Wet Granulation of Fumed Silica

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Pin mixers are frequently used to convert fine particles into larger ones. Powder, liquid (preferably water), and binder are fed into a pin mixer, which consists of an agitator comprised of pins on a horizontal rotating shaft inside an enclosed chamber. The relative rates of coalescence and attrition and the time inside the pin mixer determine the final particle size distribution.

The "green" or wet agglomerates that leave the pin mixer must have sufficient strength to withstand the downstream drying process. The strength of a green pellet highly depends on its saturation state, which is the fraction of the void volume of the agglomerates that contain water. Green agglomerates from a pin mixer are the most durable when at their funicular state of saturation, when exists when water begins at accumulate at the contact points between individual particles. At lower moisture contents, dry particles exist, which can turn into dust. Too high a moisture content will reduce the capillary forces that hold the particles together, and if appreciably high, a paste or slurry may be created. Saturation states are illustrated in Figure 1.

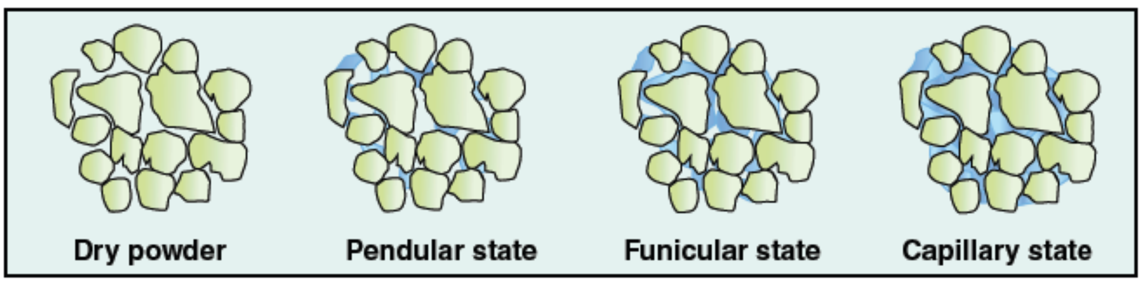

Figure 1. Saturation states.

<u>Bulk solids handling fundamentals</u>

There are two primary flow patterns that can occur in a bin: *mass flow* and *funnel flow*. 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. Mass flow is illustrated in Figure 2.

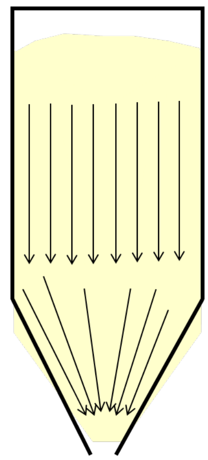

Figure 2. Mass flow pattern.

The steep hopper walls provide a more uniform flow, making mass flow hoppers suitable for agglomeration processes where steady feeds are required. Discharge rates are predictable and steady, since the bulk density of the material is nearly independent of the head of the material inside the vessel.

In *funnel flow*, an active flow channel forms above the outlet, with stagnant material remaining (*i.e.,* ratholes) at the periphery. 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. It is equal to the diameter of a round outlet or the diagonal of a slotted outlet. Collapsing ratholes can lead to variable density as the vessel will become comprised of both consolidated and aerated powder. Funnel flow is illustrated in Figure 3.

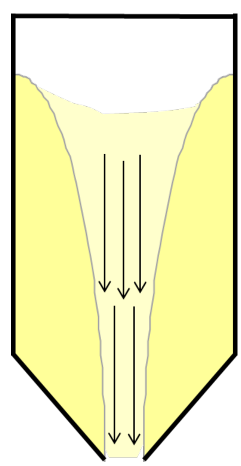

Figure 3. Funnel flow pattern.

If a stable dome, bridge, or arch forms over the outlet of a bin, the bulk solid will not flow when the feeder is started or gate is opened. If a stable rathole forms in a vessel in which flow only occurs in a narrow channel above the outlet, material will stop flowing when the flow channel empties. Obstructions to flow are illustrated in Figure 4.

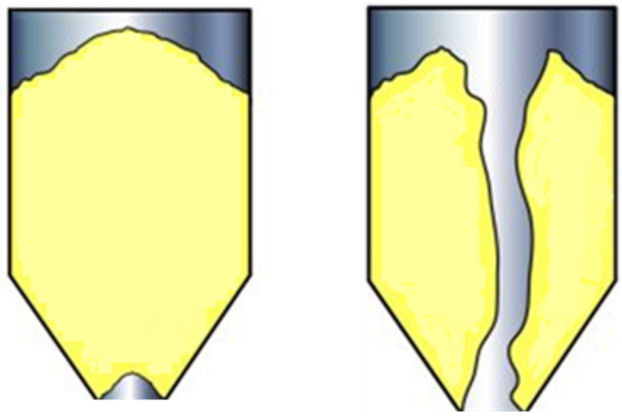

Figure 4. Obstructions to flow – cohesive arch (left), stable rathole (right).

To design a hopper for reliable flow, the following bulk solids flow properties must be known: (1) cohesive strength, (2) internal friction, (3) compressibility, (4) wall friction, and (5) permeability. These properties are measured using shear cell, wall friction, and permeability testers. The relationship between a bulk material's unconfined yield strength and major consolidation stress is the flow function. The effective yield locus provides the relationship between the major consolidation stress and the effective angle of friction. Compressibility is the relationship between a material's bulk density and major consolidation stress. Permeability relates the pressure drop through a bed of material and the superficial gas velocity. Details of the test equipment and testing procedures are given in the 9th edition of Perry's Handbook.

Two critical design parameters are specified for mass flow hoppers: the hopper angle and the size of the outlet. The hopper angle required to allow mass flow depends on the effective angle of friction δ, the wall friction angle, and the geometry of the hopper. Figures 5 and 6 provide recommended mass flow hopper angles for conical bins and bins with flat walls and slotted outlets based on analyses developed by Andrew Jenike. Values of the allowable hopper angle θ' are on the horizontal axis, and values of angle of wall friction ϕ' are on the vertical axis. The boundaries between mass flow and funnel flow depend on the effective angle of friction δ. Any combination of θ' and ϕ' that falls within the limiting mass flow region of the chart will provide mass flow.

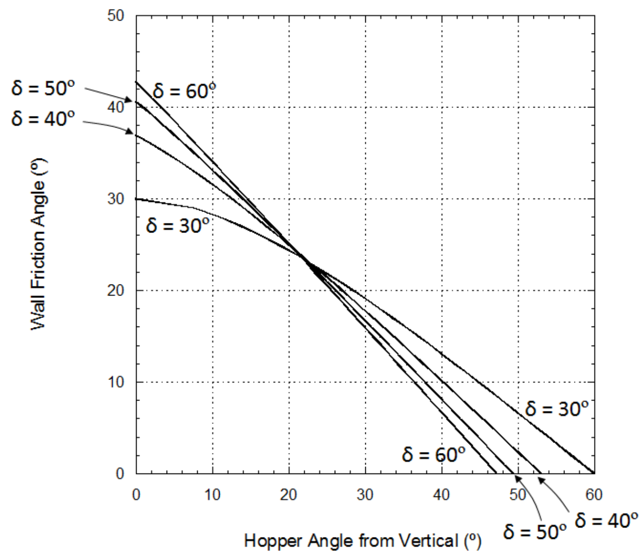

Figure 5. ***Theoretical*** **mass flow hopper angles for hoppers with round or square outlets.** ***Note: a minimum safety factor of 2 to 3° should be used.***

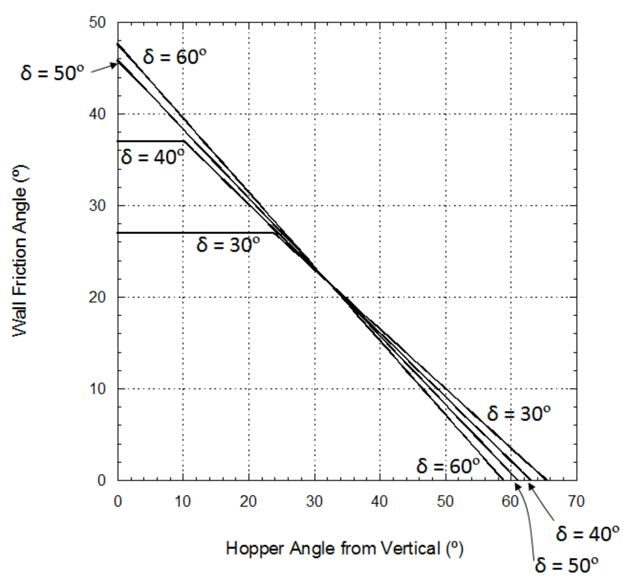

Figure 6. Recommended mass flow hopper angles for wedge-shaped hoppers.

A 2-3° safety factor with respect to the theoretical mass flow hopper angle is recommended in the design of a conical mass flow bin. Hopper angles of bins with flat walls and slotted outlets can be 5-10° greater than recommended in Figure 6 without risking funnel flow. The outlet of a planar flow bin, *i.e.,* one having a wedge-shaped or transition hopper section, must be at least two times as long as it is wide for Figure 6 to apply if it has vertical end walls and three times as long if its end walls are converging.

To prevent the formation of a stable cohesive arch at the outlet of a hopper, the external stress must be greater than the powder's unconfined yield strength. Jenike defined the flow factor *ff* as the ratio of the major consolidation stress *σ1* to the stress on the abutment of the arch that naturally forms at the outlet $\bar{\sigma}$:

$$ff = \frac{\sigma_1}{\bar{\sigma}}$$

The flow factor depends on the powder's effective angle of friction *δ*, the wall friction angle *ϕ'*, and the hopper angle *θ'*. Charts that provide flow factors can be found in Jenike's Bulletin 123 and the 9th edition of Perry's Chemical Engineers' Handbook. Examples are given in Figures 7 and 8.

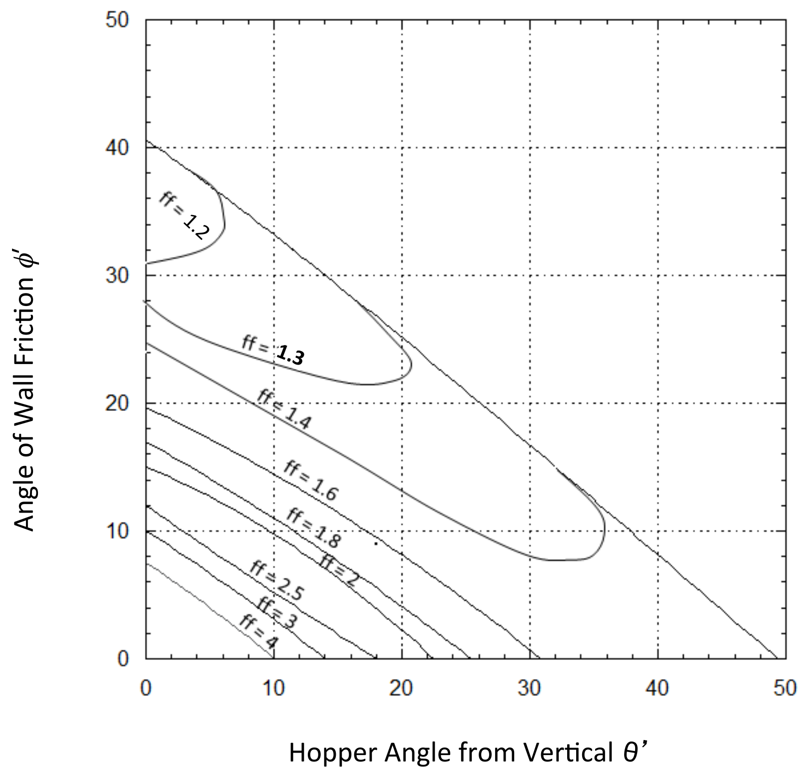

Figure 7. Flow factors for conical hoppers, *δ* = 50°.

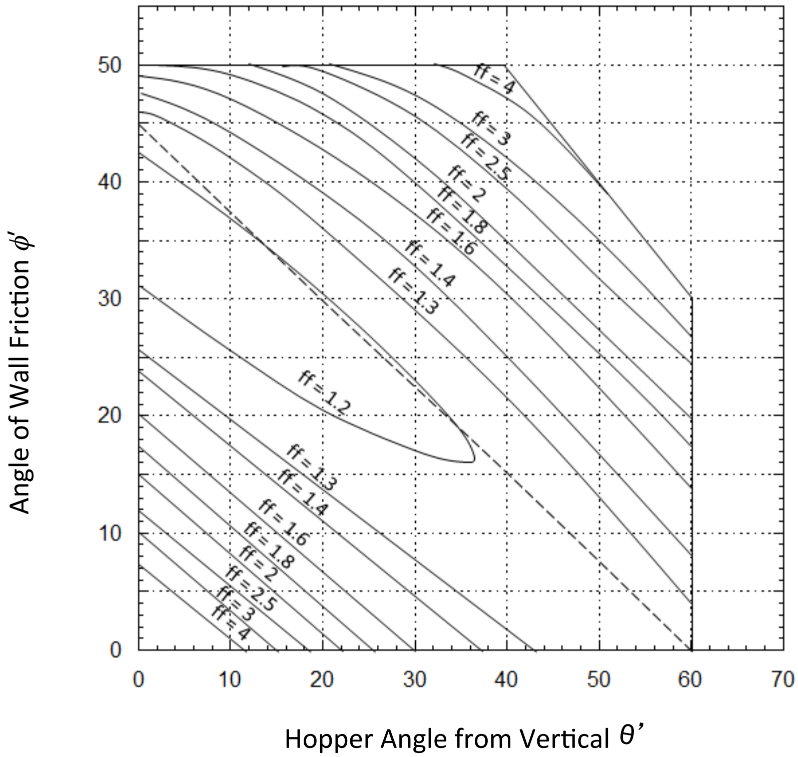

Figure 8. Flow factors for planar flow hoppers with slotted outlets, δ = 50°.

The size of the outlet required to prevent a cohesive arch from developing in a mass flow bin can be determined by first superimposing the flow factor and flow function on the same graph. The flow factor is constructed by drawing a line having a slope equal to $1/ff$ through the origin. As shown in Figure 9, three possibilities exist:

1. The flow function lies below the flow factor, and the two do not intersect. When this is the case, the stress imparted on the abutments of the arch is always greater than the material's cohesive strength, and therefore no minimum outlet dimension requirement to prevent cohesive arching exists. Instead, the outlet dimension B is determined by other considerations such as the required discharge rate.
2. The flow function lies above the flow factor and the curves do not intersect. The powder will not flow due to gravity alone. Consideration should be given to changing the flow properties of the material, such as increasing its particle size or using a flow aid, or using a standpipe.
3. The flow function and flow factor intersect. At the intersection of the two lines, the arch stress and the cohesive strength of the bulk solid are the identical and equal to the critical stress σ_{crit}. The hopper outlet diameter that must be exceeded to prevent arching, B_{min}, can be calculated from

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

where $H(\theta')$ is a shape factor equal to approximately 1 for slotted outlets and 2 for round outlets.

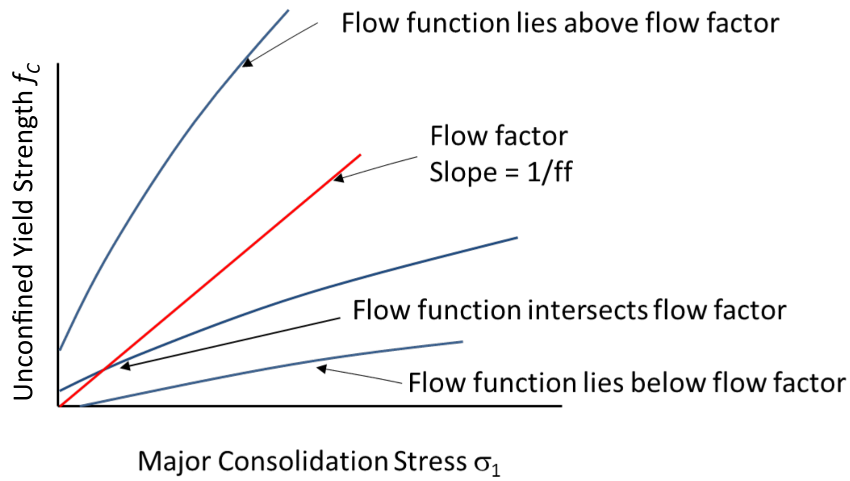

Figure 9. Plot showing both flow factor and Flow Function.

The outlet of a hopper, bin, and silo, should also be large enough to provide the desired solids discharge rate. Permeability and compressibility test results are used to determine the required outlet dimension. The design procedure is described in Perry's 9th edition.

<u>Properties of fumed silica.</u>

The results of cohesive strength tests and wall friction tests on 316 #2B finish stainless steel for Cabot Corporation's CAB-O-SIL M-5 are shown in Figures 10 and 11, respectively.

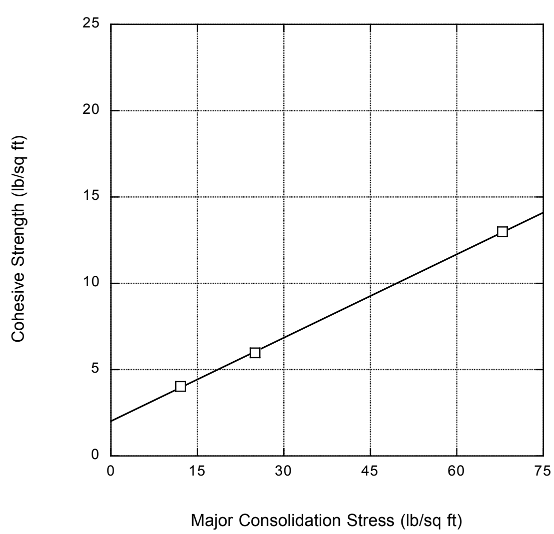

Figure 10. Cohesive strength of CAB-O-SIL M-5.

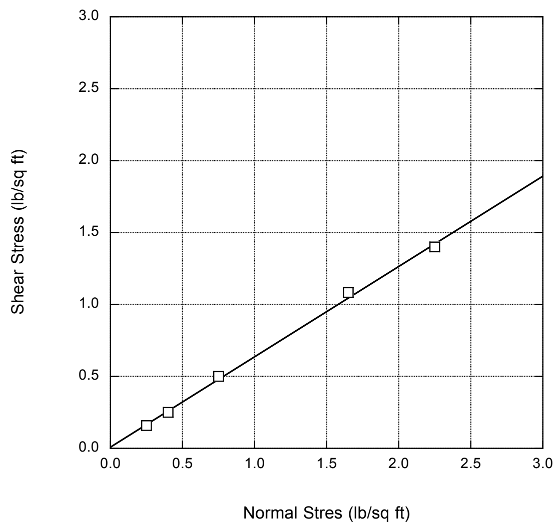

Figure 11. Wall friction of CAB-O-SIL M-5 on 316 #2B stainless steel.

Bulk density at low consolidation was measured to be 2.5 lb/cu ft.

The test results were used to determine the size of a hopper outlet required to prevent arching in a mass flow conical hopper and the recommended conical mass flow hopper angle. Calculations suggested that a conical hopper with a 3-in. diameter and walls sloped 5° from vertical were required. Obviously, such a design is impractical.

<u>Air assistance</u>

A conical hopper with gas permeable walls was fabricated. The strategy was to reduce the wall friction by injecting a small amount of air the lift the fumed silica off the hopper walls, thus greatly reducing the material's wall friction and allowing mass flow. Additionally, the plenum through which the air flowed on the non-process side was divided, so that additional air could be added near the outlet to reduce the fumed silica's cohesive strength. The goal was not to necessarily fluidize the powder. Rather, the main purpose of the gas was to create an "air-hockey" table effect and reduce wall friction.

To modulate flow, a 1-in. double-diaphragm pump was installed at the hopper outlet, as double-diaphragm pumps are frequently used to transfer fumed silica. The set up is shown in Figure 12.

Having a pump created an additional complexity. Given the pump curve and knowing the system curve, a pump can be operated to achieve a desired volumetric flow rate of air. The system curve is described by Darcy's law, *i.e.*,

$$v_g = -AC\frac{dP}{dz}$$

where v_g is the gas volumetric flow rate, A is the cross-sectional area, C is the permeability, and dP/dz is the pressure gradient.

Figure 12. Air-assist hopper and double-diaphragm pump for handling fumed silica.

From an equilibrium force balance, the solids discharge rate $\dot{m}_s$ is given by

$$\dot{m}_s = \rho_{bo} \frac{\pi B^2}{4} \sqrt{\frac{Bg}{4\tan\theta'} \left(1 + \frac{\left.\frac{dP}{dz}\right|_o}{\rho_{bo} g} \right)}$$

where ρ_b is the bulk density, θ' is the hopper angle from vertical, g is acceleration due to gravity, B is the outlet diameter, and the subscript o denotes the outlet. Note that the gas discharge rate depends on the pressure gradient. As the pressure gradient decreases, the solids discharge decreases. However, as the pressure drop decreases, the volumetric gas flow rate increases. The key was therefore to keep sufficient material inside the cylinder section of the hopper to keep the pressure drop relatively constant so that a constant solids discharge could be maintained.

The assembly was connected to a continuous pin mixer using flexible tubing and a transfer chute. To ensure flow on a transfer chute, the following design equation was used:

$$\alpha > \phi'$$

where α is the chute angle referenced from horizontal. Based on the wall friction test results chute angle of 45° was chosen. A dramatic safety factor was used since wall friction tests were conducted on 316 #2B stainless steel, whereas the chute was fabricated from pVC. Figure 13 is a photograph of the entire assembly.

Figure 14 is a plot of the solids content of the agglomerates during three campaigns. The composition was remarkably constant considering that a load cell was not available to measure the loss in weight of the hopper and allow control of the mass flow rate.

Figure 13. Air-assist hopper, double-diaphragm pump, transfer chute, and pin mixer.

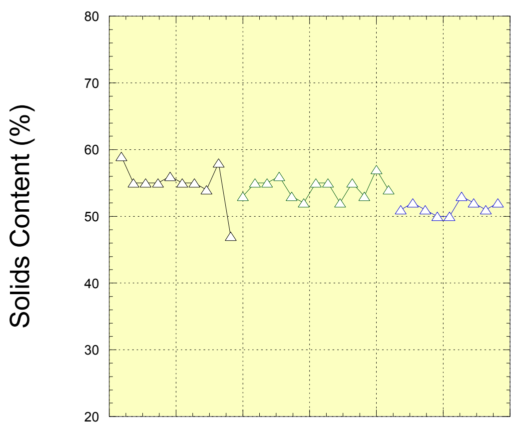

Figure 14. Solids content of granulated fumed silica.

Final remarks

The key to producing granules with acceptable strength using a wet agglomeration process is to maintain a proper ratio of solids, liquid, and binder, usually near the funicular state of saturation. The hoppers from which solids are discharged should be designed for mass flow and based on the materials' fundamental flow properties. For highly frictional, cohesive powders, air assistance should be considered.