The consolidation of a powder bed can occur in different ways. Consolidation during a transportation or processing can be due to vibration, with powder subject to normal and lateral stresses. This is often simulated by using a jolting volumeter or autotapper to tap a powder sample within a cylinder which causes rearrangement of the particle packing structure. Consolidation can also occur during storage where a powder is primarily subject to the normal stress associated with its own weight. To simulate this in a test, materials can be consolidated by applying a direct pressure using a piston, for example.

Hausner Ratio has traditionally been used to assess powder flow by comparing the poured and tapped density and is calculated using:

$$Hausner\ Ratio = \frac{Tapped\ Density}{Poured\ Density}$$

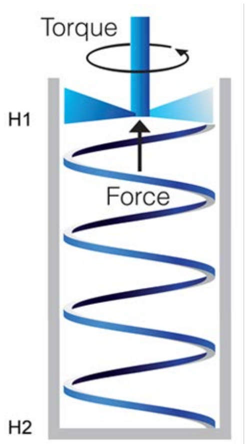
Powder flow is then categorised as follows:

Flow Character	Hausner Ratio
Excellent	1.10-1.11
Good	1.12-1.18
Fair	1.19-1.25
Passable	1.26-1.34
Poor	1.35-1.45
Very Poor	1.46-1.59
Non-Flow	>1.6

THE FT4 POWDER RHEOMETER®

The FT4 Powder Rheometer® is a universal powder tester that provides automated, reliable and comprehensive measurement of bulk material characteristics. This information can be correlated with process experience to improve processing efficiency and aid quality control. Specialising in the measurement of dynamic flow properties, the FT4 also incorporates a shear cell, and the ability to measure bulk properties such as density, compressibility and permeability, enabling a comprehensive characterisation of the powder in a process relevant context.

Dynamic testing employs a unique measurement technique to determine a powder's resistance to flow. A specially shaped blade traverses along a prescribed path through a precise volume of the powder. The force and the torque acting on the blade, as it moves axially and rotationally, are combined to generate a value for flow energy[1].



EXPERIMENTAL SET UP

A selection of ten powders used in a range of industries were assessed. Two methods were used to assess the impact of different types of consolidation. Method 1 is based on tapping the powder bed, simulating a transportation process and Method 2 on directly compressing the powder bed, simulating long-term storage.

Prior to each measurement, a Conditioning cycle was carried out, ensuring that the sample was in a homogenous, loosely packed state. Note that standard Hausner Ratio measurements do not employ Conditioning prior to measuring the poured density and therefore repeatability is likely to be adversely affected by operator variability.

Method 1: Two tests were conducted; the first measured Basic Flowability Energy (BFE) using the twisted blade, as described above. The test protocol also provides of the powder bed in a loosely packed state, i.e. Conditioned Bulk Density (CBD). For the second, the powder was tapped 50 times using an using a Copley JV Autotapper and the Consolidated Energy measured using the same technique for the BFE. The test also provides the density of the consolidated sample (BD_{Tap50}).
Method 2: The powder was compacted with a vented piston applying a Normal Stress of 15kPa and the percentage change in volume was measured.

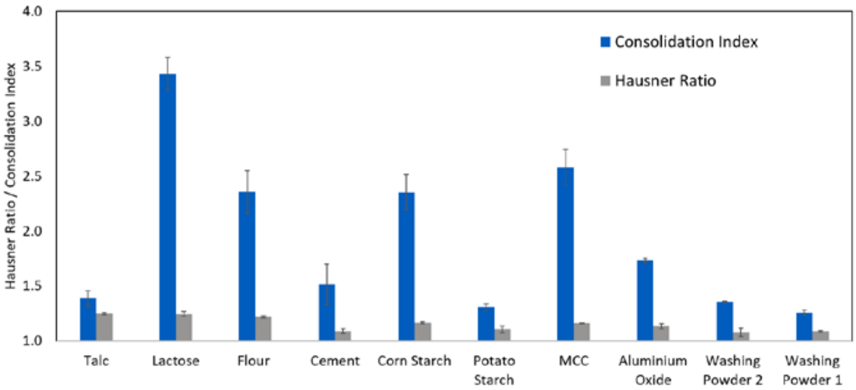
All tests were carried out in triplicate. The Consolidation Index is calculated using the following equation:

$$Consolidation\ Index = \frac{Consolidated\ Energy}{Basic\ Flowability\ Energy}$$

CBD and the BD_{Tap50} were then used as the Poured Density and Tapped Density respectively to calculate Hausner Ratio.

Interquartile Range (IQR) was used to quantify spread in the data. IQR represents the middle (50%) spread of the data. A low IQR indicates a low spread and therefore limited differentiation between samples. In order to ensure a fair representation, data was normalised prior to calculating the IQR.

METHOD 1: CONSOLIDATION INDEX AND HAUSNER RATIO



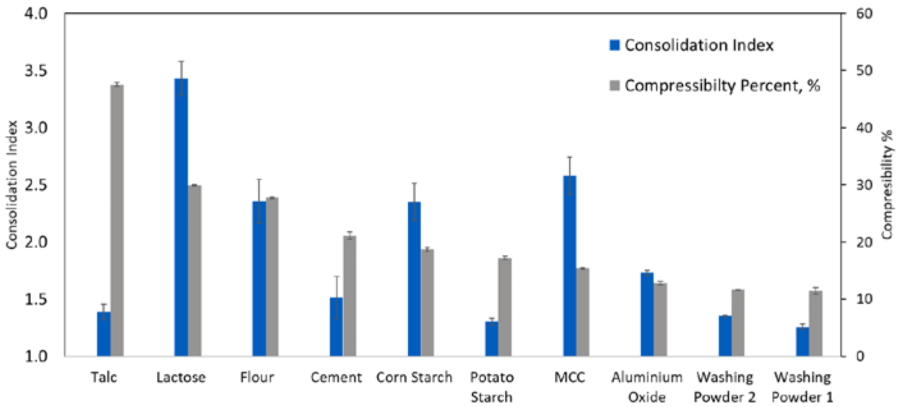
Comparing the 10 different samples, there is a wider variation with Consolidation Index (IQR = 1.0) compared to the Hausner Ratio (IQR = 0.1). This demonstrates the lack of sensitivity associated with using Hausner Ratio to compare extremely different types of materials.

Based on Hausner Ratio, three samples are in the 'Fair' category (Talc, Lactose and Flour) three are 'Good' (Corn Starch, MCC and Aluminium Oxide) and the remaining four are 'Excellent' (Cement, Potato Starch, Washing Powder 1 and Washing Powder 2).

Reviewing the Consolidation Index, there are four samples (Lactose, Flour, Corn Starch and MCC) which are significantly more sensitive to tapping or vibration with a Consolidation Index > 2.

In general, comparing different metrics from the same type of consolidation method highlights expected trends, for example. Lactose has the highest Hausner Ratio and also the highest Consolidation Index. However, there are exceptions, Talc, for example, has a relatively high Hausner Ratio but a low Consolidation Index. Of the materials studied, none exhibited an increase in density greater than 25%, whereas the Flow Energy of several samples increased by more than 200%. With materials such as Lactose, the change in packing efficiency results in more particle-particle interactions and therefore particle morphology starts to dominate the flow behaviour. The changes in bulk density alone do not necessarily inform on the flow of the consolidated material in a given process.

METHOD 2: DIFFERENCE BETWEEN CONSOLIDATION METHODS



Comparing the different consolidation methods, Consolidation Index (tapping) and Compressibility Percent (direct pressure) rank the powders differently. For example, Talc is more sensitive to direct pressure, which could represent long term storage issues, whereas Lactose is most sensitive to tapping, which simulates vibration during transportation or processing. These different responses are likely due to variations in particle properties and packing structure: fine, cohesive powders are likely to agglomerate, entrain more air and therefore be more sensitive to compression. Rough irregular particles may pack efficiently and therefore not compress significantly but when forced to rearrange, their morphology can significantly inhibit flow. This highlights the need for characterising samples using a method which is relevant to the processes in which they will be used and the conditions they will be exposed to.

CONCLUSIONS

Powder flowability is not an inherent material property but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched, and it is not uncommon for the same powder to perform well in one process but poorly in another. Multi-faceted characterisation provides an essential foundation for understanding the variable behaviour of powders, enabling the properties that are most relevant to in-process performance in any unit operation to be identified and quantified.

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[1] Freeman R., Measuring the flow properties of consolidated, conditioned and aerated powders – A comparative study using a powder rheometer and a rotational shear cell. Powder Technology, 25-33, 174, 1-2, 2007