
14. Flocculation					yes
15. Hydroclone flow					yes
16. Wicking and Expression					yes

Reference

Hughes M A (1977) 'Coagulation and Flocculation', in Svarovsky L, editor, Solid-Liquid Separation, Butterworths, London.

Perry R H and Green D W (1997) Perry's Chemical Engineers' Handbook, Seventh Edition, chapter 18, p.63.

Christian J B (1994 July) 'Improve Clarifier and Thickener Design and Operation', Chemical Engineering Progress p.50-56.

Perry R H and Green D W (1997) Perry's Chemical Engineers' Handbook, Seventh Edition, chapter 18, p.125-130.

Ludwig E E (1995) Applied Process Design for Chemical and Petrochemical Plants, Gulf Publishing Company, Houston.

Brown R L and Wines T H (1993 December) 'Improve suspended water removal from fuels', Hydrocarbon Processing p.95-100.

Wines T H and Brown R L (1997 December) 'Difficult Liquid-Liquid Separations', Chemical Engineering p.104-109.

Katona A, Darde T and Wines T H (2001 August) 'Improve haze removal for FCC gasoline', Hydrocarbon Processing p.103-108.

Snow R H and Allen T (1992 May) 'Effectively Measure Particle-Size-Classifiers Performance', Chemical Engineering Progress p.29-33.

DeCenso A J (2000 April) 'Dry Screening of Granular Solids', Chemical Engineering p.76-83.

Klumpar I V (1992 April) 'Control and Scale-up Air Classifiers', Chemical Engineering Progress p.50-55.

Hixon L (1992 July) 'Sizing Up Air Classifiers', Chemical Engineering Progress p.59-62.

Crawley G, Malcolmson A, Crosley I and McLeish A (2002 April) 'Particle Classification: Making the Grade', Chemical Engineering p.54-60.

Bloch H P (2000 August) 'Solve your hydrocracker compressor problems', Hydrocarbon Processing p.39-42.

Phillips H W (2000 September) 'Select the Proper Gas Cleaning Equipment', Chemical Engineering Progress p.19-38.

Sigales B (1975 June 23) 'How to Design Settling Drums', Chemical Engineering p.141-144.