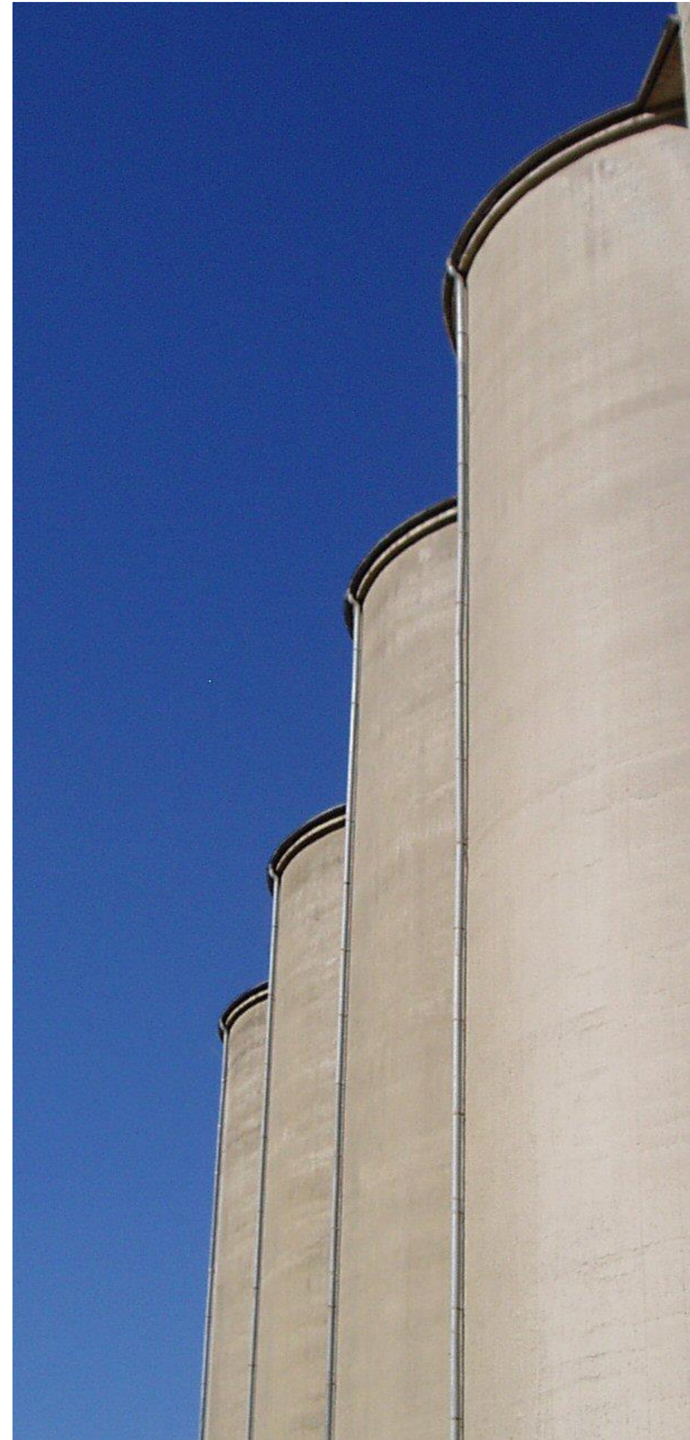
UNIVERSITY of GREENWICH

The Wolfson Centre
for Bulk Solids Handling Technology

IAgrE
Engineering for
Food and Drinks SIG



A novel approach to measure adhesion between particles and to predict the powder flowability

Presented by
Vivek Garg

Principal Investigator
Prof. Michael Bradley

What is Powder Flowability?

- Ability of the powder to flow or stick during the process or during any stage of formulation.
- Classification of powder flowability
  - Free flowing
  - Easy flowing
  - Cohesive
  - Very cohesive
  - Non-flowing

Flow Problems with Powder Blends

Blends for

Direct compression

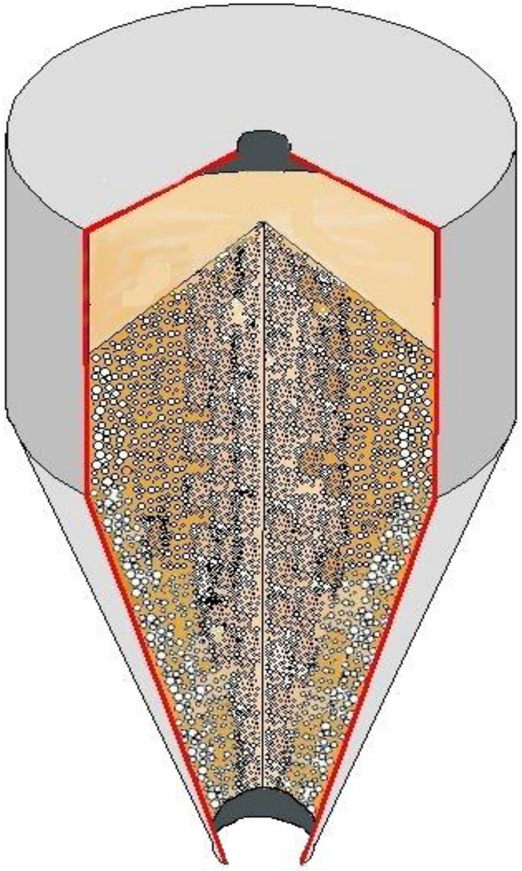
Post-granulation additions blended

Pre-compaction processing of dry-compaction blends

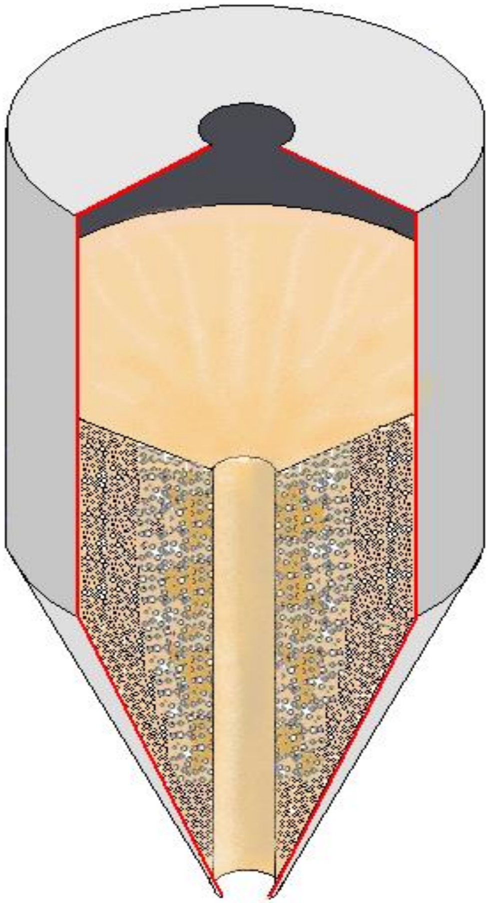
- **If too cohesive:**
- Difficulty in obtaining regular, fast die filling

  Slow production (low press speed)
- Difficulty in roller compaction
- **If too free flowing:**
- Segregation giving rise to
  - Content uniformity problems
  - Variable compaction
- Cost of a "failed batch"

Flow Stoppages "Cohesive Arching"



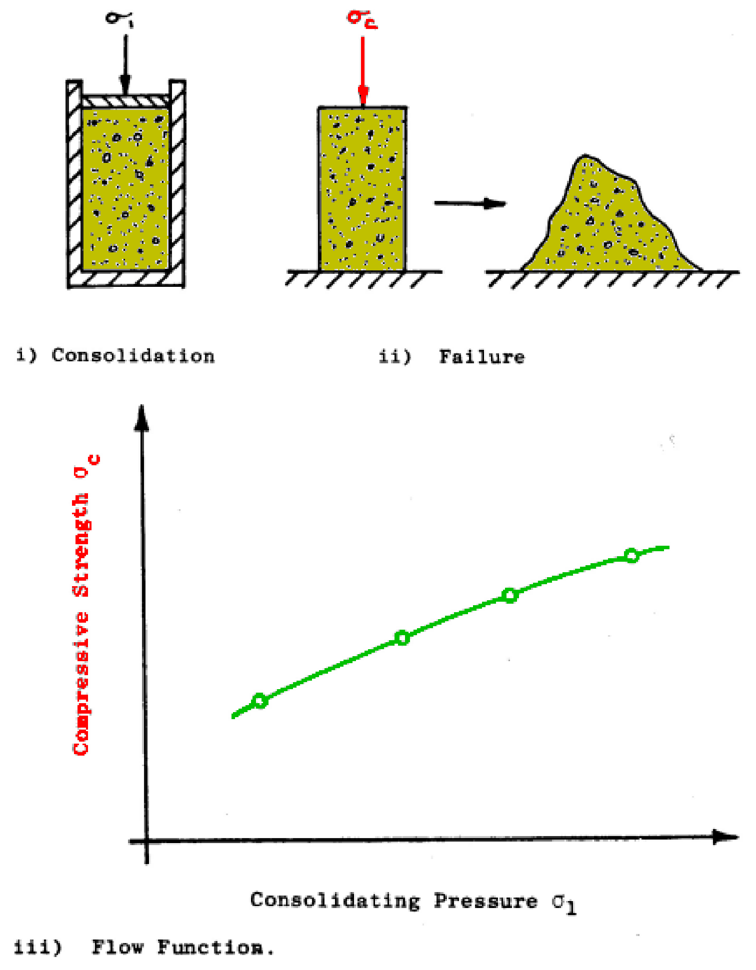
Flow Stoppages “Rat-hole”



Why is Powder Flowability Important?

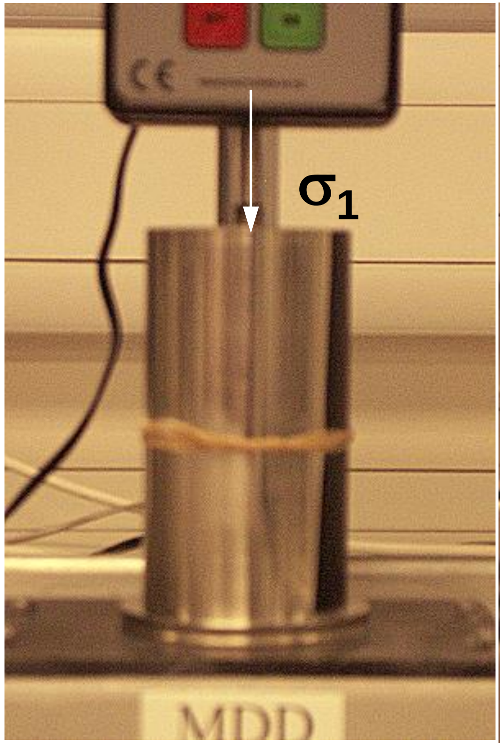
- Arches, rat-holes, hammered hoppers etc
- Flow Function allows prediction of either:
  - What equipment is needed for a given powders
  - Whether a powder will go through existing equipment or not
- Manufacturability of a blend

Characterisation of a Powder's Flowability

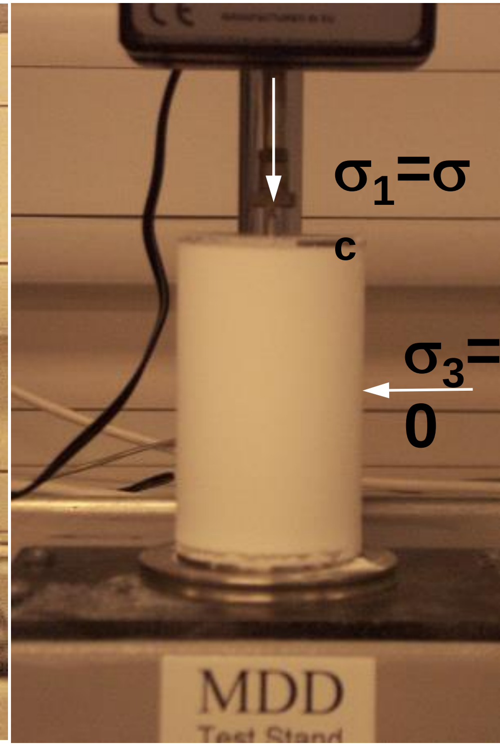


Idealised ***"Flow Function"*** Test

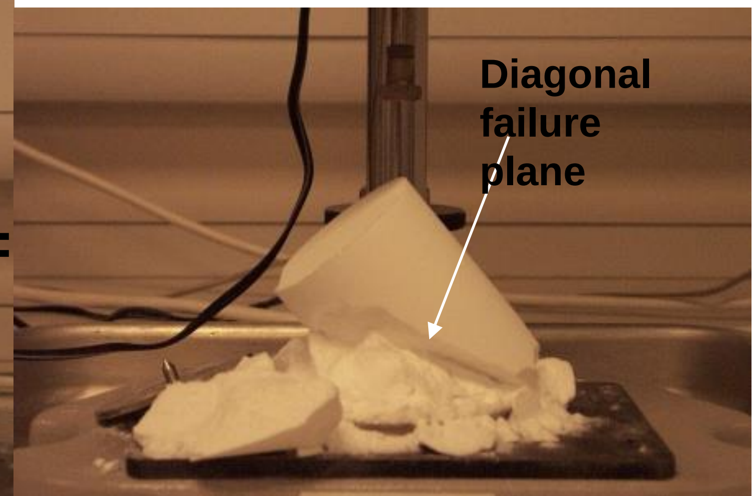
“Sand Castle” Test - Unconfined Failure Test



Consolidation stage



Failure stage



Broken sample

Brookfield Powder Flow Tester

- Software controlled
- 4 basic tests
- 2 – 30 minute test time
- Powder volume required
    - 263cc standard cell
    - 30cc pharma cell

Features of Using a Shear Tester

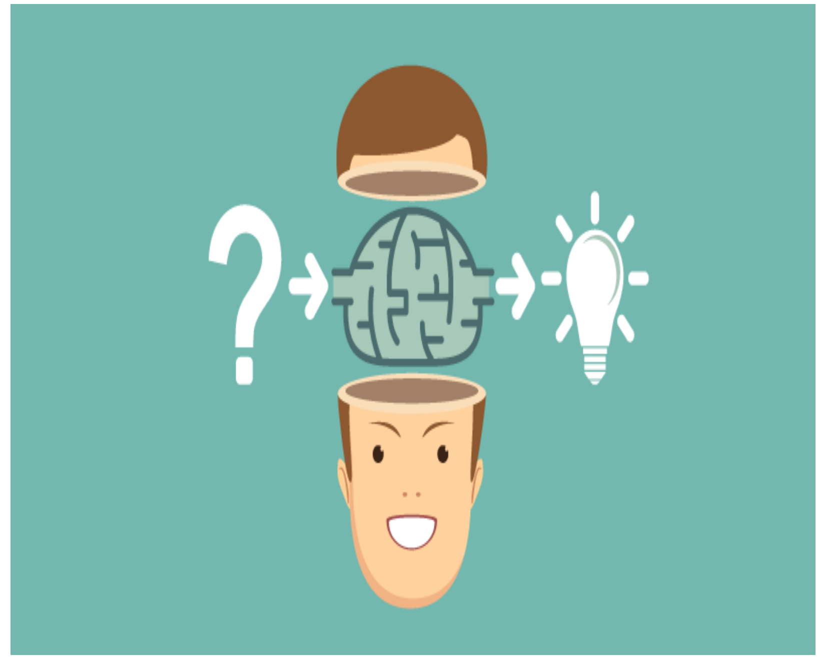
- Measure the flow function accurately and repeatability
- Generates a high level of discrimination between the powders
- Quantity of powder required
- Need to have the final PSD of powder to test – in quantity
- At formulation stage, blend often not in final PSD (Particle Size Distribution)

SSSPin Tester to measure flow function

- smaller sample volume required (a few cc)
- Limitations
  - Need to have the final PSD of powder to test – in quantity
  - High influence of the sample preparation
  - Flowability data highly dependent on value of centrifugal force
  - Inconsistent level of compaction for reproducible consolidation (deviation of about ±12%)

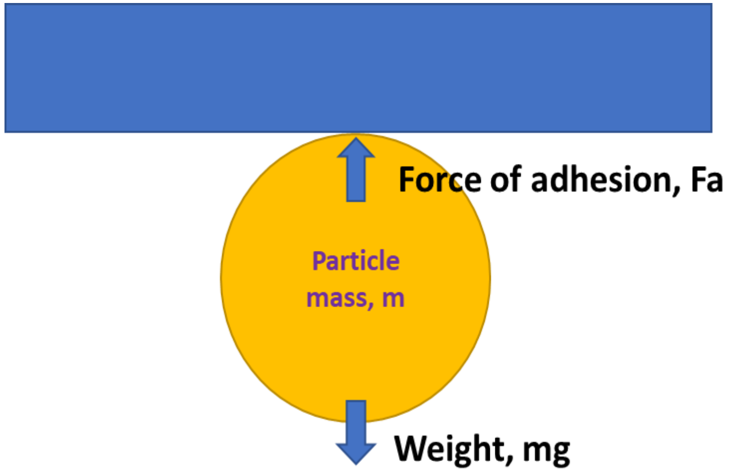
Key novel output of the VFL project

- A new method to determine the flow properties of new material at early stage using a small quantity of particles
  - By measuring the force between a few particles and a surface (Bond Number)
- Infer the “surface energy” of the substance - independent of particle size
- Model to predict flowability of any chosen PSD of bulk material in blend
- Also, a model to predict effect of blending with other components



Concept of “Bond Number”

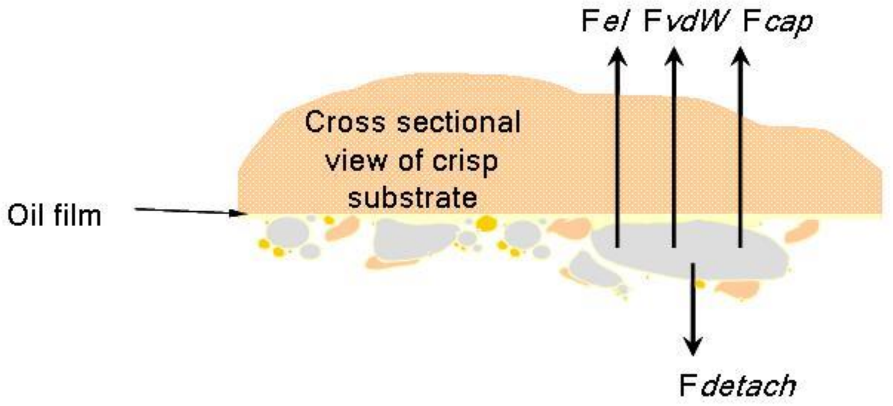
- Bo controls the flowability of a powder



Bond number
Bo = Fa/mg

- Measurement of detachment force – infer surface energy
- From which the flowability of the material in any size distribution can be inferred

Adhesion



$$F_{vdW} = \frac{A_{eff} R_p}{6h^2}$$

A: hamaker constant,
R: radius of the particle,
h: distance of seperation

$$F_{el} = \frac{q^2}{48\pi\varepsilon_0\varepsilon_r\left(R_p + h\right)^2}$$

q : net charge of the particle,
ε_r : permittivity of vacuum,
ε_o : dielectric constant of the intervening medium

$$F_{cap} = 4\pi\sigma R_p \cos\phi$$

σ : surface tension of oil ,
ϕ : contact angle of oil to the crisp surface

the sum is the <u>total adhesion force</u>

Why Measuring Adhesion Strength of Flavour Particles?

- control flavour to minimise waste in manufacture and downtime for cleaning the equipment
- control flavour to minimise salt/fat intake to consumers

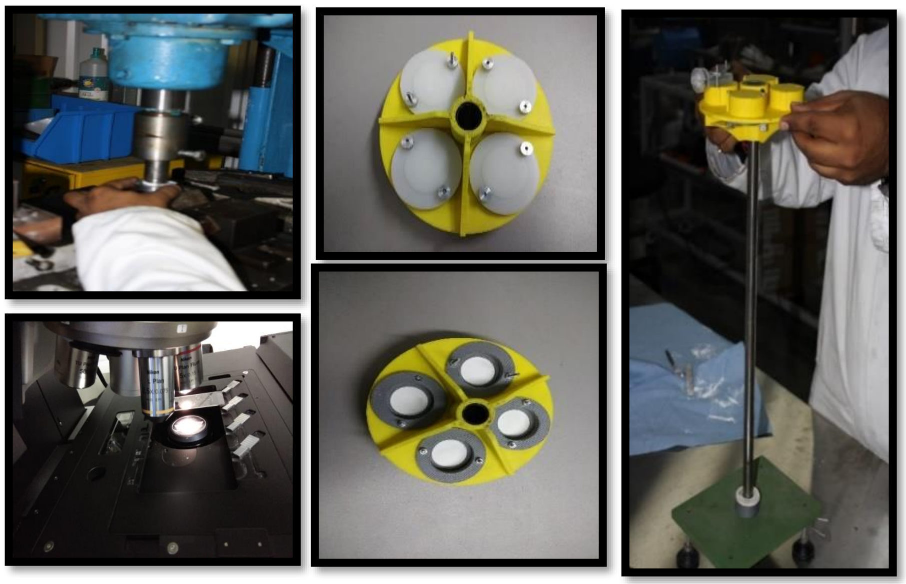
A New Technique to Measure the Particle Forces

Introduction:

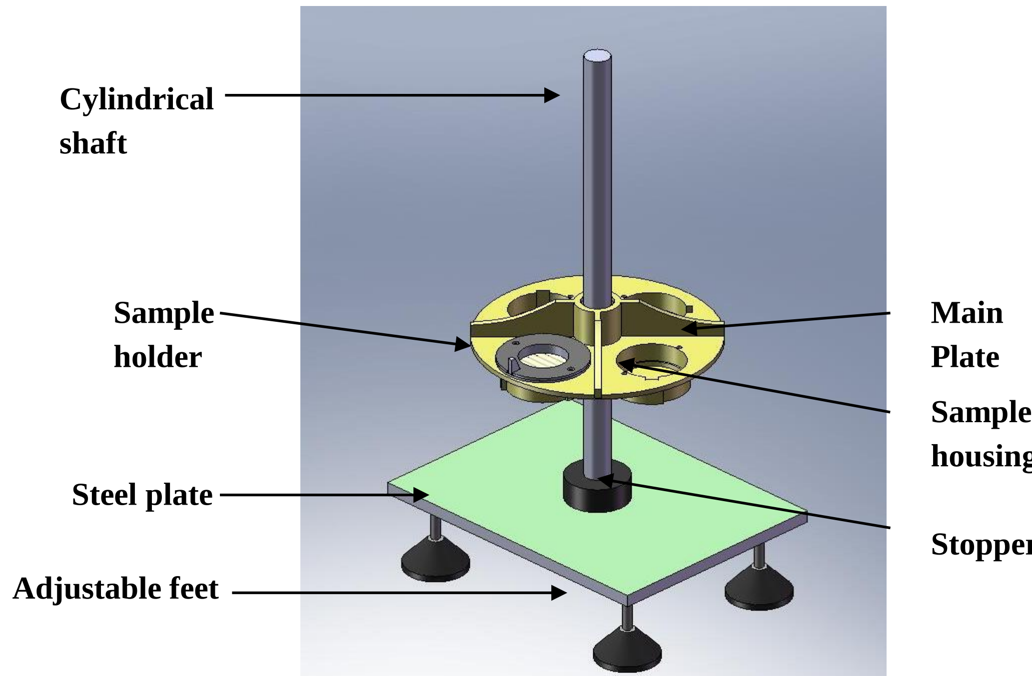
- A new technique (modified version of Ermis' Drop Test) developed to determine the **<u>particle-surface forces</u>** of the cohesive powders using a few particles

Objectives:

- To relate the results with the flowability of the powder as indicated by Flow Function



Modification of sample holder and introduction of detachment lids

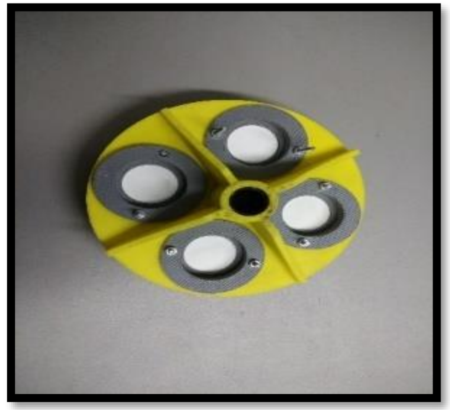


Drop test (Ermis et.al. 2009)

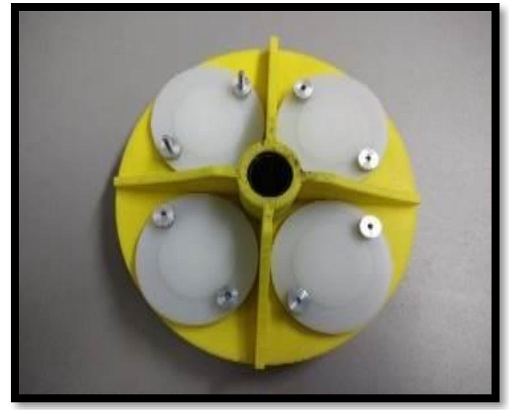
Test Technique

Compaction

Dispersion, weighing

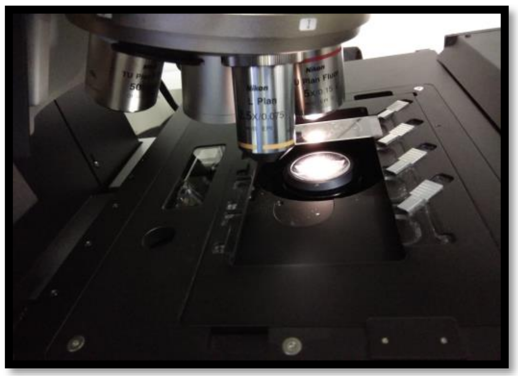


Placing of die into sample holder



Covering the tablets with lids

Drop-off the holder



G3 Analysis

- Measure proportion of powder detached at different decelerations
- Deceleration at which 50% of material is detached, versus mass of d_{50} particle.
- Indicates particle force on d_{50} particle.

Features of New Technique (novel features in green)

DESCRIPTION OF TEST

- Smooth, flat substrate of powder prepared using high stress compaction of powder under polished-face punch
- Disperse particles - either
  - Particles ***(up to 50 milligrams)*** of New Chemical Entity (API) scattered on this using air dispersion unit of Malvern G3
  - Independent stand-alone dispersion unit could be produced if required
- Substrate with particles on is weighed
- Substrate with particle mounted in holder (up to 4 at a time)
- Dropped against buffer to generate controlled deceleration
- Detached particles are caught and weighed
  - With use of Malvern G3 can also be analysed for size distribution
  - Detached particles tend to be larger ones
  - With addition of Raman accessory, chemistry of dispersed/retained materials can be studied
- Optionally repeat for several deceleration levels (height of drop)

Parameters obtained from the MSET

- **Deceleration value at which 50% particles detached.**

Inferred parameters from the MSET

- **Detachment forces**; $\boldsymbol{F_{adhesion}}$

$$\boldsymbol{F_{adhesion} = ma};$$

$$where\ m = Mass\ of\ d_{50}\ particles\ (kg);\ a = measured\ deceleration\ (m/s^2)$$

- **Gravity forces**; $\boldsymbol{F_{gravity}}$

$$\boldsymbol{F_{gravity} = mg};\ where\ m = Mass\ of\ d_{50}\ particles\ (kg)$$

- **Bond Number**; $\boldsymbol{Bo}$

$$\boldsymbol{Bo = F_{adhesion} / F_{gravity}}$$

- **Effective surface energy**; $\boldsymbol{\gamma_{effective}}$

Equations Used

$$F_{adhesion} = ma;$$

$$where\ m = Mass\ of\ d_{50}\ particles\ (kg)$$

$$a = measured\ deceleration\ (m/s^2)$$

$$F_{gravity} = mg;$$

$$where\ m = Mass\ of\ dispersed\ particles\ (kg)$$

$$ff_c = \frac{\sigma_1}{\sigma_c}\ (from\ Powder\ Flow\ Tester);$$

$$where\ \sigma_1 = Major\ principal\ consolidation\ stress$$

$$\sigma_c = unconfined\ failure\ stress\ (kPa)$$

$$Bo = \frac{F_{adhesion}}{F_{gravity}};\ where\ Bo = Bond\ Number$$

$$F_{adhesion} = \frac{0.0002 * \gamma * (r_{50})^{2.6}}{A};$$

$$where\ r_{50} = Particle\ radius\ (m)$$

$$\gamma = surface\ energy\ (J/m^2)$$

$$A = Hamaker\ constant = 0.4 * 10^{-9}\ (m)$$

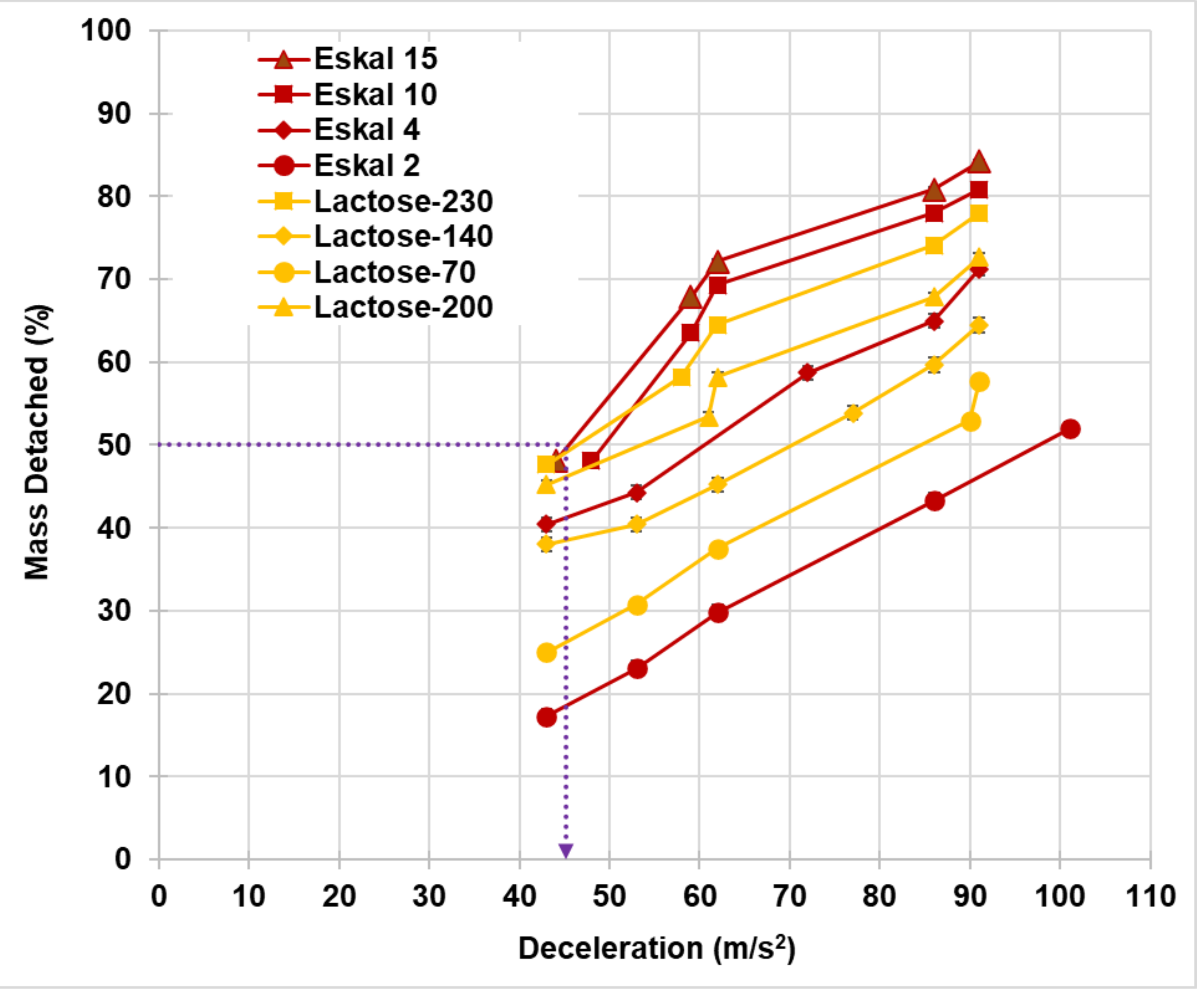
Determination of Surface Energy

Results from the MSET

Results for several different powders:

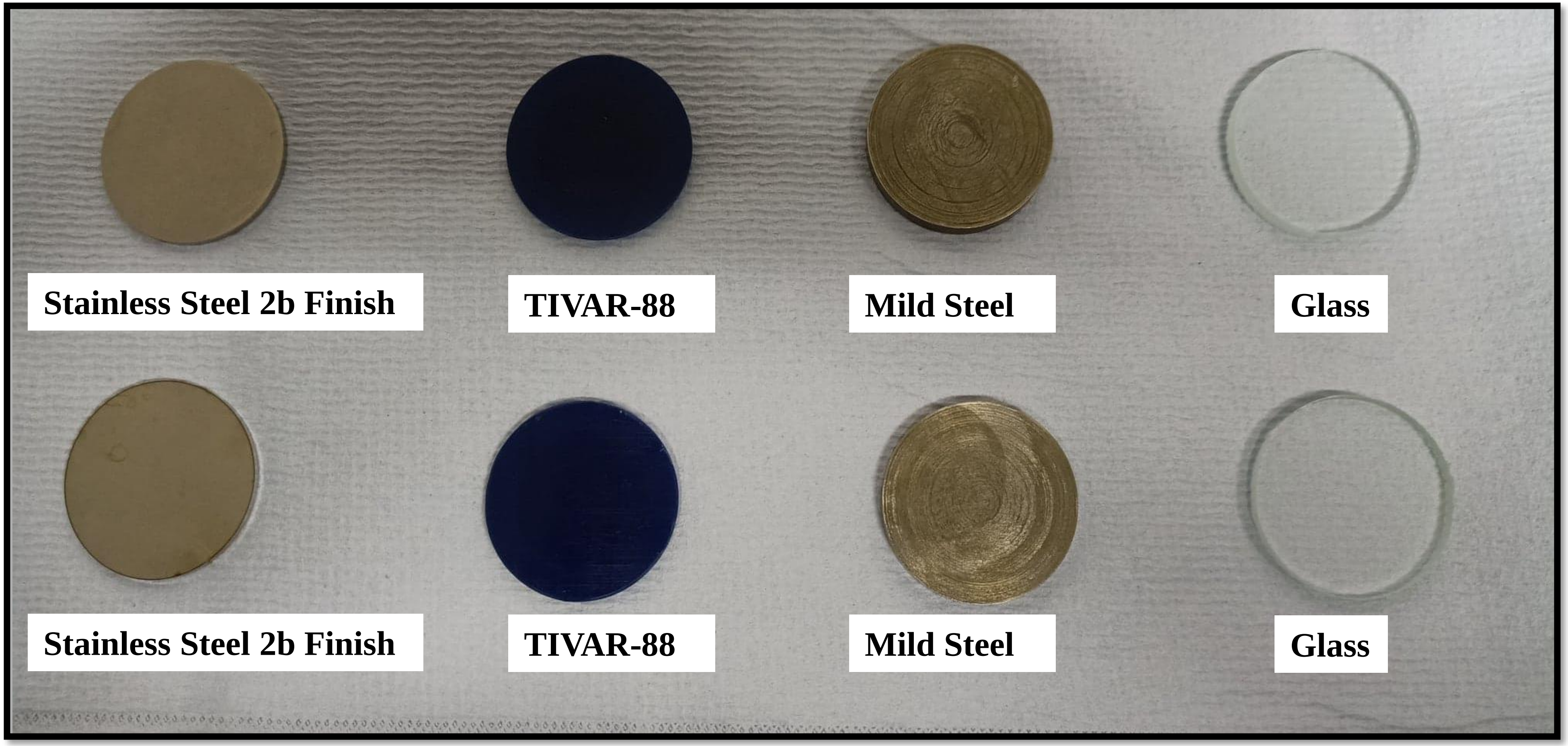
- Some are different size distributions of the same powder
- Some are entirely different powders

For each powder, the proportion of powder detached increases with deceleration level (right)



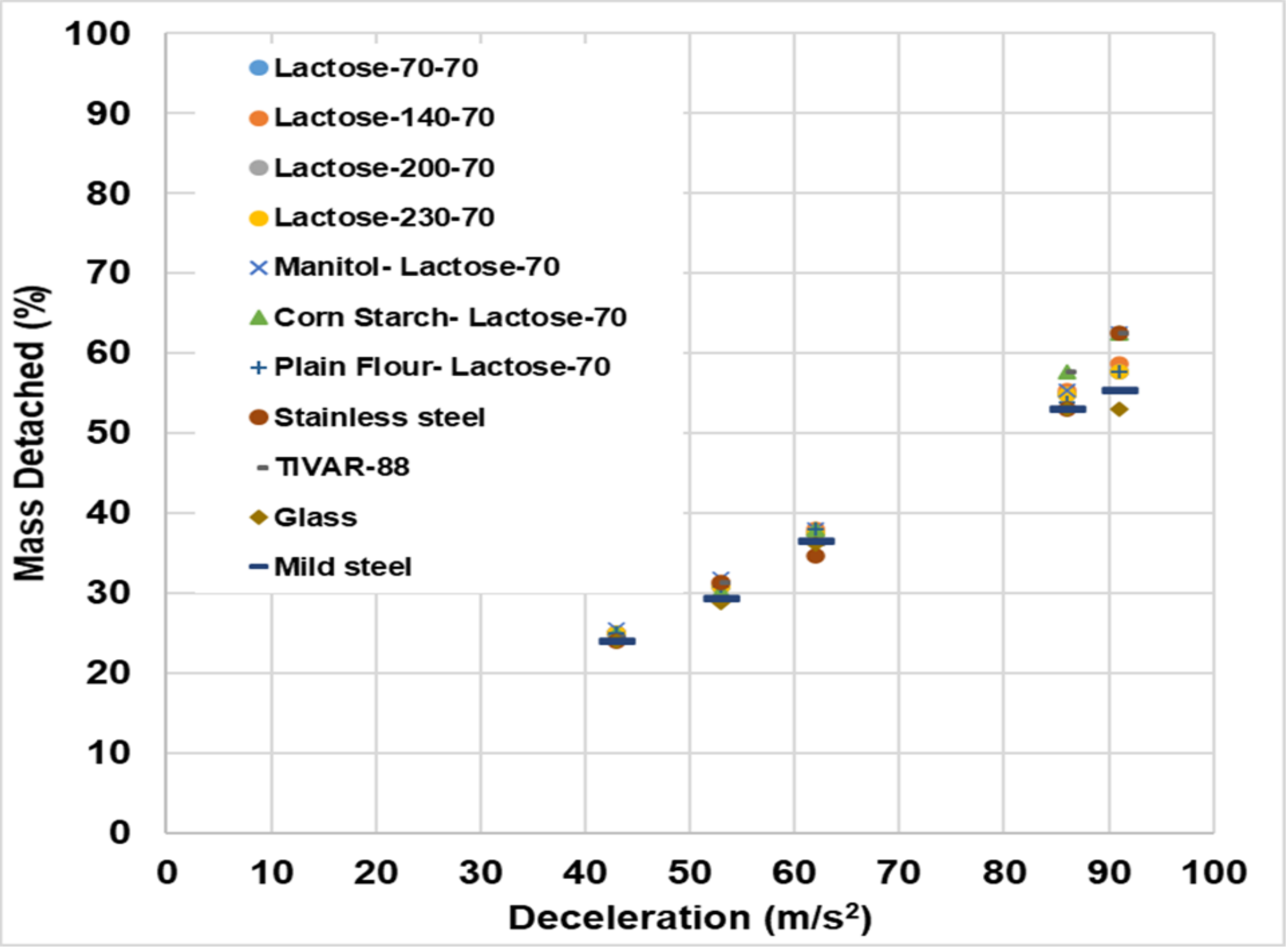
Notes on materials:
- Eskal = calcium carbonate
- Number following name is size in microns

Different Substrates



Influence of Different Substrate

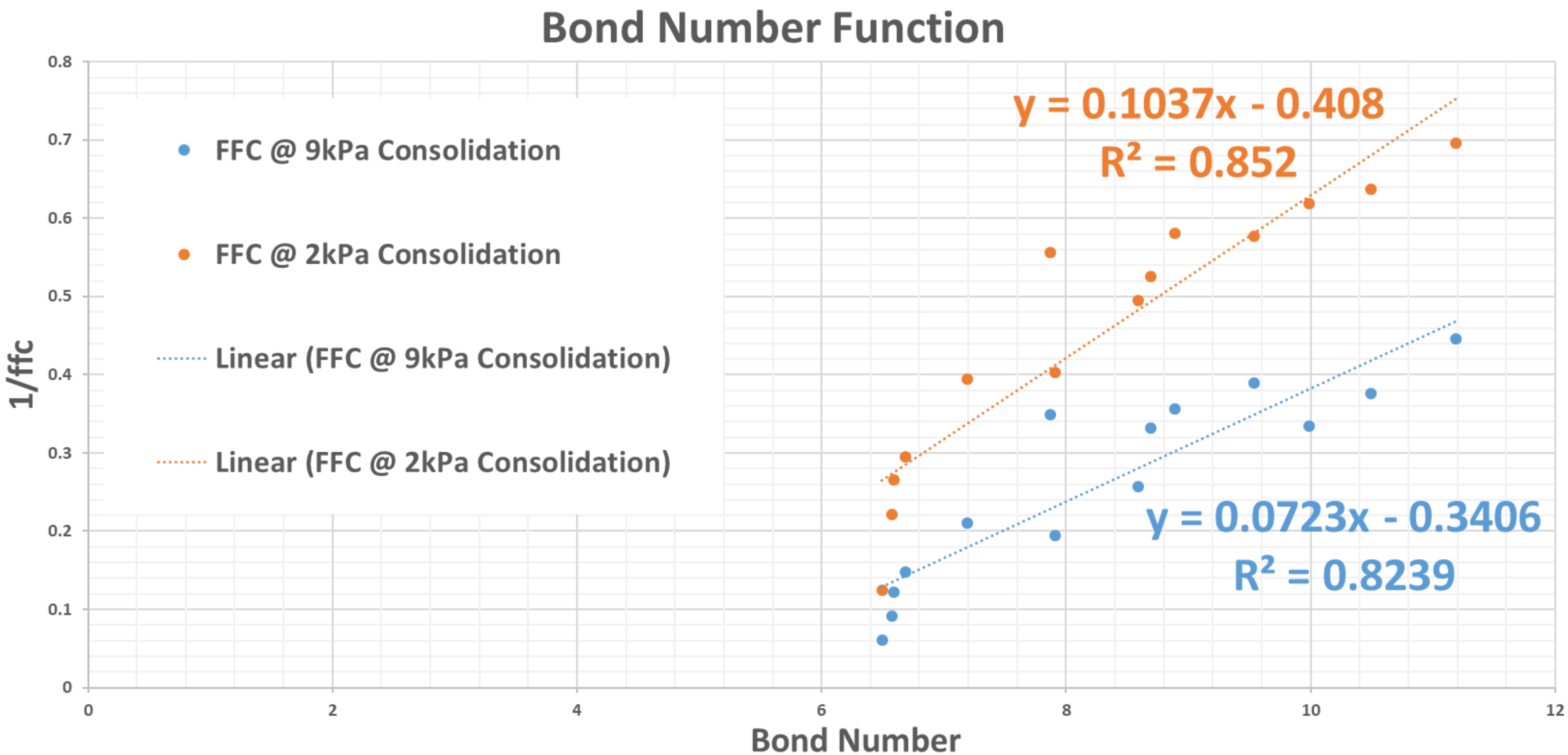
Different Substrate Material and Lactose 70 as dispersed material, in this case



- Deviation of about ± 2.5% from the averaged value of effective substance
- Tested with other different dispersed materials and found that there is a negligible effect of substrate

Relationship between Bond Number and Flow Function

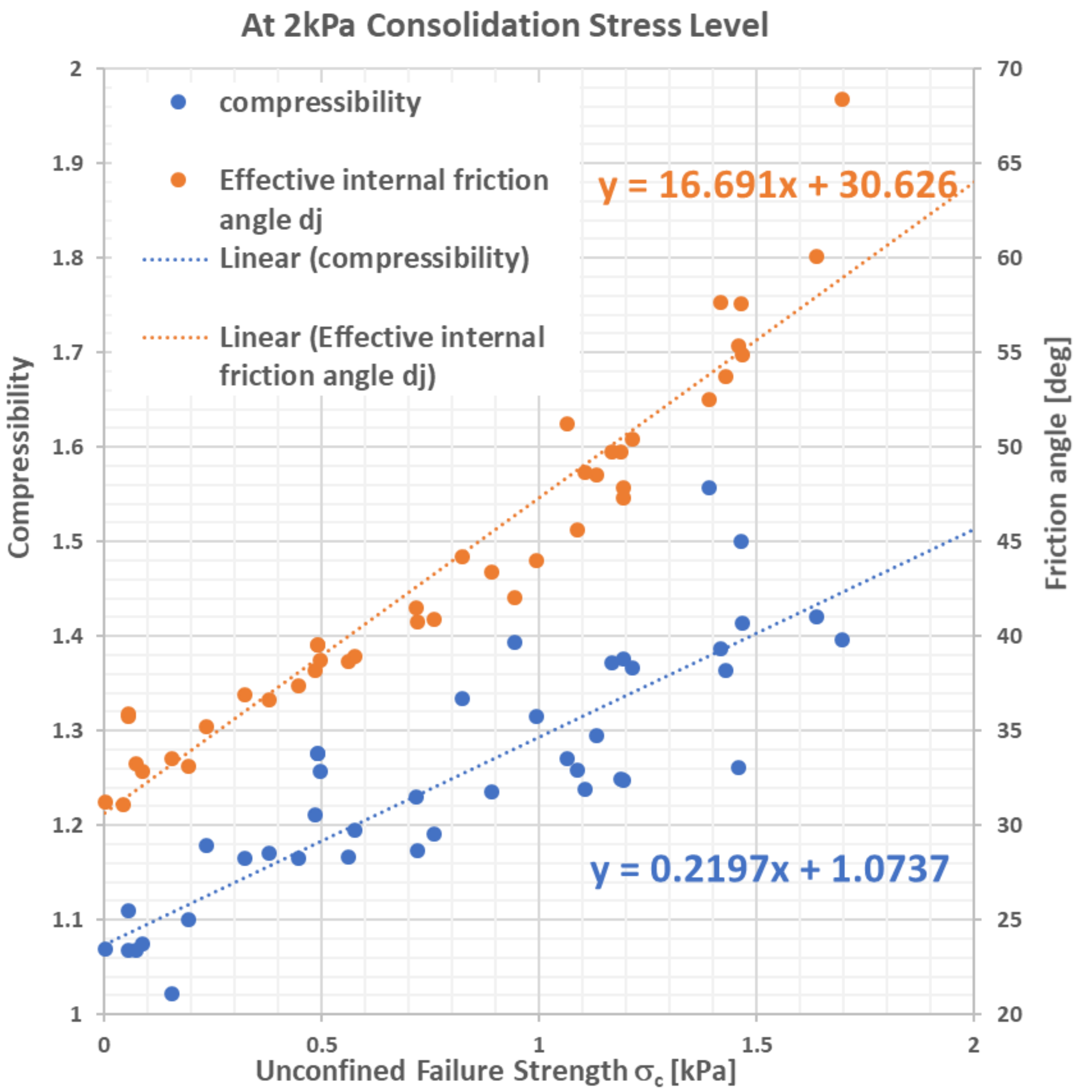
14 Different powders (some same substance with different sizes but also with range of different substances)

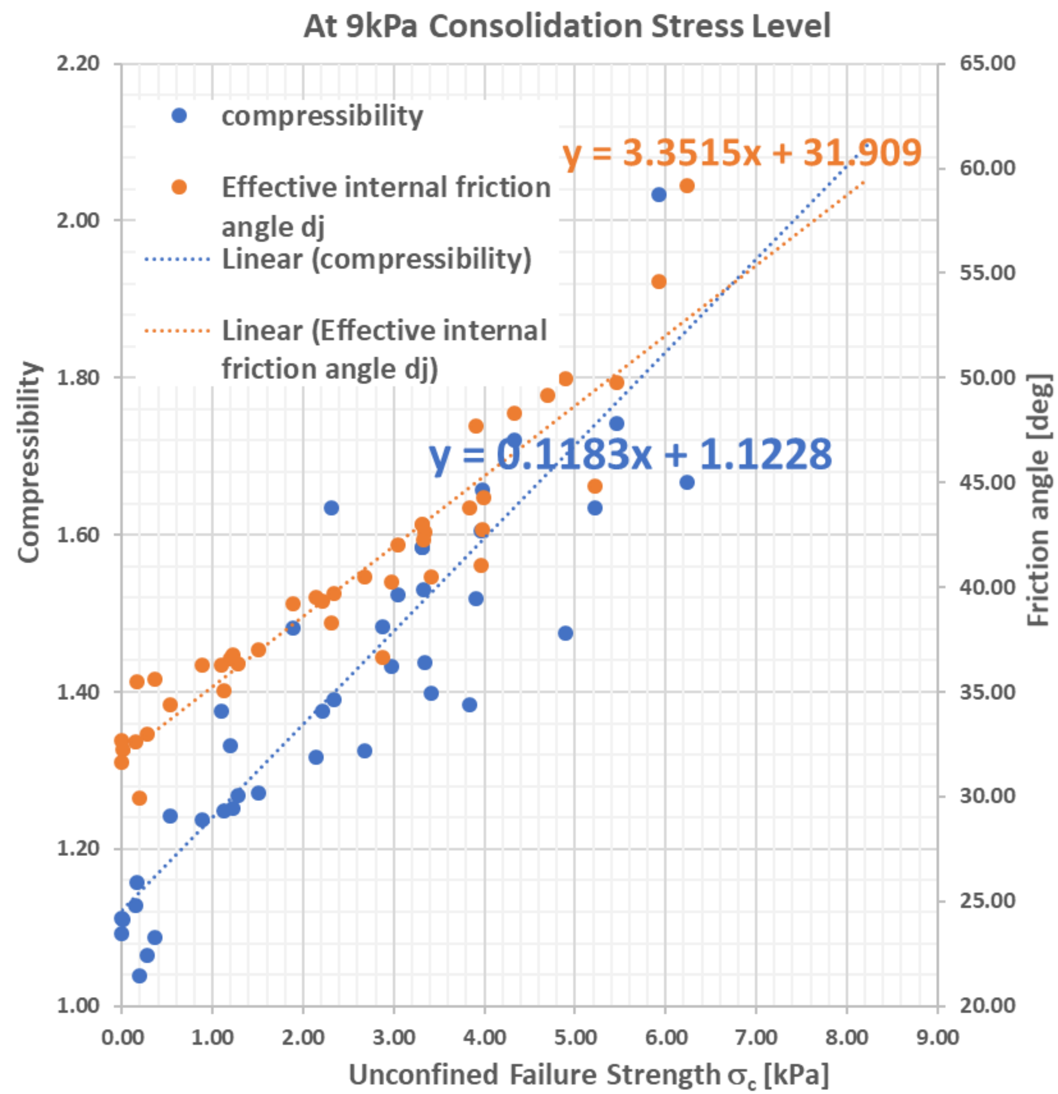


Verified across other 60 different powders

Relationship between Compressibility, Friction Angle, Unconfined Failure Strength

40 different materials





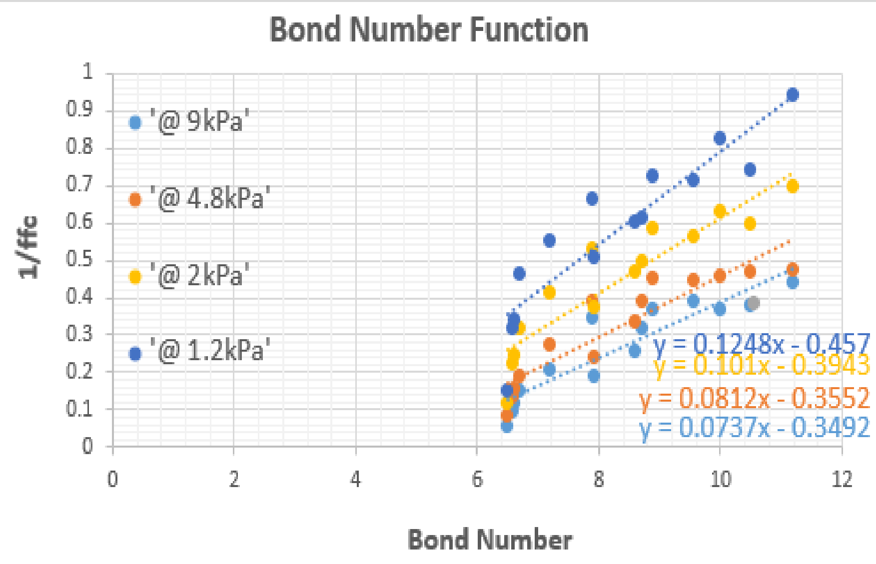
Empirical Model to Predict Flow Function Curve from Measured Bond Number

Inputs

- Measured bond number (from MSET)
  - Practical values between 3 and 14 for d_{50} particles across wide range of real material.
- Required consolidation stress

Outputs

- Unconfined failure strength
- flow function ff_c
- Compressibility
- Effective internal friction
- True friction (gradient of failure locus)



Inputs					
Input measured Bond number:	10.545				
Input Consolidation stress required :	9	4.5	2.25	1.25	kPa
Input material (particle) density:	4475	4475	4475	4475	kg/m^3
m	0.068869	0.086198	0.103527	0.118221	
c	-0.33774	-0.36959	-0.40444	-0.43655	
1/ffc	0.388488	0.539373	0.687254	0.810094	
Bulk flow property outputs					
Unconfined failure strength σc	3.496396	2.427177	1.546321	1.012618	kPa
Compressibility	1.50	1.52	1.39	1.27	
Effective internal friction angle	43.92	47.91	52.58	56.88	deg
Failure locus gradient	31.14	26.96	26.18	26.93	deg
Voidage	0.529355	0.574579	0.618245	0.655735	-
Bulk density	2106.138	1903.759	1708.353	1540.584	kg/m^3

Ways to use the test?

Simplest approach

- Use a standard fixed deceleration level (drop height) for all testing
- Compare the proportion of powder detached
- This is a measure of the adhesion of that particular chemical entity in its tested PSD

More sophisticated approach (recommended) –

- Test across a range of drop heights
- Obtain the deceleration that detaches 50% of the powder
- This gives an objective figure for the mechanical surface energy of the substance (when combined with particle density and size)
- This can be used to predict the behaviour of the same chemical entity in other particle sizes, and of the formulation
- This can be used to predict the flow function of the same material with any chosen particle size distribution

USP (Unique Selling Point) of New Technique

- Can evaluate particle-surface forces for a range of different particle materials (new entities) on any surface
- Suggest use a tablet of the “blank” formulation as the substrate
  - Ongoing research shows this actually makes little difference to the value measured
  - In practice a glass surface can be used
- Can infer the detachment force, bond number and effective surface energy from few particles

Provides the critical information for development of understanding and control of the flow behaviour of the formulation

Formulation is more likely to run first-time on production machinery = saving $$$$$

Industrial and Academic Contribution

- Produced a method where from a few particles at an early stage of formulation, a user can predict whether a bulk of this material will be easy or hard to process (will it cause hang-ups, poor dispersion etc.)
- Developed a simple method to study the detachment of particles from the dispersed surface of same material or different material.
- A new empirical model developed for predicting the flow function from Bond Number/ Adhesion forces.
- Importance of contribution
    - Avoidance of unexpected flow problems with new formulations.
    - Improved time to market.
    - Cost effective.

Summary

- A novel technique developed to study the detachment force with the effect of particle size-with same dispersed material on the substrate as well as with different dispersed material.
- A novel and well-validated method developed to predict the flowability of the powder using parameters inferred from the novel technique.
- An empirical model developed to predict the flowability of the powder using detachment force and effective mechanical surface energy of the material.

Publications

- Deng, T., Garg, V., & Bradley, M. S. (2021). A study of particle adhesion for cohesive powders using a novel mechanical surface energy tester. Powder Technology, 391, 46-56
- Deng, T., Garg, V., Salehi, H., & Bradley, M. S. (2021). Correlations between segregation intensity and material properties such as particle sizes and adhesions and novel methods for assessment. Powder Technology, 387, 215-226.
- Garg, V., Deng, T., & Bradley, M. S. (2021). A New Method for Assessing Powder Flowability Based on Physical Properties and Cohesiveness of Particles Using a Small Quantity of Samples. Powder Technology (*Under Review*)
- Garg, V., Deng, T., & Bradley, M. S. (2021). Influence of surface texture and surface energy on flowability of cohesive powders. International Journal of Pharmaceutics (*Under Review*)

Acknowledgement

- I am and will be ever grateful to **Dr. Robert Berry** for his assistance and support
- I would like to thank my supervisor **Prof. Michael Bradley** who helped me in every stage of developing this technique/toolkit.
- I would also like to thank **Mr. Richard Farnish**, **Dr. Tong Deng**, **Dr. Baldeep Kaur**, **Dr. Hamid Salehi**, **Dr. Stefan Zigan** and **Dr. Pablo Garcia Trinanes** for supporting the work
- I would also like to acknowledge all the academic and industrial partners of the **Virtual Formulation Laboratory** for their support.
- I would also like to thank the funding agency **EPSRC** for supporting VFL project that in turn stimulated and supported this work.

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Thank You

For more information, please feel free to contact:

Vivek Garg

The Wolfson Centre for Bulk Solids Handling Technology

Email id: **V.Garg@gre.ac.uk**