powder flow

Using fundamental powder properties to optimize flowability

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Many methods of measuring powder flowability aren't predictive and can't be used to select or design the optimal hopper or bin. Shear cells offer a better approach. This article details how to use them to quantify flow behavior and to design vessels that ensure reliable flow.

Formulators have a choice of tests to quantify the flowability of powders, including angle of repose, Carr's compressibility index, Hausner ratio, and the time required to discharge powder through an orifice. Unfortunately, none of these methods is predictive, because they don't simulate actual conditions. At best, they can be used to rank the flowability of similar powders.

The most useful test method is one that measures a powder's fundamental properties under consolidation stresses that simulate those anticipated when the powder is handled and measures fundamental properties. The fundamental solids flow properties are cohesive strength, internal friction, compressibility, wall friction, and permeability. By acquiring these data from tests conducted on small samples, it's possible to replicate the conditions within larger-scale systems. This means that you can predict flow behavior from fundamental principles.

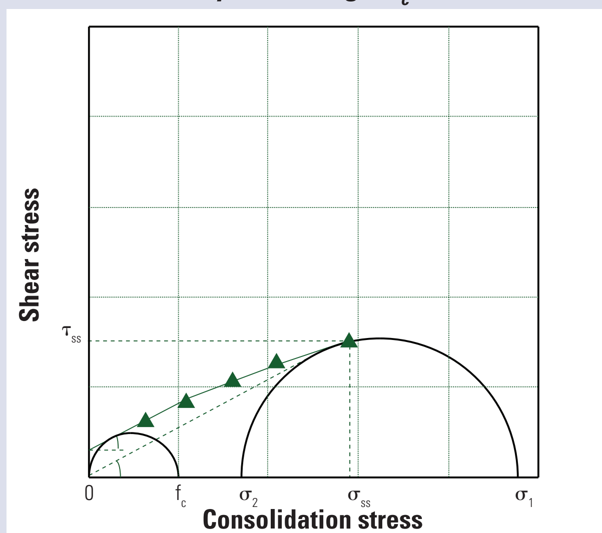
Methods for measuring fundamental flow properties

Shear cell testers measure the cohesive strength, internal friction, compressibility, and wall friction of a powder. To do so, a sample is placed in a cell and then presheared, i.e., consolidated by exerting a normal stress σ and then shearing it until the measured shear stress τ is steady. This establishes a state of consolidation in the sample that replicates the pressures that would be experienced at a particular position in a storage vessel or container. Next, the shear step is conducted, in which the vertical compacting load is replaced with a reduced load, and the sample is again sheared until it fails. The preshear and shear steps are repeated at the same consolidation level for a number of reduced normal stresses, and the yield locus is then determined by plotting the failure shear stress against normal stress.

The flow properties of the powder are determined from the yield locus (Figure 1). The semicircles in this plot are called Mohr's circles. They allow us to determine the normal and shear stresses within conveniently chosen frames of reference.

Figure 1

Construction of a yield locus and determination of the effective angle of friction δ, kinematic angle of internal friction ϕ, major consolidation stress σ_1, minor consolidation stress σ_2, and unconfined yield strength f_c

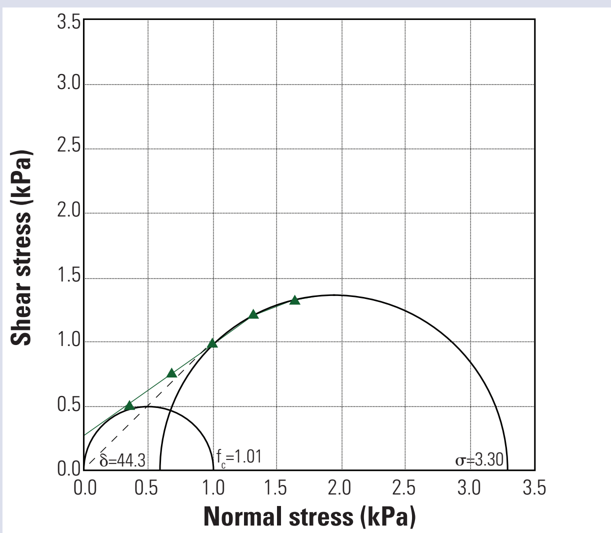


The major consolidation stress is determined from a Mohr's circle that intersects the point of steady-state flow (σ_{ss}, τ_{ss}) and is tangent to the yield locus. The larger point of intersection of the Mohr's circle with the horizontal axis is the major consolidating stress σ_1. The effective angle of friction δ is determined from the angle formed when a line passing through the origin is tangent to the Mohr's circle. The unconfined yield strength f_c is determined by a Mohr's circle that is tangent to the yield locus and passes through the origin. Because the sample volume is recorded, the powder's bulk density is also measured and is typically expressed in kilograms per cubic meter (kg/m^3).

The major consolidation stress describes the stress state that was used to consolidate the sample during the pre-shear step; it is a combined stress state that includes the normal and shear stresses present during steady flow. The unconfined yield strength is a measure of the cohesive strength that the powder gained due to its consolidation during the pre-shear step. The effective angle of friction is related to the friction between powder particles during flow. Figure 2 shows the yield locus of a blend containing 67 percent acetaminophen (APAP), 29 percent microcrystalline cellulose (MCC), and 4 percent hydroxypropyl cellulose (HPC).

FIGURE 2

Yield locus of APAP-MCC-HPC blend, σ_{ss} = 1.63 kPa



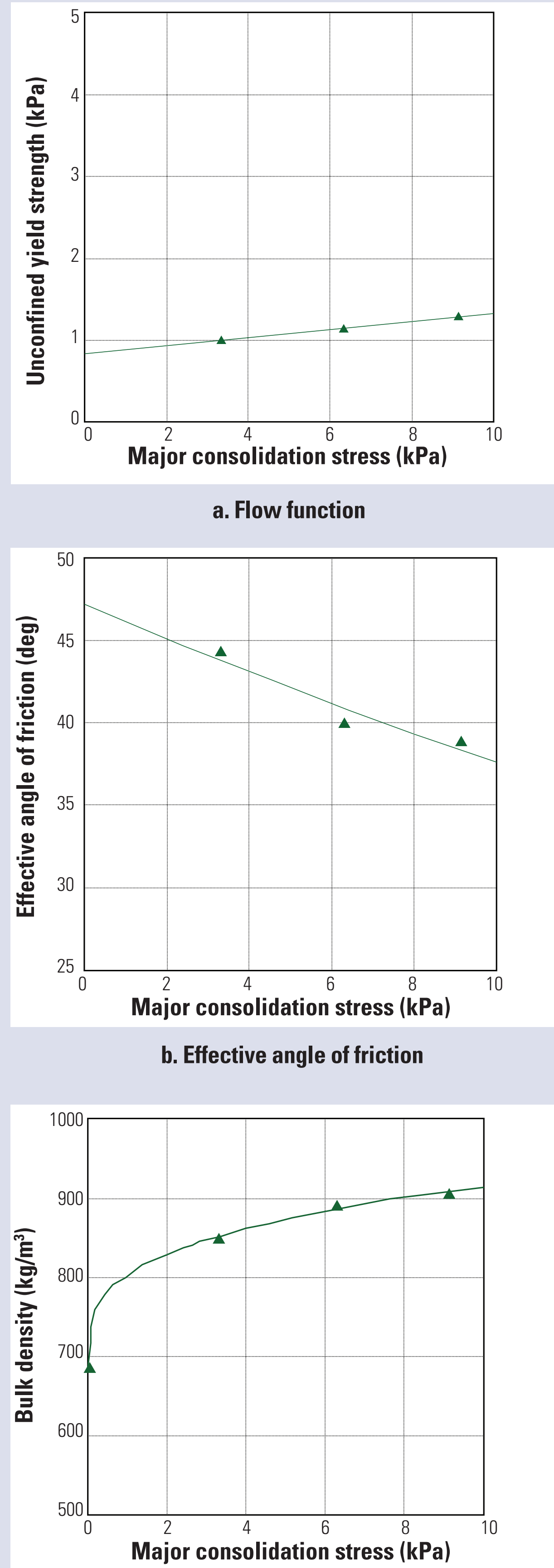
By conducting the test over a range of consolidation states, the relationship between the powder's major consolidation stress and its cohesive strength can be established. This relationship is commonly called the material's flow function. The flow function relates the cohesive strength that a powder develops due to its consolidation in a hopper to the major consolidation stress on the material. The powder's compressibility is the relationship between its bulk density and the major consolidation stress. The flow function, effective angle of friction, and compressibility of the APAP-MCC-HPC blend are shown in Figure 3.

Testing wall friction entails measuring the shear stress required to allow a sample of powder to slide along a coupon, or section, of wall material. A plot of the shear stress versus normal stress provides the wall yield locus. The angle of wall friction ϕ' is the angle that is formed when a line is drawn from the origin to a point on the wall yield locus. Figure 4 presents the wall yield locus determined from wall friction tests of the APAP-MCC-HPC blend on a section of 314 stainless steel with a 2B finish.

Permeability is determined by measuring the pressure drop that results when gas is fed into a bed of powder at

FIGURE 3

Calculated properties of an APAP-MCC-HPC blend



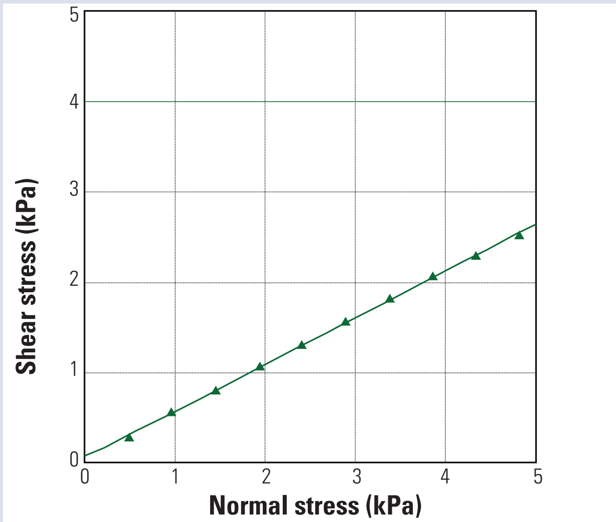
a. Flow function

b. Effective angle of friction

c. Effective bulk density

FIGURE 4

Wall friction of APAP-MCC-HPC blend on 304 stainless steel with 2B finish



low velocity. The superficial gas velocity is related to the gas pressure gradient by:

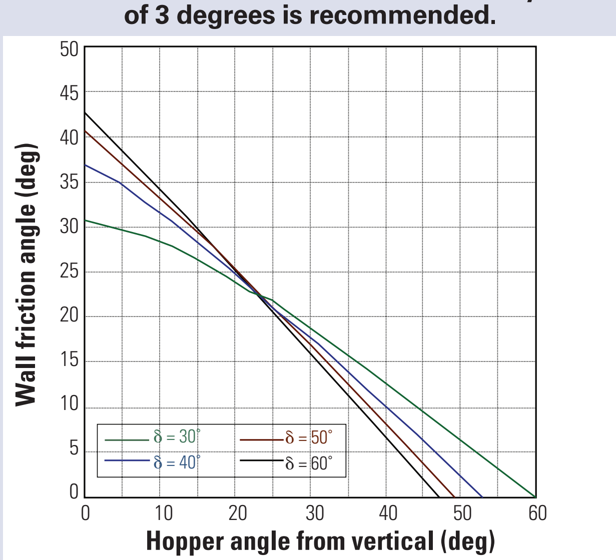
$$u_s = -\frac{K}{\rho_b g}\,\frac{dP}{dz} \qquad (1)$$

where u_s is the gas superficial velocity; K is the powder's permeability; ρ_b is its bulk density; g is equal to acceleration due to gravity; and dP/dz is the gas pressure gradient. Permeability test results are useful for estimating solids discharge rates.

Many investigators use the metric FFC—the ratio of the major consolidation stress σ_1 to the unconfined yield strength f_c determined from yield locus measurements—as a metric for flowability. FFC is frequently misused because it is generally evaluated at high stresses, where FFC is more readily distinguishable. But in reality, the stresses are low at the outlet of hoppers designed to prevent ratholing. In addition, the ratio ignores the influences of wall

FIGURE 5

Theoretical mass flow boundaries. A safety factor of 3 degrees is recommended.



friction and bulk density on flow behavior. A better approach to optimize a formulation is to use the measured flow properties in order to determine the size of the outlet of a hopper that will prevent flow obstructions, the slope of its walls that will prevent ratholing, and the outlet size that will achieve the desired discharge rate.

Mass-flow hopper angle

Two flow patterns can occur in a hopper: mass flow and funnel flow. In mass flow, the entire bed of solids is in motion when material is discharged from the outlet. This behavior eliminates the formation of ratholes in the vessel, affords a "first-in, first-out" flow sequence, and provides a more uniform velocity profile during operation. A uniform velocity profile mitigates sifting segregation, which would result in side-to-side separation of particles by size.

In funnel flow, an active flow channel forms above the outlet, with stagnant material (i.e., ratholes) remaining at the periphery of the hopper. Hoppers in which funnel flow occurs may require a very large outlet to ensure that the ratholes collapse and the hopper empties. Funnel flow can cause erratic flow and exacerbate segregation, and material that forms the ratholes may spoil or cake. Mass-flow hoppers are therefore preferable for handling pharmaceutical formulations.

The hopper angle required to allow mass flow depends on the effective angle of friction δ, the wall friction angle ϕ′, and the geometry of the hopper. Figure 7 provides mass flow boundaries for conical hoppers based on analyses developed by Jenike [1]. Values of the allowable hopper angle θ′ are on the horizontal axis, and values of the angle of wall friction ϕ′ are on the vertical axis. The theoretical 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. A 2- to 3-degree safety factor with respect to the theoretical mass-flow hopper angle is recommended when designing a conical mass-flow hopper. The figure confirms that mass flow is more likely in hoppers with steep walls (small values of θ′) and low friction (small values of ϕ′).

An analytical expression for the recommended mass flow boundary is given by Equation 2 [2]:

$$\theta' = 90^\circ - \frac{1}{2}\cos^{-1}\left(\frac{1-\sin\delta}{2\sin\delta}\right) - \beta - (2\text{-}3^\circ) \qquad (2)$$

where

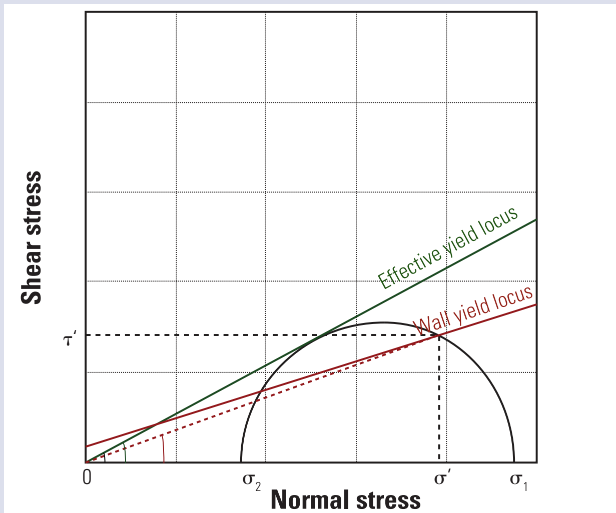
$$\beta = \frac{1}{2}\left(\phi' + \sin^{-1}\left(\frac{\sin\phi'}{\sin\delta}\right)\right) \qquad (3)$$

Note that the equation includes a 2- to 3-degree margin of safety.

The wall friction angle ϕ′ depends on the effective angle of friction δ and the major consolidation stress σ_1. It is determined from the intersection of the Mohr's circle

Figure 6

Determination of the wall friction angle from the major consolidation stress and effective angle of friction



associated with σ_1 and δ and the wall yield locus. See Figure 6. The Mohr's circle is given by

$$(\sigma-\sigma_{avg})^2+\tau^2=R^2 \tag{4}$$

where

$$R=\frac{\sigma_1-\sigma_2}{2} \tag{5}$$

and

$$\sigma_{avg}=\frac{\sigma_1+\sigma_2}{2} \tag{6}$$

The minor principle stress σ_2 is related to the effective angle of friction δ by:

$$\frac{\sigma_2}{\sigma_1}=\frac{1-\sin(\delta)}{1+\sin(\delta)} \tag{7}$$

If the wall yield locus is linear, which is often true at low stresses, it can be described by:

$$\tau'=a\sigma'+b \tag{8}$$

where τ' and σ' are the shear and normal stresses at the wall surface, respectively, and a and b are empirical constants determined from regression. The normal stress can then be calculated from:

$$\sigma'=\frac{-\beta+\sqrt{\beta^2-4\alpha\gamma}}{2\alpha} \tag{9}$$

where

$$\alpha=a^2+1 \tag{10}$$

$$\beta=2(ab-\sigma_{avg}) \tag{11}$$

and

$$\gamma=b^2+\sigma^2_{avg}-R^2 \tag{12}$$

The wall friction angle is then calculated from:

$$\phi'=\tan^{-1}\left(\frac{\tau'}{\sigma'}\right) \tag{13}$$

where the shear stress at the wall τ' is calculated from Equation 8.

Minimum outlet dimension

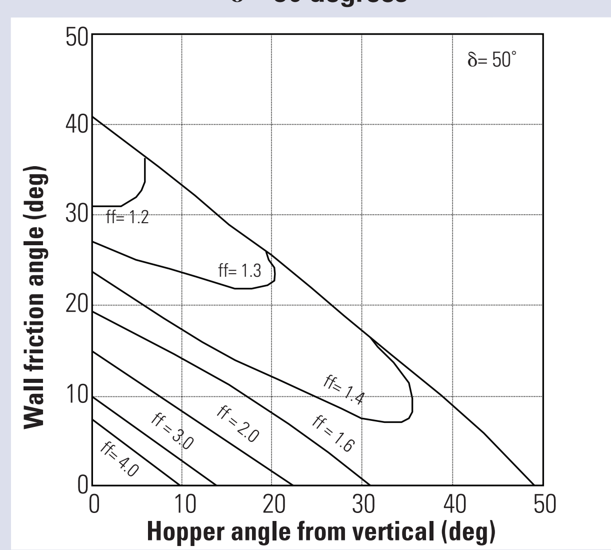
To prevent the formation of a stable cohesive arch at the outlet of a hopper, the external stress must be greater than the powder's unconfined yield strength. Jenike [1] defined the flow factor *ff* as the ratio of the major consolidation stress σ_1 to the stress on the abutment of the arch that naturally forms at the outlet $\bar{\sigma}$:

$$ff=\frac{\sigma_1}{\bar{\sigma}} \tag{14}$$

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 are provided by Jenike [1], and an example of a chart is given in Figure 7, which shows the flow factor for powders having an effective angle of friction equal to 50 degrees that are handled in conical hoppers.

Figure 7

Flow factor chart for conical hoppers, δ = 50 degrees



Analytical expressions of the flow factor are provided by Arnold and McLean [3,4]. For conical hoppers, the flow factor can be determined from:

$$ff=\frac{Y(1+\sin\delta)H(\theta')}{2(X-1)(\sin\theta')} \tag{15}$$

where

$$X=\frac{2\sin\delta}{1-\sin\delta}\left[\frac{\sin(2\beta+\theta')}{\sin\alpha}\right] \tag{16}$$

$$Y=\frac{[2(1\text{-}\cos(\beta+\theta'))]\sin\theta'+\sin\beta\sin2(\beta+\theta')}{(1\text{-}\sin\delta)\sin^3(\beta+\theta')} \tag{17}$$

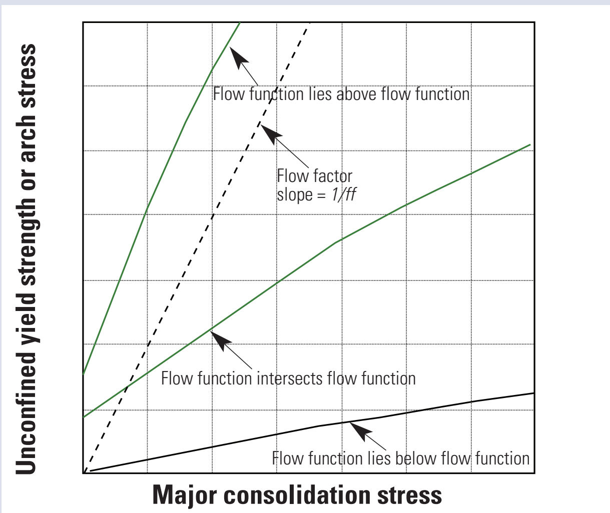
and

$$H(\theta')=\frac{130°+\theta'}{65} \tag{18}$$

These equations may be serpentine, but they can readily be input into spreadsheets and other software. Flow factors typically range between 1.2 and 1.6 but can be much greater if wall friction is exceptionally low and hopper walls are very steep.

FIGURE 8

Plot of flow function and flow factor



The cohesive strength and arch stress can be compared by 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 8, three possibilities exist:

1. The flow function lies below the flow factor, and the two do not intersect. In this case, the stress imparted on the abutment of the arch is always greater than the material's cohesive strength, and there is no minimum outlet dimension requirement to prevent cohesive arching. Instead, the outlet dimension B is determined by other considerations, such as the discharge rate required. The hopper angle required for mass flow requires the major consolidation stress σ_1 at the outlet to be known. This stress is calculated from:

$$\sigma_1=ff\,\frac{\rho_b gB}{H(\theta')} \tag{19}$$

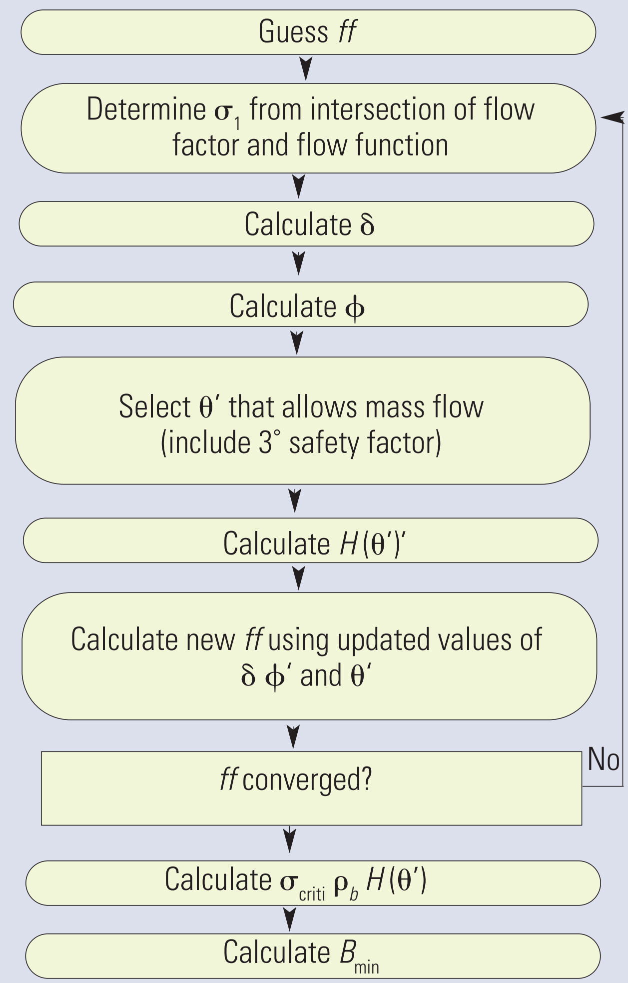
2. The flow function lies above the flow factor and the curves do not intersect. The powder will not flow due to gravity alone. Consideration should be given to changing the flow properties of the material, perhaps by increasing its particle size or using a flow aid.

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 same 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:

$$B_{min}=\frac{H(\theta')\sigma_{crit}}{\rho_b g} \tag{20}$$

FIGURE 9

Flowchart for determining critical hopper outlet size and mass-flow hopper angle

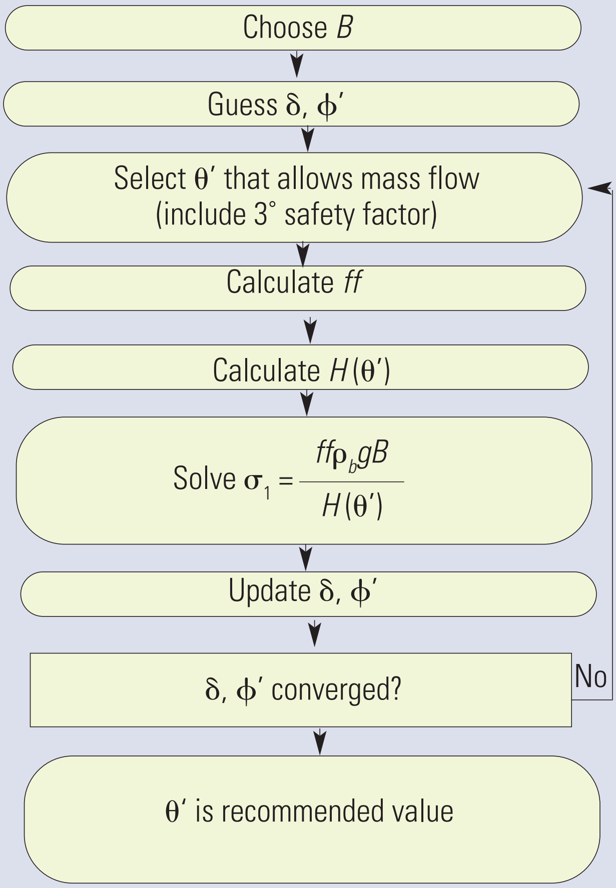


The bulk density ρ_b the effective angle of friction δ, and the angle of wall friction ϕ' depend on stress, and therefore calculating critical hopper angles and arching dimensions is an iterative procedure. Figure 9 is a flowchart for calculating the minimum hopper outlet diameter and recommended mass-flow hopper angle for the case where the flow function and flow factor intersect. A flowchart for determining the recommended mass-flow hopper angle for selected outlet diameters is given in Figure 10.

The outlet must be large enough to provide the desired discharge rate. For coarse powders, the solids discharge rate m_s is given by:

FIGURE 10

Flowchart for determining recommended mass-flow hopper angle for a specified outlet diameter



$$\dot{m}_s = \rho_{bo} \frac{\pi B^2}{4} \sqrt{\frac{Bg}{4\tan(\theta')}} \tag{21}$$

The maximum discharge rate of a fine powder can be orders of magnitude less than that of a coarse powder due to an adverse gradient that naturally develops near the outlet as the powder dilates. For fine powders, the following equation provides a conservative estimate of the maximum discharge rate [5]:

$$\dot{m}_s = \rho_{bo} \frac{\pi B^2 K_o}{4\left(1 - \frac{\rho_{bo}}{\rho_{bmax}}\right)} \tag{22}$$

where the subscripts *o* and *max* denote the value at the outlet and the maximum value, respectively. The maximum bulk density ρ_{bmax} develops at the major consolidation stress at the hopper cylinder junction, which is calculated from the Janssen equation [6]:

$$\sigma_1 = \frac{\rho_b g D}{4k\tan(\phi')}\left[1 - \exp\left(-\frac{4k\tan(\phi')h}{D}\right)\right] \tag{23}$$

where D is the cylinder diameter, h is the depth of powder in the cylinder section, and k is the Janssen coefficient. Average values of the bulk density and wall friction angle are used. The Janssen coefficient can be assumed equal to 0.4.

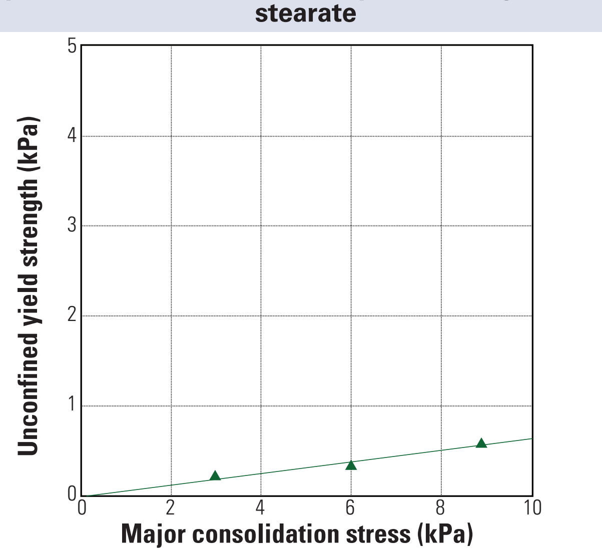
Sample calculations

Consider the powder blend containing 67 percent APAP, 29 pecent MCC, and 4 percent HPC. Its powder flow properties appear in figures 3 and 4, and follow these steps to design a suitable mass-flow hopper:

1. Choose 1.2 as an initial estimate for *ff*.
2. Determine the major consolidation stress at the intersection of the flow function and flow factor. The major consolidation stress σ_1 is equal to 1.07 kilopascals (kPa).
3. Determine δ. From Figure 3b, δ equals 45.8 degrees (°).
4. Calculate ϕ'. From equations 4 to 13, σ_2 = 0.18 kPa, σ_{avg} = 0.62 kPa, R = 0.45 kPa. The normal and shear stresses at the wall equal 0.72 kPa and 0.44 kPa, respectively, and ϕ' = 31.0°.
5. Select the mass-flow hopper angle. From equations 2 and 3, θ' = 9.2°.
6. Calculate H(θ'). From Equation 18, H(θ') = 2.14.
7. Update the flow factor. Using δ = 45.8°, ϕ' = 31.0°, and θ' = 9.2° in equations 13 to 15 gives *ff* = 1.26.
8. Determine the major consolidation stress at the intersection of the flow function and flow factor. The major consolidation stress σ_1 is equal to 1.12 kPa.
9. Update δ and ϕ'. From Figure 3b, δ = 45.7°; from equations 4 to 13, ϕ' = 30.7°.
10. Update the recommended mass-flow hopper angle. From equations 2 and 3, θ' = 9.5°.
11. Update H(θ') and flow factor. H(θ') = 2.15 and *ff* = 1.25. Solution has converged.
12. Calculate the critical stress; σ_{crit} = 1.12/1.25 = 0.90 kPa.
13. Calculate the bulk density. From Figure 3c, ρ_b = 804 kg/m^3.
14. Calculate the critical outlet diameter. From Equation 20, B_{min} = (2.15)(900)/[(804)(9.8)] = 0.24 meter

FIGURE 11

Flow function of APAP-MCC-HPC blend with 0.2 percent fumed silica and 1.0 percent magnesium stearate



(10 inches).

The recommended hopper for reliable storage and handling the APAP-MCC-HPC formulation has a 10-inch-diameter outlet and conical walls fabricated from 304 stainless steel with a 2B finish sloped 9 degrees from vertical. Because such a design is impractical, the formulation must be altered to improve its flowability.

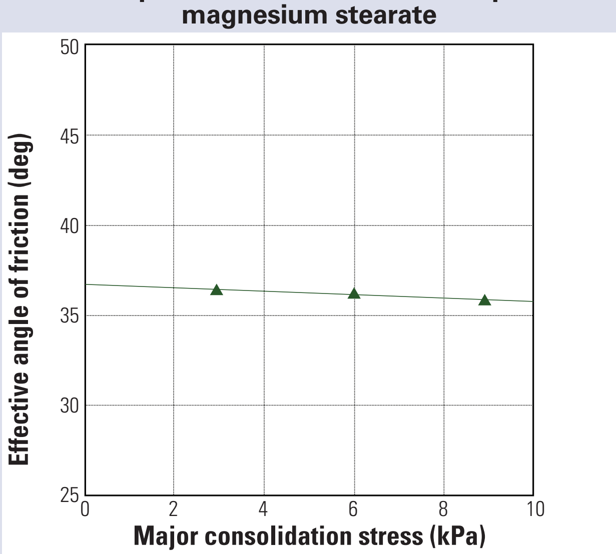
Blends containing fumed silica at levels ranging from 0.25 to 0.75 percent and magnesium stearate levels between 0.5 and 1.0 percent were prepared. Flow property tests performed on the blend showed that an APAP-MCC-HPC blend containing 0.25 percent fumed silica and 1.0 percent magnesium stearate was the least cohesive and had the lowest wall friction. The flow properties of this formulation are shown in Figures 11 to 14. Additionally, the permeability of the formulation was determined to equal 0.0024 meter per second at its loose fill bulk density (652 kg/m^3).

Design of a mass flow bin for this formulation is as follows:

1. Inspection of the material's flow function shows that it will lie below any flow factor if plotted together on the same graph. Therefore, choose a hopper outlet diameter that is appropriate for the downstream equipment. Set B = 2 inches (0.051 m).
2. Estimate the values of the wall friction angle and effective angle of friction. Based on Figures 12 and 14, choose $\delta = 37°$, $\phi' = 20°$.

FIGURE 12

Effective angle of friction of APAP-MCC-HPC blend with 0.2 percent fumed silica and 1.0 percent magnesium stearate



3. Select the mass-flow hopper angle. From equations 2 to 3, $\theta' = 24.3°$.
4. Determine the flow factor. From Equations 15 to 18, $ff = 1.52$.
5. Calculate $H(\theta')$. From Equation 18, $H(\theta') = 2.37$.
6. Calculate the outlet major consolidation stress σ_1 from Equation 19. (The bulk density ρb is determined from Figure 13.) Solving gives $\sigma_1 = 0.21$ kPa and $\rho_b = 664$ kg/m^3.
7. Update the effective angle of friction. From Figure 12, $\delta = 36.7°$.
8. Determine the wall friction angle. From equations 4 to 13, $\phi' = 20.9°$.
9. Update the mass-flow hopper angle. Equations 2 and 3a are solved to give $\theta' = 23.1°$.
10. Update the flow factor and $H(\theta')$. From equations 15 to 18, $ff = 1.56$; $H(\theta') = 2.35$.
11. Update the estimate of the major consolidation stress and bulk density at the hopper outlet. $\sigma_1 = 0.22$ kPa and $\rho_b = 664$ kg/m^3.
12. Update the effective angle of friction and angle of wall friction. $\delta = 36.7°$; $\phi' = 20.6°$.
13. Update the mass-flow hopper angle. $\theta' = 23.5°$.
14. Update the flow factor and $H(\theta')$. $ff = 1.54$. Solution has converged.

A conical hopper with a 2-inch-diameter outlet and fabricated from 304 stainless steel with a 2B finish must have walls sloped 23.5 degrees or steeper to ensure mass flow.

The maximum powder discharge rate will depend on the diameter of the cylinder and the height of the powder inside the cylinder. For a 24-inch- (0.61-m) diameter, 36-inch- (0.91-m) tall cylinder completely filled with the powder, the maximum major consolidation stress in the cylinder is calculated from the Janssen Equation:

σ_1 = (690)(9.8)(0.61)/[(4)(0.4)tan(14°)]{1-exp[-(4)(0.4)(tan(14°)(0.91)/0.61]}/1000 = 4.6 kPa.

From Figure 13, ρ_{bmax} = 712 kg/m^3. From Equation 22, the maximum solids discharge rate is

= (652)π(0.058)2(0.0024)/[4(1-652/712)](3600) = 135 kg/hr

This discharge rate is expected to meet the downstream requirements.

Hoppers with outlets of other sizes can be chosen to meet the process requirements. Obviously, hoppers with smaller outlets will have a lower maximum solids discharge rate. Less apparent is that if a smaller outlet is specified, the hopper walls may need to be steeper to allow mass flow as the angle of wall friction often increases with decreasing outlet size.

Closing remarks

Shear cell testers are frequently used by formulators to assess the flowability of powders. By using the fundamental properties measured by shear cell testers, cohesive strength, effective angle of friction, compressibility, and wall friction, together with permeability measurements, formulations can be readily optimized to ensure that they can be handled reliably in feed hoppers, storage bins, and other vessels. *T&C*

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FIGURE 13

Bulk density of APAP-MCC-HPC blend with 0.2 percent fumed silica and 1.0 percent magnesium stearate

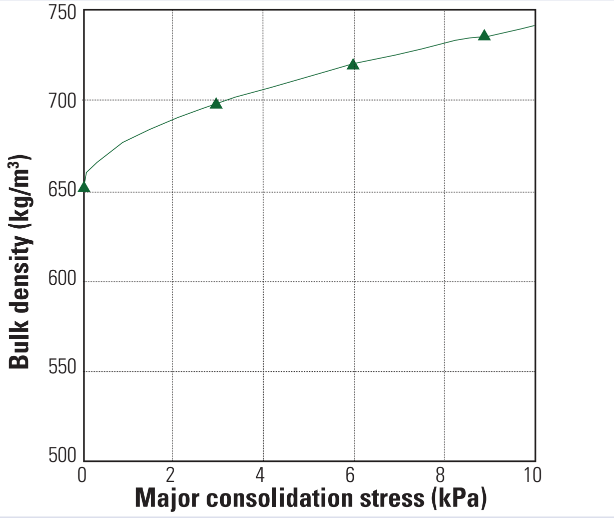
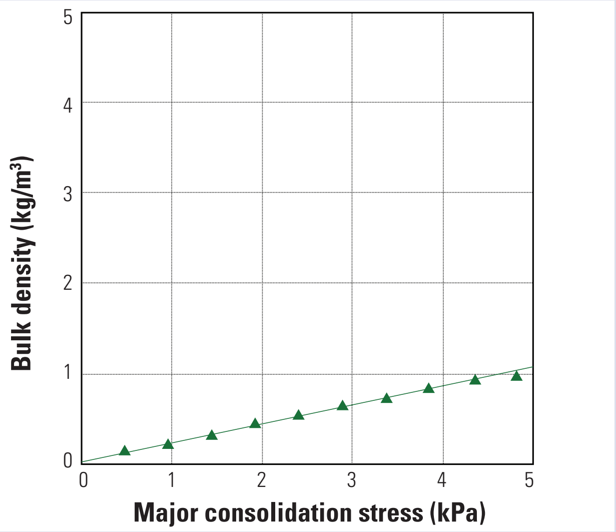


FIGURE 14

Wall friction of APAP-MCC-HPC blend that includes 0.2 percent fumed silica and 1.0 percent magnesium stearate on 304 stainless steel with 2B finish



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