Improvement in Powder Flowability by Spheronizing

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Agglomeration is often desirable because the product that has a higher bulk density and improved flowability. This is especially the case in the processing of solid dosage forms in pharmaceutical equipment. Figure 1 shows samples of a blend of fine acetaminophen (APAP), microcrystalline cellulose (MCC), and hydroxypropylmethylcellulose (HPMC) powders before and after agglomeration.

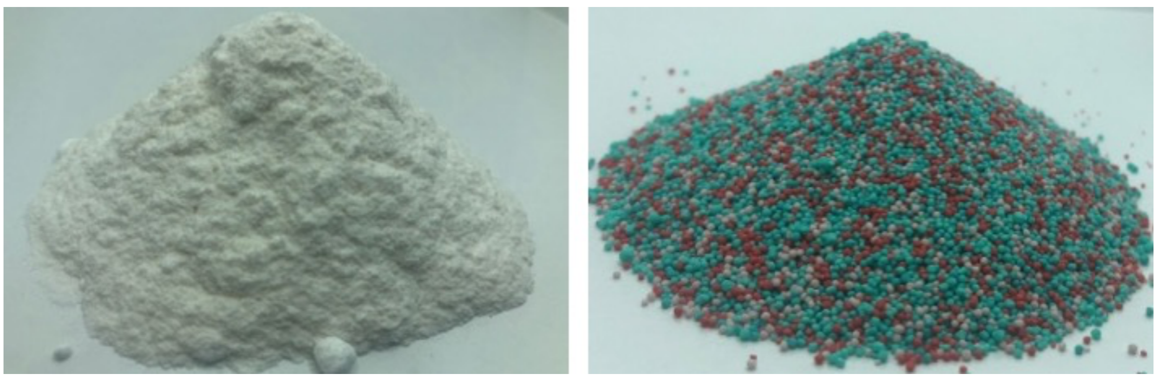

Figure 1. APAP/MCC/HPMC blend – fine (left) and spheronized (right).

Fine powders often exhibit flow problems in a hopper or bin, such as flow stoppages, erratic flow, and sluggish discharge rates. Flow stoppages can occur if a cohesive arch develops at the vessel outlet. In some cases, a stable rathole can develop and the bin will not completely empty.

The likelihood of a solids flow problem often depends on the flow pattern present inside a bin. There are two primary flow patterns that can occur: mass flow and funnel flow (see Figure 2).

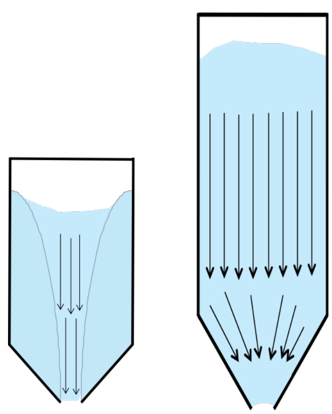

Figure 2. Flow patterns – funnel flow (left) and mass flow (right).

In funnel flow, an active flow channel forms above the outlet, with stagnant material remaining at the periphery (*i.e.*, ratholes). This occurs when the walls of the hopper section of the storage vessel are not steep enough or have low enough friction to allow flow along them. The size of the resultant flow channel is approximately the largest dimension of the outlet, and for a conical funnel flow hopper, the fraction of its volume that is active can be dramatically small. If the bulk material is cohesive, the ratholes may be stable and the effective capacity of the bin will be just a small fraction of its intended capacity.

In mass flow, the entire bed of solids is in motion when material is discharged from the outlet, including material along the walls. Mass flow hoppers typically have steep and/or low-friction walls. Provided that the outlet is large enough to prevent arching, all material will be discharged from the bin, as ratholes will not form. Eliminating flow problems can often be accomplished by ensuring that a mass flow pattern exists in the vessel.

The flow pattern inside a bin or hopper – funnel flow or mass flow – can be predicted by measuring the friction between the powder and the hopper wall material. Wall friction is measured by applying various normal loads to a sample of powder and forcing it to slide along a coupon of wall material. The resulting shear force is measured as a function of the applied normal force, and a wall yield locus is constructed by plotting shear force against normal force. The angle of wall friction at a particular pressure (ϕ') is the angle that is formed when a line is drawn from the origin to a point on the wall yield locus. A wall yield locus determined using a Jenike direct shear cell test on a sample of the fine APAP/MCC/HPMC powder on 304 # 2B finish stainless steel is shown in Figure 3.

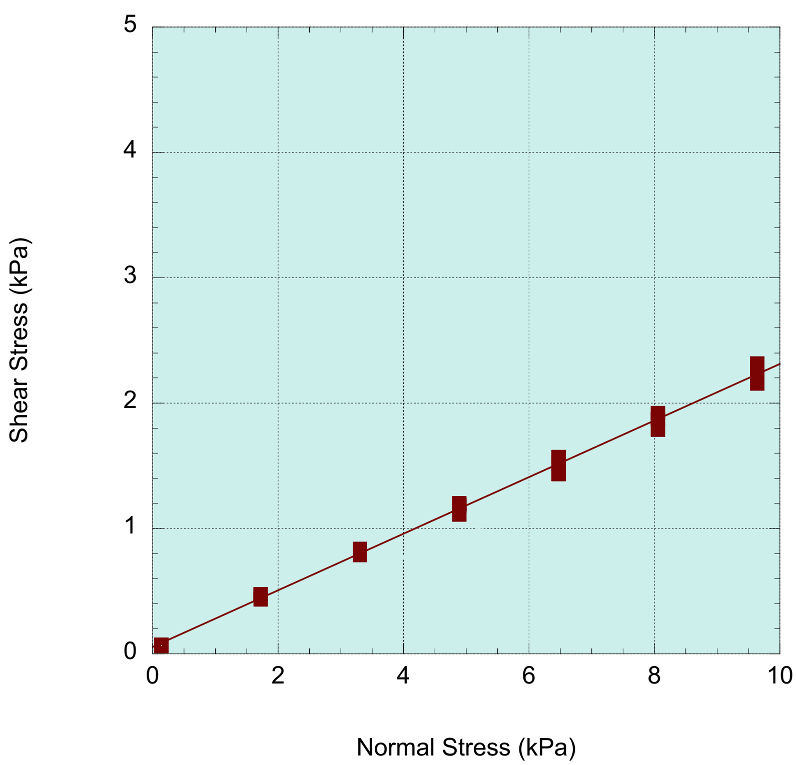

Figure 3. Wall friction between APAP/MCC/HPMC powder and 304 #2B stainless steel.

Jenike [1] developed design charts that provide allowable hopper angles for mass flow given values of the wall friction angle and the effective angle of friction δ, which is determined by shear cell testing. Jenike's design charts for axisymmetric (*e.g.*, conical, pyramidal) and planar (*e.g.*, wedge, transition, chisel) are shown in Figure 4.

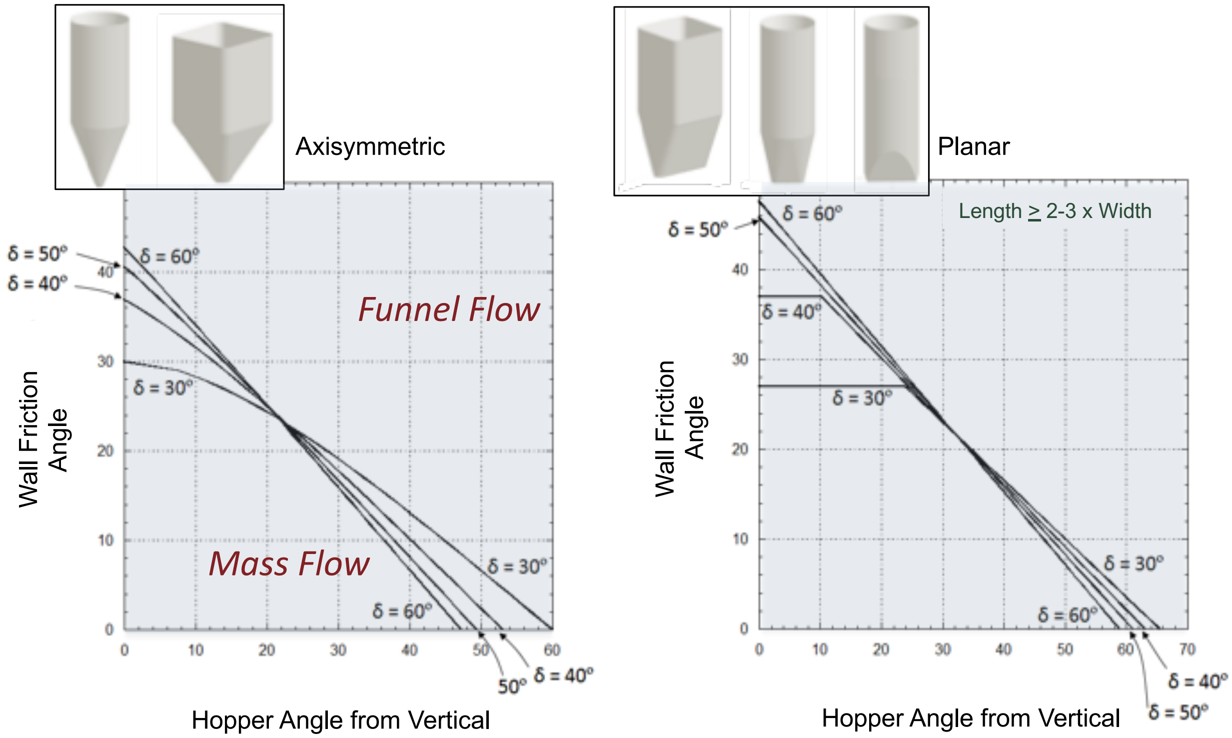

Figure 4. Jenike's recommended mass flow hopper angles – axisymmetric (left) and planar (right); a safety factor of 3-4 degrees should be used for axisymmetric hoppers.

Values of the allowable hopper angle are on the horizontal axis, and values of the wall friction angle (ϕ') are on the vertical axis. Any combinations of ϕ' and the hopper angle (from vertical) θ that lie within the mass flow region of the chart will provide mass flow. Designing right to the limit of the mass flow region is not recommended for conical bins. If the combination of wall friction angle and hopper angle lies too close to the funnel-flow line, a switch to funnel flow can occur. Hence, a 3-4-degree margin of safety is employed with respect to the mass flow boundary. For planar hoppers, mass flow can take place in hoppers shallower than Jenike's recommendations.

Because the wall friction angle depends on the wall stress, which decreases with decreasing outlet size, the recommended mass flow hopper angle depends on the size of the hopper outlet. Figure 5 presents recommended mass flow hopper angles for conical and planar hoppers that store and handle the APAP/MCC/HPMC powder having wall friction described by Figure 3.

Flow stoppages will be prevented if the stresses imparted on an obstruction to flow (such as a cohesive arch or stable rathole) are greater than the cohesive strength that the material gains due to its consolidation in a bin or hopper. The cohesive strength of a bulk solid can be determined using a shear cell tester to measure the failure strength of a material under varying consolidation pressures. The relationship between strength and pressure is called the flow function. The flow function for the sample of fine powder, measured using a Schulze RST-01.pc ring shear tester, is shown in Figure 6.

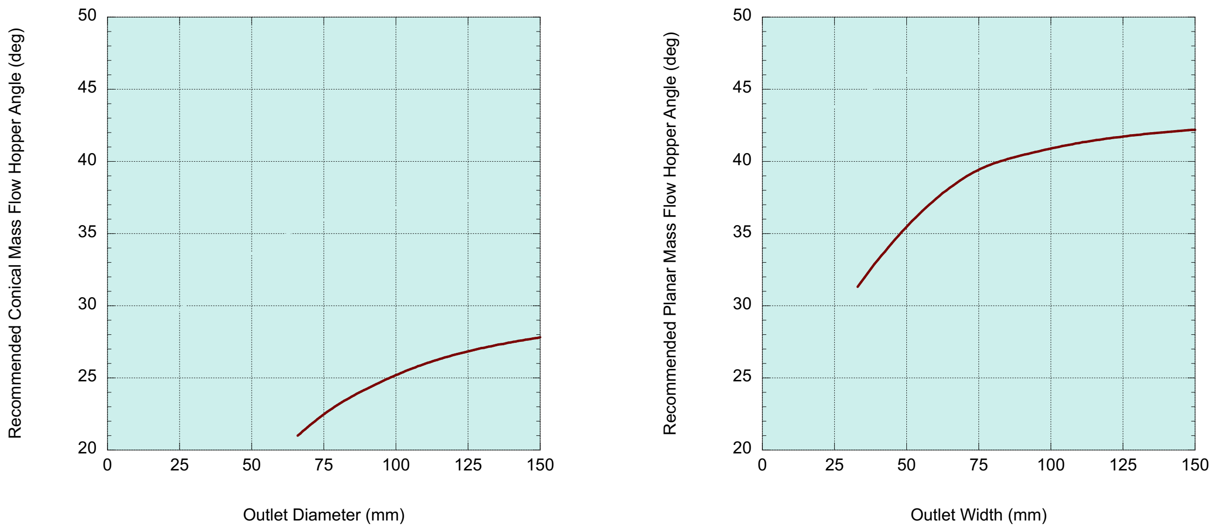

Figure 5. Recommended mass flow hopper angles – axisymmetric (left) and planar (right).

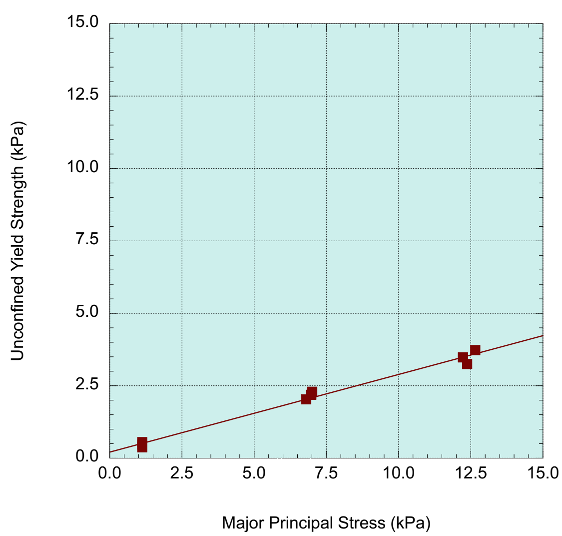

Figure 6. Flow function of fine APAP/MCC/HPMC powder.

The stress imparted on an arch of powder that forms at the vessel outlet is proportional to the material's bulk density. Provided that this stress is greater than the powder's cohesive strength, arching will not occur. Once a material's flow function has been determined and its bulk density has been measured, the minimum outlet diameter that will prevent a cohesive arch from developing can be calculated following an analysis developed by Jenike [1]. The critical arching outlet dimension can be calculated from

$$B_{min} = H(\theta') \frac{\sigma_{crit}}{\rho_b g}$$

where B_{min} is the minimum outlet diameter or width to prevent arching, $H(\theta')$ is a function defined by Jenike [1] (approximately equal to 2 for round outlets, 1 for slotted outlets), σ_{crit} is the critical stress, ρ_b is the bulk density, and g is acceleration due to gravity. The critical stress is that where the stress on the abutments of an arch is equal to the bulk material's cohesive strength, which is described by the material's flow function. Jenike's method to determine the minimum outlet required to prevent arching in a mass flow hopper is described by Mehos [2].

The analysis shows that a conical mass flow hopper requires a 65-mm diameter outlet to prevent arching when handling fine APAP/MCC/HPMC powder. For a planar hopper with a slotted outlet, the width of the outlet should exceed 30 mm. (Recall that $H(\theta')$ is approximately equal to 2 for round outlets and 1 for elongated outlets.)

In general, flowability improves with increasing particle size. Cohesive strength usually increases with decreasing particle size due to greater specific surface-area and a greater number of contacts between particles. To improve the flowability of the APAP/MCC/HPMC powder, the blend was agglomerated using an LCI Corporation blender, extruder and spheronizer (see Figure 7).

Figure 7. LCI Corporation MG-55 extruder and QJ-230T Marumerizer spheronizer.

The cohesive strength, internal friction, wall friction and bulk densities of the fine and spheronized blend are compared in Figure 8. Note that spheronizing dramatically reduces the cohesive strength of the material and its wall friction and increases its bulk density.

While a 65-mm diameter outlet is recommended to prevent the fine powder from arching in a conical hopper, the shear cell tests and subsequent analysis reveal that cohesive arching will not occur in a hopper or bin that handles the spheronized product. Instead, the outlet size of a vessel that handles the agglomerates should be selected by consideration of particle interlocking or desired discharge rates. For 1-mm diameter particles, a 6-mm diameter outlet is sufficiently large to prevent arching due to mechanical interlocking.

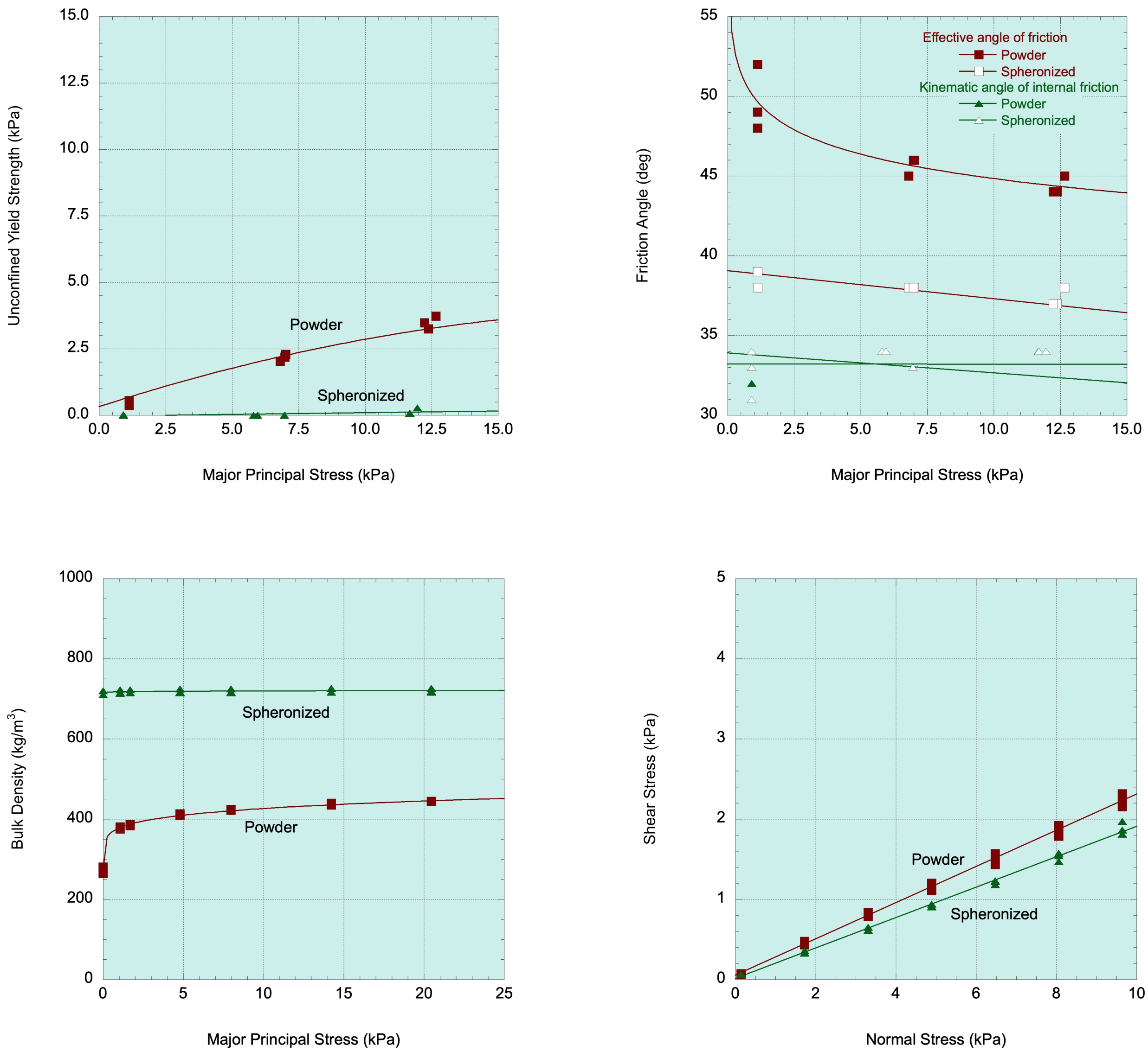

Figure 8. Flow properties of powdered and spheronized APAP/MMC/HPMC – cohesive strength (upper left), internal friction (upper right), bulk density (lower left), and wall friction (lower right).

In addition, wall friction test results reveal that the spheronized material requires hopper walls that are significantly less steep to allow mass flow. Recommended mass flow hopper angles for axisymmetric and planar hoppers handling fine and spheronized powder are given in Figure 9. If a conical hopper, bin, or silo with a 65-mm diameter outlet (*i.e.*, the minimum arching diameter) is fabricated or lined with 304 #2B finish stainless steel, walls sloped 22 degrees from vertical are recommended to ensure mass flow. A hopper fabricated using the same wall material sloped 44 degrees from vertical and a 5-mm diameter outlet will discharge spheronized material in mass flow.

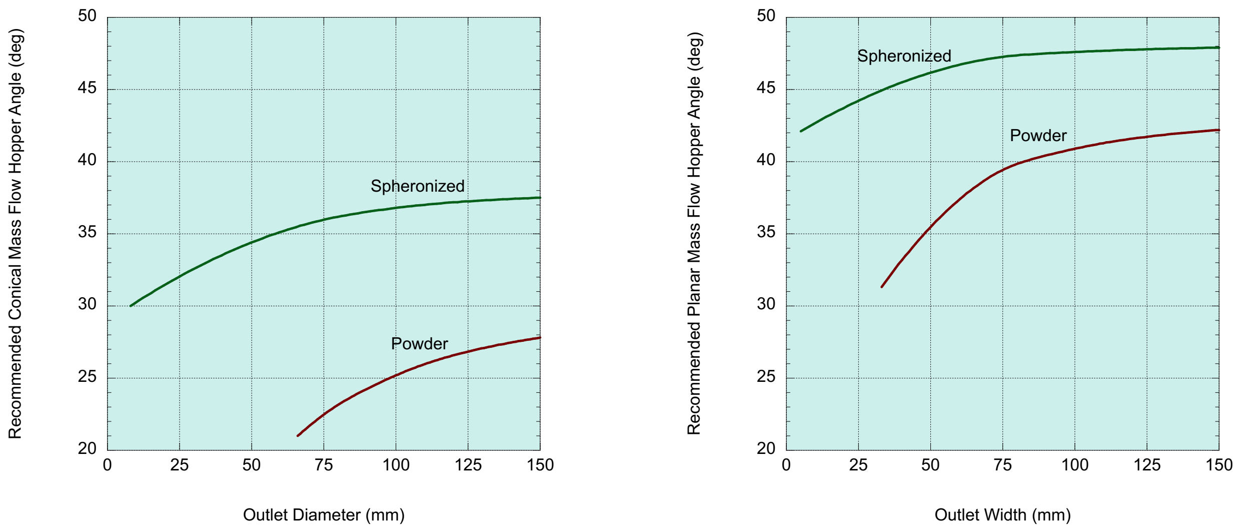

Figure 9. Comparison of recommended mass flow hopper angles – axisymmetric (left) and planar (right) – for bins handling fine and spheronized APAP/MCC/HPMC.

A funnel flow hopper or bin can be used provided its outlet is large enough to prevent a stable rathole from forming. From Jenike [1], D_F, the minimum outlet diameter of a funnel flow conical hopper or diagonal of a planar funnel flow hopper with a slotted outlet, can be calculated from

$$D_f = \frac{G(\phi) f_{Cmax}}{\rho_b g}$$

where $G(\phi)$ is a function defined by Jenike [1], ϕ is the kinematic angle of internal friction (determined from shear cell testing), and f_{Cmax} is the cohesive strength at the maximum solids stress level in a funnel flow hopper. The maximum solids stress is assumed equal to that at the junction of the straight-walled and converging sections of the bin or hopper and can be calculated from the Janssen equation:

$$\sigma_v = \frac{\rho_b g R_H}{k \tan \phi'} \left[1 - \exp \frac{(-k \tan \phi')}{R_H} z \right]$$

where σ_v is the solids stress, R_H is the hydraulic radius, k is the Janssen coefficient, assumed to equal 0.4, and z is the solids depth in the cylinder. The critical outlet diameter of a funnel flow bin will therefore depend on the maximum solids stress inside the hopper, which depends on the properties of the powder, the diameter of the cylinder section, and the powder depth.

Critical rathole outlet diameters of funnel flow bins handling fine and spheronized APAP/MCC/HPMC are summarized in Figure 10. Funnel flow bins used to store the fine powder require large outlets to ensure that the ratholes that develop will collapse. For example, a funnel flow bin with a 1-m diameter, 0.5-m tall cylinder completely filled with fine powder requires a 380-mm to prevent a stable rathole.

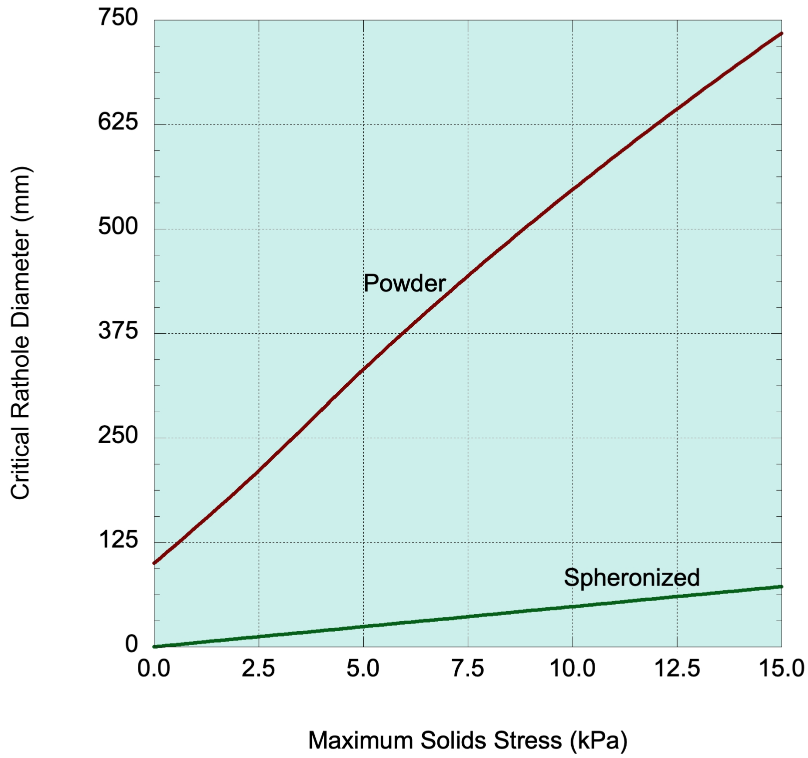

Figure 10. Critical rathole diameters.

The outlet of a planar funnel flow must be wide enough to prevent arching. The minimum width of a slotted outlet of a planar funnel flow hopper is 30 mm when the fine powder is handled. A cohesive arch will not develop if spheronized material is stored.

Concluding remarks

Fine powders are often challenging to handle due to their high cohesive strength, low bulk density, and high wall friction on most surfaces. Spheronization improves the flowability of a powder by dramatically reducing its cohesive strength, increasing its bulk density, and reducing its wall friction. When storing and handling spheronized powders, hoppers with shallow walls can often be used since mass flow is readily achieved and if not, ratholes are likely to be unstable.

References

1. Jenike, A.W., Storage and Flow of Solids, Bulletin 123, University of Utah Engineering Station, 1964 (revised, 1976).
2. Mehos, G., "Using Solids Flow Properties to Design Mass- and Funnel-Flow Hoppers", Powder Bulk Engr., 34, 2 (February 2020).