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THE BENEFITS OF POWDER CHARACTERISATION

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1. Handling and Storage of Bulk Solids

A large number of industries including power generation, petrochemical and plastics, bulk and fine chemicals, pigments, pharmaceuticals and agrochemicals, detergents, water treatment, food processing etc, are involved in handling bulk solids in raw ingredient form, during intermediate processing or right up to the 'point of sale'. One international company, DuPont, reckons that 60 per cent of their products and intermediates are manufactured in particle form and only 20 per cent of their products are not involved with particles at all (Davies, 1984). Consequently a prodigious variety of bulk solids are stored in hoppers or silos in quantities ranging from a few kilos to hundreds and, in some cases, thousands of tonnes.

It is an unfortunate fact that many installations regardless of their size are plagued by discharge and handling problems. Common difficulties include the inability to discharge on demand, hold up of a significant irretrievable inventory and unfitness for subsequent process or purpose, as with difficulties associated with segregation or uncontrollable 'flushing'. It has long been recognised that these issues result in severe restrictions on plant performance (Merrow, 1988). Figure 1 illustrates the unfortunate performance shortfall of plants handling solids compared with plants that only handle liquids or gases.

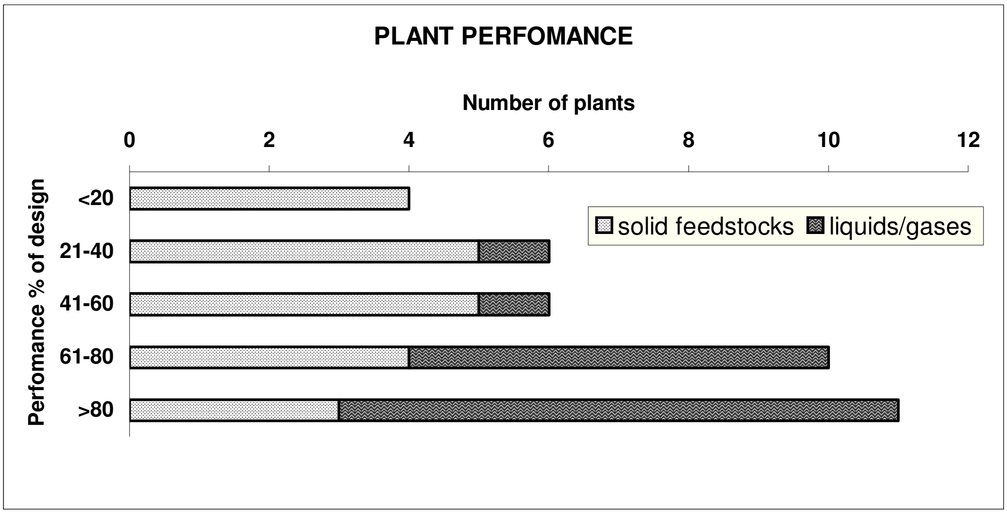

Figure 1: Performance of plants handling solids and liquids compared (Merrow, 1988)

Merrow identified that the main issues for solids processing plants were not in the chemistry of processes but rather that most problems resulted from the tendency of bulk solids to cause blockages, stick, flow erratically or go where they should not. The lack of a thoughtful design approach to 'low tech' items like hoppers is a major factor contributing to production shortcomings (Neale, 1998). Standing on a loading shovel to beat a hopper with a length of piping is one way of promoting flow - but it's not the best option. Figure 2 shows the consequences of not carrying out a design based on proper flow characterisation.

Figure 2: Promoting flow by brute force.

2. Bulk Solids Flow Properties

Because of the way they flow powders cannot be treated in the same predictable way that we can confidently assess liquids or gases. Powders can sustain stresses to variable threshold according to their structure and stress history, with a degree of elastic deformation. Failure (or flow) when it does occur is rarely uniform. Despite the complexities, Andrew Jenike pioneered a methodology of powder testing and reliable hopper design based upon a theory of flow of bulk solids (Jenike, 1961, 1964). This put science into this aspect of bulk solids handling technology, but the testing method is elaborate and tends to find limited use.

Characteristic descriptions, such as 'free flowing' or 'poor flowing' are subjective and only reflect a specific condition in particular circumstances. A powder can appear to be 'free flowing' when it is loosely poured, but may settle to a very firm and stable condition when de-aerated or subject to compacting stresses. A dry, crystalline product will usually flow through a relatively small orifice, but have extreme reluctance to deform if damp or 'caked' due to the presence of tiny crystal bridges binding particles together.

There are a variety of techniques that attempt to quantify 'flowability' e.g. angle of repose (Tenou et al, 1995), Hausner ratio (Grey and Beddow, 1969), Carr Index (Carr, 1965) and of course the more scientific Jenike Flow Function (Jenike, 1961). Some techniques pursue dynamic flow conditions using devices such as the Stable Micro Systems 'Powder Flow Analyser' with its propeller penetrating a bed of powder (Cowell et al, 2005) and the Aeroflow device which measures how chaotic powder avalanches (Kaye, 1996). Their results however tend to be difficult to relate to actual equipment design. Of course a 'single number' approach is limited, as it is only relevant to one particular aspect of the bulk solid. It is the complexity of the multiple attributes of a bulk solid and their interaction with many facets of equipment design that defines the actual bulk flow behaviour.

Perhaps the most important aspects for flow are how the product slides on a contact surface e.g. hopper wall, (wall friction), and the resistance offered by the powder bulk to deformation/flow (shear strength). These characteristics are influenced by the 'condition' or 'compaction' of the bulk – a tightly packed bed is less free flowing than a loose aerated powder. This 'condition' is directly related to the bulk density. In hoppers and conveying systems the stress influences the bulk density.

The actual flow behaviour in plant is not only contingent on the material characteristics - the equipment form has a major influence too. For example steep hoppers with large outlet sizes often have flow benefits: they prevent arches and rat holes, they generate mass flow and mitigate the effects of segregation. By contrast, a 'rat hole' may form if the hopper slope is shallow, so the effective storage capacity of the hopper is reduced. If the outlet is too small, an 'arch' may stop the flow altogether.

3. Powder Testing for Flow

3.1 Wall Friction

The wall friction angle, ϕ_w, is significant because powders are expected to slip against a contact surface – the wall of a hopper, the blade of a screw conveyor – if optimum performance is to be achieved. A translational shear device such as the AJAX wall friction tester can be used to determine friction between a contact surface (the wall material) and powder constrained within a test cell. Figure 3.

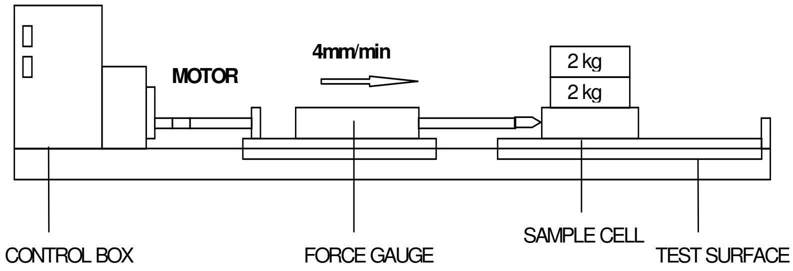

Figure 3: AJAX wall friction tester

During the test two measurements are recorded:

1) the force perpendicular (or 'normal') to the test surface due to the total weight acting on the powder/wall interface comprising the supplementary load, the cell and the sample itself

2) the shear force measured on the force gauge required to push the cell over the surface.

The measurements are converted to normal and shear stresses and plotted on a graph as a wall yield locus.

A typical graph obtained with a herbicide powder against 2B mill finish stainless steel is shown in Figure 4.

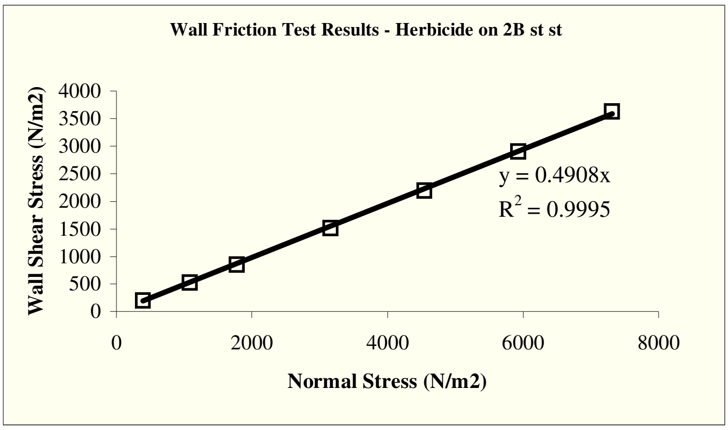

Figure 4: Wall friction characteristic for herbicide on 2B stainless steel

The friction angle ϕ_w is simply the slope of the best fit line through the data points.

For this herbicide ϕ_w is well defined by the inverse tangent of the constant gradient 0.4908 i.e. 26.1 degrees. Whilst graphical methods are available for defining the mass flow hopper wall angle from this data satisfactory results can be obtained using equation 1.

$$\beta_c = 1.2\,\phi_w + 43 \qquad \text{Eq.(1)}$$

Although a straight line through the origin is common, other characteristic relationships are observed in practice including curved loci (where the friction angle varies with stress) and loci with an intercept at zero normal load. This latter aspect indicates an element of adhesion against the surface even at very low stresses and that care is needed with the hopper geometry e.g. radiused walls and in the design of flights of screw feeders. Figure 5 shows the type of adhesive build up which can clog a standard full face flight form or tight pitches in a screw feeder. Using slender ribbon screw geometry is one solution for avoiding such problems.

Figure 5: Titanium dioxide sticks to hopper walls and clogs screw feeder flights

If the appropriate value of ϕ_w is used then good agreement is seen between calculated wall angle for mass flow and the observed behaviour in real hoppers (McGee and McGlinchey, 2004). Analysis from the results from a large number of wall friction tests with a wide variety of bulk solids shows some interesting results. Figure 6 shows two data sets - McGee & McGlinchey (2004) from over 200 bulk materials tested on the Ajax wall friction machine and this is compared with some optimisation work (best bulk solid/wall combination) carried out by ter Borg (1981).

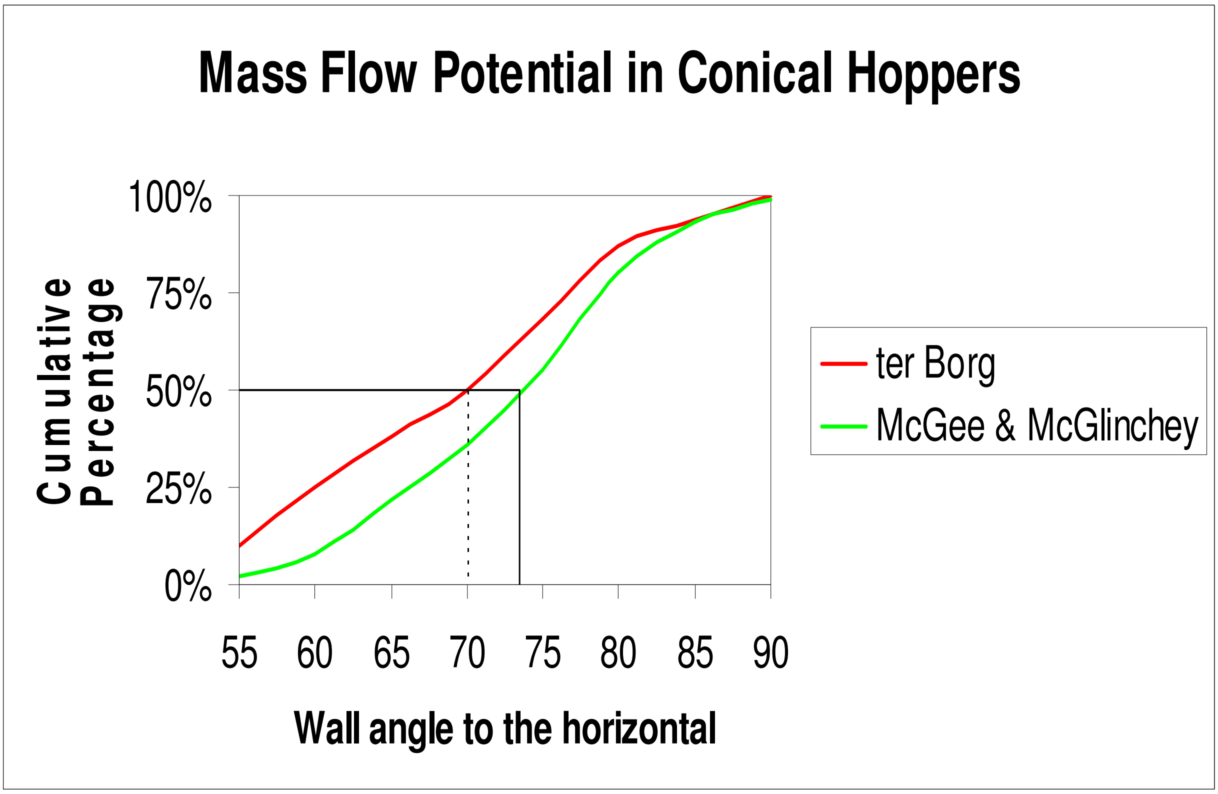

Figure 6: Wall friction data for 214 powder/wall surface tests

This graph shows that the very commonly used wall angle of 60 degrees to the horizontal has a very poor prospect of generating mass flow. Of course mass flow may not be mandatory but at least if you have sufficiently steep walls you know a rat hole cannot form. Whilst the steepest walls are needed with conical hoppers, the wall angle requirements for Vee shaped hoppers (where the convergence towards the outlet is only in one plane) can be about ten degrees shallower.

3.2 Shear Strength

In most industrial situations it is the resistance to the initiation of flow of a bulk solid that is important. A hopper is usually filled when the outlet is closed or its feeder is idle. The commencement of flow when the hopper outlet is opened or feeder started is dependent upon the nature of the material and the conditions to which it has been subjected. The vertical shear test reflects the need to initiate flow from a filled condition. A sample is compacted in the test cell and after exposing an unconfined surface a plug of bulk solid within the bed is forced out of the cell. Figure 7.

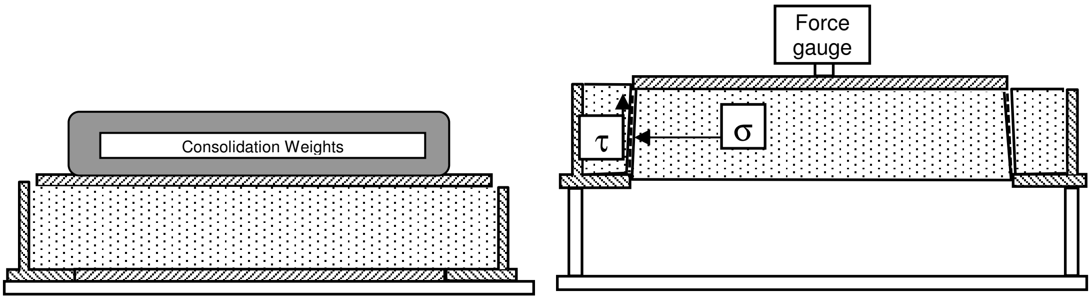

Fig. 7: The consolidation and shearing stages with the Ajax vertical shear cell tester

The consolidating load can either be a reference standard or selected from knowledge of the hopper dimensions. The mass of material and the volume occupied are used to establish bulk density ρ_b. The load to cause failure and the surface area this acts over are used to establish the shear strength, τ_s. Using a force balance analysis of the conditions at failure indicates that a minimum diameter, D_{crit} for destabilising a rat hole or arch can be given by equation 1, where g is constant due to gravity (9.8 m/s^2):-

$$D_{crit} = \frac{4 * \tau_s}{\rho_b * g} \qquad \text{Eq. (2)}$$

The equation indicates that as the diameter of the outlet increases the peripheral area (resisting shear) which supports a blockage goes up in direct proportion to the diameter, whilst the weight of product to overcome resistance to shear goes up with the diameter squared. Increasing the outlet size eventually means the stress due to the weight of the bulk will exceed the resistance that the powder's shear strength offers. The measured shear stress and the bulk density are two parameters that affect the size of outlet needed for flow. (Note that the measured shear strength is not – and does not equal - the unconfined yield stress in the Jenike method.)

Using the data from a large number of tests a graph, Figure 8, can be plotted with the y-axis representing the numerator ($4 * \tau_s$) and x-axis representing the denominator ($\rho_b * g$) from equation 2 (McGee, 2005). The various materials' data points are then plotted in a space where lines of constant outlet size can be superimposed to partition a 'flowability' grouping for bulk solids. This helps characterise materials in a space where a sensitive plant parameter (outlet size/rat hole diameter) defines flow boundaries. Note the partitioning lines identify constant outlet sizes as follows: - 1m – poor flowing, 0.5m average and 0.15m easy flowing.

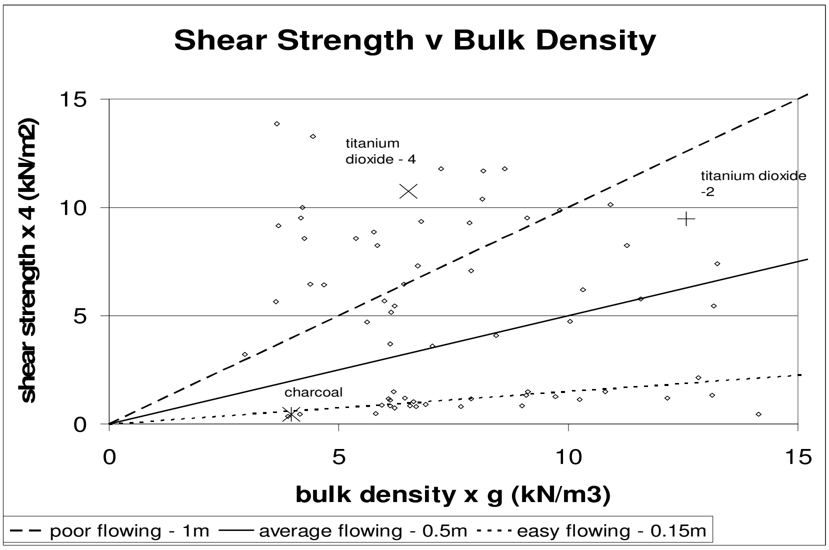

Figure 8: Data from shear tests plotted in sectors partitioned by constant outlet size

By way of example the tests indicate that titanium dioxide - which is prone to forming a rat hole or arch is towards or in the 'poor flowing' region whilst a charcoal is likely to flow through an outlet less than 15cm diameter and is 'easy flowing'.

A benefit of this form of data presentation is products that have similar strength e.g. grades 2 and 4 of titanium dioxide can actually be better distinguished from the Jenike flow function construct because this graph incorporates bulk density. This is highlighted in Figure 8 by the placement of the grade 2 results midway between 'average flowing' and 'poor flowing' whilst grade 4 is deeply placed into the 'poor flowing' area.

3.3 Bulk Density

Density is defined by the ratio of mass divided by the volume occupied. However with bulk solids this property can be almost notional. Packing depends upon the shape of the particles and their sizes as well as what opportunity the particles have had for arranging themselves in the bulk. This is influenced by vibration, confining and consolidating stresses. Therefore the 'conditions of interest' may define a range of bulk densities e.g. a bulk solid stored in a hopper may assume one density condition that is different from that when the bulk solid flows into a conveyor. A stable bulk density can indicate the flow behaviour can fairly insensitive to how the material is handled. Bulk solids which have a variable bulk density tend to be more sensitive to how they are handled. This aspect was noted by Hausner who identified the change in density under vibration as one aspect of flowability (Hausner 1967). The ratio of tapped to loose bulk density is the Hausner Ratio and the greater the ratio is, the more sensitive the powder is and hence flowability worsens. An alternative to the original ratio is the Compacted Hausner Ratio where the bulk density obtained under compaction e.g. in from a vertical shear cell test replaces the tapped density.

4. Spider Diagrams

One way of bringing the flow characterisation data together is to build a 'spider' diagram (McGee and McGlinchey, 2005). The diagram is built from a series of three concentric circles each divided by an axis for each of the characteristics (wall friction (ϕ_w), shear strength (τ_s), bulk density (ρ_b), hopper mass flow wall angle (β_c), outlet size (strength/bulk density ratio, D_{crit}) and Hausner ratio (H.R.). The axes intersect with the smallest diameter circle where that particular characteristic describes 'easy flow' behaviour with subsequent bigger diameter circles defining 'modest' or 'average' and 'poor flow'. Two idealised situations can then be presented. Figure 9 shows the spider diagrams for an 'easy flow' material and a 'poor flow' one with the in-filled part of the 'web' detailing the particular characterisation attributes.

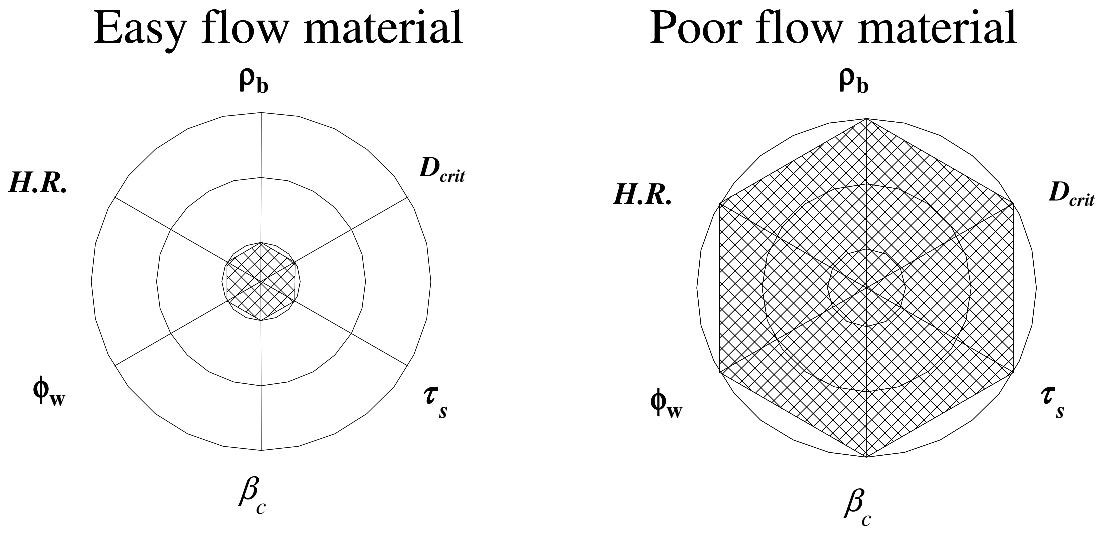

Figure 9 Spider diagrams for ideal 'easy' flow and 'poor' flow material

The diagrams can be more than qualitative if the data from the tests on the large number of materials (over 200) reported in (McGee, 2005) is used to define the 'easy', 'average' and 'poor' flow circles, Table 1.

Table 1 Parameters suggested by the tests reported in McGee 2005.

Circle	Wall friction (deg)	Bulk Density (kg/m3)	Shear strength (N/m2)	Hausner ratio	Outlet size (cm)	Mass flow Wall angle (deg)
Easy flow	< 20	1200	300	1.1	15	65
Average	25	800	1000	1.25	50	73
Poor flow	> 30	400	2000	1.5	100	80

Note that the bulk density axis is the reverse of the others because decreasing bulk density usually means poorer flow. A practical example is that most milling operations lower bulk density and worsen flowability of powders when they are stored.

This tabulated data would indicate that materials had the following characteristics:

Typical 'easy flow' material This would be a low friction material (<20 degrees), which would mass flow in a conical hopper with a wall angle of 65 degrees to the horizontal. It would have a high bulk density (around 1200 kg/m^3) but not be affected much by compaction or vibration so would have a low Hausner ratio (up to 1.1). Its low shear strength (maximum 300 N/m^2) coupled with the high bulk density would guarantee flow through a small outlet (<15cm diameter). A practical example would be a free flowing grade of lactose with wall friction angle of 17 degrees against stainless steel, shear strength 197N/m2, Hausner ratio of 1.1, rat hole diameter 9cm and requiring a 64 degree wall angle for mass flow in a conical hopper. With a bulk density 867kg/m3 this particular example would have a small spike on the density axis of the spider diagram indicating a slight deviation from the ideal flow material.

Typical 'poor flow' material A high friction material (>30 degrees), which would barely mass flow in even the steepest conical hopper (>80 degrees) (in fact probably require a Vee shaped hopper). It would have a low bulk density (about 400kg/m^3), which would be significantly affected by compaction indicated by a high Hausner ratio (about 1.5). Its high shear strength (2000 N/m^2) coupled with the low bulk density would mean very large outlets (>100cm) would be needed to ensure flow. A practical example would be a fine milled icing sugar with wall friction of 30.5 degrees against stainless steel, bulk density of 540 kg/m3, shear strength 2144N/m2, Hausner ratio of 1.49, rat hole diameter 149cm and requiring a wall angle for mass flow in a conical hopper of 80 degrees to the horizontal.

The technique when applied to two real examples highlights particular aspects of the 'profile' that merit special attention. Figure 10 shows the resultant diagram for a chemical Intermediate 1 and a pharmaceutical powder. For the intermediate all aspects for flow are good except the shear strength and outlet size. Indeed practical application for batch handling of this material required invertible bins that upset the consolidation of the material to ensure reliable flow to process.

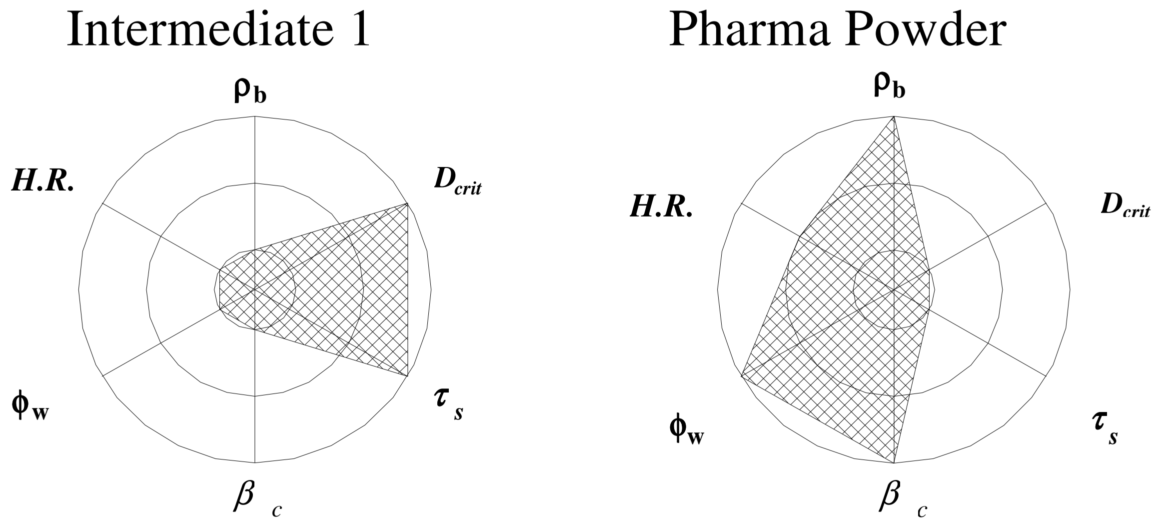

Figure 10 Spider diagrams for Chemical Intermediate 1 and a Pharmaceutical powder

The pharmaceutical powder however has high wall friction but low shear strength. Difficulties with chute work featuring insufficiently steep slope and sharp corners would have been quickly identified with this data and practical solutions like examining the effects of surface finish examined.

5. Summary

Flow behaviour of granular materials is complicated by a host of characteristics which can manifest themselves at the bulk scale, causing essential plant functions like storage and feeding to be fraught with difficulties. Flow characterisation of those aspects relevant to bulk behaviour (how the material slides against a contact surface, how strong it gets and how the bulk density varies under compaction) is essential if one wants to avoid poor plant performance. These characteristics can be measured and the data translated into meaningful plant design criteria like reliable outlet size and wall angle for flow. From the measurements, a number of calculated parameters and ratios can be used to describe and quantify the ‘flowability’ of the bulk solid. The various aspects can be combined into a spider diagram which provides a characterising ‘identikit’ of the bulk solid. This is a useful means of presenting a fuller picture of the flow relevant data for a particular bulk solid. Developments of this profiling technique can focus on individual materials (e.g. different grades, batches, suppliers, seasonal variations, etc) and set acceptable boundaries which could be modified by plant performance or indicate operating strategies for optimum performance.

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