13
Unit Operations Involving Particulate Solids

Objective

When you have completed study of this chapter you should be able to:

- Understand the concepts concerned with solids storage;
- Know the various size reduction equipments;
- Apply the principles of crystallization;
- Be familiar with the different mechanical separation techniques.

13.1 Storage of Solids

The frequently faced problems associated with the storage of bulk solids in bins and silos can be avoided if they are designed with respect to the flow properties of the bulk solid which has to be stored.

13.1.1 Bins & Silos

The basic considerations for the design of silos are mentioned below:

Stresses associated in silos

Figure 13.1 indicates a typical silo and the pressures and stresses acting on it. While the pressure (for fluids the term pressure" is used) would increase linearly downwards if the silo would have been filled with a fluid (a), the course of the vertical stress (for bulk solids the term stress is used) in a silo filled with a bulk solid is rather different (b ,c).

In the latter case in the vertical (cylindrical) section of the silo the vertical stress increases in a degressive way. If the height to diameter ratio of the silo is sufficiently large (usually: > 3), a constant vertical stress is attained. This indiactes that the vertical stress will not increase further even if the filling height is much larger. The main reason for this is that the shear stresses acting between the bulk solid and the silo walls even if the bulk solid is at rest. Due to the shear stresses, the silo walls share a part of the weight of the bulk solid.

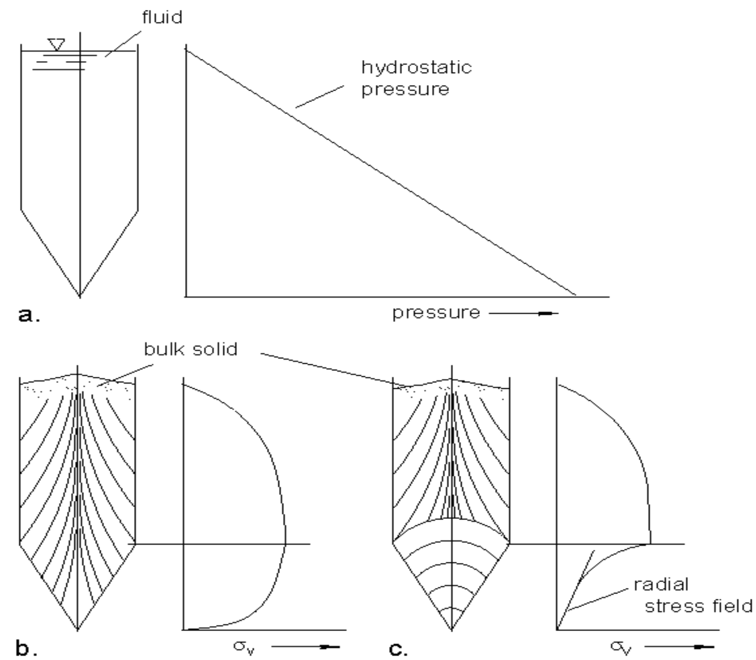


Figure 13.1
a) Pressure in a Silo Filled With a Fluid (Imaginary);
b) Vertical Stress after Filling the Silo With a Bulk Solid
c) ***Vertical Stress After the Discharge of Some Bulk Solid***

The stresses acting in a hopper are different from those in the vertical section. After filling an empty silo, the so called filling stress state (also: active stress state, figure 13.1(b) acts, where the vertical stress in the hopper decreases less in the upper part of the hopper and then more near the imaginary hopper apex. As soon as some bulk solid is discharged for the first time after filling, the stresses in the hopper change and the so-called emptying stress state (also : passive stress state) prevails (figure 13.1c). When flowing downwards in the hopper, the bulk solid is compressed in the horizontal direction so that the walls of the hopper carry a larger part of the weight of the bulk solid and, hence, the vertical stress in the lower part of the hopper is apparently lesser than after filling. In the emptying stress state the vertical stresses in the lower part of the hopper are nearly proportional to the distance to the imaginary hopper tip .In other words, the stresses are proportional to the local hopper diameter. This linear course of stress is called the radial stress field . In principle, in the vertical section of the silo the stresses remain unchanged at discharge.

Flow Profiles : Mass & Funnel Flow

Two different pattern of flow can be observed (figure 13.2a),if a bulk solid is discharged from a silo:

- mass flow
- funnel flow

In case of mass flow, the whole contents of the silo are in motion at discharge. Mass flow is only possible, if the hopper walls are sufficiently steep and/or smooth, and the bulk solid is discharged across the whole outlet opening. If a hopper wall is too flat or too rough, funnel flow will appear. In case of funnel flow (figure 13.2b), only that bulk solid is in motion first, which is placed in the area more or less above the outlet. The bulk solid adjacent to the hopper walls remains at rest and is called “dead" or “stagnant" zone. This bulk solid can be discharged only when the silo is emptied completely. The dead zones can reach the surface of the bulk solid filling so that funnel flow becomes obviously when observing the surface. It is possible as well that the dead zones are located only in the lower part of the silo so that funnel flow cannot be recognized by observing the surface of the silo filling.

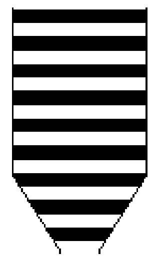


Figure 13.2a
Mass flow

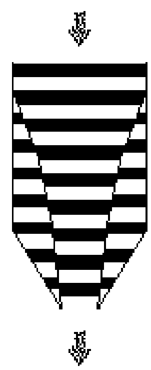


Figure 13.2(b)
Funnel flow

Flow Problems

Typical incidents which occur at the storage of bulk solids are:

- ***Arching***

If a stable arch is formed above the outlet so that the flow of the bulk solid is obstructed, then this situation is called arching (figure 13.3a). In case of fine grained, cohesive bulk solid, the reason of arching is the strength (unconfined yield strength) of the bulk solid which is caused by the adhesion forces acting between the particles. In case of coarse grained bulk solid, arching is caused by blocking of single particles. Arching can be prevented by providing sufficiently large outlets.

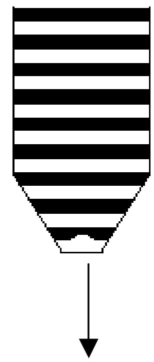


Figure 13.3a
Arching

- ***Ratholing***

This occurs in case of funnel flow if only the bulk solid above the outlet is flowing out, and the remaining bulk solid (the dead zones)- keeps on its place and creates the rathole. The reason for this is the strength (unconfined yield strength) of the bulk solid. If the bulk solid consolidates increasingly with increasing period of storage at rest, the risk of ratholing increases. If a funnel flow silo is not emptied completely in sufficiently small regular time intervals, the period of storage at rest can become very large thus causing a strong time consolidation.

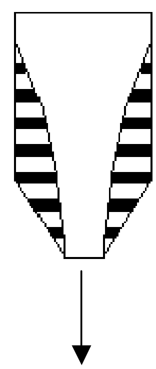


Figure 13.3b
Ratholing

Irregular flow occurs if arches and ratholes are formed and collapse alternately. Thereby fine grained bulk solids can become fluidized when falling downwards to the outlet opening, so that they flow out of the silo like a fluid. This concept r is termed as flooding. Flooding can result in a lot of dust, a continuous discharge becomes impossible.

- ***Wide residence time distribution***

If dead zones are progressively formed (funnel flow), the bulk solid in this zones is discharged only at the complete emptying of the silo, whereas bulk solid, which is filled in later, but located closer to the axis of the silo, is discharged earlier. Because of that, a wide distribution of residence time appears which is disadvantageous in some cases (e.g. in case of storage of food or other products changing their properties with time).

- ***Segregation***

Segregation of particles can occur with many bulk solids as they are being handled and the results can be quite costly. For example the pharmaceutical industry is prone to problems with particle segregation. If fine and coarse particles segregate, the final tablet or capsule quality can be altered such that valuable drugs have to be remmoved.

If a heap is formed on the bulk solid's surface at filling of the silo, segregation is possible according to particle size or particle density .In case of centric filling as shown in figure 13.3c ,the larger particles accumulate close to the silo walls, while the smaller particles collect in the centre. In case of funnel flow, the finer particles, which are placed close to the centre, are discharged first while the coarser particles are discharged at the end.

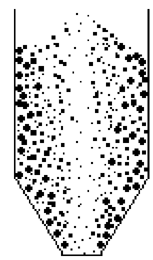


Figure 13.3c
Segregation

In a funnel flow silo, all issues mentioned above can occur generally, while in case of mass flow only arching has to be considered: segregation, ratholing, irregular flow and flooding of the bulk solid do not appear in a well designed mass flow silo. The residence time distribution of a mass flow silo is narrow, because it acts as a „first in - first out" system .

There are several processes which cause segregation. Among them are sifting, particles sliding and air entrainment.

Sifting

Sifting occurs when small particles trickle down through a body of larger particles. This is the most common means for particles to separate. In order for sifting to occur, the particles must be free flowing, different sizes, fairly large (+100 mesh)and have some means of inter-particle motion .The particles segregate in a horizontal or "side-to-side" pattern.

Sliding Particles

Particles sliding on a surface can segregate because fine particles have the capacity to be more frictional than coarse ones. If a chute is used, the fine particles in motion settle to the bottom of the chute due to sifting.

The increased friction of the finer particles results in drag and velocity differences between particles as they are sliding on the chute surface. When the particles discharge from the chute, the fine particles concentrate at the end, while the coarse ones have a trajectory which carries them further away.

Air Entrainment

Air entrainment. affects fine particles as they tend to remain airborne longer than coarse or heavier particles As a bin is filled, the fine, light particles tend to settle on top, while the coarse particles fall rapidly, creating a vertical or "top-to-bottom" segregation pattern.

Practical Solutions

- Minimize sifting by always ensuring a mass flow pattern (first-in-first-out). Even though the material can segregate side-to-side, the coarse and fine particles will be reunited at the outlet because of mass flow.
- Maintain a minimum head of material above the hopper section in a mass flow bin to reduce the velocity gradient that occurs as the material reaches the hopper section in a mass-flow bin.
- Keep the material more cohesive by adding water or oil. One of the prerequisites for segregation is inter-particle motion, increasing cohesiveness causes the particles to stick together and reduces this motion. Warning: do not make the material too cohesive or it will not flow.
- Use a tangential entry into bins when handling fine solids that segregate by air entrainment. Instead of filling the bin from the center of the top of the bin, introduce the material in at the top but on the side, tangential to the circumference of the bin. This will actually minimize a vertically segregating material by causing side-to-side segregation, the particles will be reunited when they are discharged at the outlet of your mass-flow bin

Factors That Impact a Bulk Solid's Flowability

Moisture content, temperature, particle size, and time of storage at rest can have a significant impact on your material's flow properties. The fluid flow is a function of it's cohesive properties. As such, conditions that effect the cohesive strength of a solid will have an impact on its handling capabilities.

Moisture Content

The rule of thumb is "as the moisture content of a solid increases, so does it's cohesive strength". Even though there may be only a slight increase in moisture, say from 1% to 1.5%, the flowability of the material can be significantly impacted. Hygroscopic materials can experience significant moisture increases simply by being exposed to humid air. The material flow properties should be measured using a representative value for moisture content.

Temperature

Cohesiveness is also impacted by the solid's temperature. Some materials are sensitive to increases in temperature (e.g. room temperature to 150 deg F). Others are sensitive to constant temperature. These conditions can be simulated in the laboratory through flow properties tests.

Particle Size

As a rule of thumb - "as a bulk solid becomes finer, it also becomes more cohesive and subsequently more difficult to handle". Fibrous and angular particles are usually more cohesive than particles that are rounded. Typically, particles that are consistently ¼" and larger are not cohesive arching problems. They can however, form interlocking arches.

Time of Storage at Rest

As a solid remains at rest in a bin or hopper, it can become more cohesive and difficult flowing. At rest, the compaction loads due to head pressure can produce a strong cohesive bond.

A chemical reaction, crystallization, or adhesive bonding can also cause this. Sometimes, after a cohesive arch is broken up, say by somehow initiating flow, the material can revert back to its original flow condition and not exhibit a similar cohesion if left at rest again. On the other hand, some materials will time and time again, bridge and rathole even after flow is re-initiated.

This is why it is absolutely necessary to measure the flow properties of your material to determine the affects of the environmental conditions discussed above

13.1.2 Feeders

It's easy to under estimate how important feeder selection is for consistent material flow. "The Fix" usually entails wither retrofitting an existing funnel-flow bin or designing a new bin to ensure a mass-flow pattern. This fix can be an expensive liner or steeper hopper angles and as such, you can destroy this effort simply by selecting an improperly-designed feeder.

Bin and feeder design go hand-in-hand. The feeder must work in unison with the bin and:

- Suit the materials flow properties
- Work with the bin outlet shape
- Withdraw material uniformly across the outlet's entire cross-sectional area
- Minimize the vertical loads applied to the feeder Accurately control the discharge rate

There are many types of feeders available to handle bulk solids and they can be divided into two categories: volumetric and gravimetric. A volumetric feeder discharges a volume of material as a function of time while a gravimetric feeder weighs material.

Volumetric feeders

Volumetric feeding is adequate for many solids feeding applications. Feed accuracy in the range of 2-5% can be achieved with most volumetric designs.

Volumetric feeding can, however become inaccurate if the bulk density of the solid that is being handled varies. The feeder cannot recognize a density change because it simply discharges a certain volume per unit time. Examples of volumetric type feeders are: screws, belts, rotary valves, louvered type, and vibratory.

Gravimetric feeders

A gravimetric feeder relies on weighing the material to achieve a required discharge rate or batch weight. This approach should be used when:

- Accuracy of less than 5% is required
- The material's bulk density varies
- A record the weight of material used for a particular process.
- Feed accuracy of 0.25% is sometimes obtainable with a properly designed
- gravimetric feed system. A disadvantage of a feeder that weighs material is that it is usually more expensive than a volumetric device.

There are basically two ways to feed gravimetrically, continuous and batch. A continuous gravimetric system controls the weight/unit time such as lbs./hr or kg/hr. A batch system controls simply the weight of material such as 50 lbs. of material to a mixer. Examples of gravimetric feeders are: weigh-belts, loss-in-weight systems, and gain-in-weight systems.

13.1.3 Transferring Solids with Chutes

Oftentimes, it is required to transfer solids from the outlet of a bin to processing equipment, into a truck, or another bin. Conveying material using equipment designed to mechanically convey or transfer solids works quite well; however, for short distances, this can be an expensive approach.

Chutes can be used as an substitute for expensive conveyors to transfer solids short distances. A chute is simply a pipe or trough that is sized properly and at the correct slope angle to ensure sliding of the material to be transferred. (See Fig. 13.4)

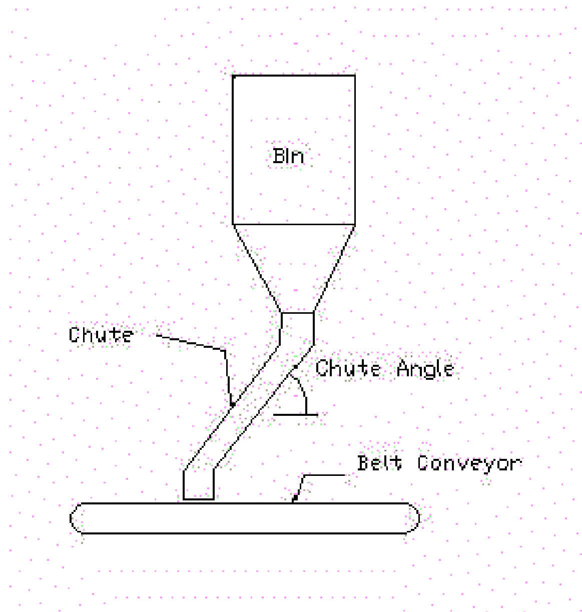


Figure 13.4
Feeding Through Chute

A chute must be steep and smooth enough to ensure sliding along its entire length. The impact of material on the chute is extremely important.

Whether the material is dropping in free fall from a bin or being transferred from another chute, impact pressures and the velocities necessary to keep material moving along a chute are critical. Impact pressure can be calculated as follows:

$$\textbf{Impact pressure} = \frac{(\text{bulk density})\cdot(\text{velocity})^2\cdot(\text{sine of impact angle})^2}{\text{acceleration due to gravity}}$$

Where:

- impact pressure is in pounds per square feet (psf),
- velocity before impact is in feet per second (ft/s),
- bulk density is in pounds per cubic feet (lb./ft^3),
- impact angle of incoming stream is in degrees
- acceleration due to gravity is 32 feet per $second^2$

A test can be run in the laboratory to measure the critical chute angle resulting from impact pressure. This information can then be used to develop the minimum chute angle at the point at which it impacts the chute.

The tests involves placing a sample of solid on a surface representative of the surface to be used as the chute. A load is applied to the sample (using weights) and removed after a short time (say 20 sec.) to simulate impact pressure. The surface is then tilted until the sample begins to slide.

That angle of slide is then recorded. This test is repeated several times to ensure accuracy and under a range of loads to simulate a range of impact pressures. A safety factor of about 5° is added to the results.

Other considerations when designing chutes is chute cross-sectional area and chute wear.

- Do not converge chutes, because the change in cross-sectional area will likely cause material to stop sliding.
- Wear can be overcome by using wear liners or abrasion-resistant steel.
- However, that the rougher the surface, the steeper the angle required to keep material moving in the chute.

13.1.4 Handling Titanium Dioxide

Titanium dioxide (TiO_2) is the most difficult material to handle. Titanium dioxide is one of the best pigments which , provides one of the quality whiteness, opacity and refractive index improvements to paints, coatings, plastics, paper, inks, fibers, food and cosmetics. The reason is that it is with it's small particle size and irregular shape, it sticks everywhere, to everything and to everyone.

Most commercially available TiO_2 is nearly 100% minus 325 mesh and has a bulk density of about 50 lb./cu.ft. Strangely, of the various grades of TiO_2 supplied by the major manufactures, the better the grade, the more difficult it is to handle.TiO_2 can bridge over circular outlets of 12-inches to up to 5-feet. Equally important is it's ability to cling to any surface.

One of the best surface for solids to slide is a 2B finish stainless steel. TiO_2 can even adhere to this smooth finish and effect a hopper's ability to flow properly. It is important to measure and evaluate your specific material's properties to ensure proper bin and feeder design. Properties vary from manufacture to manufacture.

Another area of critical concern is maintenance. There are not many machines that work well when product builds up on the sliding surfaces. A material as "sticky" as TiO_2 presents an even greater problem. It's propensity to cling creates constant work for those who maintain equipment that stores, feeds, mixes or conveys it. Work, not on a semi-annual or quarterly basis, but weekly or even daily.

Mechanical assistance for stubborn-flowing TiO_2 is not out of the question. Devices such as air blasters, air sweeps and vibrators, if designed, strategically-placed and used properly, will lengthen the time between scheduled maintenance. Some users have even gone so far as to pelletize their TiO_2 to improve it's handling characteristics.

To sum up , it is important to:

- Respect TiO_2's ability to bridge, rathole and cling
- Test for and be aware of the flow properties of the TiO_2 you use
- Allow time for scheduled maintenance
- Use flow-enhancing devices, when needed, but with caution

13.2 Size Reduction

A size reduction machine employs one of several methods -- cutting, pulverizing, crushing, impacting, or impingement grinding -- to reduce a material's particle size. Most processors operate several types of size reduction equipment because no universal size reduction machine exists that can do it all. When determining which size reduction equipment to use, a processor looks at the properties of the material to be reduced. These include material hardness (typically classified according to the Mohs' hardness scale), heat sensitivity, moisture sensitivity, abrasiveness, friability, explosiveness, and the initial and desired particle size.

13.2.1 Crushers

Crushers and mills are typical process equipment for reducing solid chemicals, materials and other solid products to a desired particle or aggregate size range in dry or wet (slurry) forms. Mills are also utilized for mixing or dispersing solids in liquids. Feed size, material and hardness are some of the factors utilized in selecting the proper crusher or mill. Types of crushers and mills include: ball or media mills, cone and gyratory crushers, disk attrition mills, colloid and roll mills, screen mills and granulators, hammer and cage mills, pin and universal mills, impact mills and breakers, jaw crushers, jet and fluid energy mills, roll crushers, disc mills, and vertical rollers and dry pans.

- Ball and media mills reduce material to particle size by tumbling the feed with grinding media such as balls, rods or other shapes. Ball mills are typically wet, batch units. Water or another liquids and additives aid the grinding process by reducing friction, deflocculating or cooling. Media mills are also employed to disperse a powder into a liquid product such as pigment in a paint base. Motion is imparted to the media through tumbling or rotating the vessel, stirring rods or vibration. They are also known as pebble, rod, tube, compartment, tumbling, vibratory, stirred, dispersion, conical or tri-cone mills.
- Cone and gyratory crushers comprises of a cone shape bowl with a gyrating central head. Feed is crushed between the cone and head.
- Disk attrition mills (including double disk mills), are modern versions of the ancient buhrstone mill where the stones are replaced with opposing disks or plates. The disks may be grooved, serrated or spiked.
- Colloid and roll mills emulsify and disperse media by using high-speed rotors within a liquid media. The rotors often have a serrated outer surface. Some dispersion mills with larger gaps also use fine beads within the liquid to enhance dispersion. Roller mills or 3-roll mills disperse and refine a fine powder or pigment into a liquid by passing the paste between a series of rolls rotating at different speeds. Three roll, colloid or other dispersion mills are commonly applied in paint, resin and adhesive applications.
- Screen mill and cutters produce a uniformly sized product or granules. Some granulators use a rotating knifes while other types employ a crushing or shearing action against an integrated screen or grate to control product granule size.

- Hammer and cage impact mills use fixed or swinging hardened steel hammers, chain or a cage for coarse crushing to fine milling. Hammer crushers and cage mills are available in vertical and horizontal rotor configurations with one or many rows of hammers.
- Pin and mills use a rotor with one or more rows of rods that impact and/or propel particles into stationary pins or surfaces. Pin mills (including cross beaters and universal mills) fall into the category of high-speed rotor pulverizers or disintegrators. They tend to produce a finer product then coarse crushers or impactors.
- Impact mills and breakers crush feed material by forcing it against a breaking surface. The feed material is propelled by gravity or by a rotating impeller or rotor. The impellers or rotors may be vertically or horizontally orientated. Vertical impact mills, cage mills, Bradford breakers, hammer mills, granulators are types of impact mills
- Jaw crushers and mills pulverize feed materials between fixed and reciprocating plates, producing coarse granuales. Blake, swing, overhead eccentric and Dodge jaw crushers are common variations.
- High-speed impact mills include hammermills, pin mills, counter-rotating pin mills, cage mills, turbo mills, and universal mills. A high-speed impact mill (Figure 13.5) reduces nonfriable and friable materials such as wood waste, sheet pulp, plastics, coal, chemicals, limestone, and fertilizer to medium-fine and fine (10- to 200-mesh) pieces. The material to be reduced enters the mill's housing and is impacted by a rotating assembly of hammers, pins, or cages. As it rotates, the assembly throws the material centrifugally outward where the hammers, pins, or cages grind it against a perforated screen for further size reduction. The final product's size is controlled by the assembly's rotating speed and the perforated screen at the discharge port. A high-speed impact mill is available in several sizes ranging from small laboratory equipment up to large production machines.

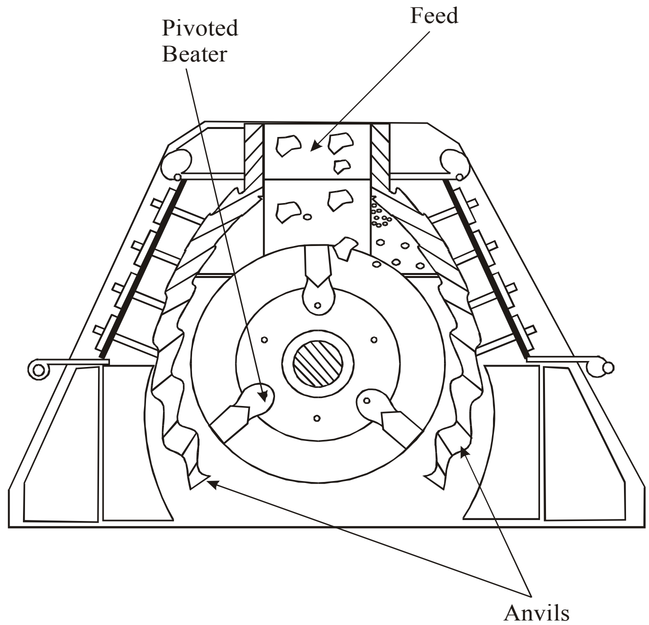


Figure 13.5
Impactor

- Jet and fluid energy mills are used for fine grinding. They function by impacting a stream or jet of feed particles against a wall or an opposing jet of particles.
- Roll crushers crush feed between the nip of two rolls or between a single roll and a fixed surface. They are used for intermediate grinding. Rolls crushers tend to produce weaker shaped product particles then impact mills.
- Disc mills are used for shredding fibrous or tough materials such as wood products, cellulose, rubber or polymers.
- Vertical roller and dry pan crushers and mills use a vertically orientated crushing wheel or muller that revolves around a solid or perforated pan, or screen. Alternately, the pan can rotate or both the rollers and pan or grinding table can rotate. These mills are often used in foundries, and mineral and ore processing applications. They can reduce relatively coarse feed to a coarse powder in one step (e.g., minus 2 inch feed to -20 mesh product).

13.2.2 Cutting Machines

Rotary knife cutting

Rotary knife cutters include precision cutters, granulators, blow-through cutters, pelletizers, and guillotine cutters. A rotary knife cutter reduces large thin pieces or small thick pieces of nonfriable materials such as paper, plastics, and rubbers to medium-coarse (1/8- to 1- inch) pieces. The rotary knife cutter typically employs a shaft with a mounted knife (or knives) that rotates toward a stationary bed knife (or knives) to cut and shear materials between the blades. A perforated metal screen, located below the knives, retains oversized material until it's processed to the proper size. Various screen mesh sizes allow particles to be reduced to multiple size ranges. The number of rotating knives and fixed knives depends on the machine's size and function. The rotary knife cutter is available in several sizes (listed as knife tip-to-tip length by shaft length) ranging from small laboratory equipment up to large production machines and can be powered by a motor ranging from 2 horsepower up to hundreds of horsepower. The rotary knife cutter can be used in applications as varied as recycling thin plastic film and reducing full bales of rubber.

13.3 Crystallization

Crystallization refers to the formation of solid crystals from a homogeneous solution. It is essentially a solid-liquid separation technique and a very important one at that.

Typical example of crystallization

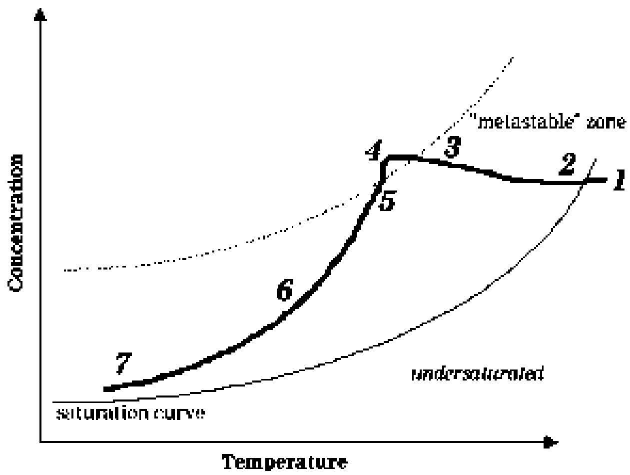
1.Waterfreezing

2. Removing sucrose from beet solutions

3. Removing KCl from an aqueous solution

Crystals are grown in different shapes, which are dependent upon downstream processing or final product requirements. Crystal shapes can include cubic, tetragonal, orthorhombic, hexagonal, monoclinic, triclinic, and trigonal. In order for crystallization to take place a solution must be "supersaturated". Supersaturation refers to a state in which the liquid (solvent) contains more dissolved solids (solute) than can ordinarily be included at that temperature.

On an commercial scale, a large supersaturation driving force is necessary to initiate primary nucleation. The initiation of primary nucleation via this driving force is not fully understood which makes it difficult to model (experiments are the best guide). Usually, the instantaneous formation of many nuclei can be observed "crashing out" of the solution. You can think of the supersaturation driving force as being created by a combination of high solute concentration and rapid cooling. In the salt example, cooling will be gradual so we need to provide a "seed" for the crystals to grow on. In continuous crystallization, once primary nucleation has begun, the crystal size distribution begins to take shape.



1 Feed location, undersaturated
2 Solution cools to saturation
3 Enter "metastable" zone, nucleation begins
4 Rapid nucleation
5 Concentration decreases with crystal growth
6 Crystal growth during main cooling cycle
7 Exit location, supersaturated

Figure 13.6
Crystal Growth

The second important mechanism in crystallization is called secondary nucleation. In this phase of crystallization, crystal growth is initiated with contact. The contact can be between the solution and other crystals, a mixer blade, a pipe, a vessel wall, etc. This phase of crystallization occurs at lower supersaturation (than primary nucleation) where crystal growth is optimal.
Again, no perfect theory is available to model secondary nucleation and it's behavior can only be anticipated by experimentation. Mathematic relationships do exist to correlate experimental data. However, correlating experimental data to model crystallization is time consuming and often considered extreme for batch operations, but can easily be justified for continuous processes where larger capital expenditures are necessary. For batch operations, only preliminary data measurements are truly necessary.

Since the solubility of salt in water decreases with decreasing temperature, as the solution cools, its saturation increases until it reaches supersaturation and crystallization begins (Figure 3). Cooling is one of the four most common methods of achieving supersaturation. It should be noted that cooling will only help reach supersaturation in systems where solubility and temperature are directly related. Although this is nearly always the case, there are exceptions. In Figure 13.7, note that $Ce_2(SO_4)_3$ actually becomes less soluble in water at higher temperatures.

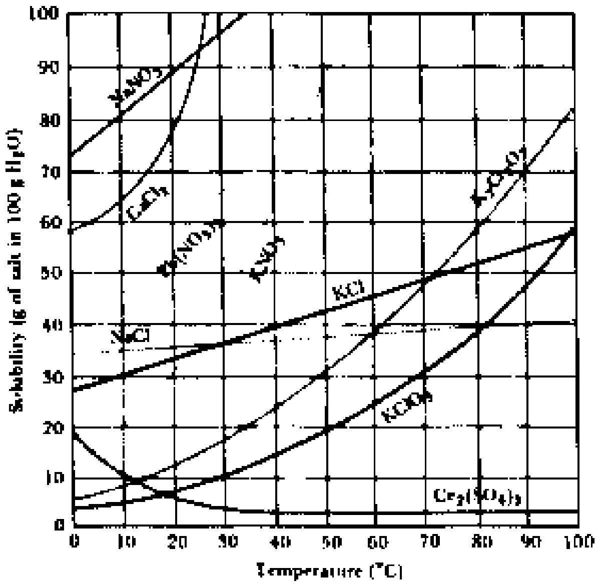


Figure 13.7
Typical Solubilities Curves

The four most common methods of reaching supersaturation in industrial processes are:

1. Cooling (with some exceptions)
2. Solvent Evaporation
3. Drowning
4. Chemical Reaction

Drowning describes the addition of a nonsolvent to the solution which decreases the solubility of the solid. A chemical reaction can be used to alter the dissolved solid to decrease its solubility in the solvent, thus working toward supersaturation. Each method of achieving supersaturation has its own benefits. For cooling and evaporative crystallization, supersaturation can be generated near a heat transfer surface and usually at moderate rates. Drowning or reactive crystallization allows for localized, rapid crystallization where the mixing mechanism can exert significant influence on the product characteristics.

Equipment Used in Crystallization

- **Tank Crystallizers**

  This is probably the age old and most basic method of crystallization. In fact, the "pot of salt water" is a good example of tank crystallization**.** Hot, saturated solutions are allowed to cool in open tanks. After crystallization, the mother liquor is drained and the crystals are collected. Controlling nucleation and the size of the crystals is difficult. The crystallization is essentially just "allowed to happen". Heat transfer coils and agitation can be used. Labor costs are high, thus this type of crystallization is typically used only in the fine chemical or pharmaceutical industries where the product value and preservation can justify the high operating costs.

- **Scraped Surface Crystallizers**

  An classic example may be the Swenson-Walker crystallizer consisting of a trough about 2 feet wide with a semi-circular bottom. The outside is jacketed with cooling coils and an agitator blade gently passes close to the trough wall removing crystals that grow on the vessel wall.

- **Forced Circulating Liquid Evaporator-Crystallizer**

  These crystallizers combine crystallization and evaporation, thus the driving forces toward supersaturation. The circulating liquid is forced through the tubeside of a steam heater. The heated liquid flows into the vapor space of the crystallization vessel. Here, flash evaporation occurs, reducing the amount of solvent in the solution (increasing solute concentration), thus driving the mother liquor towards supersaturation. The supersaturated liquor flows down through a tube, then up through a fluidized area of crystals and liquor where crystallization takes place via secondary nucleation. Larger product crystals are withdrawn while the liquor is recycled, mixed with the feed, and reheated.A typical configuration is given below in figure 13.8.

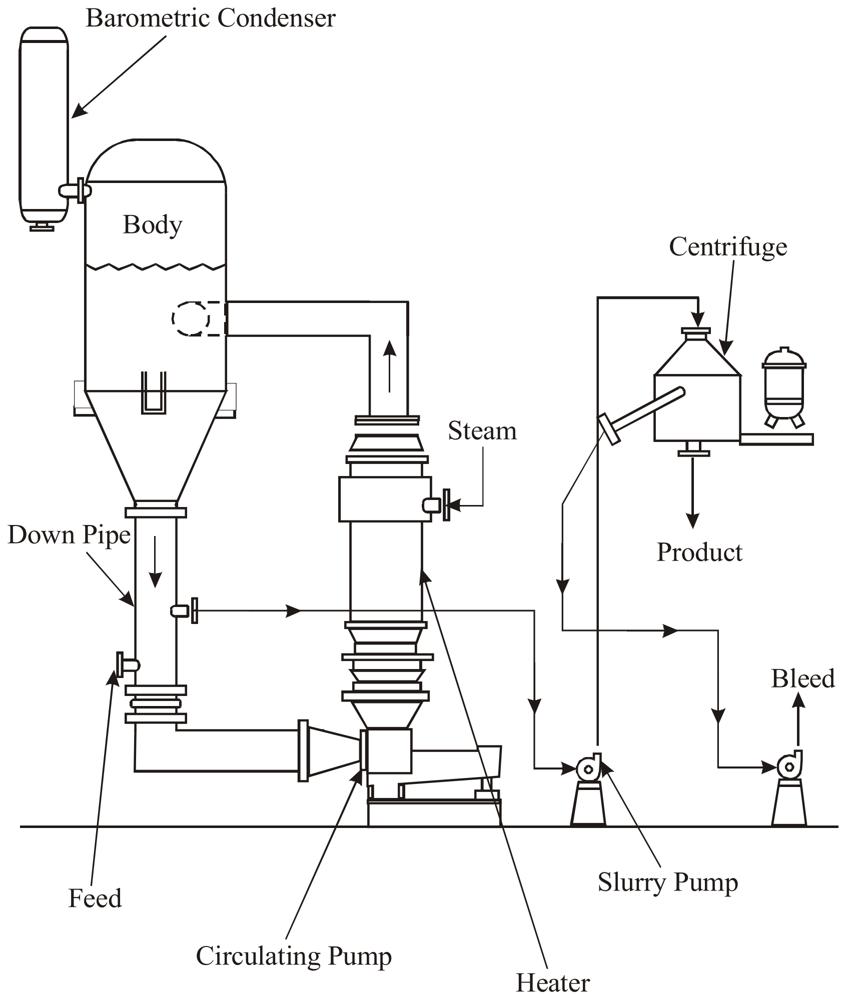


Figure 13.8
Continuous Crystallizer

13.4 Mixers

Mixers serve to put liquid in motion in order to achieve homogeneity of composition and eliminate the sedimentation process. They are driven by auxiliary equipment, such as a shaft, speed reducer or electric motor, to provide mixing action. They function by forcing sediment to flow in one direction and overcome the resistance during a liquid circulation flow in open reservoirs, ditches and canals. Mixers are also used to intensify physical and chemical processes in liquids, particularly the processes of gas and solid dissolution. Gas dissolution is usually used in sediment / waste water / anaerobic process. The intensified mixing operation is applied in order to lengthen the distance covered by gas bubbles and to prevent smaller bubbles from joining into bigger ones.

Direct drive, fast rotating mixers may also be employed to prevent surface scum from coming into existence and to destroy any surface scum that has already appeared.

Mixers are commonly categorized by the flow pattern they produce relative to the shaft centerline or the impeller axis in a fully baffled tank. Further, classfication is based on relative shear produced. The resulting divisions are: axial flow, radial flow, hi-shear, low-shear or high flow and specialized impellers (those which are normally used in unbaffled tanks).

- Mixers are also categorized by their specific action, or the action the cause within the liquid media.
- Agitators are used for mixing a product inside a vessel.
- Magnetic drive mixers are ideally suited for continuous stirred reactors (CSTR) and batch reactors where mixing and agitation must be contamination-free and leakage cannot be tolerated. The magnetic drive eliminates seals, and also the problems associated with rotating seals, such as leakage, contamination, and constant maintenance.
- Impeller / propeller mixers pump out in a radial direction generating a recirculating mixing pattern above and below the disc. This high shear, high power design is stable under varying liquid depths and is an excellent choice as a rapid mixer in shallow basins, solids suspensions in shallow or varying water depths, and is often used as the lower impeller in a multiple impeller design.
- Static or motionless mixers are fins, obstructions, or channels mounted in pipes, designed to promote mixing as fluid flows through the mixer. Most static mixers first divide the flow, then rotate, channel or divert it, before recombining the flow. Some static mixers create additional turbulence to enhance mixing.
- Turbine mixers include a wide range of general purpose mixing equipment, operating at reduced speeds provided by an enclosed gear drive with one or more multi-bladed impellers mounted on an overhung shaft. These mixers may be used on open tanks, when supported by a beam structure, or in closed tanks with a variety of seal and support arrangements. Because of the general-purpose capabilities of turbine mixers they may be used on almost any shape tank, of any size, with other drives or impellers.
- Extruder feed mixers have an integral extruder screw to mix and then extrude its contents.
- Kneaders provide a kneading motion to mix the contents of the mixer.

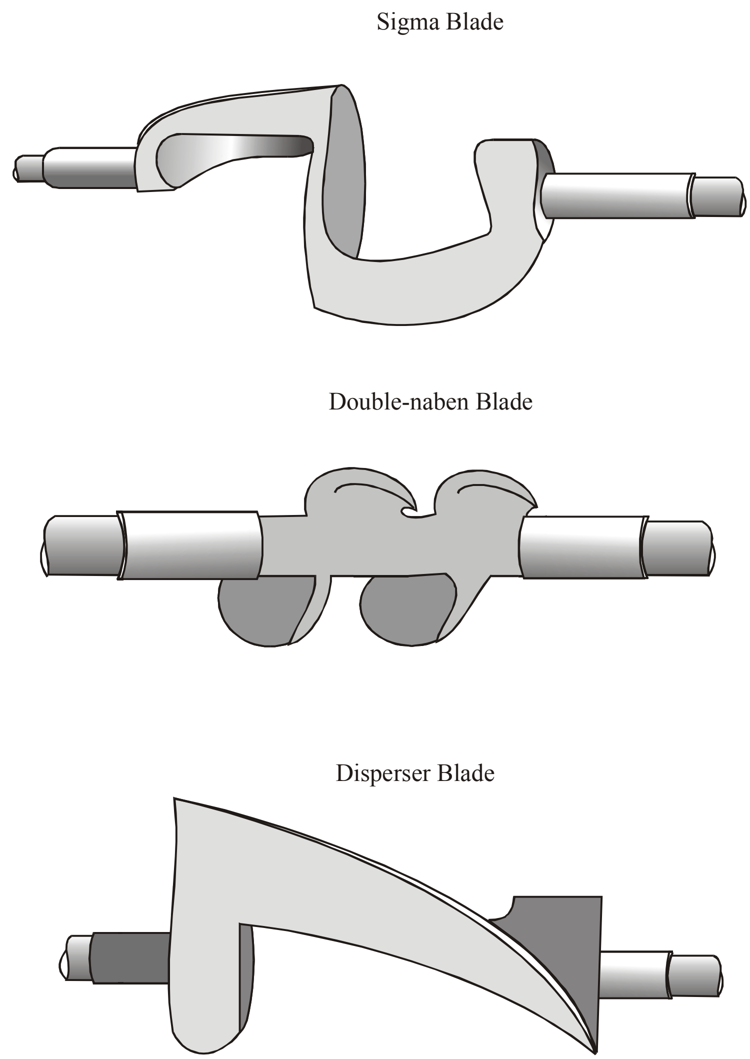


Figure 13.9
Kneader & Disperser Blades

- Planetary mixers have two mixing blades that rotate around individual shafts and the two blades further rotate around a center axis. The net effect is intermixing and stirring and shear.
- Turbine mixers have a circular trough with a housing in the center around which revolves a spider or a series of legs with plow shares or mold boards on each leg. This type of mixer is also known as a plow mixer.
- Screw mixers use a rotating screw that progresses around the periphery of a conical hopper. The screw lifts solids from the bottom of the hopper to the top, where the mixture flows by gravity back into the screw. Mixing occurs around the open screw, where the solids transported by the screw exit at various levels and are replaced by other solids at that level. The screw's shearing action also

intimately mixes the various components. Gross mixing action also occurs within the mixture by the velocity profile created in the conical hopper as it feeds the screw. This gross mixing action is most effective when the solids move along the conical hopper walls.

13.5 Mechanical Separation

Screeners, classifiers, shakers and separators are all used for classification of powders or other bulk materials by particle size as well as separation of particles by density, magnetic properties or electrical characteristics. Round and rectangular screeners, magnetic separators, electrostatic separators, rotary sifters, wet or concentrating tables, rake classifiers, classifying hydrocyclones, floatation systems and trommels are included in the category.

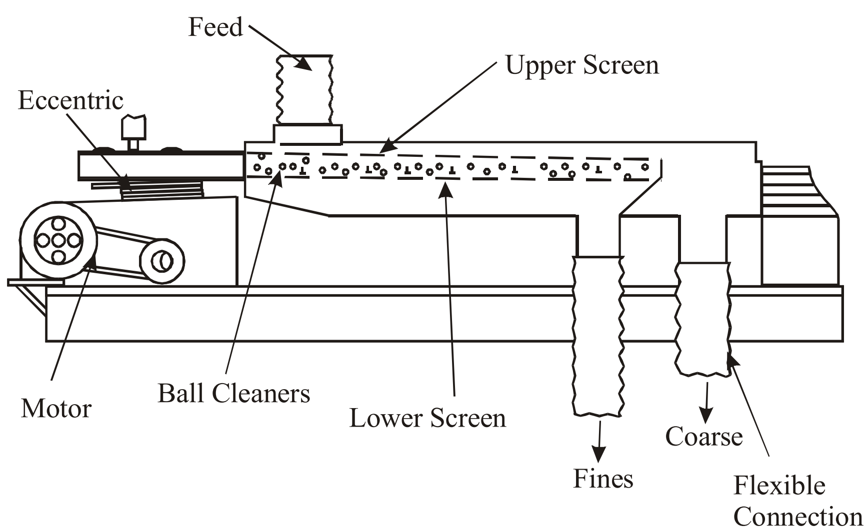
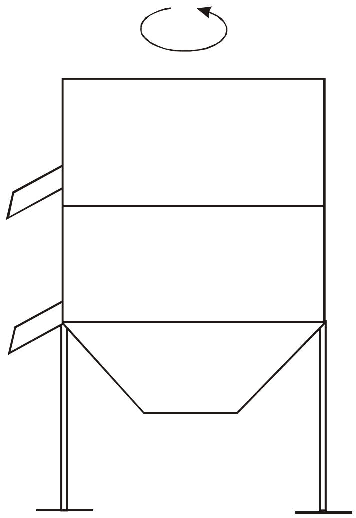
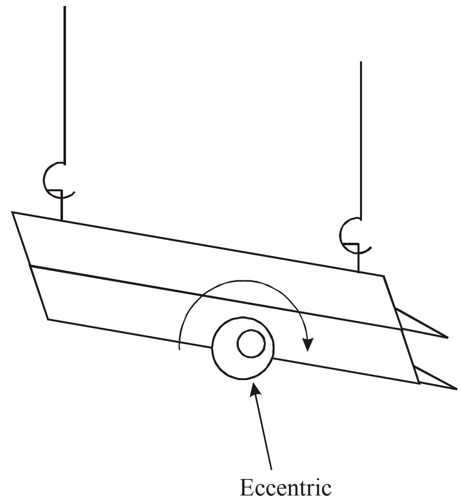


Figure 13.10
Gyrating Screen

Screeners are sifting units that are rotated as powder is fed into their interior. The finer particles fall through the sieve opening and oversized particles are ejected off the end. Rotary sifters or drum screeners are often used for deagglomerating or delumping type operations. Screeners are available in three main types: drum sifter, rectangular deck, and round deck. The various configurations are illustrated in figure 13.11.



(A)



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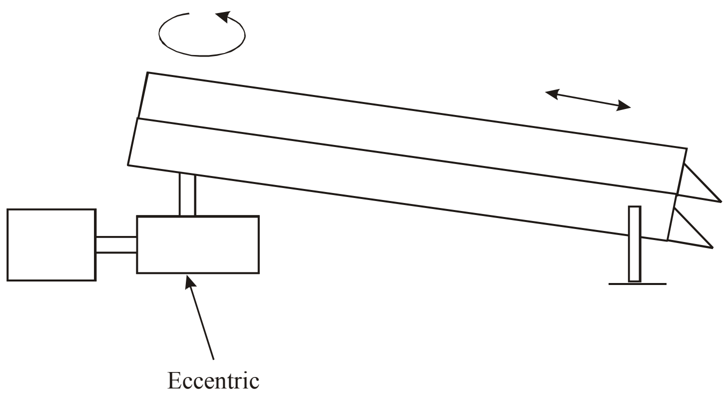

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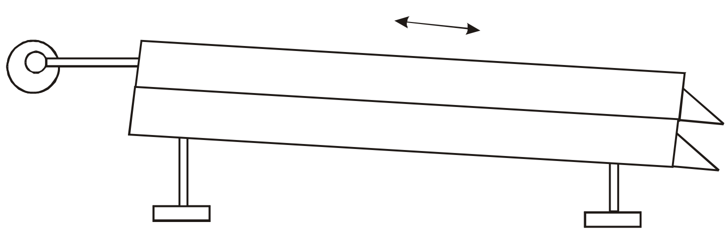

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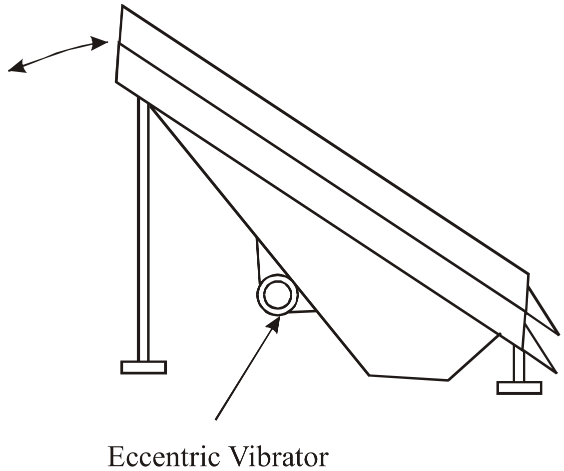

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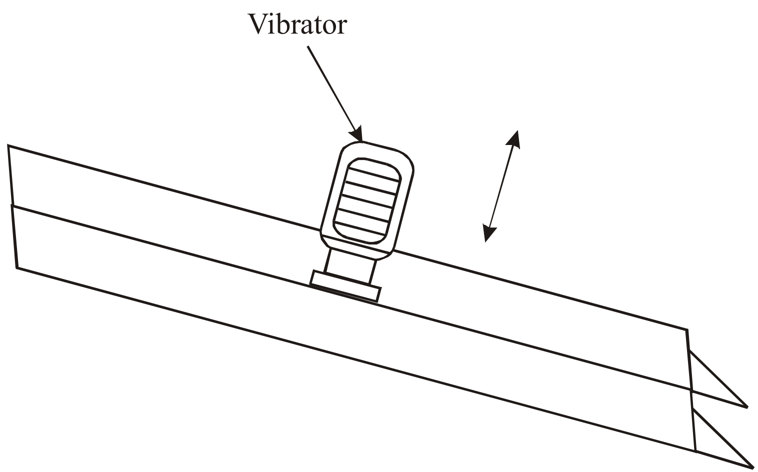

(F)

Figure 13.11
Screen Motions

Note:

(A) Gyrations in Horizontal Plane

(B) Gyrations in Vertical Plane

(C) Gyrations at one end Shaking

(D) Shaking

(E) Mechanically Vibrated

(F) Electrically Vibrated

- Air classifiers, cones or cyclones employ the spiral air flow action or acceleration within a chamber to separate or classify solid particles. Powders suspended in air or gas enter the cyclone and the heavier particles spiral out and down where they are collected. The air and finer particles flow up to the top where they may be passed to another cyclone with finer classification capability. A cyclone is essentially a settling chamber where the effects of gravity (acceleration) have been replaced with centrifugal acceleration. An air-classifying mill reduces friable materials such as polyesters, epoxies, acrylics, and sugar to fine and superfine (1 50- to 400-mesh) pieces. The material to be reduced first enters the mill's high-speed impact grinding chamber where a fixed-speed, rotating grinding plate with fixed hammers reduces it. Air moving through the mill then carries the particles to the classifying chamber where the classifier wheel rejects oversized particles and directs them back to the grinding chamber for further size reduction. The material circulates through this closed-loop environment until it's been reduced to the appropriate particle size. The classifier wheel's speed and the mill's airflow rate are adjustable to allow for a wide range of particle sizes. Heated or chilled air can enhance an air-classifying mill's performance.

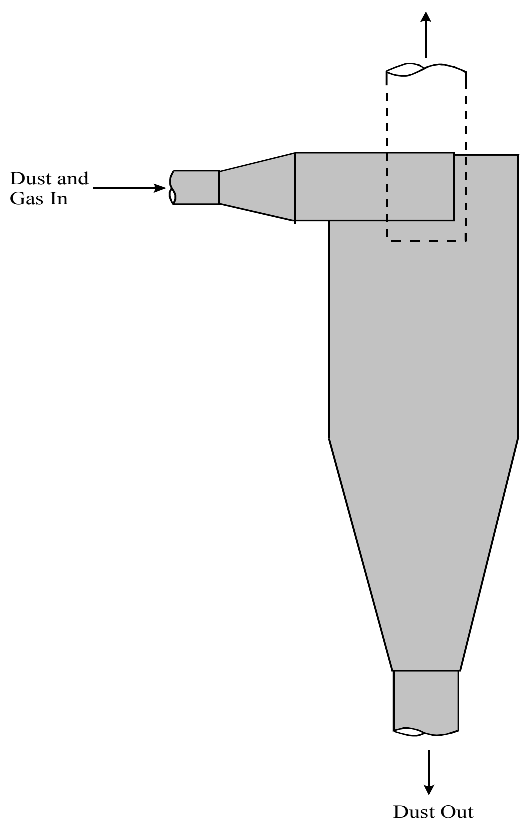

Figure 13.12
Cyclone Separator

- Concentrating tables or density separators screen bulk materials or minerals based on the density (specific gravity), size and shape of the particles. This group includes jigging equipment, hindered-bed settling devices, shaking table, spiral concentrators, concentrating or wet tables, hydraulic concentrating tables, constriction plate separators or specialized settling vessels. Most concentrating or density separation equipment are hydraulic or water-based, although pneumatic or air-based systems are also available.

- Electrostatic separators employ preferential ionization or charging of particles to separate conductors from dielectrics (nonconductors). The charged dielectric particles are attracted to an oppositely charged electrode and collected. The particles may be charged through contact electrification, conductive induction or high tension (ion bombardment).

- Floatation systems separate hydrophobic particulates from hydrophilic particulates by passing fine air bubbles up through a solid-liquid mixture. The fine bubbles attach to and lift or float the hydrophobic particles up where they are collected.

- Magnetic separators use powerful magnetic fields to separate iron, steel, ferrosilicon or other ferromagnetic materials from non-magnetic bulk materials. The magnetic field may be generated by permanent magnets or electromagnets.

- Rake, spiral and bowl classifiers use mechanical action to dewater, deslime or separate coarse bulk materials from finer materials or liquids. Drag classifiers consist of a chain-link conveyor or endless belt that is dragged through a solid-liquid mixture. Rake classifiers lift solid-liquid mixtures up onto a plate with a screen or rake. Spiral classifiers use an Archimedes pump screw to lift solid-liquid mixtures up onto a screen for dewatering. Bowl classifiers, bowl desilters, hydroseparators or countercurrent classifiers are other types or mechanical classifiers.

- Trommels are large rotary drum shaped with a grate-like surface with large openings. Trommels are used to separate very coarse materials from bulk materials such as coarse plastics from finer aluminum recycled material, coarse inorganic materials from organic wastes or large ore chunks from finer minerals.

- The demand for materials with finer and sharper particle size distributions is becoming the norm in many bulk solids processing industries. But often the only way plants can afford to accomplish these types of particle size distributions is to use a processor. Depending on your application's requirements, a processor can achieve particle classification by one of two methods. The simplest method uses sieves, which are primarily for coarse through fine grades of material. Sieves use screens with a specified mesh size to separate the particles, and vibration or air fluidization is applied to the sieves to maintain particle flow through the screens. The toll processor can stack the sieves to classify a range of particle sizes greater than 100 microns. The second classification method is air classification. An air classification machine uses air velocity to separate materials based on particle weight and size. It classifies particles ranging from 1 to 100 microns. Many air classifiers use a vaned wheel to control the particle size distribution. Water classifiers such as elutriators and classifying hydrocyclones use settling or flow in water or a liquid to separate or classify powdered materials based on particle size or shape.

13.6 Powder Compacting Equipments

These are used to shape powders as part of a forming process as well as to compress a wide range of materials into compact shapes for ease of transportation and ease of handling. Materials compressed by powder compacting equipment include powdered metals, ceramics, carbides, composites, pharmaceuticals, carbon/graphite, ferrites, explosives, chemicals, foods, nuclear fuel or other materials into compact shapes. Metal or ceramic powder compacts require additional processing such as sintering or forging to provide a finished part.

There are six main configurations of powder compacting equipment (although custom varieties are available). These types are defined either by the shape of the product they produce or the technology used to process materials.

- Briquetters and roll compactors turn fine, powdered materials into a briquettes, chunks, or sheets to improve handling, transportation, scrap disposal, storage or secondary processing. Briquetters often consist of a roll compactor with a serrated roll or a smooth roll combined with a granulator / chopper. Briquetters that form discrete cylindrical compacts also exist. Roll compactors with smooth rolls compact a powdered material into a sheet for the continuous production of ceramic or metal powder sheet or strip for filter applications or for clad / bimetal production. Some briquetters are used for fluid extraction and recovery.

- Cold isostatic presses (CIP) use a chamber to compact the powder or material placed in a sealed tool, bag or other flexible tooling. Cold isostatic presses use an oil-water mixture pressurized up to 100,000 psi. Flexible rubber or plastic tooling and steel mandrels are used in CIPing to produce performs with more complex shapes. CIP applications include refractory nozzles, blocks, and crucibles; cemented carbides, isotropic graphite, ceramic insulators, tubes for special chemical applications, ferrites, metal filters, preforms, and plastic tubes and rods.

- Hot isostatic presses (HIP) use an argon atmosphere or other gas mixtures heated up to 3000º F and pressurized up to 100,000 psi. Evacuated steel or metal cans or a sintered surface are used to contain and maintain a seal during HIPing. HIPs are used for densifying high performance ceramics, ferrites and cemented carbides, net-shape forming of nickel-base superalloy and titanium powders, compacting of high-speed tool steel, diffusion bonding of similar and dissimilar materials, and eliminating voids in aerospace castings or creep damaged blades.

- Pellet mills compress or extrude particles or fibrous materials into a cavity or die to form uniform cylindrical pellets. Compacted pellets are also formed using briquetters or tableting presses. Extruding pelletizers generate discrete and uniformly sized particles from a melt or a polymer (reclaimed scrap, post consumer or virgin plastic), liquid-solid pastes with a binder or other meltable materials. The melt or paste is extruded through a die with multiple orifices. The pellet is sheared off or chopped after cooling / drying. Several types of pelletizers are available such as hot face, air, and cold cutting and underwater.

- Rotary and multi-station tableting presses have multiple stations or punches for compacting pharmaceuticals into tablets or metal powders into simple flat or multilevel shaped parts like gears, cams, or fittings. Rotary types have a series of stations or tool sets (dies and punches) arranged in a ring in a rotary turret. As the turret rotates, a series of cams and press rolls control filling, pressing and ejection. Pharmaceutical tablet and high volume metal part production facilities often use high-speed automatic rotary presses.

- Single station presses are a type of powder compacting equipment that use a single action ram press with a die on both upper and lower punches. Single station powder compacting presses are available in several types basic types such as cam, toggle / knuckle and eccentric / rank presses with varying capabilities such as single action, double action, floating die, movable platen, opposed ram, screw, impact, hot pressing, coining or sizing.

13.7 Filtration

Liquid-solid filtration equipment is normally used to filter, thicken or clarify a mixture of different elements. Examples of liquid-solid filtration and separation equipment types include sedimentation equipment, gravity filtration equipment, vacuum filtration equipment, pressure filtration equipment, thickeners, clarifiers, and centrifugal separators. Sedimentation is a gravitational or chemical process that causes particles to settle to the bottom. Sedimentation equipment includes gravity sedimentation filters and flocculation systems.

- Gravity filtration uses the hydrostatic pressure of the prefilt column above the filter surface to generate the flow of the filtrate. Gravity filtration equipment includes bag filters, gravity nutshces and sand filters.

- Vacuum filters are available in batch (vacuum nutsches and vacuum leaf filters) and continuous (drum filters, disk filters and horizontal filters) operating cycles. Continuous vacuum filters are widely used in the process industry. The three main classes of continuous vacuum filters are drum, disk, and horizontal filters. All of these vacuum filters have the following common features:

  - A filtering surface that moves from a point where a cake is deposited under a vacuum to a point of solids removal, where the cake is discharged through mechanical or pneumatic means, and then back to the point of slurry application.

- A valve to regulate pressure below the surface.

- An apparently continuous operating cycle that is actually a series of closely spaced batch cycles.

Vacuum filtration equipment includes disc filters, horizontal belt filters, rotary drum filters (including precoat varieties), table filters, tilting pan filters, tray filters, and vacuum nutsche filters. A typical rotary vacuum filter is given in figure 13.13.

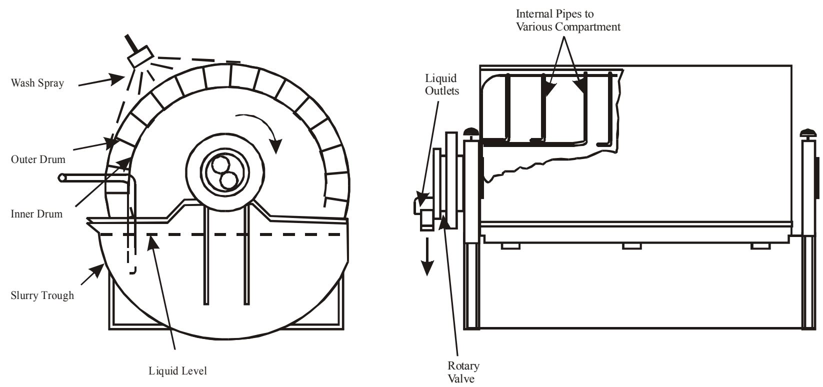

Figure 13.13
Rotary Vacuum Filter

- Pressure filters operate at superatmospheric pressures at the filtering surface. The media is fed to the machine by diaphragm, plunger, screw and centrifugal pumps, blowcases and streams from pressure reactors. Most pressure filters are batch, or semi-continuous, machines. Rotary drum pressure filters and some others have continuous operating cycles. Continuous machines are more expensive and less flexible than batch machines. Pressure filtration equipment includes automatic pressure filters, candle filters, filterpresses, horizontal plate pressure filters, nutsche pressure filters and vertical pressure leaf filters.

- Thickeners (figure 13.14) are used to separate solids from liquids by means of gravity sedimentation. Most thickeners are larger, continuous operation pieces of equipment. They are used for heavy-duty applications such as coal, iron ore taconites, copper pyrite, phosphates and other beneficiation processes. Common thickener types include conventional thickeners, high rate thickeners, lamella thickeners and tray thickeners.

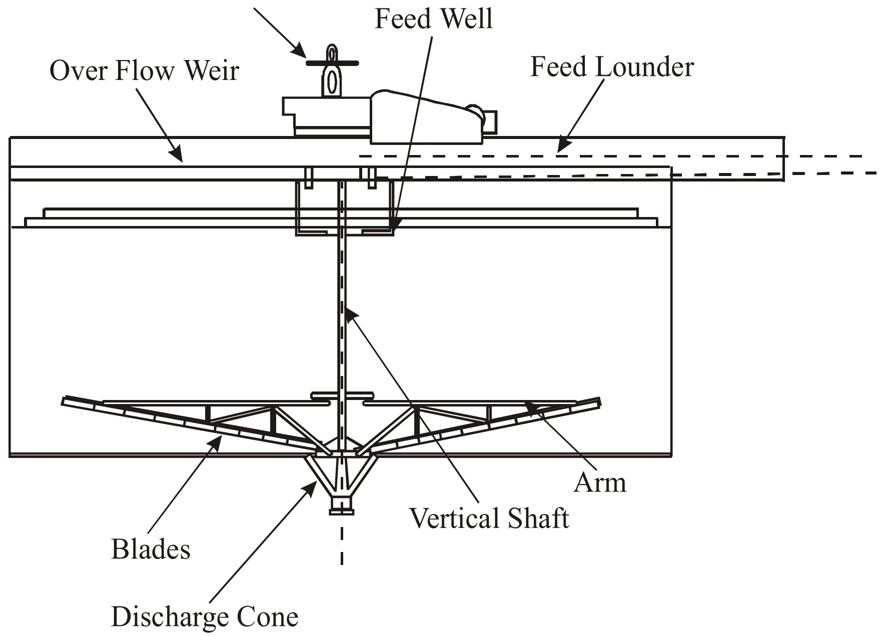

Figure 13.14
Gravity Thickener

The primary end product of clarifiers is a clarified liquid. They are virtually identical in design to thickeners, but have a lighter duty drive mechanism. They are generally used for industrial and residential waste. Clarifiers include conventional clarifiers, reverse osmosis equipment, sludge-blanket clarifiers and suction clarifiers.

13.8 Cryogenic Grinding

This technology can be used to most of the size reduction equipment. It's most commonly used with high-speed impact mills and attrition mills. Cryogenic grinding reduces heat-sensitive and nonfriable materials such as spices, plastics, organic dyes, and rubbers to medium-fine and fine (20- to 200-mesh) pieces. When reducing a heat-sensitive, nonfriable material, a toll processor either mixes cryogenic fluid directly with the material in the grinding chamber during grinding or embrittles the material by exposing it to a cryogenic fluid prior to grinding. The most frequently used cryogenic fluids (called cryogens) are liquid nitrogen and liquid carbon dioxide. A cryogen can lower material and grinding temperatures to -300 F, which increases the machine's particle size reduction capabilities by making a nonfriable material friable and minimizing the heat generated during grinding. Cryogenic grinding technology is also used in size reduction operations requiring inert atmospheres, such as those handling explosive or flammable materials.

13.9 Blending

The type of equipment used in mixing and blending operations depends on the materials to be combined. Certain blenders cause more degradation or generate more fines than is acceptable in a particular application, while others generate friction that can be detrimental to heat-sensitive materials. That's why a toll processor generally has several types of mixing and blending equipment available. For most powder blending applications a toll processor uses either mechanical agitation blenders or rotating vessel blenders. A mechanical agitation blender uses motor-driven agitators to agitate the materials in its stationary vessel until they are mixed together. Examples are ribbon blenders and conical-screw blenders, which both can die cohesive materials, such as plastics, pharmaceuticals, and spices. A rotating vessel blender has a rotating vessel that spins until the materials are mixed together. Examples are double-cone mixers (also called V-mixers) and drum tumblers, which both typically operate in batch mode and handle materials such as chemical powder blends, fertilizers, and plastic compound preblends.