

Recent developments in characterizing powder flow properties show promise in predicting the success or failure of tableting presses, and related powder processing and handling equipment, with untried formulations. Derived from basic powder flow properties, the recently developed flow indices quantify various basic powder flow phenomena related to blenders, feeders, chutes, bins and tableting presses. This article describes the indices, their measurement using small samples, and their application to pharmaceutical equipment selection, design and operation.

An indices classification system

A system for classifying the physical properties of powders can be used to quantitatively predict powder behavior in whatever processing equipment may be available or needed to make the final product. Such a classification system must:

- Be based on all the fundamental rheological properties of powders
- Consist of operator-independent, reproducible tests that can be run quickly
- Make measurements at consolidation pressures and atmospheric conditions that simulate the processing equipment, and
- Predict quantitative indices that relate directly to powder handling and process equipment parameters.

A series of flow indices can be used to predict how a powder will behave in various processing situations

Flow indices in the prediction of powder behavior

*Dr Jerry Johanson
JR Johanson Incorporated*

The basic rheological powder properties are unconfined yield strength, bulk density, air-flow resistance (permeability), surface and internal friction angles, surface adhesion and elastic spring-back modulus.¹ All of these are a function not only of chemical composition, particle size distribution and particle shape (all of which are sample-dependent), but also of the compaction pressure that any

given sample is subjected to during handling or testing. Consequently, detailed measurements of these properties produce a multiplicity of data that is difficult to interpret.

When developing a simple index to quantify the behavior of solids in equipment, one must consider a combination of rheological properties. For example, indices predicting hang-ups must be based on the ratio of unconfined yield strength and bulk density, both of which are measured at the solids' contact pressures associated with the hang-up. Powder-flow rates depend on air-flow resistance and bulk density changes as the powder flows from the lower part of the bin to the hopper outlet.

The test methods must be rigorously defined, meticulously followed and automated to produce reproducible, quickly run, operator-independent tests. The indices must be derived using a combination of practical experience and mathematical linkage between the powder properties and the process equipment behavior.

The indices classification system developed by the author consists of eight powder rheological (flow) property indices derived using uni-axial test cells, as shown in Figure 1.² The sample is uni-axially compressed by the two-piece top cylinder to the pressure associated with the process equipment. The outside cylinder has loose fit tolerances to the test cell to prevent powder from squeezing out of the test cell. The consolidation pressure is measured only on

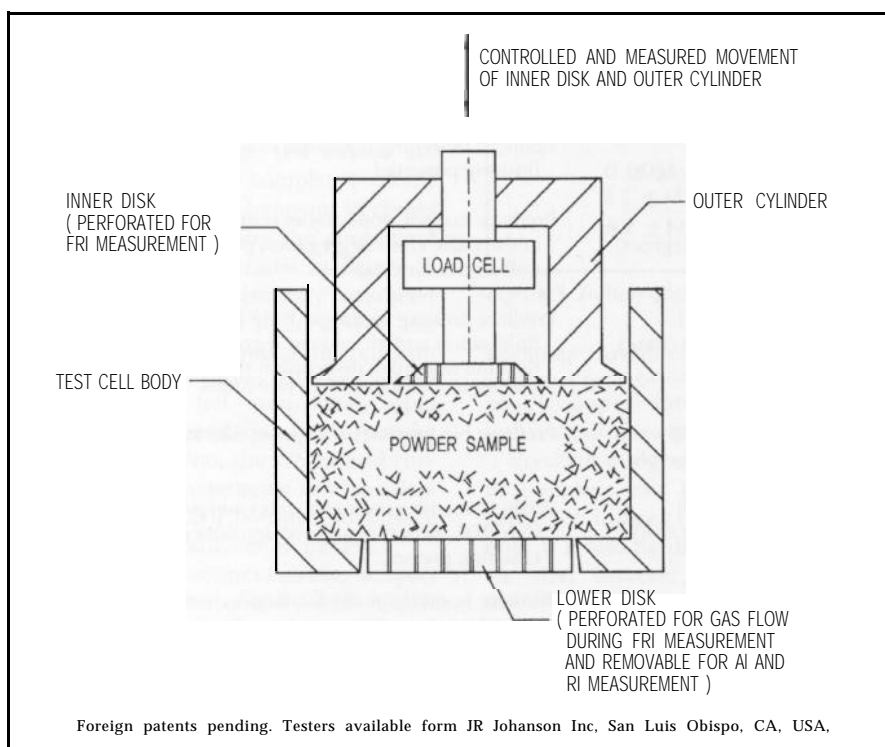


Figure 1. Uni-axial test cell, US Patent 5289727.

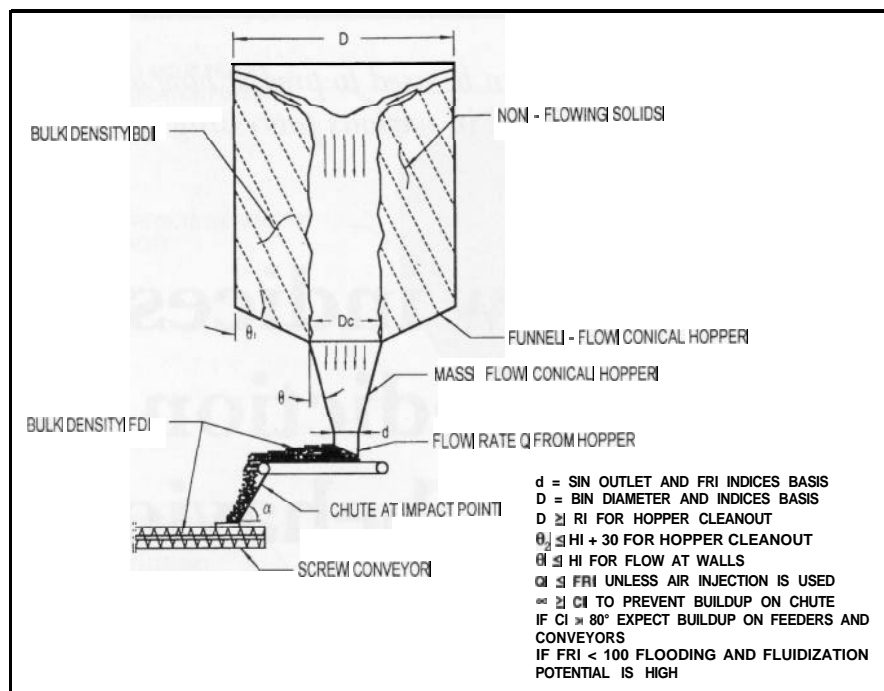


Figure 2. Flow characteristics indices as applied to a powder handling System.

the central disk by the local cell. This eliminates the frictional effects that occur as the outer piston moves relative to the test cell. This basic uni-axial load application is used for most of the indices tests. This test cell, in combination with the automated tester," is attached to measure simultaneously the compaction pressure and bulk density of the sample.

Tests defining the indices

The various indices, their measurement and basic derivation and use are outlined below.

The *Arching Index (AI)* is determined by compacting the sample uni-axially at a pressure associated with a hopper outlet. The upper piston and lower disk (as shown in Figure 1) are removed, leaving the test sample suspended and supported only by the outer lip of the test cell. The suspended sample is then failed as the upper piston is lowered. The peak failure load is recorded, interpreted as the unconfined yield strength and used to calculate the critical arching dimension or AI under these conditions. This index is presently undergoing a standardisation procedure by the American Society for Testing Materials (ASTM). The AI is used directly to determine the required outlet size (see Figure 2) and indirectly to indicate segregation or de-mixing tendencies.

The *Rat-holing Index (RI)* is determined by applying the uni-axial pressure associated with solids in a funnel-flow bin of diameter D as specified. The unconfined yield stress and

bulk density are then measured as with the AI and used to calculate the critical rat-hole diameter and RI under the conditions measured. The RI is used directly to determine the size of the flow channel required to eliminate hang-ups on funnel-flow hopper walls (see Figure 2) and to determine agglomeration in packages, blenders, drums and railcars.

The *Flow Rate Index (FRI)* is deter-

mined by compacting the sample uni-axially at a pressure associated with solids at the hopper outlet specified. The *Feeder Density Index (FDI)* and air-flow resistance (permeability) are then measured under these conditions. The sample is then further compacted uni-axially to the pressure associated with the vertical portion of the bin and the *Bin Density Index (BDI)* is then measured. The compaction load is then released and the percentage of spring-back of the powder sample is measured; this is called the *Springback Index (SBI)*. These measurements are then interpreted as the maximum powder flow from the hopper outlet when the powder is fully de-aerated, or the FRI. This index is used to determine powder flow rate from hoppers, the need for air injection, fluidization and air current segregation potential, flooding tendency, flow rate limits into tableting presses, capping potential of tablets, fill rate limits for capsules, de-aeration time in packages, drums and bins, and limiting speeds for rotating shell and ribbon blenders.

The last two indices, the *Hopper Index (HI)* and *Chute Index (CI)*, are determined by the angle of slide or friction angle on a flat plate. The angle of slide is determined by placing a predetermined load on a powder sample confined in a circular ring on a flat plate. The plate is then tipped until the ring, solids and load slip continuously down the plate. The angle at which this occurs is the angle of slide. The arc tan

Index	Symbol (units)	Application in the pharmaceutical industry
Arching Index	AI (length)	Predicts: hopper outlets to eliminate arching, angle of repose segregation potential, tablet and capsule fill weight variations.
Rat-hole Index	RI (length)	Predicts: rat-holing hang-ups, lump formation and flushing potential.
Hopper Index	HI (degrees)	Predicts: hopper slope angles required to cause flow at the walls, chute segregation potential and hopper wall angles for clean-out.
Flow Rate Index	FRI (wt/time)	Predicts: limiting feed rates from hopper outlets, fluidization and air current segregation potential, flushing potential, de-aeration time in containers, pre rate limits and capsule till rate limits.
Bin Density Index	BDI (wt/vol)	Predicts: bin gravimetric capacity and loads on bin walls
Feeder Density Index	FDI (wt/vol)	Predicts: feeder gravimetric rates and, when compared with BDI, gives the range of densities possible at tableting presses.
Chute Index	CI (degrees)	Predicts: build-up in chutes, feeders, conveyors and press feed shoes.
Springback Index	SBI (percent)	Predicts: special hang-up problems with elastic wind-up.

Table 1. The Johanson indices and their applications.

of this angle is the surface friction coefficient between the powder and the plate at the applied pressure. This angle of slide is then interpreted as the HI or the conical half-angle required to cause mass flow.

The value of the CI is similarly determined, except the powder confined in the ring on the plate is first uni-axially compressed onto the plate at 100psf. This compaction load is carefully removed to leave the sample undisturbed on the plate. The plate is then slowly tilted until the unloaded sample breaks away and slides. This slide angle is interpreted as the Chute Angle Index to prevent build-up on chutes.

The indices test procedures described above provide the basic concepts. The actual tests are more explicitly defined by the automated Johanson Indicizer System,³ which has carefully controlled load and displacement application rates, simultaneous density and load measurements, and automated calculations that eliminate interpretation errors.

Using the indices

Because the indices represent all of the basic rheological powder properties, they can be used to understand and predict flow behavior whenever a bulk powder is squeezed, conveyed, stored, fed, mixed, packaged or compacted. Table 1 indicates some of the uses of various indices. Often, new formulations may need to be used in existing pre-qualified equipment, even perhaps in an entire existing set-up. The indices allow one to determine if this can be done with a sample of the new formulation equal to about 300ml. There is no need to produce large quantities to try in full-size systems.

In the case of a typical handling system using conical hoppers, the indices are used as they occur to completely specify the geometry, predict hang-ups in chutes and feeders, and to size the equipment needed. This is illustrated in Figure 2. Nearly every critical geometry parameter is determined by the indices. With the indices of a new formulation, one can determine if an existing handling system will work and tell immediately what changes, if any, need to be made to make it work. In addition, the feeder and conveyor power requirements can be calculated using the FDI density and surface friction angles contained in the HI.

Other hopper shapes can also be specified by the indices. Figure 3 shows the indices design criteria for six different hopper shapes. Notice that some shapes allow much smaller outlet diameters to prevent arching. This allows the plant designer to extend the range of materials handled by a given hopper by incor-

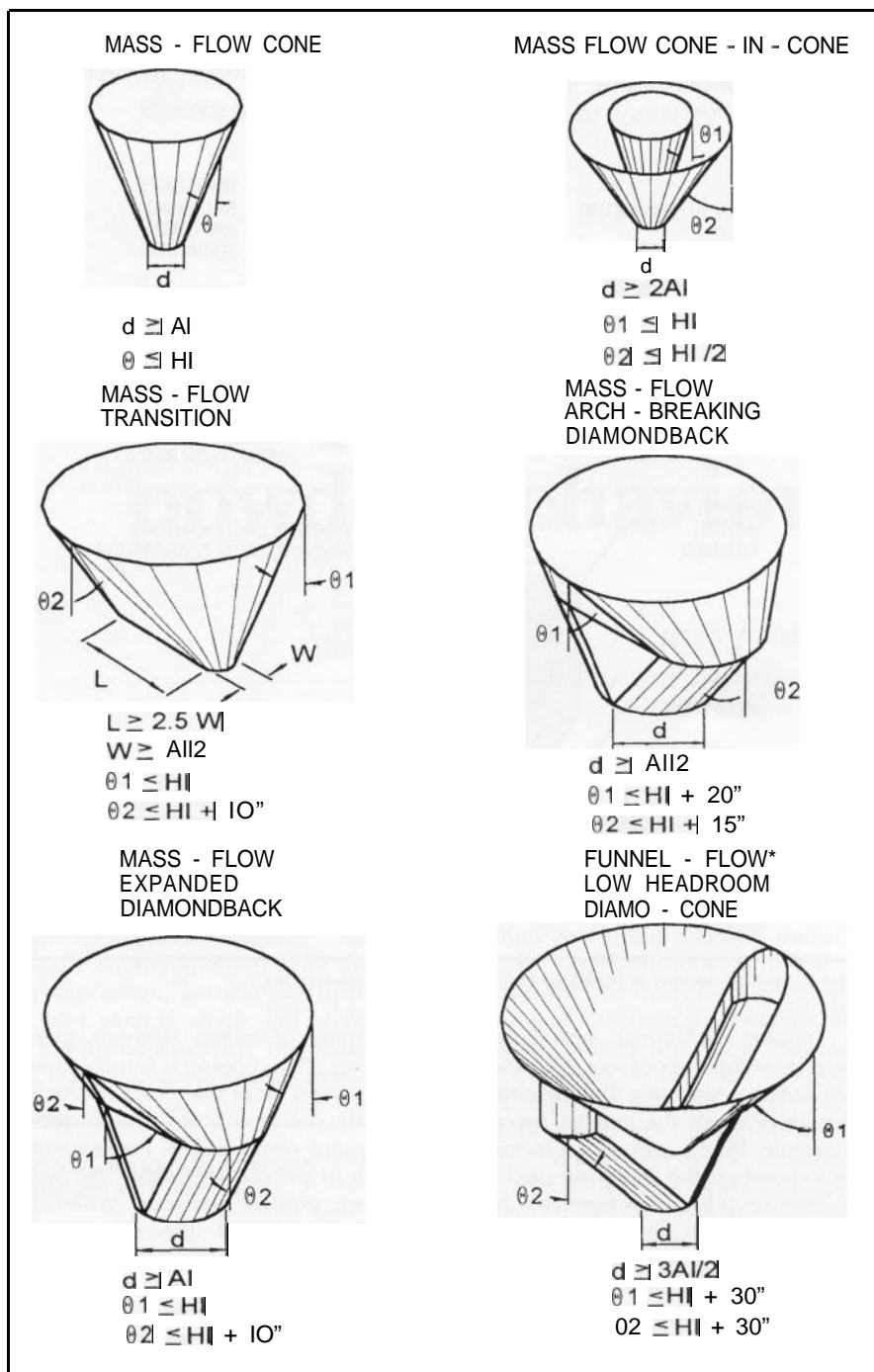


Figure 3. Indices applied to various hopper shapes.

porating Archbreaking Diamondback Hoppers or a headroom saving cone-in-cone hopper into the system. The indices indicate when this is necessary.

Predicting segregation and demixing in blenders can be greatly aided by using the indices of individual ingredients. It is also helpful to know the particle size; however, the FRI gives an excellent indirect measure of particle size without using laborious screening or more expensive particle counting techniques.

Tableting and capsule filling indices applications are summarized in Figure 4. Usually, the system already exists and a

new formulation needs to be used with the system. In general, a powder with lower AI, RI and CI indices, and larger FRI and HI indices, will likely work satisfactorily in the old system, especially if the BDI/FDI ratio is smaller. The only negative with this new formulation is that it may segregate more easily than the previous formula.

Figure 4 offers some guidelines as to what indices to look at when various problems occur. The absolute magnitude of these indices with respect to hoppers and feeders has been confined experimentally. Quantifying actual capsule and tablet dye filling operations

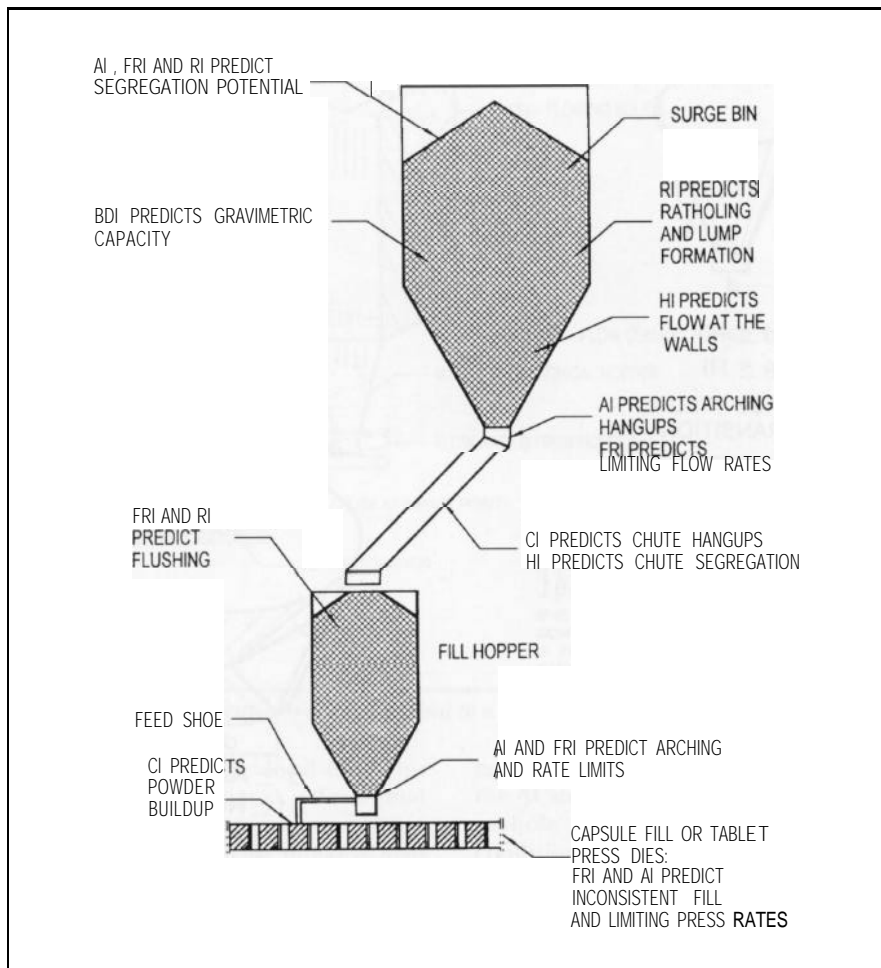


Figure 4. Indices applied to tableting and capsule-filling operations.

will require additional correlations before these operations are quantified by the indices. Assuming that the chutes and hopper satisfy the indices, successful capsule filling and tablet-forming then depend on the following parameters: absence of lumps as predicted by a low RI; absence of sticking to the feed shoe as predicted by a low CI; absence of arching over the capsule diameter as indicated by a low AI; the ability to fill the capsule in the allotted time as indicated by a large FRI; and the consistency of the density in the capsule as indicated by a small BDI/FDI density ratio. With a few controlled tests, the allowable magnitude of the indices for a given press or fill system can be quantified.

Capping of tablets deserves special mention. This capping is usually caused by entrained air in the tablets. If the FRI is small, the air cannot escape during filling and compression; consequently, the air is pressurised within the voids between powder particles. When the specific gravity of the substance is known, this pressure can be calculated. If, after elastic springback, the pressure exceeds the strength of the tablet, then capping occurs. Sometimes this capping is aided by powder adhesion to the press plunger as indicated by a large CI. Since the tableting pressures are generally much higher than encountered in bins, the indices provide only relative guides. Special high pressure CI and RI

are needed to provide absolute measures of capping. These testing devices would follow the same uni-axial format indicated for the other indices testing, but would likely involve smaller test cell diameters.

Conclusion

The indices technique, although developed specifically for predicting flow in hoppers, feeders and chutes, shows great promise in the broader application of characterizing the rheological properties of various formulations. Because the indices are a combination of basic properties, they can be extrapolated to predict the success or failure of new formulations in existing processing systems. The 300 ml quantity of powder used makes testing much less expensive than pilot studies or full-scale testing. □

DR Jerry Johanson is a world-renowned expert on solids flow handling and storage, having spent 30 years helping industries to eliminate problems in processing and handling equipment. After receiving a PhD in mechanical engineering from the University of Utah, in 1962, he joined US Steel where he headed the materials-handling research section. In 1966, he teamed up with his former University of Utah, professor, Dr Andrew Jenike, to form Jenike & Johanson Incorporated, and was appointed president of the company in 1979. In 1985, he left to form his own consulting business, JR Johanson Incorporated, and today specializes in a variety of solids flow problems. Dr Johanson has authored more than 100 technical articles, holds 14 US and several international patents in the field of solids flow, and is a frequent lecturer at conferences and exhibitions worldwide.

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