

Flowability Assessment of Weakly Consolidated Powders

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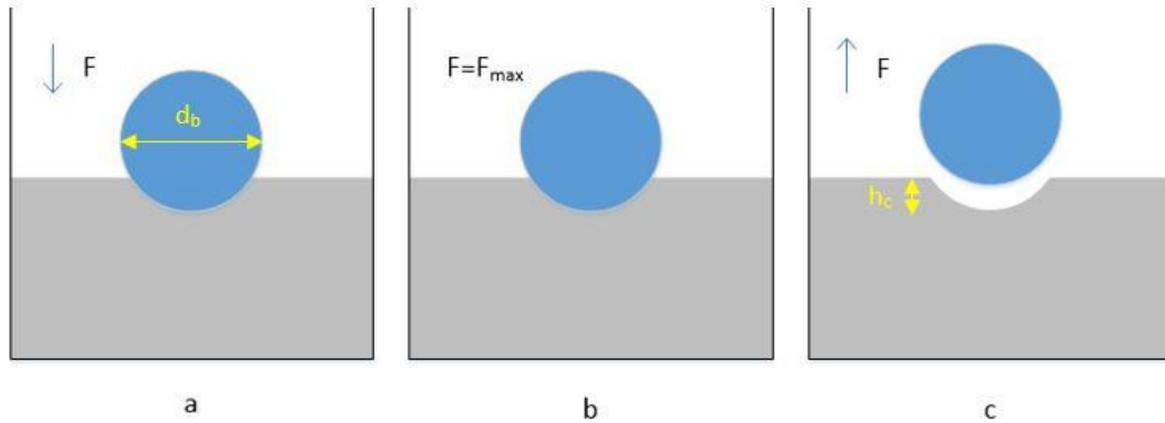
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Introduction

- In traditional flowability measurement devices:
 - Reproducibility of unconfined yield strength is greatly reduced at low stresses.
 - Or inconsistent with observed behaviour.
 - Materials found to be cohesionless may have practical differences.
 - Onset of flow is measured – may not be complete flow description.
- IFPRI seek to develop a theoretical understanding of flow of weakly consolidated & weakly cohesive powders.
 - Development of practical means of making measurement to support theory.
 - Results should be generalisable to broad class of powders.

Ball indentation¹

- Spherical indenter driven into powder bed²



- Force & displacement measured throughout

☐ Hardness determined:

$$H = \frac{F}{A} = \frac{F}{\pi(d_b h_c - h_c^2)}$$

- Indentation zone is confined by powder
- Hence, hardness related to unconfined yield stress by **constraint factor, C**:

$$H = C \cdot \sigma_c$$

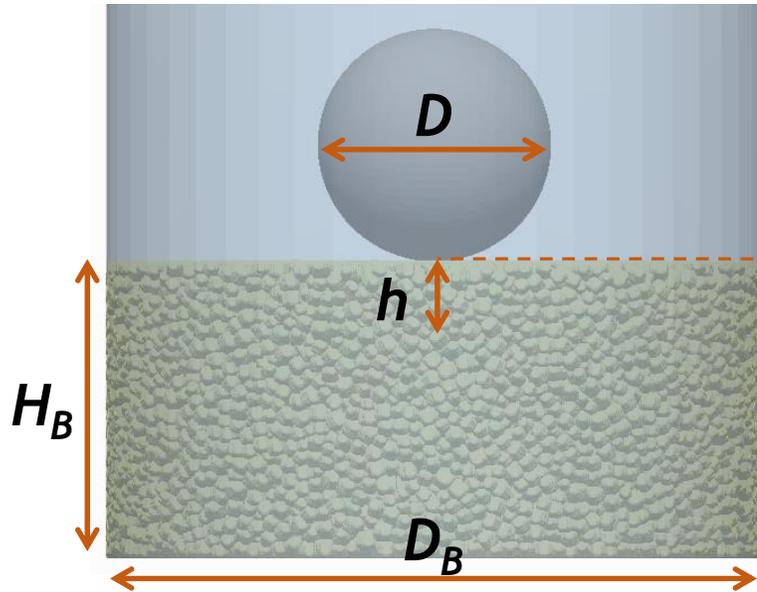
- The value of C is not known a priori for a given powder
- Can be determined by complimentary σ_c measurement

¹Hassanpour, A., Ghadiri, M., 2007, *Particle & Particle Systems Characterization*, 24 (2), 117-123.

²Stavrou, A., Hare, C., Hassanpour, A., Wu, C-Y., 2020, *Chemical Engineering Science*, 211, 115307.

Ball indentation dimensions

- Criteria established by Zafar *et al.* (2017)³



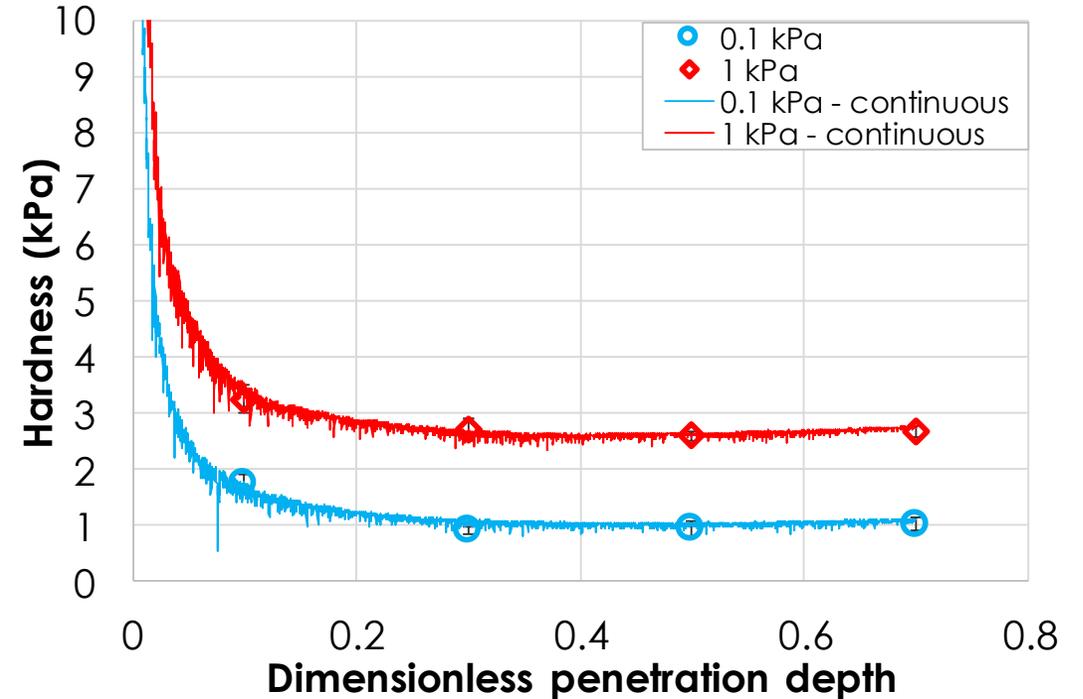
$$D \geq 16d_p$$

$$D_B \geq 45d_p$$

$$H_B \geq 20d_p$$

- Suitable penetration depth required
- Dimensionless penetration depth,

$$h_d = \frac{h}{R}$$



- Stable hardness region found²
- Negligible elastic recovery
 - H determined without unloading (<1 % error)

²Stavrou, A., Hare, C., Hassanpour, A., Wu, C-Y., 2020, *Chemical Engineering Science*, 211, 115307.

³Zafar, U., Hare, C., Hassanpour, A., Ghadiri, M., *Powder Technology*, 310, 300-306.

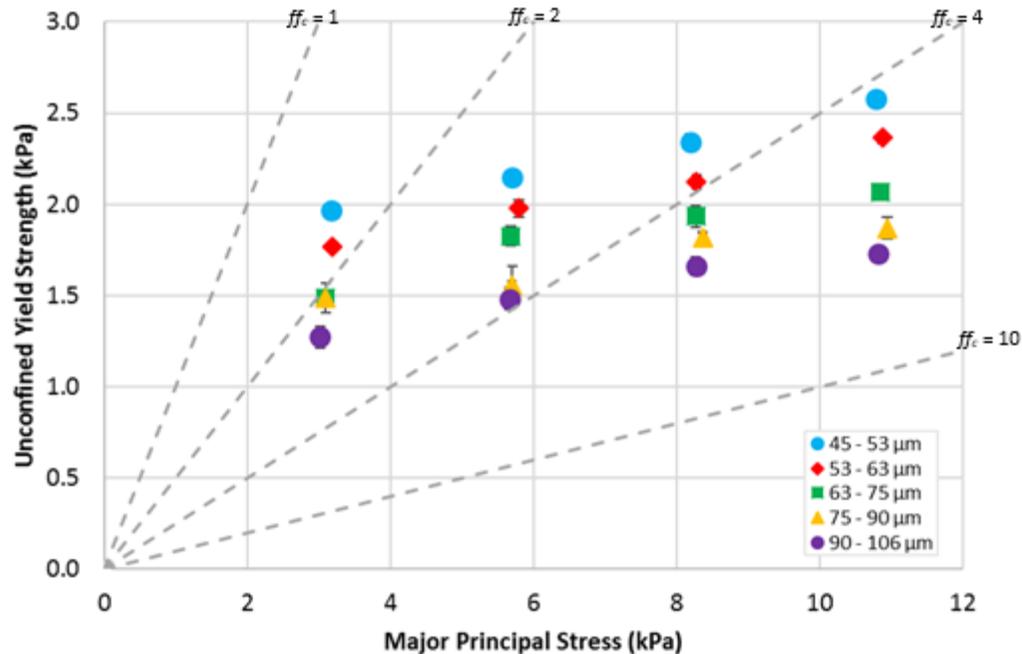
Flow measurement methods used

| | Shear cell | Uniaxial compression | Ball indentation (original method) ¹ | Ball indentation (critical method) |
|---------------|--|--|---|------------------------------------|
| Consolidation | | | | |
| Failure | | | | |
| Measurement | Control: σ Measure: τ Determine: σ_1 & σ_c | Control: σ (σ_1) Measure: σ_c (uUYS) | | |

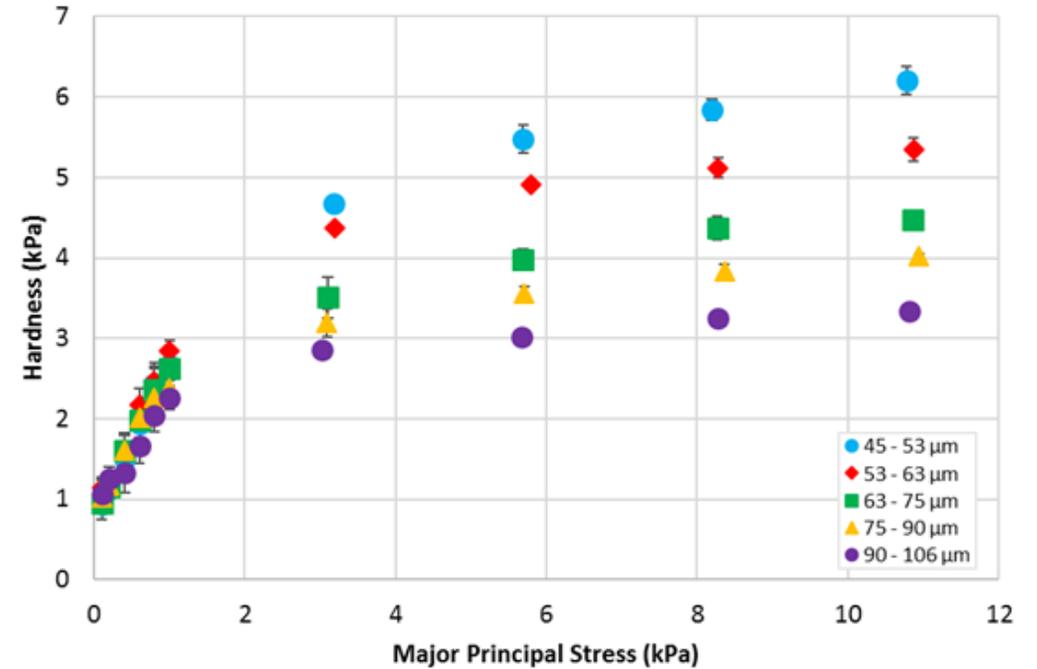
¹Hassanpour, A., Ghadiri, M., 2007, *Particle & Particle Systems Characterization*, 24 (2), 117-123.

Cohesive glass beads: size effect

➤ Effect of particle size



➤ Increase in size \rightarrow decrease in σ_c



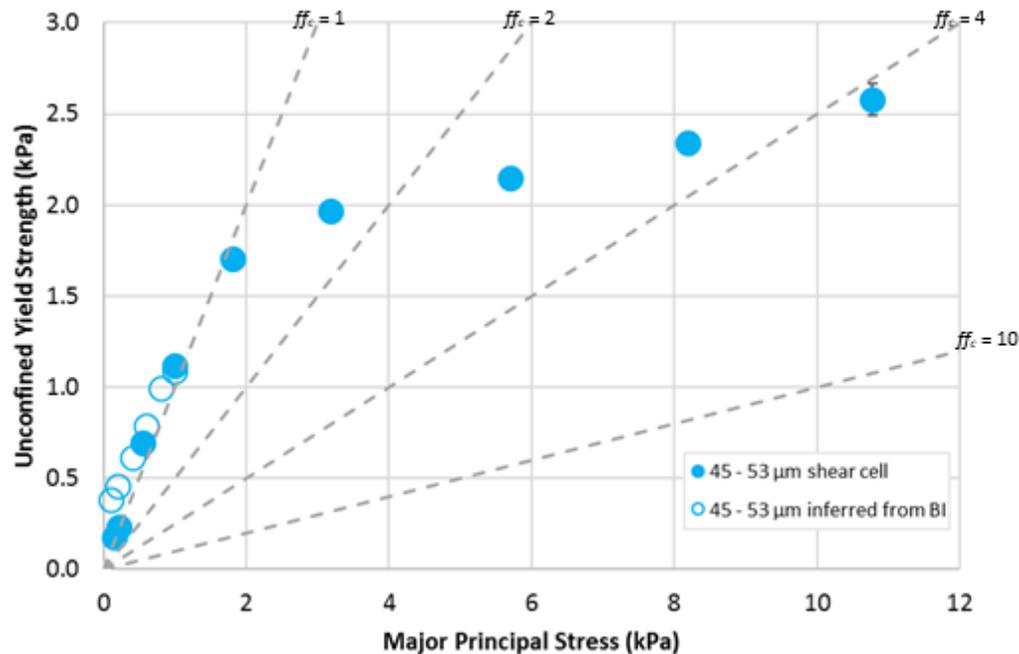
➤ Increase in size \rightarrow decrease in H

➤ H increase is much steeper at low stress

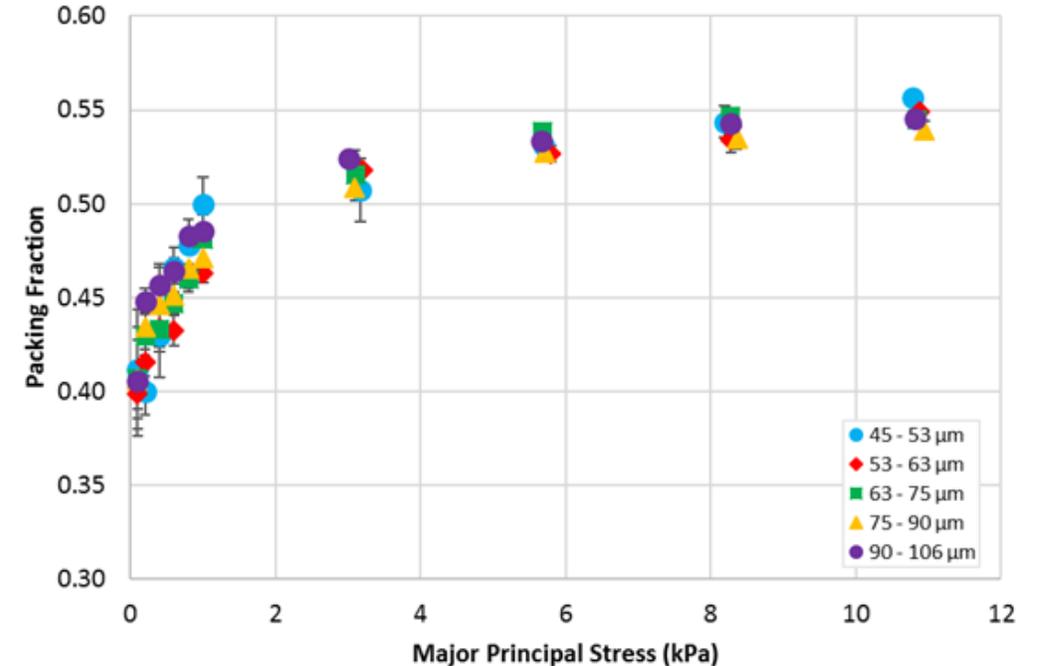
➤ Constraint factor found: $C = H/\sigma_c$

Cohesive glass beads: low stress behaviour

- Shear cells often not reliable at such low stresses
- Yield loci reliable for 45-53 μm sample

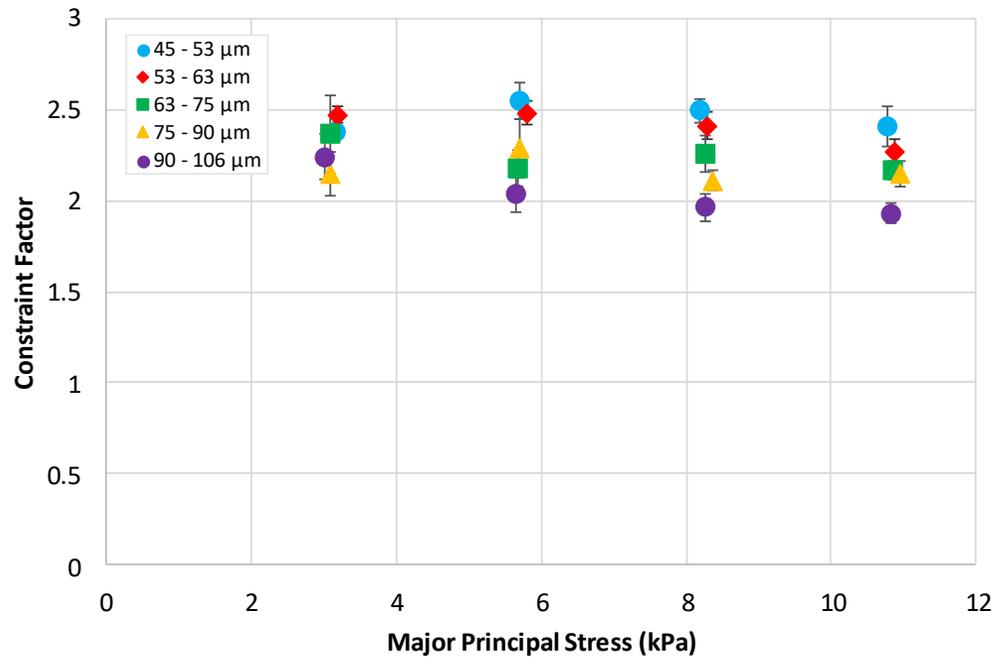


- σ_c agrees with that inferred from ball indentation



- Low stress behaviour:
 - ❑ H, σ_c increase sharply with stress
 - ❑ Corresponds with sharp increase in packing fraction

Cohesive glass beads: size distribution effect



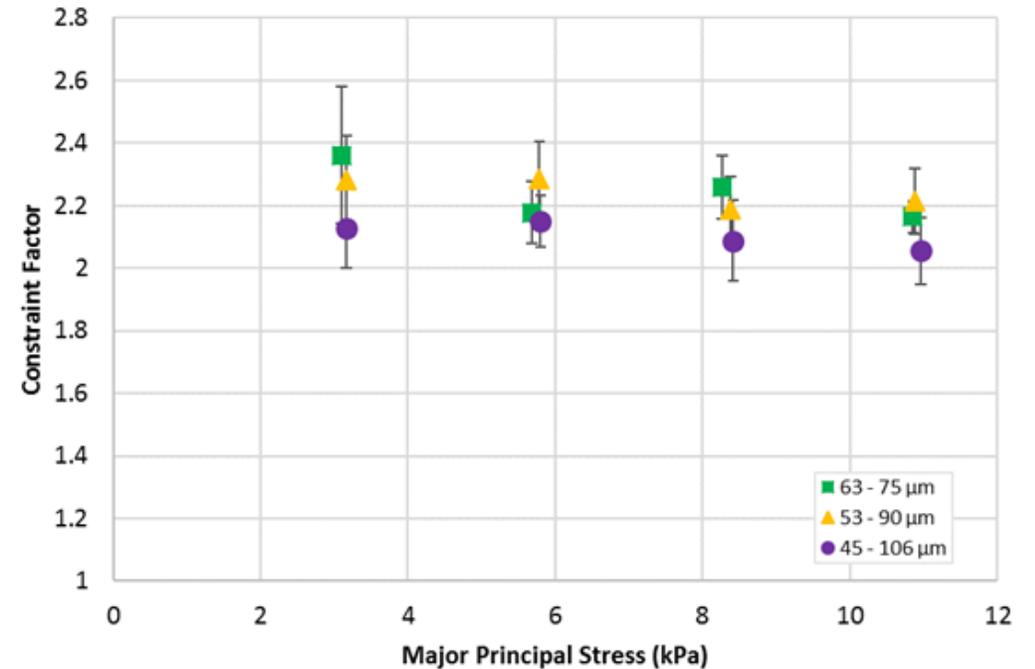
90-106 μm

75-90 μm

63-75 μm

53-63 μm

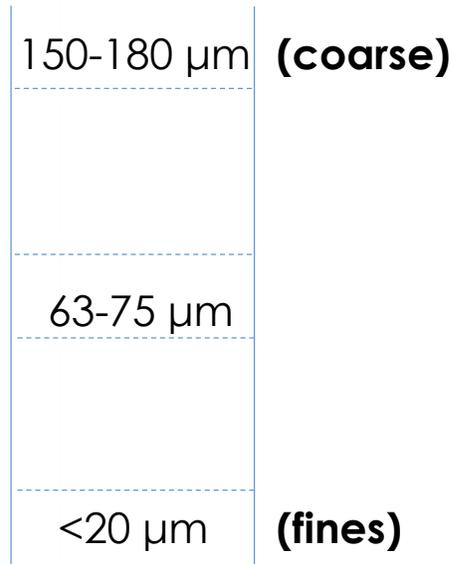
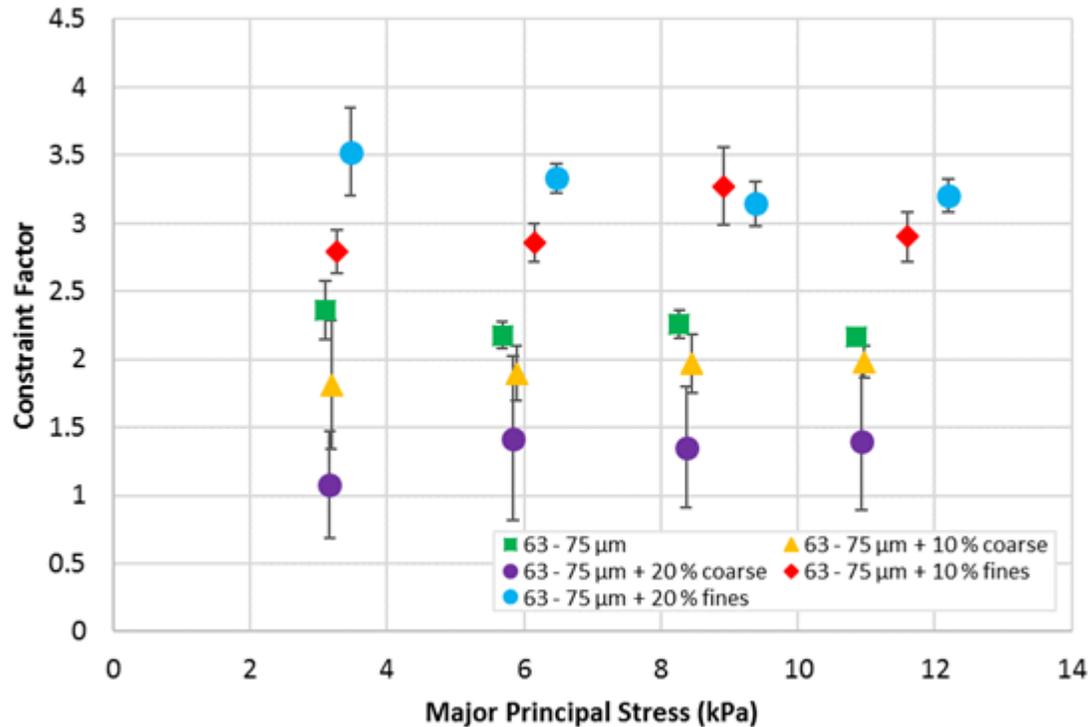
45-53 μm



➤ C generally decreases with increasing size

➤ C decreases as distribution span increased

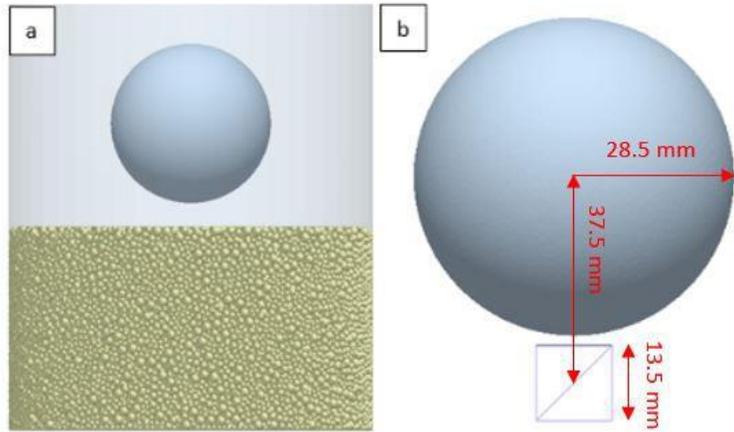
Cohesive glass beads: size distribution effect (2)



- Increased fines → C increases
- Increases coarse → C reduces
- Competing effects led to less pronounced C changes for wider span

Simulation results

- Discrete Element Method (DEM) used to manipulate particle properties²



- Stresses determined below indenter:

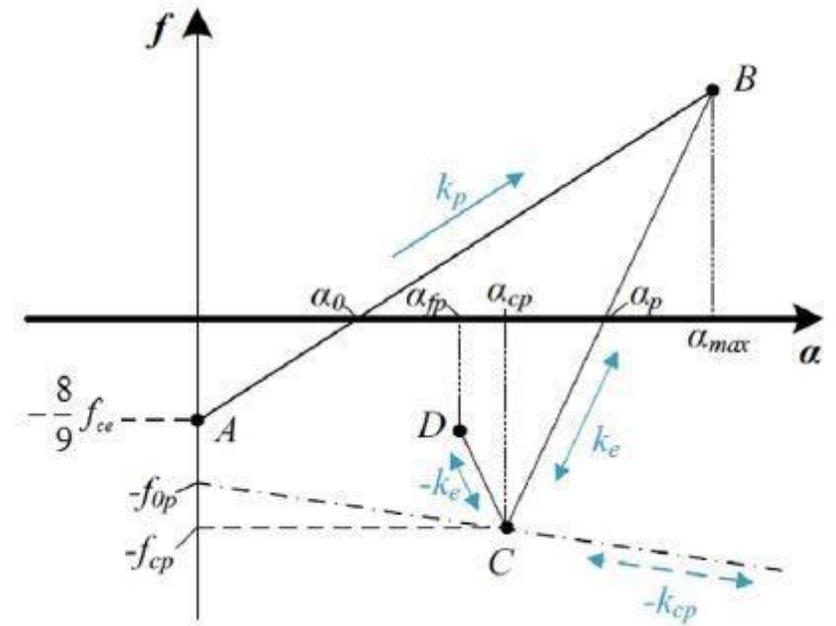
$$\sigma_{ij} = -\frac{1}{V} \sum_{N_p} \sum_{N_c} |x_i^c - x_i^p| n_i F_j$$

$$\tau_D = \sqrt{\frac{(\sigma_1 - \sigma_3)^2 + (\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2}{6}}$$

- $C' = H/\tau_D$

- Pasha *et al.* (2014)⁵ model, accounts for:

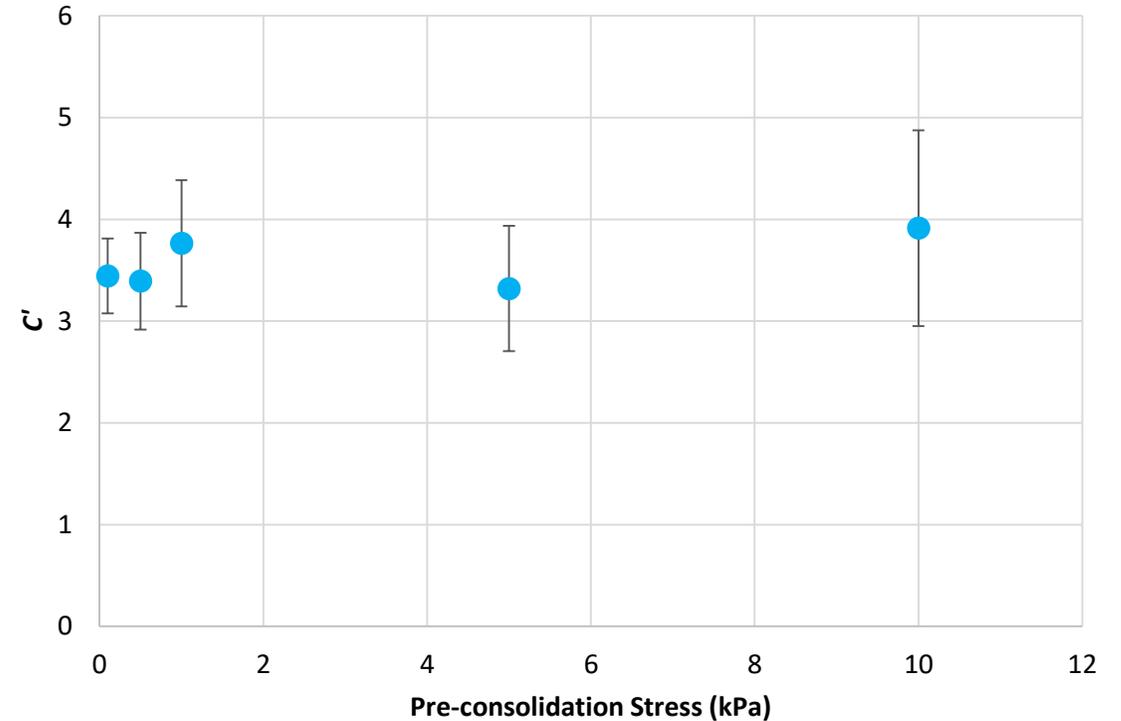
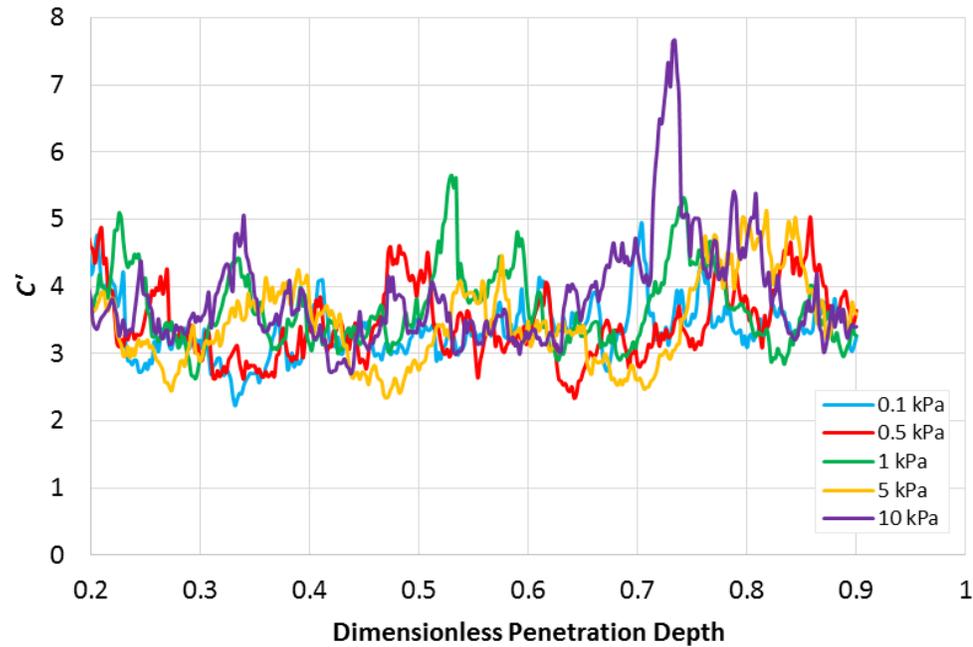
- ❑ Plasticity
- ❑ Adhesion



²Stavrou, A., Hare, C., Hassanpour, A., Wu, C-Y., 2020, *Chemical Engineering Science*, 211, 115307.

⁵Pasha, M., Dogbe, S., Hare, C., Hassanpour, A., Ghadiri, M., 2014, *Granular Matter*, 16, 151-162.

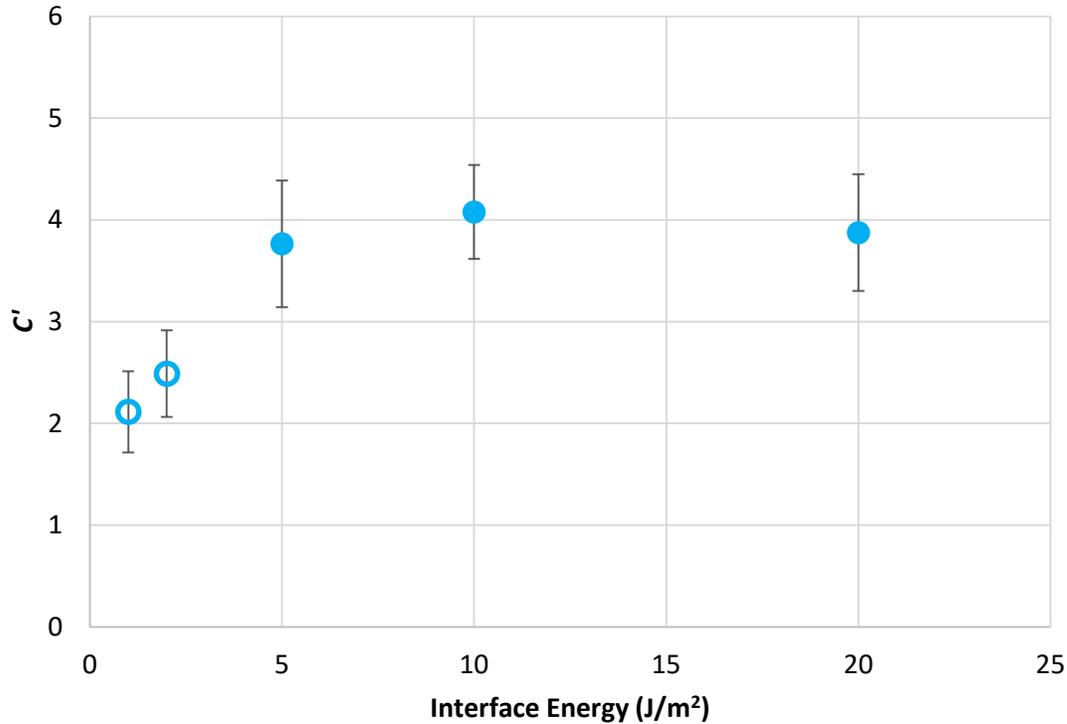
Stability of constraint factor



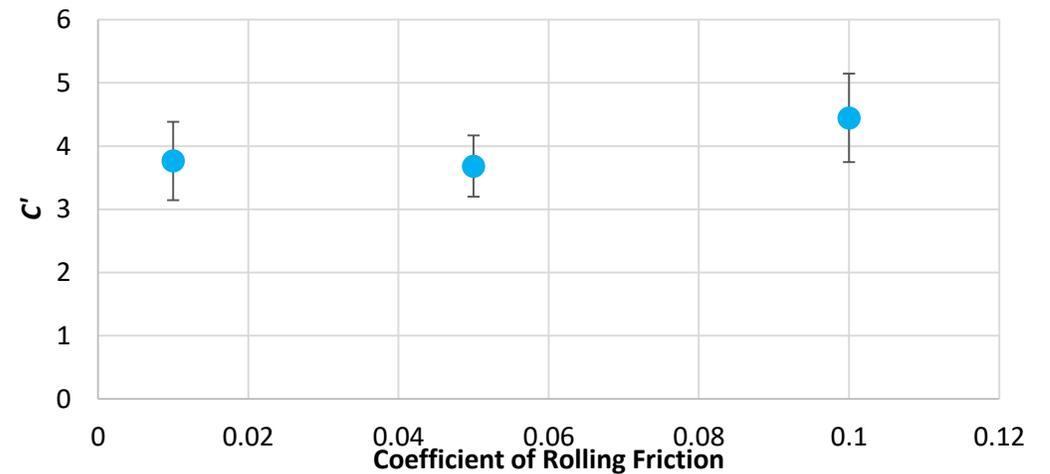
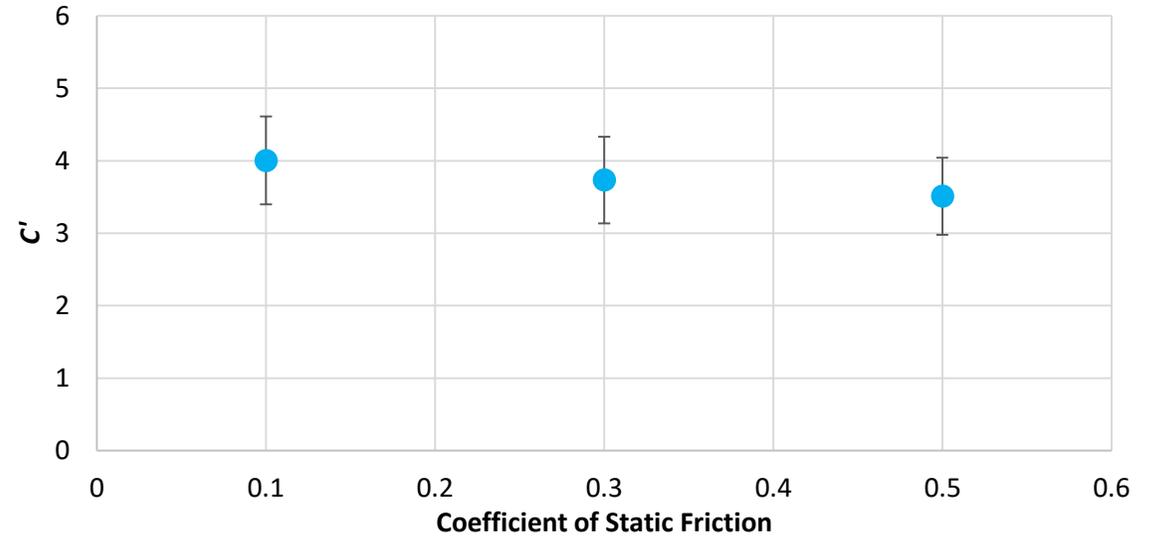
➤ Significant C' fluctuations with depth

➤ Average C' independent of stress

Effects of surface energy & friction

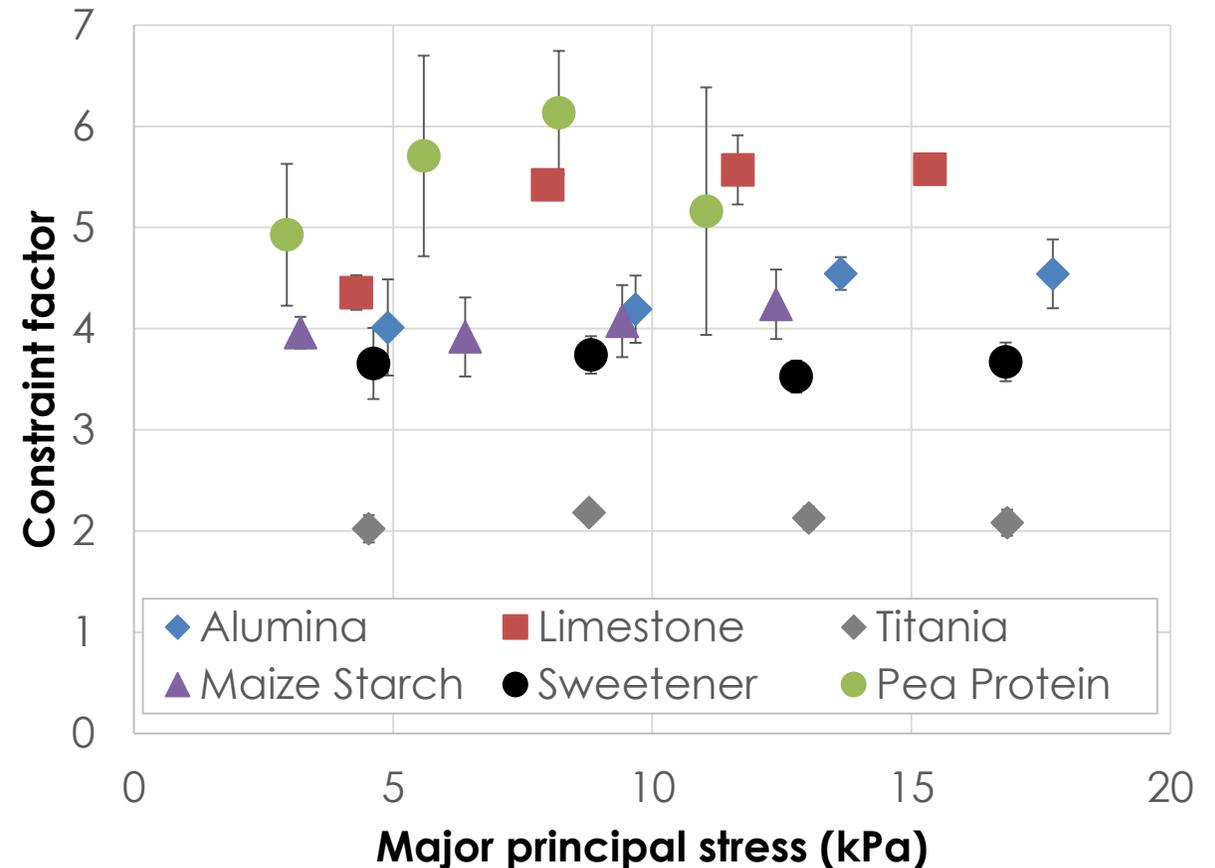


- C' increases with Γ for slight cohesion
- C' virtually independent of μ_s
- C' increases slightly for increasing μ_r

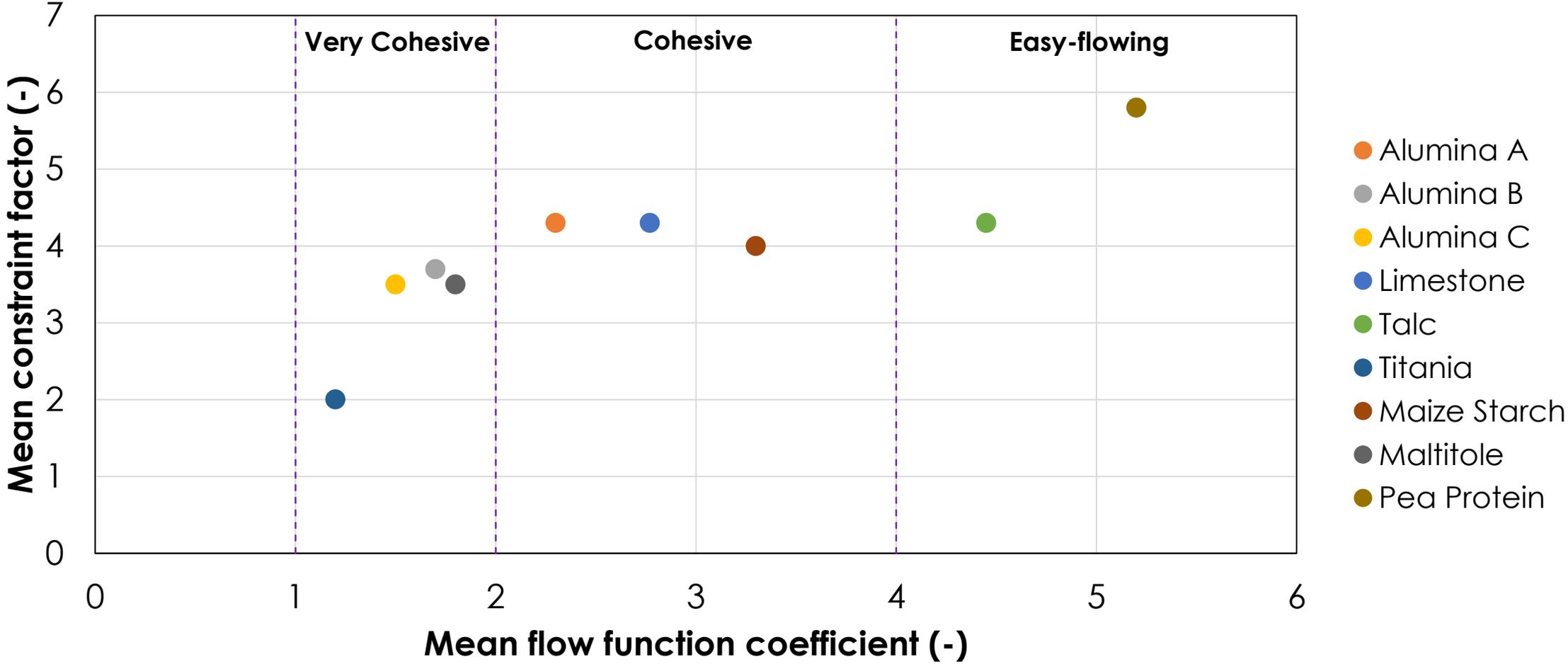


Real materials

- Methods applied to non-ideal powders
 - ❑ Shear cell measurements at $\sigma_{pre} = 2, 4, 6, 8$ kPa
 - ❑ Indentation at σ_1
- Constraint factor stable for most
 - ❑ Pea protein exhibited slip-stick behaviour
- Value of C ranges from 2-6
 - ❑ For materials tested
- Actual value of C depends on method used to determine σ_c



Influence of mean flow function coefficient



Relationship between C and ff_c

➤ Since: $ff_c = \frac{\sigma_1}{\sigma_c}$ (1),

$$C = \frac{H}{\sigma_c} \quad (2)$$

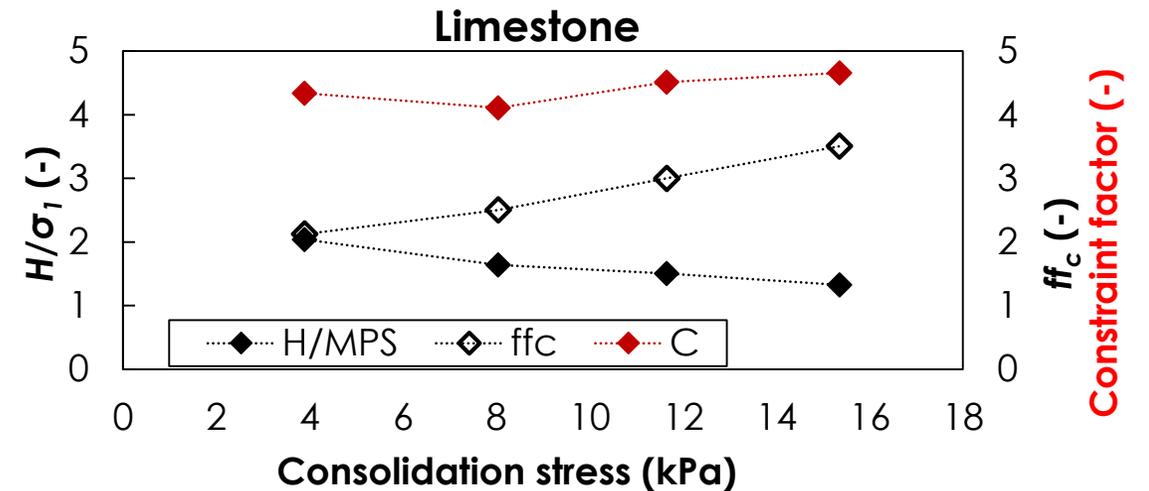
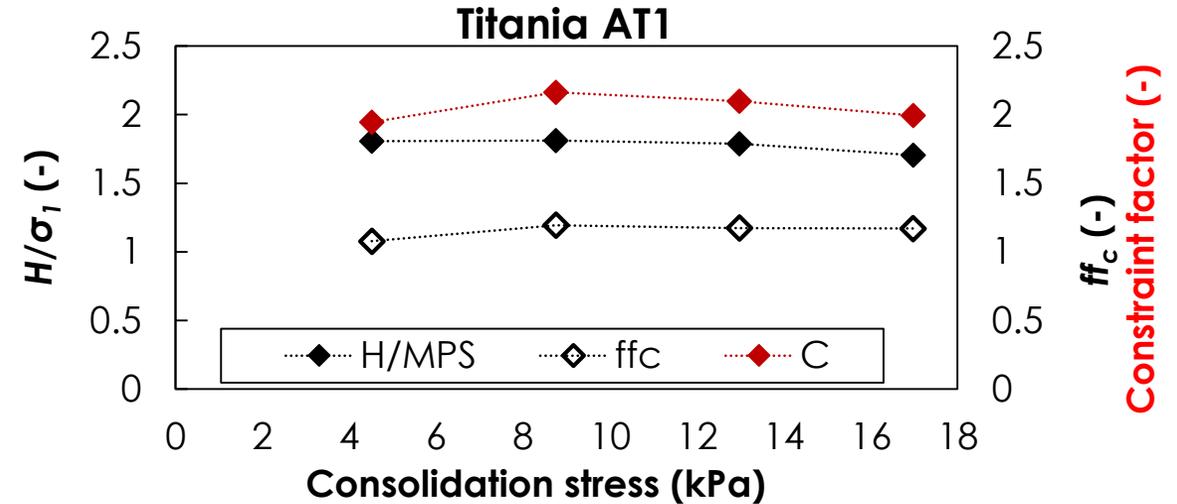
➤ Then: $C = \frac{H}{\sigma_1} ff_c$ (3)

➤ Constraint factor (C) will be constant if:

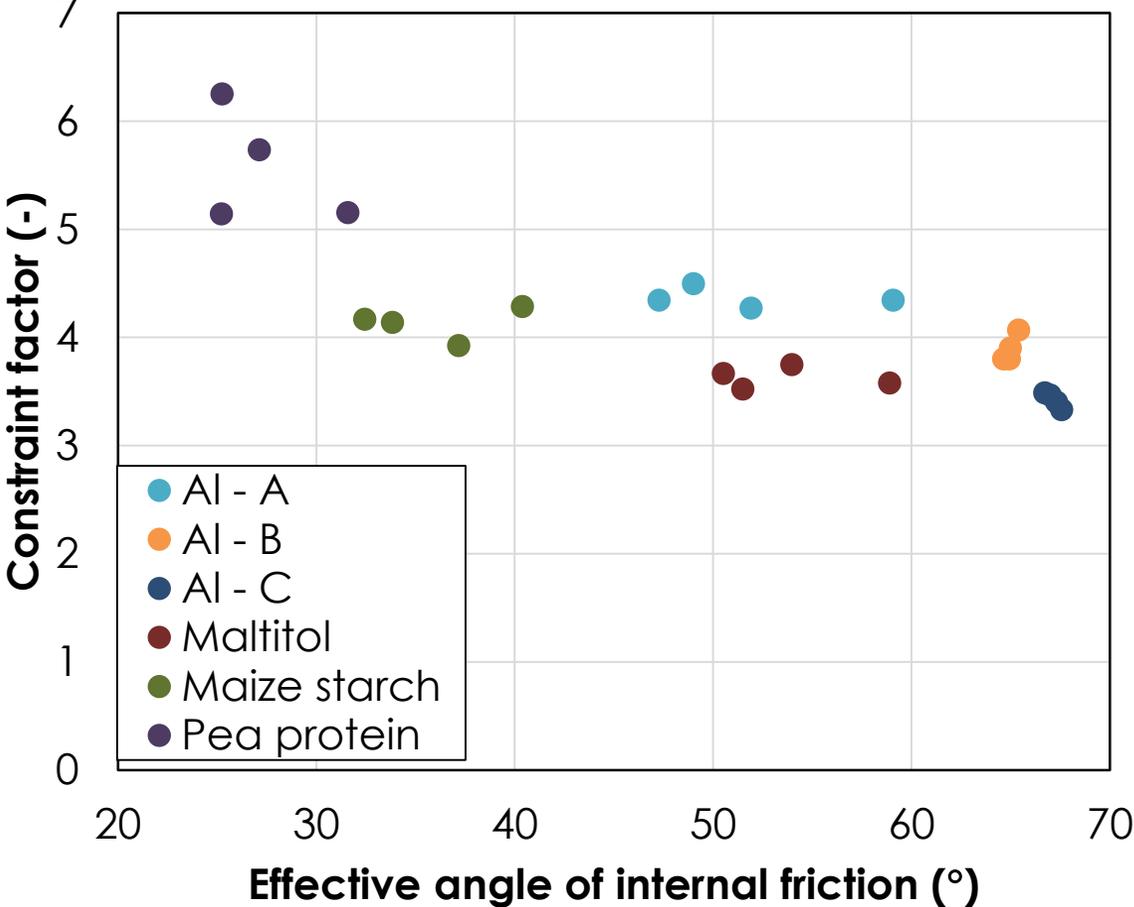
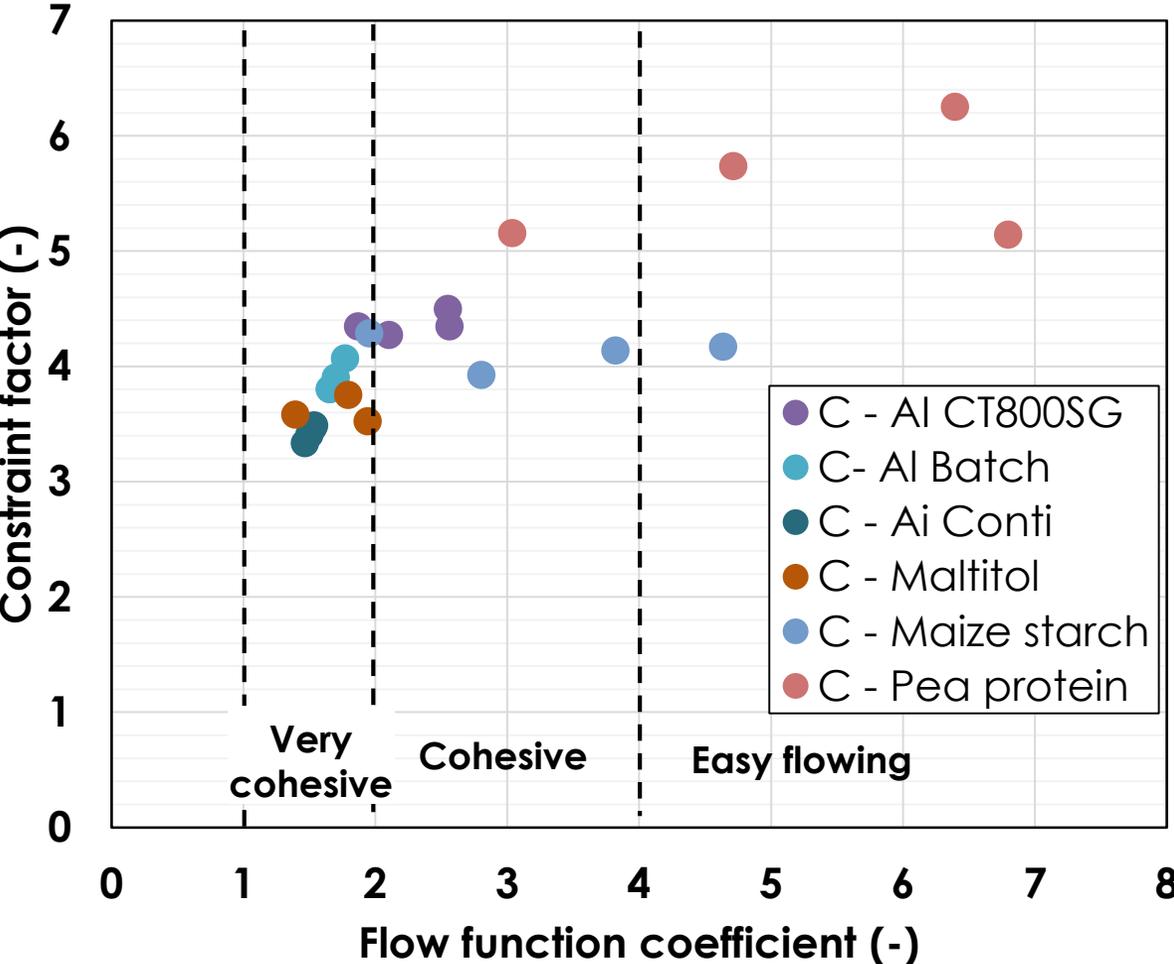
□ ff_c and (H/σ_1) are independent of consolidation stress,

or

□ ff_c increases, while (H/σ_1) decreases at the same rate, with increasing stress



Influence of flow function coefficient and friction angle



Critical method

| | Shear cell | Uniaxial compression | Ball indentation (original method) ¹ | Ball indentation (critical method) |
|---------------|--|--|---|--|
| Consolidation | | | | |
| Failure | | | | |
| Measurement | Control: σ Measure: τ Determine: σ_1 & σ_c | Control: σ (σ_1) Measure: σ_c (uUYS) | | Control: σ (σ_1) Measure: H Determine: C, σ_c |

¹Hassanpour, A., Ghadiri, M., 2007, Particle & Particle Systems Characterization, 24 (2), 117-123.

Indentation onto sheared beds

Standard shear test: requires extreme cohesion



Shear test using wall friction head: insufficient pre-shear



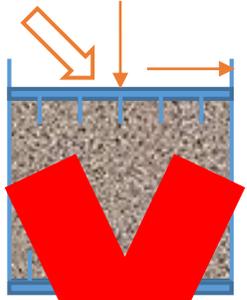
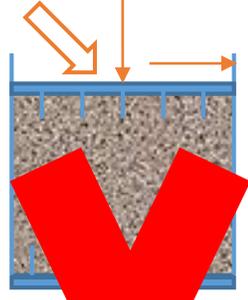
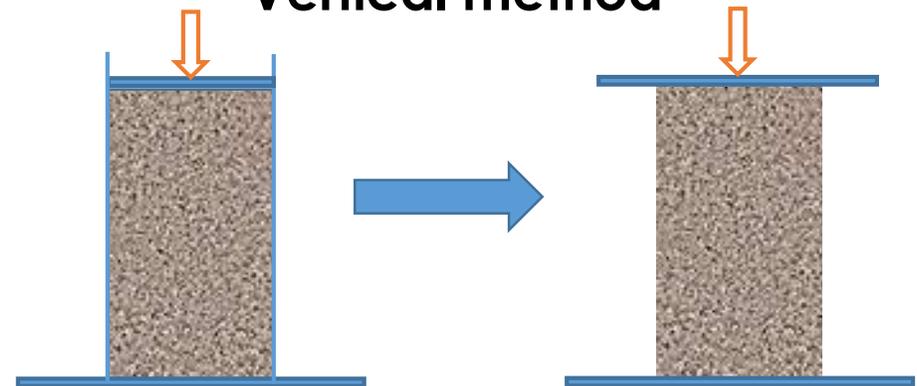
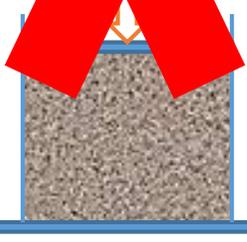
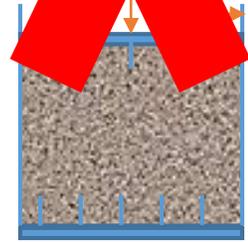
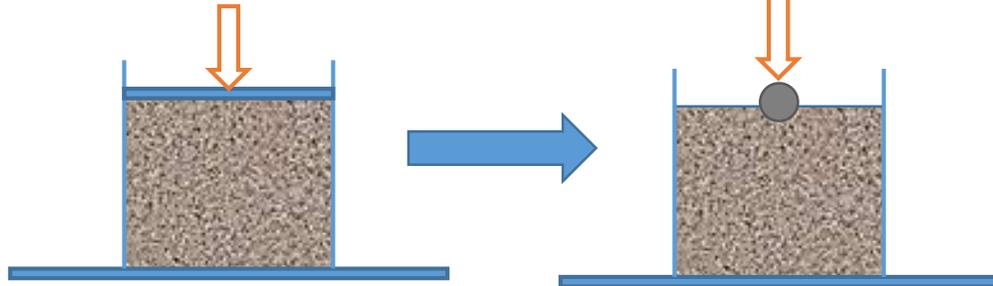
Splitting after shear test: induces unknown shear stress



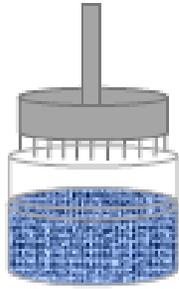
Vacuum suction after shear test: unknown suction force



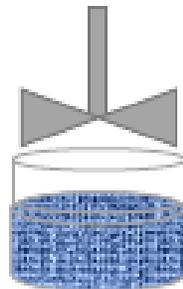
Defining constraint factor

| | Mixed method | Critical method | Vertical method |
|---------------------------------|--|---|---|
| Unconfined yield strength |  <p>Shell</p> |  <p>Shell</p> |  <p>Uniaxial compression</p> |
| Bed preparation for indentation |  |  |  |
| Comment | Packing state differs | Difficult to provide a flat surface for indentation | Packing state replicated in indentation test $\sigma_{uUYS} < \sigma_c$, though reliable as flow indicator |

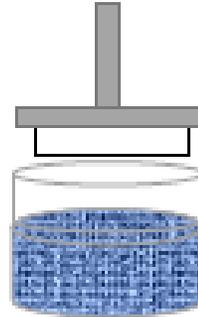
Bed preparation



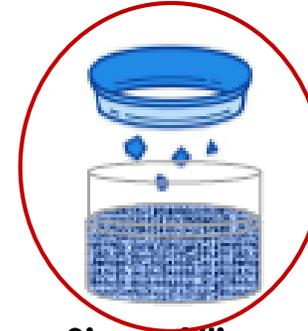
Pre-shearing



Blade conditioning



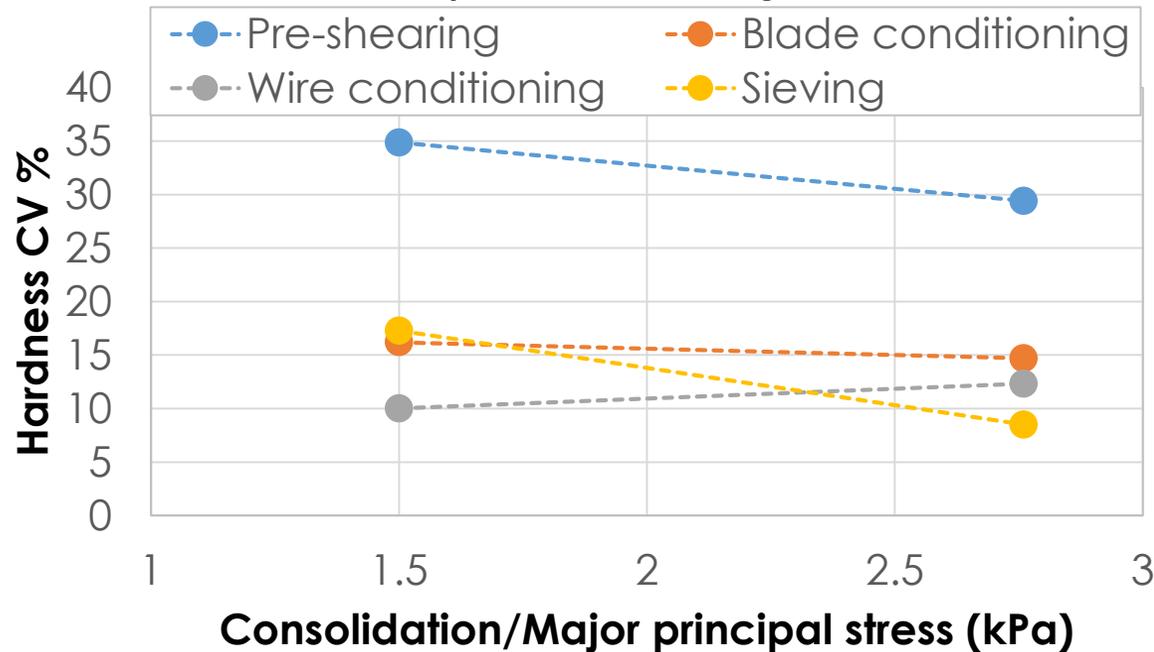
Wire conditioning



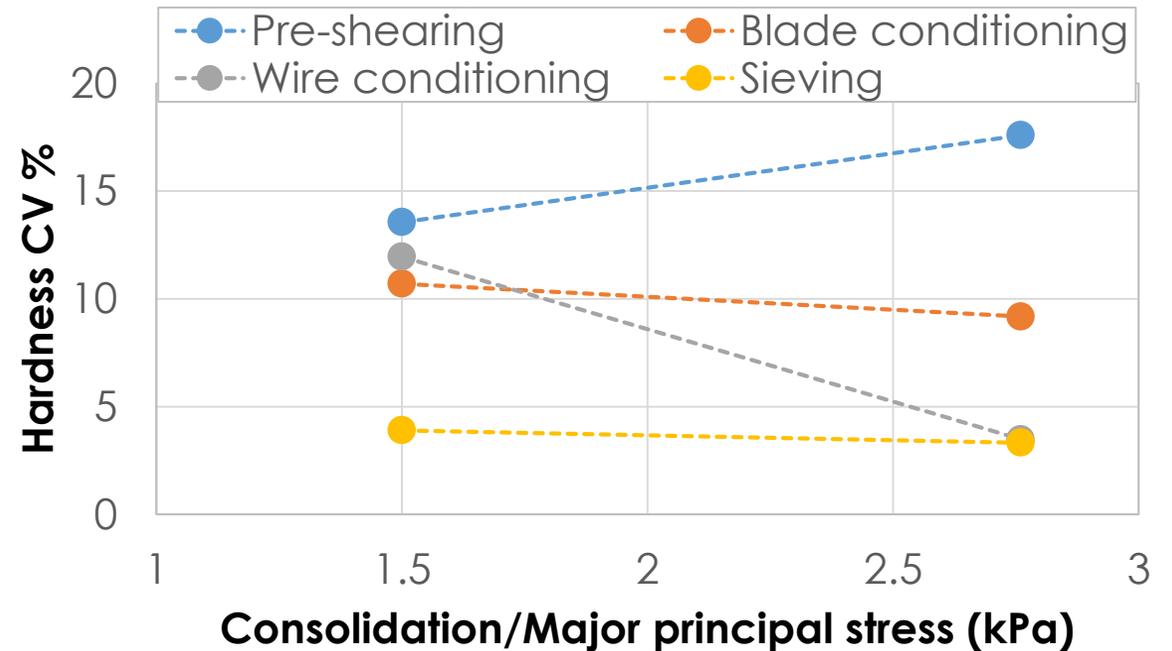
Sieve-filling

Best reproducibility (CV < 5%)

Variation for 3 indents on a single bed



Variation for indents on 5 separate beds



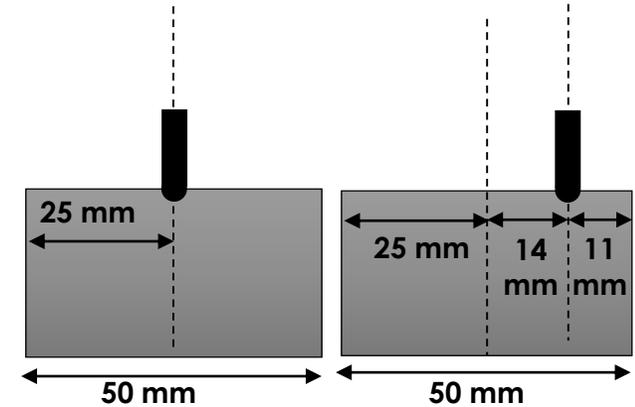
Influence of indentation method setup

How indentation test arrangements may affect

- Bed hardness
- C value
- Inferred σ_c

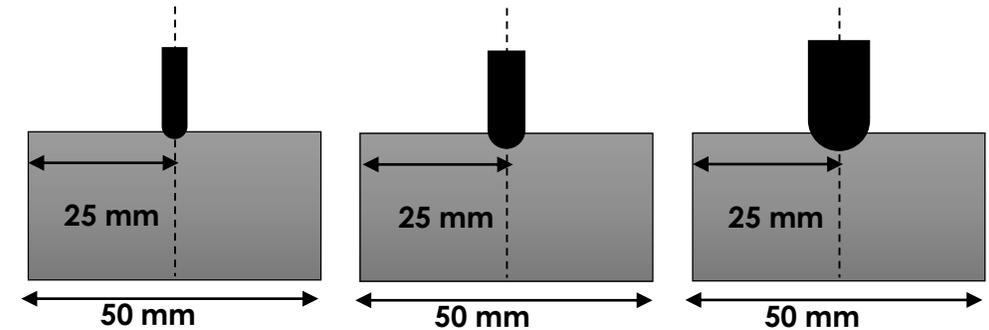
➤ Indentation position

- At central and radial positions (4 mm indenter)



➤ Indenter size

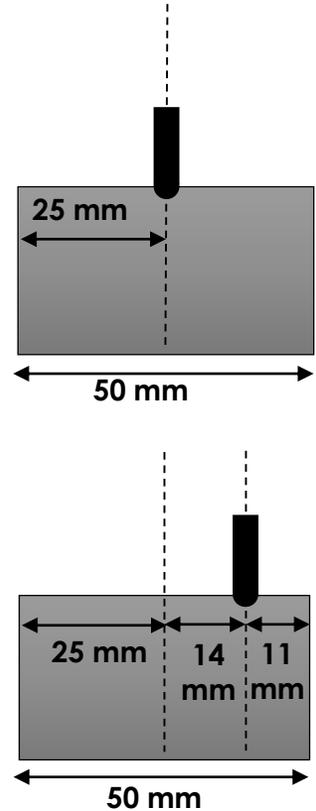
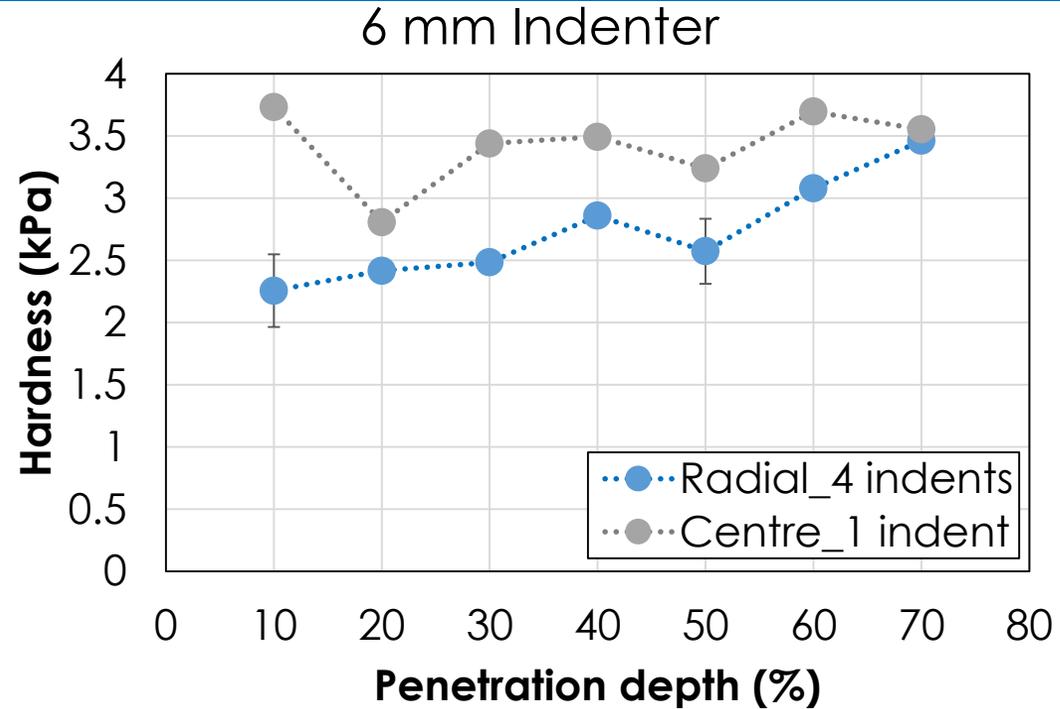
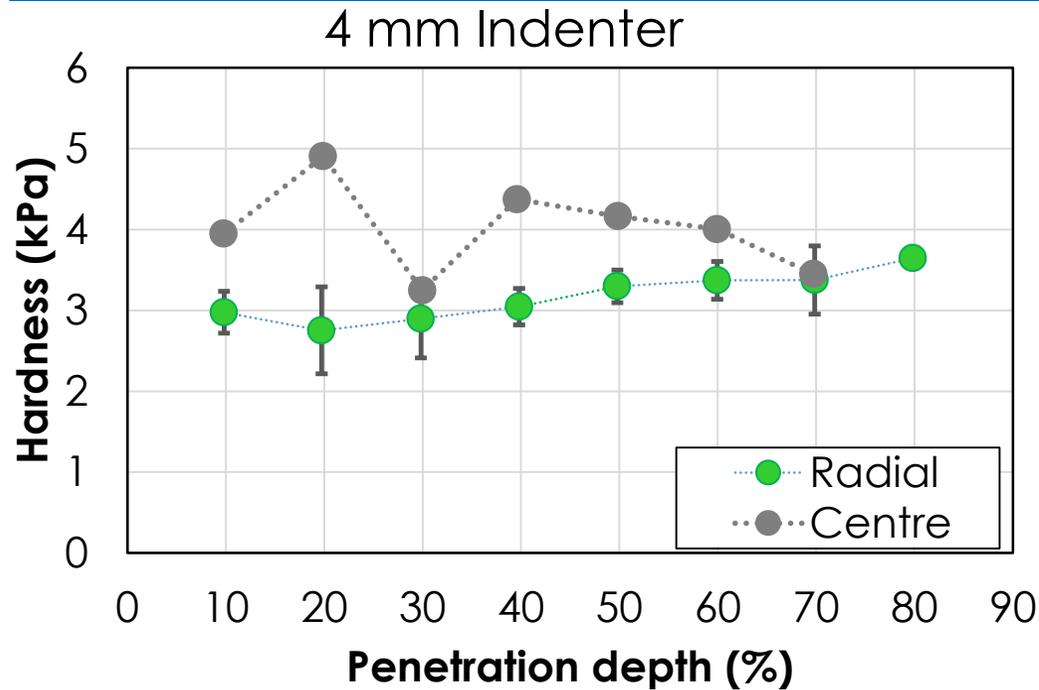
- 4, 6 and 10 mm indenters, indenting at the centre



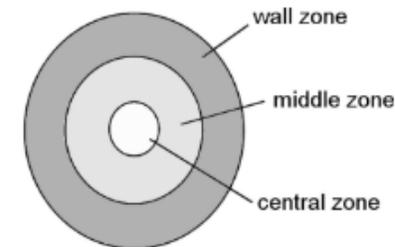
➤ Penetration depth

- At penetration depths of **20%** and **50 %** of indenter radius (4 mm indenter)

Indenter position



- Hardness greater in the centre
 - ☐ Agrees with findings of Hassanpour and Ghadiri (2007)¹
- This may be due to greater packing fraction in the centre

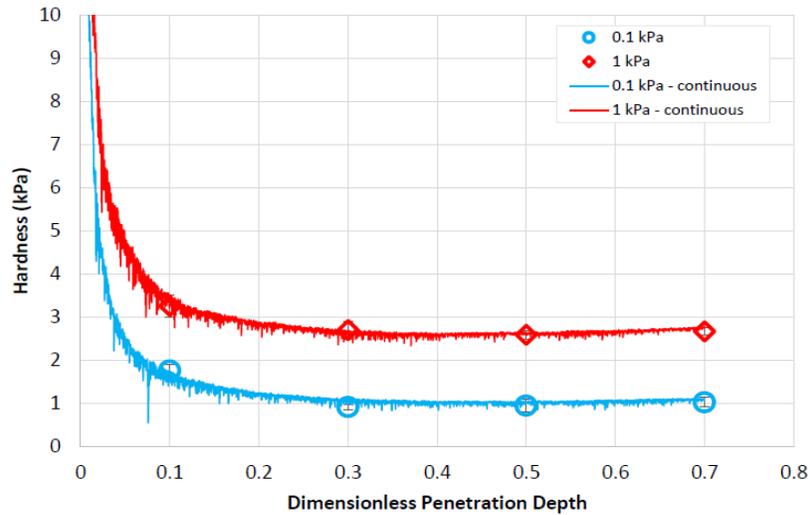


Indentation zone classification¹

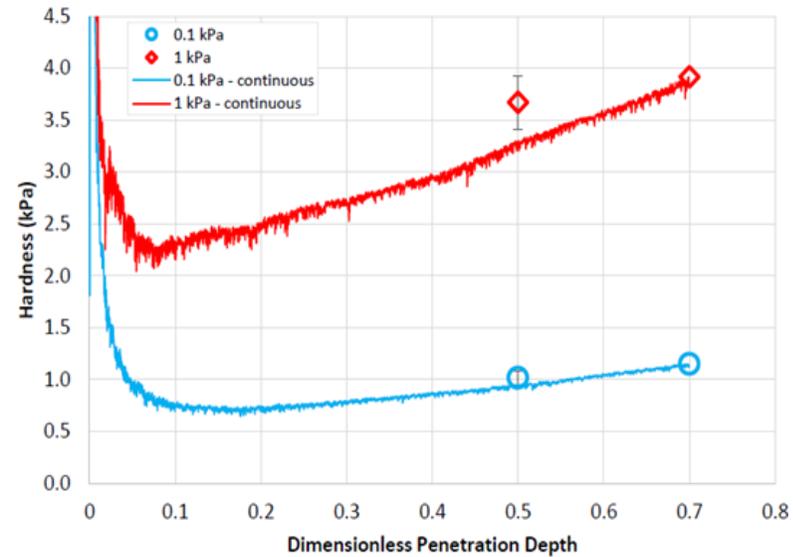
¹Hassanpour, A., Ghadiri, M., 2007, *Particle & Particle Systems Characterization*, 24 (2), 117-123.

Penetration depth: influence on hardness

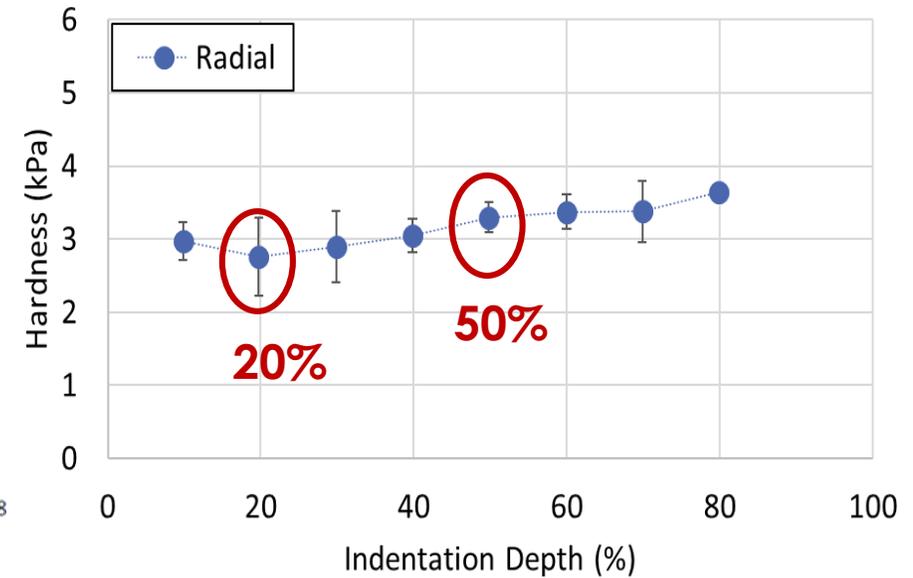
Silanised glass beads 63-75 μ m⁴



Alumina⁶



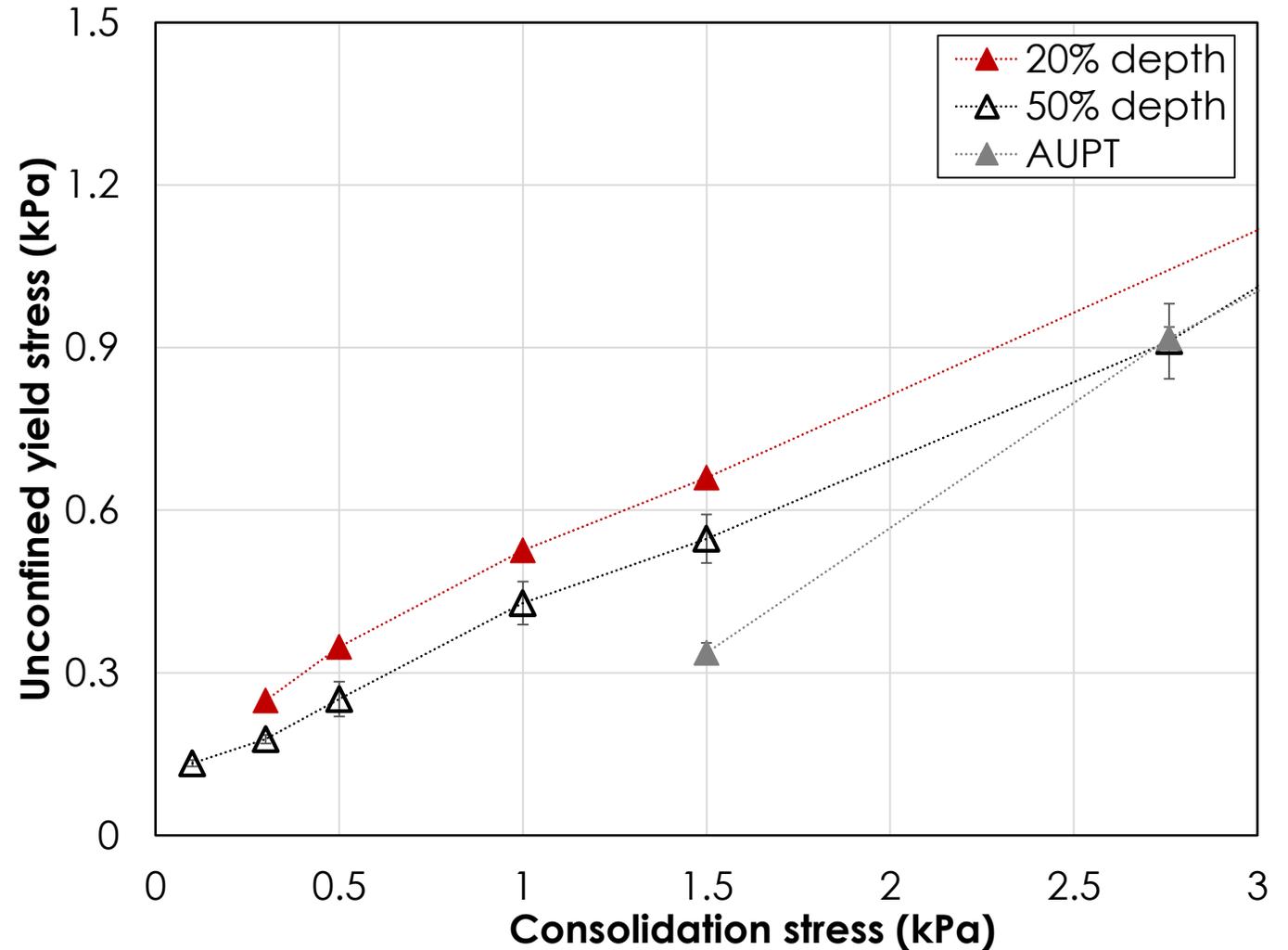
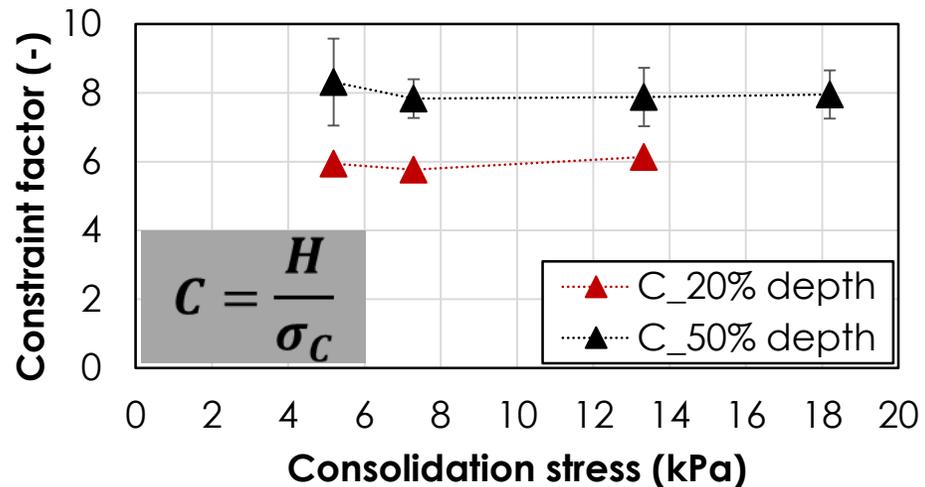
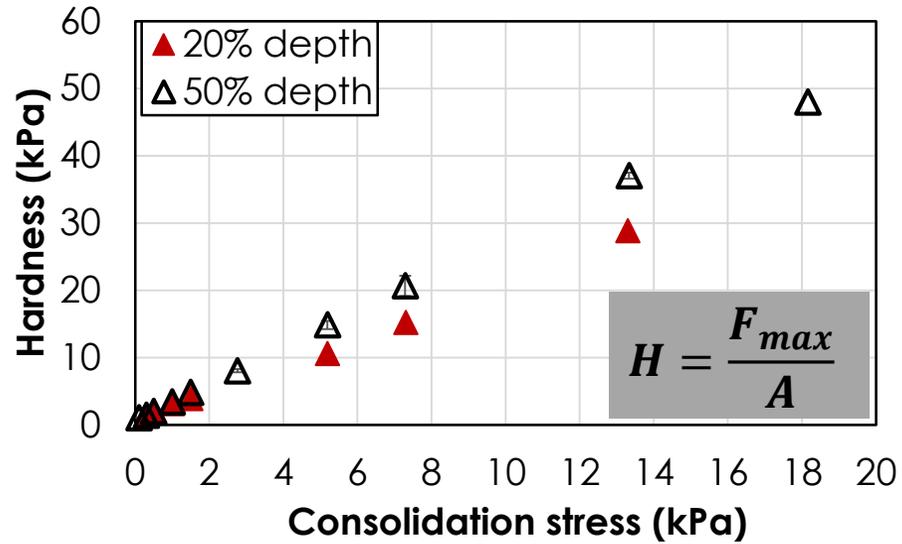
Titania – at 1 kPa



⁴Stavrou, A., Hare, C., Hassanpour, A., Wu, C-Y., 2020, *Powder Technology*, 364, 98-114.

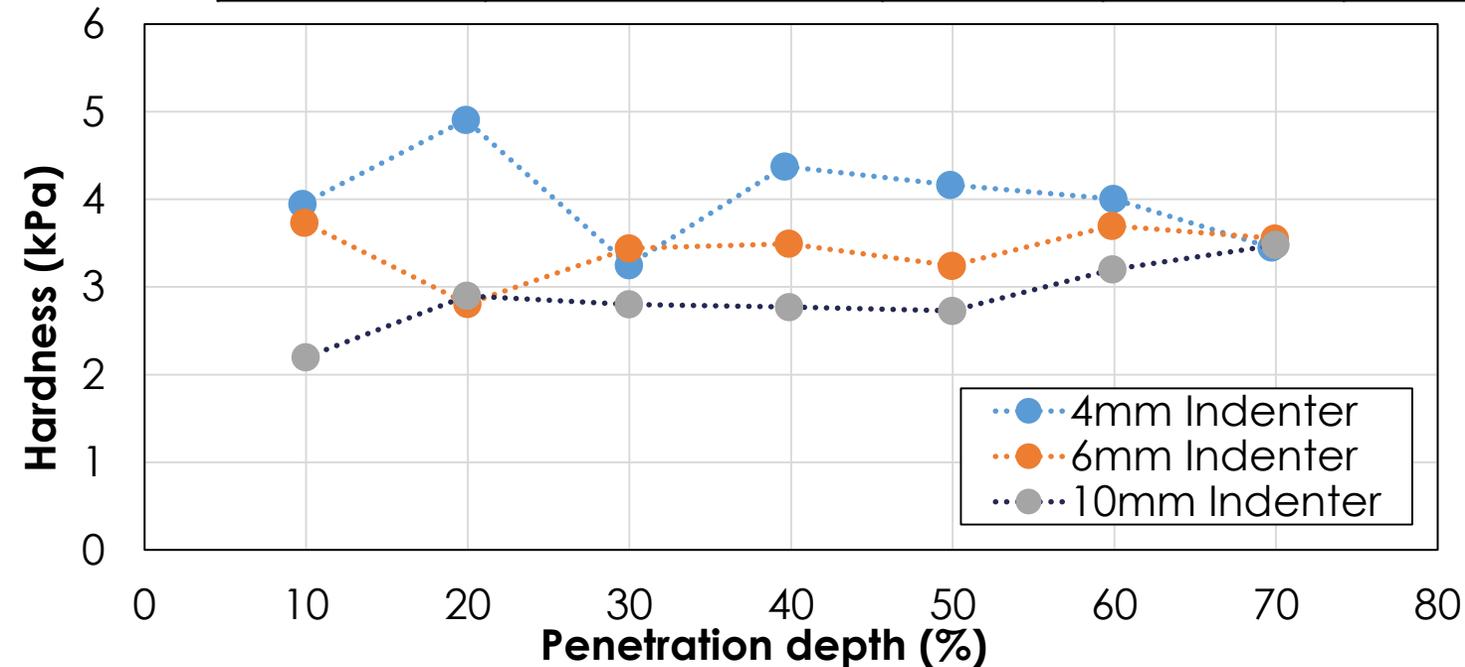
⁶Stavrou, A., 2019, Doctoral Thesis, *University of Surrey*.

Penetration depth: influence on C and νUYS



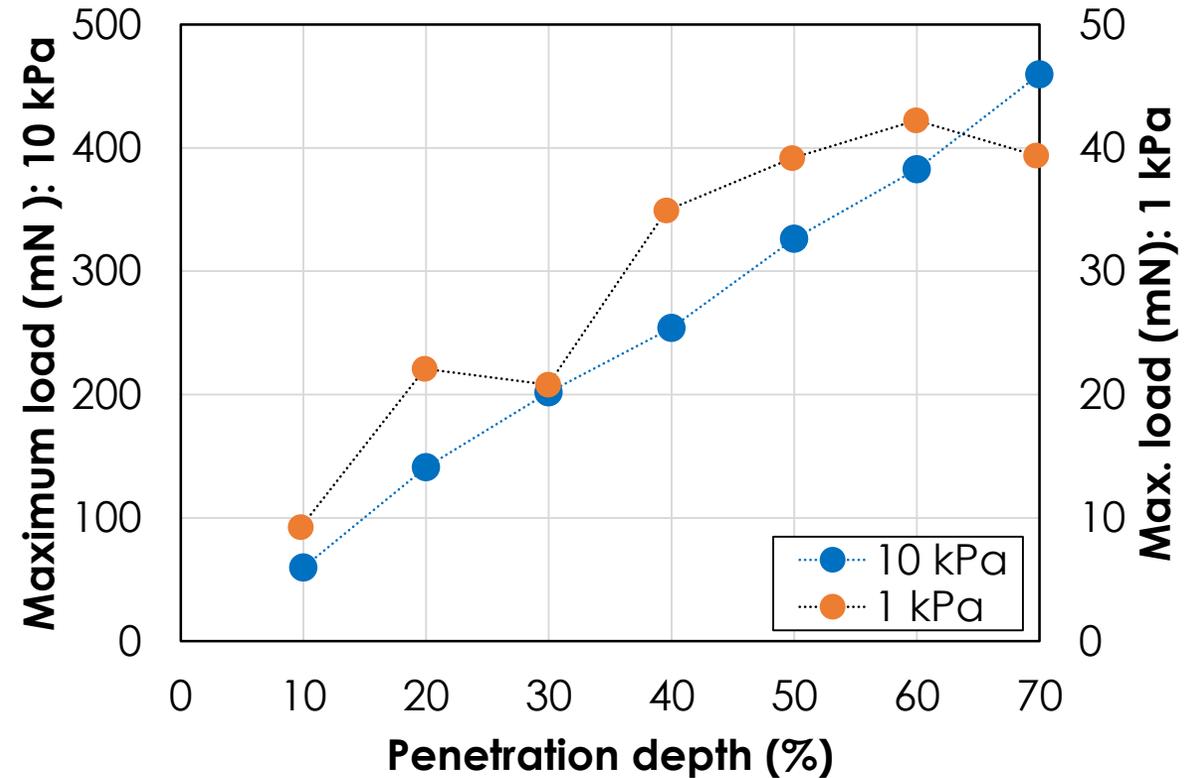
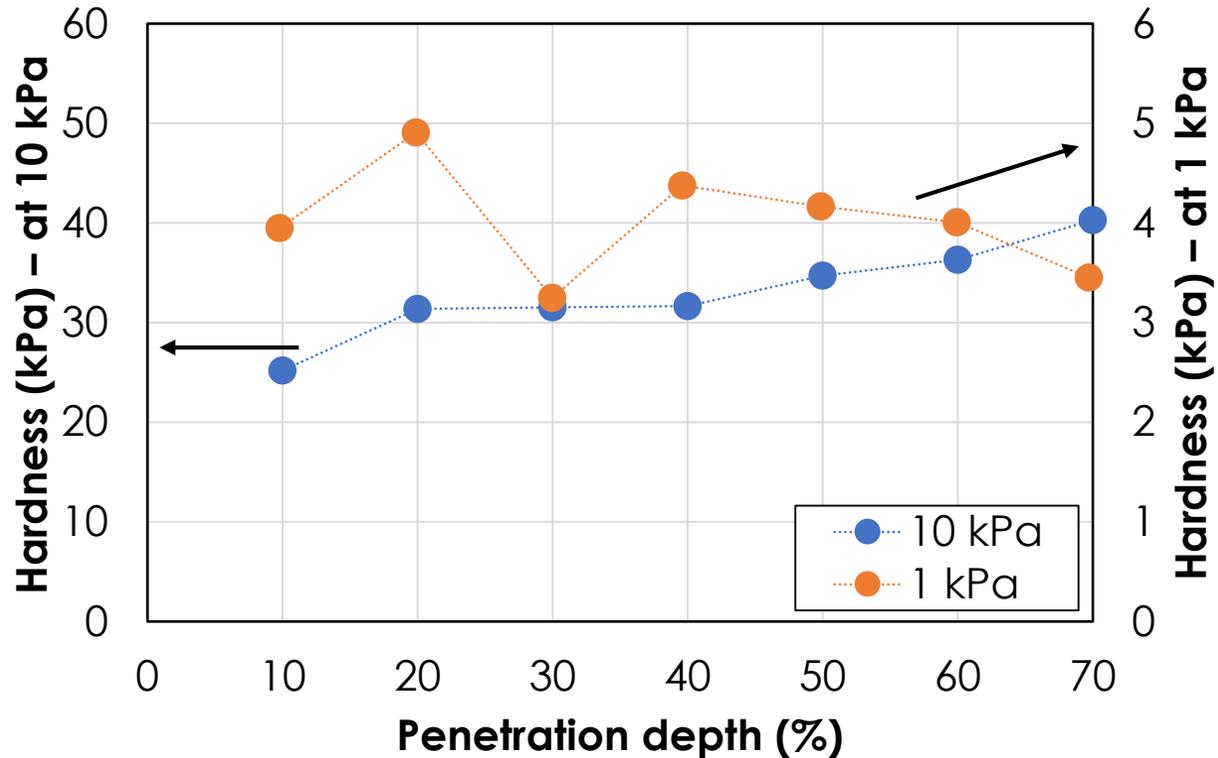
Indenter size

| d_{Ind} | Depth | 10 % | | 20 % | | 30 % | | 40 % | | 50 % | | 60 % | | 70 % | |
|-----------|-------|--------|----------------|------|-----------|------|-----------|------|-----------|------|-----------|------|-----------|------|-----------|
| | | h (mm) | F_{max} (mN) | h | F_{max} |
| 4 mm | | 0.2 | 9.23 | 0.4 | 22.1 | 0.6 | 20.8 | 0.8 | 34.9 | 1 | 39.2 | 1.2 | 42.3 | 1.4 | 39.4 |
| 6 mm | | 0.3 | 19.9 | 0.6 | 28.5 | 0.9 | 50 | 1.2 | 63 | 1.5 | 68.7 | 1.8 | 87.7 | 2.1 | 91.4 |
| 10 | | 0.5 | 32.7 | 1 | 81.7 | 1.5 | 112.1 | 2 | 139 | 2.5 | 160 | 3 | 211 | 3.5 | 248.6 |



- Fluctuations largest for smallest size
 - ❑ Smaller force measured
- Largest indenter gives most consistent results
- H is lower for larger indenters
 - ❑ Suggests F_{max} is overestimated for small indenters

Measurement accuracy: consolidation stress

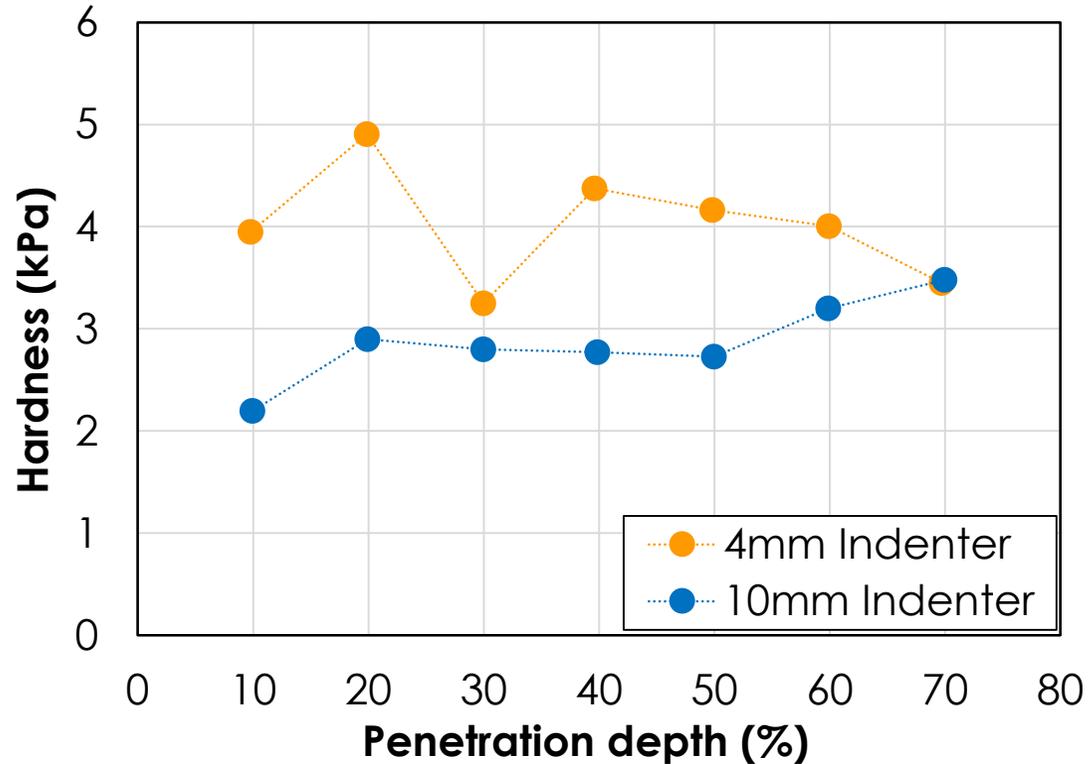


- Lower force measured at low consolidation stress
- Hence greater fluctuations in hardness with depth

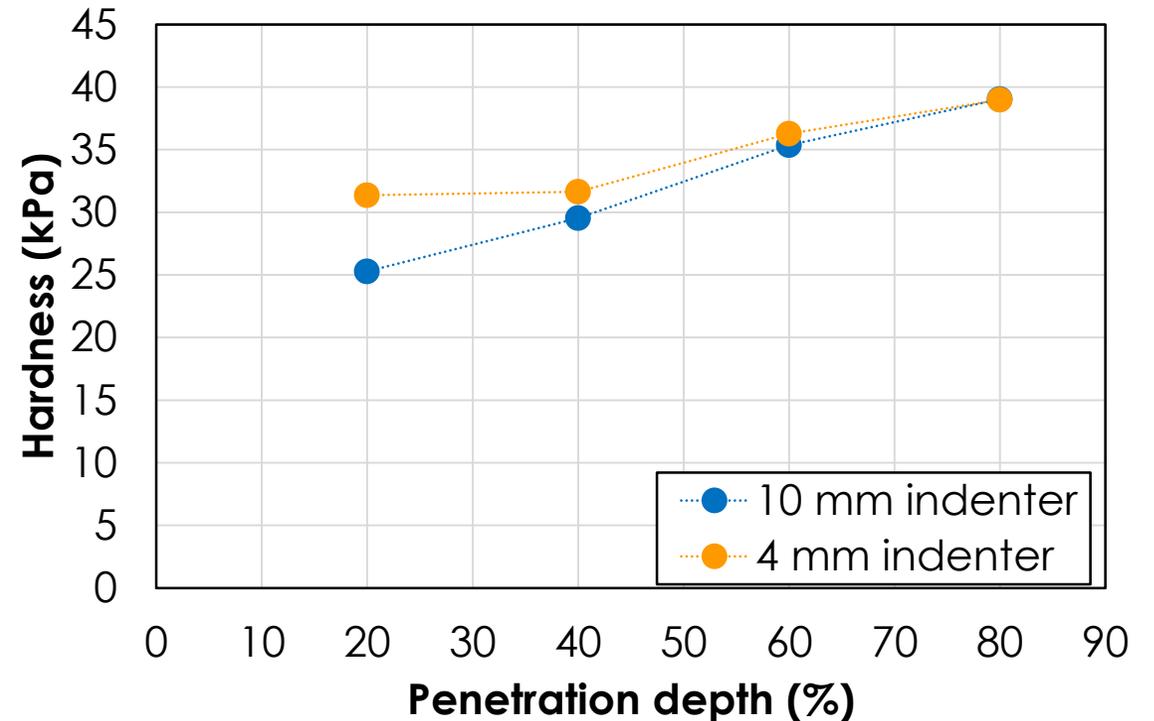
$$H = \frac{F_{max}}{A} = \frac{F_{max}}{\pi(dh - h^2)}$$

Measurement accuracy: indenter size

1 kPa consolidation



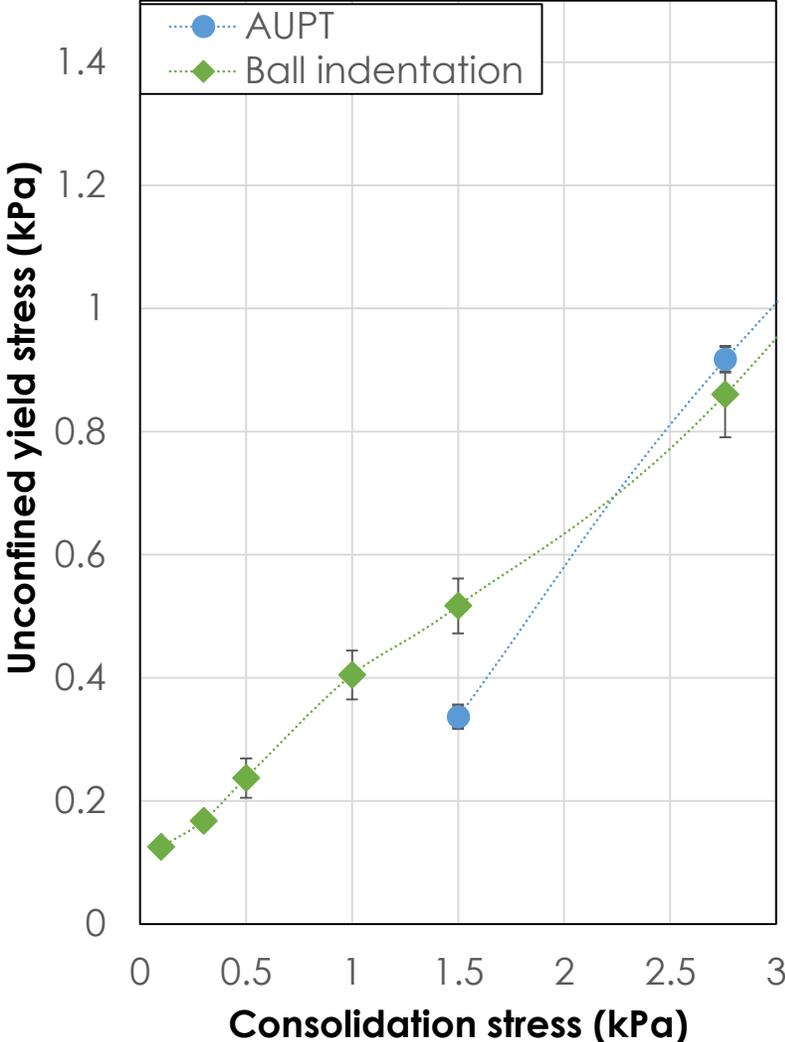
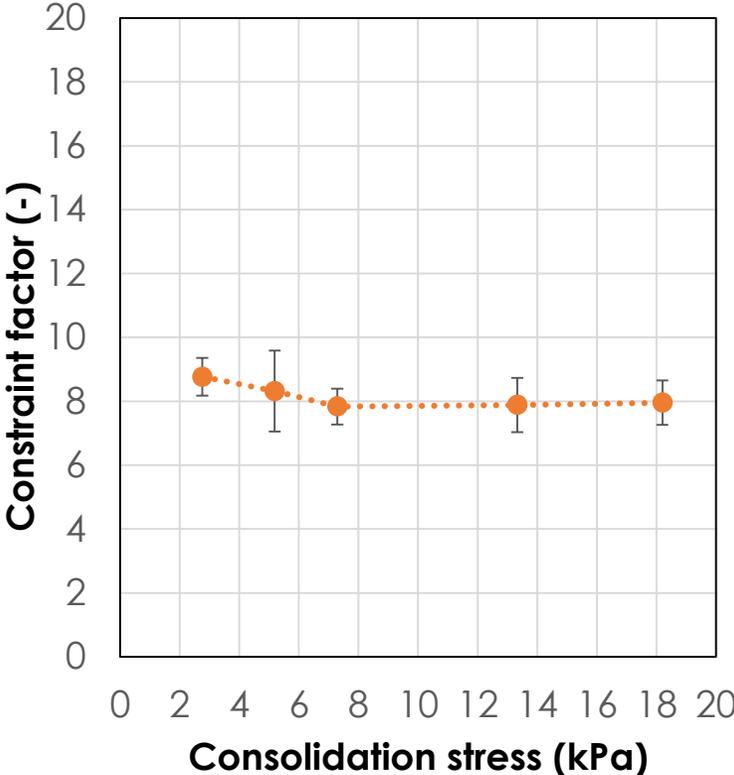
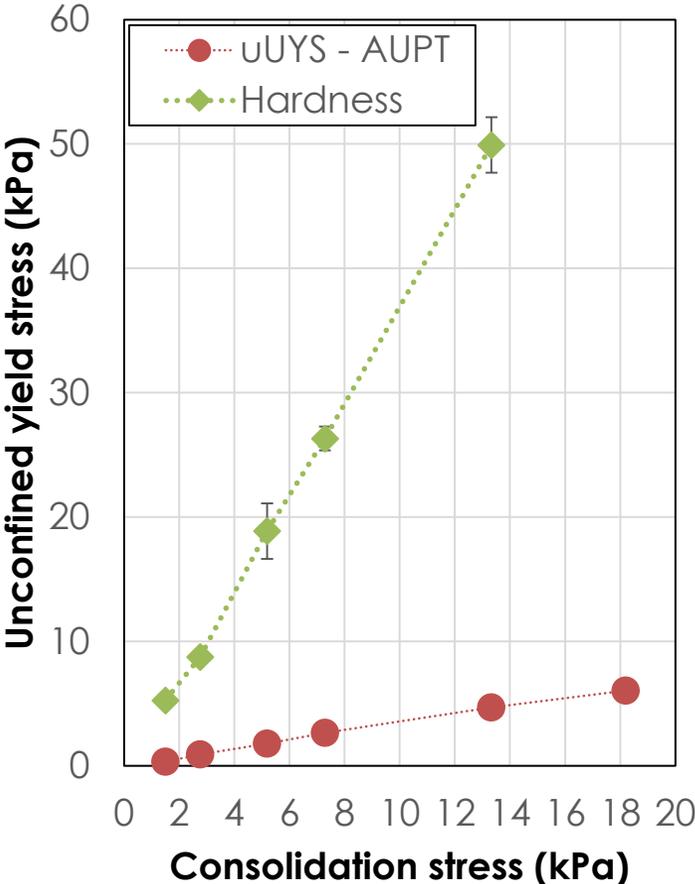
10 kPa consolidation



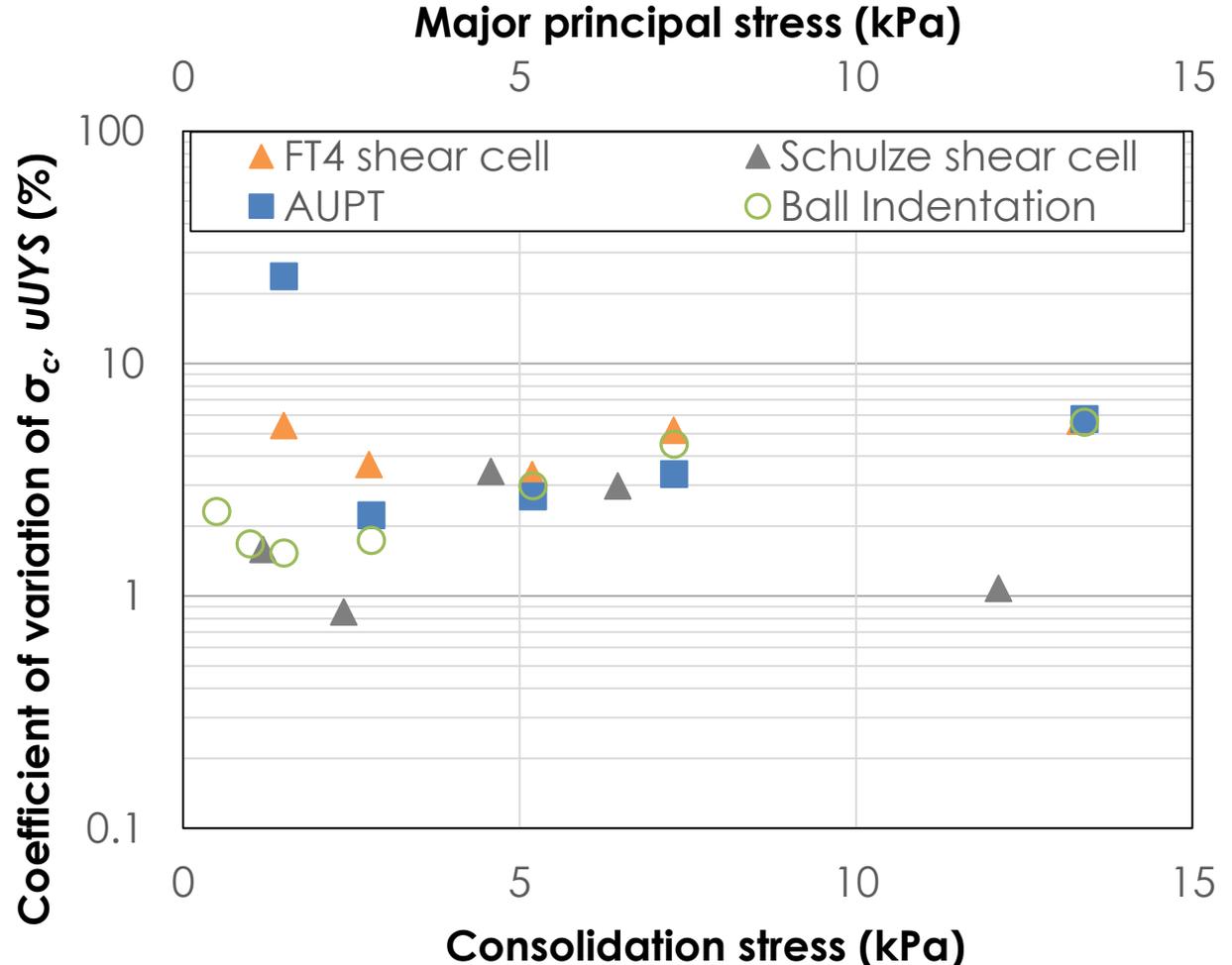
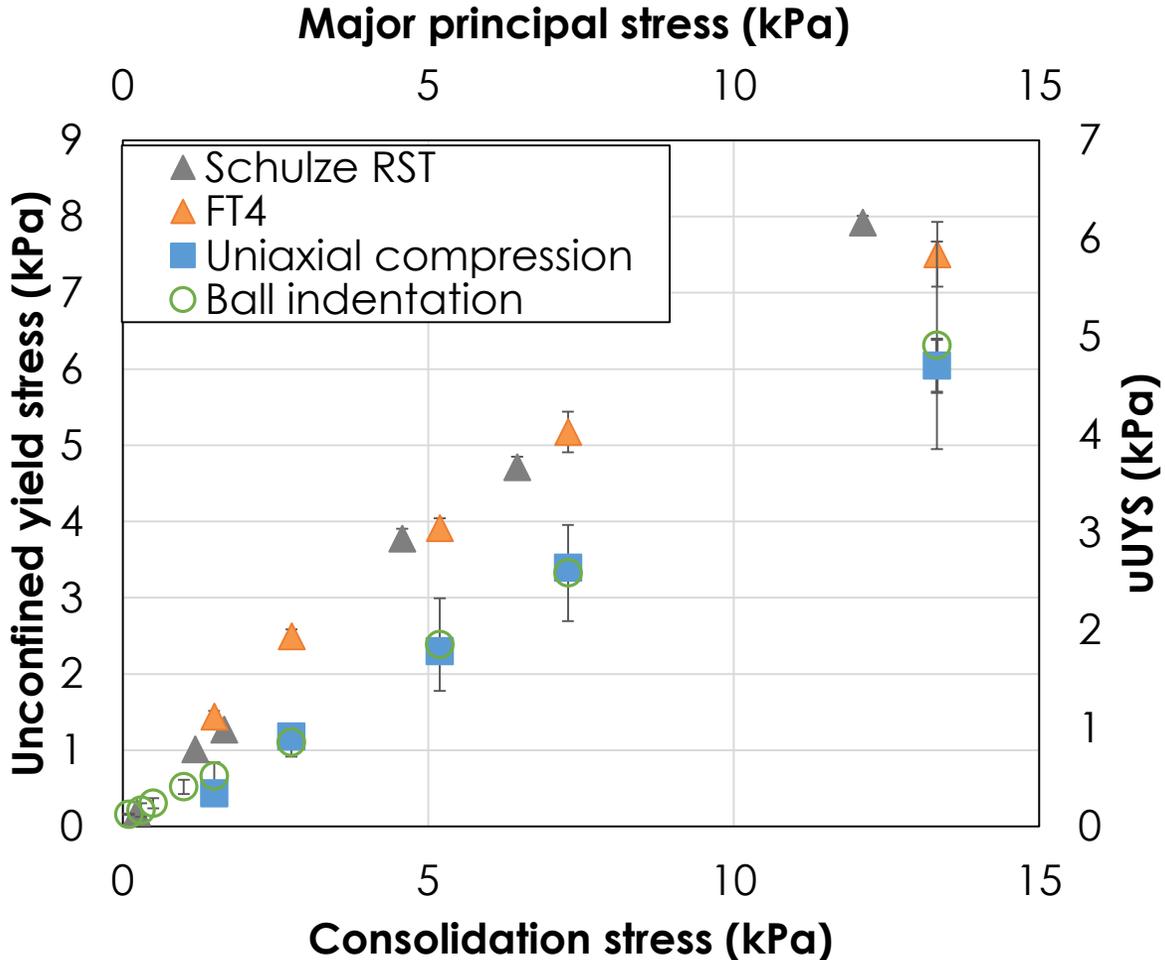
➤ For given dimensionless penetration depth, force increases with indenter size & consolidation stress

☐ At higher consolidation the 4 & 10 mm indenters agree

Indentation onto sieved beds - titania



Comparison of techniques



Measurement accuracy: test flow through a hopper

➤ Powder flow properties required for the design:

- Flow function** (from **shear cell tests**) – or from **ball indentation**
- Effective angle of friction** (from shear cell test)
- Wall friction angle** (wall friction test)
- Bulk density** (compressibility test)

➤ Need to determine:

- Hopper half angle, θ** (from vertical)
- Outlet size, B**



Critical mass flow hopper angle depends on:

- The hopper geometry (conical or planner)
- The powder's **effective angle of friction, δ_e**
- The powder's **wall friction angle, ϕ**

Analytical description of the theoretical boundary between the mass flow and funnel flow regions for conical hoppers^{7,8}:

Where β is the angle formed between the major principal axis and a line normal to the hopper wall

$$\theta = \frac{\pi}{2} - \frac{1}{2} \cos^{-1} \left(\frac{1 - \sin \delta_e}{2 \sin \delta_e} \right) - \beta$$

$$2\beta = \phi + \sin^{-1} \left(\frac{\sin \phi}{\sin \delta_e} \right)$$

⁷Mehos, G., *Hopper Design Principles for Chemical Engineers*.

⁸Enstadt, G., *Chemical Engineering Science*, 30, 1273-1283.

Hopper design:

- Jenike (1961)⁹ defined ratio of σ_1 to the arch support stress ($\bar{\sigma}$) as the flow factor, ff
- The flow factor is a function of θ , ϕ , and δ :

$$ff = \frac{Y(1 + \sin\delta_e)H(\theta)}{2(X - 1)(\sin\theta)}$$

$$X = \frac{2^m \sin\delta}{1 - \sin\delta} \left[\frac{\sin(2\beta + \theta)}{\sin\theta} + 1 \right]$$

$$Y = \frac{[2(1 - \cos(\beta + \theta))]^m \sin\theta (\beta + \theta)^{1-m} + \sin\beta \sin^{1+m}(\beta + \theta)}{(1 - \sin\delta) \sin^{2+m}(\beta + \theta)}$$

where m is equal to 1 for circular outlets and 0 for slotted outlets

Hopper design – Outlet size (B):

$$B = H(\theta) \cdot \frac{\bar{\sigma}_1}{g \cdot \rho_b}$$

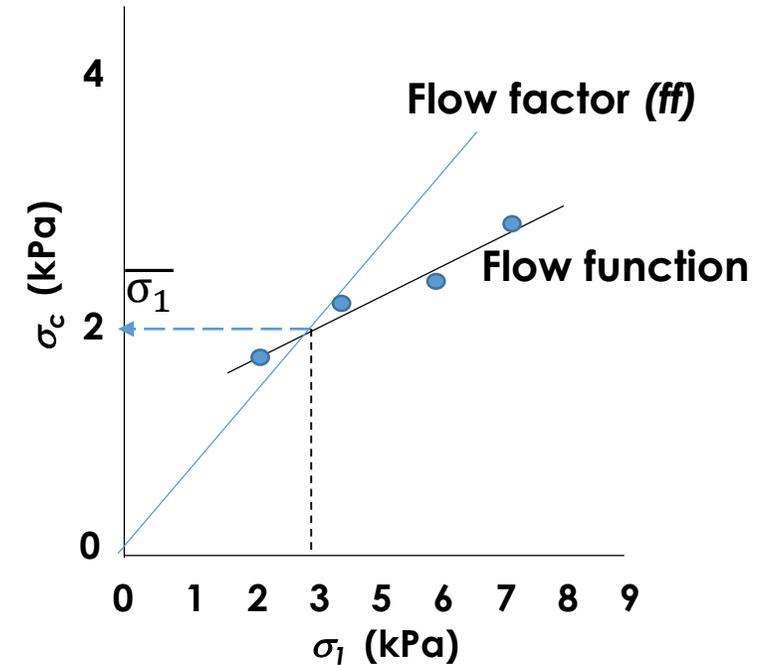
$$H(\theta) = \left(\frac{130^\circ + \theta}{65} \right)^m \left(\frac{200^\circ + \theta}{200^\circ} \right)^{1-m}$$

B : minimum outlet diameter to avoid arching (m)

$H(\theta)$: Factor depends on angles of internal friction and wall friction

$\bar{\sigma}_1$: Critical unconfined yield strength (Pa)

ρ_b : bulk density (kg/m³)



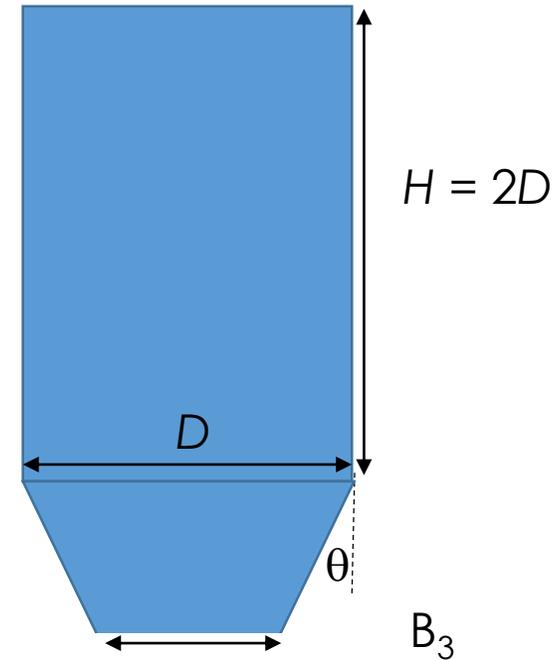
⁹Jenike, A. W., 1961, University of Utah Engineering station, Bulletin 108.

Actual outlet size required for flow

- We aim to use a small volume of powder ~ 5 litres
 - ❑ Require small outlet size
- Require arch support stress, $\bar{\sigma} = 0.1-1$ kPa

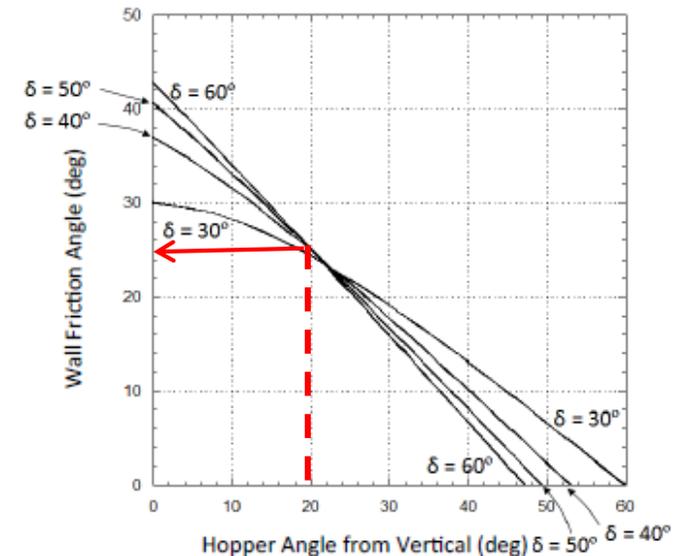
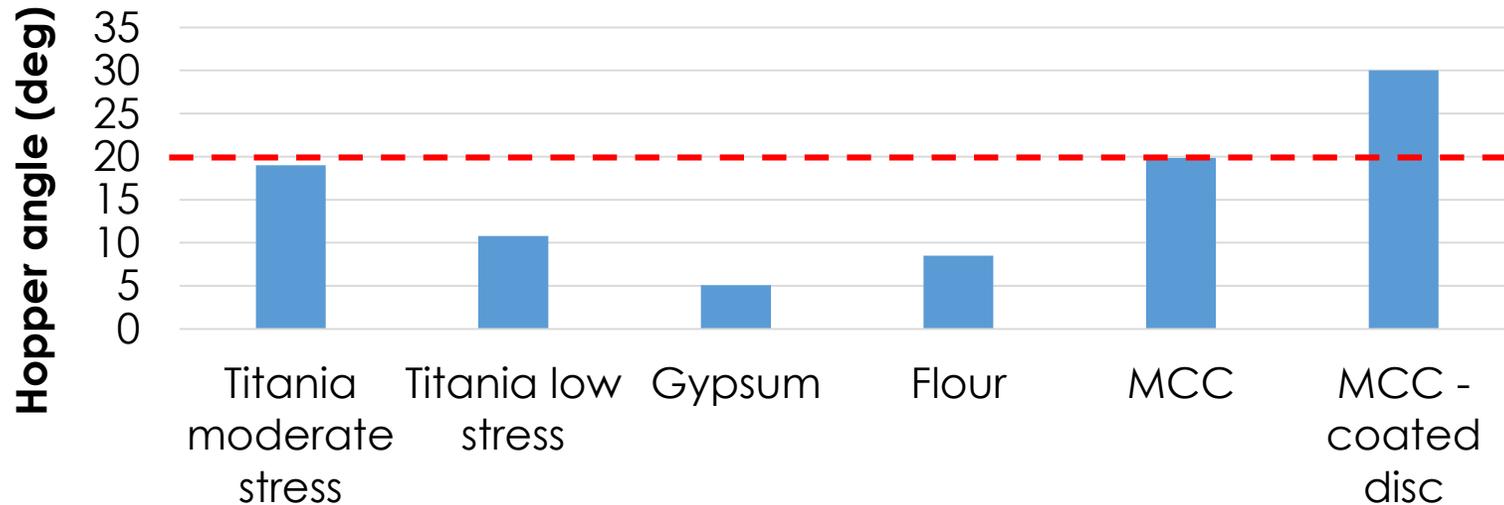
- Therefore need powder to be:
 - ❑ Cohesive
 - ❑ Moderately high bulk density

- Preferably
 - ❑ Hopper angle should be fixed
 - ❑ Outlet size free to vary
 - Without significant change in hopper height
 - Requires hopper angle $> 20^\circ$



Hopper design for tested powders

| | Stress range (σ_{pre} , kPa) | FF_c (-) | Loose Bulk density (g/ml) | δ_e (°) | ϕ (°) | $\bar{\sigma}_1$ (kPa) | ρ (g/ml) | θ (°) before subtracting safety factor | Outlet size, B (cm) |
|--|--------------------------------------|------------|---------------------------|----------------|-------------|------------------------|---------------|---|---------------------|
| Titania moderate σ_{pre} | 2-16 | 1.8 – 5 | 0.34 | 51 | 25.9 | 3.24 | 0.5 | 19 | 145 |
| Titania low σ_{pre} | 0.2 - 1.6 | 1.1 – 1.2 | 0.34 | 66.5 | 33.7 | 0.43 | 0.38 | 10.8 | 25 |
| Gypsum | 0.2-0.8 | 1.2 – 1.7 | 0.74 | 67 | 38.2 | 0.34 | 0.85 | 5.1 | 10 |
| Flour | 0.1-0.8 | 1.1 - 2 | 0.54 | 66 | 35.4 | 0.24 | 0.61 | 9 | 9 |
| MCC | 0.1-1 | 2.5-4.2 | 0.32 | 44.4 | 25.2 | 0.06 | 0.37 | 19.8 | 4 |
| MCC - coated disc | 0.1-1 | - | 0.32 | 44.4 | 17.6 | 0.06 | 0.37 | 29 | 4 |



Conclusions

- For ball indentation, constraint factor correlations found for:
 - ❑ Particle properties: size, size distribution, surface energy, friction
 - ❑ Flow properties: flow function coefficient, angle of internal friction

- Challenges of powder flow measurement at low stress
 - ❑ Reproducibility of bed preparation method
 - ❑ Resolution & accuracy of the force sensor

- Reproducible measurements provided by:
 - ❑ Indentation – sieve-filled bed, vertical consolidation, multiple indents on bed
 - ❑ Schulze RST.XS.s with low stress cell

Future work

- Accuracy of the measurement to be tested by hopper flow behaviour
 - ❑ Based on flow functions from
 - Ball indentation
 - Schulze RST.XS.s

- Packing fraction distribution in ball indentation measured by X-Ray tomography

- Ball indentation, uniaxial compression and shear cell measurements carried out for further materials, to:
 - ❑ Determine constraint factor
 - ❑ Compare the measurements
 - ❑ Establish the operability range