Virtual Formulation Laboratory

for prediction and optimisation of manufacturability of advanced solids based formulations

Powder Flow 2018: Cohesive Powder Flow

organised by

Formulation Science and Technology group (FSTG) of the Royal Society of Chemistry

12 April 2018 Burlington House, London

Academic Collaborators

- Csaba Sinka, Ruslan Davidchack, Ben Edmans, Nicodemo Di Pasquale

  University of Leicester

- Mojtaba Ghadiri, Xiaodong Jia, Mehrdad Pasha

  University of Leeds

- Mike Bradley, Rob Berry, Pablo Garcia Trinanes, Baldeep Kaur

  University of Greenwich

- Jerry Heng, Vikram Karde

  Imperial College

Industrial Partners

- Centre for Process Innovation (CPI)
- Procter & Gamble
- GlaxoSmithKline
- AstraZeneca
- Nestle
- KP Snacks
- Chemours
- Malvern Instrument
- Brookfield
- Britest
- Process Systems Enterprise (PSE)
- Griffiths Food
- Freeman Technology
- DEM Solutions

VFL: 4 Processes/ 4 Problems

Molecule level information

Particle level information

Bulk level information

Prediction of flow/ arching, flooding

Prediction of mixing/ segregation

Prediction of storage/ caking

Prediction of compact/ breakage

Hierarchical input structure

Manufacturability indicators (MI)

Surface Free Energy Predictions

Dr Nicodemo Di Pasquale and Prof. Ruslan Davidchack

- Prediction of Adhesive Interactions by Molecular dynamics (MD), using Cleaving Method

- Comparison of results from MD simulation with FD-IGC experimental work at ICL

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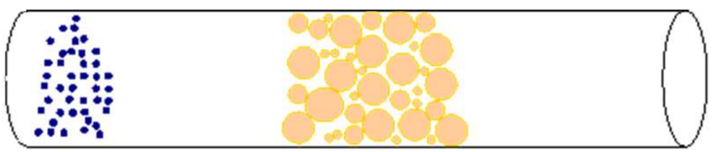
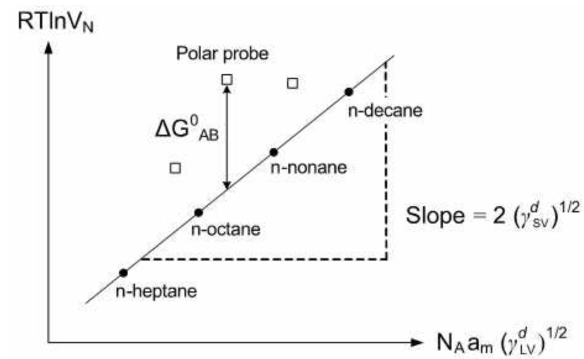
Surface Energy Characterisation using Inverse Gas Chromatography (FD-IGC)

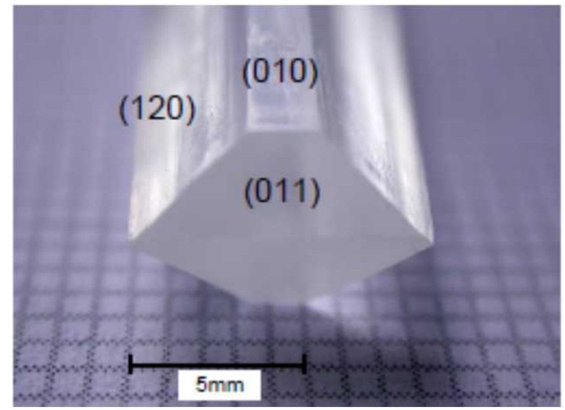
Dr Vikram Karde and Dr Jerry Heng

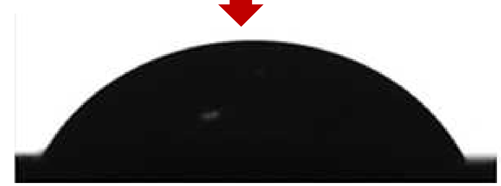
Surface energy determination using IGC





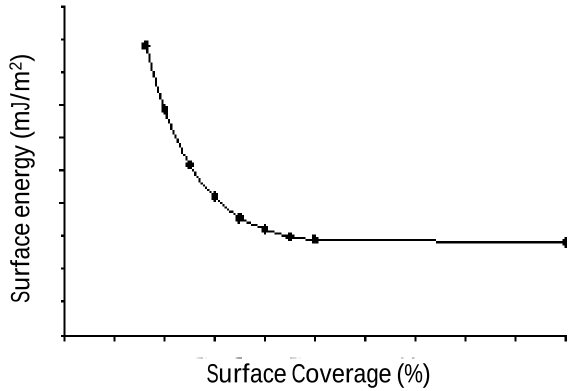
Anisotropy in crystalline solids (Heterogeneous surfaces)





Facet specific surface energy using Contact angle

Surface energy heterogeneity using Finite Dilution IGC (FD-IGC)



Surface energy heterogeneity profile

Flowability, Mixing, Segregation

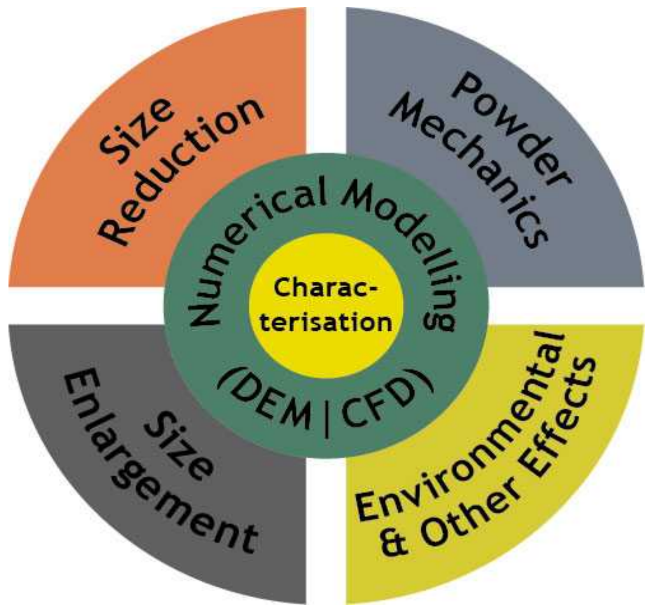
Dr Mehrdad Pasha, Dr Xiaodong Jia and Prof. Mojtaba Ghadiri

Single particle characterisation

Particle assembly behaviour prediction by DEM

Experimental validation

VFL Toolkit development in a collaborative way



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Modelling Powder Compaction

Dr Ben Edmans and Dr Csaba Sinka

Constitutive Model
State variables

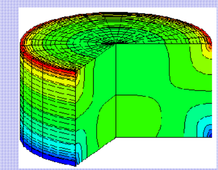
Friction
Dependencies

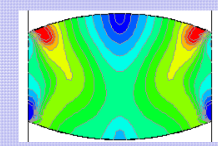
Geometry
Tablet image

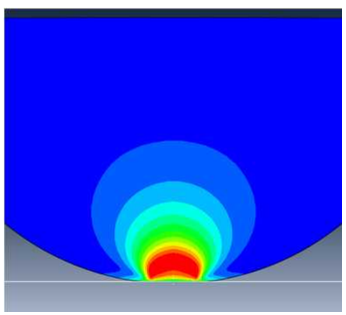
Loading schedule
Sequence of punch motion

Initial conditions
Die fill

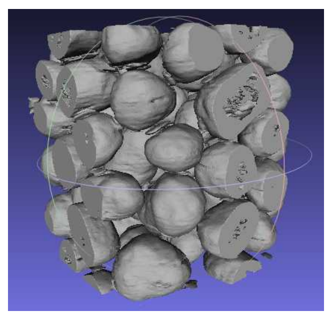
Tablet compaction modeling
FEA - ABAQUS
Solving equations:
- Equilibrium
- Compatibility
- Constitutive



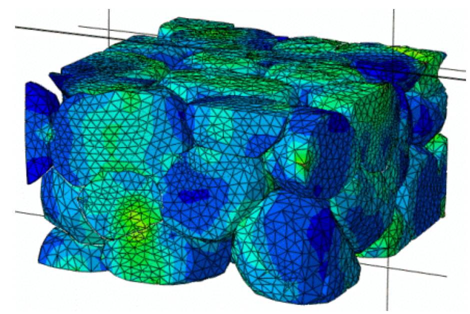




Contact stress between particles



X-ray CT particle assembly



Numerical constitutive law

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Particle and Bulk Scale Measurements

Dr Pablo Garcia Trinanes, Dr Rob Berry and Prof. Michael Bradley

- Particle size and shape measurement
  - G3 morphologi – shape/ size
  - Air-swept sieve – size
  - Pycnometer – material density
- Bulk flow properties
  - Brookfield (PFT) - freeman for high stress tests? – flow function, friction, bulk density (voidage)
  - Uniaxial compaction test – for high stress tests
- Segregation properties
  - Free surface (rolling segregation) for coarse particles > approx. 100 mm
  - Air induced (elutriation) for separation of fines (sub 50 mm) from wider distribution
- Caking properties
  - Capability for measuring cake strengths driven by:
    - moisture migration, chemical reaction or plastic flow mechanisms in storage

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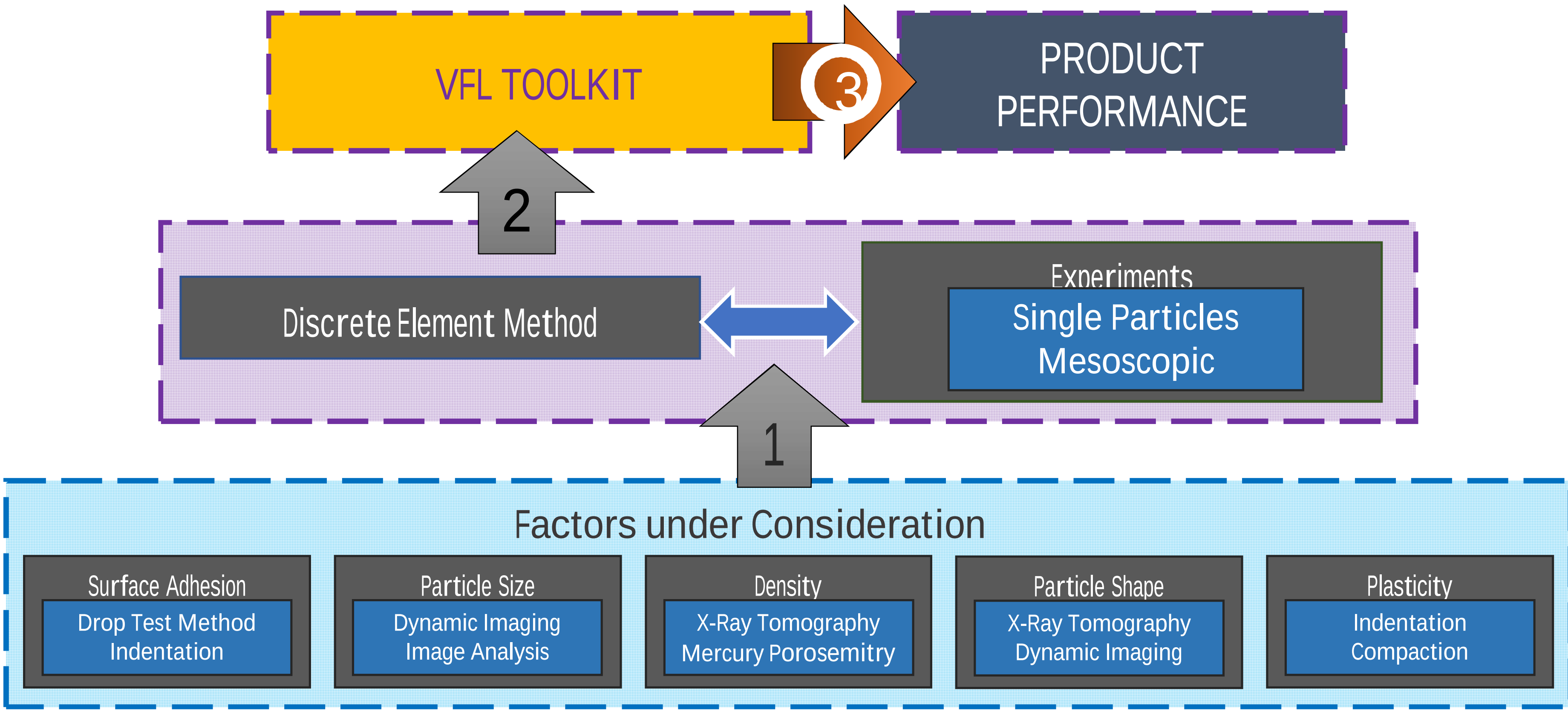
Work Plan of Leeds

Flowability | Segregation | Mixing



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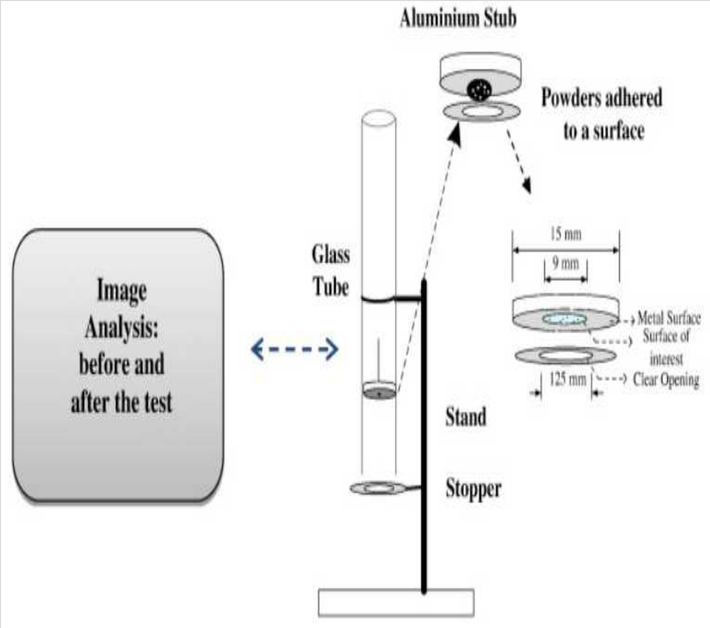
Material Characterisation

Surface Adhesion

Method

- The powder will be dispersed into a flat target (material of interest) using Malvern G3 Morphologi disperser.
- The target will then be dropped from a range of heights until a satisfactory detachment of particles is observed by image analysis.
- Two images, before and after the drop, are taken by SEM to assess the detached and attached particles on the surface of the target

Schematic



Calculations

$$\frac{F_d}{F_{ad}} = \frac{mv/\Delta t}{\frac{3}{2}\pi R \Gamma} = L$$

$$R = \frac{\text{smallest detached particle} + \text{largest attached particle}}{2}$$

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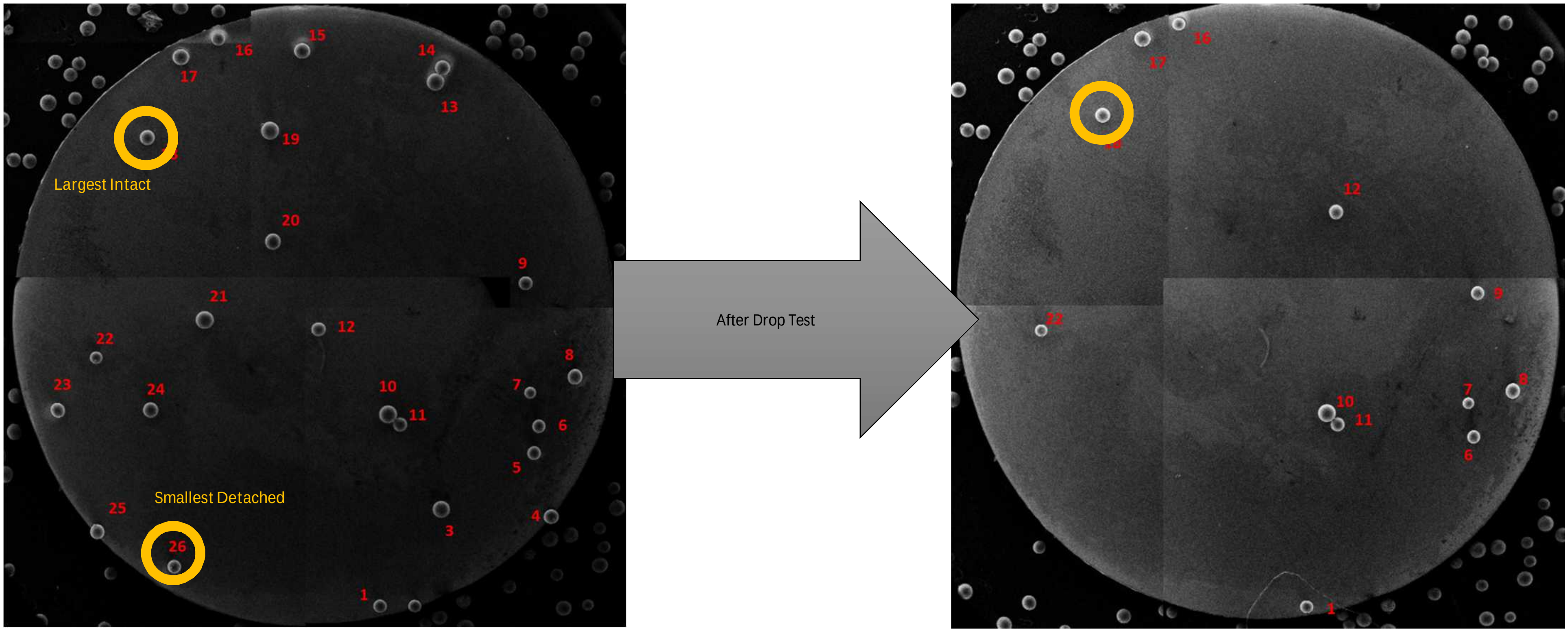
Material Characterisation

Surface Adhesion



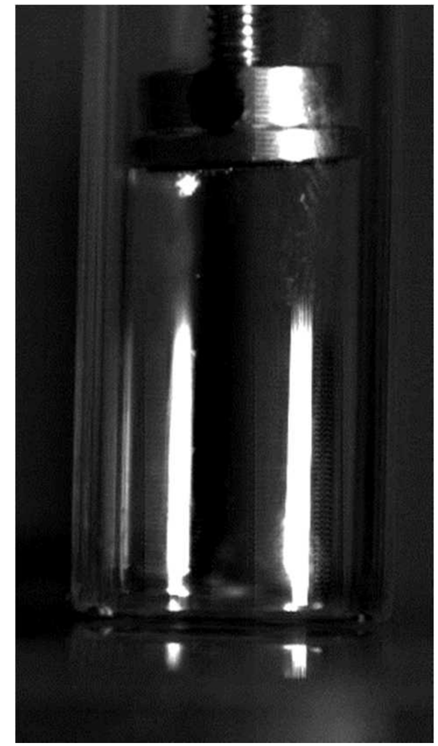
Measurement of Surface Energy

Leeds Drop Test Method: Results

Materials: Glass Ballotini (90 – 200 µm), Glass Plate (5 mm in diameter), Steel Plate (5 mm in diameter)

Interactions:

1) Silanised Glass Ballotini vs Silanised Glass Ballotini/Plate (SGB-SGB)
2) Silanised Glass Ballotini vs Non-Silanised Glass Ballotini/Plate (SGB-NSGB)
3) Silanised Glass Ballotini vs. Steel Plate (SGB-SP)



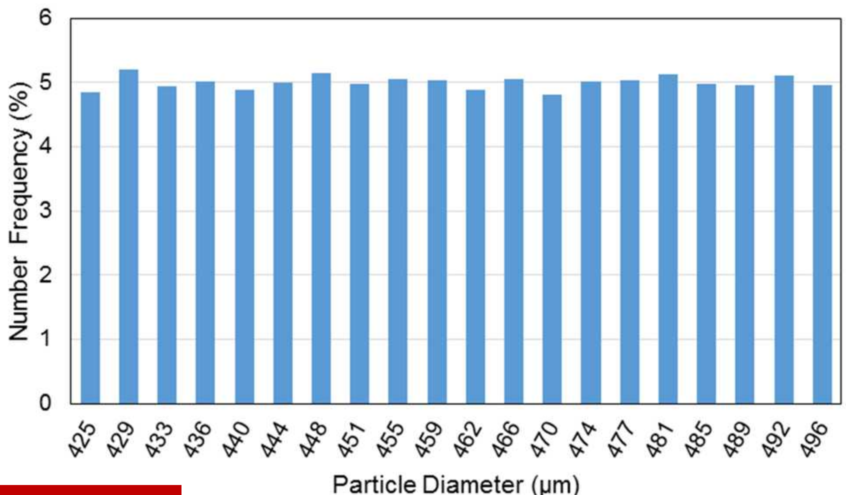
<u>Drop Test Results</u>

SGB – SGB	SGB – NSGB	SGB – SP
$G_{SGB-SGB} = 27.4 \left[mJ/m^2 \right]$	$G_{SGB-NSGB} = 20.6 \left[mJ/m^2 \right]$	$G_{SGB-SP} = 24.4 \left[mJ/m^2 \right]$

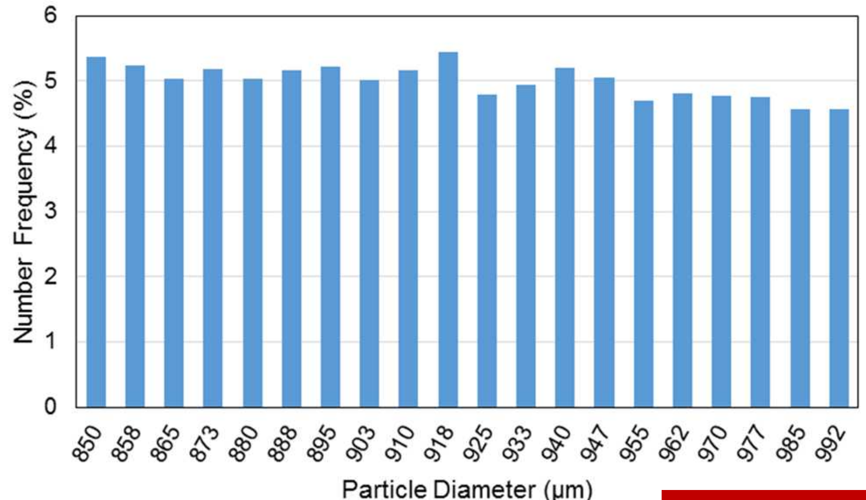
Flowability by FT4

Effect of Particle Size: Material

- Two size classes of glass ballotini were chosen:
  - 425 – 500 µm (on the left)
  - 850 – 1000 µm (on the right)



425 – 500 µm



850 – 1000 µm

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Flowability

Effect of Particle Size: Material

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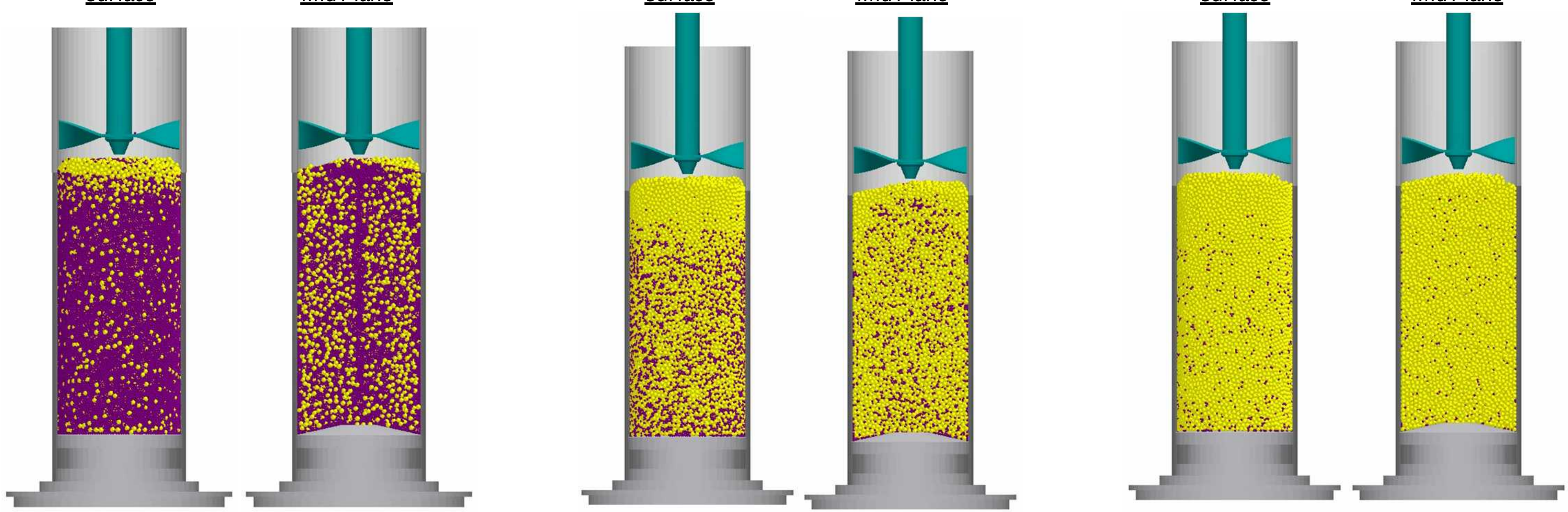
Three mixtures are considered as follow based on number ratio

- 10% (425 – 500 µm) & 90% (850 – 1000 µm) referred to 10S_90L
- 50% (425 – 500 µm) & 50% (850 – 1000 µm) referred to 50S_50L
- 90% (425 – 500 µm) & 10% (850 – 1000 µm) referred to 90S_10L

90S_10L — *Surface* / *Mid Plane*

50S_50L — *Surface* / *Mid Plane*

10S_90L — *Surface* / *Mid Plane*



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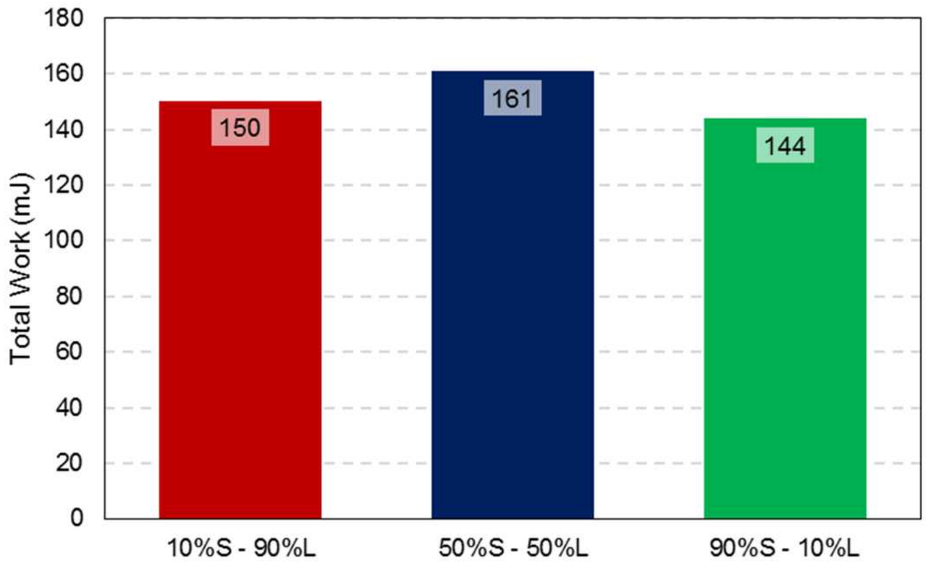
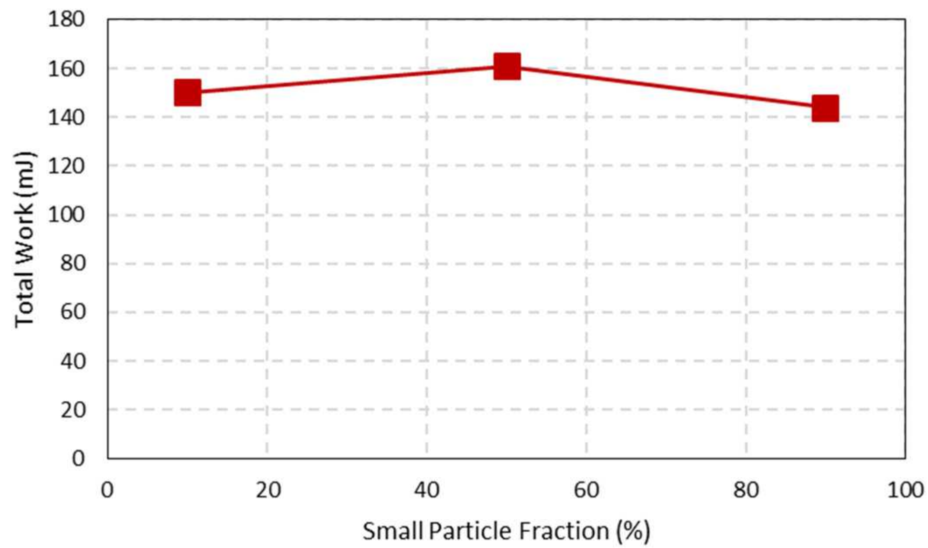
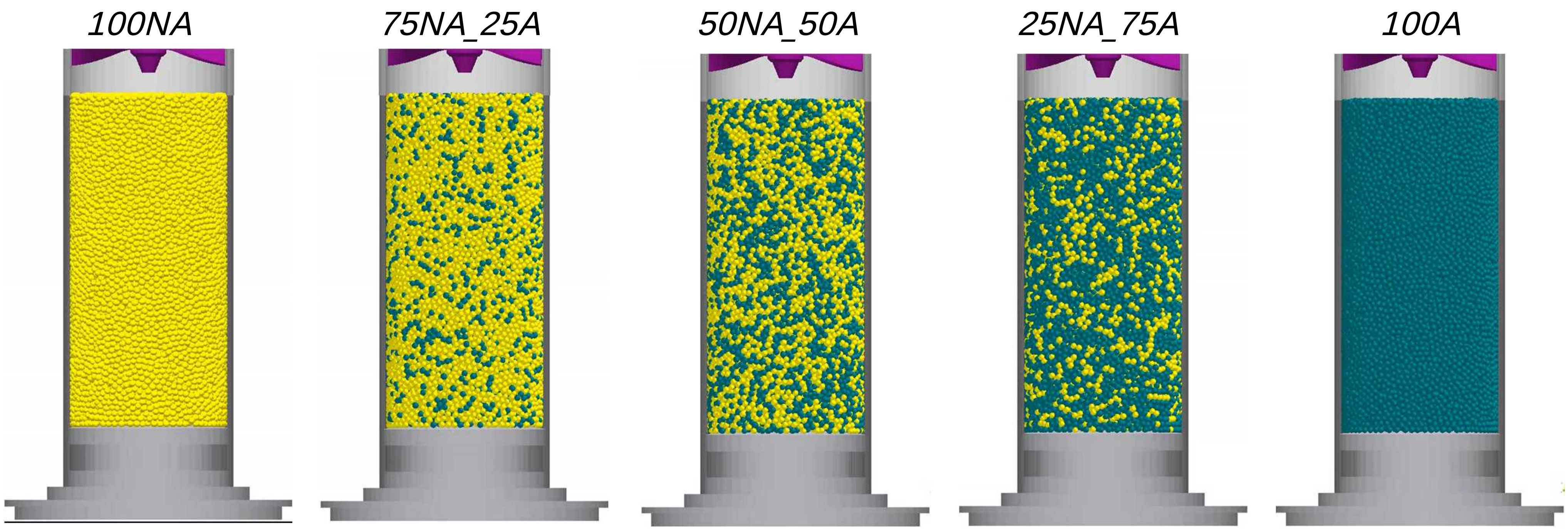

180
160
140
120
100
80
60
40
20
0
Total Work (mJ)
150
161
144
10%S - 90%L
50%S - 50%L
90%S - 10%L


180
160
140
120
100
80
60
40
20
0
Total Work (mJ)
0
20
40
60
80
100
Small Particle Fraction (%)

Flowability

Effect of Particle Adhesion: Downward Test Results



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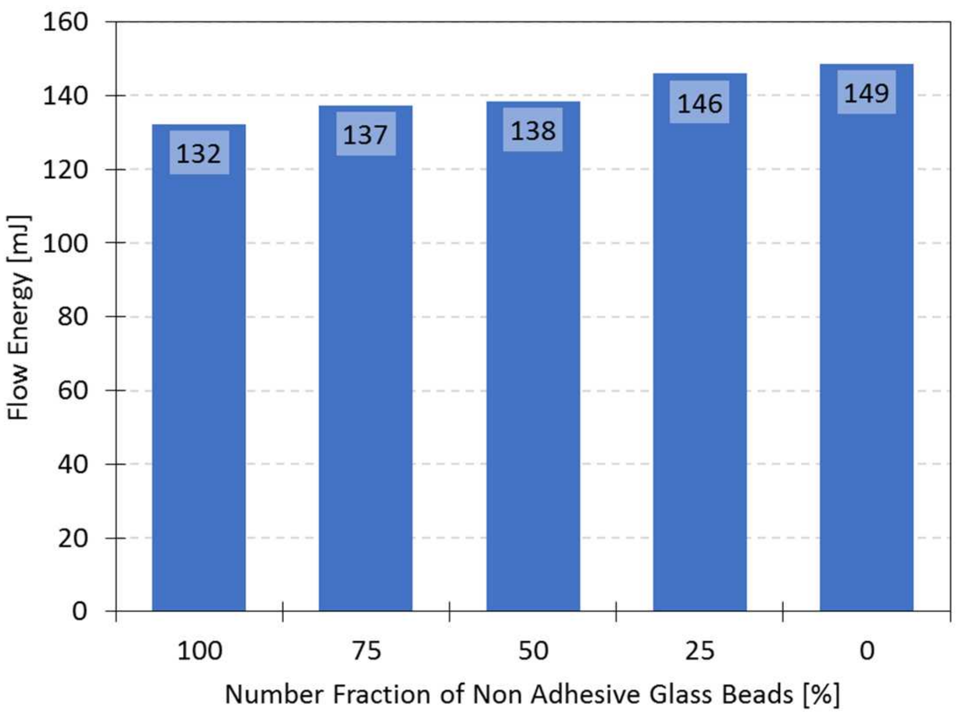
Flowability

Effect of Particle Adhesion: Downward Test Results



Number Fraction NSGB [%]	Number Fraction SGB [%]	Flow Energy [mJ]
100	0	132.3
75	25	137.4
50	50	138.5
25	75	145.9
0	100	148.7

Manufacturability Index for Powder Flow

The Approach of Capece *et al.**

Flow Function and Granular Bond Number For Multi-Component Powder Bed

$$ff_{c,mix} = \alpha \left(Bo_{g,mix} \right)^{-\beta}$$

$$Bo_{g,Mix} = \left(\sum_{i=1}^{N} \sum_{j=1}^{N} \frac{w_{ij}}{Bo_{g,ij}} \right)^{-1}$$

w_{ij} is the interaction weighting factor

where α and β are the fitting parameters

$$Bo_{g,ij} = \frac{F_{ad,ij}}{W_{ij}}$$

$$W_{ij} = \frac{2W_i W_j}{W_i + W_j}$$

α is the flow function at the cohesive-non-cohesive boundary ($Bo_{g,mix}$=1)

$$w_{ij} = f_{SA,i} f_{SA,j}$$

f_{SA} is the fractional surface area that gives the likelihood that the two material (i and j) come into contact

*Capece *et al.* (2015), Powder Technology 286 561–571

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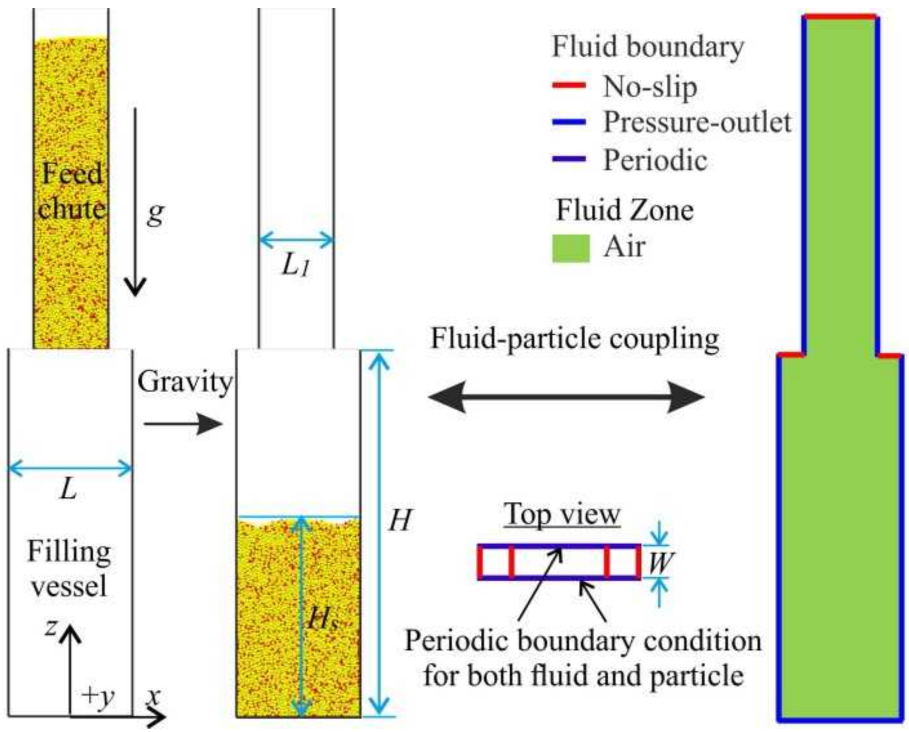
Elutriation Segregation

(Dr W. Nan)



Aspects under investigation:

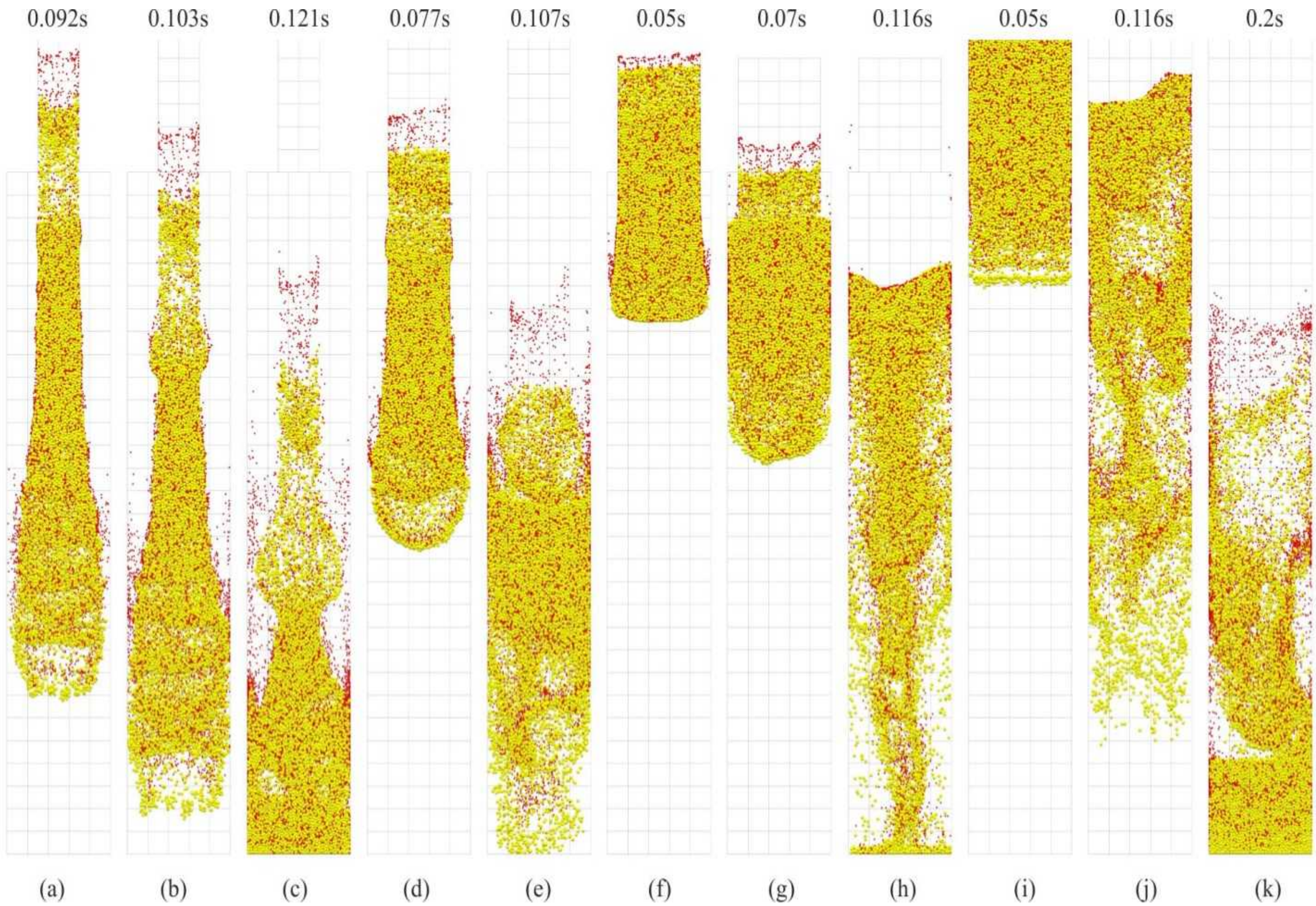
- Effect of the depth of filling vessel
- Effect of inlet ratio
- Effect of size ratio and density ratio

Simulation Parameters

Parameters	Basic Value	Value range
Geometry		
Length (mm), L	10	-
Depth (mm), H	60	30-180
Width (mm), W	2	-
Inlet ratio, IR	0.6	0.4-1.0
Gas (air)		
Density (kg/m³), ρ_f	1.2	-
Viscosity (Pa·s), μ_f	1.8×10^{-5}	-
Particle		
Diameter (mm), d_p		
Fine particle, $d_{p,f}$	0.1375	-
Coarse particle, $d_{p,c}$	0.275	0.275, 0.55
Density (kg/m³), ρ		
Fine particle, $d_{p,f}$	1300	-
Coarse particle, $d_{p,c}$	1300	1300, 7800
Volume fraction of fine particle, x_f	0.1	-
Poisson's ratio, ν	0.3	-
Shear modulus (MPa), G	10	-
Restitution coefficient, e	0.42	-
Friction coefficient, μ	0.5	-

Effect of Inlet Ratio

IR=0.4 (a-c), IR=0.6 (d-e), IR=0.8 (f-h) and IR=1.0 (j-k)



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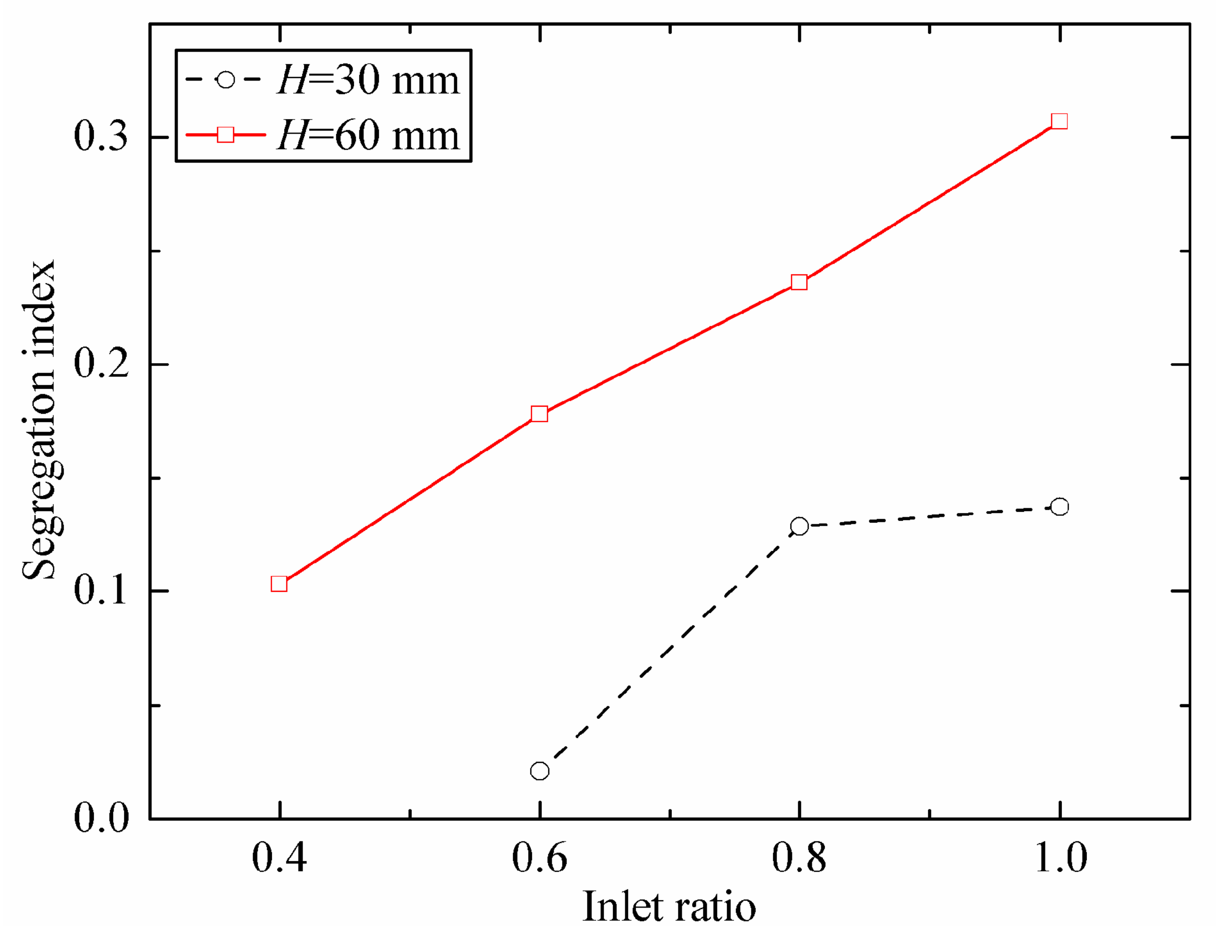
Effect of the inlet ratio on the vertical segregation index



$$SI = \frac{\sigma}{\bar{x}}$$

Mixing and Segregation

Concluding remarks

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VFL

- develop the science base for understanding of particle surfaces, structures and bulk behaviour to address physical, chemical and mechanical properties and behaviour during processing and storage
- develop formulation science to link molecule to manufacturability (through experimental characterisation and numerical modelling)
- establish methodologies to formulate new materials through developing functional relationships, considering the limits and uncertainties
- Develop a software tool for prediction and optimisation of manufacturability and stability of advanced solids-based formulations

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