Food Powders Flowability Characterization: Theory, Methods, and Applications

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Key Words

flowability, flow, food powder, physical property, failure, hopper

Abstract

Characterization of food powders flowability is required for predicting powder flow from hoppers in small-scale systems such as vending machines or at the industrial scale from storage silos or bins dispensing into powder mixing systems or packaging machines. This review covers conventional and new methods used to measure flowability in food powders. The method developed by Jenike (1964) for determining hopper outlet diameter and hopper angle has become a standard for the design of bins and is regarded as a standard method to characterize flowability. Moreover, there are a number of shear cells that can be used to determine failure properties defined by Jenike's theory. Other classic methods (compression, angle of repose) and nonconventional methods (Hall flowmeter, Johanson Indicizer, Hosokawa powder tester, tensile strength tester, powder rheometer), used mainly for the characterization of food powder cohesiveness, are described. The effect of some factors preventing flow, such as water content, temperature, time consolidation, particle composition and size distribution, is summarized for the characterization of specific food powders with conventional and other methods. Whereas time-consuming standard methods established for hopper design provide flow properties, there is yet little comparative evidence demonstrating that other rapid methods may provide similar flow prediction.

INTRODUCTION

A large number of food industries currently handle food powders. The wide variety of powders involved in the manufacture of food products range from ingredients, such as flours and spices, to end products such as instant coffee and powdered milk. Particulate ingredients' handling and storage play an important role in most food processes. Part of the design of processing plants involves ensuring the satisfactory flow of materials, which is essential to the profitability of the process. In fact, a study by Merrow (1988) showed that two of the most common problems encountered in solids processing plants occur during the transporting of solids and handling of fines. In particular, problems arise from the difficulty encountered in withdrawing material from a storage hopper without interruption and at the required rate. These problems are caused by failure to incorporate accurate flowability measurements into the design. The end result is frequent stoppage of the process, involving costly loss of production time and inefficient use of staff to restore the flow.

The realm of powder flowability, in a wide sense of the word, has not yet been mastered, although important efforts toward this end have been conducted since the early 1960s. Powder characteristics such as composition, moisture content, particle size distribution, and shape (surface properties) are established by the particle production method, ambient relative humidity and temperature, and storage time, and can powerfully influence flow behavior. Many ideas, methods, and types of testing equipment exist to measure the flowability of bulk solids. Flow characterization is not only relevant for equipment design, storage location of bulk solids, and transporting or otherwise handling of solids, but is also needed to fulfill the requirements of quality control and for modeling processes.

Starting with the term powder, we find that proposed definitions are unclear not only in the food engineering field, but also in soil, metallurgic, pharmaceutical, and polymer engineering. A common convention considers powders to be any group of particulates with an approximate median size less than one millimeter (Peleg 1983, Barbosa-Cánovas et al. 2005). However, in practice, food and other industries often must handle larger particulate materials such as agglomerates or encapsulated particles, in which case the terms coarse and fine are used to roughly describe these materials. Food powders can be irregular in shape and size, and any given powdered product may be composed of several different types of particles. Given these preliminary conditions, the goal of this review is to provide an update on powder flowability characterization methods and factors that prevent food powder flow.

POWDER FLOWABILITY: DEFINITION AND APPLICATIONS

The concept of flow is generally related to fluids. However, under the proper conditions, industries such as the food processing industry can effectively convey solids in a flow-like regime. Powder flow can be defined as the relative movement of a bulk of particles among neighboring particles or along the container wall surface (Peleg 1978).

From a rheological point of view, powder flow has been studied from several different perspectives: as a fluid, as a solid body undergoing elastic/plastic deformation (Sutton 1976), and as the mechanical failure of a solid structure (Jenike 1964). In the first of these approaches, the consideration of powder flowing as a fluid is not a very practical assumption. This approach departs from observed behavior that the flow of powders does not depend on the head of a container (or static pressure generated by the fluid over a container opening). It is based on the fact that the flow rate of powders flowing through an orifice is independent of the head, provided the orifice diameter is at least 2.5 times smaller than the bed height above it (Peleg 1978). However, the two latter approaches, elastic/plastic deformation and mechanical failure, can describe powder flow more accurately and therefore have been widely adopted to explain powder flow.

The deformation and flow of powders under unconfined stress refer to a situation in which the physical configuration of the system allows the powder to flow before massive comminution or deformation of the particles occurs. The first reaction of a powder subjected to a continuous stress is to deform elastically when the particles press against each other, in which case a true particle elastic deformation occurs.

The material (that is, the assemblage of particulates) will return to its original condition if the stress is released at this point. Yet as the stress increases, the interparticle forces increase up to a certain limit, at which time interlocking particles begin to slip or rub against each other, and the powder starts to yield. Once slippage begins, a shear failure plane is established, and the whole structure eventually fails, leading to powder flow. From this point on deformation is completely plastic, the material dilates, and flow occurs by means of slippage—of one layer of particles over the other.

For the purpose of this discussion, only unaided flow will be considered; hence, the force of gravity is the only source of energy promoting flow. Powder flow from fluidized beds and pneumatic conveying escape the scope of this contribution.

FACTORS PREVENTING POWDER FLOW

The flow of powders is a complex phenomenon in which both the powder characteristics and physical and chemical features of the system determine the behavior of the powder flow system. Flowability also depends on the powder's bulk properties (moisture content, density, composition, shape, and particle size distribution), some of which can change as a result of impact during handling, air relative humidity, temperature, and storage time conditions.

Many studies have shown the effects of ambient conditions on the flow properties described above (Peleg et al. 1973; Schubert 1987a,b; Duffy & Puri 1996; Teunou et al. 1999). Even though much has been reported, it is important to consider that reported values are very specific in terms of specific bulk properties of a selected food powder, their experimental conditions, and the specific method and equipment used. In addition to particle size, moisture and powder composition flowability are affected by surface and material properties, particle shape, ruggedness, hardness, and level of surface lubrication owing to the presence of water or fat. However, not much has been reported on the effect of surface properties on food powder flow descriptors.

Table 1 shows some of the more obvious variables affecting powder and system characteristics, which make food powders among the least predictable materials in terms of flowability because of the large number of factors that can significantly change the rheological properties of flowing powder.

Bulk and Interparticle Interactions During Flow

As mentioned, powder particles can stick together and form stable structures, preventing flow. Powder flow can be decreased by mechanical forces related to the shape and size of particles. Some irregularities on the surface of particles can cause physical interlocking of the particles, restricting the slippage required for flow. The method used for particle production (e.g., grinding, spray, drum and freeze drying, agglomeration, milling, mixing, and so forth) has a strong influence on the surface mechanical characteristics. Also, the formation of stable mechanical structures, such

Table 1 Some particle/powder characteristics and factors affecting powder flowability (adapted from Freeman 2000)

Particle properties	Intrinsic factors
Composition (type of material)	Temperature
Density (voidage)	Air relative humidity
Particle size	Compaction level
Particle shape	Coating, agglomeration
Particle roughness	Segregation
Surface friction (coating)	Anticaking agents
Particle compressibility (hardness, elasticity, ductility)	
Moisture	
Electrical properties (conductivity, capacitance, propensity to electrostatic charge)	
Powder properties	**External factors**
Size distribution	Feeding rate
Bulk density	Vibration
Homogeneity (mixture type)	Hopper dimensions and design
Attrition level	Discharge aids
Powder compressibility	
Cohesiveness (powder stickiness)	
Coefficient of internal friction	
Coefficient of wall friction	

as arches above the aperture of silos and containers, is possible, but it depends on the three-dimensional shape and size of particles (Jenike 1964).

All powders possess sufficient strength to form stable heaps or piles and bridges across openings. This strength results from a variety of factors, including van der Waals forces, electrostatics, elastic deformation, plastic deformation, chemical bonding, and mechanical interlocking. In order for solids to flow, gravitational forces must exceed the strength of the powder. During flow, individual particles slide across each other in a shearing action. Thus, the resistance of a powder to flow can be thought of as shear strength, which also can be a measure of flowability.

Physical characteristics that determine the flowability of a given powder include particle surface properties, particle shape and size distribution (**Figure 1**), and the geometry of the system. Particles with irregular fibrous shapes and plate-shaped particles can mechanically interlock. Mechanical interlocking is used to describe the hooking and twisting together of the packed material. With the aid of vibration or pressure, these particles can become mutually orientated and then physically bound.

Flowability can be affected by the amount of free and associated water inside each particle. Other components such as fats, sugars, proteins, and fibers also determine the flowability of a powder. The ability to associate water within a powder bulk mass depends on the structural distribution of these components within each particle. Furthermore, surface properties such as friction, ductility, and interlocking capacity may depend on the powder's composition and structural distribution on the surface.

Lower particle size generally provides lower flowability (Thomson 1997). When particles become smaller, they provide a greater surface area for surface cohesive forces to interact as well as friction to resist flow (Fitzpatrick et al. 2004a). Changes in flowability can be noticeable if particle size reduces at least ten times its original average diameter (Fitzpatrick et al. 2004b). An increase of surface-to-surface ratio becomes greater as the particles become smaller, increasing surface interactions (Peleg & Hollenbach 1984, Griffith 1991).

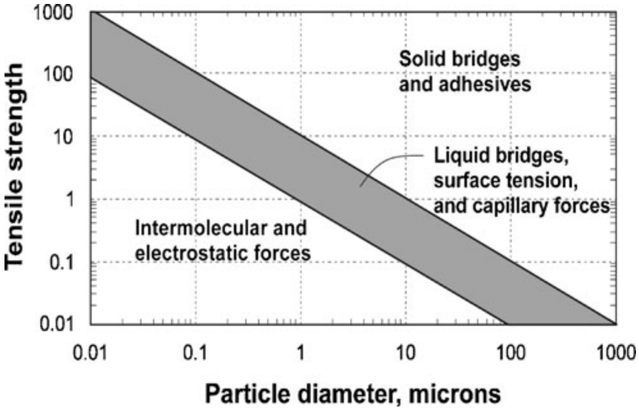


Figure 1

Interparticle strength attributed to intermolecular and electrostatic forces, liquid bridges and capillary forces, and solid bridges (from Barbosa-Cánovas & Juliano 2005).

Particle Forces Affecting Flowability

The forces opposing flow are friction, attraction between particles (cohesion), attraction between the particles and system walls (adhesion), and mechanical resistance or interlocking (that is, formation of stable arches) (Peleg 1978).

During handling and storage, food powders can easily uptake moisture from the air, as the relative humidity of ambient air is generally much higher than the equilibrium relative humidity of most dry food powders (Fitzpatrick et al. 2004b). As a rule of thumb, food powders can be considered as noncohesive only when dry (that is, moisture content below 0.01% wet base) and when particle size is above 100 microns (Peleg 1978). The increase of cohesivity with moisture increase is mainly caused by an increase in the surface tension forces. These forces depend on the presence of liquid (usually water) at the outer surface layer of the particles.

The presence of a liquid can be the result of not only moisture absorption by hygroscopic materials but also melting of some particle components, chemical reactions that liberate liquid, excessive addition of a liquid ingredient, moisture liberation during crystallization, and accidental wetting of powder or equipment (Peleg 1978). Liquid layers on the surface of particles promote cohesion by creating a meniscus between the particles. The more viscous the liquid, the stronger are the cohesive forces.

Food powder caking. Food powders with poor flowability usually, but not always, tend to cake readily. Caking is a deleterious phenomenon by which low-moisture, free-flowing powder is first transformed into lumps, then into an agglomerated solid and ultimately into a sticky material, resulting in loss of functionality and lowered quality (Aguilera et al. 1995). Caking is controlled by several factors, including heat and moisture transfer within the particle bed, heat and moisture transfer to individual particle surfaces, and heat and moisture transfer within the particles themselves. Given that changing ambient conditions can affect each of these transfers differently, the number of possible combinations almost guarantees that caking will be an irregular, unpredictable process.

Caking can occur as a result of recrystallization by surface wetting, followed by moisture equilibration or cooling, or because of electrostatic attraction between particles. The caking mechanism involves bridging, agglomeration, compaction, and liquefaction (Aguilera et al. 1995). Bridging occurs as a result of surface deformation and sticking at the contact point between particles. Agglomeration is a later stage and involves an irreversible consolidation of bridges resulting in particle clumps with structural integrity and larger size (Schubert 1981). When compaction occurs, interparticle spaces are reduced, and deformation of particle clumps under pressure leads to a loss in the system integrity because of thickening of the already formed interparticle bridges. Finally, higher moisture leads to liquefaction, where interparticle bridges disappear, and as a result of the high moisture content, low molecular weight fractions are solubilized. At any given stage, lumps may be few or numerous, of different sizes, and of varying degrees of hardness, providing potential variations for flowability.

In soluble particles, on the order of 100 μm to 1000 μm in size, liquid bridging is by far the most common cause of caking (**Figure 1**). The same applies to fat rich powders (for example, cheeses, soup mixes) exposed to elevated temperatures, at which all or part of the fat melts. Hydrophilic particles are more unlikely to adhere to one another as a result of surface tension in a moist atmosphere. Conversely, hydrophobic particles have good adhesion until very low moisture values are reached, at which point adhesion may decrease sharply due to breaking up of the meniscus (Sutton 1976).

Powders such as instant coffee, powdered tea, powdered cream, and even powdered soup must be easy flowing, especially when vending machines are used (Schumann & Gerhards 1997). The powders must be dispensed in measured quantities and, if caked, the drink or food will otherwise be too concentrated or too diluted when delivered. Other food powders, especially those containing soluble or amorphous components such as sugar, salt, lactose, or lipids, have a very strong tendency to cake when exposed to an atmosphere of high humidity or elevated temperature (Griffith 1991, Roge & Mathlouthi 2003). For example, skim milk and whey permeate cake at 44% and 33% relative humidity (Teunou et al. 1999), respectively. Furthermore, plastic-flow caking can also happen when the particle's yield stress value is exceeded, forcing particles to stick together. Caking usually occurs with amorphous materials such as tars, gels, lipids, waxes, or some soft crystalline substances that stick together when subjected to either pressure or higher temperature.

Glass transition temperature. Glass transition temperature (Tg) is the most representative parameter of a powder's state transformation from minerals and dry polymers (glassy state) to materials in a rubbery state. A dramatic reduction in viscosity (or mechanical modulus) of an amorphous solid occurs at the so-called glass-rubber transition once the glass transition temperature boundary Tg is reached, provoking an increase in the amorphous solid plasticity. Consequently, physicochemical changes occur during this transformation, allowing the flow of liquids, which contributes to the formation of liquid bridges. Temperature and moisture directly influence the structural transformations that occur above the glass transition temperature and are related to increased adhesiveness (Adhikari et al. 2001). Water addition decreases the Tg of amorphous foods. Furthermore, high dextrose-equivalent carbohydrate constituents have the greatest influence on the glass transition temperature of an amorphous dried food material. Common low-molecular weight sugars such as fructose, glucose, and sucrose have very low glass transition temperatures, so their influence on reducing Tg (which can increase their formation of liquid and solid bridges) is very notable in sugar-rich foods.

Anticaking agents. It was previously shown that flow problems are mainly dependent on inter/intraparticular forces, powder particle size, and shape, as well as moisture and fat content. Flow conditioners (or anticaking agents) enhance powder flow by reducing the cohesiveness and compressibility of interparticle forces while increasing bulk density (Peleg & Mannheim 1973). Anticaking agents are finely divided powders that are usually added to the host powder at concentrations on the order of 0.5% to 2%. Generally, anticaking agents are very fine powders (approximately 40 μm to 100 μm) with low bulk density and surface areas of hundreds of square meters per gram. They are also considered chemically inert. The main types of agents are silica and silicates, phosphates, stearates, polysaccharides, and iron salts.

Anticaking agents compete with the host powder for moisture (Peleg & Hollenbach 1984). They act as physical barriers between particles by interfering with liquid bridging (for example, silicon dioxide), by reducing or neutralizing superficial molecular attractive forces (mainly electrostatic), and by inhibiting crystal growth and altering its lattice pattern (Hollenbach & Peleg 1983). Adding high molecular weight carbohydrates as anticaking agents can increase Tg, which increases product stability. For example, the stability of various dairy-based products increases when increasing the concentration of high molecular weight carbohydrates as a result of a rise in the sample's Tg (Aguilera et al. 1995), subsequently changing the food from a rubbery amorphous state to a crystalline state, lowering internal fluid mobility, and preventing bridging. Anticaking agents can also be moisture-protective barriers when applied to the surface of the powder. Stearates in particular can serve as lubricants, thus reducing internal friction (Peleg 1983).

POWDER FLOW PROPERTIES CHARACTERIZATION

Flow of powders, as described above, implies the collapse of a mechanical structure formed by powder particles. The collapse of this structure occurs when the load applied, mainly characterized by gravity force, exceeds the structure's mechanical resistance. The load received by a given powder structure depends on the amount of mass composing the structure, whereas the mechanical resistance of the powder bed basically depends on the cohesive forces holding the mechanical structure together. Thus, generally, noncohesive powders are considered as easy- or free-flowing powders. Flowability is also strongly affected by the state of compaction of the powder, ambient humidity, and temperature.

The situations mentioned above can be characterized with different properties. This section will introduce flowability properties that are used quantitatively for bin storage design, such as angle of internal friction, the flow function, cohesion, and tensile strength. Compressibility, Hausner's ratio, bulk density, angle of repose, and friction, as well as other studied parameters, will shortly be described as qualitative descriptors of flowability (**Table 2**). Conventional, nonconventional, and new methods for measuring quantitative and qualitative flow properties in food powders will also be presented. Special attention will be given to Jenike's method and angle of repose, as explored by different researchers over the years, for their common application in the food industry.

Failure Properties

The conventional basic functions describing the conditions in which a mechanical structure collapses are known as failure properties. These properties include the angle of wall friction, the effective angle of internal friction, flow function, cohesion, and tensile strength (**Table 2**). These failure properties take into account the state of compaction of the powder as this strongly affects its flowability, unless of course the powder is cohesionless, e.g., dry coarse salt, and gains no strength upon compression.

The angle of internal friction (α) is a measure of the interaction between particles. It is calculated from the slope of the plot of shear force against normal load (or stresses) after running the tests with a shear tester.

The angle of wall friction (ϕ) is a measure of the friction between the powder and the walls of the confining system, and is equivalent to the angle of friction except that, during the shear test, one of the surfaces is the confining bin wall. The angle describes the friction and adhesive strength between the powder and the material of construction used to confine the powder (e.g, a hopper wall). This is the simplest powder failure property to test, as it depends very little on the state of consolidation of the powder. The wall friction causes some of the powder weight to be supported by the walls of the hopper. This parameter will help decide the material used in hopper design. It can be used as an index for adhesiveness between the hopper material and the powder, and as complimentary information to characterize the powder's ability to flow. It tends to increase with lower particle size (Thomson 1997).

The effective angle of internal friction (δ) is a measure of the friction between particles that takes into account particle size, shape, roughness, and hardness. For noncohesive powders, the α coincides with the δ. It can be measured indirectly, in shear cells, for example, or directly by the grooved plate method.

The flow function (FF) is a complex function that gives the strength of the cohesive material in the surface of an arch as a function of the stress under which the arch was formed. This function is the most important of any single property, as it can be directly used as a flowability index.

The cohesion (C), as mentioned earlier, is a function of interparticle attraction and is due to the effect of internal forces within the bulk, which tend to prevent planar sliding of one internal particle's surface against another.

The tensile strength (T) of a compact powder represents the minimum force required to cause separation of the bulk structure, producing a plane of failure.

There are several, both direct and indirect, methods of testing the five failure properties defined previously. Basically, all methods can be determined using a shear cell, but simplified or alternative procedures can be adopted when the aim is to monitor the flowability of the output from a process or to compare a number of materials.

Measuring equipment. Several different pieces of laboratory equipment and experimental procedures have been suggested for direct measurement of the failure properties of powders. Jenike's shear cell and the annular shear cell are among the most typical types of test equipment used to characterize powder flowability. Other testers, also tried and compared for food powder measurement, are the direct shear cell, biaxial and triaxial testers and the rotational split-level shear tester commonly used in soil mechanics. To date, Jenike's direct shear cell tester and his proven procedure for designing bins for flow have become benchmarks in industrial practice and research. Kamath et al. (1993) have provided a historical review of the different shear testers created since Jenike's cell, some of which will be described briefly, but special attention will be given to Jenike's shear cell.

Jenike's cell. Among shear cells, Jenike's shear cell (**Figure 2**) is the most common instrument used for flowability evaluation and determination of failure properties (Peleg 1978). The resulting comprehensive theory (Jenike 1964) generated to describe the flow of bulk solids has been applied and perfected over the years, but is truly recognized worldwide as the only scientific guide for evaluating bulk solids flow. Jenike was the first to use the concepts of plastic failure to develop a flow-nonflow criterion for analyzing the flow of solids in bins and hoppers. Jenike's quantitative method is used to design storage bins for the gravity flow of solids and is applied in the food engineering field. The method has been established as an international standard by the American Society for Testing and Material (ASTM International), which has published two standards describing this theory, D6128-00 (ASTM D6128-00) and D6773-02 (ASTM D6773-02).

Table 2 List of terms employed

A	effective cross sectional area of the shear cell (m^2)
b	mechanical compressibility (dimensionless)
C	cohesion or cohesiveness (kPa)
EYL	effective yield locus
F	unconfined yield strength (N)
F_t	unconfined yield strength after time consolidation (N)
f_c	unconfined yield stress (kPa)
ff	ratio of σ_1 and f_c
FF	flow function
h	height of a powder bed (m)
H_R	Hausner ratio (dimensionless)
M	torsional moment (N.m)
m	powder mass (kg)
n	shear index (dimensionless)
T	tensile stress (kPa)
R_o	outer radii of the annular trough (m)
R_i	inner radii of the annular trough (m)
R	Mohr circle radii (kPa)
S	shearing force reached during consolidation (preshear) (N)
$\bar{S}$	shearing reached during shear at selected $\bar{V}$ (N)
V_0	loose bulk volume (m^3)
V_n	bulk volume after n taps (m^3)
V_1	maximum consolidation force (N)
V_2	minor consolidation force (N)
V_t	vertical force applied to a shear cell during twisting (preconsolidation) (N)
V	normal force applied to a shear cell during consolidation (preshear) (N)
$\bar{V}$	selected normal force applied to a shear cell during shear (N)
x	horizontal displacement in a direct shear cell (m)
y	vertical displacement in a direct shear cell (m)
α	angle of internal friction (°)
ϕ	angle of wall friction (°)
ρ	bulk density (kg/m^3)
ρ_p	particle density (kg/m^3)
ρ_0	initial loose bulk density (kg/m^3)
ρ_n	tapped bulk density after n taps (kg/m^3)
ρ_∞	asymptotic density (kg/m^3)
δ	effective angle of internal friction (°)
θ	angle of repose (°)
τ	shear stress (kPa)
σ	vertical stress (kPa)
σ_0	atmospheric stress (100 kPa)
σ_t	vertical stress caused by V_t (kPa)
σ_1	maximum consolidation stress (kPa)
σ_2	minor consolidation stress (kPa)
ω	angular speed (rad/s)
μ	friction coefficient (dimensionless)

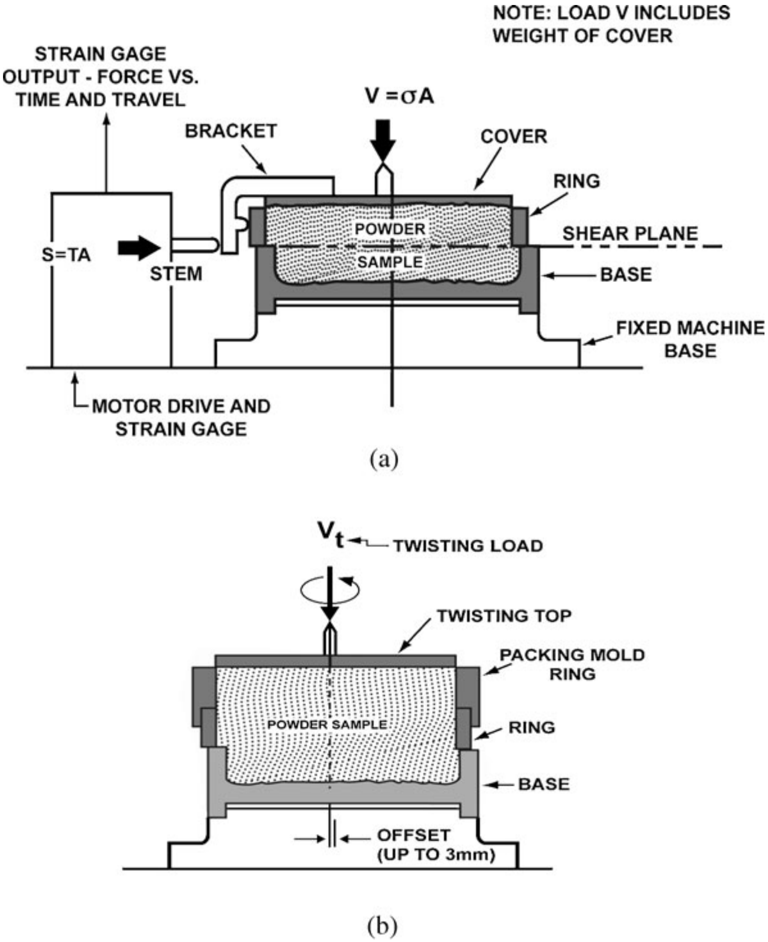


Figure 2

(*a*) Jenike's shear cell and (*b*) the preconsolidation system (from Barbosa-Cánovas et al. 2005).

Furthermore, the Commission of the European Communities (Akers 1992) has certified the use of limestone powder as a standard reference material for Jenike shear testing.

In each shear test, a point describing a pair of normal and shear stresses is obtained, both of which act on a single plane. This point is assumed to lie on the yield locus of the powder (defined later), which represents the region of failure in shear stress of the powder, or the limit of elasticity. The Mohr circle describes the stresses in different directions and is determined tangential to the yield locus. This shear test is, however, only a two-dimensional representation of the stresses within the powder, whereas in practice any powder is subjected to three-dimensional stressing. Three Mohr circles can represent the three-dimensional situation, but it is accepted that only the largest of the three circles is significant in powder flow. Therefore, the so-called translational devices, such as the Jenike or annular shear cells, are able to characterize flowability for applications such as hopper or bin design. However, due to the lack of high compaction forces in the system, they may not be sufficient in predicting performance on powder dosage from small-scale systems such as vending machines.

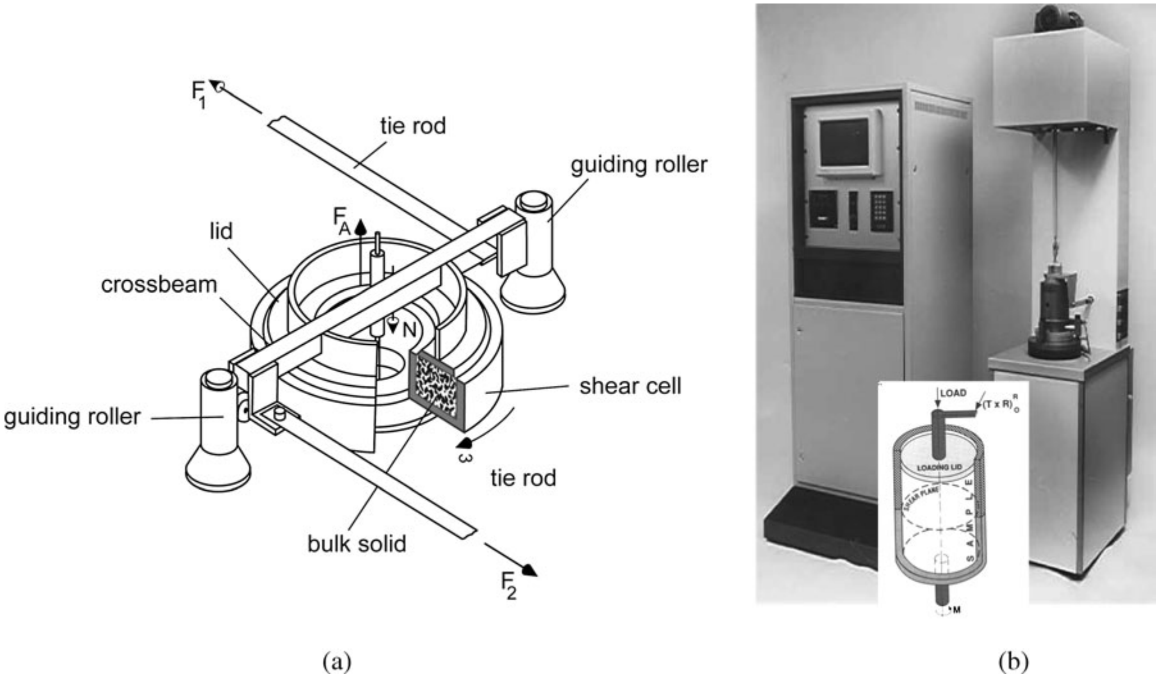


Figure 3

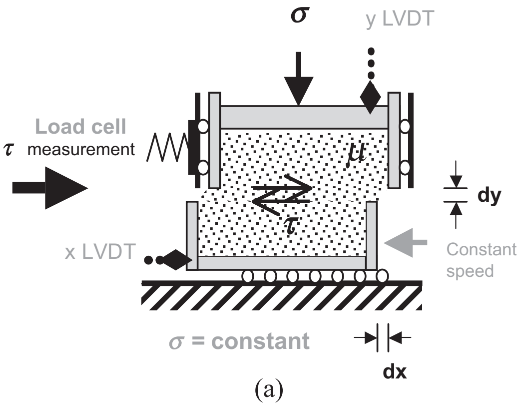
The annular shear cell (*a*) standard diagram (from Barbosa-Cánovas et al. 2005) and (*b*) Peschl shear tester (IPT 2006a, Peschl 1989).

Annular shear cell. Shear force measurements at very low normal stresses are possible by using an annular shear cell (or ring cell), as shown in **Figure 3**. The powder is contained in a shear trough, which rotates at angular speed (ω). The torsional moment (M) is measured at the nonrotating loading ring, which transmits the normal load (V). From M, the shear force and hence the shear stress can be derived by means of the equation:

$$\tau = \frac{3M}{2\pi(R_o^3 - R_i^3)}. \tag{1}$$

This has been confirmed theoretically and experimentally (Gebhard 1981, Ramachandruni & Hoag 2001). The radii R_o and R_i are the outer and inner radii of the annular trough, respectively. The purpose of this equipment is to find accurate shear stress measurements, particularly in the range of extremely low normal stresses. Hence, the annular shear cell is preferred for flowability studies over the Jenike cell (Walker 1967), especially in cases when the steady state (or failure) cannot be achieved during shearing. This device can be connected to a computer and is thus suitable for automatic operation.

Annular shear cells are based on the same principles as Jenike's (Schulze 2000) but can allow coverage of much larger shear distances, in both sample preparation and testing. Furthermore, the area of shear is constant as opposed to Jenike's, which varies in shearing area during tests. The test requires a shorter time for a complete measurement than with Jenike's device. Furthermore, the cell system also allows for time consolidation studies. The ASTM D6682-01 (2001) standard establishes four procedures for measuring the shear stresses of powders as a function of normal stress and determination of internal friction, wall friction, powder bulk density, and powder degradation.



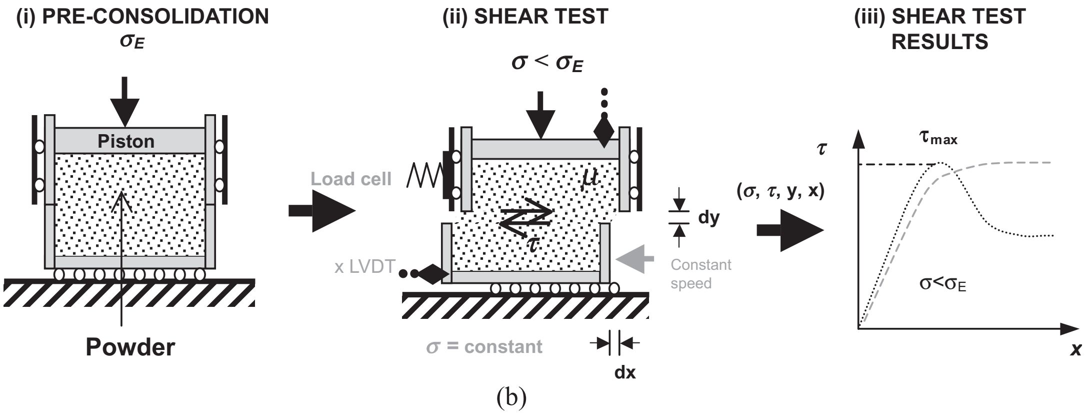


Figure 4

Direct shear cell. (*a*) Schematic diagram (two boxes confining the powder, a horizontal and vertical LVDT, a constant vertical load, a shear force gauge and motor providing constant speed to the lower half of the cell) and (*b*) the shear test procedure (from Juliano et al. 2006).

Direct shear cell. The direct shear cell is an apparatus designed for direct measurement of the shear strength of a consolidated powder material. It consists of a shallow circular or square bracket on which a matching cover of equal dimensions is placed (**Figure 4**). This method is specified as ASTM Standard D 3080 (ASTM D3080-98). Once the cell is filled with the test material, the excess is scraped off with a flat spatula. The lid is then placed on top of the specimen and a weight placed immediately on the lid. Using a rack and a pinion arrangement driven by a dynamometer at constant speed, a shear force is applied at the top half of the cell until the shear force becomes constant.

Other models displace the lower ring at constant velocity. The upper ring is connected to a piston located opposite to the movement direction that measures the resistance in shear of the consolidated powder. The vertical and horizontal displacements of the cells are controlled by two linear variable displacement transducer (or LVDT) systems.

Biaxial and triaxial testers. As mentioned before, enough information can be obtained in terms of flowability by geometrically quantifying parameters determined from the principal shear stress or failure plane (that is, the largest stress selected within the three Mohr circles that correspond to each of three possible stressing dimensions). Different researchers have proven this assumption by using many different versions of biaxial or triaxial testers (Eelkman & Haaker 1977). Most versions have given similar or identical yield loci compared with those measured with the Jenike or annular shear cells.

The principle of these testers is that the specimen can be subjected to controlled stresses in two orthogonal directions (biaxial testers), or three orthogonal directions (triaxial testers). In the case of the triaxial testers, two of the orthogonal stresses are usually equal, normally generated by liquid pressure in a pressure chamber. The specimen is isotropically consolidated by the same pressure in all three directions, which leads to volumetric strain but little or no shear strain. This is followed by anisotropic stress conditions, wherein an axial stress greater than the other orthogonal stresses is transferred to the specimen by mechanical force through the end cups. In the evaluation of results, it is assumed that the principal stresses act on horizontal and vertical planes, and Mohr circles can easily be drawn for the failure conditions. Many different modifications of the triaxial (or biaxial) tester have been proposed or developed by various researchers for powders, but no universal or standard procedure or equipment yet exists.

The triaxial test, although still only a research tool, is probably the best means for testing powder strength and stress-strain characteristics, owing to its ability to measure strains and volume changes and to apply stresses in all three directions. The flexible boundary cubical triaxial tester is one type of triaxial tester (Kamath 1996, Li & Puri 1996). It allows not only the three principal stresses to be applied independently, but also volumetric deformation and constant monitoring of deformations in three principal directions. By doing a triaxial compression test, the specimen is started at an initial isotropic state of stress. Then the same pressure is applied along the three pressure lines at the same rate to all six faces; thus, the pressure is the same (that is, $\sigma_1 = \sigma_2 = \sigma_3$) in all three principal directions. Research has shown that the cubical triaxial tester is very useful for investigating anisotropy of cohesive and noncohesive powders as well as the effects of particle shape and sample deposition methods. Wheat flour was used for the calibration of the triaxial tester in order to determine parameters related to hopper design (Kamath 1996).

Determination of failure properties. Testing shear cells requires that the powder samples are uniform and representative of field conditions. Also, ambient moisture and temperature are recommended to be controlled to ensure reproducibility of the tests. Jenike or annular shear cells require as a first step in conducting a flow test to preconsolidate the sample to a predetermined consolidation level. The consolidation of a sample is carried out in two stages. The first stage is called preconsolidation, and its purpose is to prepare a uniform specimen. With the cover off the test cell, a packing mold is placed on top of the ring, and the mold and ring are placed in an offset position on the base, as shown in **Figure 2**. A sample of the tested solid is then placed in the cell, layer after layer, until the sample is slightly packed in and up to the top of the mold. The excess material is scraped off, level with the top of the mold. A twisting top is placed over the solid. A vertical force (V_t) is applied to the top by means of a system of levers and attached weights. V_t causes a vertical pressure (σ_t) in the material. By means of a special wrench, a number of oscillating twists are then applied to the cover to assure the initial level of compaction is reached. Hence, this first procedure preconsolidates the solid and assures a uniform specimen.

Consolidation is completed in the second stage by forcing the specimen to flow under given stresses until a steady state is reached or closely approached. This is attained in the following way: the load V_t is taken off, the twisting top and the mold are removed, any excess material is scraped off level with the top of the ring, and the test cover is placed on the material. A load (V) is now positioned as shown in **Figure 2*a***.

Then, a stage called preshear takes place, which consists of preparing a critically consolidated specimen so as to develop a shear zone in which steady-state flow occurs. In this step, the stem of the shearing device is pressed against the bracket at a constant strain rate. The shearing force (S), applied on the stem to move the upper half of the ring with respect to the bottom half fixed on the base, is measured during the test. As shearing proceeds, a condition is reached where a layer of the solid is forced to flow plastically, or yield, across the whole specimen, at which point the recorded shearing force reaches a steady value (S). Consolidation conditions (V, S) determine the limit test conditions for the actual shear test (**Figure 5**).

When preshear is completed, the stem of the shearing force device is retracted. A smaller load ($\bar{V}$) replaces the vertical compacting load (V), and the corresponding shearing force needed to produce shearing failure is determined ($\bar{V}$, $\bar{S}$). The shearing force is applied until a failure plane has developed to identify a specific point in the yield locus, as explained in the following paragraphs. This fact is indicated on the recorder by the force $\bar{S}$ passing a maximum value.

After shearing, the plane of failure of the specimen is checked. The plane of failure should roughly coincide with the plane of shear of the cell, that is, if the planes deviate sufficiently, it means that the measured point ($\bar{V}$, $\bar{S}$) does not belong to the yield locus and cannot be used in the calculations. Hence, the test must be repeated. Tests are not repeated on sheared particles, as the particles may be damaged or segregated after extensive shearing, and the original sample may not have been a true representation of the source material.

Yield locus definition. Failure properties can be determined by means of the construction of a yield locus (**Figure 5**). In order to construct this graph, the measurement of three or more points on the locus, $(\bar{V}, \bar{S})_1$, $(\bar{V}, \bar{S})_2$, and $(\bar{V}, \bar{S})_3$, is required. For each point, the specimen is first consolidated to the same initial consolidation level (V, S), and then sheared under a smaller load. A common practice consists of shearing the first specimen at a normal load, which is half the consolidation load. Second and third specimens are sheared at normal loads equal to one-third and two-thirds of the consolidation load, respectively. The location of the unconfined yield force, the Mohr semicircle, is estimated by drawing a semicircle through the graph's origin tangentially to the yield locus. The yield locus is then extrapolated toward the higher values of V and S, and a Mohr stress semicircle is drawn through point (V, S) tangentially to the yield locus.

The point of tangency (E) locates the end of the yield locus. From this point on shear failure is replaced by consolidation failure. Every yield locus has an individual termination E, at which point steady-state flow is reached, that is, no further changes in stress or volume take place (Schubert 1987a). The point of intersection of the stress semicircle with the V axis determines the value of the major consolidating force V_1, whereas the point of intersection of the unconfined yield force, the Mohr semicircle, with the V axis determines the unconfined yield force F. The Jenike shear cell cannot measure the unconfined yield strength of a specimen directly. It can be determined only by the Mohr circle evaluation of the yield locus.

Yield locus dependency with moisture and bulk properties. Pierrat et al. (1998) studied the effect of moisture on the yield locus of granular noncohesive materials and derived a theory for the shift in yield locus of a dry material when it acquires moisture. The yield locus of a wet powder was found to be parallel to the dry yield locus and shifted to the left by a value depending on the tensile strength of the wet powder and the angle of internal friction of the material.

In fact, cohesion and angle of internal friction were found to increase with the moisture content of selected alimentary powders (Teunou et al. 1995, Duffy & Puri (1996), Chang et al. 1998, Teunou & Fitzpatrick 1999). Buma (1971) and Rennie et al. (1999) found a sharp increase in

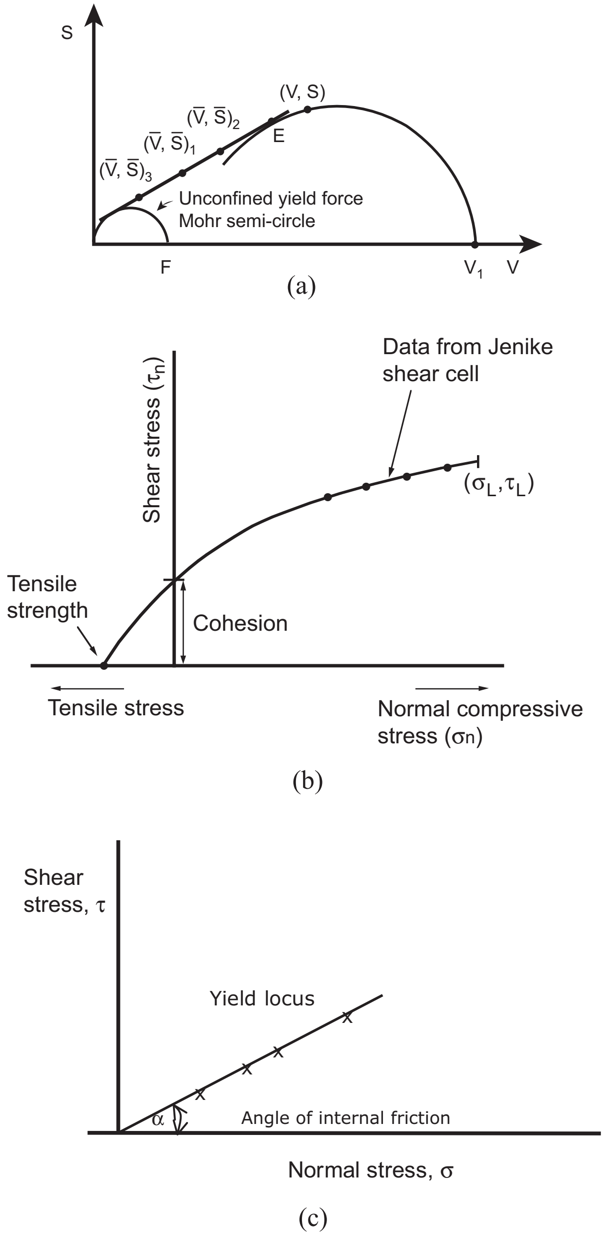


Figure 5

Construction of (*a*) a yield locus and (*b*) the shear locus for a cohesive powder and (*c*) a free flowing powder. (Adapted from Barbosa-Cánovas et al. 2005.)

cohesion of whole milk powder (determined by an unconfined yield test) when moisture content reached 6% or at higher moisture contents. Duffy & Puri (1996) found that if moisture content is increased 3% in a carbohydrate powder, such as confectionery sugar (initially 0.3% in moisture), a 14% increase in cohesion and 59% decrease in internal angle of friction are observed.

Teunou et al. (1999) found a significant combined synergistic effect owing to relative humidity and temperature in the cohesion and angle of internal friction of flour after storage at temperatures ranging between 5°C and 40°C.

Shinohara et al. (2000) found that the angle of internal friction of noncohesive powders, determined in a triaxial compression test, increased with particle shape angularity. Increased angularity decreases the initial voidage (increased density) and increases the interlocking effect.

Warren Spring equation. Provided that the yield locus is not linear for all powders, an equation suggested by Ashton et al. (1965), known as the Warren Spring equation, has been established to mathematically describe the yield locus of both cohesive and noncohesive powders. The Warren Spring equation is defined as:

$$\left(\frac{\tau}{C}\right)^n = \frac{\sigma + T}{T} \tag{2}$$

where τ is the shear stress, σ is the normal stress, C is the cohesion, T is the tensile stress, and n is the shear index.

Teunou et al. (1999) showed that the shear index increases as consolidating stress increases from shear tests in flour, powdered milk, instant tea, and whey. They also found that cohesion and tensile stress change with the consolidating stress. Therefore, flow will depend on the consolidating stress, that is, on the porosity or level of compaction the powder will have. The height of the hopper will influence powder consolidation and, consequently, its flow. As a result, it becomes fundamental to approximate the normal stresses a powder will be subjected to during storage in a bin before performing any test (Teunou & Fitzpatrick 2000). A rough approximation of the consolidation stresses or the normal stress range used during the test can be determined via the following equation:

$$\sigma = \rho_p g h \tag{3}$$

where σ (Pa) is the normal stress, ρ_p (kg/m^3) is the particle density, and h (m) is the height of powder bed in the bin.

Effective yield locus and parameter determination. A straight line passing through the origin and tangent to the Mohr stress semicircle is known as the effective yield locus (EYL) (**Figure 6**). The angle of the EYL line with respect to the normal force axis represents the effective angle of internal friction (δ).

The intersection of the yield locus with the shear stress axis gives the value of cohesion (C), which will be a function of the powder's properties and of the preconsolidation level (**Figure 6**). For noncohesive powders, $C = 0$. Given that C depends not only on the powder's properties, but also a lot on the consolidation conditions, C is less suitable as a measurement of flowability, compared with the *FF*. The tensile strength is the normal stress required for failure of the compact at zero shear stress (negative stress corresponds to tensile stress).

The final and most useful property of powders that can be derived from the yield locus is the *FF*. Jenike (1964) showed that the more easily a powder flows out of a bin, the greater the major consolidation stress (force V_1 or stress $\sigma_1 = V_1/A$) becomes relative to the unconfined yield strength (force F or stress $f_c = F/A$).

Family of yield loci and flow function. Constructing the yield locus for several initial consolidation levels of the same powder gives a family of yield loci that can be used to describe the behavior of the powder under a wide range of pressures at several different bulk densities.

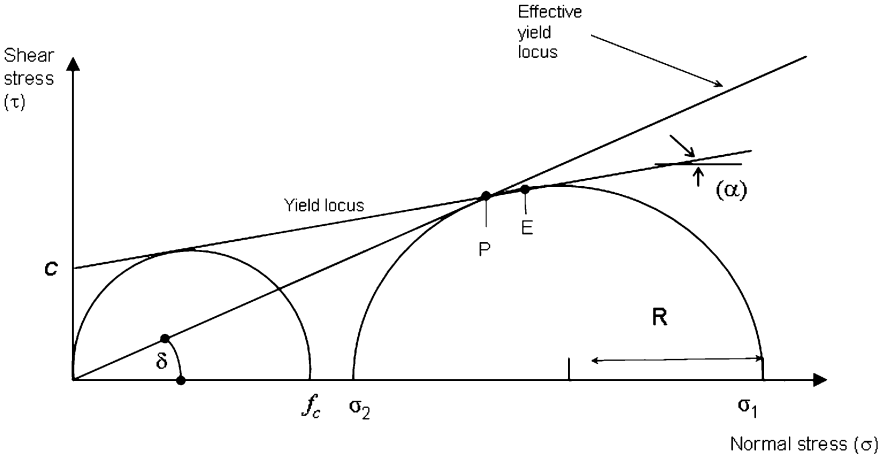


Figure 6

Flow parameter determination. *E* is the failure point necessary to make the powder flow, *P* is the tangency point of the effective yield locus in the major Mohr circle, *C* is the powder's cohesion, *F* is the unconfined yield strength, σ_1 is the maximum consolidation stress, δ is the effective angle of internal friction, α is the angle of internal friction, and *R* is the Mohr circle radii (adapted from Thomson 1997).

From the family of yield loci, pairs of unconfined yield strength (f_c) and maximum consolidation stress (σ_1) values can be obtained. An *FF*, which characterizes the flowability of a powder, can then be obtained by plotting f_c against σ_1. Different curves obtained are useful for characterizing a food powder as a function of its composition, particle size, and other environmental factors that might influence the powder under study over a given range of consolidation stresses. **Figure 7** shows different flow functions for different powders. In his work, Jenike (1964) concluded that

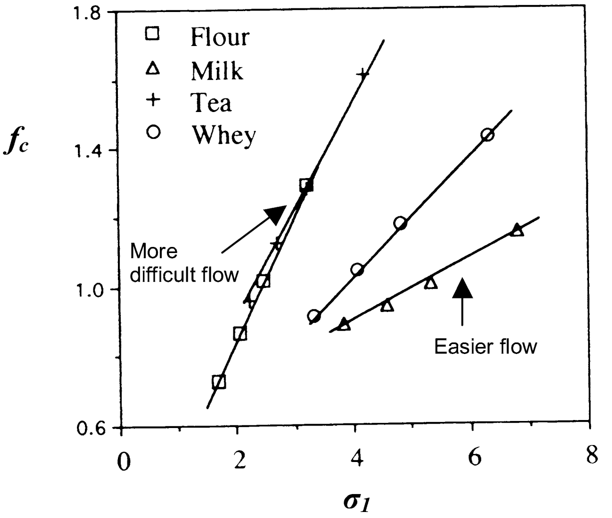


Figure 7

Instantaneous flow functions (unconfined yield strength against maximum consolidating stress) of flour, milk, tea, and whey (adapted from Teunou et al. 1999).

Table 3 Flow function index *i* (Teunou et al. 1999) and compressibility C_2 using the modified uniaxial compression test method (Ehlermann & Schubert 1987) and adapted to characterize flowability

Flowability	Flow index, *i* (ff_c)	Compressibility, C_2
Non-flowing	<2	>0.02
Cohesive	<4	>0.06
Easy-flowing	<10	>0.10
Free-flowing	>10	n/a

the more easily a powder flows out of a bin, the greater the σ_1 becomes relative to the f_c, which corresponds to a large index value.

Other authors in the chemical and food engineering fields (Thomson 1997, Rao 1992, Peleg 1978, Schubert 1987a, Jenike 1964) used the term flow function to define the following equation:

$$ff = \frac{V_1}{F} = \frac{\sigma_1}{f_c}. \tag{4}$$

This equation gives the ratio of maximum consolidation stress to the corresponding unconfined yield stress for a single pair of data values. This ratio has been a useful and quick guide to determine the flowability of powders. It does not, however, completely describe the flow behavior of a powder; still the most accurate way of flowability determination is by studying the flow index from the flow function plot (Schubert 1987a).

The flowability of powders may be classified according to the flow index (ff_c) (inverse slope of the flow function at the selected range of normal stresses), as shown in **Table 3**. Given that both consolidation stress and unconfined yield strength depend on specific conditions affecting the powders (that is, initial bulk density, humidity, or temperature), it is necessary to describe the specific field conditions every time flowability is to be determined from a yield locus (Fitzpatrick et al. 2005). For example, Teunou et al. (1999) found that the unconfined yield strength increased with the moisture content of flour and whey permeate powders. Furthermore, the unconfined yield strength for powdered tea and whey permeate increased with increasing temperature. Buma (1971) found that the unconfined yield strength of four different whole milk powders increased with temperature increase from 5°C to 20°C.

If difficulty is found in determining the yield locus in the region where a Mohr circle is plotted to determine f_c, tensile strength may be used to interpolate the yield locus by using the Warren Spring equation, hence, improving the measurement accuracy of f_c (Schubert 1987a). This is of particular importance when slightly cohesive powders, such as instant food powders, are investigated.

Friction for free-flowing powders and effective yield locus. It has been proven that particle surface friction plays a major role in the interaction between free-flowing powders. In fact, the yield locus for free-flowing, noncohesive powders forms a straight line that passes through the origin (**Figure 5**). As long as the powder is free-flowing, the major obstruction to flow is the internal friction; therefore, flow will only occur when:

$$\tau > \mu\sigma,$$

where τ is the shear stress, μ is the friction coefficient ($\mu = \tan\alpha = \tan\delta$), α is the angle of internal friction, and σ is the normal stress. For free-flowing solids, the angle of internal friction

(α) equals the effective angle of internal friction (δ), and the yield locus coincides with the effective yield locus (EYL).

Soil Mechanics Theory Applied to Flowability Characterization

A theory born in soil mechanics has been proposed to explain the concept of maximum consolidation state that gives a mechanical alternative to the theory of cohesion (Schofield 1998). When shearing a powder under a given consolidating stress (σ = constant), as already mentioned, the peak strength or the asymptotic shear force value appears to be due to the yielding between particles (**Figure 4**). This peak strength or asymptotic flow value is caused by two components: friction (μ) and interlocking. Frictional resistance occurs as particles slip during shear distortion, and interlocking of particles causes the volume to increase during shear distortion. As shown in **Figure 4**, the powder is sheared at a distance x by a shear force τ, whereas the piston is lifted during shear by a distance y in the direction of the normal stress. At peak strength, during cell displacement, the quantity dy/dx (**Figure 4**) indicates the rate of maximum interlocking in the shear zone.

The rate at which work is done at peak strength can be calculated from the work of friction plus the work of interlocking (Schofield 1998).

$$\tau dx = \mu\sigma dx + \sigma dy \tag{5}$$

(peak strength = friction + interlocking).

Equation 5 can be transformed into the following expression, dividing by σdx:

$$\frac{\tau}{\sigma} = \mu + \frac{dy}{dx}. \tag{6}$$

Friction (μ) is a property inherent to the material and therefore could be used as a flowability index. Juliano et al. (2006) proposed the friction-based index μ (Equation 6) as a means to characterize food powder strength during flow and shear. They determined the friction value μ for commercial powdered salt, sugar, cocoa, and cheese and compared this data with different bulk and flow properties such as angle of repose, cohesion, angle of internal friction, and unconfined yield stress.

Measurements of friction values showed better repeatability than the angle of repose technique, whereas lower angles of internal friction found in cohesive cocoa and cheese powders agreed with lower friction values obtained for these powders. This indicated the prevalence of interlocking and frictional forces at peak failure in comparison to adhesive and other attraction forces of lower magnitude. Tests performed in Washington State University soil and food engineering laboratories indicated that this theory works for dry free-flowing powders such as sugar and salt. However, more research is needed to understand the elastic, semisolid, and nonbrittle deformable food materials in relation to this theory.

Another advantage of this theory is that it allows determination of the effective angle of internal friction δ without the use of Mohr circles by assuming $\mu = \tan(\delta)$.

Friction can be used to calculate the flow function from the yield locus. By approximating the yield locus to a straight line, as shown in **Figure 6**, it follows that:

$$\tau = \tan(\alpha) \cdot \sigma + C \tag{7}$$

where τ is the shear stress, α is the angle of internal friction, σ is the normal stress, and C is the cohesion. The effective yield locus applies to the following equation:

$$\tau = \tan(\delta) \cdot \sigma. \tag{8}$$

In this case, friction calculated from Equation 6, using the data obtained from the tests and transformed into the effective angle of internal friction [$\mu = \tan(\delta)$], can be used to determine Equation 10. Therefore, the effective yield locus can be plotted, and point P can designate its intersection with the yield locus. The maximum consolidating strength V_1 can be then determined from drawing the major Mohr circle tangent to the yield locus at point P, and passing through the corresponding preconsolidation point E, as defined before. In soil mechanics, point P is called critical state point (**Figure 6**). The major Mohr circle can be drawn as tangent to point P with its center in the normal stress σ axis.

The ratio of the maximum consolidating strength σ_1 and the minor consolidating strength σ_2 (as shown in **Figure 6**) for the Mohr circle obtained at point E during steady flow can be expressed by the effective yield locus:

$$\frac{\sigma_1}{\sigma_2} = \frac{1 + \sin(\delta)}{1 - \sin(\delta)} \tag{9}$$

From geometrical calculations, the following equations can be obtained for the major consolidation strength σ_1 and the unconfined yield stress f_c:

$$\sigma_1 = 2R \cdot \frac{1 - \sin(\delta)}{\sin(\delta)} \tag{10}$$

where

$$R = \frac{C}{\left(1 - \frac{tg\alpha}{tg\delta}\right) \cdot \cos\alpha} \tag{11}$$

where δ is the effective angle of internal friction calculated from the friction value obtained in Equation 6, α is the angle of friction, and C is the cohesion. The unconfined yield stress can be found as explained by Juliano et al. (2006):

$$f_c = \frac{2.C.\cos(\alpha)}{1 - \sin(\alpha)}. \tag{12}$$

Thus, the flow function ratio (Equation 4) can be directly calculated from the yield locus after testing, without obtaining the fc from the Warren Spring equation or by tensile measurement. This method allows the determination of an effective yield locus with friction μ, avoiding the ambiguity in selecting the preshear normal stress (point E, **Figure 6**).

Angle of Repose

The static angle of repose is defined as the angle at which a material will rest on a stationary heap or the angle θ formed by the heap slope and the horizontal when the powder is dropped on a platform (**Figure 8**). Bulk solids such as cereals, dried milk, flour, salts, sugars, and so on, when transported, treated, or stocked, can form a stable heap or pile due to internal forces. The most obvious characteristic of this heap is the angle of repose. Depending on the environmental conditions at which the pile has been poured and how the angle is measured, different values of the angle can be obtained for the same powder, not being an intrinsic characteristic of the product. The measuring methods used as well as experimental parameters influence the results. Thus, published values of the angle of repose are not always comparable. Several types of angles of repose are used to assess food powder flowability in an empirical manner. Teunou et al. (1995) reviewed a list of common measuring methods for the angle of repose as well as other applications, which was published in the literature.

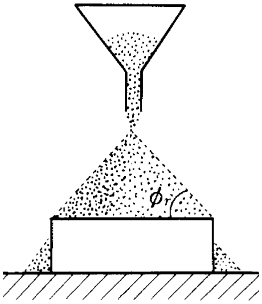


Figure 8

Static angle of repose method (adapted from Barbosa-Cánovas et al. 2005).

The angle of repose may be less accurate for predicting flowability of cohesive powders. Bell (1993) noted that difficulties are common due to cohesive forces. Consequently, a collection of a large number of data points is recommended for characterizing a single sample. Moreover, the angle of repose is not an accurate test to apply in the design of silos, when cohesive powders are under high stress, because the angle of repose does not represent how the strength varies with its state of compaction.

Juliano et al. (2006) showed that the angle of internal friction of crystalline amorphous salt and sugar coincided with the angle of repose, whereas different values were found for cohesive cocoa and cheese powders. In cohesive powders, the angle of repose was much higher, not only because of the presence of cohesive forces, but also because of differences in surface frictional characteristics.

The angle of slide is closely related to the drained angle of repose (Wilkinson et al. 1983). The angle of slide is the minimum angle of incline (measured relative to the horizontal) that will allow a bulk solid to flow out from under its own weight. This angle is presumed to be useful in designing stationary chutes, but its measurement has not been designated as standard. The value measured is thought to be highly influenced by the construction material of the chute, the amount of material used, and humidity. Augenstein & Hogg (1978) have shown that the motion of individual particles strongly depends on the nature of the surface over which they flow: highly roughened surfaces cause shear within the flowing stream, whereas smooth ones cause slip at the surface.

Compression and Compaction Methods for Flowability Characterization

Quite often the testing of bulk solids is not done for silo design purposes, but instead to study the influence of anticaking agents during flow, to check if environmental conditions influence the time-temperature-moisture history of the product, to set the specifications of a certain bulk powder for use as raw material (or ingredient) by another food processor, or to establish the statistical control limits of flow for the continuous production of a certain powder. For solving these problems, estimates of the flow function are sufficient, as long as the test procedure does not change from test to test. Annular shear cells and uniaxial testers are instruments that can easily be automated and give reproducible results although their sensitivity may vary according to the tested powder. Indirect evaluation of flow properties has also been attempted by measuring compressibility of bulk solids using uniaxial compression testers (Ehlermann & Schubert 1987), by vibration (tapping) using the Hausner ratio (Grey & Beddow 1969), and by using the rotational viscometer (Benarieh 1961).

For preliminary studies and comparative measurements, simpler methods and equipment may be preferable. Extensive works have been focused on bulk properties (such as bulk density), the Hausner ratio, and compressibility (Peleg et al. 1973; Moreyra & Peleg 1980, 1981; Baker et al. 1980; Scoville & Peleg 1981; Mannheim & Passy 1982; Passy & Mannheim 1982; Hollenbach et al. 1982; Peleg et al. 1982; Hollenbach & Peleg 1983; Malavé-López et al. 1985; Lai et al. 1986; Barbosa-Cánovas et al. 1987; Ehlermann & Schubert 1987; Molina et al. 1990; Konstance et al. 1995; Murthy & Bhattacharya 1998; Gerhards et al. 1998; Cenkowski et al. 2000; Domian & Milczarski 2003; Barbosa-Cánovas & Juliano 2005). These works have dealt with coffee, coffee creamer, milk powders, baby formula, soup mixes, sugar, gelatin, and salt, among other powders.

Bulk density. Food powders have irregular particle shapes, and the effects of friction or cohesive forces are significant. The existence or stability of a food powder's unconfined array of particles depends on the availability of mechanical forces supporting the open structure, such as friction, cohesion, and particle shape. Therefore, as a result of excessive variability inherent in the system, bulk density alone cannot predict the flowability of powders.

Hausner ratio. Subjecting a powder to vibration (tapping) or impact usually results in its compaction. During vibration, the compact density usually approaches an asymptotic value determined by the nature of the motion, that is, the vibration's amplitude and frequency, impact intensity, or drop height, and so forth. The amount of bulk density change owing to compaction may be an index to the presence of attractive forces and friction. The advantage of this method is the simplicity of the instrumentation (Sone 1972) and the test performance. It should be mentioned that the experimental results may depend to a large extent on the test procedure (for example, number of taps delivered) and such factors as particle size.

Malavé-López et al. (1985) defined the Hausner ratio according to Sone's model, as the relation between asymptotic over initial bulk density by:

$$H_R = \frac{\rho_\infty}{\rho_0} \tag{13}$$

where H_R is the Hausner ratio, ρ_∞ is the asymptotic constant density after a certain number of taps (or vertical motions), and ρ_0 is the initial bulk density. A more practical equation widely used to evaluate flow properties follows that calculates powder volume changes in a graduated cylinder after a certain period of time or number of taps (Hayes 1987):

$$H_R = \frac{\rho_n}{\rho_0} = \frac{V_0}{V_n} \tag{14}$$

where n is the number of taps administered to the sample, ρ_n and ρ_0 are the tapped and loose (initial) bulk densities, and V_0 and V_n are the loose and tapped volumes, respectively. Calculation of the Hausner ratio and evaluation of the flowability become simple when the loose and tapped volumes of the test material are known. Hayes (1987) created a classification for powder flowability similar to the one made by Jenike for the flow function. If the Hausner ratio is 1.0–1.1, the powder is classified as free-flowing; 1.1–1.25, medium flowing; 1.25–1.4, difficult flowing; and >1.4, very difficult flowing. However, if a quantitative approach is needed, the Hausner ratio may not be a reliable flowability index for most food powders (Peleg 1978). Moreover, evidence indicates that compaction patterns of food powders, including the asymptotic density, can strongly depend on the vibration/impact regime. The Hausner ratio can therefore vary dramatically, and it should not be considered as having a typical or unique value. The purpose of food powder compaction tests, therefore, is primarily to simulate density changes during handling and transportation, rather than to assess flowability.

Chang et al. (1998) evaluated tapped bulk density and the Hausner ratio of different mixtures of a starchy powder (potato starch) and proteinaceous powder (wheat protein) at different water activities. As the water activity of each mixture increased, values for these bulk flow properties increased. However, loose bulk densities were found to decrease at increasing water activities. The bulk properties evaluated fitted a quadratic model as a function of water activities with large correlation coefficients.

Compressibility. The confined uniaxial compression test involves confining a bed of powder in a cylindrical cell and measuring the force applied to a flat punch suspended between locking screws, when in contact with the top surface of the bed, as a function of the displacement of the punch. The determinations are made by applying different vertical loads to a bulk solid sample of known mass and recording the compression of the sample with a dial indicator, a scale, or some other electronic position-indicating device (Thomson 1997). The changes in bulk density or porosity of the powder bed versus the compression load are usually expressed by mathematical functions.

By compressing a powder bed and following the force-displacement curve, the change in bulk density can be attained. With these data, powder contact volume versus compressive force or stress can be represented. Bulk density of a solid is a function of consolidation stress and, during flow, it changes as the stress changes. Mechanical compressibility can be determined by examining the logarithm of ρ versus the logarithm of σ. Under low compressive loads, which may exist during powder storage, the relationship between bulk density and stress usually obeys an empirical logarithmic or semilogarithmic relationship (Schubert 1987a, Thomson 1997), as for example,

$$\log\left[\frac{\rho(\sigma)}{\rho_0}\right] = b \times \log\left[\frac{\sigma}{\sigma_0}\right] \tag{15}$$

where $\rho(\sigma)$ and ρ_0 are the compact and loose density, respectively, and σ and σ_0 are the normal stress and atmosphere pressure, respectively. The constant b can be used as a measure of the powder's mechanical compressibility. The more compressible a material is, the more flowable it will be (Carr 1965).

Peleg (1978) mentioned that the characteristic constant b in Equation 15 gives only an approximate measure of the flowability of powders. The relationship between compressibility and yield loci tests was studied on limestone, powdered sugar, semolina, and flours at different particle sizes, size distributions, and moisture contents. It was determined that the compression test may prove to be a convenient method for process control in the laboratory. For instance, the compression behavior of food powders in the low-pressure range may be used to evaluate cohesion. Furthermore, there is some correlation between compressibility and cohesion C, as shown by Schubert (1987a). **Table 3** offers a range of values to classify flowability by comparing flow function index ff_c with compressibility in a selected food powder (Ehlermann & Schubert 1987).

Alternative and Novel Methods to Measure Flowability

There are a number of novel methods that have been developed to describe powder flowability such as the Hall Flowmeter, the Johanson Indicizer, and the Postec Uniaxial. However, none or little has been published of their application in food powders. Others have developed methods to characterize specific food powders such as milk, which may be applied in other food powders.

Hall Flowmeter®. The Hall Flowmeter has been a useful device that is directly relevant in determining flow rate. The method has been standardized by the International Organization for

Standardization (ISO 4490) and described in the ASTM B213-03 (2003). The measurement is relatively reproducible, and the tester is inexpensive. In the test, a funnel of prescribed dimensions is filled with a powder, and the time necessary for the powder to drain out of the funnel is measured. The test equipment allows comparisons between test results that are independent of the funnel orifice size. As with the angle of repose method, the results of the test are affected by particle size and shape, and the user must perform various replicate measurements. This flowmeter cannot be used with powders too cohesive to flow from the outlet or with coarse materials that may form a mechanical bridge across the opening. It is useful for overall assessment of flowability of free-flowing powders (Bell 1993); however, it is not helpful for predicting the performance in circumstances significantly different from the small hoppers and funnels represented by the flowmeter.

Johanson indicizer^{TM}. Johanson (1992), a former associate of Jenike, developed a tester intended as a short cut to the Jenike procedure that directly measures the unconfined yield strength. No yield loci are constructed and testing is very rapid. Instead of reporting f_c or flow functions, the device estimates and displays the diameter of the hopper outlet opening in which the powder would arch across. A coaxial piston compresses the sample to a predetermined stress level determined by its bulk density. Force is measured on the inner piston only, with the outer piston intended to absorb wall effects. Once the consolidation is complete, the vertical load is removed, and the lower piston drops, confining the sample. The outer upper piston is raised relative to the inner piston, and shear failure is induced by the downward movement of the inner piston. The unconfined yield stress is then directly measured.

Postec research uniaxial tester. This method (Maltby & Enstad 1993) directly applies the concept of unconfined yield strength. The key design detail is the flexible membrane that separates the sample from the wall of the die. It is intended to minimize wall friction, and moves with the powder sample as it contracts under the pressure from the upper piston. The membrane is peeled back after compaction, thus permitting the sides of the sample to be completely unsupported during measurement of the unconfined yield strength. The machine is not as automated as the Johanson device, and users can select any value of major principal stress and measure the corresponding value of the unconfined yield strength. Consequently, a complete flow function can be developed. The inventors reported that the uniaxial tester produced a flow function comparable to that made with a Jenike Tester for a standard reference material (Maltby & Enstad 1993).

Pisecky's flowability index. Niro Atomizer's laboratory (1976) introduced a qualitative method suitable for milk powders with good reproducibility results (+/− 10%). The apparatus is a drum with longitudinal slots. The drum rotates around its longitudinal axis at 30 revolutions per minute while 25 ml of milk powder are introduced into the drum. The measure of flowability is the number of seconds clocked for the powder to leave the drum.

The method is suitable for all types of milk powders and recommended for nondairy powders as well (Niro Atomizer 1976). A classification of flowability and examples of performance for dairy and fatty powders are shown in **Table 4**.

Similar research has been undertaken by Chen (1994), who modeled the discharge of fine powder (coffee, sugar, flour, dried milk) from a slowly rotating slitted drum to quantify the cohesion contribution to powder flowability.

Simplified Navier-Stokes differential equations for continuous fluid flow were used to derive the rate of powder discharge from the drum as follows:

$$\frac{dm}{dt} = -K\left(\frac{\rho \cdot m}{\alpha}\right)^{0.5} \tag{16}$$

Table 4 Flowability classification using a drum test for dairy products (adapted from Pisecky 1978)

Flowability	Classification	Type of milk powder
0–10	Excellent	Re-wetted skim milk
10–20	Very good	Straight-through skim milk
20–50	Good	Straight-through whole milk
50–100	Fair	Lecithinated, straight-through whole milk
100–200	Poor	High-fat powders
>200	Very poor	High-fat powders

where K is a rate constant related to the particle cohesion but independent of powder density, m is the mass of powder remaining in the rotating drum, and α is a function of the poured density of the powder.

Hosokawa powder tester. Carr (1965) designed a combined method where flowability was calculated from a weighted average value from results obtained in testing a powder for angle of repose, angle of spatula, compressibility, and cohesion. The total value was compared on a scale ranging between 0 and 100 (from very poor to excellent flowability). The Hosakawa Micrometric Laboratory designed a powder tester based on Carr's research (1965). This tester performs mechanically and systematically many routines described by Carr (1965). This method is still used in some pharmaceutical and food industries. Ilari (2002) characterized the flow properties of 53 dairy powders. They found that the physical properties tester from Hosokawa provides reliable dairy powder flowability measurements.

Powder rheometer. The Freeman FT3 Powder Rheometer (Freeman Technology) and the ManUmit Powder Rheometer (Stable Micro Systems) (Iles 2000, Texture Technol. 2000) were designed to establish the flow patterns produced in nonconfined materials by forces exerted by a twisted blade moving along a helical path through the test sample. These flow patterns are determined by the combination of axial and rotational speeds. A downward helical path may be a small or steep helix, either left-handed (for more compaction and higher flow) or right-handed (for low compaction owing to slicing action), and depends on which direction the blade rotates. Powders that flow freely will exhibit very little resistance to force, or torque, transferred through the powder column in either the downward or the upward direction. Conversely, poorly-flowing powders exhibit a substantial amount of torque in either direction. The wave of powder displacement is virtually in steady state, allowing flow to be observed, and generally resulting in smooth, linear, or logarithmic profiles of the measured forces. It is worth mentioning that after the inception in the market of the Freeman FT3 Powder Rheometer, Freeman Technology developed an updated version, the FT4, capable of measuring a range of additional powder properties such as shear and wall friction, as well as bulk properties like permeability, compressibility, and density. This allows comprehensive characterization of powders from a fluidized state to a highly consolidated one.

FINAL REMARKS

Flowability of food powders is a complex phenomenon that depends on both the powder's characteristics and the flow system configuration. Powder friction, interlocking, and cohesion lead to the formation of stable structures and are the principal forces opposing flow. Powder characteristics such as moisture content, composition, particle size distribution, and shape (surface properties) are established by the particle production method and can powerfully influence flow behavior.

Failure properties obtained from the yield locus and flow function for each specific powder, under a particular set of conditions, can be determined using not only Jenike's shear cell, but also other custom cells. Protocols for cells such as the annular shear cell or the direct shear cell have become international standards. Biaxial and triaxial testers can also provide information about failure properties, although not much has been published on food powders. Although Jenike's method is standard for the determination of opening size and hopper angle, it still has difficulty obtaining reliable flow property measurements with high reproducibility.

Other methods and parameters exist for the characterization of powder cohesion, which, although not yet useful in the determination of hopper angle and opening size, may provide a fast way to characterize flow. Non-conventional approaches have appeared as quicker and user-friendly techniques by either accounting for overall characterization of a number of bulk properties, using unconfined yield strength or tensile stress measurements, or calculating energy requirements to rotate a blade in a powder rheometer. However, few comparisons exist regarding the sensitivity of these methods with respect to standard failure property methods.

Theory adapted from soil mechanics to food powders has shown that the effective yield locus can be determined using a friction-based index. Although more research is needed in this field, the validity of the theory has been seen in dry powders such as table sugar and salt.

Determining models and flow property charts that account for only the effects of powder composition in failure properties is already a challenging task, considering the existing variability found at the particle level (size, porosity, hardness, and surface ruggedness), and bulk level, given that different cohesive forces become predominant at different sizes, temperature, relative humidity, and composition. This includes the characterization of fat-based powders, which provide particle ductility and surface lubrication. An additional challenge would be to understand how these model particles interact in the creation of uneven flow patterns inside a bin, which produce start-up delays. With current facilities experiencing daily bulk-solids flow problems, solids flow testing and analysis can save many hours of expensive down time worth many thousands of dollars, thus progressing the industry from the dilemma of quick-fix solutions that fail to deliver to proven engineering solutions that work over long periods of time.

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