

Agglomeration Technology: Mechanisms

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Powder agglomeration is used in a wide variety of the chemical process industries (CPI), and a virtually endless number of process options are available. Selection requires engineers to make a substantial number of design decisions, such as the choice between wet or dry processing, the intensity of mixing and shear rates, continuous versus batch operation, cross contamination of products and ease of cleaning. These choices must be made in light of desirable agglomerate end-use properties. Key agglomeration mechanisms and their impact on agglomeration processing are reviewed in this article. The impact on process selection is also touched upon within the context of mechanisms.

Agglomeration processes

Agglomeration processes can be loosely broken down into agitation and compression methods. Agglomeration by agitation will be referred to as wet granulation. Processes include fluid-bed, disc, drum, and mixer granulators, as well as many hybrid designs. Here, a particulate feed is introduced to a process vessel and is agglomerated, either batch-wise or continuously, to form a granulated product. The feed typically consists of a mixture of solid ingredients, referred to as a formulation, which includes a key active product ingredient (API), binders, diluents, flow aids, surfactants, wetting agents, lubricants, fillers and end-use aids (such as sintering aids, colors and taste modifiers). Agglomeration can be induced by a solvent or slurry atomized onto the bed of particles, or by the controlled sintering or partial melting of a binder component of the feed. Product

forms generally range from spherical agglomerated or layered granules, to coated carrier cores.

In the second approach of agglomeration by compression, or compaction, a powder blend is fed to a compression device that promotes agglomeration due to large, applied compaction pressures. Continuous sheets of solid material are produced, as in roll pressing, or some solid form is made, such as a briquette or tablet. Continuous sheets or strands may either break down in subsequent handling to form a granulated material, or the material may be further processed through a variety of chopping, spheronizing or forced screening methods. Carrier fluids may be added or induced by melting, in which case the product is wet extruded. Compaction processes range from confined compression devices, such as tableting, briquetting machines and ram extrusion to unconfined devices, such as roll presses and a variety of pellet mills.

In a CPI plant, an agglomeration process involves several peripheral unit operations, such as milling, blending, drying or cooling and classification, referred to generically as an agglomeration circuit. In addition, more than one agglomeration step may be present as in the case of pharmaceutical or detergent processes. In troubleshooting process upsets or product quality deviations, it is important to consider the high degree of interaction between all unit operations involved in solids processing facilities.

Agglomeration is typically used to

create free-flowing, non-segregating, uniform blends of key ingredients, with agglomerates of controlled strength that can be reproducibly metered in subsequent operations. The desired attributes of the agglomerate clearly depend on the application at hand. Still, it is important to appreciate the generic impact of agglomerate size distribution and porosity, both of which impact final product appearance. Agglomeration is used to achieve numerous benefits*. For example, a proper size distribution of granules improves solids flow, deaeration and compaction behavior, but minimizes segregation. Granule porosity controls strength, attrition resistance and dissolution rate, impacts capsule and tablet behavior, and controls surface-to-volume ratios of catalyst applications. The generic agglomeration mechanisms of granulation and compaction are addressed in the following sections.

Wet granulation

Granulation processes produce granules of low to medium, and in some cases, high density. Ranked from lowest to highest levels of shear, these processes include fluid-bed, tumbling, and mixer granulators. Four key rate mechanisms contribute to all granulation methods, as outlined by Ennis [1]. The reader is referred elsewhere for a more complete treatment [1-7]. The key rate mechanisms include wetting and nucleation, coalescence or growth,

* For more information, see box titled "Objectives of size enlargement" in the online version of this article at www.che.com

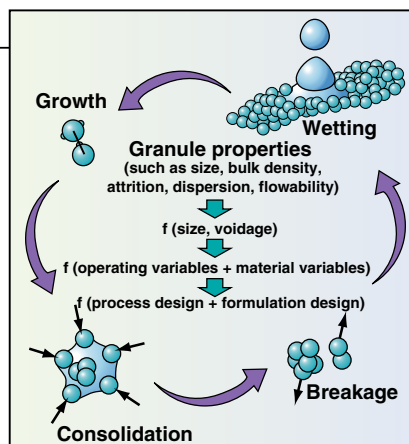


FIGURE 1. The rate mechanisms of agitation agglomeration, or granulation include powder wetting, granule growth, granule consolidation and granule attrition [1]

This review of agglomeration technology focuses on the mechanisms of agitation (wet granulation) and compression (compaction) methods

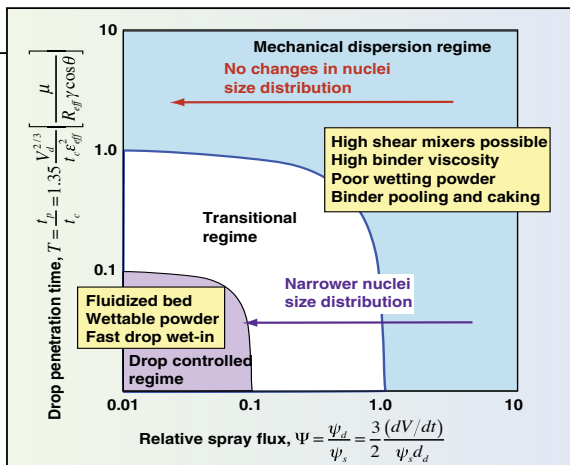


FIGURE 2. This regime map for wetting and nucleation relates spray flux, solids mixing (solids flux and circulation time) and formulation properties [3,6]

consolidation, and attrition or breakage (Figure 1). Wetting of the initial feed promotes nucleation of fine powders, or a coating for particle sizes in excess of drop size. In the coalescence or growth stage, partially wetted primary particles and previously formed nuclei coalesce to form granules composed of several particles. As granules grow, they are compacted by the forces arising from bed agitation. This consolidation stage strongly influences internal granule voidage (or porosity), and therefore end-use properties, such as granule strength, hardness and dissolution. Formed granules may be particularly susceptible to attrition if they are inherently weak or if flaws develop during drying.

Wetting. The mechanism of nucleation and wetting may be determined from a wetting regime map (Figure 2), and is controlled by two key parameters [3–6]. One is the time required for a drop to wet-in to the moving powder bed (t_p) in comparison to process circulation time (t_c). This defines a dimensionless, drop penetration time, or $T = t_p/t_c$. The second parameter is the actual spray flux of drops (ψ_d) in comparison to solids flux moving through the spray zones (ψ_s). This defines a dimensionless, relative spray flux of $\Psi = \psi_d/\psi_s$, which is a measure of the density of drops falling on the powder surface. If wet-in is rapid and spray fluxes are low ($T < 0.1$, $\Psi < 0.1$), individual drops will form discrete nuclei in a droplet-controlled regime. At the other extreme, if drop penetration is slow and spray flux is large ($T > 0.5$, $\Psi > 0.5$), drop overlap, coalescence and pooling of binder material will occur. Shear forces due to solids mixing then control the breakdown of wet mass clumps in a mechanical-dispersion re-

gime, independent of drop distribution. Drop overlap and coalescence occur to a varying extent in a transitional regime, with an increasingly wider nucleation distribution being formed for increasing spray flux and decreasing wet-in time. Spray flux is strongly influenced by process and nozzle design, whereas penetration times are a strong function of the binder-powder formulation.

In Example 1 (see box, p. 36), the spray flux is close to the limit necessary to remain in a droplet-controlled regime of wetting, which forms discrete nuclei. To lower the spray flux by a factor of two, as a safety for droplet-controlled nucleation, either two nozzles spread well apart, twice the solids velocity, or half the spray rate would be needed (or, doubling the spray cycle time). Alternatively, if five times the spray rate were required, wetting would occur in the mechanical dispersion regime, diminishing the need for spray nozzles.

For a 100-fold increase in viscosity, representative of a typical binding solution and twice the drop size, the penetration time would increase to 0.4 seconds. This time is short when compared to the circulation times of high shear systems, suggesting a move toward mechanical dispersion. Drop penetration time decreases with the powder material variables of increasing pore radius, decreasing binder viscosity and increasing adhesion tension, and the operating process variables of decreasing drop size and increasing process circulation time (t_c). Circulation time is a function of mixing and bed weight, and can change significantly with scaleup.

Granule growth. There are strong interactions between the granule growth and consolidation (Figure 3). For fine-powder feed, granule size often pro-

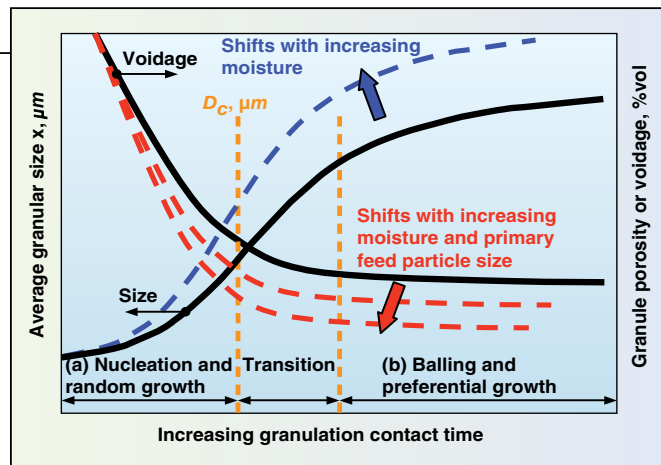


FIGURE 3. Typical regimes of granule growth and consolidation are shown [7]. Increases in growth and consolidation rate are indicated for increasing moisture and feed particle size

gresses through rapid, exponential growth in an initial nucleation stage, followed by a transition stage, finishing with very slow growth in a final balling stage [7]. In the nucleation stage, growth rate is random or independent of granule size, whereas in the balling stage, growth rate is preferentially dependent on size. While growth is occurring, granule internal porosity decreases with time as the granules are compacted. This connection between growth and densification is a dominant theme in wet granulation. Additional modes of granule size change include layering of raw material onto previously formed nuclei or granules, breakdown of wet clumps or wall buildup into stable nuclei, and rupture and attrition of wet or dry granules, respectively.

The degree of granule deformation taking place during granule collisions defines growth mechanisms (Figure 4). If little deformation takes place, the system is referred to as a low-deformability, low-shear process. This generally includes fluid-bed, drum and disc granulators. Growth is largely controlled by the extent of any surface-fluid layer and surface deformability, which acts to dissipate collisional kinetic energy and allow permanent coalescence. Growth generally occurs at a faster time scale than overall granule deformation and consolidation. This is depicted in Figure 4, where smaller granules can still be distinguished as part of a larger granule structure. As granules are compacted, they become smoother over time due to the longer time-scale process of consolidation. This separation in time scale and interaction makes low-deformability, low-shear systems (such as fluid-beds and

Feature Report

drums) easiest to scale-up and control for systems without high recycle.

For high shear rates, large granule deformation occurs during collisions, and granule growth and consolidation are intimately linked and occur on the same time scale. Such a system is referred to as a deformable-high-shear process, and includes continuous pin and plow shear mixers, as well as batch high-shear pharmaceutical mixers. In these cases, kinetic energy is dissipated through deformation of the wet mass composing the granule. Rather than the sticking-together mechanism of low deformability processes such as a fluid-bed, granules are smashed or kneaded together, and smaller granules are not distinguishable within the granule structure. High-shear, high-deformable processes generally produce denser granules than their low deformability counterpart. In addition, the combined and competing effects of granule coalescence and consolidation make high shear processes (such as mixers) difficult to scale-up with wet-mass rheology controlling granule properties, though this is still poorly understood.

Two key dimensionless groups control growth. As originally defined by Ennis [1, 8] and Tardos [9], these are the viscous and deformation Stokes numbers given respectively by:

$$St_v = \frac{4\rho u_0 d}{9\mu} \quad (7)$$

$$St_{def} = \frac{\rho u_0^2}{\sigma_y} (\text{impact}) \text{ or } \frac{\rho (du_0/dx)^2 d^2}{\sigma_y} (\text{shear}) \quad (8)$$

Both numbers represent a normalized granule kinetic energy. The viscous Stokes number is the ratio of kinetic energy to viscous work due to binding fluid occurring during granule-particle collisions. Low St_v or low granule energy represents increased likelihood of granule growth, and this occurs for small granule or particle size (d), low relative collision velocity (u_0) or granule density (ρ), and high binder phase viscosity (μ). Note d is the harmonic average of granule diameter. The deformation Stokes number (St_{def}) represents the amount of granule deformation taking place during collisions, and is a ratio of kinetic energy to wet-mass yield stress (σ_y).

EXAMPLE 1. WETTING REGIME

To illustrate wetting regime determination, consider a powder bed of width (B) equal to 0.10 m, moving past a flat spray with volumetric spray rate (dV/dt) equal to 100 mL/min at a solids velocity (w) equal to 2.5 m/s. For a given spray rate, the number of drops is determined by a drop volume or diameter (d_d) of 100 μm , which in turn defines the drop area (a_d) per unit time that will be covered by the spray, giving a spray flux (ψ_d) of:

$$\psi_d = \frac{da_d}{dt} = \frac{dV/dt}{V_d} \frac{\pi d_d^2}{4} = \frac{3}{2} \frac{(dV/dt)}{d_d} = \frac{3}{2} \frac{(100 \times 10^{-6} / 60 \text{ m}^3/\text{s})}{(100 \times 10^{-6} \text{ m})} = 0.025 \text{ m}^2/\text{s} \quad (1)$$

Where V_d is the drop volume.

As droplets contact the powder bed at a certain rate, the powder moves through the spray zone at its own velocity, or at the solids flux (ψ_s). The solids, dimensionless flux and the dimensionless, relative spray flux (Ψ) are then given for this simple example by:

$$\psi_s = \frac{dA}{dt} = Bw = 0.10 \text{ m} \times 2.5 \text{ m/s} = 0.25 \text{ m}^2/\text{s} \quad (2)$$

$$\Psi = \frac{\psi_d}{\psi_s} = \frac{0.025 \text{ m}^2/\text{s}}{0.25 \text{ m}^2/\text{s}} = 0.10 \quad (3)$$

Where A is the spray area, B is the spray width, w is the solids surface velocity, and ψ_d and ψ_s are the drop and solids fluxes respectively.

For a lactose powder of surface-to-volume average diameter of $d_{32} = 20 \mu\text{m}$, and loose packing and tapped packing voidage of $\varepsilon = 0.60$ and $\varepsilon_{tap} = 0.40$, the effective voidage and pore radius are given by:

$$\varepsilon_{eff} = \varepsilon_{tap} (1 - \varepsilon + \varepsilon_{tap}) = 0.4 (1 - 0.6 + 0.4) = 0.32 \quad (4)$$

$$R_{eff} = \frac{\varphi d_{32}}{3} \left(\frac{\varepsilon_{eff}}{1 - \varepsilon_{eff}} \right) = \frac{0.9 \times 20}{3} \left(\frac{0.32}{1 - 0.32} \right) = 2.8 \mu\text{m} \quad (5)$$

Where ε is the loose effective packing voidage; ε_{tap} is the tapped packing voidage; ε_{eff} is the effective voidage; d_{32} is the average diameter; φ is the particle sphericity; and R_{eff} is the effective pore radius.

For droplet-controlled growth, a short drop wet-in or penetration time is required, and should be no more than 10% of the circulation time (t_c). For water with a viscosity of 1 cP (0.001 Pa-s), and an adhesion tension of .033 N/m, we obtain a penetration time of:

$$t_p = 1.35 \frac{V_d^{2/3}}{\varepsilon_{eff}^2} \left[\frac{\mu}{R_{eff} \gamma \cos \theta} \right] = 1.35 \frac{(\pi (100 \times 10^{-6})^3 / 6)^{2/3}}{0.32^2} \left[\frac{0.001}{2.8 \times 10^{-6} \times 0.033} \right] = .0009 \text{ s} \quad (6)$$

Where μ is the binder viscosity; γ is the binder surface tension; θ is the contact angle; $\gamma \cos \theta$ is the adhesion tension; and t_p is the drop wet-in penetration time. □

Note that granule collisional velocities require judicious estimation.

Examples of growth behavior are illustrated with the help of Figure 5. In the limit of low deformability ($St_{def} = 0$), growth is controlled by St_v and bed moisture. This includes fluidized beds, discs and drums. For low St_v , less than some critical value, initial growth rate becomes a function of moisture and mixing only as illustrated for the (a) nucleation-random growth stage (Points 1 to 3, black curve). To a first approximation, the initial growth behavior is actually independent of St_v , granule inertia and binder viscosity. This growth curve may vary from linear at small spray rates, as in fluid beds, to exponential growth in drums or at high moisture levels. Increases in spray rate (or more generally spray flux), bed moisture, and mixing (such as drum rotation rate, or fluid-bed excess-gas velocity when ignoring attrition) in-

crease initial growth rate. The width of the granule size distribution typically increases in proportion to average size (Figure 5, inset a). An exception would be disc granulation, which possesses self-granule size classification.

Later in the (b) balling/preferential growth stage (Figure 5, Points 4 and 5, black curve), granules will reach some limiting size. Here, the granule size distribution will narrow and continue to grow only by layering of powder and smaller granules onto granules in excess of the limiting size (Figure 5, inset b). This final limit increases with decreasing St_v (shown in dashed blue), or increases with increasing binder viscosity, and decreasing granule inertia (see Example 2 on p. 38). Granule attrition will also contribute to the final growth curve.

For deformable, high shear processes, namely high shear mixers, initial growth rate increases with

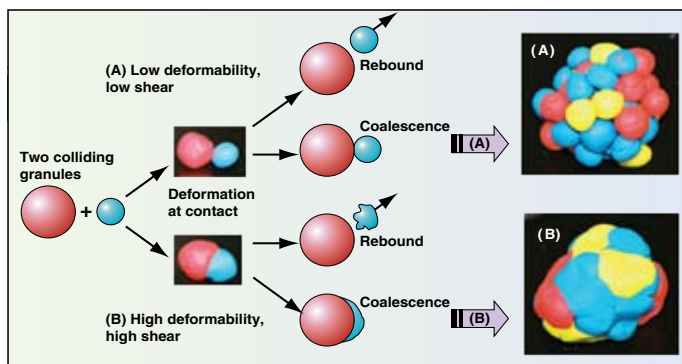


FIGURE 4. Granule structures resulting from (A) low and (B) high deformability systems are typical for fluid-bed and high-shear mixer granulators, respectively [4]

increasing deformation Stokes number (St_{def}), as illustrated in Figure 5 (shown in red), representing an increased kneading together of granules in the process. This occurs for increasing impeller speed, granule density, and decreasing formulation yield stress (σ_y). Yield stress generally decreases with increasing bed moisture (or saturation), increasing particle size or pore radius, decreasing binder viscosity, and decreasing surface tension. As granules densify in a high shear process, their yield stress rises and they become less deformable — which works to lower coalescence in the later stages of growth — and a limit of growth will again be achieved as with St_v . Often this limiting size will vary inversely with the initially observed growth rate.

Lastly, it should be noted that the process or formulation itself cannot uniquely define whether it falls into a low or high deformability category. A very stiff formulation with low deformability may behave as a high deformability system in a high shear mixer, or a very pliable formulation may act as a low deformability system in a fluid-bed granulator.

Consolidation. Granule consolidation or densification is also controlled by Stokes numbers and peak bed moisture. Consolidation typically increases for all processes with increasing residence time, shear levels, bed height, bed moisture or granule saturation, particle feed size or pore radius, surface tension, and decreasing binding-fluid viscosity. The roles of moisture, feed particle size and processing time are illustrated in Figure 3. Simultaneous drying or reaction usually acts to arrest granule densification.

Attrition. Breakage and attrition play critical roles in defining a final agglomerated product quality as well as final strength attributes for sub-

sequent processing and handling [1–4, 10]. Attrition is controlled by a combination of granule voidage and inherent bond strength, which may be assessed by measurements of fracture toughness and hardness on prepared bar composites of the formulation. In addition, direct indentation measurements of hardness of granules and particles are possible in some cases. Granule or agglomerate voidage is controlled by the mechanism of consolidation, with denser granules giving less attrition and requiring greater work for re-dispersion in solution.

Granulation processes

The granulation mechanisms described above can occur simultaneously in all wet granulation processes, and they determine the final granule size distribution, voidage and the final product quality. However, certain mechanisms may dominate in a particular process. It is vital to keep in mind the high degree of interaction between formulation properties and process equipment in making a selection of process equipment.

With small shear rates and simultaneous drying, batch fluid-bed granulators can produce some of the lowest density granules, and are an example of low deformable growth. Growth rate is controlled primarily by the wetting process, spray rate and current bed moisture. Low spray fluxes and fast drop penetration are required to prevent binder pooling and defluidization. Poorly wetting powders or binders of initially high viscosity are precluded. Consolidation of granules can be increased independent of growth through increasing bed height, bed moisture or process residence time. The inherent stability of

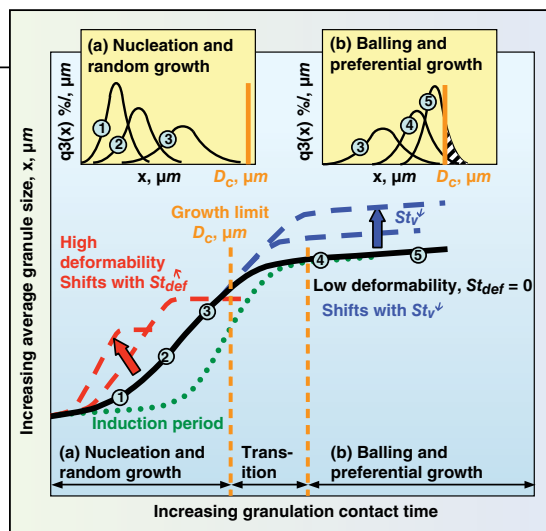


FIGURE 5. Typical granule-growth profiles for low and high deformability mechanisms are shown. The insets illustrate the corresponding evolution of size distribution, above and below the growth limit D_c [4]

low deformability processes allows a wide manipulation in granule properties, as well as ease of scaleup.

At the other extreme are high shear mixer granulators, where mechanical blades and choppers induce binder distribution and growth, producing medium to dense, sometimes irregular granules. Mixers generally operate as a deformable growth process, where in most cases it is difficult to control granule density independent of size. Mixers have an advantage in that they can process plastic, sticky or poorly wetting materials, and can spread viscous binders while operating in a mechanical dispersion regime of nucleation. However, associated with this flexibility in processing a wide variety of materials, high shear mixers can be very difficult to scale-up due to large shifts in the competition between growth and densification, wetting regimes, and powder mixing with vessel scale.

Tumbling granulators produce spherical granules of low to medium density, and lie between fluid-bed and mixer granulators in terms of shear rate and granule density. They have the highest throughput of all granulation processes. In the case of drums, processes often operate with high recycle ratios, whereas preferential segregation in disc granulators can produce very tight size distributions of uniform spherical granules.

Compaction and extrusion

Compressive techniques of agglomeration range from completely confined compaction processes, as in case of tableting, to unconfined as in the case

EXAMPLE 2. DRUM GRANULATION

For the drum granulation of ground iron ore feed ($d = 5 \mu\text{m}$, $\rho = 3 \text{ g/cm}^3$) and water ($\mu = 0.01 \text{ P}$) at a rotation rate (N) of 10 rpm, the initial starting collisional velocity (u_o) by relative shear and the viscous Stokes number are given by:

$$u_o = \omega d = \frac{2\pi N}{60} d$$

$$= \frac{2\pi 10}{60} (5 \times 10^{-4}) = 5.2 \times 10^{-4} \text{ cm/s} \quad (9)$$

$$St_v = \frac{4\rho u_o d}{9\mu}$$

$$= \frac{4 \times 3 \times 5.2 \times 10^{-4} \times 5 \times 10^{-4}}{9(0.01)} \approx 3.5 \times 10^{-5} \quad (10)$$

Where ω or N is the rotation rate; d is the average granule or particle diameter; ρ is the granule or particle density; and μ is the binder viscosity.

For such a low value of $St_v \ll 1$, all the iron ore particles will adhere to one another provided local binding water is present. As granulation continues, diameter will increase along the growth curve given in Figure 7, with the rate controlled by moisture level and rotation rate (or solids residence time). This occurs until a transition limit of size is reached where St_v approaches one. In this example, the limit is about 1 mm, after which growth of these granules continues by balling. In the presence of binder such as bentonite clay, larger diameters are possible, as this will raise the effective viscosity of the binding solution. Alternately, lowering drum speed will give a larger limit, but will decrease the initial rate of growth. □

EXAMPLE 3. STRESS TRANSMISSION

By way of example for an aspect ratio z/D equal to one for a cylindrical compact, with an effective powder friction δ equal to 40 deg. and wall friction ϕ' at 15 deg., let us determine the percentage of stress transmitted from an applied load using Janssen's relation:

$$K = (1 - \sin \delta) / (1 + \sin \delta)$$

$$= (1 - \sin 40^\circ) / (1 + \sin 40^\circ) = 0.643 \quad (12)$$

$$\mu_w = \tan \phi' = \tan (15^\circ) = 0.268 \quad (13)$$

$$\frac{\sigma_z}{\sigma_o} = e^{-4\mu_w K(z/D)}$$

$$\sigma_o = \exp(-4 \times 0.268 \times 0.643 \times 1) = 0.79 \quad (14)$$

Where K is the lateral Janssen constant; μ_w is the wall friction coefficient; D is the die diameter; z is the axial distance from the applied load; and σ_z/σ_o is the ratio of axial to applied stress.

We obtain a ratio of top-applied punch stress to bottom punch stress of 79% when pressing from one side. Twenty percent of this stress is lost to die wall friction, which could result in large density variations (Figure 7) and delamination during unloading. In this case, a decrease in wall friction (ϕ') to 3 deg due to lubricants gives an approved stress ratio of 96%. □

of roll pressing [2–4]. Due to the importance of powder friction and compression, we also include here wet extrusion techniques, such as screw extrusion and pellet-type mills. The success of these unit operations is determined by the ability of powders to freely flow, uniformly transmit stress, readily deaerate, easily compact forming permanent interparticle bonding, and maintain bonding and strength during stress unloading (Figure 6). These mechanisms are controlled in turn by the geometry of the confined space, the nature of the applied loads and the physical properties of the particulate material and of the confining walls.

Powder filling and compact weight variability are strongly impacted by bulk density control and powder flowability, as well as any segregation tendencies of the feed [11–12]. In the case of unassisted flow of free-flowing coarse material through an orifice of diameter B , the mass discharge rate is given by the Beverloo relation:

$$W_o = 0.58\rho_b \sqrt{g} (B - kd_p)^{2.5} \quad (11)$$

Here, ρ_b is bulk density, k equals 1.5 for spherical particles, and d_p is particle size. This is a maximum achievable filling rate, and it is a strong function of opening size, B . Carefully controlling gap distances for free flowing materials is critical. In practice,

mass feedrate can decrease substantially with increasing powder and wall friction, increasing powder cohesion, decreasing bulk-powder permeability, and decreasing opening size (small compacts). Reproducible powder feeding is crucial to the smooth operation of compaction techniques. High powder cohesion and low permeability can lead to wide feed fluctuations and in the worst case, can entirely arrest the flow. In fact, permeability plays a large role in determining maximum production rate in compaction processes.

Following the filling of a compression zone, stresses are applied to the powder with the aim of forming interparticle bonds. However, the frictional properties of powders prevent a uniform stress transmission. For a given applied load, wide distributions in local pressure and the resulting density can exist throughout the compact [13] as illustrated in Figure 7. The axial (σ_z) and radial (σ_r) stresses decrease exponentially with axial distance, z , from the applied load, σ_o .

The ratio σ_z/σ_o may be taken as a measure of stress uniformity, which in practice increases toward unity for decreasing aspect ratio of the compact, decreasing diameter, increasing powder friction, and most important, decreasing wall friction, as controlled by the addition of lubricants (Figure 7 and Example 3). Low stress trans-

mission results in poor compact uniformity, unnecessarily large compression loads to compact weak zones, and large residual radial stresses after stress unloading, giving rise to flaws and delamination as well as large die ejection forces.

For a local zone of applied stress, particles deform at their point contacts, including plastic deformation for forces in excess of the particle surface hardness. This allows intimate contact at surface point contacts, allowing cohesion and adhesion to develop between particles, and therefore interfacial bonding, which is a function of their interfacial surface energy. Both particle size and bond strength control final compact strength for a given compact density or voidage. While brittle fragmentation may also help increase compact density and points of interparticle bonding as well, in the end some degree of plastic deformation and interlocking is required to achieve some minimum compact strength. Successful compaction requires that a minimum critical yield pressure be exceeded to obtain significant strength. This yield pressure increases linearly with particle hardness. Strength also increases linearly with compaction pressure, with a slope inversely related to particle size.

During the short time scale of the applied load, any entrapped air must es-

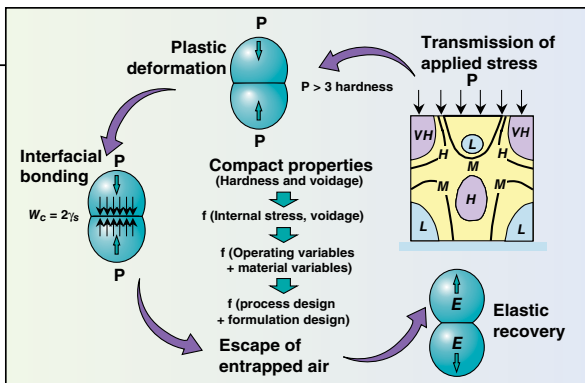


FIGURE 6. The micro-level mechanisms of compaction are shown here [4] (letters in inset refer to levels of pressure; low, high, medium and very high)

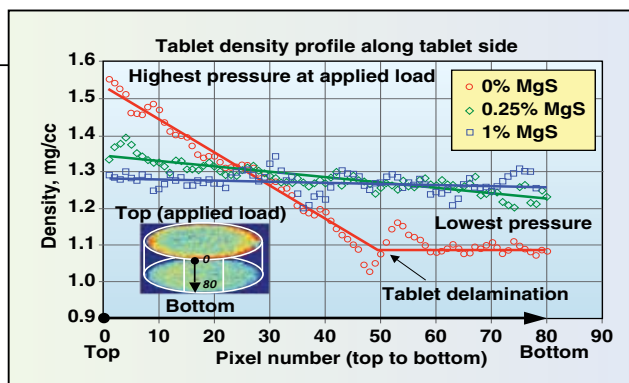


FIGURE 7. The density along a lactose tablet edge (pixel number) is shown as a function of level of magnesium stearate lubricant. The inset photo is a near infrared (NIR) chemical image of a lactose tablet density [13]

cape and a portion of the elastic strain energy must be converted into permanent plastic deformation. The developed air pressure will vary inversely with permeability, and increase with compact size and production rate. Low powder permeability and entrapped gas may act to later destroy permanent bonding, and generally lower allowable production rates.

Upon stress removal, the compact expands due to elastic recovery of the matrix, which is a function of elastic modulus and expansion of any remaining entrapped air. This can result in loss of particle bonding and flaw development, which is exacerbated for variation in compaction stress due to poor stress transmission. The final step of stress removal involves compact ejection, where any remaining radial elastic stresses are removed. If recovery is substantial, it can lead to capping or delamination of the compact. Therefore, most materials have an allowable compaction pressure range, with a minimum pressure set by hardness, and a maximum by elastic and permeability effects.

In the case of extrusion, both wet and dry techniques are strongly influenced by the frictional properties of the particulate phase and wall. In wet extrusion, wet mass rheology and friction control the pressure needed to in-

duce die extrusion, with this pressure increasing with desired throughput. On the other hand, the actual pressure that can be developed by the sliding action of the barrel from the reference frame of the screw flight decreases with increasing throughput and screw friction, and increases with decreasing barrel friction. Lastly, the rheological properties of the liquid phase are equally important. Poor rheology can lead to separation of the fluid and solid phases, large rises in pressure, and undesirable sharkskin-like surface appearance on the granulate, which is prone to high attrition [3, 4].

These mechanisms of compaction control the final flaw and density distribution throughout the compact, whether it is a roll-pressed, extruded or tableted product; and as such, control compact strength, hardness, strength characteristics and dissolution behavior. Process performance and developed compaction pressures in extrusion and dry compaction equipment are very sensitive to powder flow and mechanical properties of the feed. These processes generally produce much denser compacts or agglomerates than wet granulation.

Process equipment selection

The choice of agglomeration equipment is subject to a variety of constraints.

Ideally, the choice of equipment should be made on the basis of the desired final product attributes. Agglomerate porosity is a very important consideration in that it impacts strength and attrition resistance, hardness, internal surface area, reactivity and dissolution rate. The desired agglomerate appearance and size distribution, as well as the ability to utilize moisture or solvents are additional considerations. Wet granulation produces low- to medium-density granules of varying sphericity. Binders are typically utilized, and drying of solvents is required, with the associated energy and dust-air handling costs. If denser agglomerates are required, dry compaction or wet extrusion should be considered, although it is worth noting that reasonably dense granules are possible with two-stage mixer processing. Dry compaction is suitable for moisture sensitive materials. Appearance considerations might suggest tableting, or wet granulation or extrusion combined with spheronizing for free-flowing, nearly spherical granules. ■

Edited by Dorothy Lozowski

Author



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OBJECTIVES OF SIZE ENLARGEMENT

- Production of useful structural forms, as in pressing of intricate shapes in powder metallurgy
- Provision of a defined quantity to facilitate dispensing and metering, as in agricultural chemical granules or pharmaceutical tablets
- Elimination of dust handling hazards or losses, as in briquetting of waste fines
- Improved product appearance, or product renewal
- Reduced caking and lump formation, as in granulation of fertilizer
- Improved flow properties, generally defined as enhanced flow rates with improved flow rate uniformity, as in granulation of pharmaceuticals for tableting or ceramics for pressing
- Increased bulk density for storage and tableting feeds
- Creation of non-segregating blends of powder ingredients with ideally uniform distribution of key ingredients, as in sintering of fines for steel or agricultural chemical or pharmaceutical granules
- Control of solubility, as in instant food products
- Control of porosity and surface-to-volume ratio, as with catalyst supports
- Improvement of heat transfer characteristics, as in ores or glass for furnace feed
- Remove of particles from liquid, as with polymer additives which induce clay flocculation

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