Principles and Measurement of Dynamic Powder Behaviour

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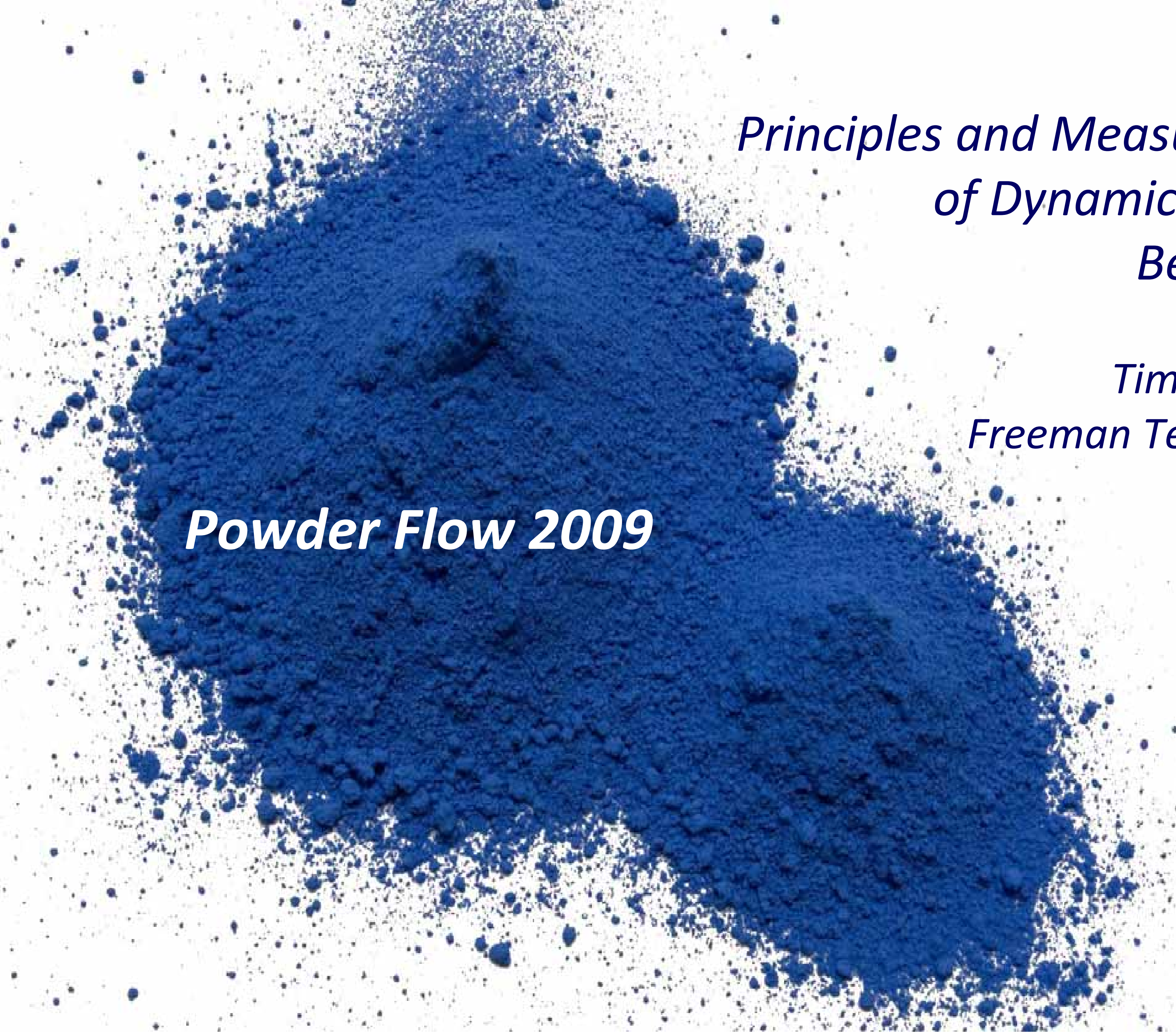
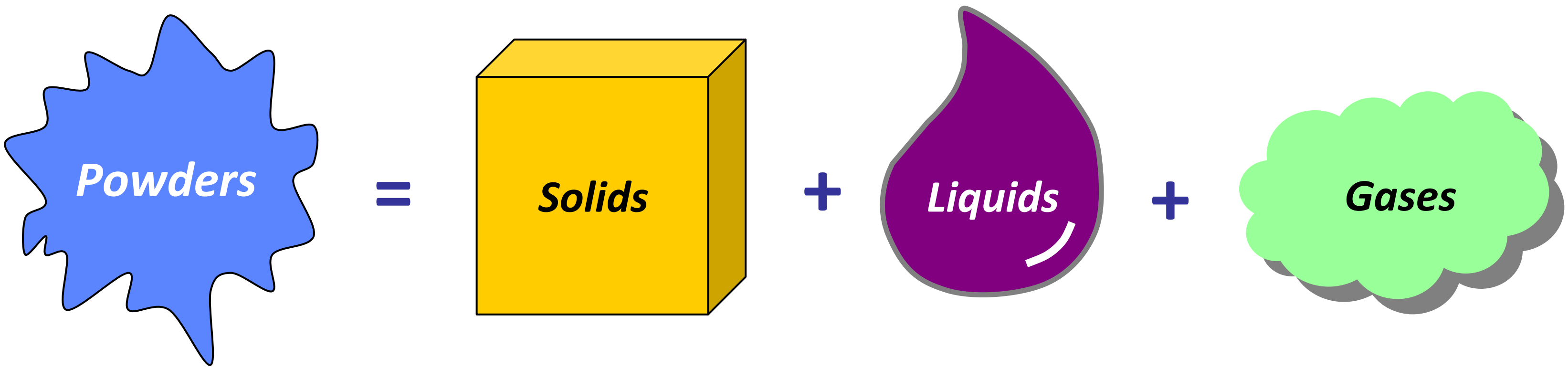
Freeman Technology



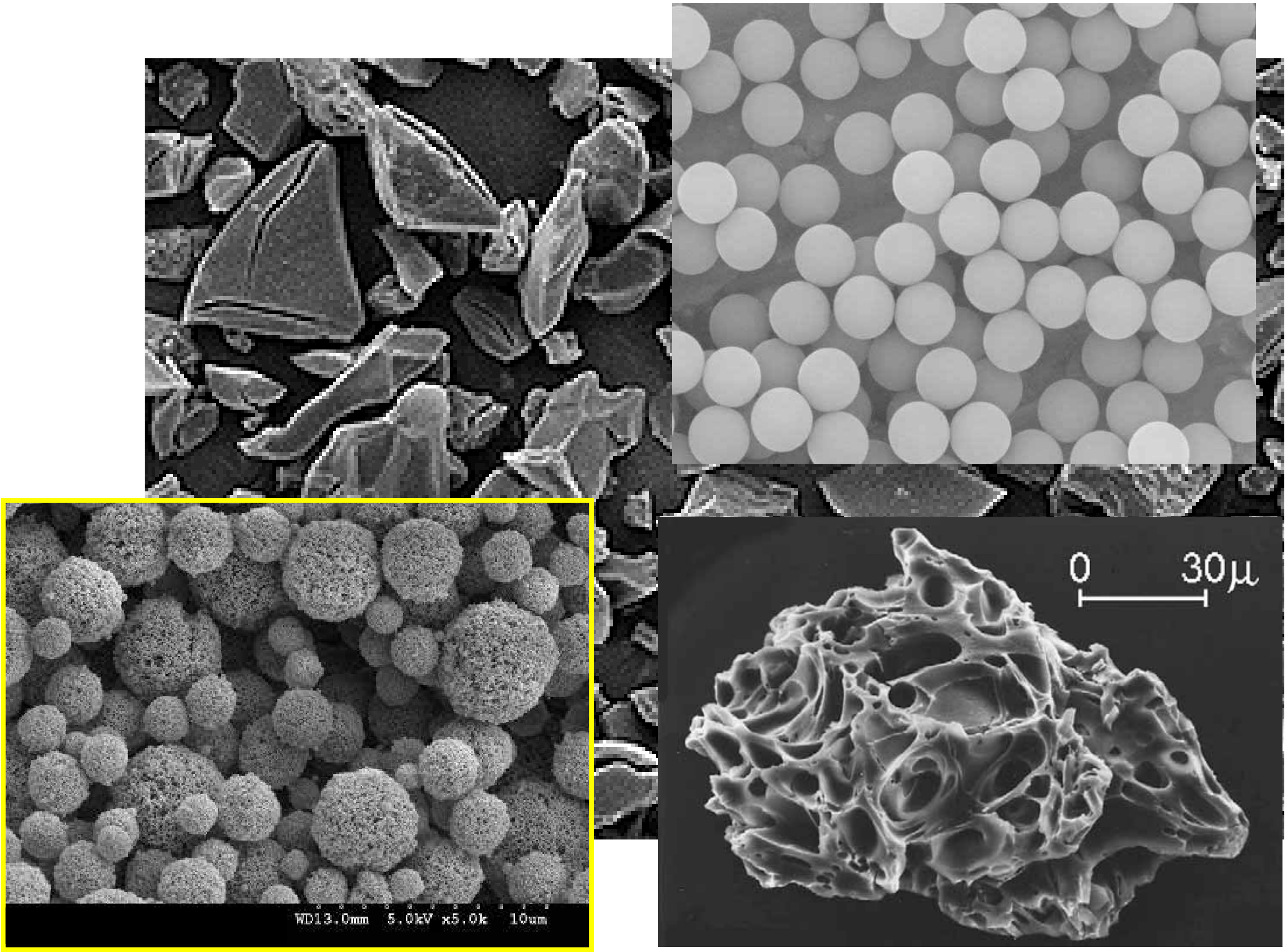
Powders are complex materials

They consist of....



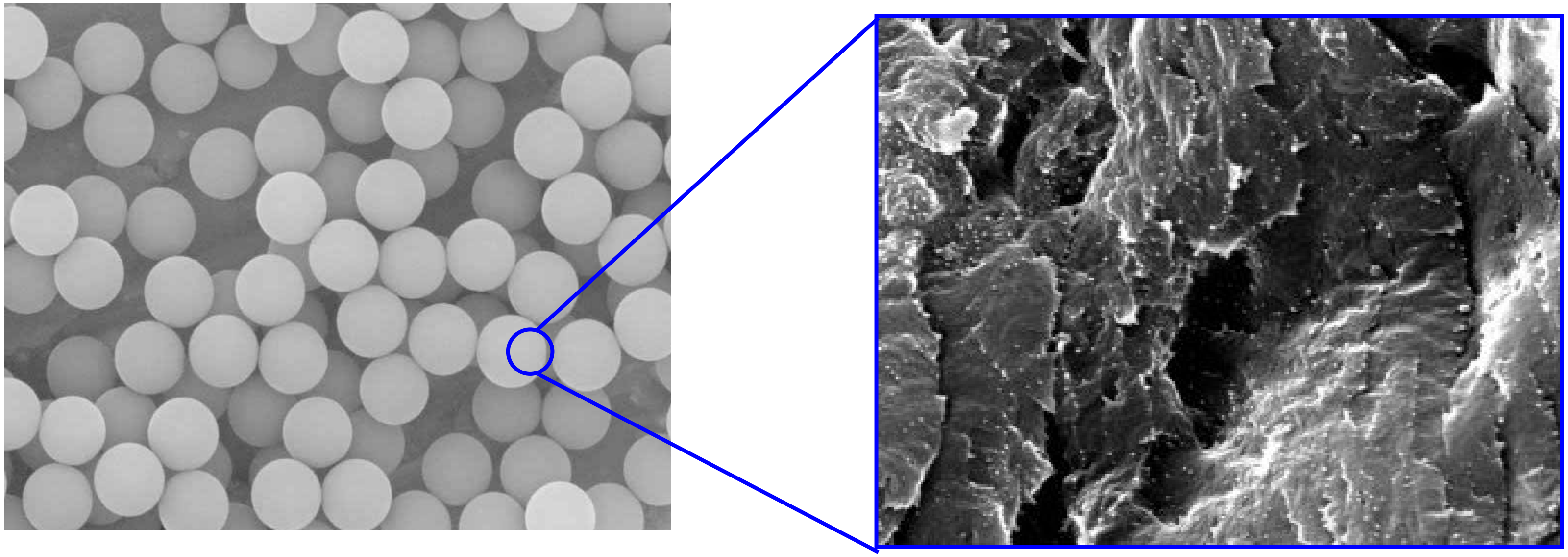
Behaviour is a function of the properties of each component and the way the components interact

SEM Images of Particles



Ref: Microparticles
SciELO
Trinity College Dublin
U.S. Geological Survey

Surface Texture



Ref: Microparticles

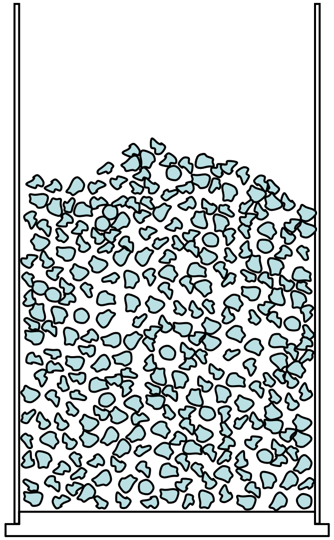
Solid components - variable and complex

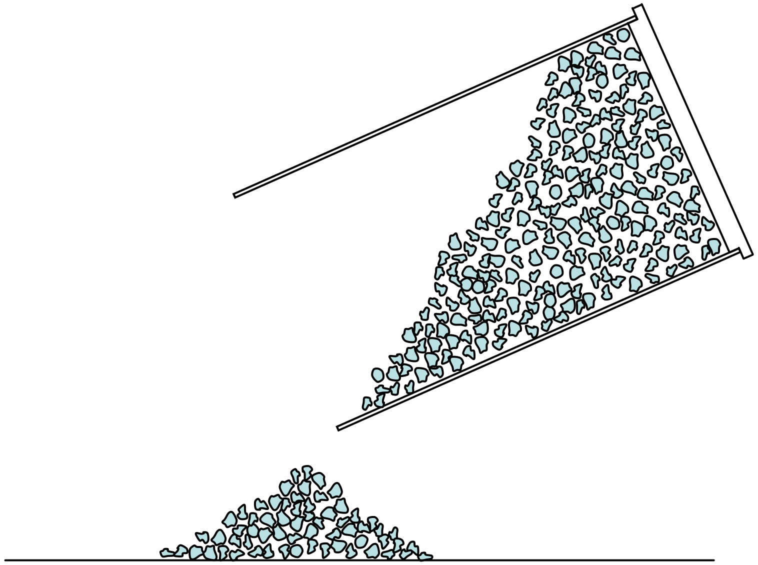
Each particle defined by a set of physical and chemical properties

- Particle Size & Distribution
- Shape
- Surface Texture
- Surface Area
- Density
- Hardness / Friability
- Hygroscopicity
- Elasticity
- Plasticity
- Porosity
- Potential for Charge
- Cohesivity
- Adhesivity
- Amorphous content

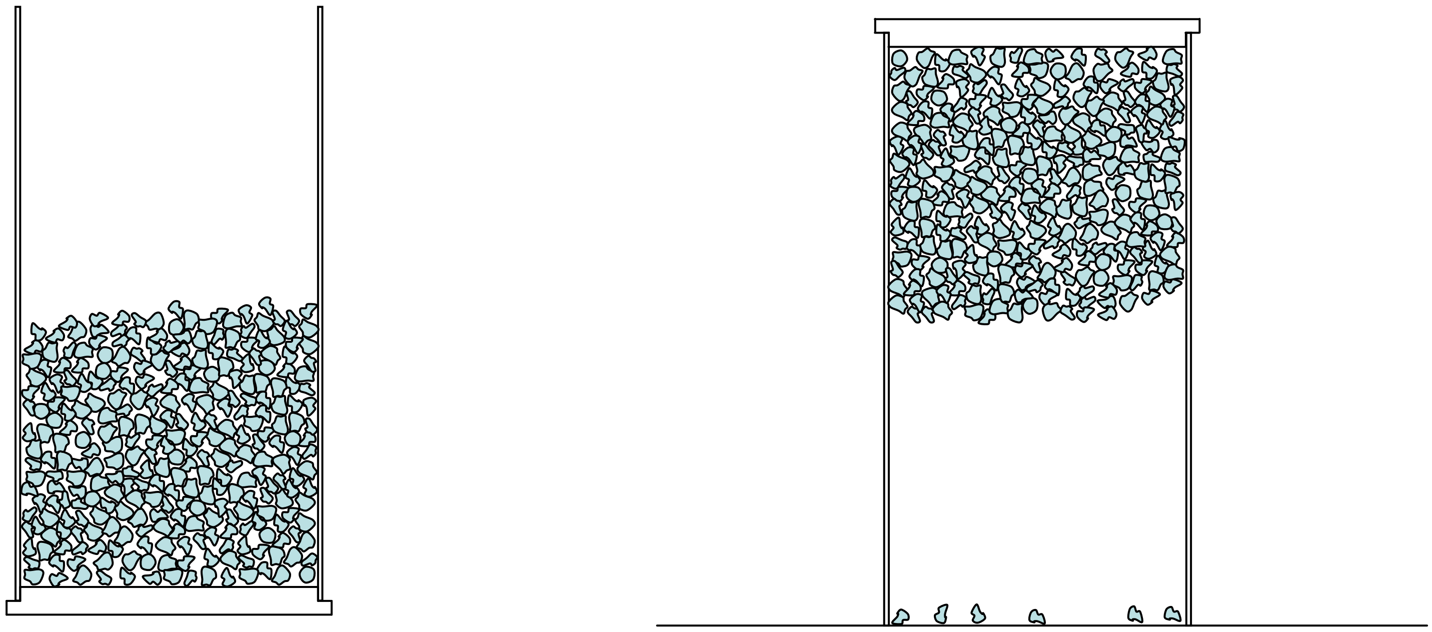
Examples of Powder Behaviour (observational behaviour)

Loose packing state

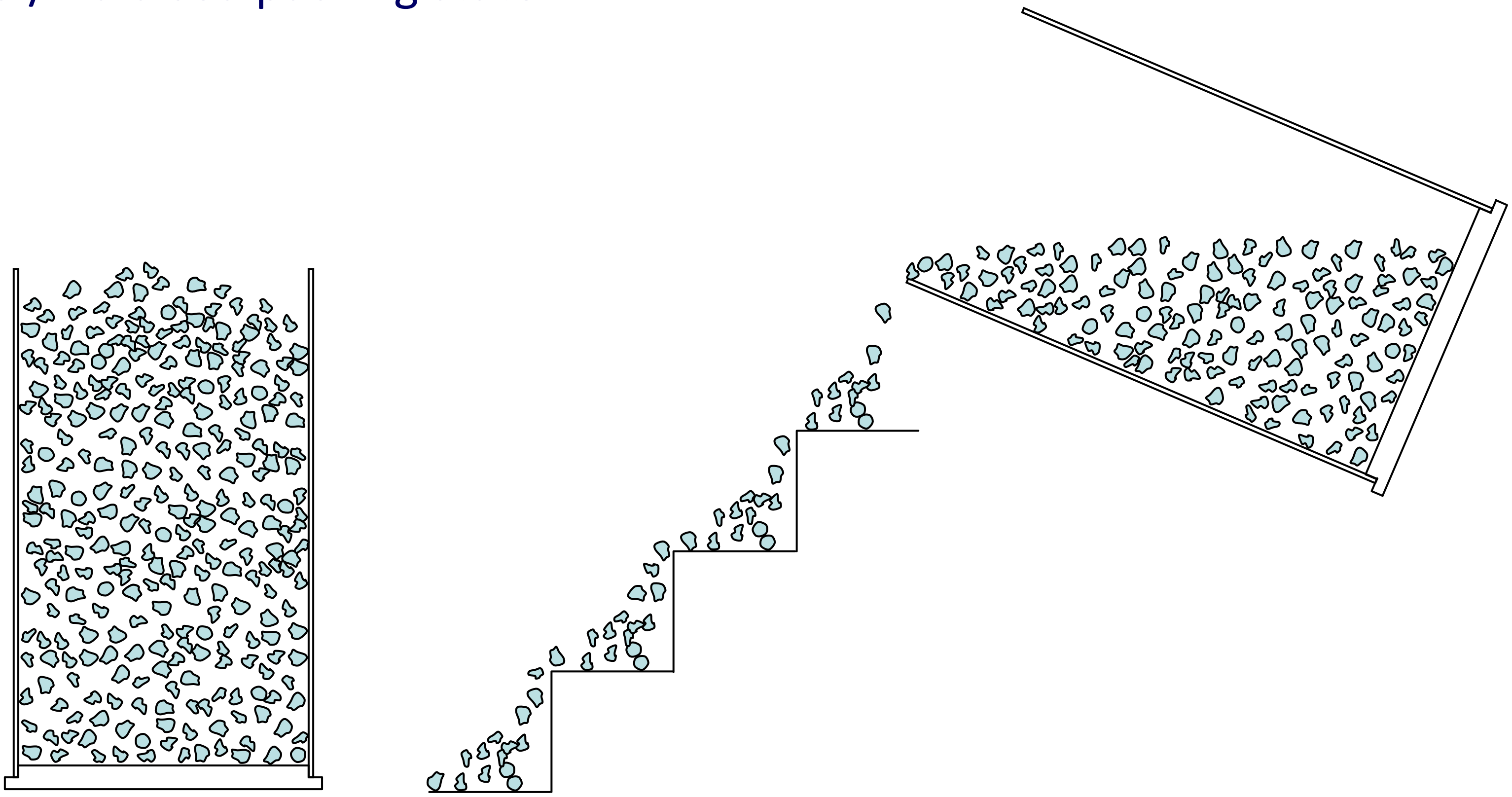




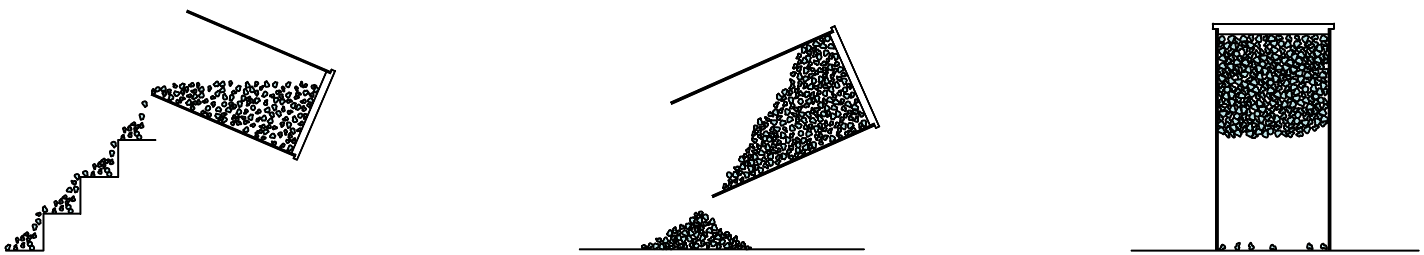
Consolidated packing state



Aerated / fluidised packing state



Variation in behaviour with level of aeration and consolidation



Fluidised → Consolidated

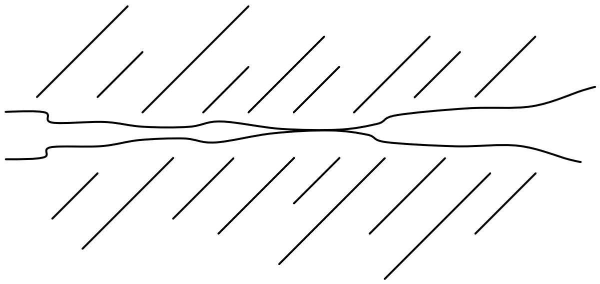
Stress: Low Moderate High

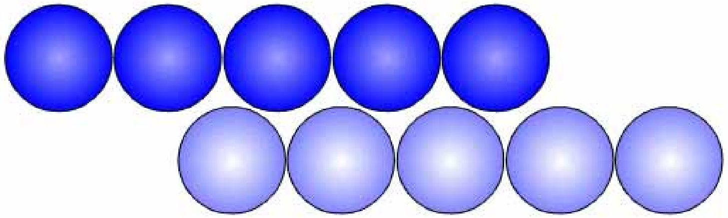
External Variables

- Consolidation
  - Vibration / tapping
  - Direct pressure - container, keg, hopper
- Aeration
  - Gravity discharge, filling, blending, aerosolisation, pneumatic transfer
- Flow Rate
  - Powder/powder, powder/container wall
- Moisture
  - Increases particle adhesion, reduces particle stiffness
- Electrostatic Charge
  - Discharge, high shear mixing, high flow rates
- Storage Time
  - Consolidation, caking

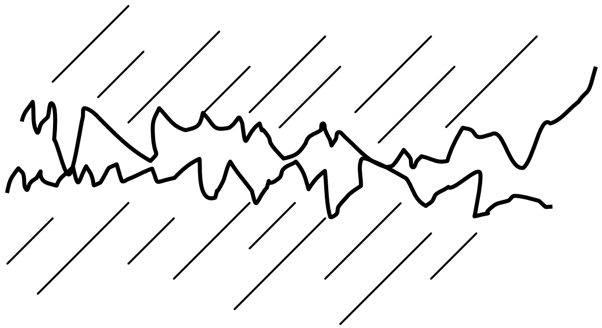
Mechanisms of Powder Flow

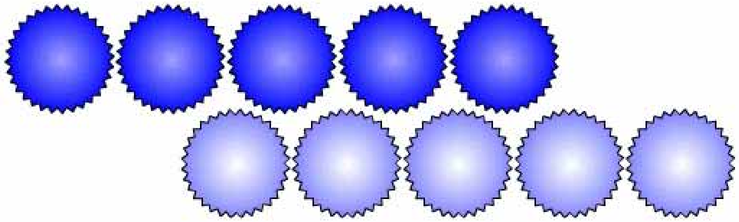
- Friction between particles





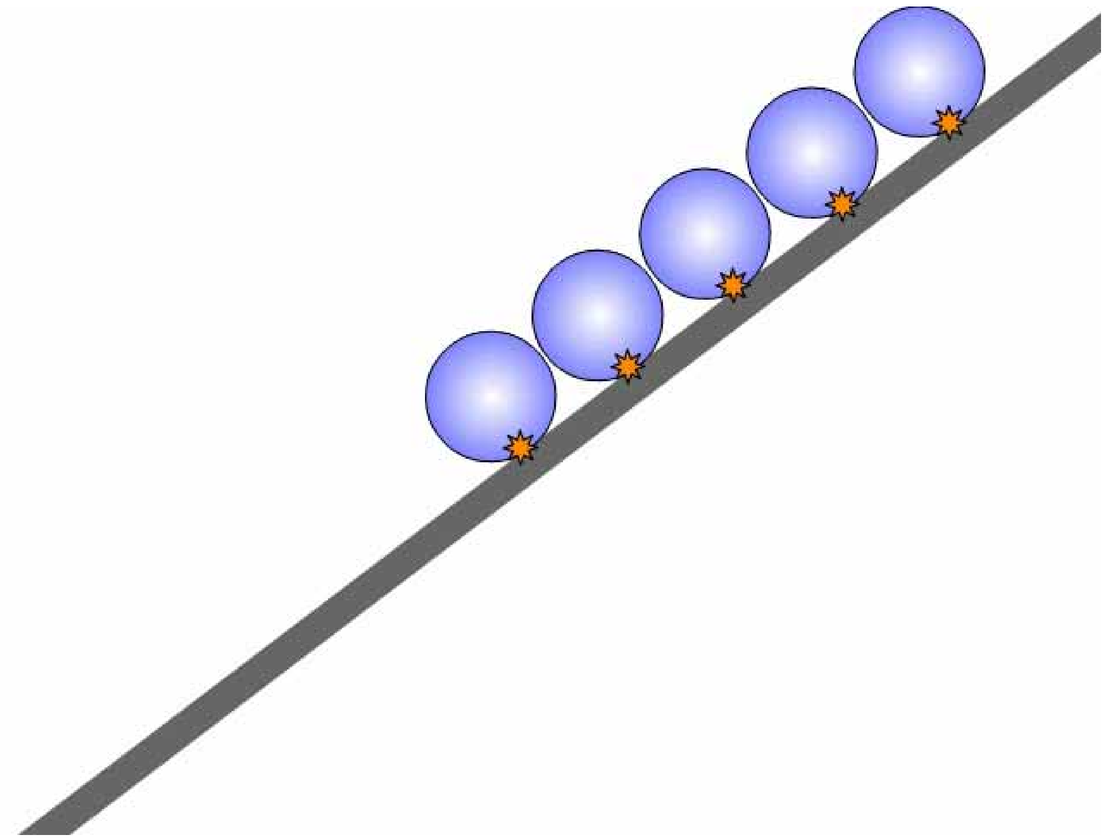
Smooth / low friction particles





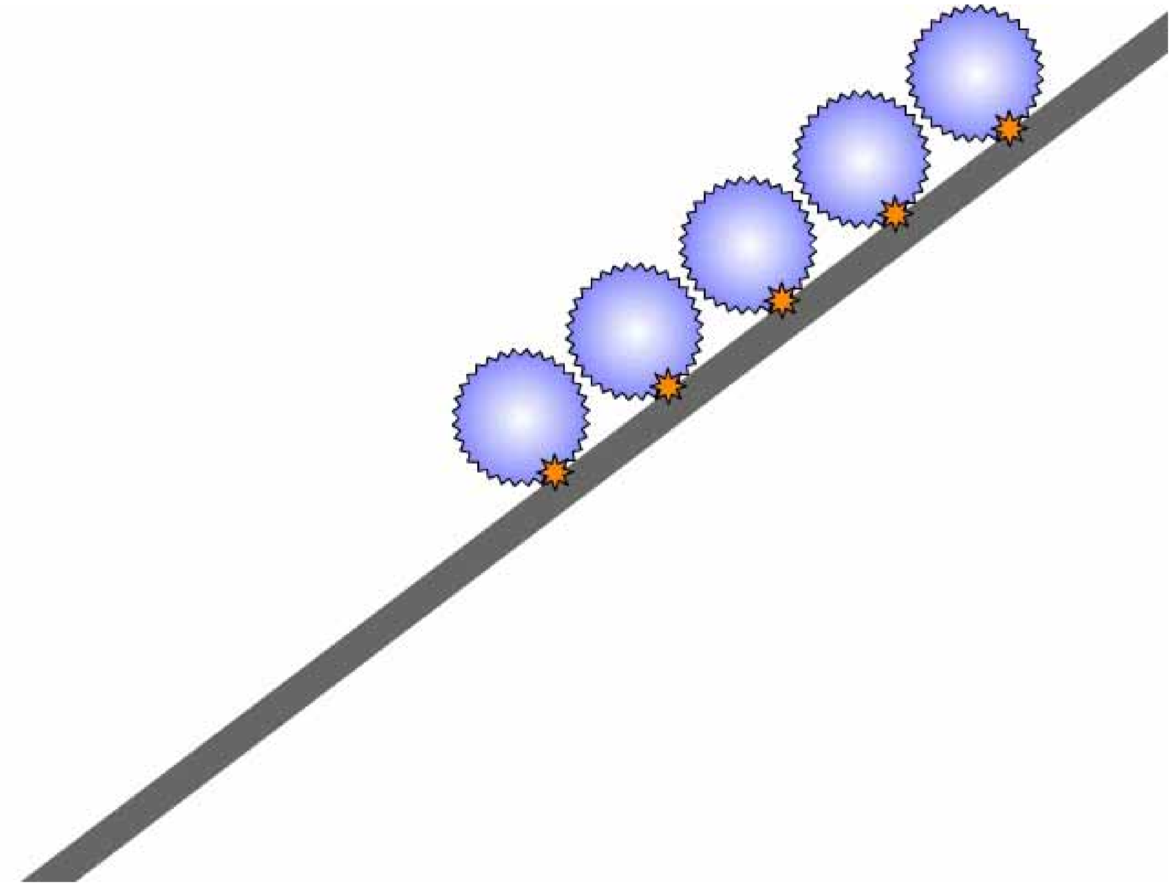
Rough / high friction particles

- Friction between particles and wall



Smooth / low friction particles

➢ Friction between particles and wall



Rough / high friction particles

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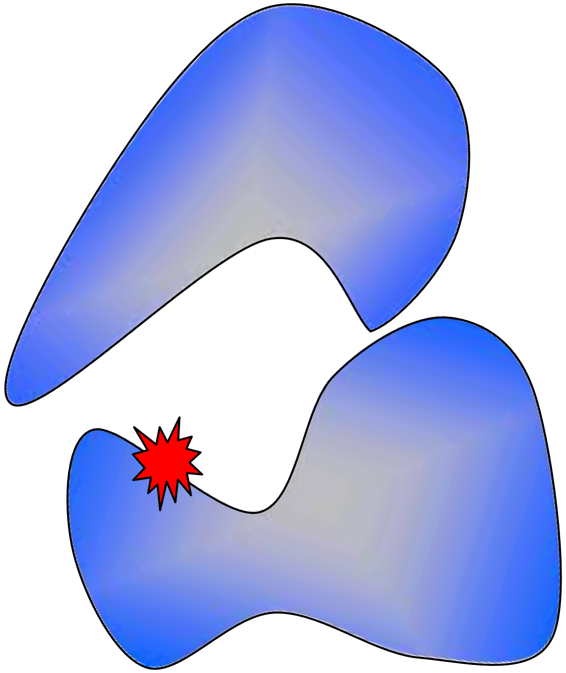
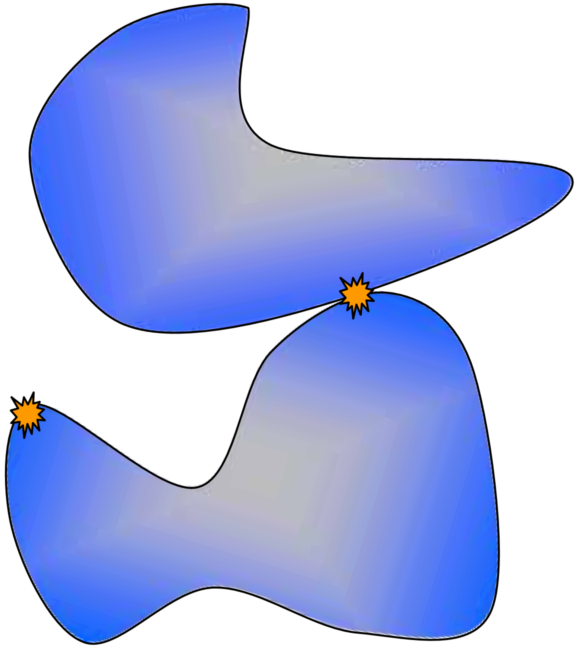
Mechanical Interlocking of particles



Strong interlocking

High force required for separation of particles

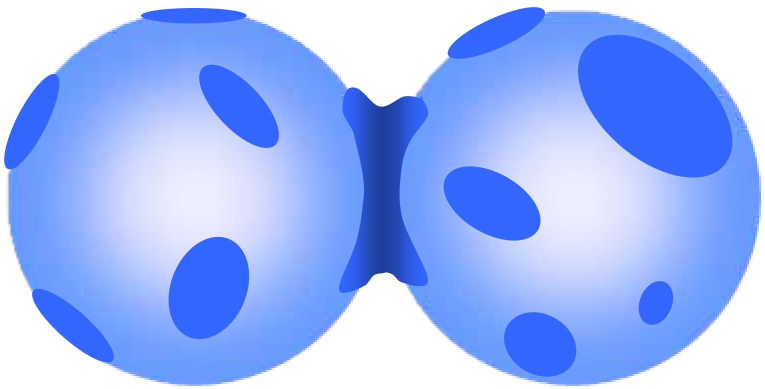
Same particles, but different orientation....



Weak interlocking

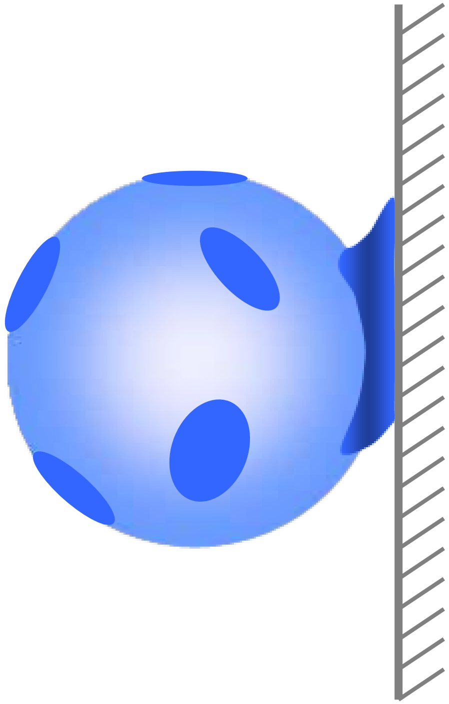
Relatively low force required for separation of particles

Liquid bridges between particles



Increased particle – particle adhesion

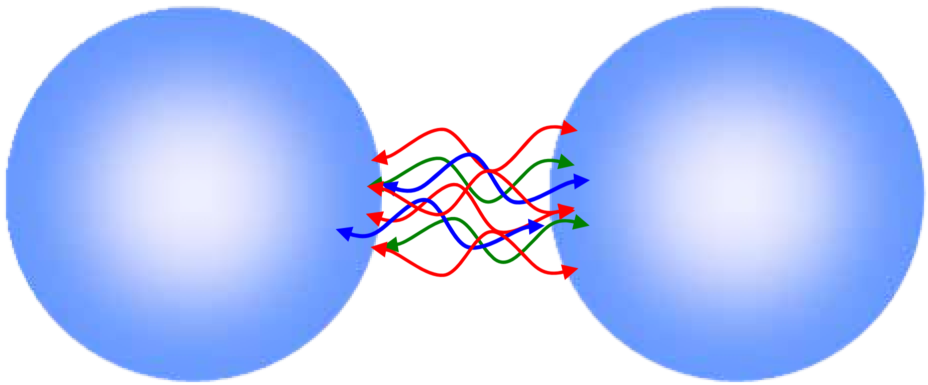
Liquid bridges between particle and wall



Increased particle – wall adhesion

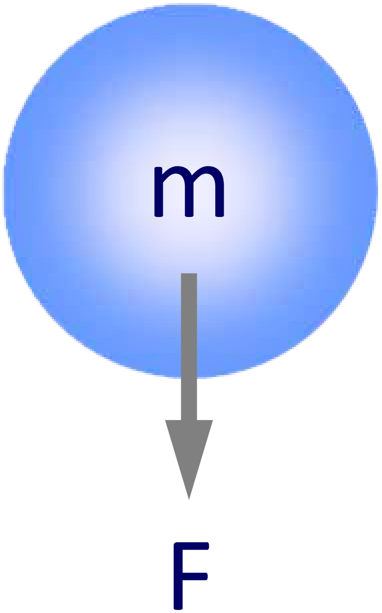
Cohesive, inter-particulate forces

- *Van der Waals*
- *Electrostatics*
- *Covalent*



Increased particle – particle attraction

Gravitational forces

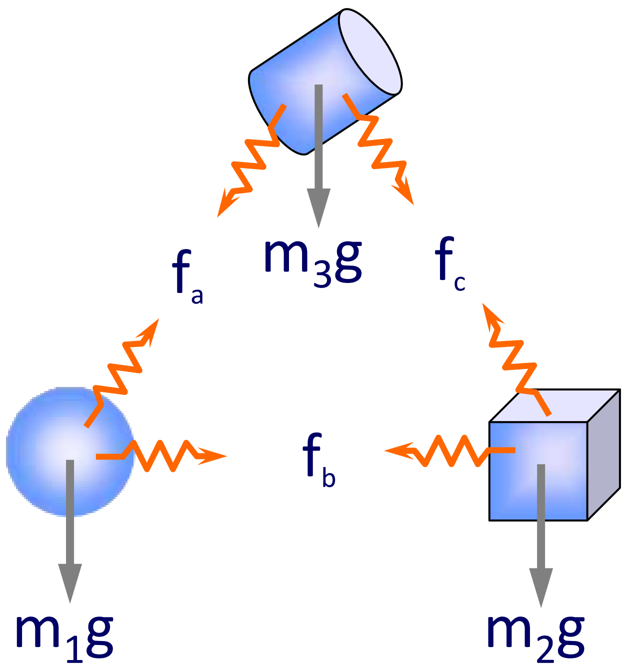


$$F = mg$$

g = acceleration due to gravity

Mechanisms of particle interaction

- Friction
  - Particle – Particle, Particle - Wall
- Mechanical Interlocking
- Adhesion / Liquid Bridges
  - Particle – Particle, Particle - Wall
- Cohesion
  - Van der Waals, Electrostatics, Covalent
- Gravity



Interparticulate forces (f_n) are a function of: -

- Frictional forces
- Mechanical interlocking
- Adhesion
- Cohesion

The relationship between f_n and mg depends on **particle physical properties** and **process environment**.

When powder is in **low stress state** (die filling / mixing), **cohesive forces** most influential.

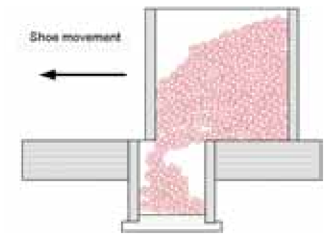
When powder is **consolidated** (hopper / extrusion / compression), **mechanical friction** and **particle interlocking** are most important.

Cohesion still contributes to flow, but it's effect is less significant.

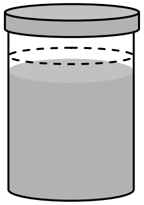
Examples of processes and applications

- Flow in the hopper
  - High stress, low flow rates, gravitationally induced flow

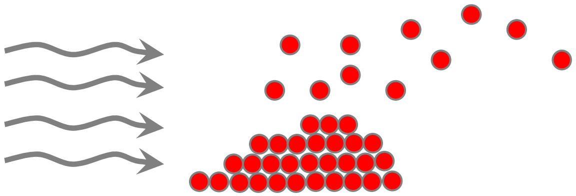
- Filling
  - Low stress, dynamic, gravitationally induced flow



- Transport / storage
  - Moderate / high stress, de-aeration, consolidation, caking



- Aerosolisation
  - Very low stress, powder needs to fluidise



Athletic ability!

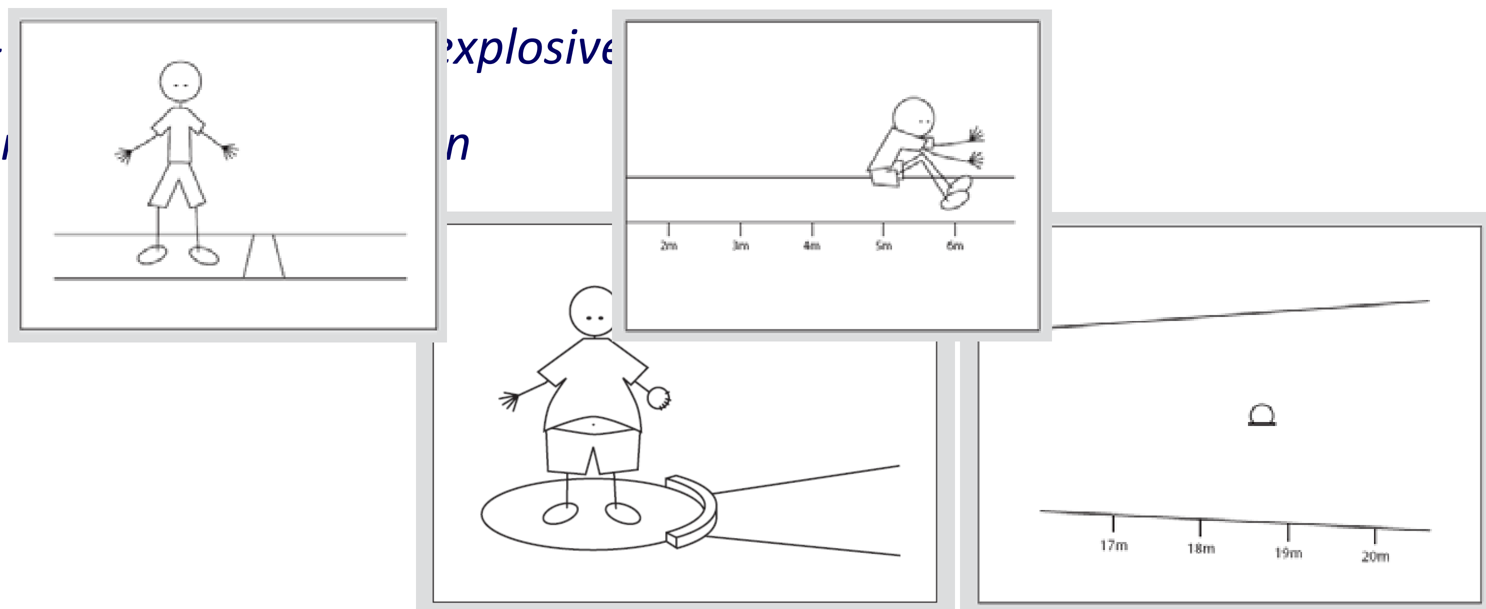
Consider an athlete competing at the Olympic Games

Properties: - *Tall, long stride length, slim, fast runner, stamina*

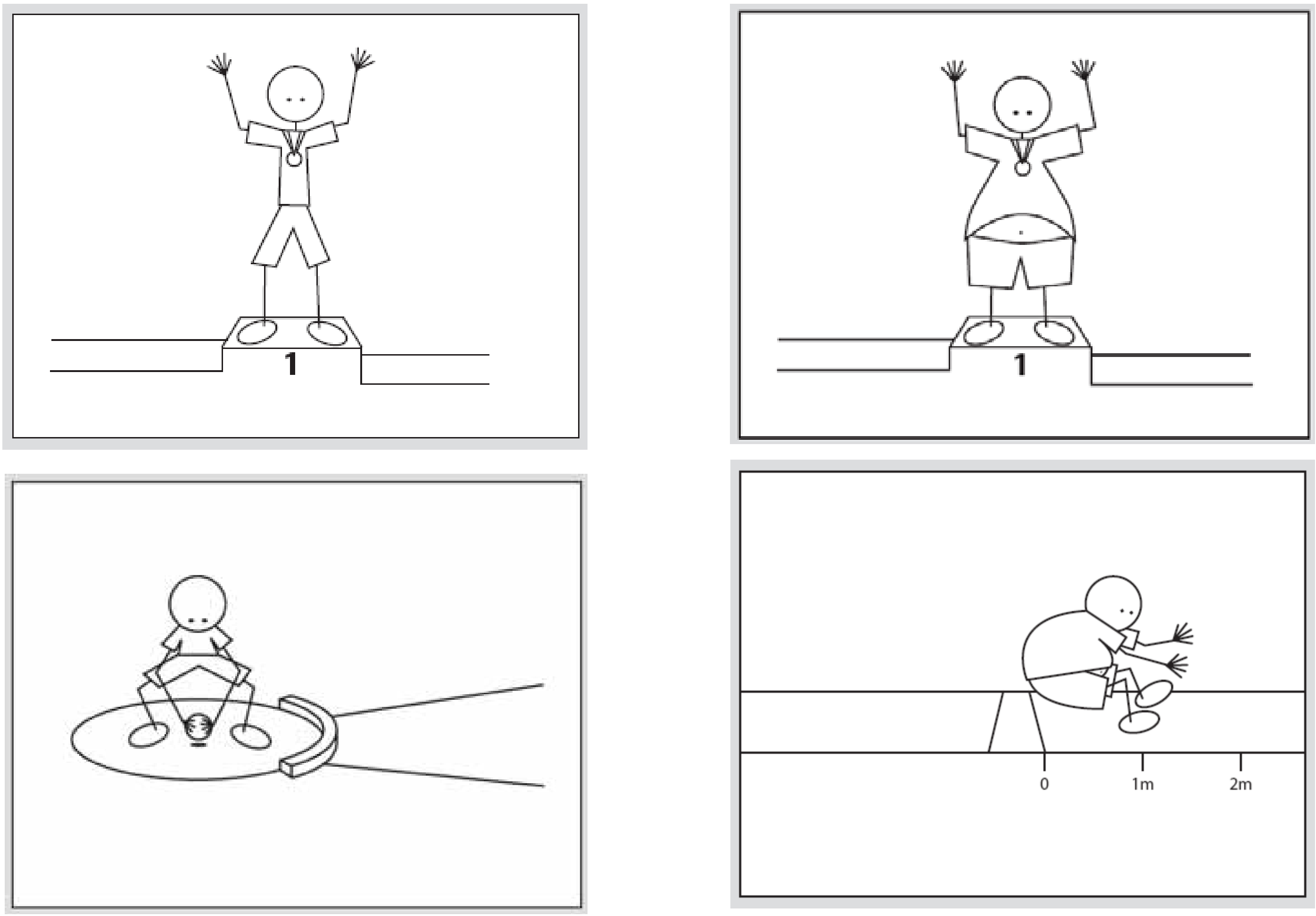
Suitable event: - *Long jump, runner, pole vault*

Properties: - *xplosive*

Suitable eve *n*



Are they "good" athletes?



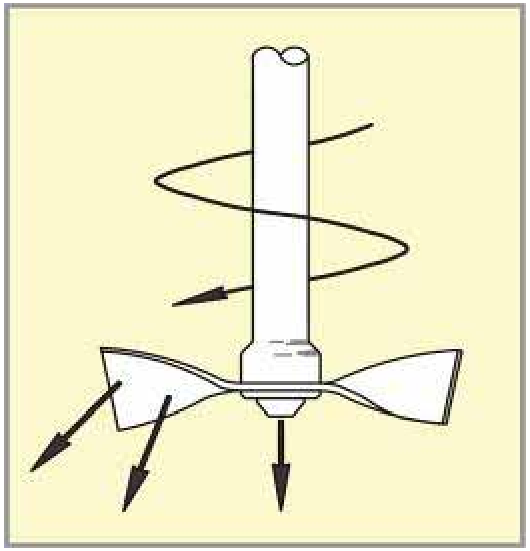
So what can be measured?

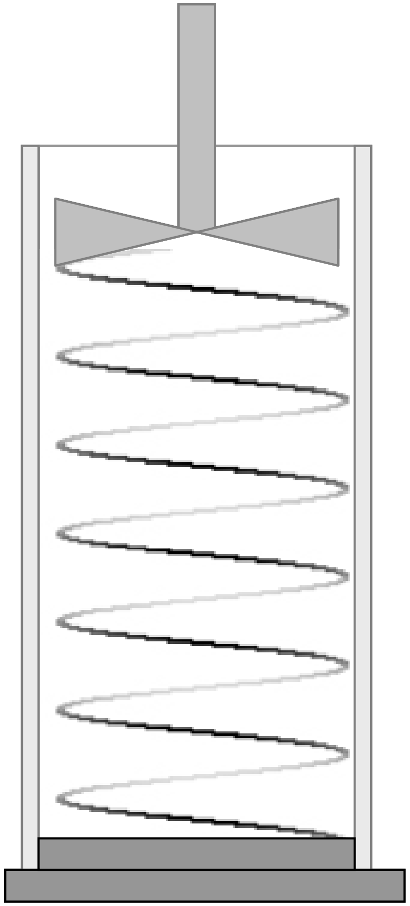
- Several traditional techniques
  - Carr's Index, Hausner Ratio, Angle of Repose, Flow through a funnel
- Pragmatic solution for their day
- Basic & insensitive, developed before modern technologies existed
- Don't represent conditions that powders experience in processing and application
- Each method typically produces one parameter

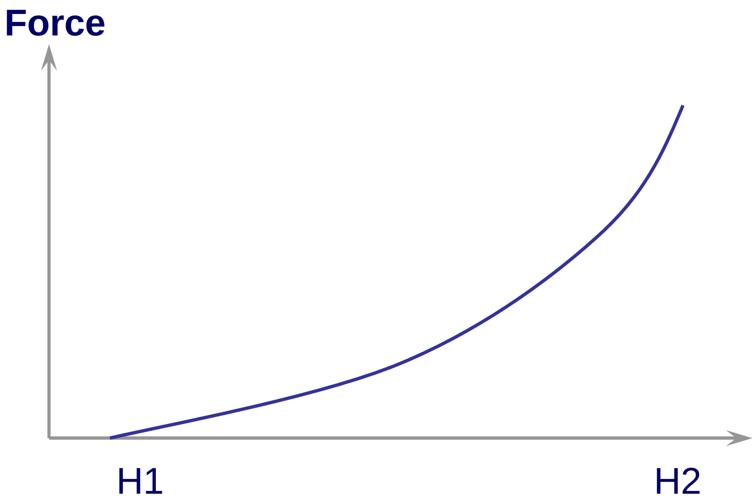
- More recently, shear cells -
  - Measure powders under consolidation
  - Measure the onset of flow, transition from static to dynamic
  - Good for understanding behaviour in hoppers

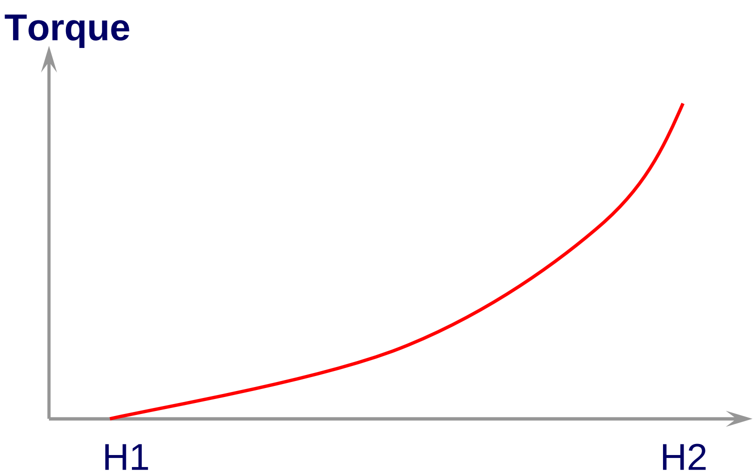
- Most recently, dynamic characterisation methods
  - Measure response of powder to various environments
  - Simulating range of process conditions
  - Measure directly response to aeration, consolidation, moisture, flow rate
  - Measure bulk properties of density, compressibility and permeability

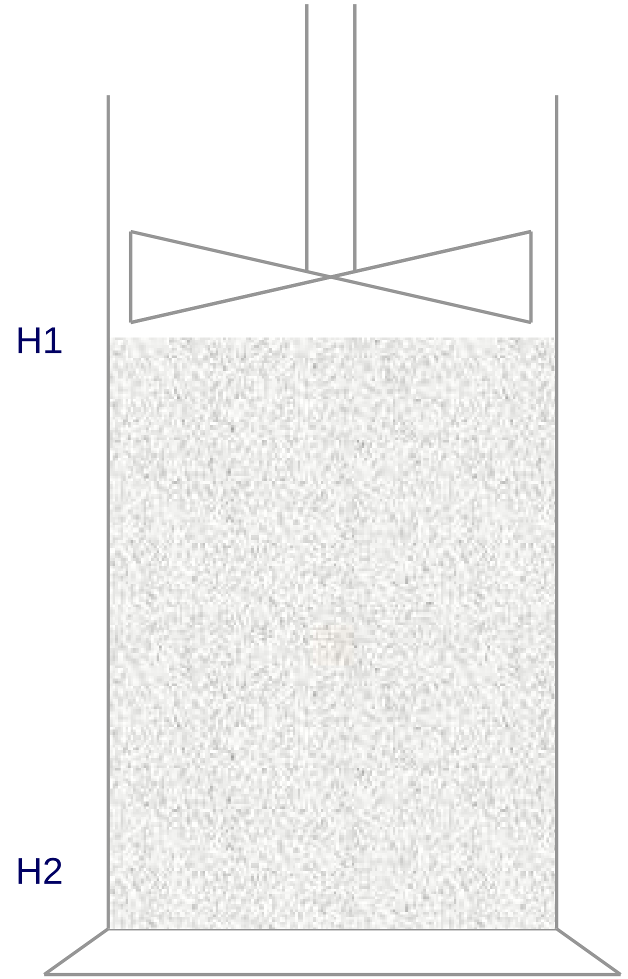
Principles of Operation





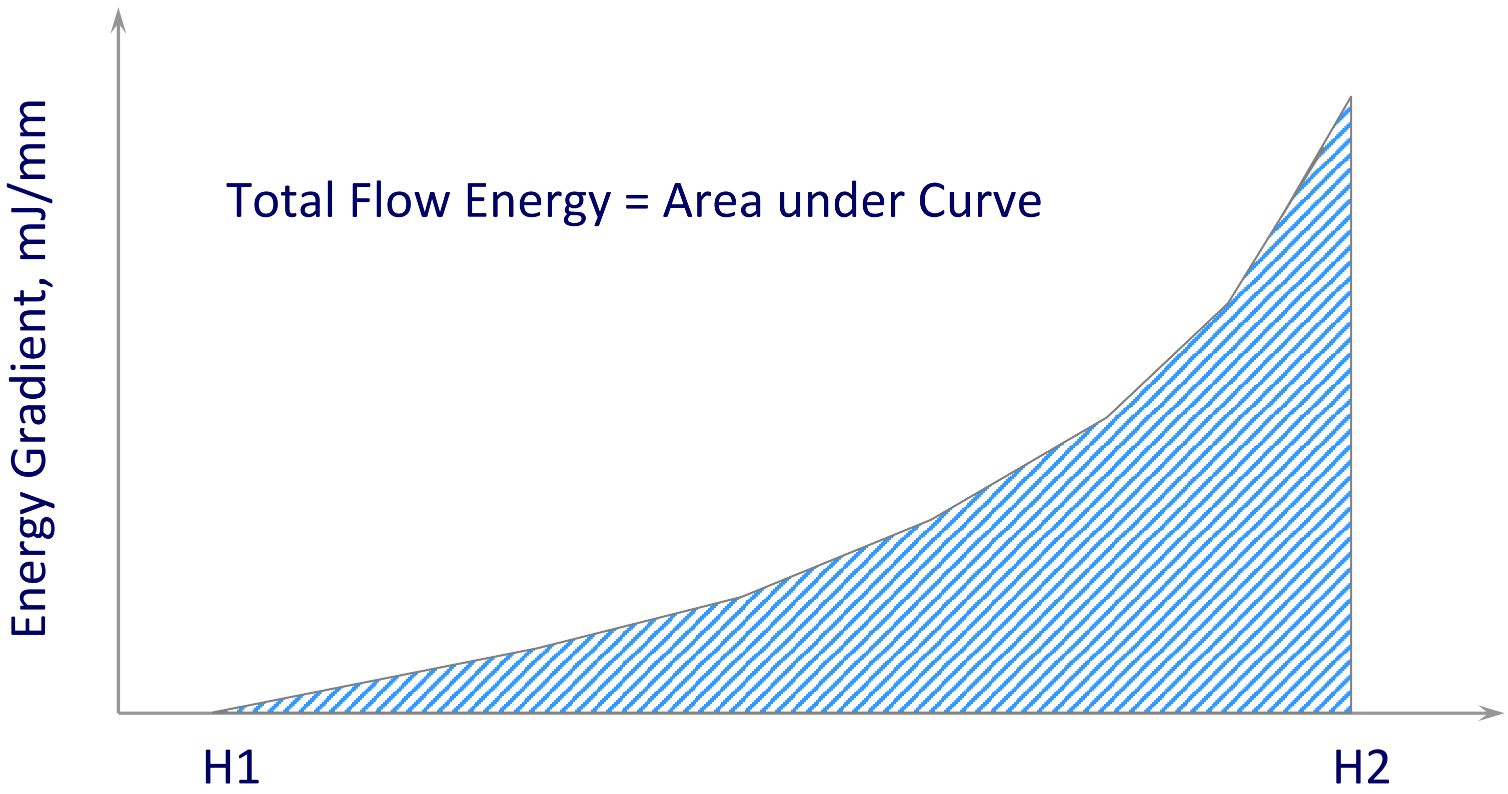

Force
H1
H2


Torque
H1
H2

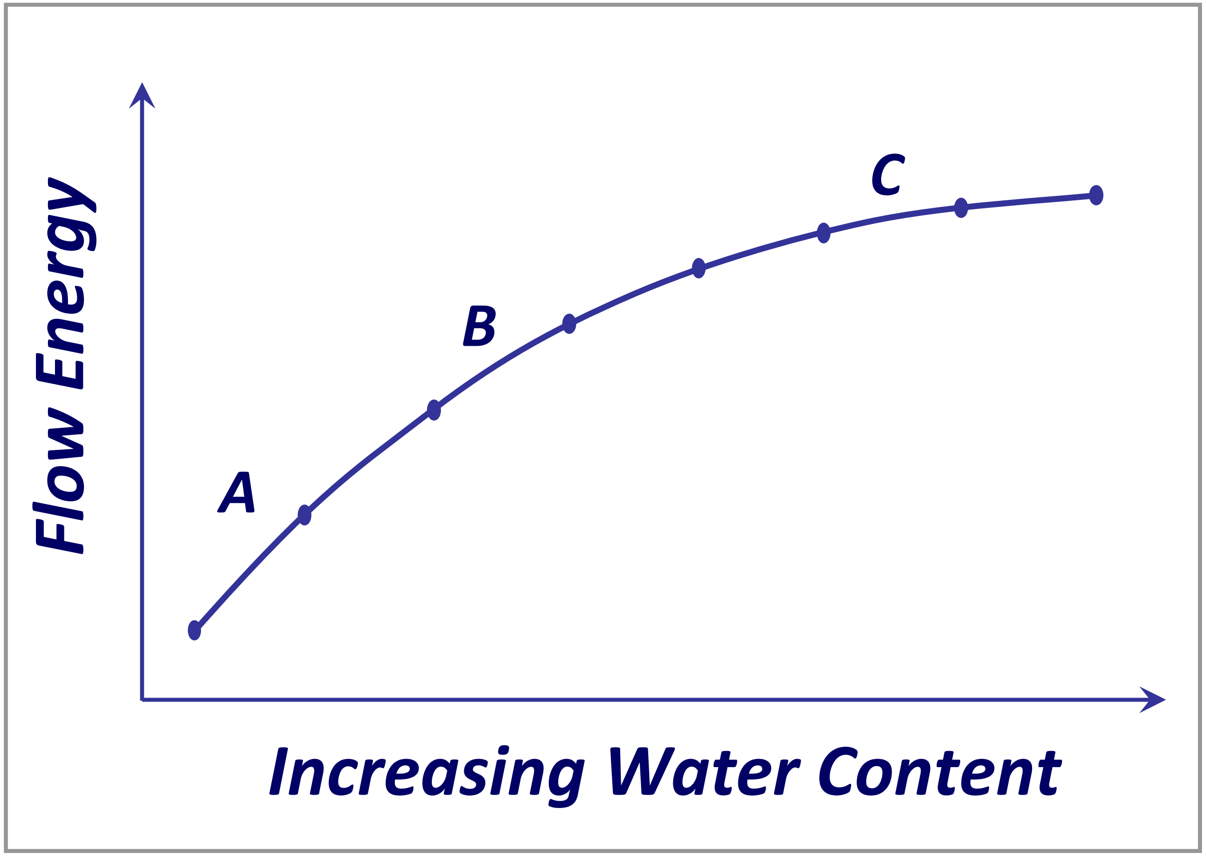

H1
H2

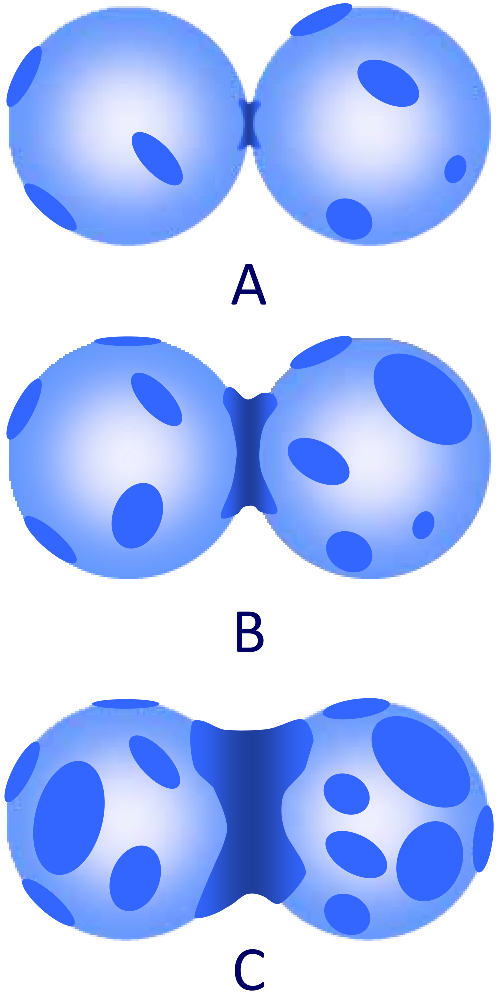
Work Done = Energy = "Force" x Distance = (Force + Torque) x Distance

Energy Gradient = Work Done per mm

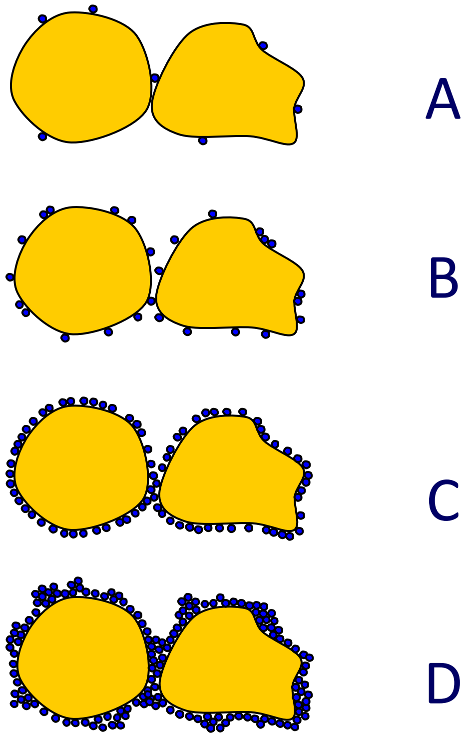


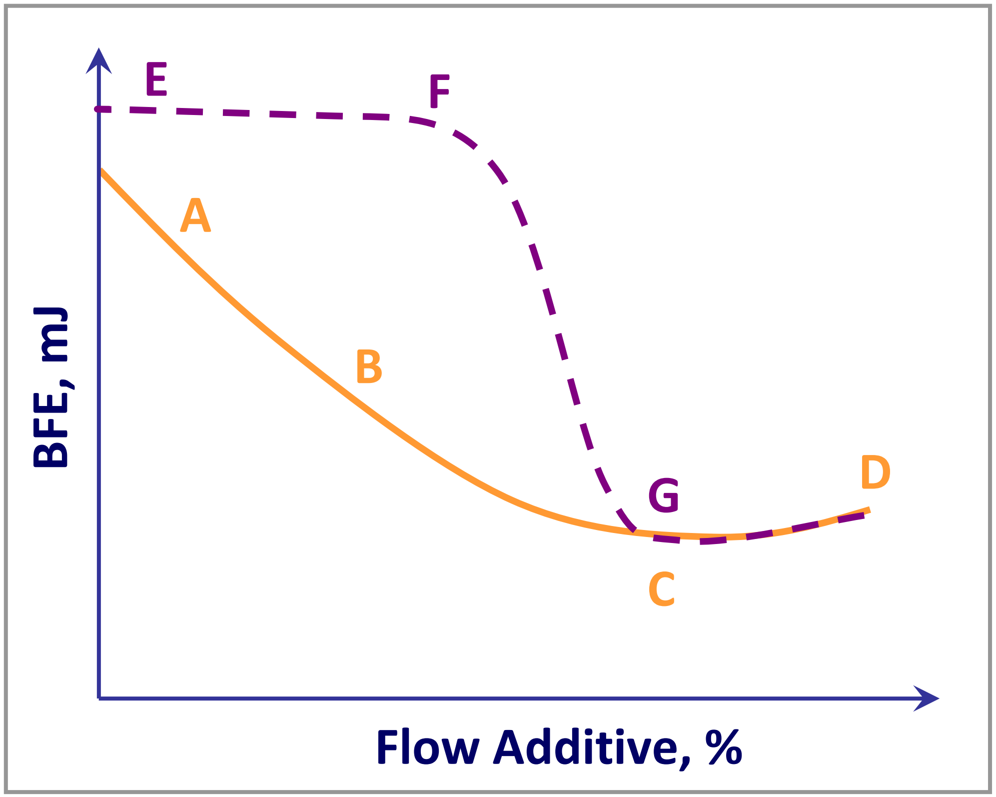
Flow Energy vs. Moisture Content

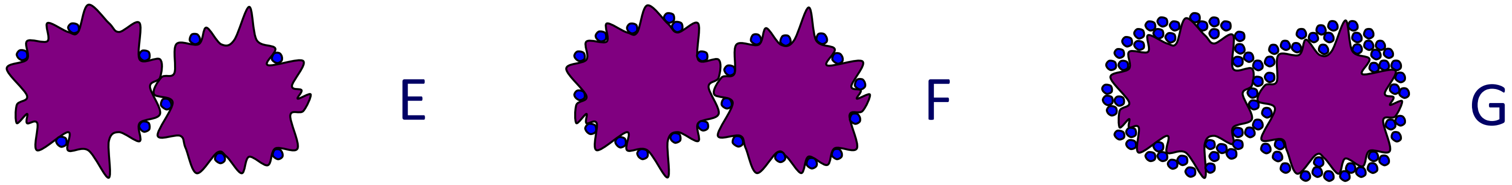




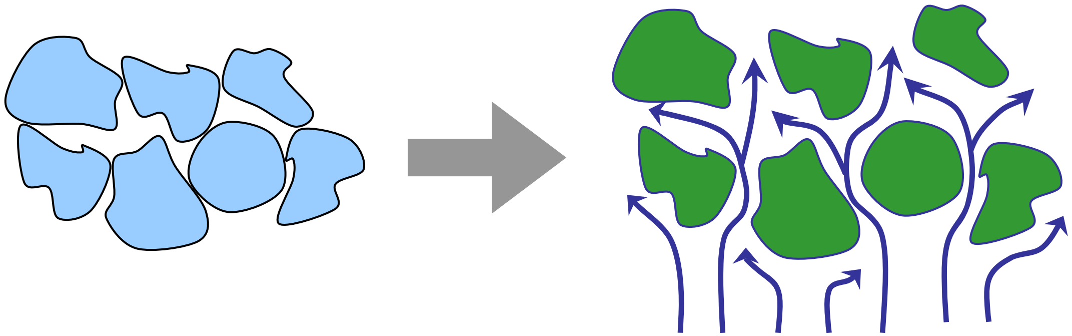
Flow Energy vs. Additive & Morphology



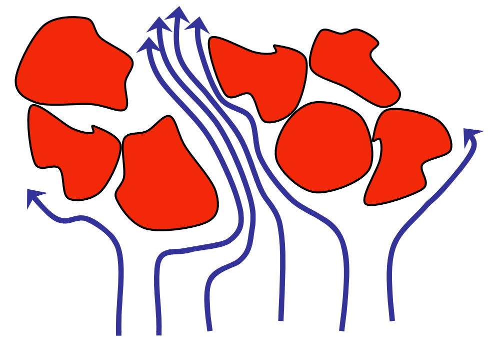




Aeration

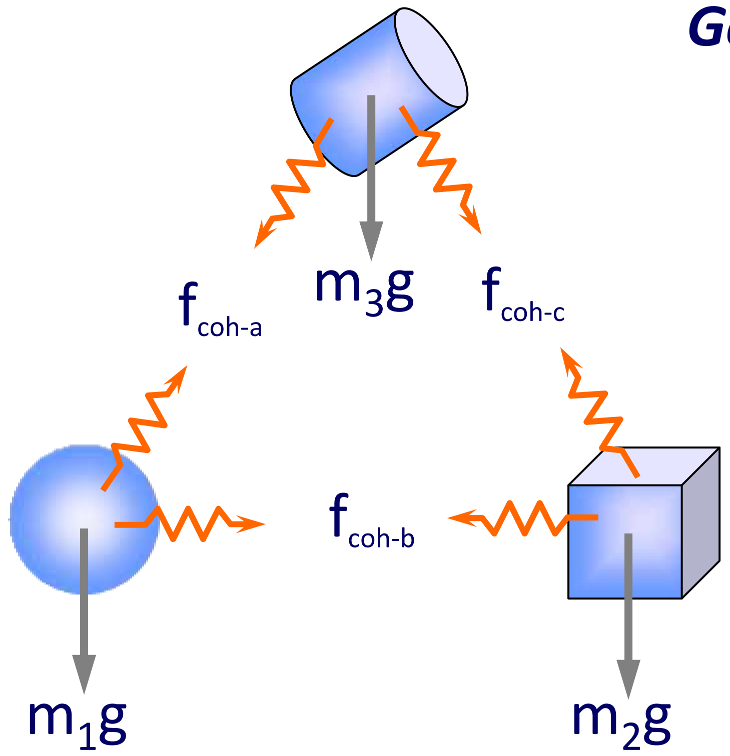


or



Non – Cohesive Powder

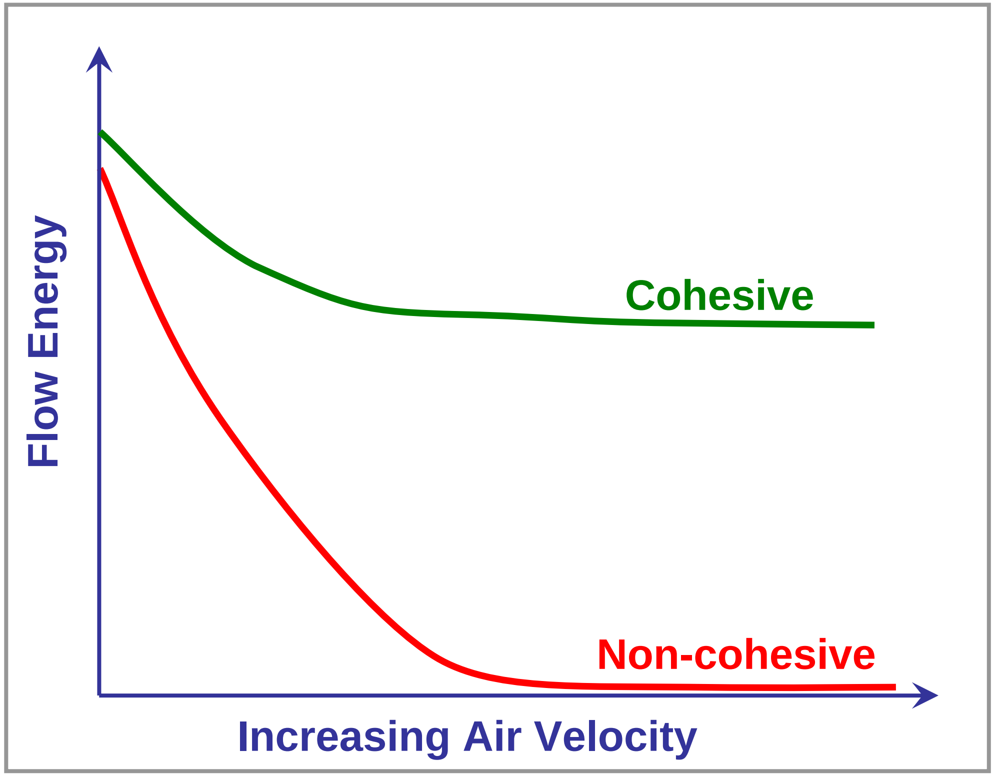
Cohesive Powder



Generally, if: -

$mg > f_{coh}$ powder is non-cohesive

$mg < f_{coh}$ – powder is cohesive



Used for measuring: -

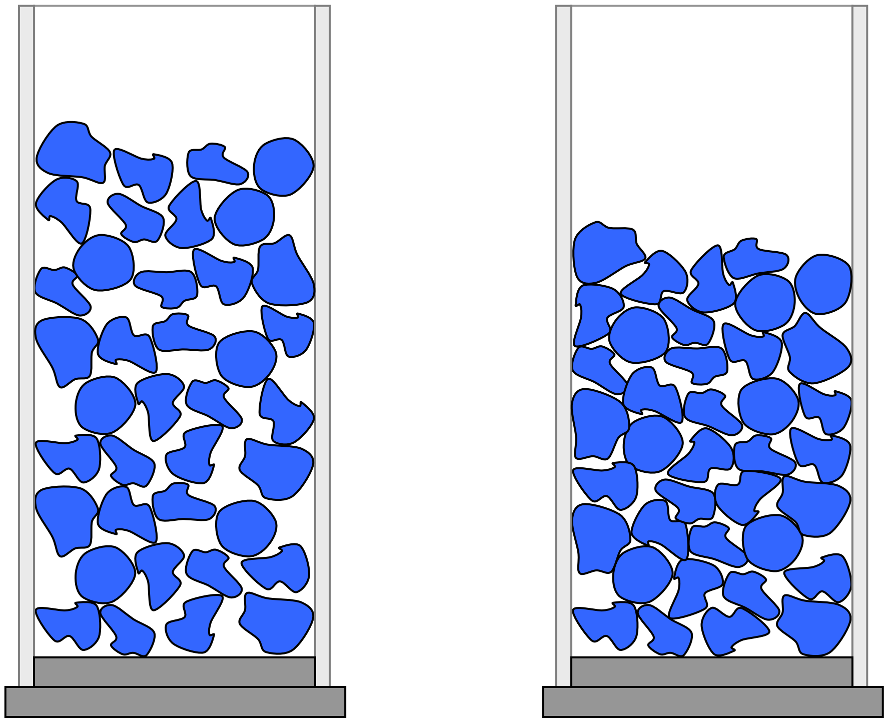
- *Cohesion*
- *Low stress, gravitationally induced flow*
- *Dosing / Mass Uniformity*
- *Aerosolisation / DPI*
- *Fluidisation behaviour*
- *Blending / mixing*
- *Segregation potential*

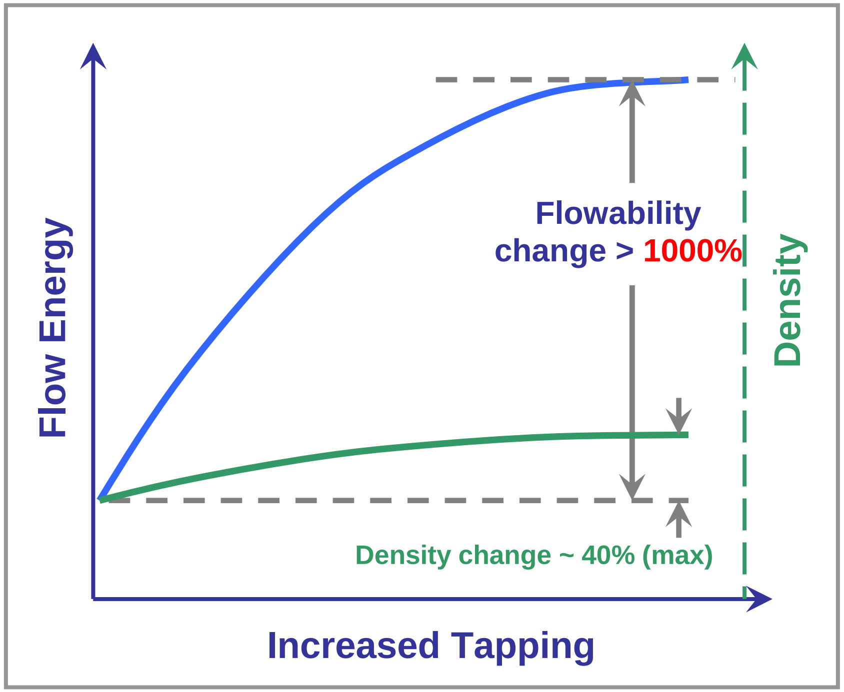
Consolidation

- Direct Pressure
- Tapping / vibration

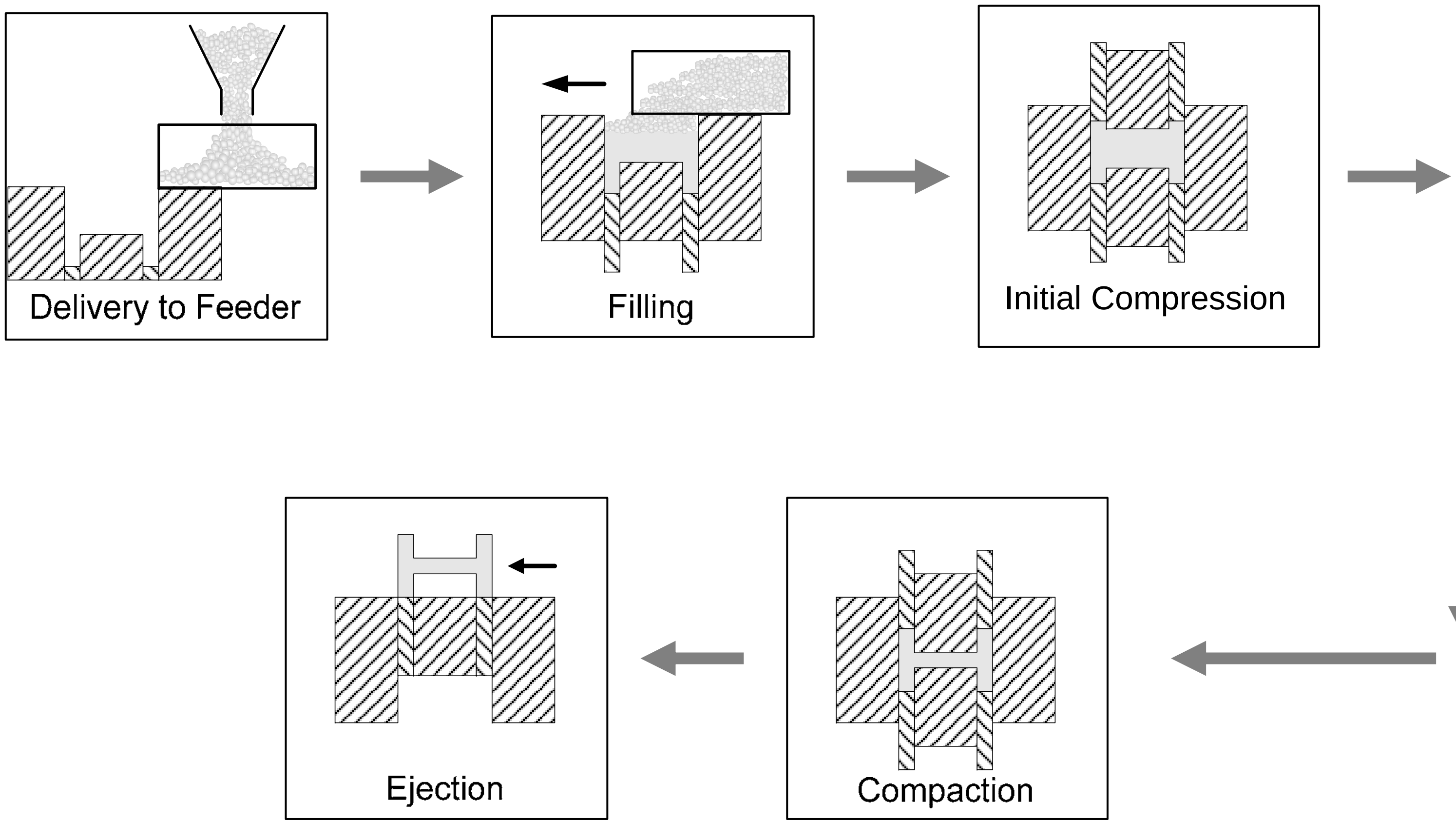
Understanding the effects of: -

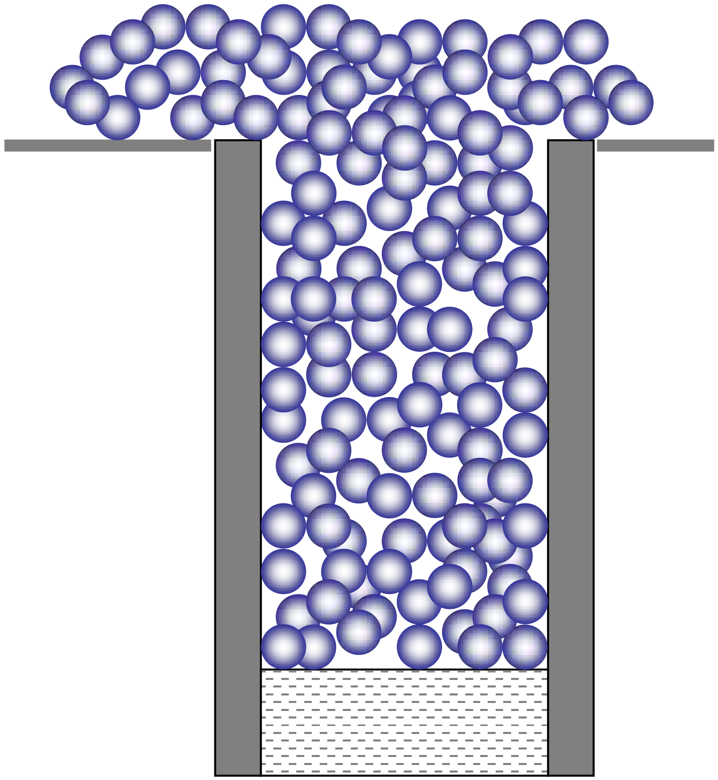
- Transport
- Storage
- Processing
- Caking



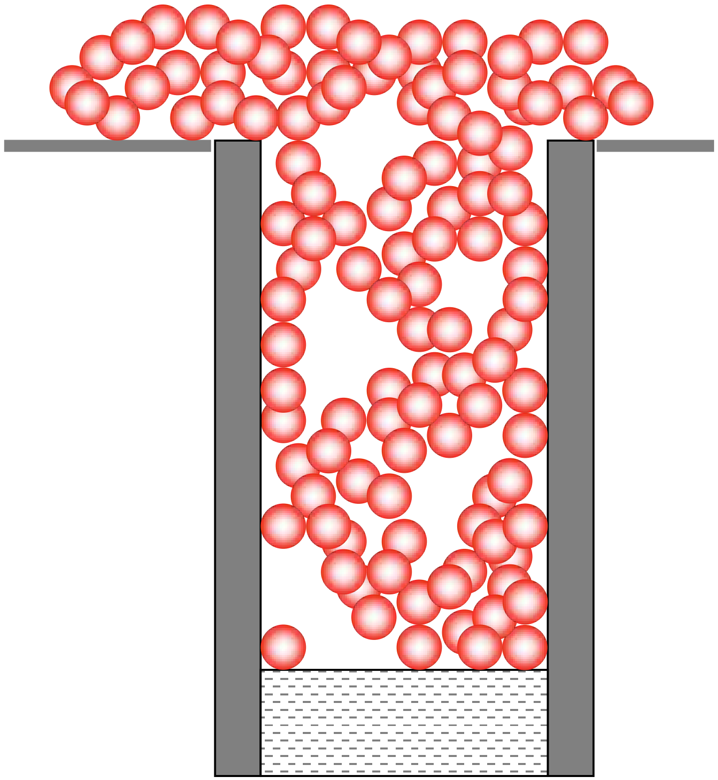

Flow Energy
Flowability
change > 1000%
Density
Density change ~ 40% (max)
Increased Tapping

Filling and Compression case study



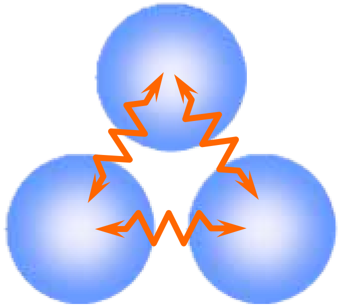


Good
Uniform filling, low porosity



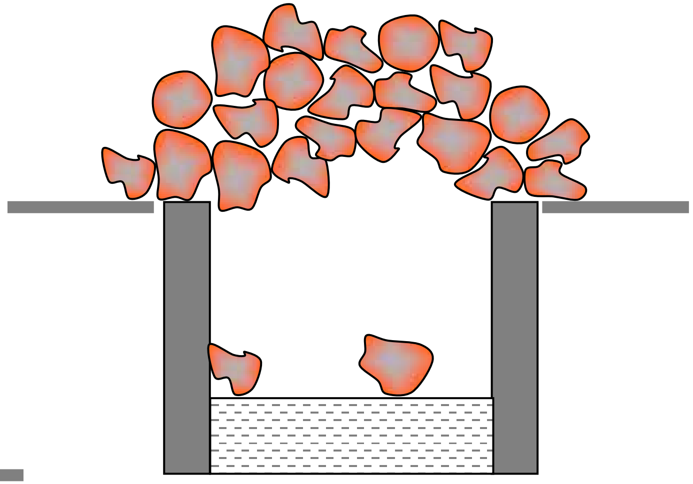
Poor
Non-uniform filling, high porosity

- *High cohesion*

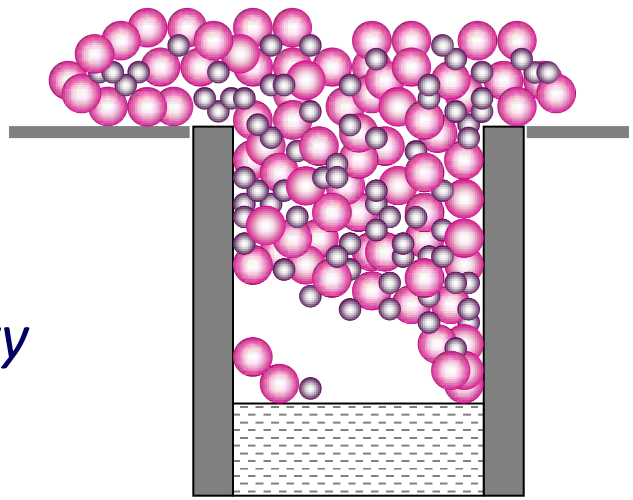


Cohesive forces >> mg

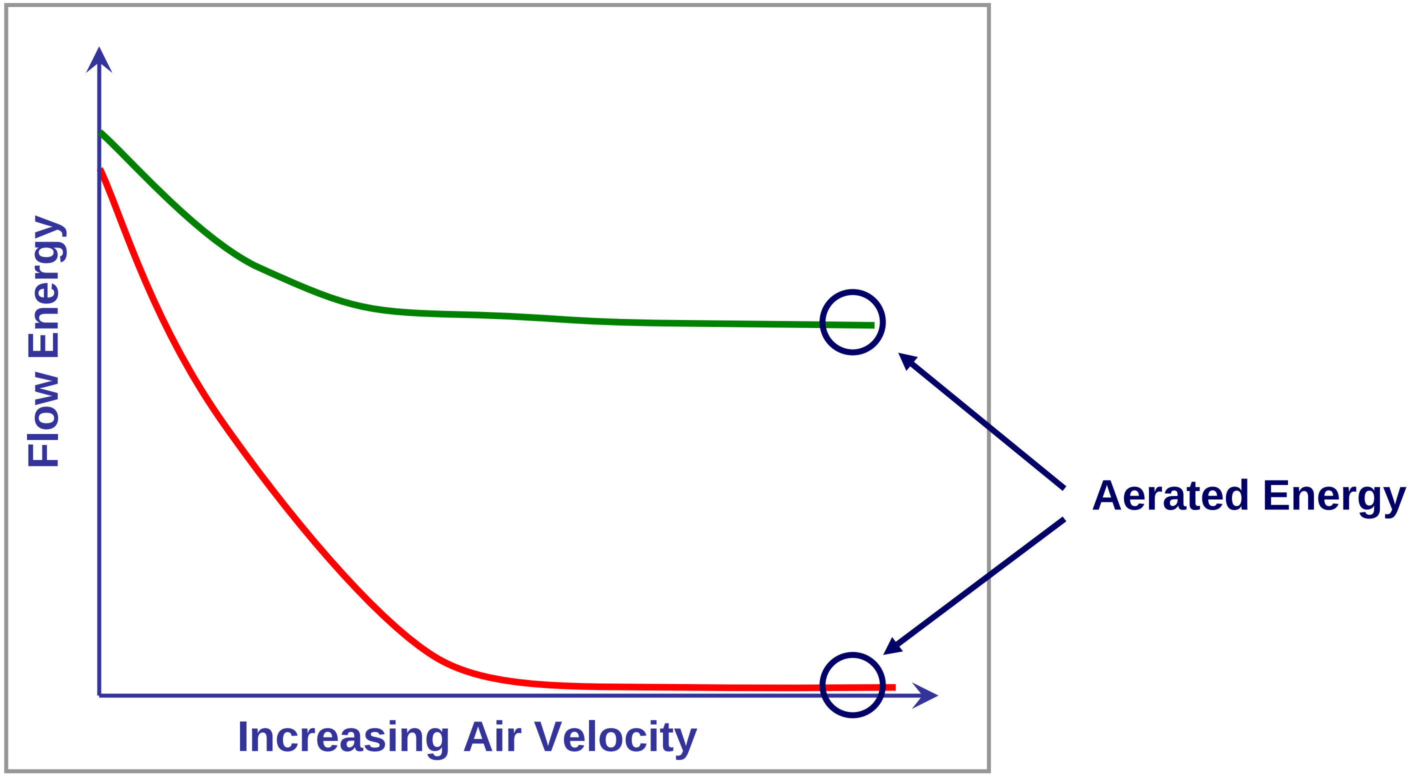
- *High mechanical friction & Interlocking*



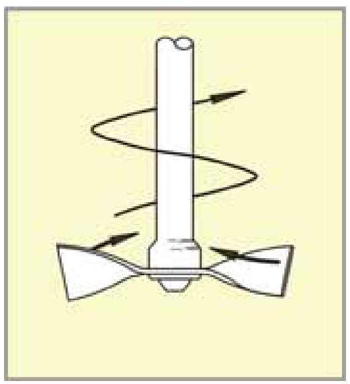
- *Low permeability*

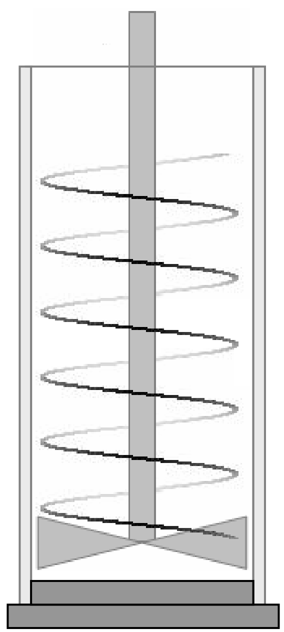


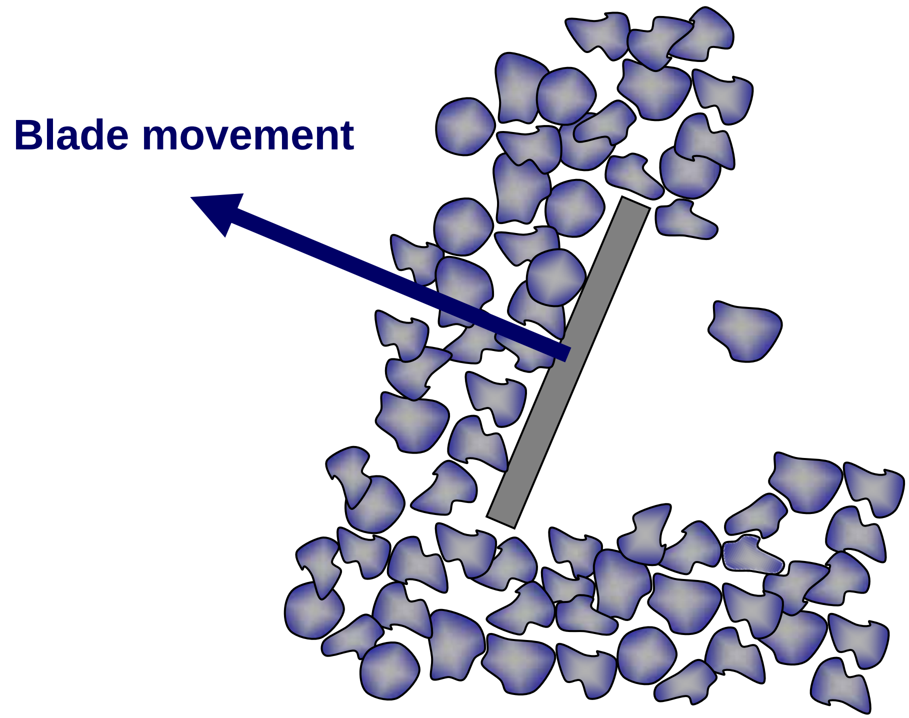
- *Cohesion*
  Aeration Test – low value of Aerated Energy equals low cohesion



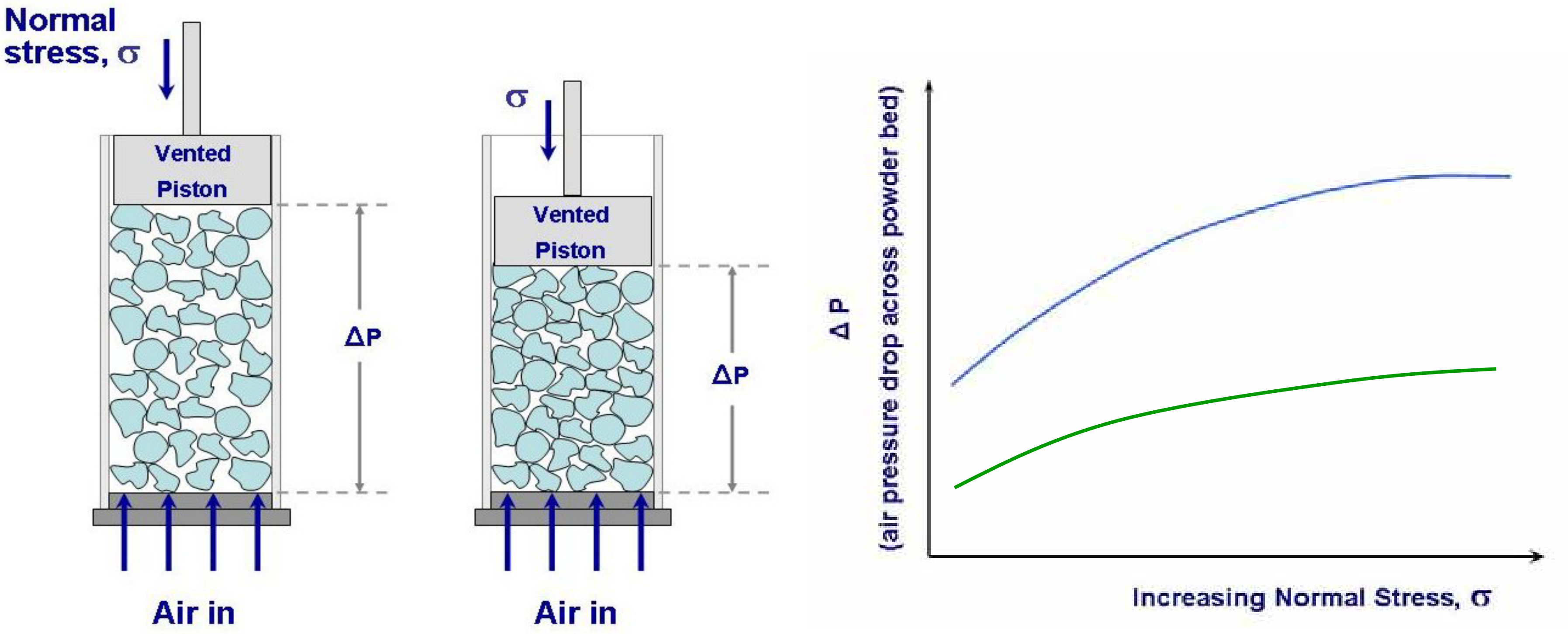
- *Mechanical interlocking & friction*
  Specific Energy – low value equals low interlocking and friction

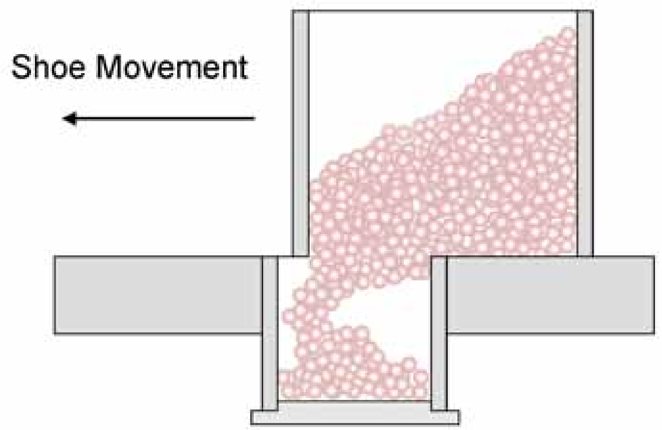




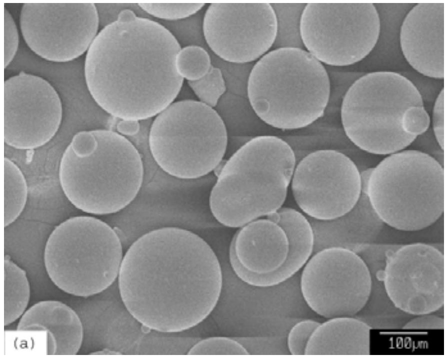


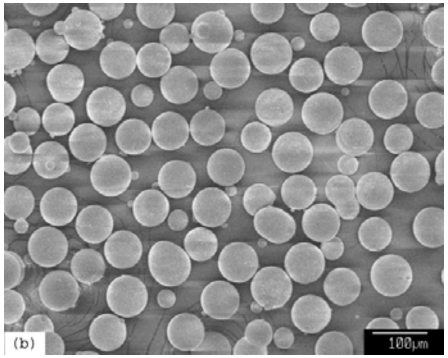
- *Permeability*
  High Permeability – entrained air can easily escape

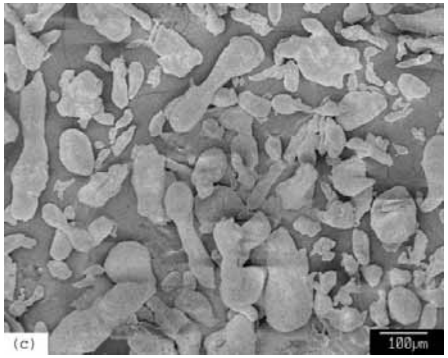


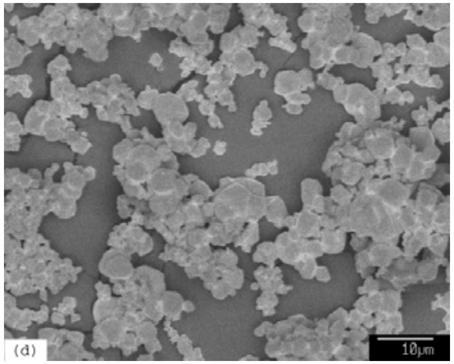


Material	Material/Powder description	D_{50} (µm)	Shape
(a)	GL Glass beads	174	Spherical
(b)	GS Glass beads	68	Spherical
(c)	Granular Aluminium powder	134	Irregular
(d)	Tungsten powder	4	Angular



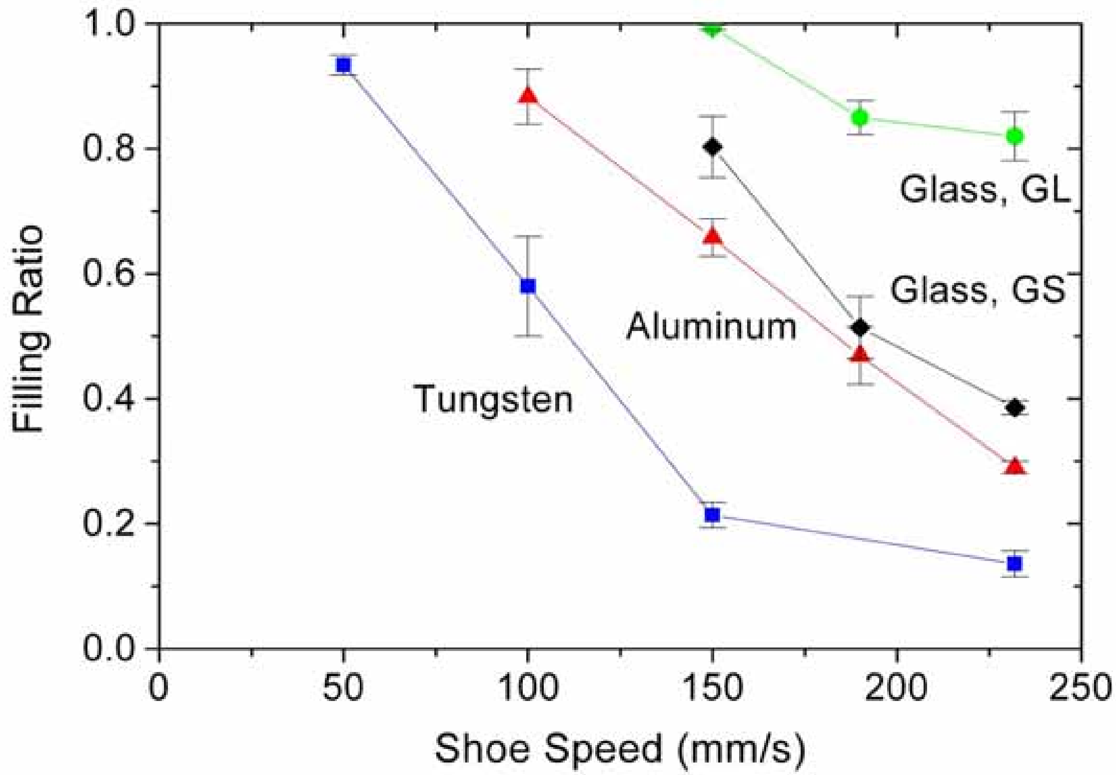


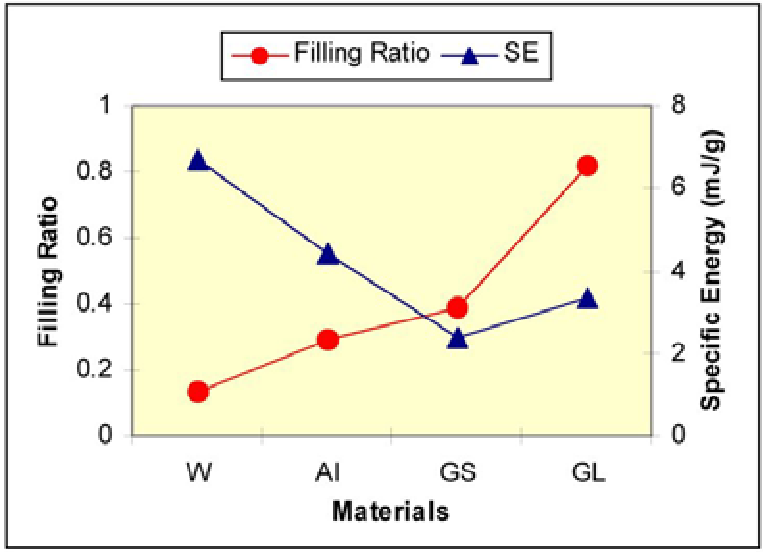




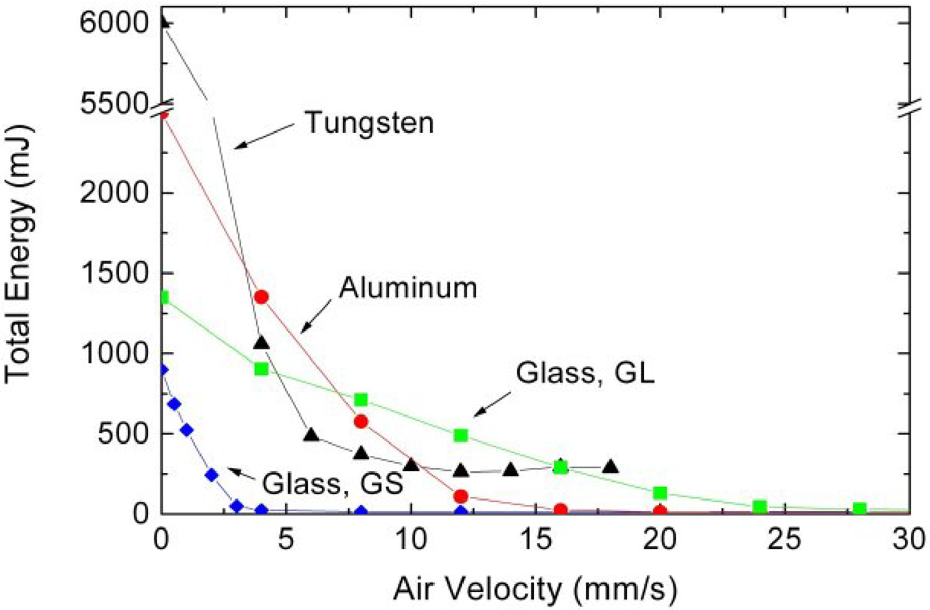
Filling Ratio

Ratio of actual mass in die compared to mass calculated from bulk density and volume of die



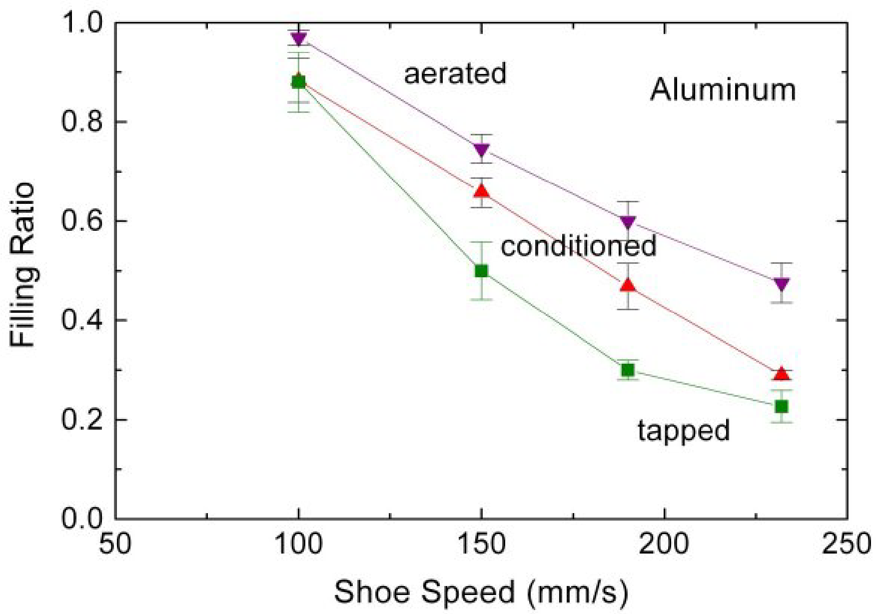


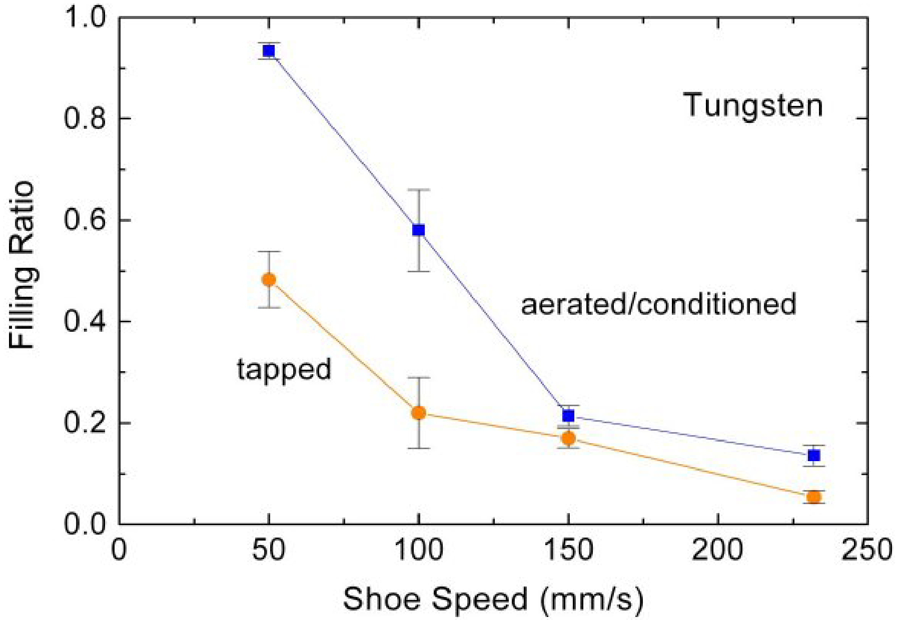
Filling Ratio & Specific Energy



Aeration

Measurements:	Glass GL	Glass GS	Aluminium	Tungsten
Fill Ratio	0.82	0.40	0.29	0.14
Aeration Energy, AE (mJ)	< 10	< 10	< 10	~300
Specific Energy, SE (mJ/g)	3.4	2.4	4.4	6.7
Pressure Drop across the powder bed at 2mm/s air velocity, PD_{15} (mbar)	0.8	**5.2**	1.4	15.3
Consolidation Index, CI_{20Taps} - factor by which flow energy increases after tapping, relative to BFE	1.31	1.11	**1.43**	**2.32**





Other applications

- Mixing / Blending – Cohesion, Aeration, Specific Energy
- Compression – Basic Flowability Energy, Permeability, Compressibility
- Dry Powder Inhalers – Aeration, Permeability
- Storage – Consolidation, Caking
- Size Reduction – Particle attrition, Basic Flowability Energy
- Hopper Flow – Shear Cell, Wall Friction, Permeability

Conclusions

1. Powder behaviour is complex
2. Flow properties will change depending on the conditions imposed
3. Each process / application will subject the powder to a specific set of conditions
4. Essential that powder characteristics match the demands of the process or application
5. Now possible to simulate range of conditions and measure powder response
6. Important to measure powder in representative state – e.g. aeration tests for aerosolisation & cohesion, shear cell tests for behaviour in hoppers, etc
7. Database of flow properties can be established, leading to enhanced process understanding, efficient formulation and development of new materials and higher quality of final product

Thank you for your attention

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