Kirk-Othmer Encyclopedia of Chemical Technology.

SIZE ENLARGEMENT

Size enlargement concerns those processes that bring together fine powders into larger masses in order to improve the powder properties. Usually, they produce relatively permanent entities in which the original particles can still be identified, but this is not always necessary, as demonstrated in the formation of amorphous agglomerates by the cooling of melts, or by the production of weak and transient "instant" food agglomerates in which product strength need only be sufficient to withstand downstream handling, packaging, and transportation.

Size enlargement methods have been known for hundreds of years. The roots of the processes can be traced to such ancient techniques as the formation of clay bricks and other building materials, the hammering of implements from sponge iron, the manufacture of various items from precious metal powder, and the preparation of solid molded forms of medicinal agents (1). Agglomeration by heating and roasting techniques became established as a practical operation during the nineteenth century with the need to beneficiate and process fine coals and ores. In this century use of size enlargement has grown rapidly for a number of reasons. The application of high analysis nitrogen fertilizers in intensive agriculture led to development of noncaking and granulated, rather than powdered, products (2). The lower quality of available iron ore resulted in the need to upgrade the resource by grinding and rejection of the liberated impurities, followed by pelletization of the resulting fines into an acceptably coarse product (3, 4). Environmental considerations have led to the recovery of many dusts and fine waste powders that can be recycled after size enlargement (5). In addition, modern high volume processing requires consistent feeds with good flow properties, requirements that for powders can often only be met through some form of agglomeration.

Many diverse industries benefit from the use of size enlargement, ranging from the high value, relatively low volume requirements of pharmaceutical manufacturers to the tonnage requirements of the fertilizer and minerals processing industries. Benefits gained from size enlargement are as diverse as the industries in which the operation is used (1, 6–8). Dusting losses and caking and lump formation are avoided with improved product appearance through the granulation of fertilizers. The wet granulation of pharmaceutical powders produces nonsegregating powder blends with consistent flow properties. The granulation then feeds high capacity tableting devices which yield tablets of a defined and consistent dosage. Objects with useful structural forms and shapes are produced in powder metallurgy and ceramic forming. The pelleting of carbon black increases bulk density and improves handling qualities. Control of powder properties such as solubility, porosity, and heat-transfer capability can be attained through agglomeration procedures. Agglomeration in liquid suspension, such as the selective oil agglomeration of coal in water suspension, not only recovers the fine particles from the liquid, but does so selectively in that unagglomerated impurity particles remain in suspension (see Carbon, carbon black; Ceramics; Coal conversion processes, cleaning and desulfurization; Coffee; Drying; Fertilizers; Iron; Metallurgy, powder metallurgy; Pharmaceuticals; Plastics processing).

Table 1. Classification of Binding Mechanisms According to Rumpf[a]

Class	Mechanism	Representative examples	Refs.
solid bridges	sintering, heat hardening	induration of iron-ore pellets	(3, 4)
		sintering of compacts in powder metallurgy	10
	chemical reaction, hardening binders, “curing”	cement binder for flue-dust pellets	11
		ammoniation–granulation of mixed fertilizers	12
		oxidation of tar binders	13
	incipient melting owing to pressure, friction	briquetting of metals, plastics	(14, 15)
	deposition through drying	crystallization of salts in fertilizer granulation	16
		deposition of colloidal bentonite in dry iron-ore balls	(3, 4)
immobile liquids	viscous binders, adhesives	sugars, glues, gums in pharma-ceutical tablets	17
	adsorption layers	instantizing food powders by steam condensation	18
		humidity effects in flow of fine powders	
mobile liquids	liquid bridges (pendu-lar state)	flocculation of fine particles in liquid suspension by immiscible liquid wetting	8
		moistening–mixing of iron-ore sinter mix	(3, 4)
	void space filled or partly filled with liquid (capillary and funicular states)	balling (wet pelletization of ores)	(3, 4)
		soft plastic forming of ceramic powders	
intermolecular and long-range forces	van der Waals, electrostatic, and magnetic	adhesion of fine powders during storage, flow, and handling	19
		spontaneous dry pelletization of fine powders, eg, carbon black, zinc oxide	
mechanical interlocking	shape-related bonding	fracturing and deformation of particles under pressure	20
		fibrous particles, eg, peat moss	21

[a]Refs. 8 and 9.

1. Particle-Bonding Mechanisms

The mechanisms by which particles bond together and grow into agglomerates are affected by the specific size enlargement method. Nevertheless there are certain aspects of the bonding process that are essentially independent of the equipment and method. These aspects are described herein in general terms, followed by more detailed examination of the various size enlargement processes (see Coating processes, powder technology).

A classification of bonding mechanisms based on the fundamental nature of the interparticle bonds has been widely adopted in the literature and will be used here, together with the theoretical model used to estimate agglomerate strength (9). Rumpf’s classification into five categories of particle–particle bridging is summarized in Table 1 together with references from the literature to examples in each category. More than one bonding mechanism is likely to act in a given process. For example, in bonding by tar deposited through solvent evaporation, it is likely that oxidative hardening will also occur. In sintering ores, bonding through chemical reaction also contributes to strength. With very fine powders it is difficult to determine whether bonding through long-range forces or vapor adsorption predominates. Although mechanical interlocking of particles influences agglomerate strength, its contribution is generally considered small in comparison to other mechanisms.

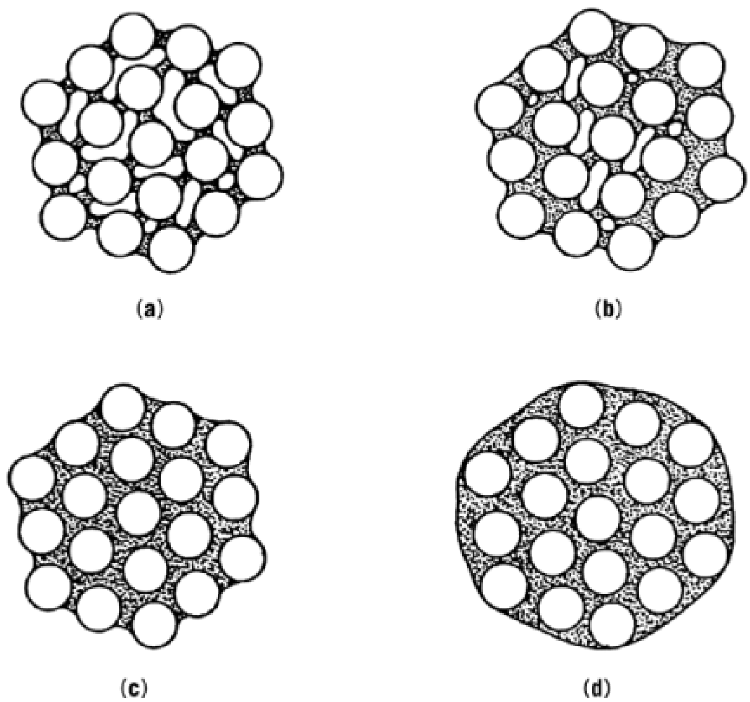

Fig. 1. States of liquid content in bonding by low viscosity liquids: (**a**) pendular, (**b**) funicular, (**c**) capillary, and (**d**) particles surrounded by liquid droplet (8).

1.1. Theoretical Strength of Agglomerates

Based on statistical-geometrical considerations, Rumpf developed the following equation for the mean tensile strength of an agglomerate in which bonds are localized at the points of particle contact (9):

$$\sigma_T \approx \left(\frac{1-\epsilon}{\epsilon}\right)\frac{H}{d^2} \qquad (1)$$

where σ_T is the mean tensile strength per unit section area, Pa; ϵ is the void fraction of the agglomerate;d is the diameter of the (assumed) monosized spherical particles in m; and H is the tensile strength in N, of a single particle–particle bond. To convert Pa to psi, multiply by 0.145×10^{-3}.

In a second main class of agglomerate bonding, particles are embedded in or surrounded by an essentially continuous matrix of binding material rather than having bonding localized at points of particle contact. An important example of this second type of binding is the case in which particles are held together by mobile liquids where adhesion occurs as a result of interfacial tension at the liquid surface and the pressure deficiency (capillary suction) created within the liquid phase by curvature at the liquid surface (22). The various regimes of low viscosity liquid which can exist in an agglomerate are shown in Figure 1.

In the pendular state, shown in Figure 1**a**, particles are held together by discrete lens-shaped rings at the points of contact or near-contact. For two uniformly sized spherical particles, the adhesive force in the pendular state for a wetting liquid (contact angle zero degree) can be calculated (19, 23) and substituted for H in equation 1 to yield the following, where γ is the liquid surface tension in N/m.

$$\sigma_T = \frac{9}{4}\left(\frac{1-\epsilon}{\epsilon}\right)\frac{\gamma}{d} \qquad (2)$$

When the void space in an agglomerate is completely filled with a liquid (Fig. 1**c**), the capillary state of wetting is reached, and the tensile strength of the wet particle matrix arises from the pressure deficiency in the liquid network owing to the concave liquid interfaces at the agglomerate surface. This pressure deficiency can be calculated from the Laplace equation for circular capillaries to yield, for liquids which completely wet the particles:

$$\sigma_T = C\left(\frac{1-\epsilon}{\epsilon}\right)\frac{\gamma}{d} \qquad (3)$$

where the parameter C has a theoretical value of 6 for uniform spherical particles and ranges between 6.5–8 for nonuniform particles (22, 24).

It is evident, by comparing equations 2 and 3, that tensile strength in the pendular state is about one-third that in the capillary state. Intermediate liquid contents in the funicular state (Fig. 1**b**) yield intermediate values that can be approximated as follows:

$$\sigma_T = sC\left(\frac{1-\epsilon}{\epsilon}\right)\frac{\gamma}{d} \qquad (4)$$

where s is the fractional filling of the agglomerate voids. This classical approach to predicting the strength of wet agglomerates has been reviewed (25) and some systems for which this approach needs to be modified are provided.

This discussion of agglomerate strength applies to established and static agglomerates. It has been demonstrated (26) that the viscosity of the binder liquid plays a key role during agglomerate formation, where it provides strength under dynamic strain, such as would occur with intensive mixing. It has been found that under industrially relevant conditions, the strength of the dynamic bridge exceeds the static strength by more than an order of magnitude.

1.2. Measuring and Correlating Agglomerate Strength

In practice, simple and quick test methods are most often used to assess the quality of bonding and other desirable properties of product agglomerates. Compression, impact, abrasion, and other types of tests are widely accepted in industry to characterize agglomerate quality in relation to subsequent handling and processing. The mode of agglomerate failure is complex in these tests, making it difficult to relate the results directly to theory.

Compression tests, in which agglomerates are crushed between parallel platens, are probably most universal. To obtain reproducible and accurate results, the rate of loading and method of load application must be strictly controlled. A variety of commercial testers are available to allow this needed control over the compression process. Several means of distributing the load uniformly at the point of contact are used, including covering the platen surface with compressible board. With pliable agglomerates especially, the fracture load is highly dependent on the rate of loading (27, 28).

During compression of spherical agglomerates, flattening takes place at the points of contact as particle–particle bonds fail locally and particles are driven into adjoining voids. Small, dense, wedge-like elements form in the regions adjacent to the platens and the agglomerate fails in tension along a circumferential crack joining the poles of loading (double-cone failure) (28, 29). Internal frictional effects must be overcome in this compression process, in addition to the tensile strength of the particle matrix given by equations 1–4. For corresponding tensile and compression measurements on wet limestone pellets in the range for which the mean tensile strength is $\sigma_T = 20 - 83$ kPa (3–12 psi), Rumpf found the ratio of tensile to compressive strength to be $\sigma_T/\sigma_C \approx 0.5 - 0.77$ (9).

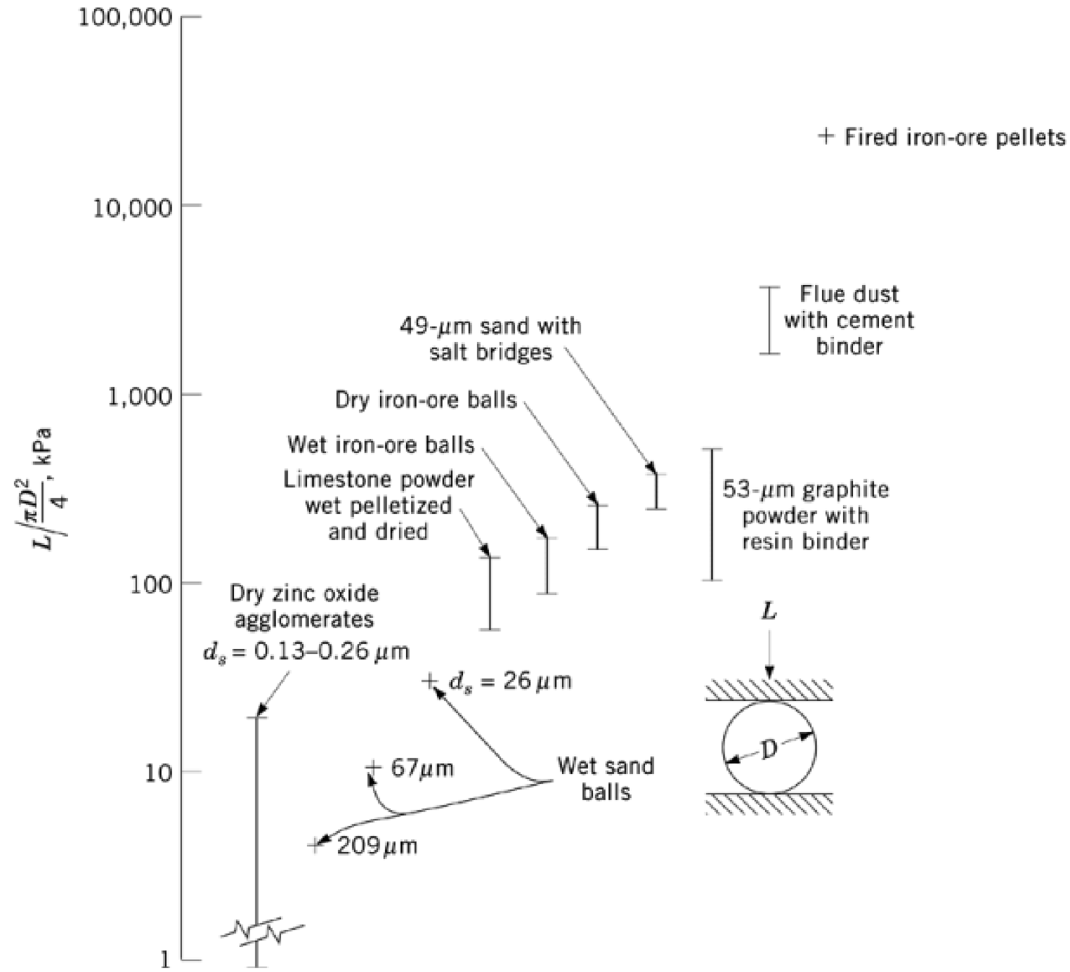

Fig. 2. Compression strength of agglomerates formed into spherical shape by tumbling where d_s=mean disperse particle diameter (8). To convert kPa to psi, multiply by 0.145.

For approximately spherical agglomerates, compression strength is calculated as follows:

$$\sigma_C = L/\left(\pi D^2/4\right) \qquad (5)$$

where L is the compression force at failure in N, and D is the agglomerate diameter in m. Some typical compressive strengths of various agglomerates are indicated in Figure 2.

According to equation 5, compression test data can usually be correlated through a log–log plot of load at failure against pellet diameter to yield a straight line whose slope is often, but not always, equal to 2 (30, 31). The intercept of such a plot is related to the compressive strength, σ_C. Because ultimate failure occurs in tension as explained above for the double-cone failure mechanism, this compressive strength factor can subsequently be correlated with a bonding mechanism through equations 1–4 to account for the effect of such parameters as particle diameter, agglomerate porosity, and the strength of particle–particle bonding (22, 32).

Alternatively, empirical relationships can be developed to account for the effects of these parameters describing the particle matrix (33, 34).

Several other tests of agglomerate quality are done routinely, the details depending on the practice accepted in a specific industry. For iron ore, a drop test is used on wet agglomerates as a measure of their ability to withstand handling up to the point in the process at which they are dried and fired (3). A test might consist of dropping a number of agglomerates from a height of 300 or 450 mm onto a steel plate. The average number of drops required to cause fracture is the drop number. In the ASTM tumbler test for iron-ore pellets (E279) an 11.3-kg sample in the size range 6.4–38.1 mm is placed in a tumbler drum ca 910 mm diameter by 460 mm long and rotated at 24 rpm for a total of 200 revolutions. The drum is equipped with two equally spaced lifters 51 mm high. The abrasion index is given by the weight percentage of plus 6.4 mm material surviving the test and the dust index by the yield of minus 0.6 mm (30 mesh) material. In addition to the tumble drum attrition test, there is wide industry use of screen abrasion tests (35). Preweighed samples of rock salt or potassium granules, for example, are shaken on sieve shakers, with the abraded fines recorded as abrasion index. This can be done with or without the addition of steel ball grinding media in the sieve shaker. Other strength tests such as the Linder rotating-furnace procedure are used with iron-ore pellets in an attempt to determine their reducibility and breakdown under reducing conditions simulating those of a blast furnace (36).

Laboratory tests to assess the caking tendency of fertilizer granules consist of two parts (37). The first entails cake formation in a compression chamber under controlled conditions of air flow, humidity, temperature, etc. In the second part the cake is removed and its crushing strength determined as a measure of the degree of caking. In the tablet disintegration test outlined in USP XX, pharmaceutical tablets are contained in a basket-rack assembly which is immersed in a suitable dissolution fluid in a container and raised and lowered to agitate the tablets at a constant rate of 29–32 cycles per minute for a specified period of time (38). Acceptable tablets disintegrate completely by the end of the test period.

2. Types of Size Enlargement

Classification of size enlargement methods reveals two distinct categories (8, 39). The first is forming-type processes in which the shape, dimensions, composition, and density of the individual larger pieces formed from finely divided materials are of importance. The second is those processes in which creation of a coarse granular material from fines is the objective, and the characteristics of the individual agglomerates are important only in their effect on the properties of the bulk granular product.

Four principal mechanisms are used to bring fine particles together into larger agglomerates: agglomeration by tumbling and other agitation methods; pressure compaction and extrusion methods; heat reaction, fusion, and drying methods; and agglomeration from liquid suspensions.

3. Agglomeration by Tumbling and Other Agitation Methods

When fine particles, usually in a moist state, are brought into intimate contact through agitation, binding forces come into action to hold the particles together as an agglomerate. Capillary binding forces caused by wetting with water or aqueous solutions is the most common binding mechanism, but others such as intermolecular forces developed in extremely fine dry powders may also be used to form the agglomerates. Several different forms of agitation may be used. The rolling cascading action of disk and drum devices produces rounded or roughly spherical agglomerates; other types of mixers, described below, generally yield more irregular shapes.

Agglomerate growth can occur through a number of mechanisms, such as coalescence, crushing and layering, layering of fines, and abrasion transfer (40–42). More than one mechanism may occur simultaneously in a given process. Agglomerates of the order of 1–3 mm diameter formed by coalescence of fine feed particles, or recycled undersize product agglomerates act as seed nuclei for the process. The nuclei grow to larger sizes by the addition of layers of fines supplied continuously to the process.

To survive and grow in an agitated system, agglomerates must be able to withstand the destructive forces generated by the moving charge of powder. Equation 3 indicates that for given agitation conditions there is a maximum particle size that can produce sufficient tensile strength in the particle matrix to form agglomerates satisfactorily. The addition of fines to a given size distribution of feed material usually improves agglomeration not only because it reduces the mean particle size, but also because it improves packing and reduces voids in the system. Similarly, more intensive agitation conditions generally reduce the size of agglomerates produced from a given material. For wet agglomeration on disk pelletizers top feed size is usually 300–600 μm (ca 30–50 mesh) and at least 25% of the feed powder should be finer than 75 μm (ca 200 mesh) (43). In iron-ore balling, a grind with 40–80% of the material below 44 μm (325 mesh) is normally used.

Agglomerates formed by wet pelletization in balling drums and disks are generally considered to have their internal pores saturated with binding liquid, that is, to be in the capillary state of wetting (see Fig. **1c**) (41, 44). The weight fraction for the theoretical liquid content of such agglomerates is given by equation 6 where W is the weight fraction of liquid (wet basis); ϵ is the void

$$W = \frac{\epsilon\rho_L}{\epsilon\rho_L + (1-\epsilon)\,\rho_S} \qquad (6)$$

fraction in the agglomerates; and ρ_L, ρ_S are liquid and particle densities, respectively.

Equation 6 has been fitted to a wide variety of literature data in which solid density ranged from about 1–6 g/cm^3 and liquid densities were generally close to 1 g/cm^3 (44). The following relationships were found.

For average feed-particle diameters $<30\ \mu$m:

$$W = \frac{1}{1 + 1.85\frac{\rho_S}{\rho_L}} \qquad (7)$$

For average feed-particle diameters $>30\ \mu$m:

$$W = \frac{1}{1 + 2.17\frac{\rho_S}{\rho_L}} \qquad (8)$$

These relationships predict the binding liquid content for wet agglomeration with an accuracy of only ca 30%. The liquid content required to agglomerate a particular feed material depends, for example, on the interfacial properties of the system (45). Typical values of moisture content required for balling a variety of materials are listed in Table 2. Very accurate information on the optimum liquid content to agglomerate a particular feed material must be obtained from experimental tests.

3.1. Drum and Inclined-Disk Agglomerators

Although a wide variety of agitation equipment is used industrially to produce agglomerates, rotary drums or cylinders and inclined disks or pans are the most important equipment in terms of tonnages produced. Fertilizer granulation and iron-ore balling represent two of the largest applications (see Fertilizers; Iron).

Drum agglomerators (Fig. 3) consist of an inclined rotary cylinder powered by a fixed- or variable-speed drive. Feed material, containing the correct amount of liquid phase, agglomerates under the rolling, tumbling action of the rotating drum. The pitch of the drum (up to 10° from the horizontal) assists material transport down the length of the cylinder. A retaining ring is often fitted at the feed end to prevent spillback of feed. A dam ring may also be used at the exit to increase the depth of material and residence time in the drum. Liquid phase may be introduced either before or immediately after the solids enter the cylinder. With iron ores, the feed is usually premoistened wet filter cake, but water sprays may also be located inside the drum for moisture

Table 2. Moisture Requirements for Balling a Variety of Materials[a]

Raw material	Approximate size analysis of raw material, less than indicated μm (mesh)[b]	Moisture content of balled product, wt % H_2O
precipitated calcium carbonate	75 (200)	29.5–32.1
hydrated lime	45 (325)	25.7–26.6
pulverized coal	300 (48)	20.8–22.1
calcined ammonium metavandate	75 (200)	20.9–21.8
lead–zinc concentrate	850 (20)	6.9–7.2
iron pyrite calcine	150 (100)	12.2–12.8
specular hematite concentrate	106 (150)	9.4–9.9
taconite concentrate	106 (150)	9.2–10.1
magnetic concentrate	45 (325)	9.8–10.2
direct shipping open-pit ores	1680 (10)	10.3–10.9
underground iron ore	6.4 mm	10.4–10.7
basic oxygen-converter fume	1	9.2–9.6
raw cement meal	106 (150)	13.0–13.9
utilities–fly ash	106 (150)	24.9–25.8
fly ash–sewage sludge composite	106 (150)	25.7–27.1
fly ash–clay slurry composite	106 (150)	22.4–24.9
coal–limestone composite	150 (100)	21.3–22.8
coal–iron-ore composite	300 (48)	12.8–13.9
iron-ore–limestone composite	150 (100)	9.7–10.9
coal–iron-ore–limestone composite	1180 (14)	13.3–14.8

[a]Courtesy of Dravo Engineers and Constructors.
[b]Tyler equivalent scale.

addition to aid control. In mixed fertilizer granulation, various solutions and slurries may be used, but water is the usual wetting agent. Various types of internal scrapers are in use to limit buildup of material on the inside surface and to provide a uniform layer to promote the correct rolling, tumbling action in the drum. Rubber flaps and liners as well as external knockers are used to limit buildup in fertilizer processing.

The drums used for fertilizer granulation range from 1.5 m in diameter by 3 m long, with installed power of 11 kW and rotation rates of 10–17 rpm that process 8 t/h, up to 3 m diameter by 6 m long, with installed power of 112 kW, rotation rates of 7–12 rpm, and a capacity of 51 t/h. These capacities exclude recycle so that the actual throughput of the drums may be much higher. Capacity depends primarily on the quantity of undersize and crushed oversize material recycled to the drum during continuous operation. The recycle ratio (amount recycled/output) varies from <1 ($>50\%$ output) to 5 or 6 for hard-to-granulate grades of fertilizer. The grade being granulated, differing formulations for the same grade, differing plant operations, ambient temperatures, and skill all influence capacity in fertilizer granulation (46).

A typical drum used to ball iron ore is 3 m in diameter by 9.5 m long, is driven at 12–14 rpm by a 45-kW drive, and produces 60 t/h product (3, 4, 47).

An inclined-disk agglomerator consists of a tilted rotating plate equipped with a rim to contain the agglomerating charge (Fig. 4). Solids are fed continuously from above or from the front onto the central part of the disk, and product agglomerates discharge over the rim. Moisture or other binding agents can be sprayed on at various locations on the plate surface. Adjustable scrapers and plows maintain a uniform protective layer of product over the disk surface and also control the flow pattern of material on the disk. Plate angle can be adjusted from 40 to 70° to the horizontal to obtain the best results, and both constant-speed and variable-speed motors are available as disk drives. Dust covers can be fitted when required. Characteristics of some of the range of inclined disks offered by one manufacturer are given in Table 3.

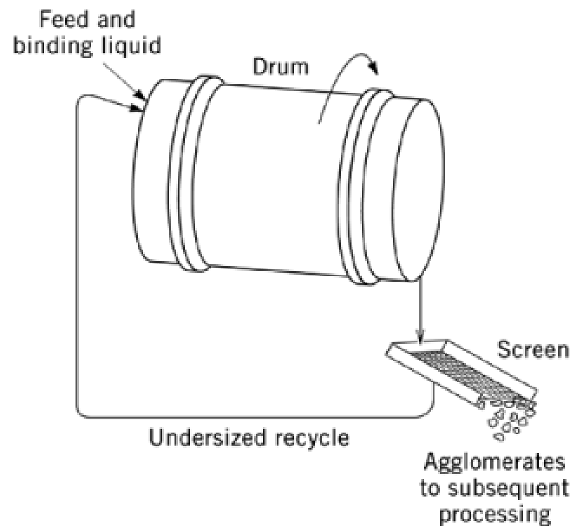

Fig. 3. Schematic of a drum agglomerator.

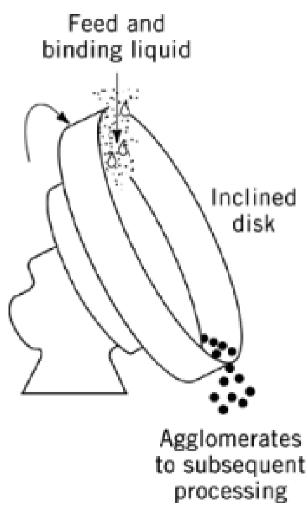

Fig. 4. Schematic of an inclined-disk agglomerator.

Operation and control of tumbling agglomerators is affected primarily by the character of the feed powder (size distribution, solubility), by its optimum liquid content for agglomeration, and by retention time in the device (3, 48, 49). An important parameter for drum agglomeration is the amount of undersize product that must be recycled. A disk agglomerator requires experimental adjustment in angle of inclination, speed of rotation, and position of spray(s) and scraper(s). Temperature of operation can be a significant variable for soluble ingredients.

An important feature of the inclined-disk agglomerator is its size separating ability. Feed particles and the smaller agglomerates sift down to the bottom of the tumbling load where, because of their high coefficient of friction, they are carried to the highest part of the disk before rolling downward in an even stream. Larger agglomerates remain closer to the top of the bed, where they travel shorter paths. In continuous operation the

Table 3. Characteristics of a Range of Inclined Disks[a]

Diameter, m	Depth, cm	Motor, kW	Speed, rpm	Approximate capacity[b], t/h
0.41	8.9	0.19	12–36 variable	
0.91	20.3	0.75	9–27	0.3
1.40	22.9	2.2	6.7–20.2	0.9
1.83	27.9	3.7	8.1–16.2	1.8
2.44	33.0	11.2	7.5–15	4.1
3.05	39.4	18.6	12.8 fixed	6.4
3.66	44.5	29.8	11.9	11
4.27	49.5	44.7	11.3	15
4.88	55.9	55.9	10.7	24
5.49	61.0	74.6	10.4	32
6.10	66.0	93.3	10.0	40
7.01	76.2	111.9	8.0	53
7.62	76.2	149.2	6.0	63

[a]Courtesy of Feeco International, Inc., 1980.
[b]Based on dry dust at 961 kg/m^3. Capacity depends on type of material and desired product (rates are average for nominal 1.3-cm pellets).

largest agglomerates are discharged from the top of the bed over the rim, while smaller ones and feed fines are retained for further growth. Because rotary drums do not possess the inherent classifying action of the inclined disk, agglomerates of a wide size distribution are discharged. Drums are therefore operated in closed circuit with screens to recycle the undersized (and crushed oversized, if present) material.

The inherent classifying action of inclined disks offers an advantage in applications that require accurate agglomerate sizing. Other advantages claimed for the inclined disk include less space requirements and lower cost than drums, as well as sensitivity to operating controls and easy observation of the agglomeration process. These latter features lend versatility in agglomerating a wide variety of materials of different degrees of ease of agglomeration.

Advantages claimed for the drum compared with the disk agglomerator are greater capacity, longer residence time for difficult materials, and less sensitivity to upsets owing to the damping effect of a larger recirculating load. Dusty materials and simultaneous processing steps (chemical reaction or drying during agglomeration) can be handled more easily in a drum.

Many variations on the design of the basic inclined-disk and drum agglomerators are in use. A well-known addition to the inclined disk is a separate reroll ring beyond the rim of the main disk. Normally disks are relatively shallow in depth with a rim height ca 20% of the disk diameter, but deeper configurations are also in use, including those with multistepped sidewalls, deep pan or deep drum designs and a cone pelletizer. Drum agglomerators may incorporate internal baffles (50), lifting blades, or independent paddle shafts in certain applications (51).

3.2. Mixer Agglomerators

Mixers are used by various industries in which, by contrast with drum or disk equipment, internal agitators of several designs provide a positive rubbing and shearing action to accomplish both mixing and size enlargement. Horizontal pan mixers, pugmills, and other types of intensive agitation devices are used. The positive cutting-out action of such equipment can handle plastic and sticky powder feeds and its kneading action is claimed to produce denser and stronger granules than the tumbling methods although agglomerates of more irregular shape usually result (see Mixing and blending).

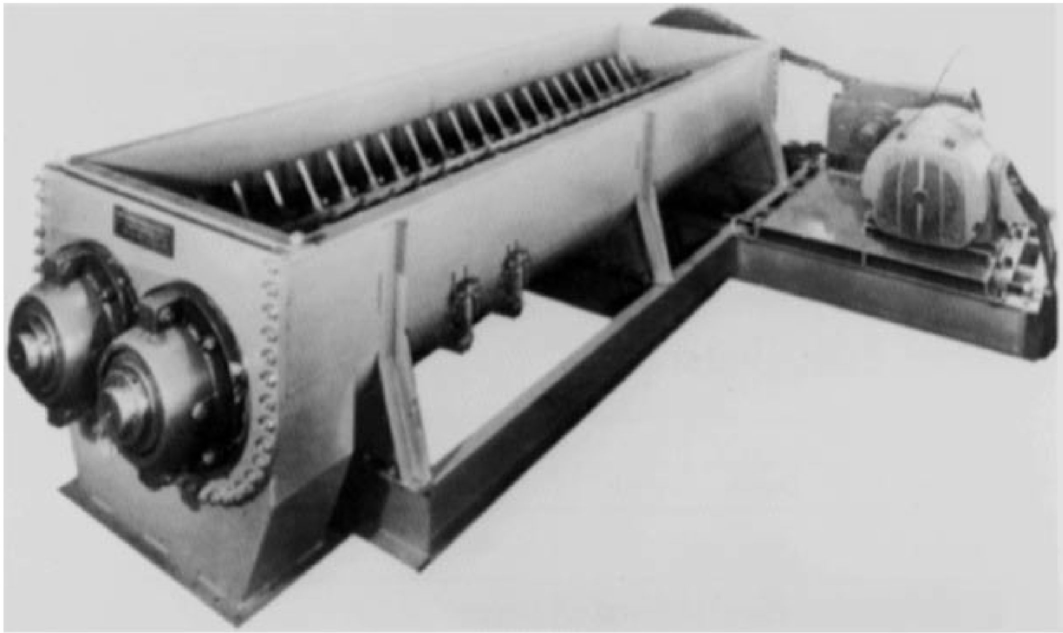

Fig. 5. Double-shaft mixer used in fertilizer granulation. Courtesy of Renneburg Division of Heyl & Patterson.

Pug mixers (blungers, pugmills, paddle mixers) such as that shown in Figure 5 have been widely used in the granulation of fertilizer materials and for the mixing, moistening, and microagglomeration of sinter-strand feed in both the ferrous and nonferrous metallurgical industries. These mixers consist of a horizontal trough with a rotating shaft to which mixing blades or paddles of various designs may be attached. The vessel may be of a single-trough design although a double-trough arrangement is most popular. Twin shafts rotate in opposite directions throwing the materials forward and to the center as the pitched blades on the shaft pass through the charge. Construction is robust, with the body of heavy plate (6.4 or 9.5 mm thick) and hardened agitators or tip inserts. Operational features include fume hoods, spray systems, and stainless steel construction. Provision can be made to feed materials at different points along the mixer as well as at the end to ensure that the entire mixing length is used and to add processing versatility. Capacities of these mixers cover a broad range, from typical levels of 20–30 up to several hundred tons per hour.

An intensive countercurrent pan mixer can be used to homogenize feed powders while adding binding liquid to help pregranulate extreme fines before pelletizing. This mixer consists of a rotating mixing pan and two eccentrically mounted mixing stars which operate in the direction opposite to the pan rotation. This equipment operates in a batch mode to handle typically about 30 t/h of fertilizer materials.

Shaft mixers operating at very high rotational speeds are also used to granulate extreme fines, such as clays and carbon black, which may be highly aerated when dry, and plastic or sticky when wet. These machines are generally single-shaft devices in which the paddles are replaced by a series of pins, pegs, or blades. The peg granulator (52) used to agglomerate ceramic clays in the china clay industry and the pinmixer (53) used in the wet pelleting of carbon black and pharmaceutical powders (54) are representative examples. As shown in Figure 6, these mixers consist of a cylindrical shell within which rotates a shaft carrying a multitude of cylindrical rods (pegs or pins) arranged in a helix. The shaft rotates at speeds critical to machine performance. Wet feed or dry feed that is immediately moistened enters the machine at one end and emerges as pellets at the opposite end.

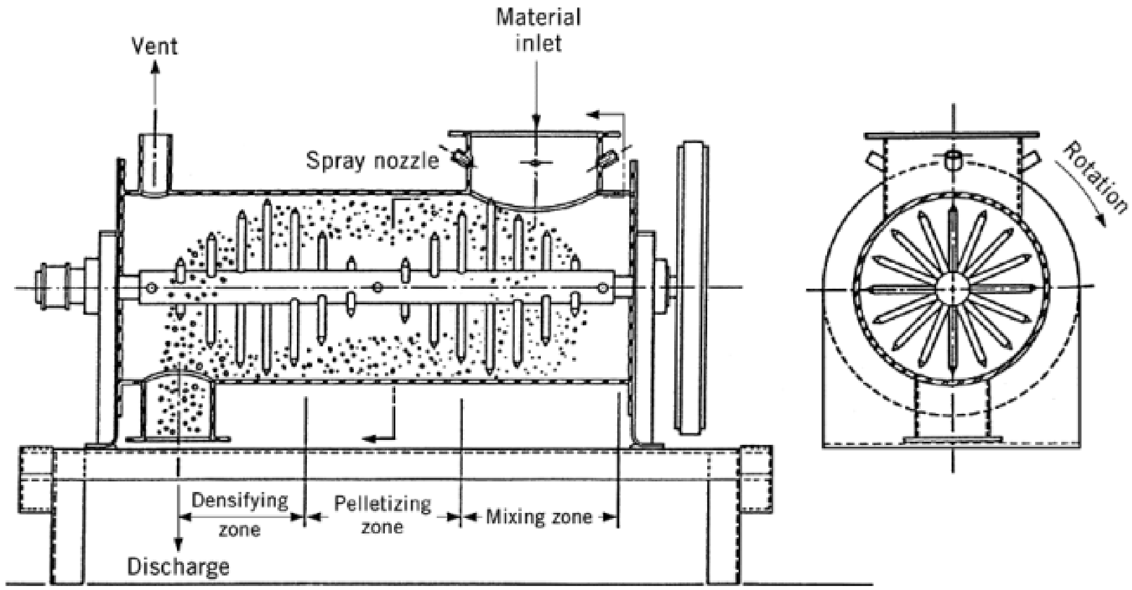

Fig. 6. Pinmixer used in wet-pelleting carbon black (53).

For intensive mixing and agglomerating at high speeds and very short retention times, the vertical plow mixer, also known as the Schugi mixer, can be used (55, 56). These high speed mechanical vertical plow-type agglomerators produce agglomerates in the 200 and 2000 μm size range, at typical retention times of 1–2 s.

3.3. Powder Clustering

Many applications of size enlargement require only relatively weak, small, cluster-type agglomerates to improve behavior of the original powder in flow, wetting, dispersion, or dissolution. Tableting feeds in pharmaceutical manufacture, detergent powders, and "instant" food products are examples. In these cases, agglomeration is accomplished by superficially wetting the feed powder, often with less than 5% of bridging liquid in the form of a spray, steam, mist, etc. The wetting is carried out in a relatively dry state in standard or specialized powder mixers in which the mass becomes moist rather than wet or pasty. Equipment used (57) includes sigma-blade and heavy-duty planetary mixers (33), horizontal cylindrical vessels containing mixing and chopping tools (58), and rotary drums of special design (59) to form a constant-density falling curtain along the drum axis into which dispersed binding liquid is directed to form small agglomerates.

Continuous-flow mixing systems are commonly used in the agglomeration of powdered food products. The instantizer agglomerator in Figure 7 is representative of these systems. In Figure 7 the feed powder is introduced to the moistening zone by a pneumatic conveyor and rotary valve. The dry powder falls in a narrow stream between two jet tubes which inject the agglomerating fluid in a highly dispersed state. Steam, water, solvents, or a combination of these are used. Air at ambient temperature is also introduced through radial wall slots in the moistening chamber to induce a vortex motion. Control of this air flow controls the flow pattern and particle temperature. The reduced temperature serves to condense fluid onto the particles while the vortex motion induces particle collisions. Clustered material then drops through an air-heated chamber onto a conditioning conveyor where sufficient time is allowed to reach a uniform moisture distribution. The material then passes to an afterdryer, cooler, and sifter, and is finally bagged (see Food processing).

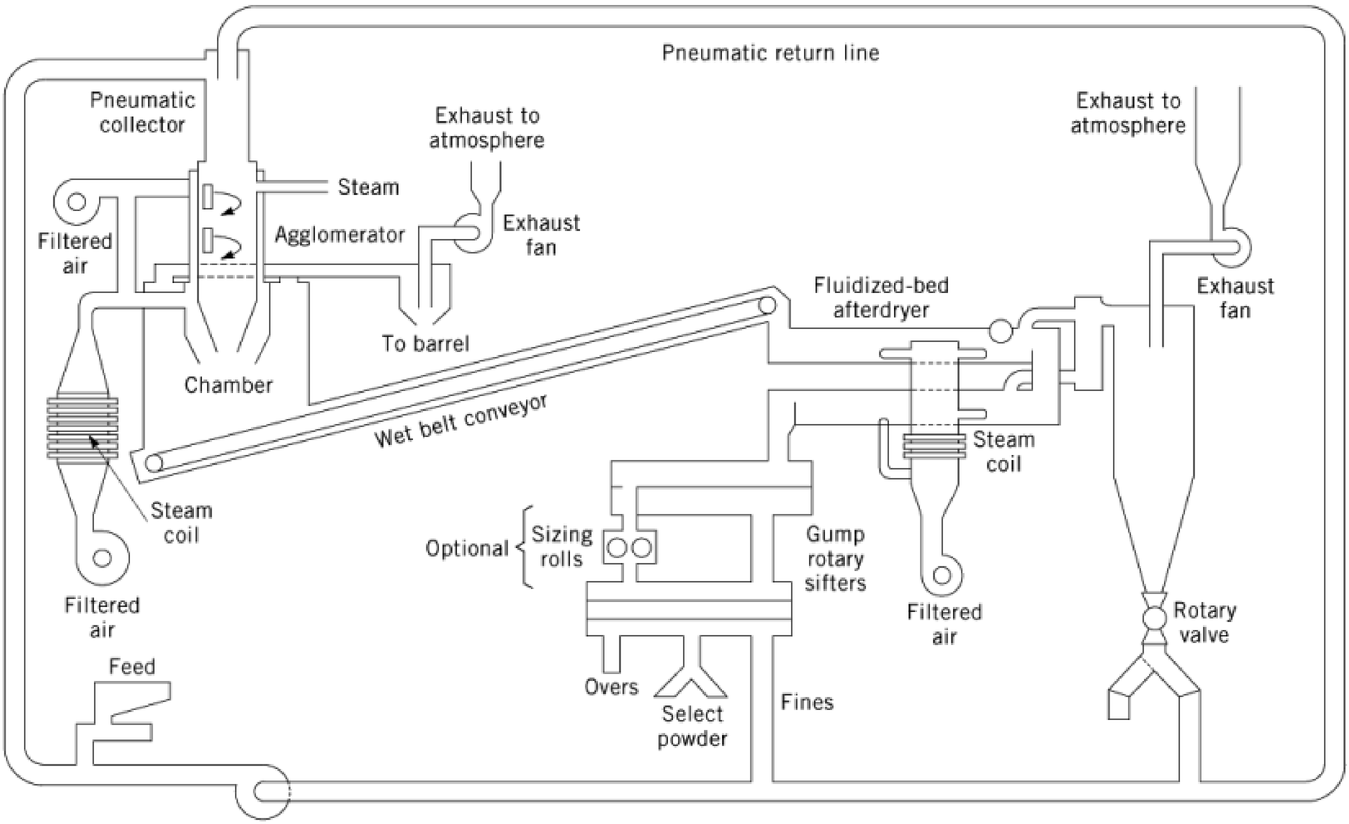

Fig. 7. Blaw-Knox Instantizer-Agglomerator. Courtesy of Buffalo Technologies Corp.

4. Pressure Compaction and Extrusion Methods

The compression techniques of size enlargement produce agglomeration by application of suitable forces to particulates held in a confined space. The various methods in use differ in both the means of pressure application and the method used to confine the powder. Unidirectional compaction in punch and die assemblies and in molding presses makes use of a reciprocating punch or ram acting on the particulates in a closed die or mold. In roll-pressing equipment, the particulate material is compacted by squeezing as it is carried into the gap between two rotating rolls. In extrusion systems the particulates undergo shearing and mixing as they are consolidated while being forced through a die or orifice under the action of a screw or roller.

Tableting, pressing, molding, and extrusion operations are commonly used to produce agglomerates of well-defined shape, dimensions, and uniformity in which the properties of each item are important and output is measured in pieces per hour (see Ceramics, ceramics processing; Pharmaceuticals; Metallurgy, powder metallurgy; Plastics processing).

The compaction process of void reduction may be considered to occur by two essentially independent mechanisms (60). The first is the filling of the holes of the same order of size as the original particles. This occurs as particles slide past one another; it is distinguished by the voids being filled with original particles that have undergone only slight size modification by fracture or by plastic deformation. The second process consists of the filling of voids that are substantially smaller than the original particles by plastic flow or by fragmentation. Many quantitative relationships have been suggested to represent this compaction process, usually in cylindrical die cavities (61–65). A successful theoretical design analysis for roll presses has been developed using small-scale laboratory measurements of the flow and compression properties of the feed powder (66–69).

The success of the compaction operation depends partly on the effective utilization and transmission of applied forces and partly on the physical properties and condition of the mixture being compressed. Friction at the die surface opposes the transmission of the applied pressure in this region, results in unequal distribution of forces within the compact, and hence leads to density and strength maldistribution within the agglomerate (70). Lubricants, both external ones applied to the mold surfaces and internal ones mixed with the powder, are often used to reduce undesirable friction effects (71). For strong compacts, external lubricants are preferable as they do not interfere with the optimum cohesion of clean particulate surfaces. Binder materials may be used to improve strength and also to act as lubricants.

4.1. Compacting Presses

In the automotive industry and other metal-working industries, coarse scrap-metal particulates are compressed and recycled to melting operations through piston-type briquetting presses (72, 73). Feed materials are typically cast-iron and steel borings or turnings, which tend to bond under pressure at least partially by mechanical interlocking. A reciprocating hydraulic press working on such materials might use a 56-kW hydraulics actuating pump with a rating of 318 t on a 12.7-cm diameter die. Briquettes of cylindrical shape 7.6 cm in length are produced at ca 3–4 t/h. Such compacting presses are not suited to larger tonnages when a small briquette is required. Their reciprocating nature is a disadvantage since this produces nonuniform loads on the drive motors.

4.2. Roll Briquetting and Compacting Machines

In roll presses, particulate material is compacted by squeezing as it is carried into the gap between two rolls rotating at equal speed. This is probably the most versatile method of size enlargement because most materials can be agglomerated by this technique with the aid of binders, heat, and/or very high pressures if needed (7, 74). The method generally requires less binder and therefore there is little or no requirement for drying the agglomerates. Simplified briquetting and compaction flow diagrams are shown in Figure 8. In briquetting machines, pillow shapes are formed by matching indentations in the rolls. Special shapes, such as the well-known fertilizer spikes, are also made by these machines. Precise design of these pockets based on practical experience is important to ensure optimum briquette density, minimum incidental feather (fines) production, and dependable pocket release of finished briquettes. In compaction machines the agglomerated product is in sheet form as produced by smooth or corrugated rolls. The compacted product can remain in sheet form or can be granulated into the desired particle size on conventional size reduction equipment. Polishing of the particles with or without additional moisture can be done in a rotary drum to round the particles and prevent additional dusting during handling.

Roll presses consist of the frame, the two rolls that do the pressing, and the associated bearings, reduction gear, and fixed or variable-speed drive (Fig. 9). Spacers between bearing housings prevent roll contact and allow adjustment of roll spacing. The frame of the press is designed so that all forces are absorbed internally. The rolls are forced together by a hydraulic system which may incorporate a safety valve to prevent overpressure if foreign material intrudes between the roll faces. The rolls consist of a continuous roll shaft, the roll body, and attached molding equipment. The molding surface may be either solid or divided into segments. Segmented rolls are preferred for hot briquetting, as the thermal expansion of the equipment can be controlled more easily. Segmented rolls can be made from harder materials more resistant to wear than can one-piece rolls.

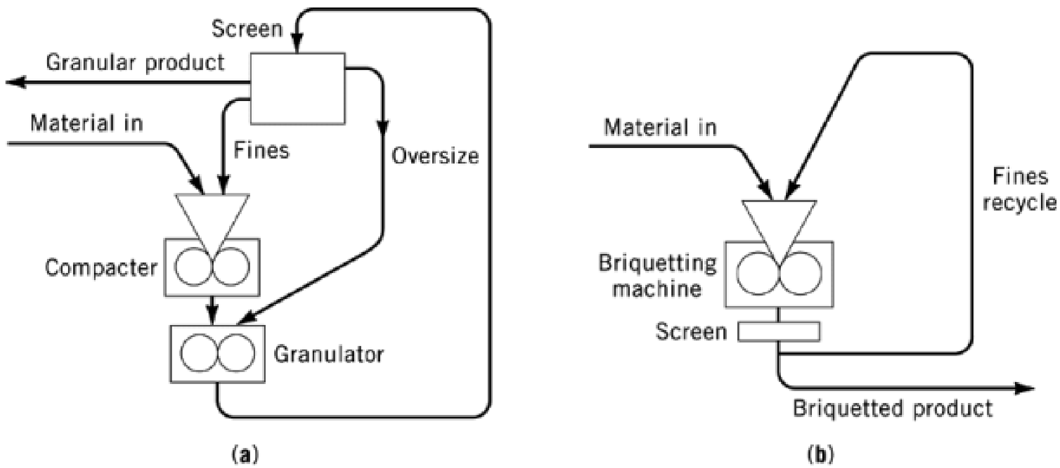

Fig. 8. Simplified compacting and briquetting systems: (**a**) compaction–granulation flow diagram and (**b**) briquetting flow diagram. Courtesy of Hosokawa Bepex Corp.

For fine powders that tend to bridge or stick and are of low bulk density, some form of forced feed, such as the tapered screw feeder shown in Figure 9, must be used to deaerate, precompact, and pressurize the feed into the nip. Large machines are available with up to five screw feeders to spread the flow across the rolls, and vacuum hoppers are also used to remove air when densifying low density feeds.

Capacity data for a variety of materials using a range of roll-press sizes are given in Table 4.

4.3. Pellet Mills

Pellet mills differ from roll briquetting and compacting machines in that the particulates are compressed and formed into agglomerates by extrusion through a die rather than by squeezing as they are carried into the nip between two rolls. Several types of equipment that use the extrusion principle are available. The die may be a horizontal perforated plate with rollers acting on its upper surface to press material through the plate. Rolls may be either side-by-side with material extruded through one or both of the rolls, or one or more small rolls may be fitted inside a larger die roll. In yet another design, two intermeshed gears are used and material is extruded through die holes located in the gear root.

The action of the roller and die assembly to produce a shearing and mixing action yields a plastic mix to be pushed through the die. Binders, plasticizing agents, and lubricants may be used to facilitate the process. Probably the most popular design of pellet mills in use is that shown in Figure 10, which utilizes a ring-type die and two or three rollers mounted in a vertical plane. Power is applied to the die to rotate it around the roller assembly, which has a fixed axis. Capacities range from <1 to ≤ 75 t/h operating from 75–300 rpm with a drive power of 7.5–592 kW. Dies with hole sizes from 2 or 3 to 30 mm are used. Capacities vary greatly depending on speed, hole and die size, moisture content, and other characteristics of the material being pelleted. Scores of materials can be pelleted, from catalysts and carbon materials to rubber crumb, wood pulp and bark, compound animal feeds, and many chemicals.

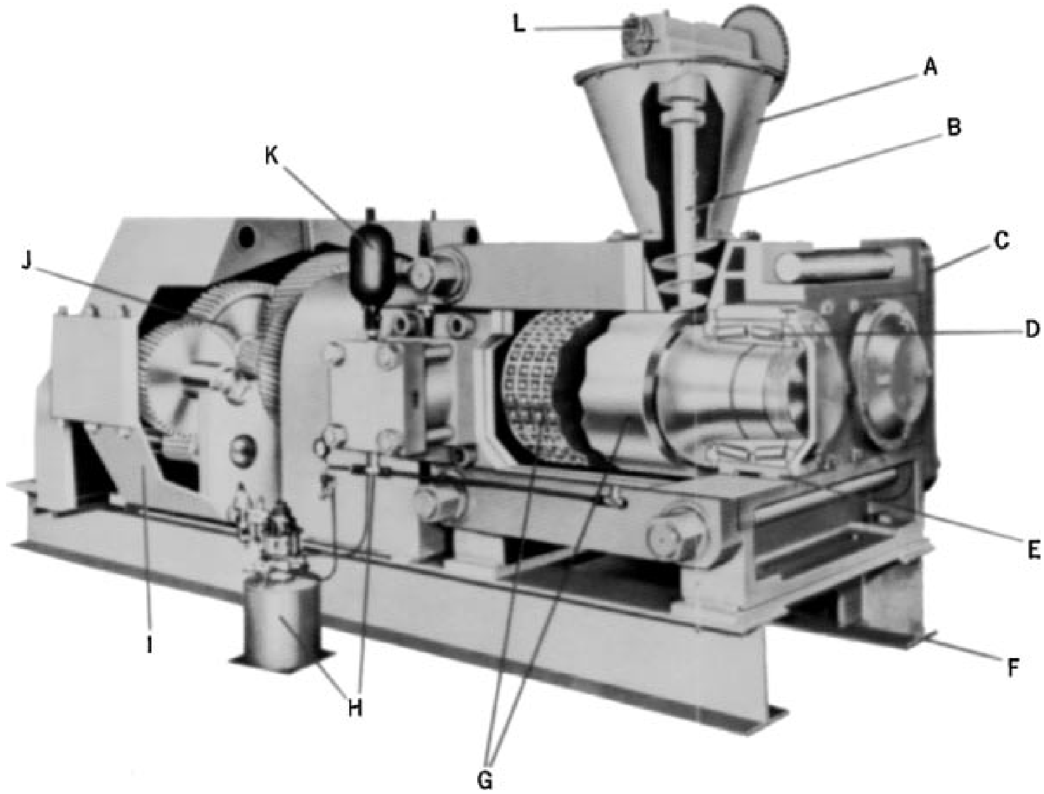

Fig. 9. Cutaway view of a briquetting–compacting machine. A, predensifying feeder; B, feeder screw; C, machine housing; D, antifriction bearing; E, machined bearing block; F, base frame; G, pocketed or corrugated rolls; H, hydraulic system; I, speed reducer; J, gears; K, hydraulic accumulator; and L, feeder drive. Courtesy of Hosokawa Bepex Corp.

5. Heat Reaction, Fusion, and Drying Methods

These methods of size enlargement depend on heat transfer to accomplish particle bonding. Heat may be transferred to the particle agglomerates, as in the drying of a concentrated slurry or paste, the fusion of a mass of fines, or chemical reaction between particles at elevated temperatures. Alternatively, heat may be removed from the material to cause agglomeration by chilling, as in the solidification–crystallization of a melt or concentrated suspension. Heat transfer may be direct from a heat-transfer fluid or indirect across a heat-transfer surface. As a consequence, heat transfer and drying equipment used is quite varied and includes packed-bed systems of particulates and aggregates, dispersed particle–fluid systems, and heating and cooling on moving surfaces. An equally wide variety of preagglomeration equipment is used to preform powders and pastes into agglomerates suitable for drying, firing, or chilling. Included are balling devices and pellet mills as described above and extruders using rollers, bars, or wiper blades to force plastic pastes through perforated plates or grids to preform the paste into rods and other shapes (75).

5.1. Sintering and Pelletizing

In extractive metallurgy (qv), sintering and pelletizing processes have been developed to allow processing of fine ores, concentrates, and recyclable dusts (3, 4). In this connection, sintering refers to a process in which

Table 4. Capacities for Roll Presses, t / h[a]

Roll diameter, cm	25.4	40.6	30.5	26.2	33.0	52.1	71.1	91.4	110
Maximum roll face width, cm	8.3	15.2	10.2	15.2	20.3	34.3	68.6	25.4	137
Roll separating force, t	23	45	36	45	68	136	272	327	600
Carbon									
coal, coke		1.8	0.91		2.7	5.4	23		70
charcoal			7.3			12			
activated					2.7	15			
Metals and ores									
alumina					4.5	9.1	25		
aluminum				1.8	3.6	7.3	18		
brass, copper	0.5			1.4	2.7	5.4	15		
steel-mill waste					4.5	9.1			
iron				2.7	5.4	14	33		
nickel powder					2.3	4.5			
nickel ore						18	33		
stainless steel				1.8	4.5	9.1			
steel								23	
bauxite		1.4				9.1	18		
ferro-metals						9.1			50
Chemicals									
copper sulfate	0.5	1.4		0.91	2.7	5.4	14		
potassium hydroxide				0.9	3.6	7.3			
soda ash	0.5				2.7	5.4	14		
urea	0.23					9.1			
dimethyl terephthalate	0.23				1.8	5.4			
Minerals									
potash						18	73		150
salt				1.8	4.5	8.2			
lime					3.6	7.3	14		
calcium sulfate							12	33	
fluorspar						4.5	9.1	25	
magnesium oxide						1.4	4.5		
asbestos						1.4	2.7		
cement						4.5			
glass batch						4.5	11		

[a]Courtesy of Hosokawa Bepex Corp., 1995.

fuel (5–10% coke fines) is mixed with an ore and burned on a grate. A cake of hardened porous material is the resulting agglomerate. In the pelletizing process as applied on a large scale to iron ores, discrete "green" balls produced in balling drums, disks, or cones are dried and hardened by passing combustion gases through a bed of the agglomerates, without fusing the agglomerates as in sintering.

Four separate sequential processes take place during these high temperature operations: drying, preheating, firing or high temperature reaction, and cooling. Ceramic bond formation and grain growth by diffusion are the two important mechanisms for bonding at the high temperatures (1093–1371°C) employed. Simultaneous useful processes may also occur, such as the elimination of sulfur and the decomposition of carbonates and sulfates. The highest tonnage applications are in the beneficiation of iron ores, but nonferrous ores may also be treated (76).

A traveling-grate sintering machine is depicted in Figure 11. The machine consists of a strong frame of structural steel supporting two pallet driving gears and steel tracks or guides. Traveling on the track is an endless line of pallets with perforated bottoms or grates. The train of pallets passes first under a feeding

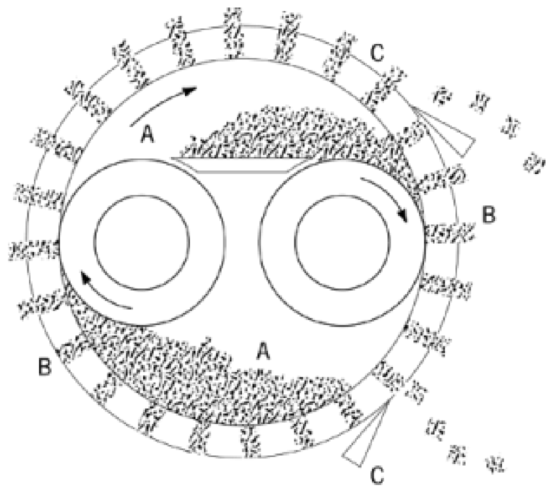

Fig. 10. Operating principle of a common design of pellet mill: A, loose material is fed into pelleting chamber; B, rotation of die and roller pressure forces material through die, compressing it into pellets; and C, adjustable knives cut pellets to desired length. Courtesy of California Pellet Mill Co.

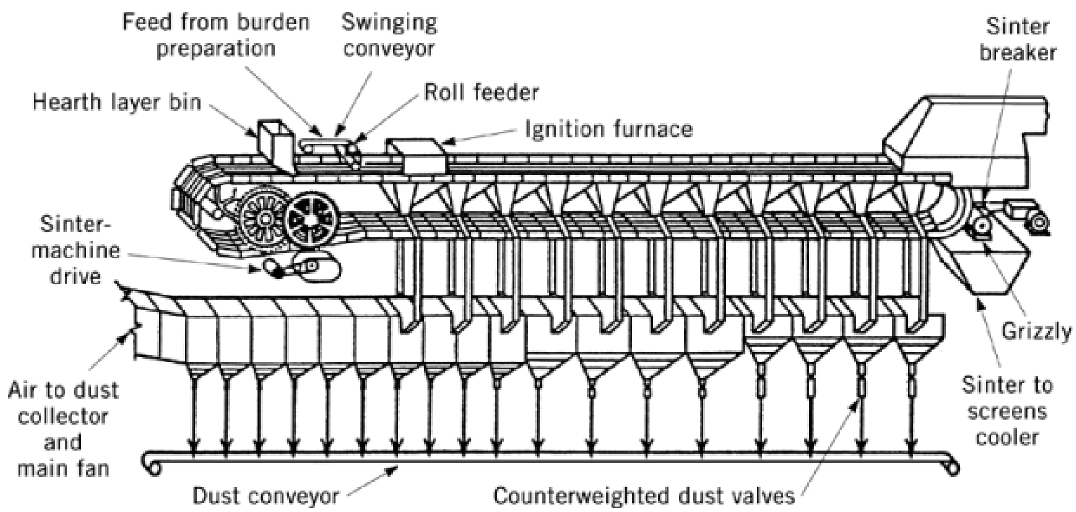

Fig. 11. Traveling-grate sintering machine (77).

mechanism which lays down a uniform layer of charge, then under an igniter, and finally over a series of wind boxes exhausted by fans. Each wind box is approximately equal in length and width. The speed of the train of pallets and the volume of air drawn through the charge are controlled so that the combustion layer reaches the grate just as the pallet passes off the machine. The sintering machine is a relatively small part of the equipment needed for a complex sintering plant. Auxiliary devices include conveying and storage equipment, mixing, nodulizing and proportioning equipment, fans, dust collectors, etc.

The capacity of a sintering strand is related directly to the rate at which the burning zone moves downward through the bed (78). This rate is controlled by the air rate through the bed, with the air acting in its function as the heat-transfer medium. The permeability of the sinter bed is thus very important in determining these rates and must be kept high for high throughput. Bed permeability is improved by preagglomeration of the feed material, proper feeding of a material to the sinter strand, and the use of 20–30% of recycled product material to support the needed coarse structure of the bed. A modern sinter strand might be 4 m wide by 61 m long with a capacity of 7200 t/d of ferrous sintered product.

In the pelletizing process for iron ore, wet balls 9.5–13 mm in diameter are dried and heat-hardened on traveling-grate machines adapted from the downdraft sintering grate. Installations differ in air-flow arrangements used to accomplish the process steps of drying, firing, and cooling (3). Pelletizing grates differ from sintering grates in a number of other ways. Pelletizing grates possess a multiplicity of windboxes divided into large groupings to allow recovery of sensible heat (eg, air from the cooling section may be used for drying or combustion purposes). This improves fuel efficiency and adds flexibility to the processing steps. In pelletizing, pellets are held for a long period, relative to that used in sintering, at closely controlled temperatures to effect hardening. In addition, pellet cooling is usually done on the same machine used for heat hardening, whereas sinter is cooled in separate equipment.

In addition to the common straight-grate machine, a circular-grate machine is also in use (79). The drying and firing of pellets may also be done in shaft furnaces or in a combined system using a traveling grate, followed by final firing in a kiln (80).

5.2. Drying and Solidification on Surfaces

In this type of equipment, granular products are formed directly from fluid pastes and melts, without intermediate preforms, by drying or solidification on solid surfaces. Surfaces formed by single or double drums are common. Drum dryers consist of one or more heated metal rolls on which solutions, slurries, or pastes are dried in a thin film. Drying takes place in less than one revolution of the slowly revolving rolls, and a doctor blade scrapes the product off in flake, chip, or granular form. A wide variety of products, such as cereals, fruits, starches, vegetables, meat and fish products, inorganic and organic chemicals, and pharmaceuticals are beneficiated in this way. In drum flakers, a thin film of molten feed is applied to the polished external surface of a revolving, internally cooled drum. Virtually any molten material that solidifies rapidly with cooling can be treated by this method. Although ambient water is normally the cooling liquid, chilled water or other coolants may be used if lower temperatures are needed. The cooled solid is scraped from the drum as a flaked or granular product. Many organic and inorganic food and chemical products are flaked in this way, including caustics, resins, detergents, sugars, waxes, pharmaceuticals, and explosives.

Molten materials can also be cooled to solid products on endless-belt systems, as shown in Figure 12. Some typical materials treated, product and feed characteristics, and capacities of belt cooling systems are given in Table 5.

As illustrated in Figure 12, a number of different feeding and discharge arrangements as well as surface sizes and speeds lend versatility to these methods based on drying and solidification on surfaces. For mobile slurries and fusible and/or soluble particles, these methods offer alternatives to more traditional size enlargement techniques.

5.3. Suspended Particle Techniques

In these methods of size enlargement, granular solids are produced directly from a liquid or semiliquid phase by dispersion in a gas to allow solidification through heat and/or mass transfer. The feed liquid, which may be a solution, gel, paste, emulsion, slurry, or melt, must be pumpable and dispersible. Equipment used includes spray dryers, prilling towers, spouted and fluidized beds, and pneumatic conveying dryers, all of which are amenable to continuous, automated, large-scale operation. Because attrition and fines carryover are common problems with this technique, provision must be made for recovery and recycling.

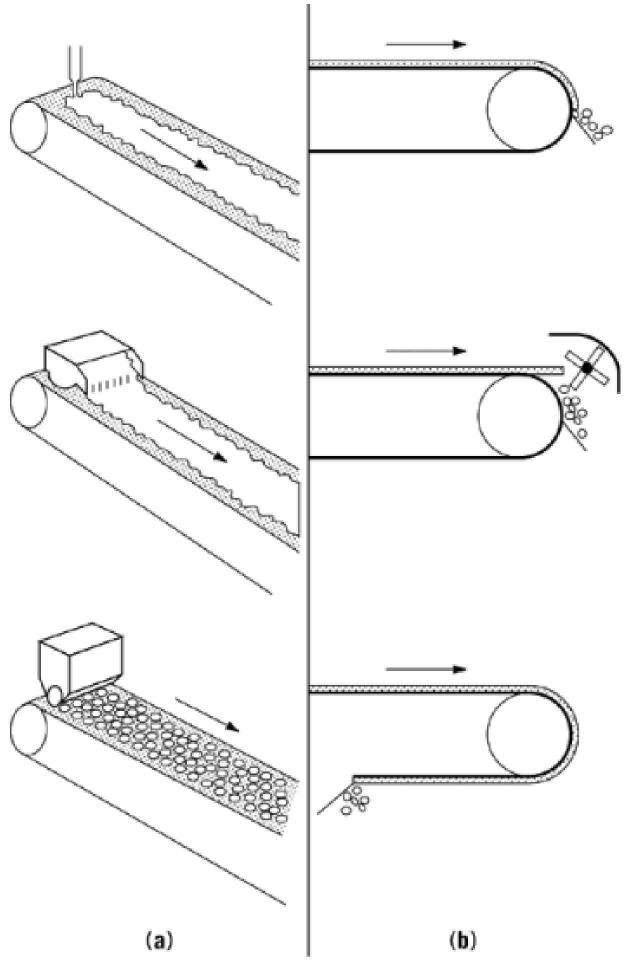

Fig. 12. (**a**) Typical-feeding and (**b**) discharge systems for endless-belt cooling of molten materials. Courtesy of Sandvik Process Systems Canada Ltd.

In the suspension methods, agglomerate formation occurs by hardening of feed droplets into solid particles, by layering of solids deposited from the feed onto existing nuclei, and by adhesion of small particles into aggregates as binding solids from the dispersed feed are deposited. The product size achievable in these methods is usually limited to ca 5 mm and is often much smaller (see Drying).

In spray drying, the largest particles produced are normally ca 1 mm diameter. Through design and operation of the dryer, however, larger agglomerated particles can be produced, and this technique is used to produce granular dried products in the pharmaceuticals and ceramics industries as well as coarse food powders with instant properties. Although the variables of spray dryer design and operation all interact to influence product characteristics, a number of these have important effects on product size and size distribution (81). In general, the product size is increased by decreasing the intensity of atomization and of spray–air contact, and by lowering the exit temperatures from the dryer. Higher liquid-feed viscosity and feed rate as well as the presence of natural or added binders also favor larger product size.

Table 5. Product Characteristics and Capacity Data for Some Materials Treated in Belt Cooling Systems[a]

Product	Thickness, mm	Feed temp, °C	Discharge temp, °C	Capacity, kg/(h·m^2)
resins				
phenolic	1.6	135	43	225
phenolic	1.2–1.3	138	33	277
sulfur	6.4	143	66	269
asphalt	3.2	218	52	90
urea	2.4	191	60	190
ammonium nitrate	1.6	204	71	439
chlorinated wax	1.6	149	38	303
sodium acetate	3.2	82	38	183
hot-melt adhesive	11.1	166	39	70
wax blend	0.6	132	29	129
epoxy resin	1.0	177	38	195

[a]Courtesy of Sandvik Process System Canada Ltd., 1995.

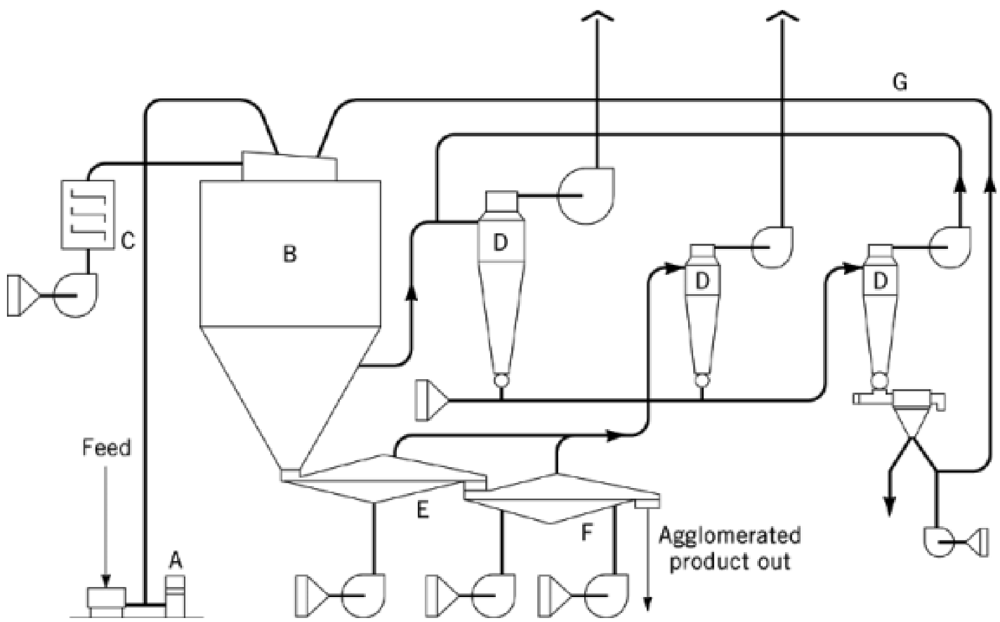

Fig. 13. Spray-dryer system designed for production of agglomerated food powders with instant properties (82): A, liquid-feed system; B, spray-dryer chamber; C, drying air heater; D, cyclones for fines recovery; E, vibrofluidizer as afterdryer; F, vibrofluidizer as aftercooler; and G, fines return to drying chamber.

The flow sheet in Figure 13 shows one example of a system designed to yield agglomerated products. Coarse spray-dried instant food powders are produced directly from liquids in this system. Two stages of agglomeration take place. The initial stage occurs in the atomization zone of the spray dryer, in which relatively cool air is used to retard the evaporation rate and enhance the agglomeration of fines. Further agglomeration is achieved by operating the spray dryer so that the powder is still moist when it leaves the dryer chamber. The agglomerated powder passes out of the bottom of the drying chamber to a vibrating fluid bed where drying is completed, and finally into a second fluid bed for cooling.

Spray cooling or solidification, more commonly known as prilling, is similar to spray drying in that liquid feed is dispersed into droplets at the top of a chamber. These congeal into a solid granular product as they travel down the chamber. The method differs from spray drying in that the liquid droplets are produced from a melt that solidifies primarily by cooling in the chamber with little, if any, drying. Product size is also generally larger (up to 3 mm dia) than in spray-dried materials. As a result of this relatively large prill size, the process is generally carried out in narrow but very tall towers to ensure that the prills are sufficiently solid when they reach the bottom. Because of the melt feed requirement, prilling is normally limited to materials of low melting point that do not decompose on fusion. Urea and ammonium nitrate fertilizers are traditionally treated by prilling (83, 84) (see Fertilizers).

Fluid-bed (85–87) and spouted-bed granulation (88, 89) both accomplish size enlargement and drying simultaneously by spraying feed liquids (solution, slurry, paste, melt) onto suspended layers of essentially dry particles. The seed-bed particles grow either by coalescence of two or more particles held together by a deposited binder material, or by layering of solids onto the surface of individual particles. Because multiple layers can be deposited, these granulation systems can produce larger granules than spray dryers. The two methods differ in the way the growing bed particles are agitated (Fig. 14). In a fluid bed, a suitable distributor such as a perforated plate passes hot gas uniformly into the base of the particle bed to suspend the particles. In the spouted-bed, hot gas is injected as a single jet into the conical base of the granulation unit causing the bed material to circulate in a fountain-like fashion. Spouted beds were originally developed as an alternative to fluidized beds as a means of contacting gas with solids. The operation of fluidized beds becomes less effective when particles greater than ca 1 mm diameter are to be treated. The gas–solids contacting efficiency is impaired at larger particle sizes, as more and more gas bypasses in the form of large bubbles. In contrast, ca 1 mm is the minimum particle size for which spouting appears to be practical. Thus, spouted-bed granulators allow larger granules to be formed than do fluid-bed granulators (see Fluidization).

6. Agglomeration from Liquid Suspensions

Size enlargement of particles contained in liquids is a frequent aid to other operations such as filtration, dewatering, settling, etc. Flocculation procedures are the traditional means used to promote such size enlargement (see Flocculating agents); the product is usually in the form of loose aggregates of an open network structure. Herein, attention is focused on less conventional techniques in which stronger bonding and specialized equipment are used to form large and more permanent agglomerates in liquid suspensions. Table 6 provides examples of such agglomeration processes, in which the objectives include not only the removal or capture of particles in suspension but also the production of granular, including spherical, material, displacement of as much suspending liquid as possible from the product, and the selective agglomeration of one or more components of a multiparticle mixture.

6.1. Agglomeration by Competitive Wetting

Fine particles in liquid suspension can readily be formed into large dense agglomerates of considerable integrity by adding a second or bridging liquid under suitable agitation conditions (103). This second liquid should be effectively immiscible with the suspending liquid and must preferentially wet the solid particles to be agglomerated. A simple example is the addition of oil to an aqueous suspension of fine coal. The oil readily adsorbs preferentially on the carbon particles and forms liquid bridges between these particles by coalescence during the collisions produced by agitation. Inorganic impurity (ash) particles are not wetted by the oil and remain in unagglomerated form in the aqueous slurry (see Coal conversion processes, cleaning and desulfurization).

The agglomeration phenomena that occur as progressively large amounts of bridging liquid are added to a solids suspension are depicted in Figure 15. The general relationships shown are not specific to a given system,

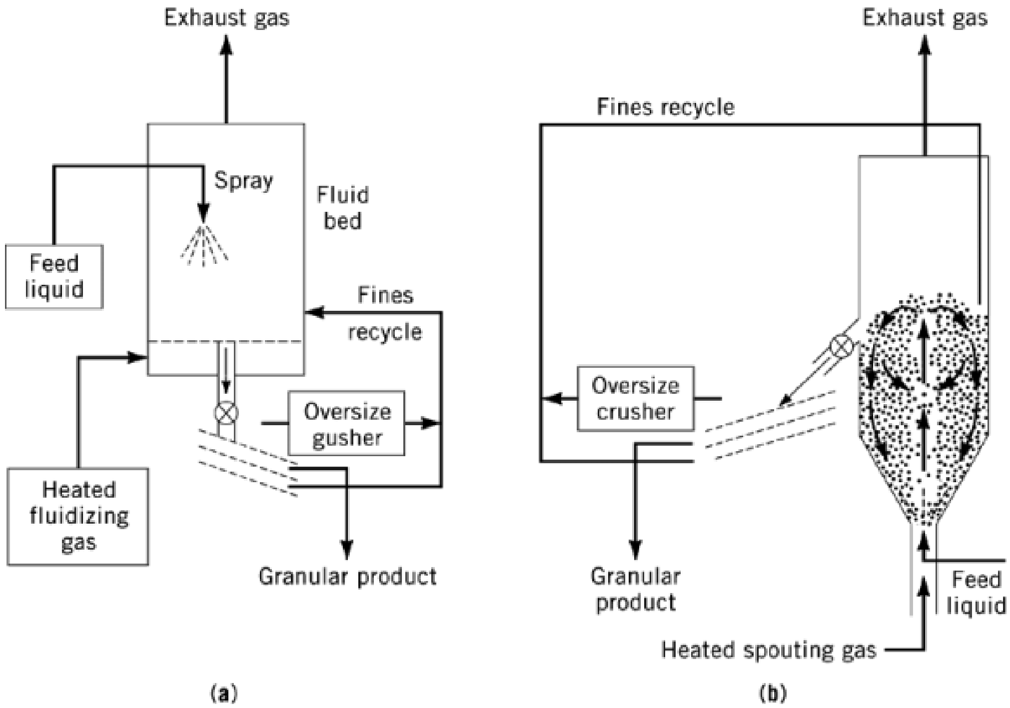

Fig. 14. Schematics of (**a**) fluid-bed and (**b**) spouted-bed granulation systems.

but relate equally well to siliceous particles suspended in oil and collected with water, or to coal particles suspended in water and agglomerated with oil. However, given the need for separation of valuable particles from associated gangue particles, the colloid and surface chemistry involved are usually quite complex. As in the flotation process, selective agglomeration by immiscible liquids depends on the relative wettability of surfaces, and the same fundamentals of surface chemistry apply to the conditioning of particles to yield the required affinity for the wetting liquid (see Flotation). A number of examples of ores, fossil fuels, and other particle mixtures in which particle conditioning and selective agglomeration are used to effect separation are given in Reference 103. Mineral recovery applications of the process have been summarized (101).

A most useful feature of the agglomeration technique is its ability to work with extreme fines. Even particles of less than nanometer size (ca 10^{-10} m) can be treated, if appropriate, so that ultrafine grinding can be applied to materials with extreme impurity dissemination to allow recovery of agglomerates of higher purity. A number of applications of liquid-phase agglomeration have reached either the commercial or semicommercial pilot scale of operation.

6.2. Oil Agglomeration for Fine Coal Cleaning

In the coal industry, increasingly greater amounts of fine coal must be processed owing to the need to crush to finer sizes for purposes of impurity liberation in low cost physical cleaning processes. Fine coal processing is a significant problem for the coal industry, owing both to the high cost of disposal of reject fines and to the mining cost of lost coal values.

The principal factors affecting coal agglomeration are similar to those affecting flotation, eg, amount and type of collecting oil, degree and type of agitation, pulp density, size consist, and wetting properties of

Table 6. Important Agglomeration Processes Carried Out in Liquid Systems

Process objective	Material treated and process used	References
sphere formation and production of coarse granular products	nuclear fuel and metal-powder production by sol–gel processes	(90, 91)
	manufacturing of small spheres from refractory and high melting point solids (eg, tungsten carbide) by immiscible liquid wetting	(92, 93)
	spherical crystallization: direct agglomeration of crystals during crystallization for drug delivery systems	94
removal and recovery of fine solids from liquid	removal of soot from refinery waters by wetting with oil	95
	recovery of fine coal from preparation plant streams to allow recycling of water	96,97
displacement of sus-pending liquid	dewatering of various sludges by flocculation, followed by mechanical drainage on filter belts, in revolving drums, etc	98,99
	displacement of moisture from fine coal by wetting with oil	96,97
selective separation of some components in a mixture of particles	removal of ash-forming impurities from coal and tar sands by selective agglomeration	96,100
	coal–gold agglomeration to recover very low concentrations of values in gold ore	101
	solvent extraction and simultaneous soil agglomeration to remediate oil-contaminated soil	102

the coal. The most significant operating parameter, however, is the oil concentration. As progressively larger amounts of bridging liquid are added to a suspension of fine particles, a variety of agglomerated products can result. Economic considerations require however, that the smallest quantity possible of oil be used, with the consequent production of very small microagglomerates. This approach requires both an efficient mixer to disperse the oil agglomerant, and centrifugal drying to reach product moisture requirements. A skimming or bubble flotation step has also been added to capture very small agglomerates lost in the screening operation. These process steps are seen in the flow sheet shown in Figure 16, and were used on commercial plants operated in the eastern United States (96) to produce 20–30 t/h of clean coal product agglomerates from 50% minus 325 mesh coal particles contained in a thickener underflow, material which was previously discarded as waste.

6.3. Liquid Waste Treatment

Persistent crude petroleum–water emulsions are produced in the recovery and extraction of heavier oils, eg, in the hot water processing of surface-mined oil sands and during *in situ* methods such as steam or water flooding. Oily sludges, formed from water contaminated by fine solids or soil and crude petroleum or bitumens, are an undesirable by-product of most oil recovery or refinery operations. Land farming as a disposal method is increasingly unacceptable, and in any case this and similar approaches ignore the opportunity to recover and reuse valuable aqueous and organic components of the oily wastes.

Liquid-phase agglomeration can play a significant role in oily waste treatment. In the examples discussed for coal, oil is added as an agglomerant to recover and beneficiate the solids. In liquid waste treatment, the waste stream provides the immiscible liquids, and it is then necessary to provide the solid adsorbent for the oil. The commercial fine coal agglomeration facility described in Figure 16 can be thought of as a sizable water treatment facility that recovers clean water containing only a few ppm of organics from the thickener at rates in excess of 2000 L/min. Dispersed, emulsified, and dissolved oils are all susceptible to removal in various degrees by this method.

This approach to liquid waste treatment requires synergy between different process stages or between various industries (industrial ecology). Suitably matched solid adsorbents and waste liquids must be available

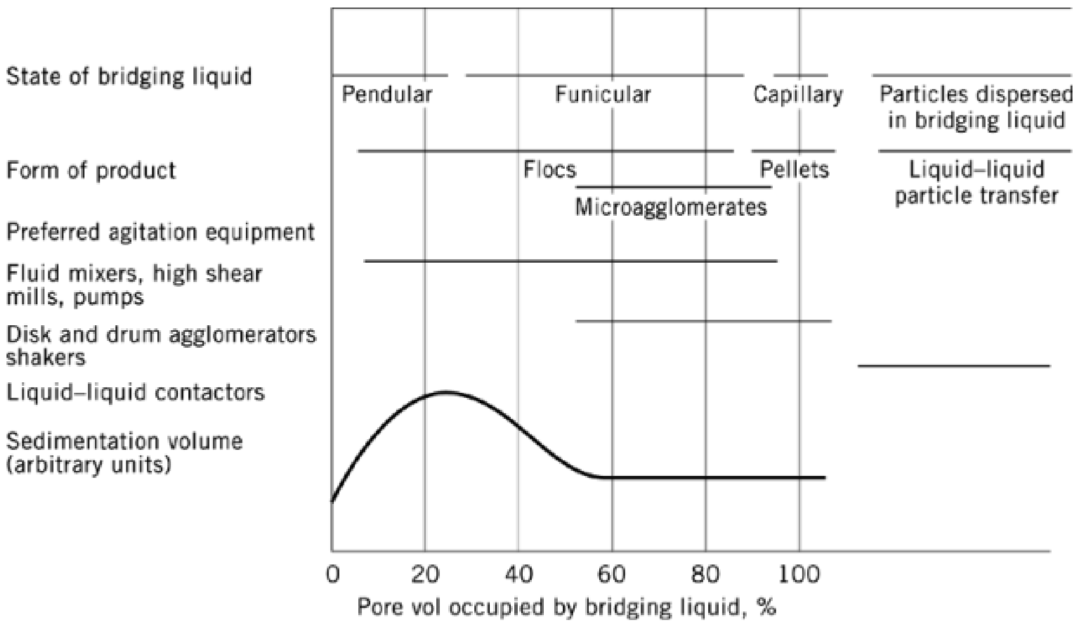

Fig. 15. Phenomena that take place as increasing amounts of an immiscible wetting liquid are added to a suspension of fine particles (103).

so that selective wetting and agglomeration take place. Combinations of acceptable feed streams are specific to a given industry or geographic location, but some examples are obvious, such as the emulsions from oil production operations scavenged by thermal coal, eg, in Alberta, Canada; oily sludges from coking/steelmaking operations treated by coal or coke; in chemical/petrochemical complexes, reject or off-spec solvents and other oily wastes treated by solid wastes such as rubber crumb or shredded plastics; and purification of oily wastewater by soot pelletization in an oil gasification plant, used commercially by Shell (95).

6.4. Spherical Oxide Fuel Particles

The sol–gel process is a related technique which has been actively developed for the preparation of spherical oxide fuel particles, up to ca 1 mm diameter, for nuclear reactors (90, 91). In agglomeration by immiscible liquid wetting, small amounts of a bridging phase adsorbed on the particles coalesce to draw the particles into larger entities. In the sol–gel process, fine particles are initially suspended in an excess of a bridging phase, the suspension is formed into spherical droplets and the excess bridging phase is removed to solidify the droplets into a particulate product. For example, an aqueous sol of colloidal particles such as thoria can be dispersed into droplets in a stream of immiscible water-extracting fluid, such as 2-ethyl-1-hexanol. As water is removed from the sol droplets, the gel formed solidifies and densifies the spherical agglomerates of the colloidal particles. Drying, calcining, and sintering of the recovered microspheres complete the process.

6.5. Soil Remediation

There are many sources of oil-contaminated soils as a result of petrochemical spills and industrial activity in general. For high levels of oil loading (eg, greater than a few weight percent), solvent extraction methods provide important cleaning process options. In general, these processes work well with coarse soils, but have

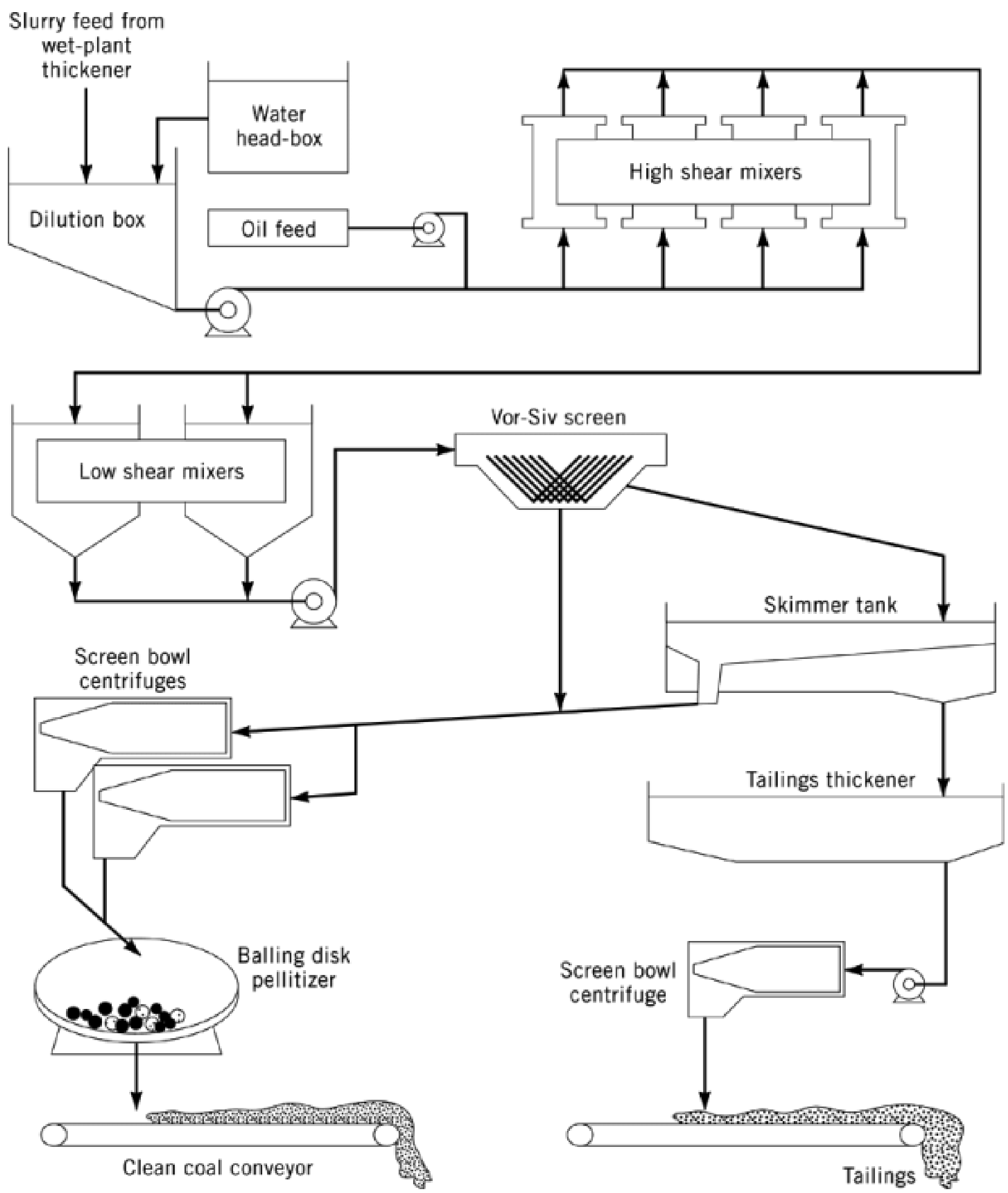

Fig. 16. Flow sheet for oil agglomeration of fine-coal slurries (96).

difficulty in handling fine, clay-containing soils because of difficulties in separating extreme fines from the extraction solvent.

Liquid-phase agglomeration can overcome the fines separation problem by forming aggregates of controlled size tailored to the chosen downstream separation method. A size range of 0.5–2 mm has been recommended (102) for soil remediation. This provides optimal drainage and aeration for subsequent bioremediation, which may be needed as a final polishing step. In addition, this size mimics the natural soil size distribution which is important in returning the clean aggregates to the remediated site for fertile agricultural use.

In this process (102), the suspending phase for the soil is selected to be a good sorbent for the oily contaminant. Naphtha is often used. Agglomeration is effected by either water alone if the particles are sufficiently hydrophilic, or by water with added wetting agents to help displace remaining organic contaminants. Process equipment consists of standard conveying, milling, mixing, screening, settling, and filtration modules. In treating soil with $\sim$30% clay in one example, the process removed 95% of a heavy oil contaminant, leaving a residue of 0.3% on the soil, down from about 6% in the feed material. A treatment cost of about $50/t has been estimated for a 240 t/d plant, based on extensive bench and pilot-scale testing of bitumen extraction from oil sands.

6.5.1. Nomenclature

Symbol	Definition	Units
C	parameter in eq. 3	
D	agglomerate diameter	m
d	diameter of monosized spherical particles	m
H	tensile strength of a single particle-particle bond	N
L	compression force at failure	N
s	fractional filling of agglomerate voids	
W	weight fraction of liquid (wet basis)	
γ	liquid surface tension	N/m
ϵ	void fraction of agglomerate	
ρ_L	liquid density	g/cm^3
ρ_S	particle density	g/cm^3
σ_C	compressive strength	Pa
σ_T	mean tensile strength per unit section area	Pa

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Related Articles

Coating processes, powder technology; Waste treatment, hazardous waste; Size reduction