Choosing Agglomeration Processes

Greg Mehos, University of Rhode Island
Chris Kozicki, Feeco International

Agglomeration is the process of converting fine powder particles into larger ones through the introduction of external forces. Agglomeration offers numerous benefits including significant dust reduction, improved handling, more complete utilization of raw materials, and densification. Agglomeration can be accomplished by a variety of means including mixing with a liquid, applying pressure, and heating.

Technologies available for size enlargement fall into three general categories: (1) tumble-growth agglomeration, (2) pressure agglomeration, and (3) agglomeration by heat or sintering. Tumble growth agglomeration includes both wet and dry methods. Stresses developed in pressure agglomeration equipment can range from moderate to extreme. For some materials, agglomeration by heat or sintering can be accomplished at relatively moderate temperatures.

Tumble growth agglomeration equipment tend to have a lower capital cost but may have high capital costs if drying is required. Agglomerates produced by tumble growth technologies tend to have a lower bulk density and a wider particle size distribution. Binders frequently must be added to ensure that "green" (*i.e.*, wet) or dry agglomerates have sufficient strength.

Pressure agglomeration equipment and equipment that agglomerate particles by heat or sintering tend to have higher capital costs but lower operating costs. The agglomerates have a higher bulk density but they may be prone to attrition if the pellets that are produced have sharp edges.

Particles are held together by a number of mechanisms. Solid bridges between particles can be the result of sintering, partial melting, and recrystallization during drying. Binders that readily adhere to solid particles are effective agglomerating aids. Liquid bridges in spaces between individual particles can form strong agglomerates as a result of capillary forces that develop when the voids are completely filled with the liquid. Attractive forces such as van der Waals forces, valence forces, and hydrogen bridges can also hold solid particles together. If adjacent particles interlock, very strong agglomerates form.

Simple tests can be used as screening tools to determine the most suitable agglomeration process for a particular powder. Liquid can be added to a fine powder in a small kitchen blender to determine if the liquid will indeed wet the powder. This simple test can also give the investigator a sense of how much liquid must added to avoid producing a slurry or paste. Hydraulic presses can be used to gauge the likelihood of pressure agglomeration to be a suitable technology. Differential scanning calorimetry (DSC) can be used to estimate the temperature required to agglomerate a fine powder by melting or sintering.

Many vendors of agglomeration equipment have test facilities for conducting feasibility studies with pilot-scale agglomerators. Often the equipment is available for rent, and customers can perform thorough evaluations at their sites.

When conducting a feasibility study, the following properties should be measured:

Crush strength. Crush strength tests determine how much pressure is required to crush an agglomerate completely. Crush tests are performed on single agglomerates, using a metal plate to gradually apply increasing pressure to the agglomerate until it fractures.

Green/wet crush strength. Green/wet crush strength tests are essentially crush strength tests performed on agglomerates made using a wet process. The tests are conducted on green agglomerates prior to drying. To prevent breakage during the drying process, the green pellets must have sufficient crush strength.

Cohesive strength. Unlike crush strength, cohesive strength is a bulk property. Cohesive strength tests are conducted using a shear cell tester and measure the stress required to cause a consolidated sample of agglomerates to fail. Together with wall friction test results, cohesive strength measurements can be used to design hoppers, bins, and silos that will reliably handle both the agglomerated product and the fine powder that is to be fed to the agglomeration equipment.

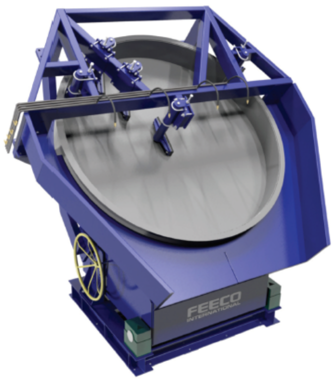
Particle size. The particle size of an agglomerated product is usually measured by sieve analysis. Sieves are made up of circular metal trays each with a screen-like wire mesh in the bottom. Using sieves having a range of sizes allows a material's particle size distribution to be measured.

Tumble-growth agglomeration

Tumble growth agglomeration can be either wet or dry. In a wet agglomeration process, the fine particles are wetted with an appropriate liquid, preferably water. In some cases, a surfactant or other chemical is added to improve the wettability of the solid particles. Binders are frequently introduced to improve the agglomerates' crush strength. In a wet tumble-growth process, powder, liquid, and additives are fed continuously into a chamber where the wetted mass is then sheared or kneaded until the liquid is evenly distributed and the granules have the desired size and strength. Examples of organic binders are waxes, rosin, starch, and alginates. Inorganic binders include alkali silicates, bentonite, and various aqueous solutions and dispersions. The major criteria for selecting a binder are cost, compatibility with the product's final use, and its ability to give agglomerates their desired crush strength.

Popular wet agglomeration equipment include pin mixers, plough mixers, disk pelletizers, fluidized beds, and other technologies. Figures 1 and 2 are photographs of a disk pelletizer and a pin mixer, respectively.

In general, wet agglomeration of fine powders occurs in three stages. The first stage is a mixing stage where powder, liquid, and binder are combined. Next, moist particles are joined together to form green agglomerates. The green particles are formed by first forming nuclei that then grow into larger aggregates by layering or coalescence. In some cases, nucleation and aggregate growth take place in two separate pieces of equipment; for example, agglomerates from a pin mixer can be fed into a disk pelletizer. The final stage is drying or curing, which takes place in a separate device. An example of a wet agglomeration process is shown in Figure 3.



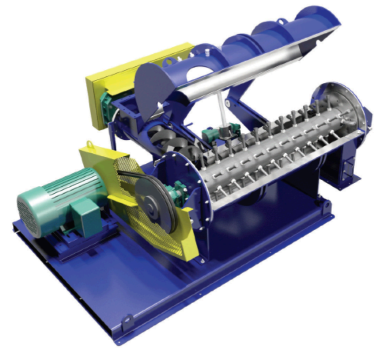


Figure 1. Pan agglomerator. **Figure 2. Pin mixer.**

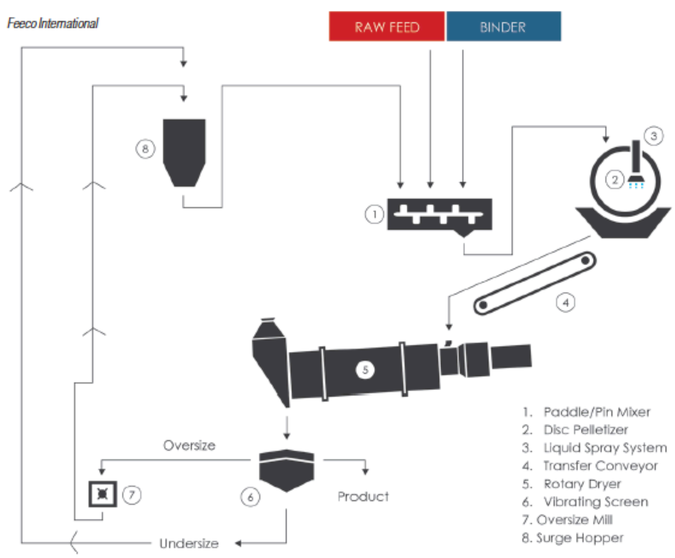


Figure 3. Wet pelletization process.

The optimal amount of liquid added to a powder, *i.e.*, the amount that gives green agglomerates their greatest strength, is typically 40-90 percent of its full saturation. Saturation is the fraction of the total void space that can be filled with a liquid. When the liquid is added to a dry powder, liquid bridges will begin to form at contact points between particles. This is known as the pendular stage of saturation. Moisture is attracted to the interfaces between powder particles by capillary forces. As saturation levels are increased, the funicular stage is approached where all internal solid surfaces become surrounded by liquid. At this point, the mixture becomes more paste-like, the tensional forces disappear, and the agglomerates become significantly weaker. When the powder becomes fully saturated, the capillary state is reached, and at higher moisture levels, the mixture begins to behave as a slurry. Saturation states of powder are illustrated in Figure 4 [1].

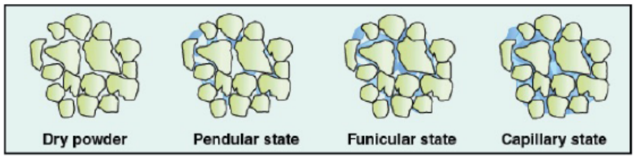


Figure 4. Saturation states.

The optimum saturation state of a powder often depends on the equipment used for size enlargement. In general, the higher the shear imparted to the powder by the equipment, the lower the optimum saturation state and the smaller the size of the agglomerate.

Since producing a green pellet with an optimum saturation state is critical, careful control of the liquid and solids feed rates is required. Providing a steady liquid stream is usually not a problem, but providing a stable powder feed rate or ensuring a constant solids to liquid ratio may be a challenge for some powders. Methods for designing reliable powder feed systems are discussed by Mehos and Morgan [2].

Dry agglomeration can be a misnomer because the technology frequently requires moisture, although significantly less than that required for wet agglomeration processes. The most common equipment used in dry agglomeration is an inclined drum. Often spray nozzles run along the interior of the drum to coat particles with liquid , which promotes adhesion and particle coalescence. A photograph of a drum agglomerator is shown in Figure 5.

Figure 5. Rotary drum agglomerator.

The major advantage of a dry tumble growth agglomerator is reduced drying costs. Disadvantages include the difficulty in predicting the time required to granulate powder, requiring seed particles during start-up, and high product recycle rates that are usually necessary.

Other tumble growth agglomerators include V-blenders and other batch mixers that are equipped with spray nozzles. Fluidized beds can also be used for size enlargement. Mechanical agitation in mixing equipment creates a turbulent environment that allows particles to contact with liquid and each other, coalesce, grow, and form agglomerates. A similar environment can exist when particles are suspended in a fluidized bed. Fluidization occurs when the upward flow of gas provides drag forces equal to the weight of the bed of particles.

In a fluidized bed agglomerator, dry powder is fed into a chamber equipped with a distributor plate through which the fluidizing gas is fed. Spray nozzles above the fluidized bed provide a fine mist of liquid. The gas is heated, which removes the added liquid while agglomeration takes place.

If the dry powder is isolated by spray drying, agglomeration can be accomplished by feeding partially dried particles into an integrated fluidized bed dryer. In a fluidized spray dryer, gas not only enters the top of a tower where it contacts the atomized suspension or solution of solids but also at the bottom of the tower through a distribution plate. Because only partial drying is achieved in the spray dryer section of the tower, particles reaching the bottom are slightly cohesive, and size enlargement will occur during fluidization. In most cases, the final drying also takes place in the fluidized bed.

Pressure agglomeration

Pressure agglomeration falls in two general categories: roll compaction and die compaction. Because attractive forces between particles intensify as the distance between them decreases, applying pressure can lead to exceptionally strong agglomerates.

Roll compactors use a mechanical force to press powder. Fine particles are first fed between two counter-rotating rolls, which draw material into the gap between them. The powder is compressed into a sheet if smooth roles are used or formed into strips if the compactor is equipped with corrugated rollers. The sheet or strips are then fed through a flake breaker. The agglomerates are irregular in shape but often have sufficient flowability. A roll compactor is shown in Figure 6.

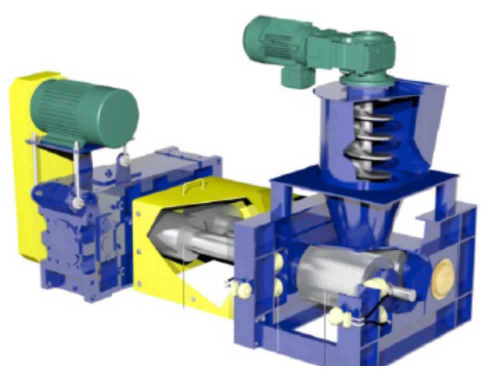


Figure 6. Roller compactor.

The starting material is fed into the gap between the rolls by gravity or with a feeder. The equipment works best when the powder is de-aerated before it reaches the rolls. This is accomplished by equipping the feed hopper with a screw that compresses the material as it is conveyed downward, fabricating the hopper with gas permeable walls and applying vacuum on the clean side, using one or two gas permeable rolls and providing vacuum in the interior, or a combination of these methods.

Die compaction also relies on mechanical forces to press fine powders but also allows agglomerates with a desired shape. Die compaction equipment include briquetters and pellet mills.

Briquetters are roll compactors that have pocketed rolls. Briquetters allow the material to be molded into a desired geometry as it is pressed into recessed cavities on the two rolls. Each roll has one half of the desired form, and as the rolls approach each other, the halves unite, pressing the material into the desired shape.

Pellet mills are devices that force fine powders into dies that are open at both ends. There two common types: flat die mills and ring die mills. Flat die mills are similar to extruders in that a screw is used to convey powder into an orifice plate. A cutter at the opposite side of the screw cuts the exposed pellets.

In a ring die mill, powder is fed into the inside of a cylindrical chamber. Two rollers inside the cylinder then compress the powder through the die holes. Cutters outside the cylinder then cut the pellets as they leave the mill. Figure 7 is a schematic of a ring die mill.

Figure 7. Ring die mill.

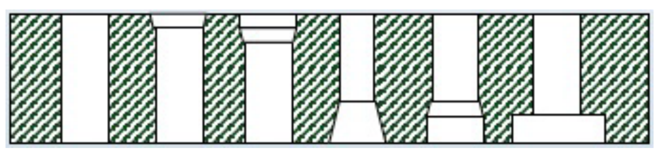


Figure 8. Typical die geometries.

Typical die geometries are shown in Figure 8. Because considerable pressures develop ahead of the die, it must be designed so that it will not fail during operation of the mill. Straight bores are rarely used. Instead, a small chamfer located at the inlet is employed to guide powder into the die. To reduce frictional resistance, the size of the die is often larger at the die inlet, outlet, or both. A tapered die outlet is beneficial because it can prevent sudden expansion and breakage due to spring back.

Temperature/sintering

Extruders are typically used to heat powders to temperatures high enough to cause melting or sintering. Two types of extruders are used: ram extruders and auger or screw extruders.

A ram extruder is used batch-wise. Powder is fed into a barrel equipped with a die plate and then isolated by a hydraulically-driven piston. The powder is then forced towards the die. The barrel may be heated, although the temperature will likely rise without the introduction of heat because of friction. The entire plug of powder does not necessarily have to reach its melting point as only a thin layer of liquid needs to be sheared between the wall and the plug.

The extruder is equipped with cutters immediately downstream of the die plate. Because high pressures are generated, the feed material must have a low moisture content. Otherwise, the liquid will flash upon leaving the die plate and potentially fracture the pellets that are formed.

Auger or screw extruders are widely used as to manufacture pellets continuously. The screw is comprised of four sections: a feed section, a conveying section, a melting or sintering section, and a pumping section. If the feed hopper is conical, the pitch of the screw beneath its outlet should equal the diameter of the hopper outlet. For cohesive materials, the hopper outlet should have a length at least twice its width, and the screw should have a tapered shaft to ensure that the feed hopper is fully activated. By reducing the diameter of the shaft linearly in the direction of flow, the capacity of the screw will be constant, and all the contents of the hopper will be able to enter the extruder.

A short conveying section follows the feed section, followed by the melt or sintering section. Friction or a heated barrel increases the temperature of the conveyed powder. Frequently, the diameter of the barrel is reduced or the shaft diameter is increased to enhance shear. Pressure is generated by pumping the material through a die plate or restricted orifice. If necessary, the extrudate leaving the die plate or orifice can be immersed in a water bath to reduce its temperature to below its melting point. Cutters are used to convert the extrudate into pellets.

Handling agglomerated products

Because agglomerated products are typically non-cohesive, they can be susceptible to segregation by particle size during handling. This is especially true if the product has a wide particle size distribution and the mean particle size exceeds *ca.* 100 μm. Such a material is prone to side to side segregation when a hopper, bin, or silo is filled [3]. Once a pile has formed, larger particles, having better flowability, will flow towards the periphery. Smaller particles will then percolated down the center of the solids bed.

Storage vessels should therefore be designed for mass flow to ensure that all material will flow when the gate is opened or the feeder is started. Otherwise, the particle size may cycle during discharge. A mass flow pattern will develop if the hopper walls are steep enough and low enough in friction to allow flow of solids along the walls.

Final remarks

Agglomeration of powders is desirable for a variety of reasons, including reduced dust levels and improved flowability. It is important to recognize that the product's intended use often dictates what type of agglomeration equipment should be used. For example, many customers must be able to return the agglomerates back to their initial particle size, which would eliminate sintering as a possible agglomeration method. Some applications require a constant particle size distribution, and therefore tumble growth processes will require classification equipment to recycle undersized agglomerates to the front end of a process and oversized agglomerates to a milling process. Simple screening tests that use bench scale presses and mixers will provide insight to the best choices. To select the most appropriate agglomeration equipment, larger scale tests should be conducted at manufacturers' facilities or rental equipment should be evaluated at the user's site. Crush tests, cohesive strength tests, and particle size measurements can then be used to select the most applicable technology. Wolfgan Pietsch [34] is an excellent reference for reviewing agglomeration technologies.

References

1. Mehos, G. and C. Kozicki, "Consider Wet Agglomeration to Improve Powder Flow", *Chem. Eng.,* 121, 11 (January 2011).
2. Mehos, G., and D. Morgan, "Hopper Design Principles", *Chem. Eng.,*126, 1 (January 2016).
3. Mehos, G., "Preventing Segregation in Agglomeration Processes", presented at the 34th biennial Institute for Briquetting and Agglomeration Conference, Scottsdale, Arizona (2015).
4. Pietsch, W., Agglomeration Processes – Phenomena, Technologies, Equipment, Wiley-VCH, Weinheim, Germany, 2002.