Measuring and Understanding Powder Flow and Powder Behaviour

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An Introduction to Powder Characterization

- Powders are used extensively throughout a range of industries
- Thousands of different formulations
- Hundreds of different manufacturing processes
- Powder characteristics will influence manufacturing and the quality and properties of final product






Introduction

- Powders are used extensively…… but still their behaviour is poorly understood
- Results in stoppages, downtime, out of spec product…… rework or scrapped material
- Expensive & inefficient use of resource
- How to address these problems…… by measuring and understanding powder behaviour!

The Nature of Powders

Particles are complex, and variable

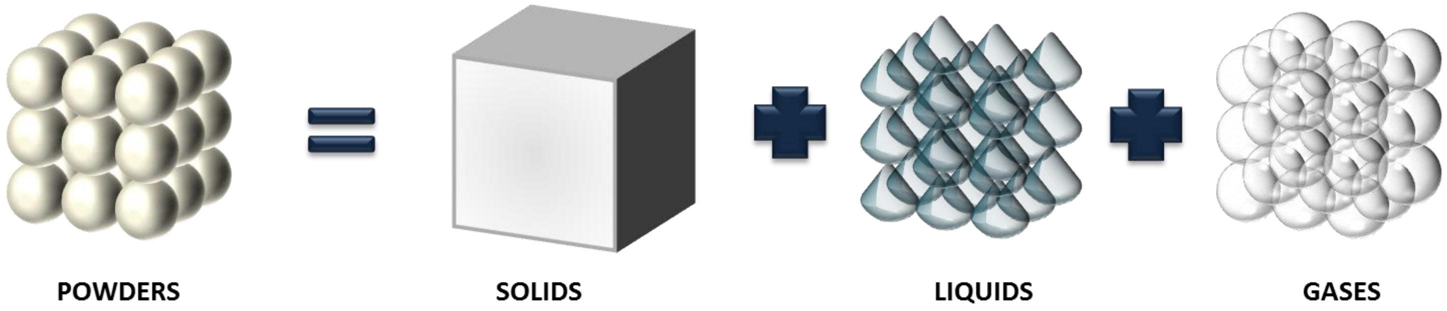
Each particle defined by a set of physical and chemical properties

- Particle Size & Distribution
- Shape
- Surface Texture
- Surface Area
- Density
- Cohesion
- Adhesion
- Elasticity
- Plasticity
- Porosity
- Potential for electrostatic charge
- Hygroscopicity
- Hardness / Friability
- Amorphous content

Each will contribute to how the powder behaves!

The Nature of Powders

- Powders are bulk materials, made from:
  - Solids (the particles)
  - Liquid (water on the surface of the particle, in the particle or in the air between particles)
  - Gas (normally air, between particles)



Bulk powder “behaviour” is complex and will depend on how these three phases interact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C2031
50mm x 160ml No. 2336
FT Powder Rheometer

What do we mean by powder behaviour?

- Powder behaviour = fn (size) + fn (shape) + fn (stiffness)
  + fn (porosity) + fn (surface texture) + fn (density)
  + fn (cohesion) + fn (adhesion) + …….

… but also the environmental conditions the powder is exposed to:

- Consolidation
- Aeration
- Humidity level
- Extent of Shear / Strain
- Equipment surface material……

No mathematical way of predicting behaviour from primary properties

If there are 12 variables, each with just 4 permutations, this gives over 16 million combinations!

The FT4 Powder Rheometer

Methodologies available with the FT4 Powder Rheometer

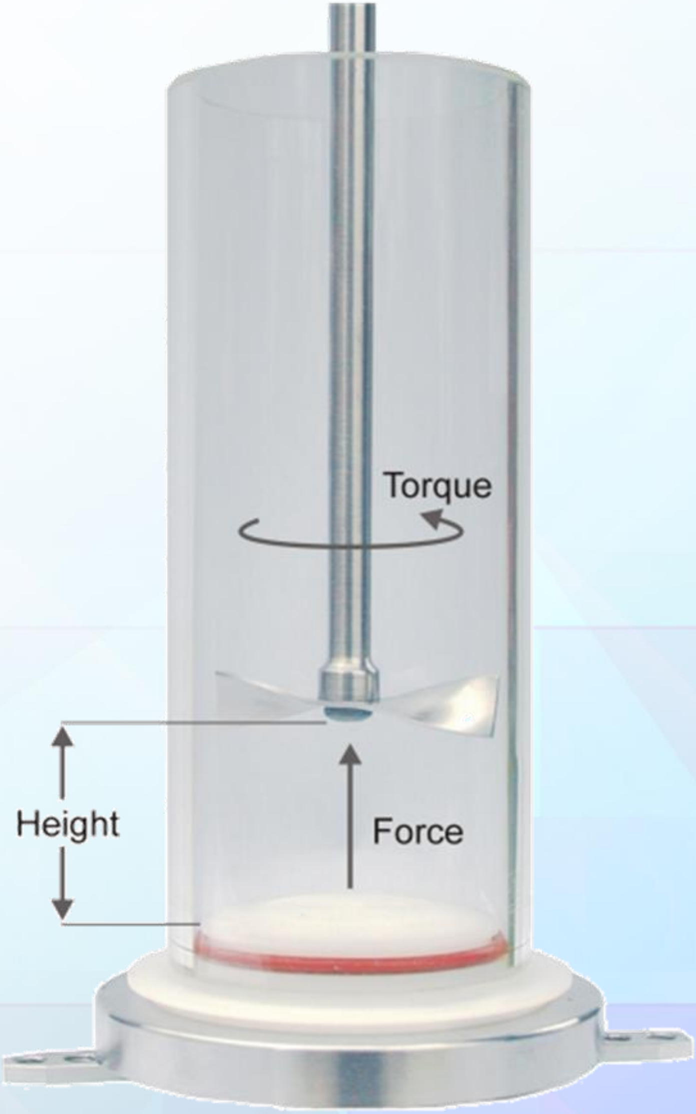
- **Bulk**
  - Density
  - Compressibility
  - Permeability
- **Dynamic Flow**
  - Basic Flowability
  - Aeration
  - Consolidation
  - Flow Rate
  - Specific Energy
- **Shear**
  - Shear Cell
  - Wall Friction
- **Process**
  - Segregation
  - Attrition
  - Caking
  - Electrostatics
  - Moisture
  - Agglomeration

FT4 Principle of Operation

Blade rotates and moves down and up through powder at a defined helix angle and speed

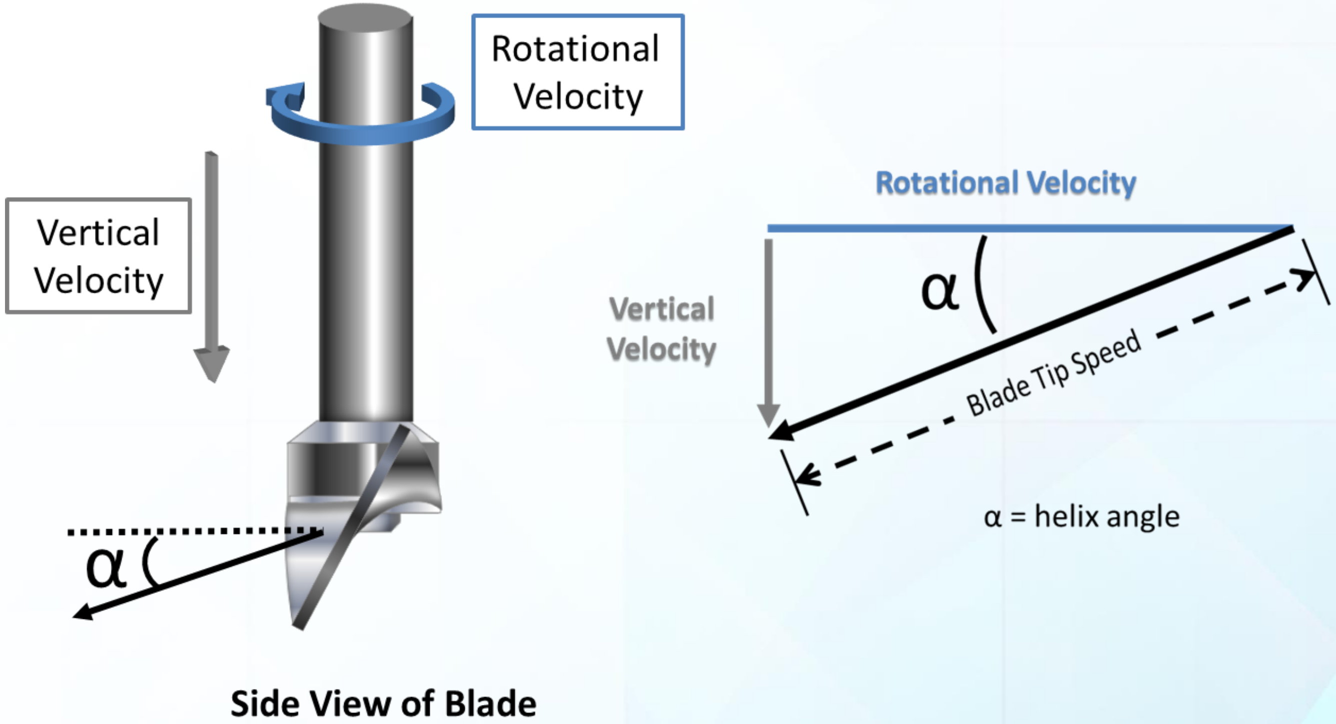
Measured Parameters are:

- Torque
- Force
- Height



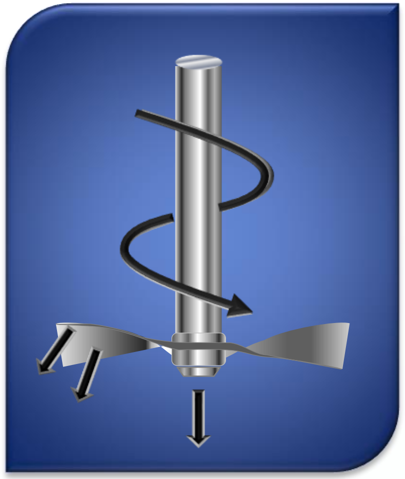
Clockwise Downwards Flow Pattern

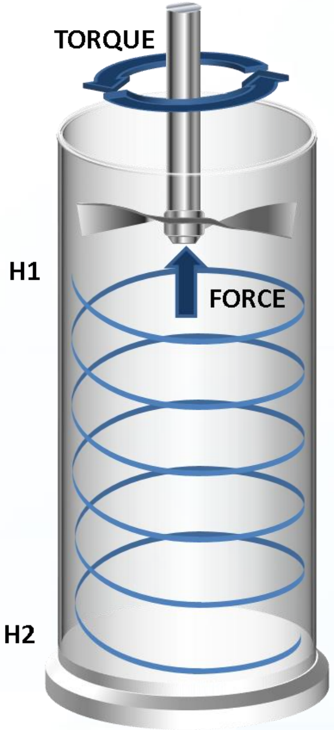
(typical Conditioning mode)



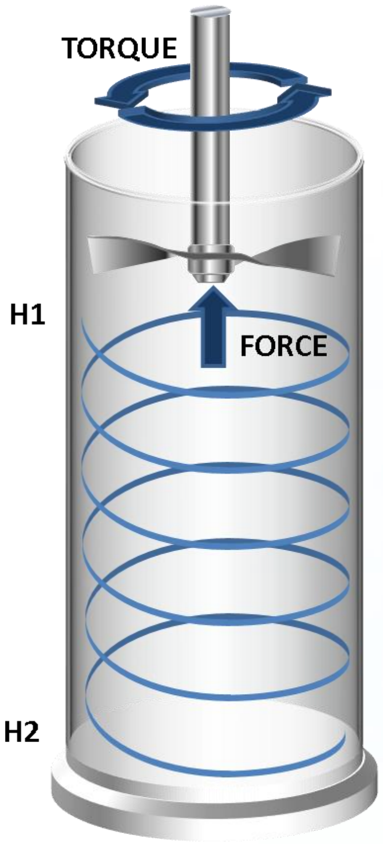
Gentle slicing flow pattern to remove stress or excess air
- results in homogenous, low stress packing state

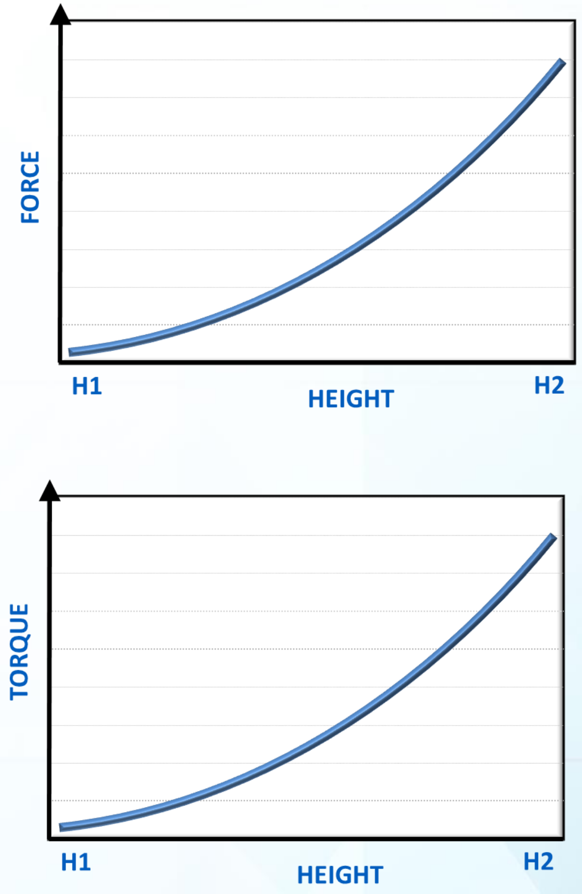
Dynamic testing





Torque & Force as a function of Height



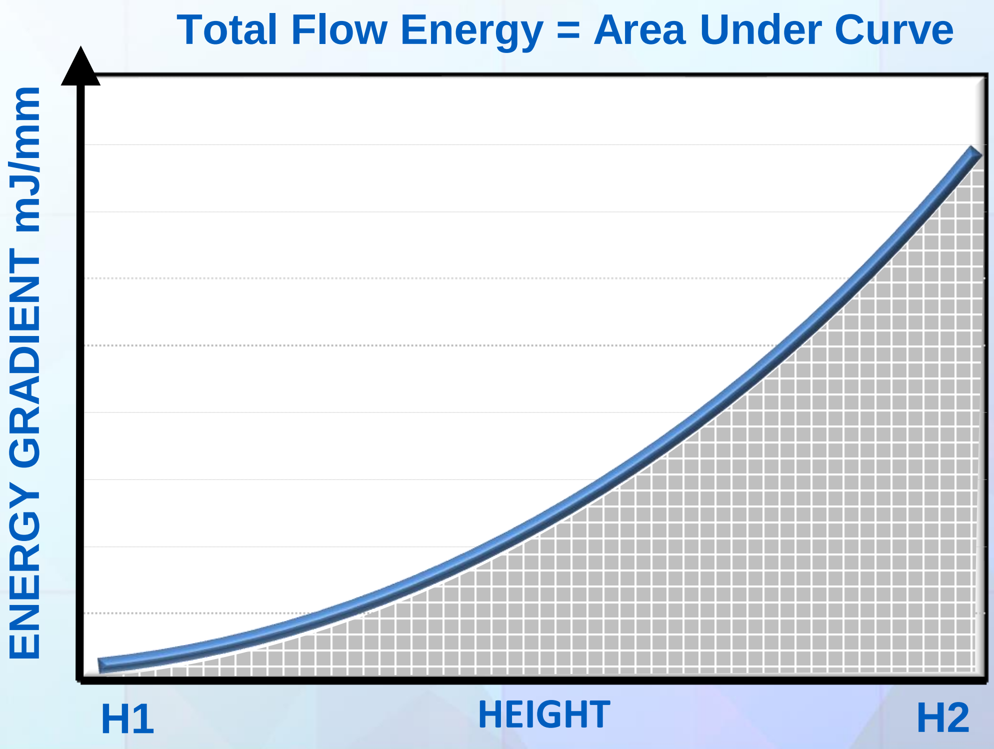


How to represent both resistances as a single parameter…

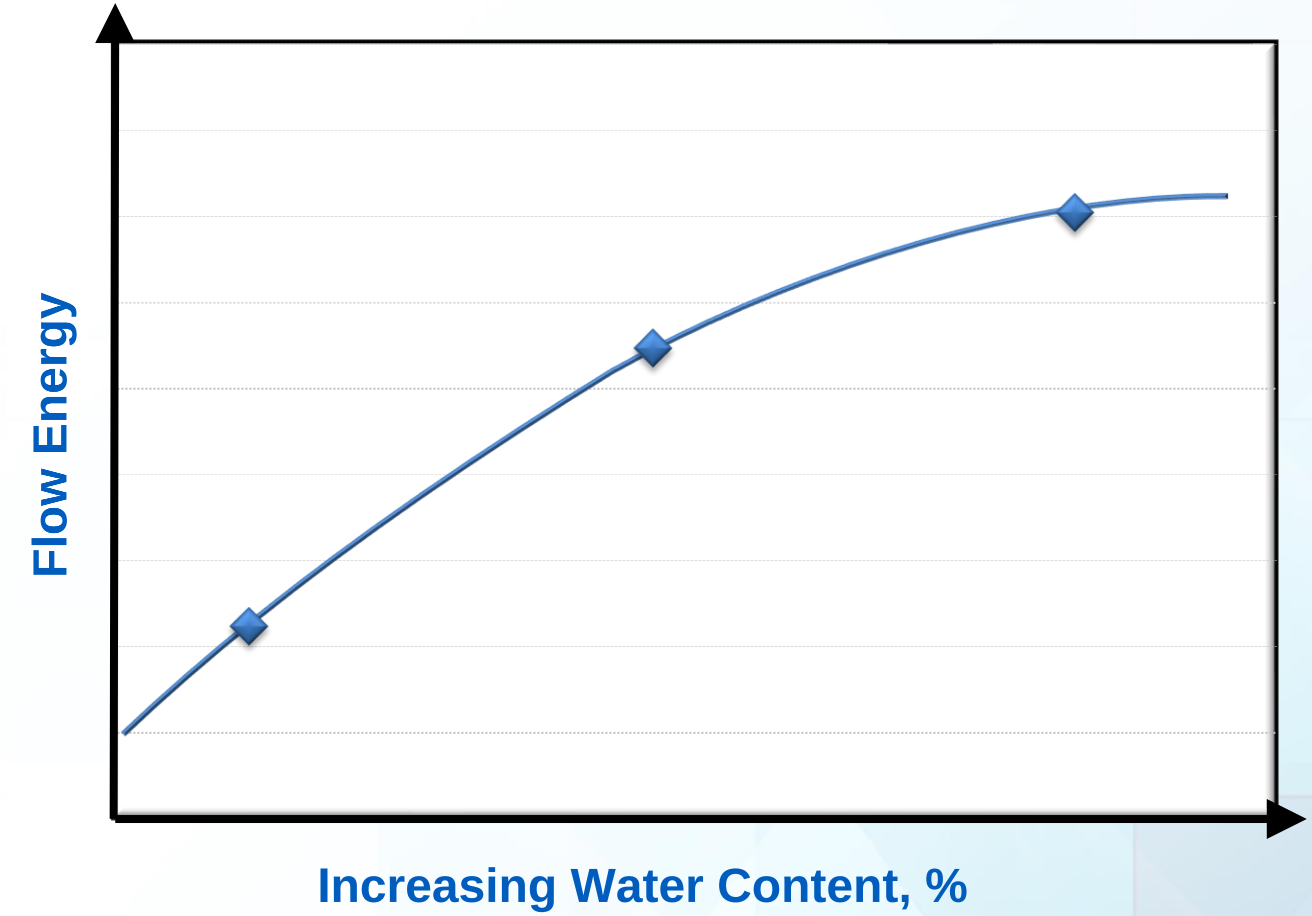
….. *the calculation of Flow Energy*

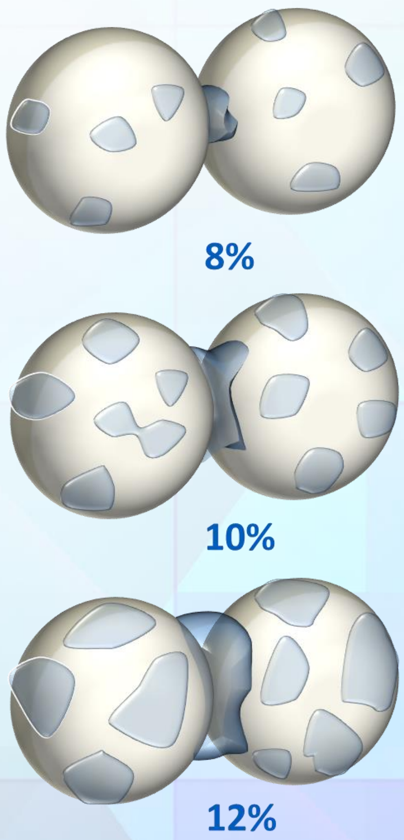
Work Done = Energy = "Force" x Distance = (Force + Torque) x Distance

Energy Gradient = Work Done per mm

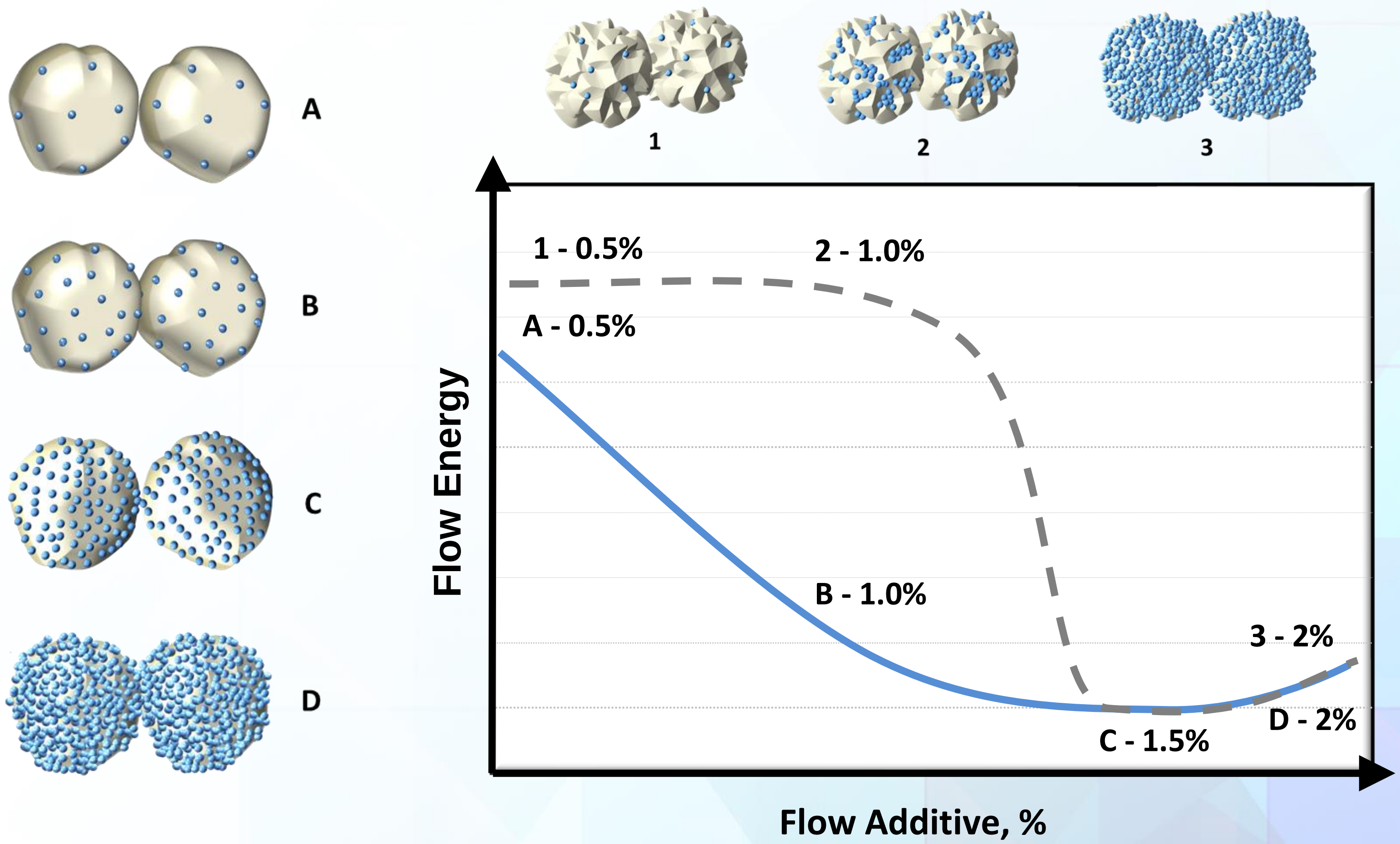


Flow Energy vs. Water Content





Basic Flow Energy vs. Additive & Morphology

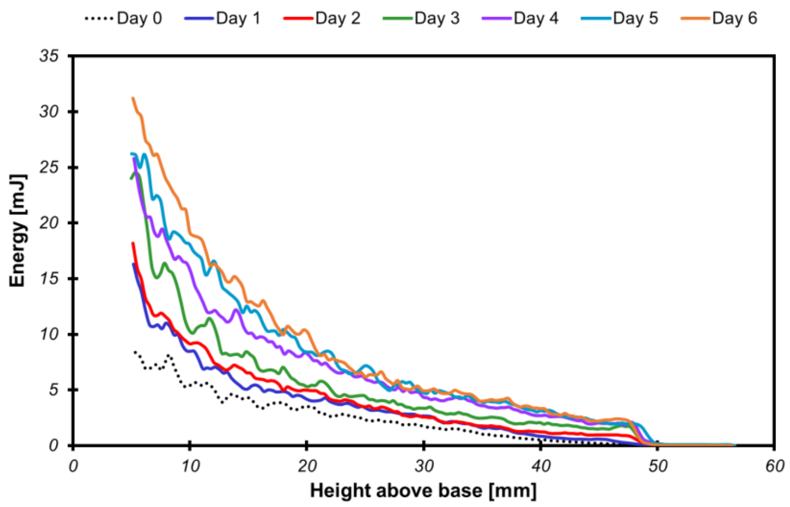


CASE STUDY - CAKING

Non-Uniform Caking

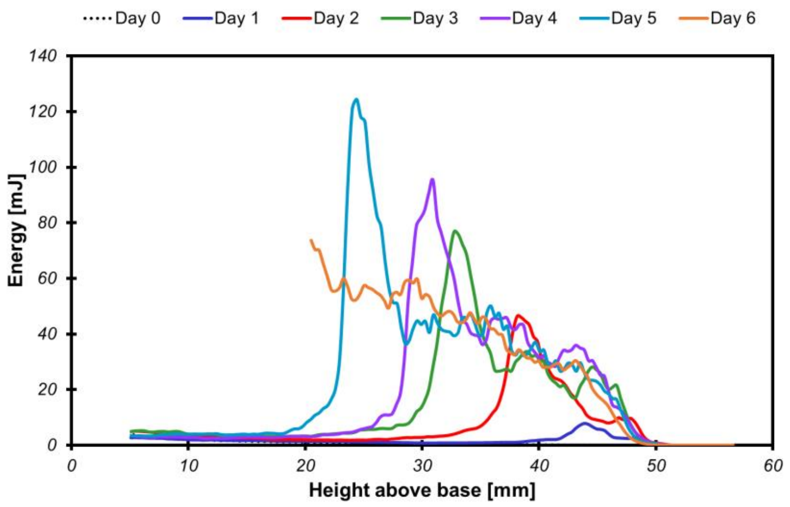
- Not all powders cake uniformly through out the powder bed
- Powders may exhibit non-homogeneous or hybrid behaviour
- It is not possible to quantify this behaviour using other testing methodologies that are used to investigate caking, e.g.
  - Shear Cell
  - Penetrometer
  - Uniaxial Testers
- Combination of measurement and data capture provides a unique method for identification caking modes and the impact on flow

Homogeneous Caking - Sorbitol



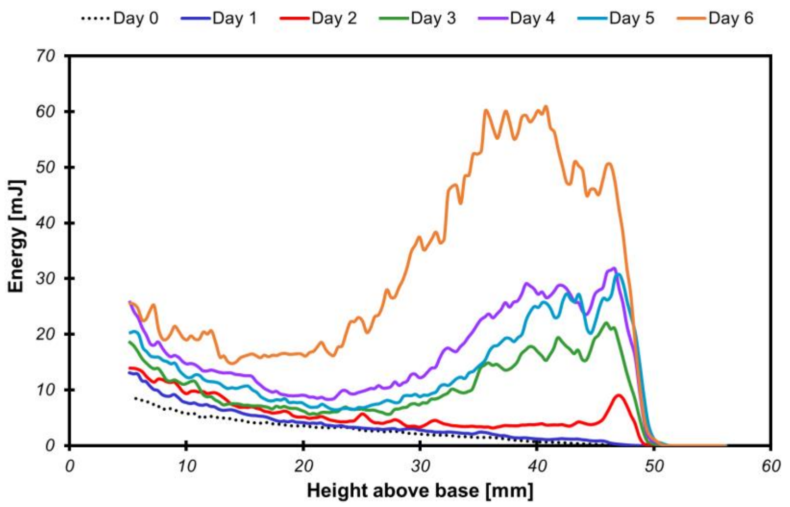
Flow Energy increases uniformly throughout the powder bed with time

Non-Homogeneous Caking / Crusting - SMP



Increase in Flow Energy is confined to a localised region which may expand or move with time

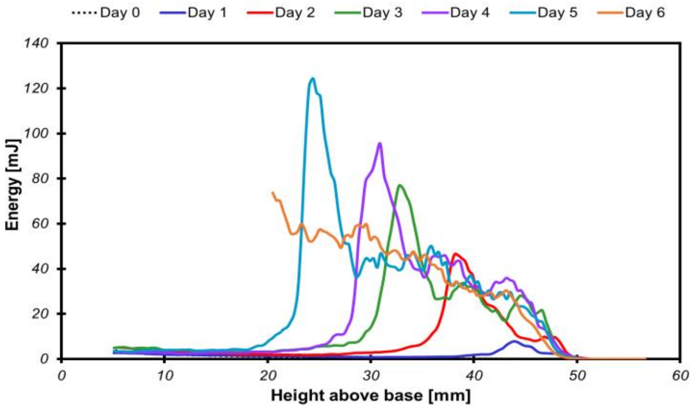
Hybrid Caking - SME



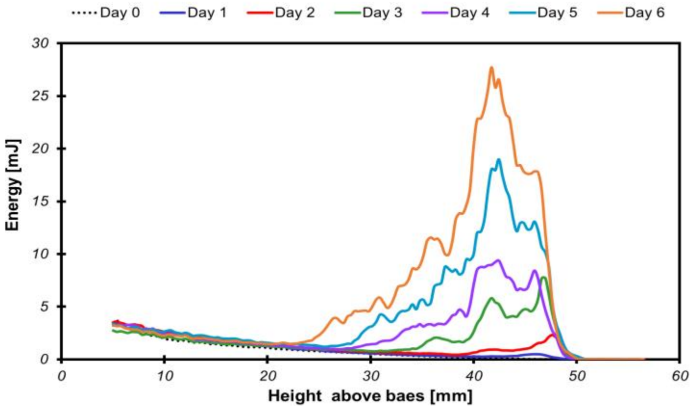
Increase in Flow Energy primarily occurs in a localised region but with smaller increases occurring thoughout powder bed.

Non-Homogenous Caking: Skimmed Milk Powder

SMP stored at 75%RH

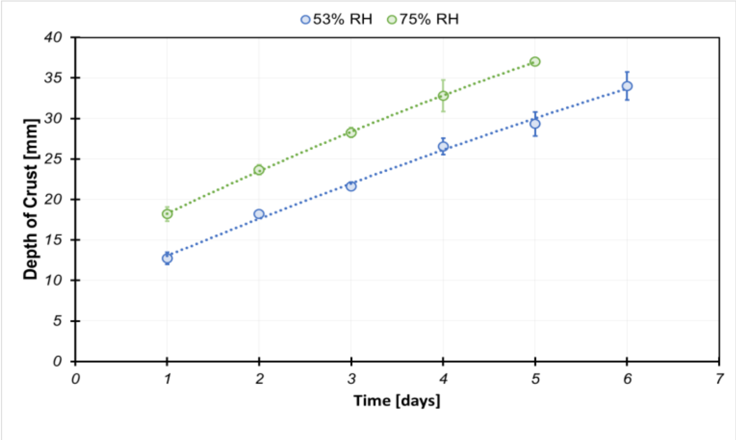


SMP stored at 53%RH

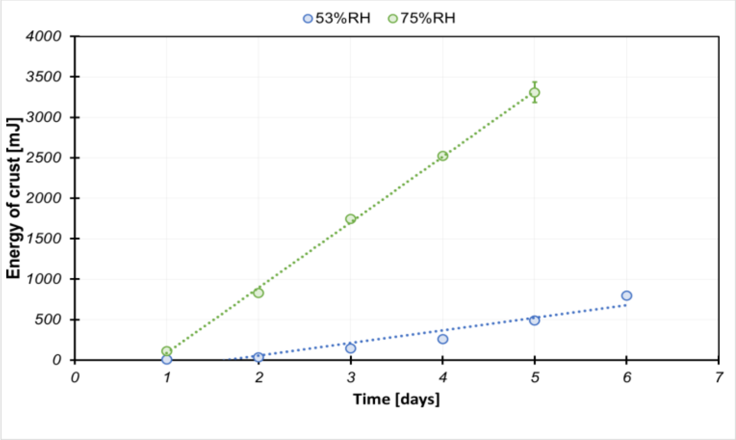


Analysis of the raw data allows the progression of the cake, and its resistance to flow to be tracked over time

Depth of Crust

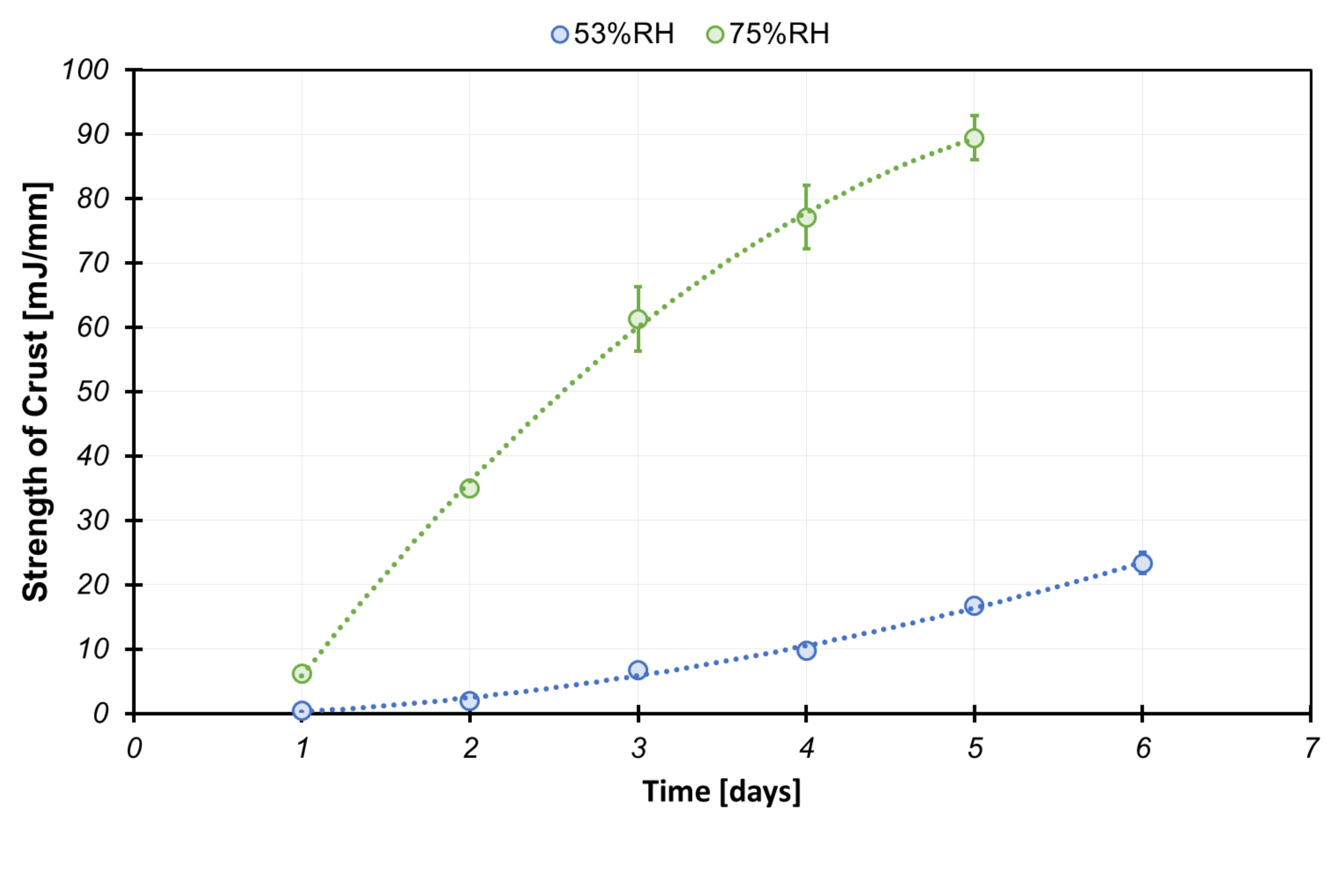


Total Energy of Crust



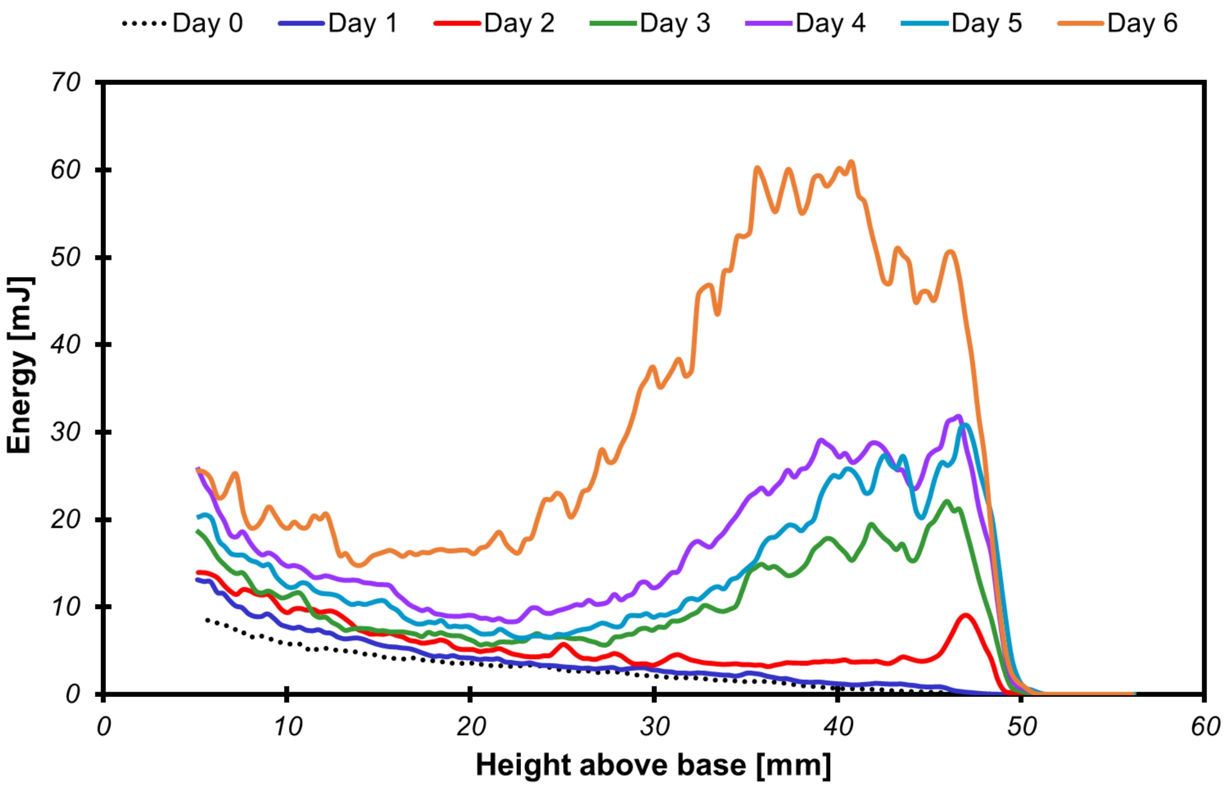
Strength of Crust can also be derived by dividing Energy of Crust by Depth of Crust

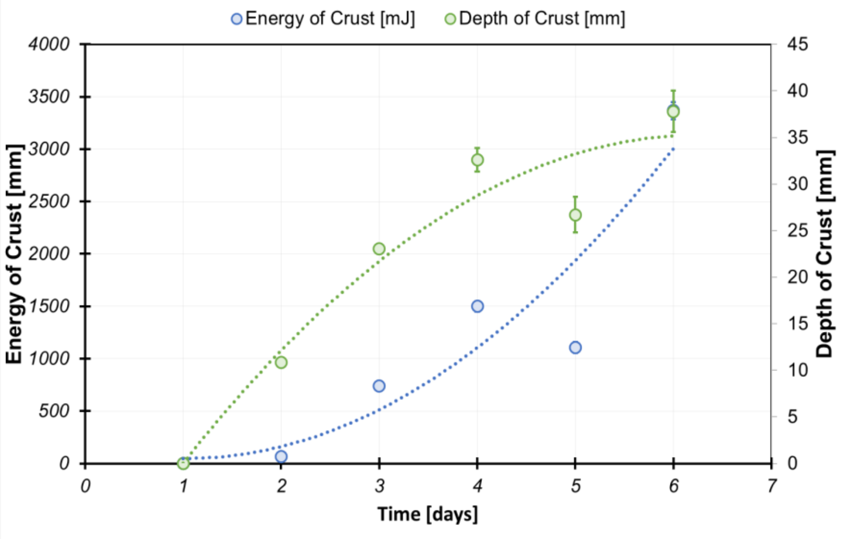
From this data it can be ascertained if the increase in Energy of Crust is solely a function of expansion of the caked region (no change in strength) or if the caked region hardens with time

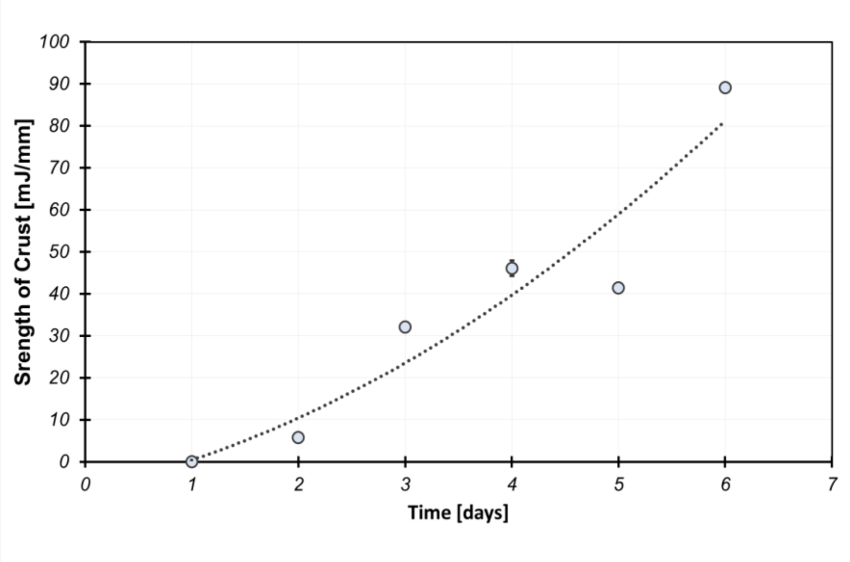


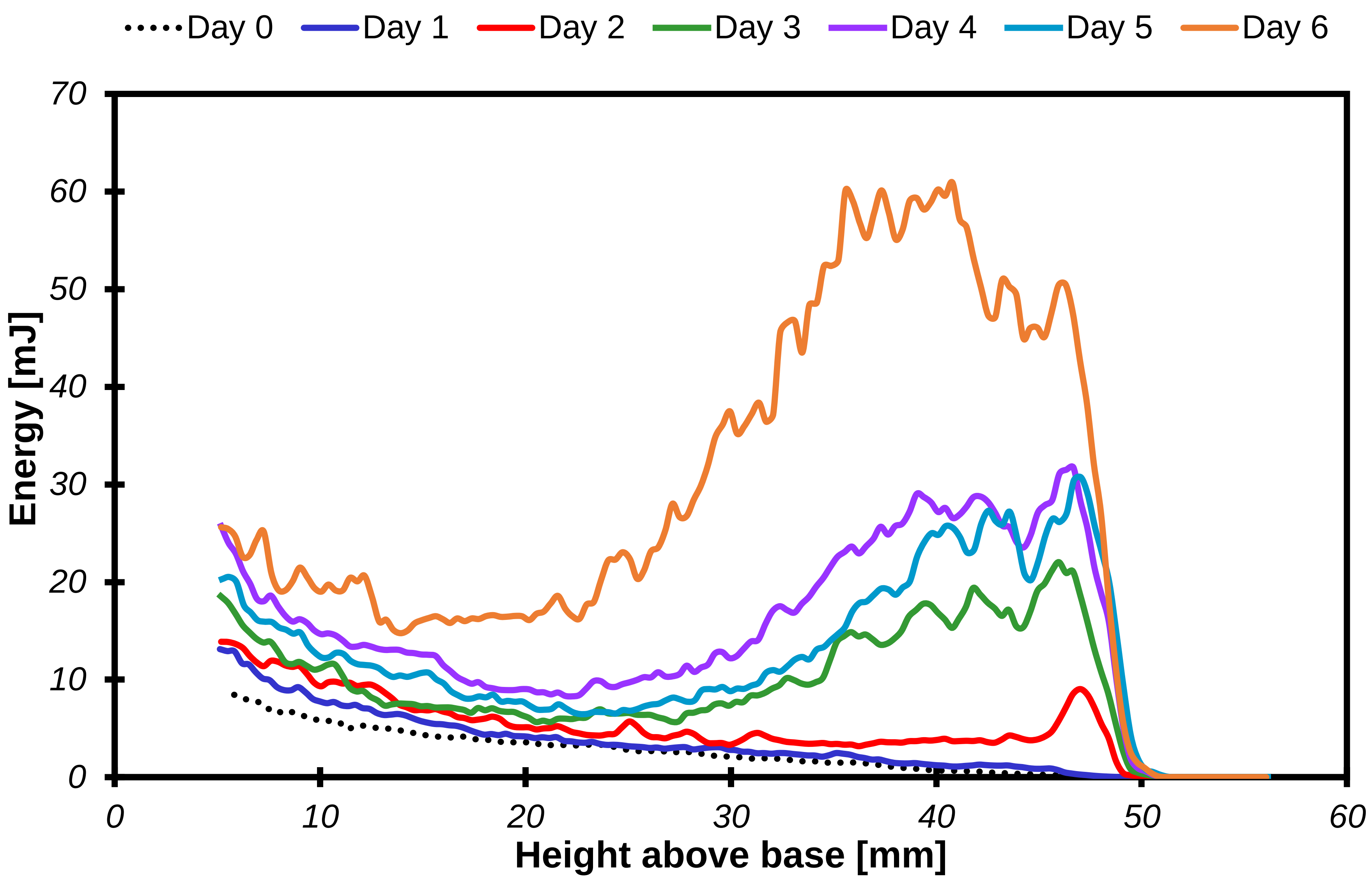
At both humidity levels, SMP exhibits an increase in Strength of Crust with time, demonstrating that the crust hardens following the initial caking phase

Hybrid Caking: Sulphonated Methyl Ester @ 75% RH







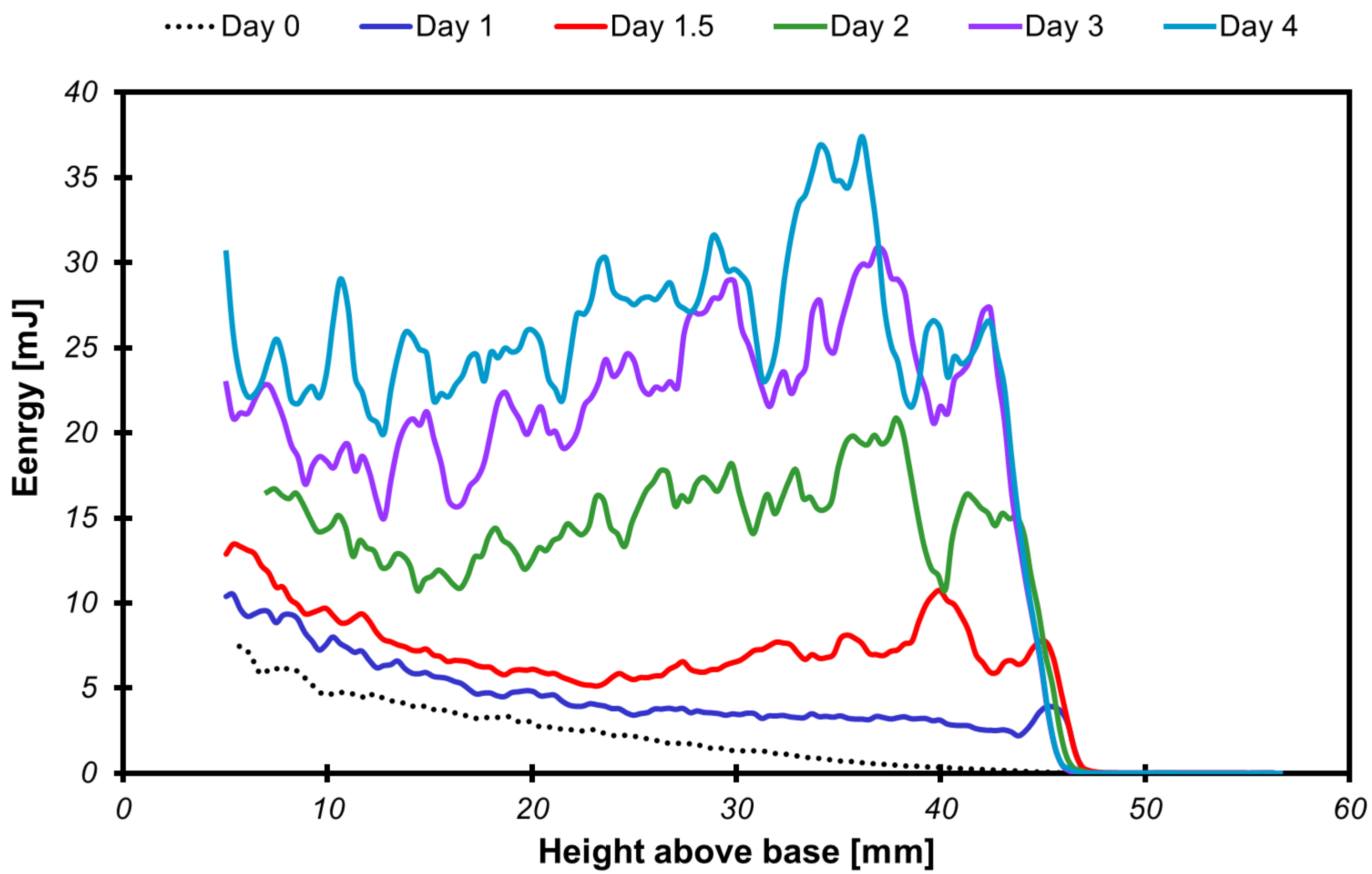


Day 0
Day 1
Day 2
Day 3
Day 4
Day 5
Day 6
Energy [mJ]
70
60
50
40
30
20
10
0
0
10
20
30
40
50
60
Height above base [mm]

Translational Behaviour: Food Flavouring

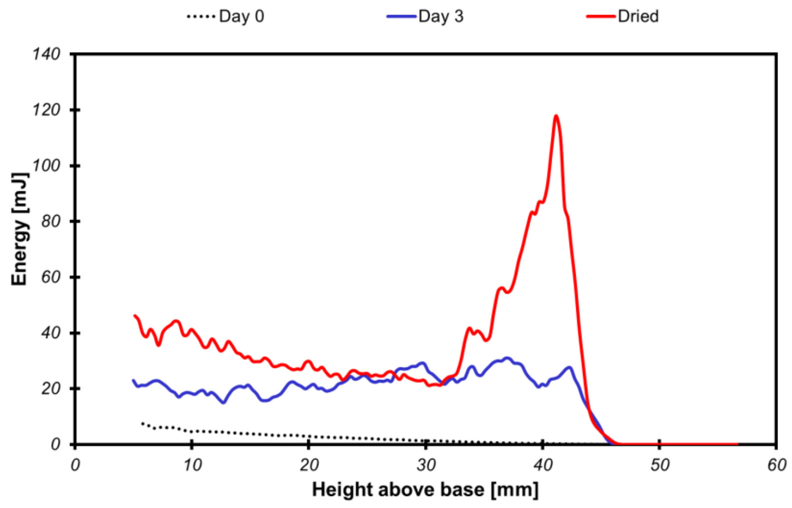
In some case, powders may transition between different caking modes.

After 1.5 to 2 days at 75% RH, this food flavouring exhibits hybrid behaviour. After this point, more homogeneous behaviour is observed.

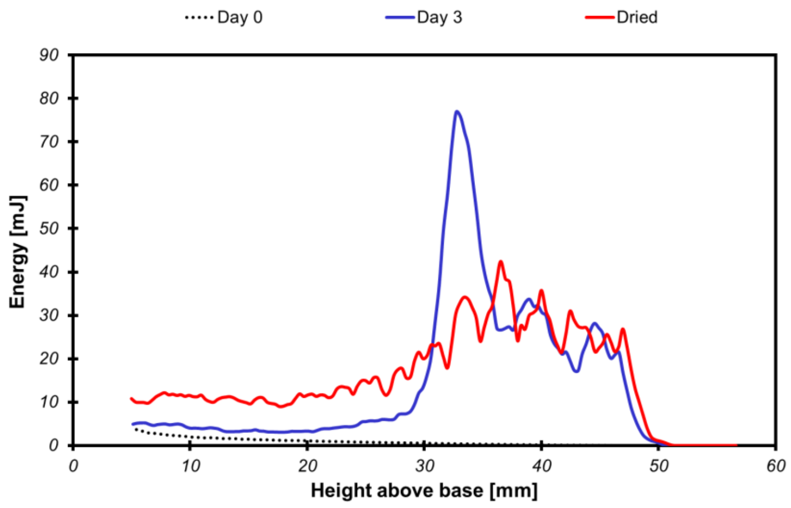


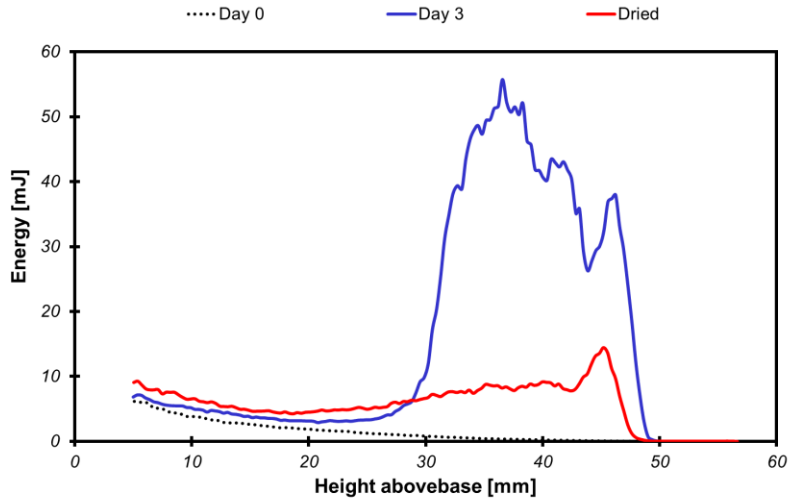
Humidity Cycling

- Powder rheometry can also be used to explore the complex caking behaviour exhibited by powders when subjected to alternating humidity
- Several samples were stored at 75% RH for 3 days before being returned to ambient conditions (33% RH) for a further 3 days
- As demonstrated by the results, each showed a different response to the change in humidity



Flavour 1 showed a significant increase in Flow Energy across the powder bed when returned to ambient, particularly at the powder/ air interface





Flavour 2 presented a marked reduction in energy when returned to ambient conditions. although it did not return its initial state

SMP: the degree of caking at the top of the powder/air interface remained unchanged, however the peak dissipated leading to increased caking below the crust

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Conclusions

- Powder interaction with humidity is a complex phenomenon manifesting in a range of different caking behaviours.
- In order to fully understand caking behaviour, variations in flowability across the powder bed should be quantified, rather than assuming that caking has occurred uniformly.
- The ability of the FT4 Powder Rheometer to measure the resistance to flow at multiple points through the powder bed makes dynamic testing the ideal tool for quantification of caking behaviour.

Further reading: Measurement and quantification of caking in excipients and food products with emphasis on the non-homogeneous interaction with ambient moisture, Brockbank *et al*., Particuology, 2021.

CASE STUDY – SACHET FILLING

Introduction

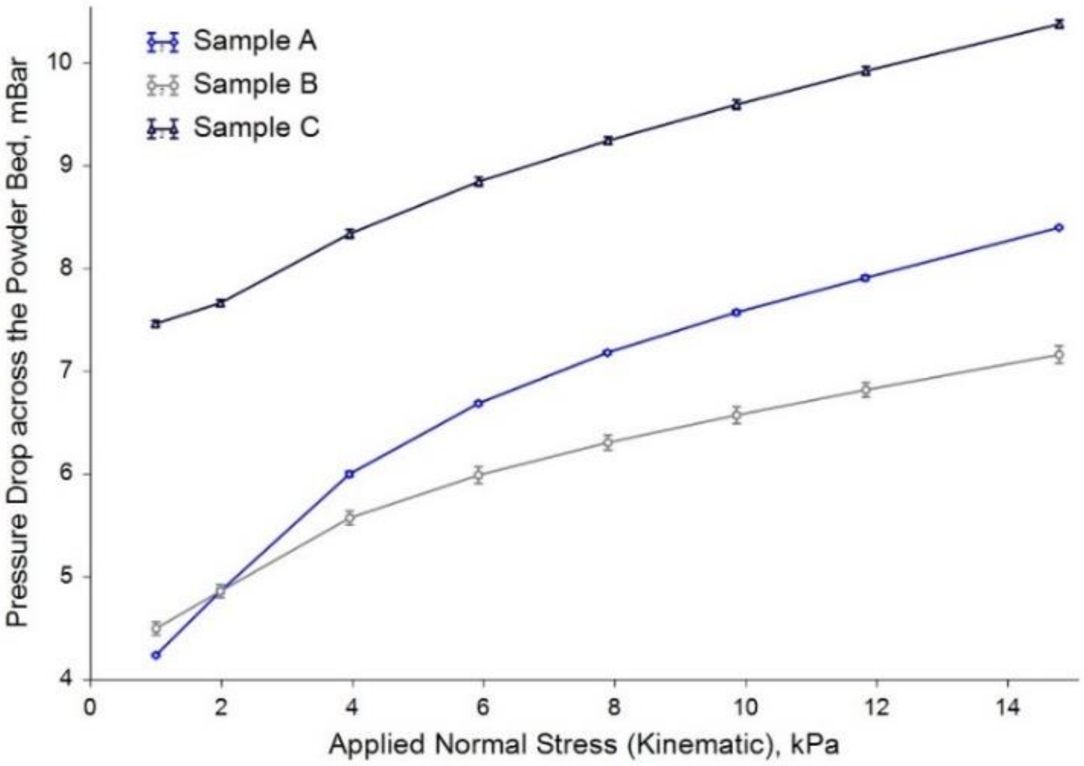
- Sachets allow small quantities of powder to be presented to the consumer in a manageable format.
- It is imperative that the sachet filling process produces consistent, uniform fill throughout an entire production run, with low weight variation and high content uniformity.
- Significant deviation can carry both financial risks, and in the case of pharmaceutical powders, endanger patient health.
- These considerations also apply to other filling operations across various scales, e.g. IBCs, sacks, capsules, dies/moulds.

Relative Performance of Different Blends

- Three batches of a pharmaceutical blend were used in a sachet filling operation.
- The three batches flowed differently from the filling shoe into the sachet, resulting in significant weight variation.
  - Sample A exhibited good performance and Sample B (with a wider particle size distribution) was classed as average.
  - Sample C had the same particle size distribution as Sample A, but performed very poorly in the process, suggesting that particle size alone did not dictate performance.
- The samples were analysed using an FT4 Powder Rheometer® to identify differences between the samples that would explain the varying in-process performance.

Bulk Testing - Permeability

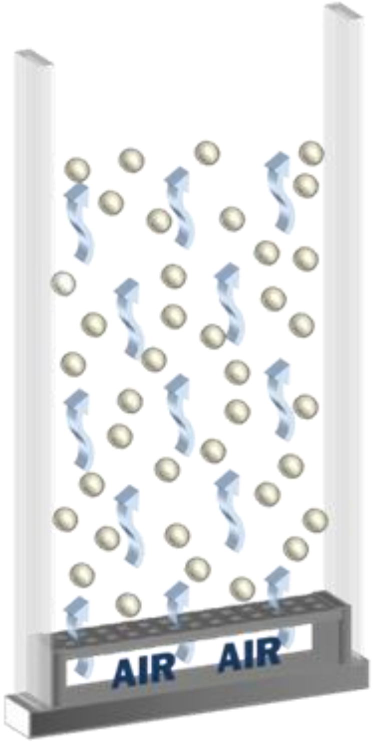
- Sample C generated the highest Pressure Drop (lowest Permeability), indicating the greatest resistance to the passage of air.
- Low Permeability means that air entrained in the powder cannot escape when it enters the sachet, leading to high weight variation across a manufacturing batch.
- Sample A was less permeable than Sample B, suggesting that an extreme value for any parameter may result in sub-optimal performance.



Dynamic Testing – Aerated Energy

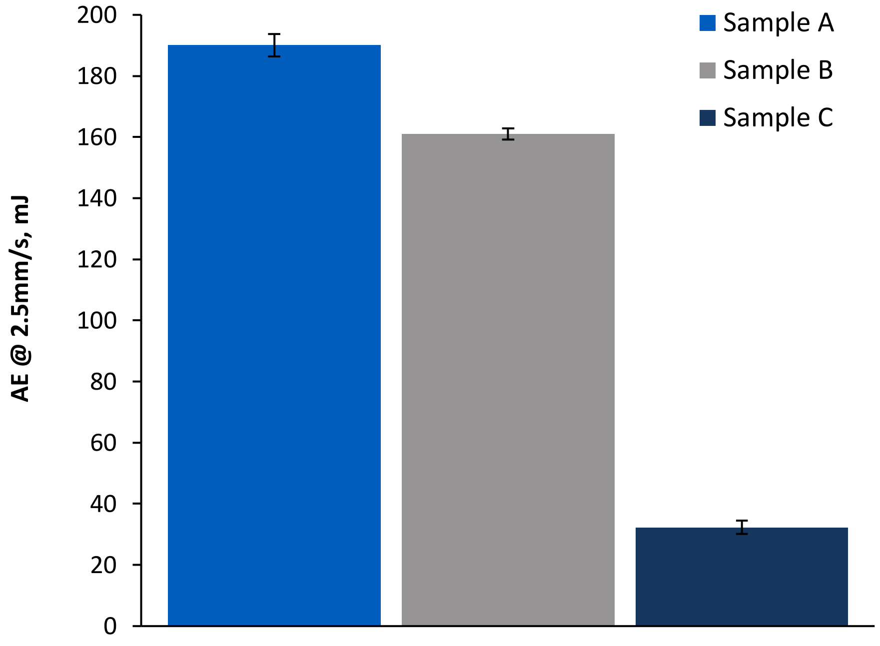
- As air is introduced to a powder, the flow of the gas lifts and separates the particles, reducing inter-particular interactions and the overall resistance to flow.

- The degree to which particles separate is a reliable indicator of the strength of the cohesive bonds.



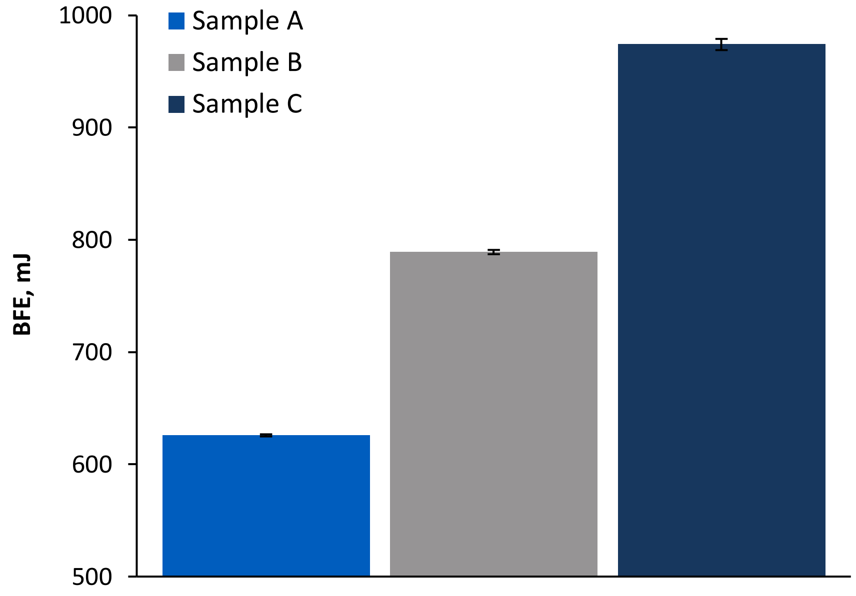
Dynamic Testing – Aerated Energy (AE)

- Sample A generated the highest AE, which is likely a consequence of its high permeability.
- Highly permeable powders allow air to traverse the bed readily with little influence on its packing structure.
- In contrast, Sample C generated the lowest AE, likely as a result of its lower permeability.
- Powders that are sensitive to aeration may also be more prone to segregation and dusting, which can both have a detrimental impact on content uniformity.



Dynamic Testing – Basic Flowability Energy (BFE)

- Sample A generated the lowest BFE, requiring less energy to move the blade through the powder.
- Sample C generated the highest, indicating greater resistance to dynamic, confined flow.
- Low BFE is indicative of a powder that is able to flow more freely under the forced flow conditions present in a shoe feeder operation.

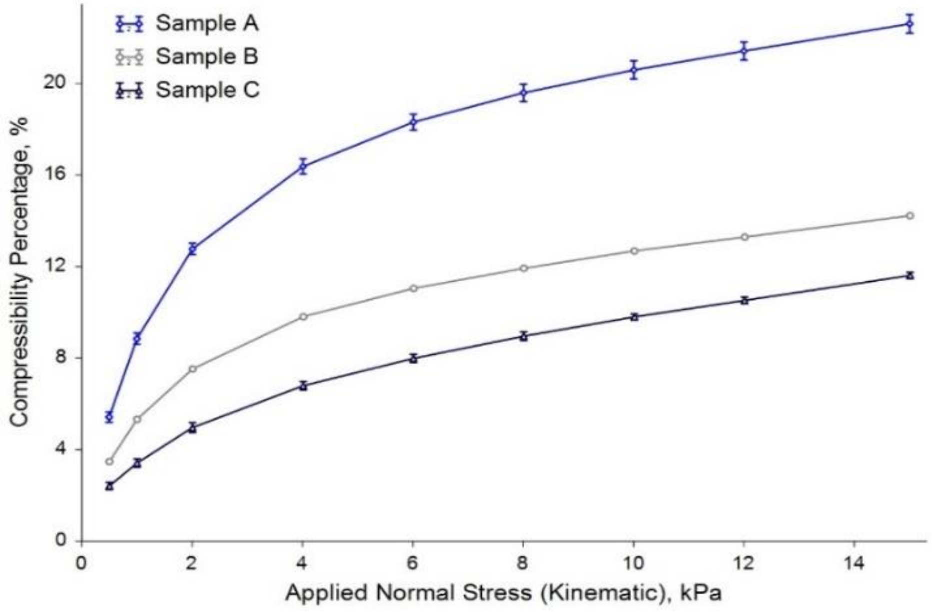


Bulk Testing - Compressibility

- Sample A generated the highest Compressibility value, likely due to the formation of stable agglomerates.

- While high compressibility is typically associated with more cohesive powders, the presence of stable agglomerates can also promote content uniformity.

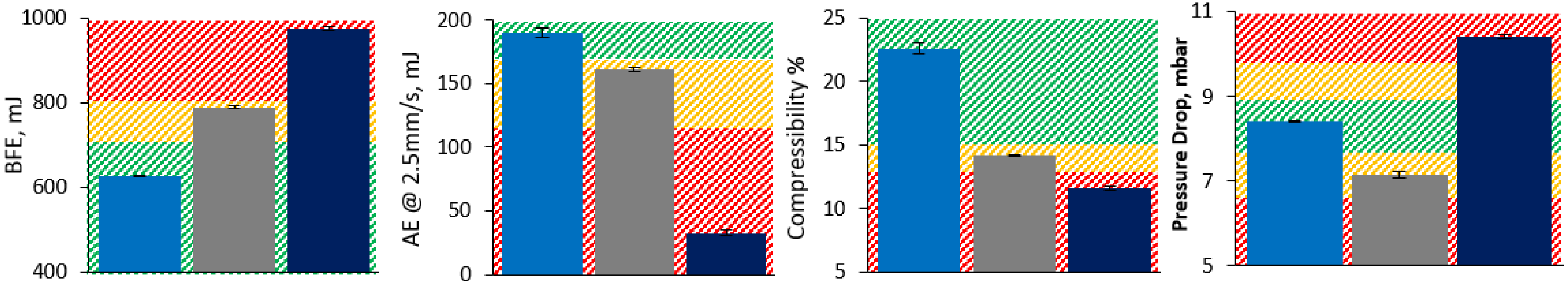
- The more compressible nature of Sample A does not appear to have a negative impact on the filling operation, probably due to the low stress conditions imposed in the process.



Design Spaces

- If a newly-tested sample has properties in the:
  - **Green** zone in each test, it can be expected that this powder will perform similarly to Sample A in the process.
  - **Amber** zone, there may be a risk of risk of poor performance and the sample should be used with caution.
  - **Red** zone, the powder is likely to perform similarly to Sample C, and prove problematic.

- Samples in the ‘red’ zone can be screened out before they enter the process, minimising poor performance thereby increasing productivity and reducing waste.



Conclusions

- The FT4’s Dynamic Flow and Bulk characterisation techniques have quantified clear and repeatable differences between three samples known to behave differently in process.
- The results demonstrate that any one single parameter may not be sufficient to fully rationalise process performance, and that a multivariate approach is required.
- The results show that powders with a low resistance to dynamic flow (low BFE), and a high Permeability (high AE, lower Pressure Drop) perform best in this operation.
- This enables a design space to be defined, against which new formulations can be assessed to predict performance.

SUMMARY

1. Powders are complex materials
2. Single number characterization, or even a single technique is not going to thoroughly describe powder behaviour in every process – multivariate analysis is required!
3. Achieving high quality of finished product requires an understanding of how the material properties and the process influence attributes of the finished product
4. Essential to identify and measure most relevant material properties in regard to in-process performance and Critical Quality Attributes (CQAs)
5. Modern instrumentation that emulates a range of process conditions, allows a database of process relevant, powder properties to be established
6. Hard earned experience (from production over many years) can now be quantified by correlating good / bad processability to powder characteristics
7. Correlation fed back into R&D to assist in new product development – the essence of QbD
8. Processing performance can be predicted with confidence, batch to batch variability can be investigated and understood
9. Productivity and product quality can be systematically enhanced through the application of the most appropriate measurements

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