Breaking up is hard to do: Particle agglomeration

Sometimes particles stick together strongly enough that they can behave as a single, larger particle. In powder technology practice, this sticking is called agglomeration. Here we’re going to discuss agglomeration in which the resulting larger particle is relatively strong. Weaker particles can be aggregates/flocs or clusters/clouds. The former corresponds to loosely bound colloids or particles in suspension. The latter corresponds to easily broken structures of particles that travel together.

In many cases, agglomeration is desired. Larger material particles produce fewer dust hazards, are generally more flowable, can have higher bulk density, and may be designed in a layered or fast-dissolving structure for a specialty product. Many products — granules, tablets, briquettes, pellets, and others — are designed with these features in mind.

In other cases, agglomeration is undesirable. Powder caking can be a menace; fouling (agglomeration of particles with a vessel wall) is a ubiquitous concern; or a material/catalyst efficacy can be completely destroyed if the particle size changes. For example, in many fluidized particle applications, such as FCC units, gasifiers, cokers, and pyrolyzers, a change in particle size can be detrimental. Large agglomerates will settle to the bottom of the bed, build on internals and component surfaces, or block cyclone exits.

Regardless of whether you want to make agglomerates or avoid agglomeration, understanding the fundamentals of the process is essential. As with many things, most agglomeration knowledge has been developed for cases where it’s desired. In this column, we’ll discuss these cases with the understanding that the same knowledge can be used when agglomeration is not desired.

Agglomerate formation

In both cases, the agglomerate formation mechanism is similar. Particle-size enlargement typically occurs through four methods: mixing, pressure (stress) and compaction, heat, and sintering.

- With mixing agglomeration, particles are either tumbled (low-shear agglomeration) or aggressively mixed with a bladed system (high-shear agglomeration). Here, particle-cohesion or adhesion forces are stronger than external forces, such as inertia, gravity, shear, and drag. These types of agglomeration are mostly limited to particles of 200 microns or smaller. Larger particles in motion tend to stay in motion, which makes agglomeration more unlikely. For the agglomeration of larger particles, a binder or wetting agent (moisture) may be necessary. Particle wetting can range from a monolayer coverage of adsorbate to dripping wet particles. With a monolayer, there can be a covalent or hydrogen bonding action. Hydroxyl groups or -OH groups are known for having strong enough bonding forces to start particle agglomeration. With visually wet particles or multilayer coverage of a particle, viscous and capillary forces predominate. Many processes intent on particle agglomeration tend to use some form of multilayer wetting or capillary force to achieve their desired particle size. A good example of this can be found with granulators, rotating blenders, powder mixers, and fluidized beds

- With pressure (stress) and compaction agglomeration, pressure (typically uniaxial) is applied to a powder resulting in compaction with material bulk densities significantly higher than the powder in a loosely packed bed. Roller presses, tabulators, punch-and-die units, and extruders provide this type of compaction agglomeration. Often, the powder is coated with a binding agent that becomes plasti-
cized during compaction. In other words, the binding agent needs to be softer than the parent powder so it can act as a wetting agent to the parent particle.

- For heat and sintering agglomeration, relatively high temperatures are used to develop a melt layer — going from monolayer to multilayer — on the particle surface. As with the other agglomeration mechanisms, wetting now becomes the driver for particle agglomeration, which becomes rigid upon cooling. This type of agglomeration is often called sintering, and it can occur well below the bulk material melt temperature. As a rule of thumb, monolayer wetting of a heated particle surface can occur at temperatures as low as $\frac{3}{4}$ the bulk material melt temperature. Sintering may also arise if solid bridges are formed at particle contacts, as may occur with sublimation, crystallization, or solute deposition.

**Agglomerate consolidation and growth mechanisms**

In agglomerating systems where surface-bound liquid is required for growth, Iveson and Litster labeled two potential regimes as steady growth and induction growth. As shown in Figure 1, steady growth is the result of a single step process where two particles form one agglomerate. This process is indicative of a high deformation system — possibly involving weak granules or high compaction forces. Surface-bound liquid is constantly squeezed so that nearly every particle collision results in sticking. Steady growth is relatively fast, continuous, and often linear.

*Induction* growth is a multistep process where the particles first need to be consolidated before any growth can occur. Progressive consolidation slowly squeezes liquid to the surface. During this time, very little growth occurs. However, once some liquid is squeezed to the surface, rapid growth occurs. Once a critical amount of wetting is achieved, agglomeration growth can be significant as long as an effective wetting agent or wet particles are available. Beyond that critical amount of wetting, agglomerate breakage becomes substantial. Induction growth is usually the result of a low deformation system such as that in a mixer, rotating drum, or fluidized bed.

An expansion of Figures 1 and 2 can be found in Figure 3, which was...
Stokes Deformation Number, low or no wetting agents results in dry powder. There isn’t enough moisture available for granulation. Also, any granules that may form are too weak to exist for very long and fall apart into dry powder.

Adding more wetting agents results in granules in the crumb regime. These granules are just too weak, or the agitation is too strong for large agglomerates to form. Only a few strong granules exist, but they continuously break and reform. Adding moisture here doesn’t do much because the formed granules are too weak and you would end up with a slurry.

In the center of the regime map, we have steady and induction growth. This center region is the “sweet spot” for promoting agglomeration. Steady growth is the result of stress and/or heat agglomeration with the right level of liquid or plasticized wetting agent to form bridges and attraction via capillary forces. This is the easiest and most effective way to make agglomerates.

For induction growth, high-contact kinetic energy is either not possible or counter-productive due to particle attrition. Thus, a wetting agent such as moisture or a binder needs to be added — or at the very least managed. This mode of growth is incredibly hard to control and may possibly be mistaken for slow, steady growth. Consequently, more moisture is added,
and then the induction growth is catastrophic. This is a good illustration of why understanding your agglomeration growth is essential.

**Improving agglomeration**
If the goal is to improve agglomeration and agglomeration growth, the agglomeration growth map in Figure 3 could serve as a good start. The “sweet spot” would be in the middle of the plot. If you are only getting a dry powder or clustering of powder, adding more wetting agent could provide the improvement needed. Of note from Figure 3 is that dry powder or nucleated agglomerates rarely form stable agglomerates by increasing the Stokes Deformation Number (increasing stress, temperature, and/or agitation). Likewise, a material that’s more slurry than agglomerate will have little effect just by changing conditions that change the Stokes Deformation Number. Most often, the only controllable change is to reduce the amount of wetting agent or moisture.

If induction agglomeration growth is too slow, changing conditions (increasing the stress, temperature, and/or agitation) that increase the Stokes Deformation Number could shift the growth mechanism to steady growth. However, too much stress and/or agitation could result in the breakage of these agglomerates.

**Mitigating agglomeration**
Most often, agglomeration mitigation involves increasing the Stokes Deformation Number (increasing stress, agitation). Attritors, which are nozzles designed to provide a high-velocity jet in a dense bed, are often used in these cases. However, that should not be the only consideration. Reducing the amount of the wetting agent or moisture also can reduce the amount of agglomeration. Temperature also should be considered. Higher temperature can make the particles sticky before they melt (the surface is already melted). Lowering the temperature even slightly could have positive results. However, a lower temperature could also stiffen granules, transitioning from problematic steady growth to catastrophic induction growth. Another option is to increase the amount of wetting agent so that a slurry forms. If a slurry is not a problem, it could be the solution.

**Summary**
Whether more or less agglomeration is needed, understanding the mechanism for agglomeration is the first step. Is the agglomeration growth related to steady growth or inductive growth? From there, the agglomeration growth map in Figure 3 can serve as a guide for the direction you need to go. More energy in the form of stress, temperature, or agitation to increase the Stokes Deformation Number or a higher amount of wetting agent can increase agglomeration rates but only up to a point. Too much energy or wetting agent can result in agglomerate degradation. In contrast, if agglomeration reduction is desired, then add more energy or keep the material dry or too wet. PBE

**References**

**For further reading**
Find more information on this topic in articles listed under “Agglomeration” in Powder and Bulk Engineering’s article index in the December 2018 issue or the Article Archive at PBE’s website, www.powderbulk.com. (All articles listed in the archive are available for free download to registered users.)

Ray Cocoo is president and CEO of Particulate Solid Research Inc. (ray.cocco@psri.org) and holds a PhD in chemical engineering from Auburn University in Auburn, AL. He has more than 25 years of experience in particle technology, holds several patents, and has published numerous technical articles on particle technology topics.

Ben Freireich is the technical director of Particulate Solid Research Inc. and holds a PhD in mechanical engineering from Purdue University in West Lafayette, IN. Ben has more than 10 years of experience in powder technology and is a recipient of the Core R&D Young Research Award and the AIChE “35 under 35” Award.

Particulate Solid Research Inc.
Chicago, IL
773-523-7227
www.psri.org