Modeling and optimization of continuous granulation

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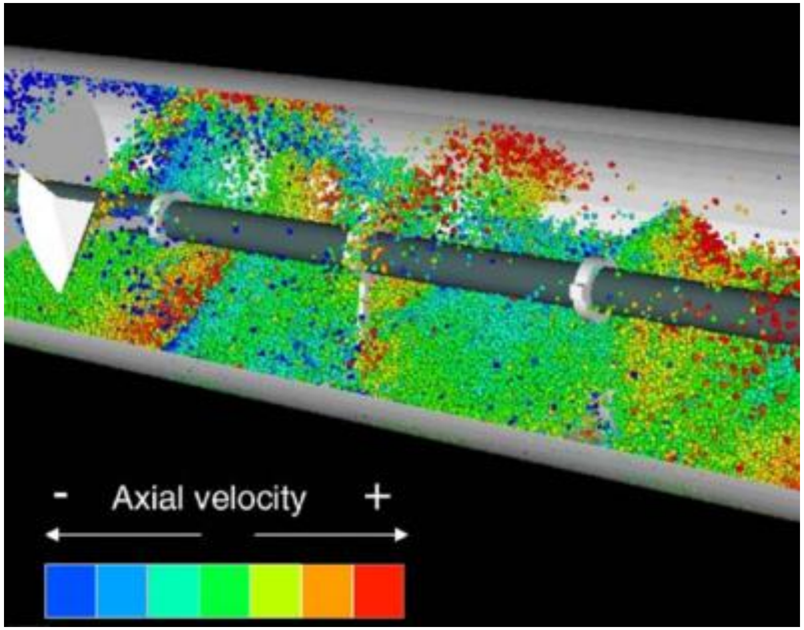
OUTLINE

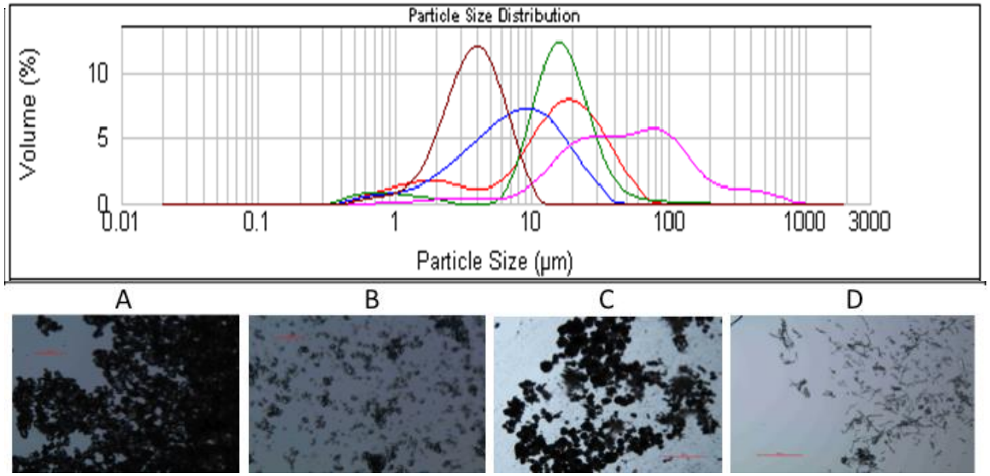
- Granulation rate processes and regime maps
- Elements of a mechanistic based process model for granulation
- Implementation in gSolids and case studies

Continuous granulation is a complex

Complex equipment

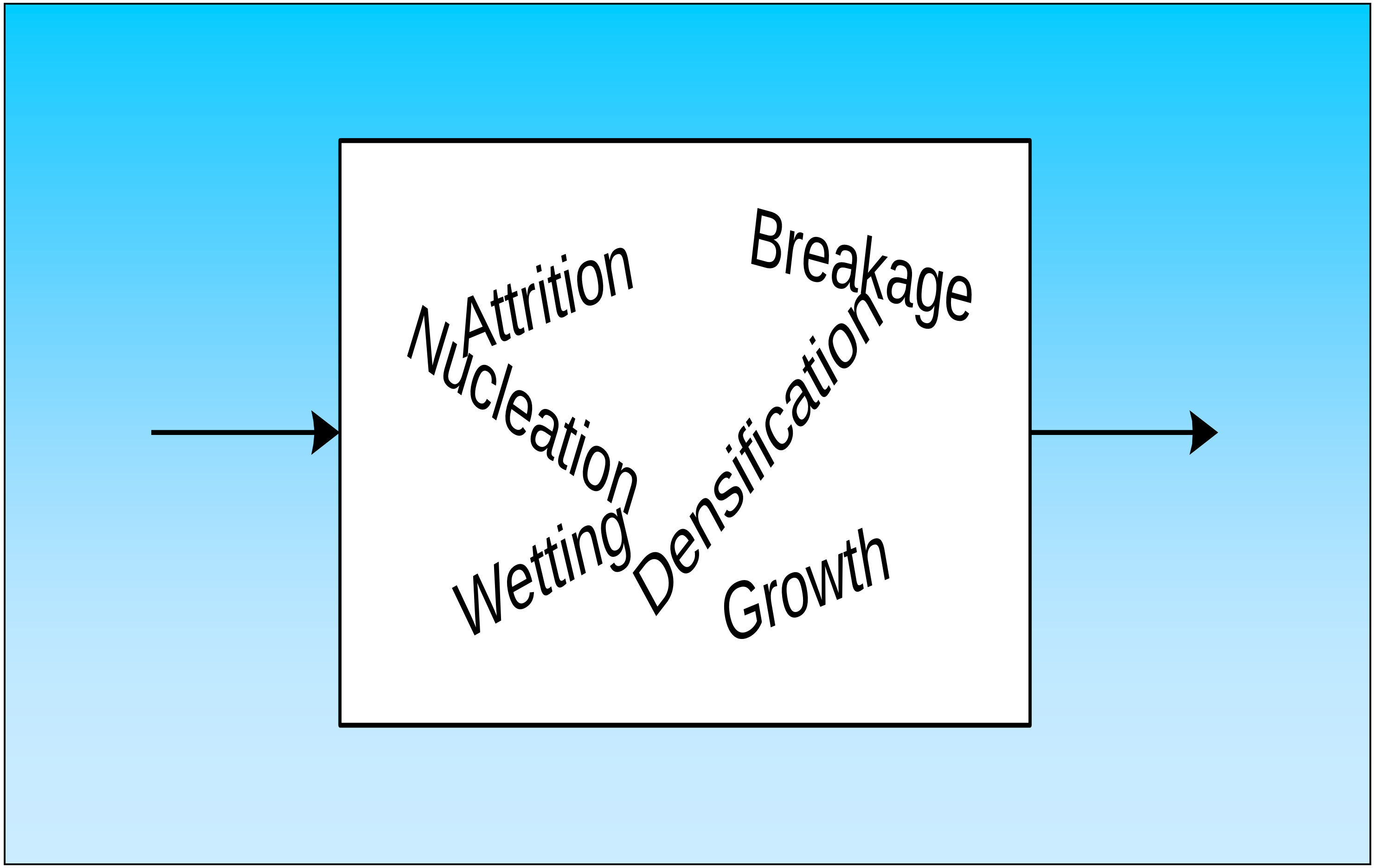


Complex formulations



Quantitative Engineering Approaches

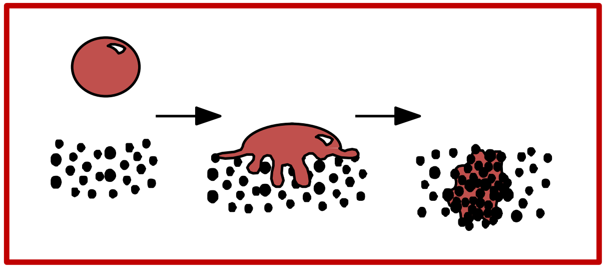
What do we know?	How do we design experiments and scale ?	Implications
Nothing except parameters we can vary	Statistical Experimental Design	Lots of experiments at all scales
Controlling mechanisms	• Careful formulation and process characterization • Designing experiments based on **dimensionless groups and regime maps**	• Reduced experiments at all scales • Use dimensionless groups to scale up
Fully predictive model	• Careful formulation and process characterization • Design min. number of experiments to validate and fine tune the model	• Least number of experiments • Pilot/full scale model validation and parameter estimation


Attrition
Breakage
Nucleation
Densification
Wetting
Growth

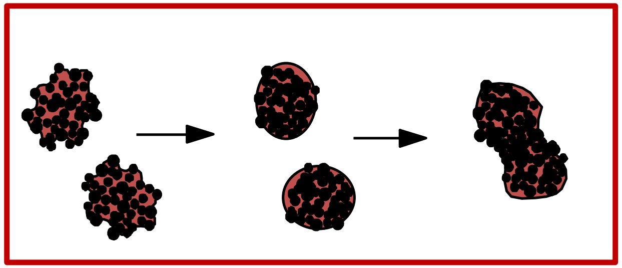
Granulation rate processes

- Study and model each process in isolation

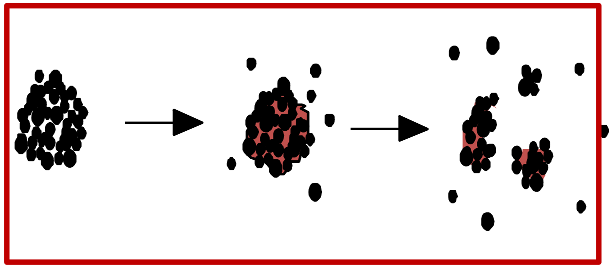
Nucleation



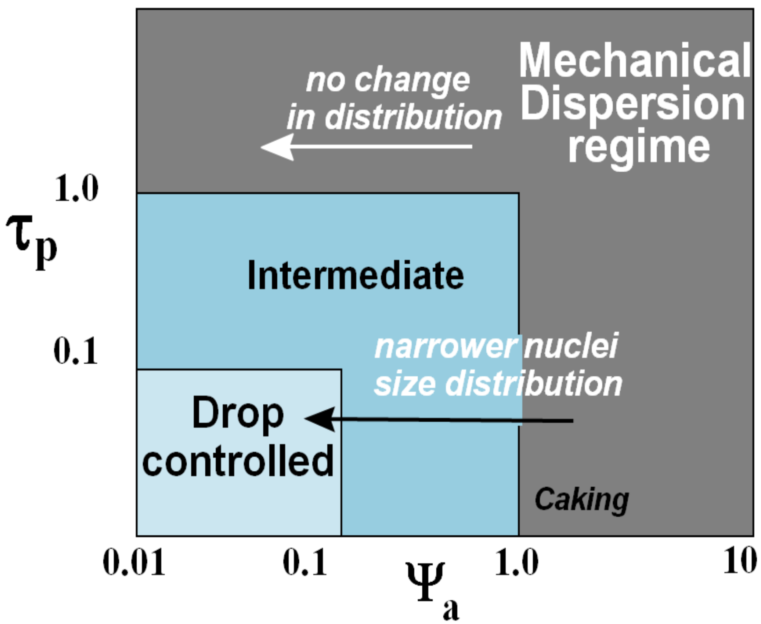
Consolidation and Growth

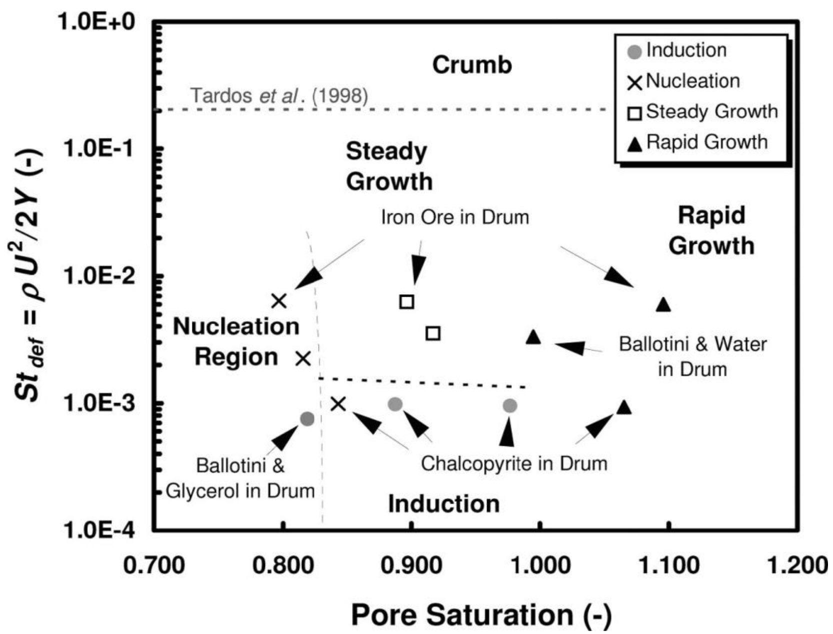


Breakage



Regime Maps for Rate Processes

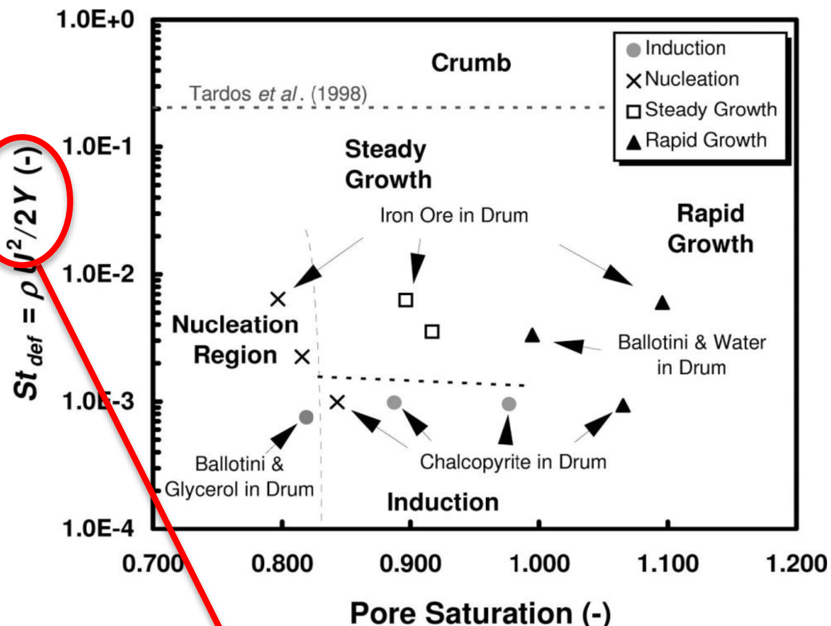


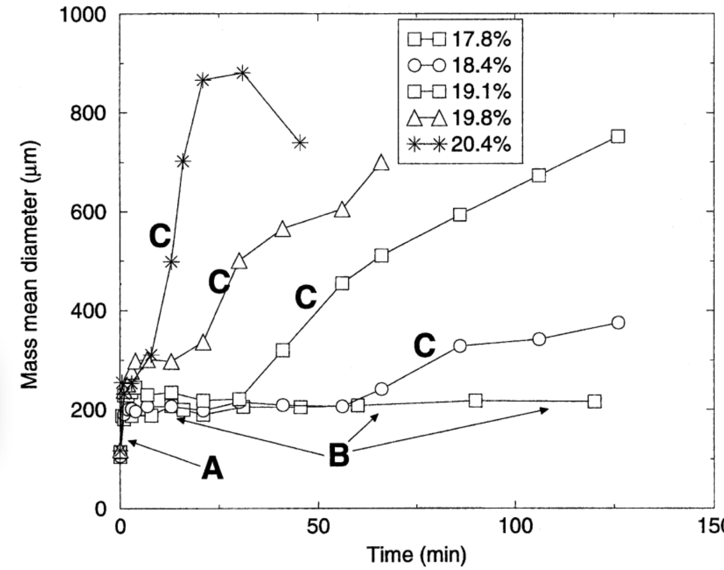


Hapgood, Litster & Smith, *AIChE J,* **49**, 350-361, 2003

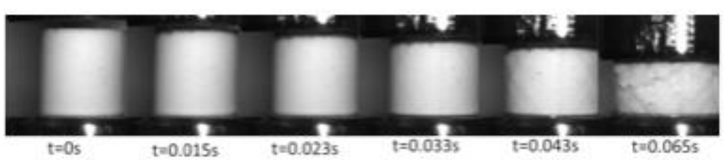
Iveson *et al.*, *Powder Technol.,* **117**, 83-87, 2001

Influence of formulation properties

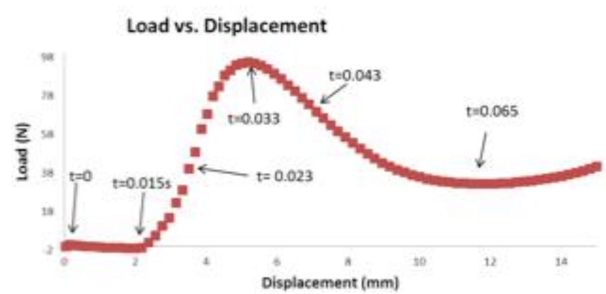




Dynamic yield strength



$$St_{def} = \frac{\rho_g U_c^2}{2Y_d}$$



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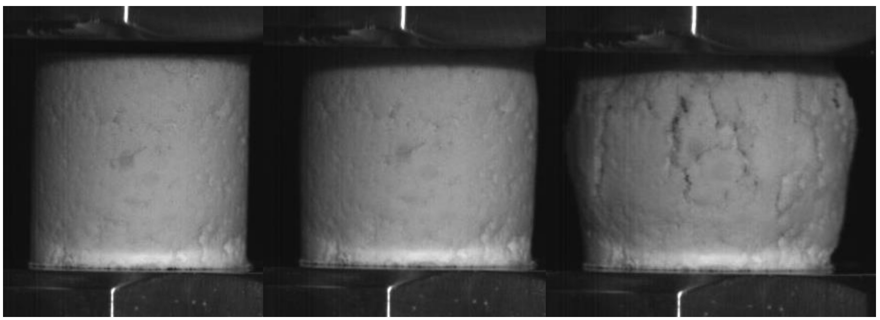
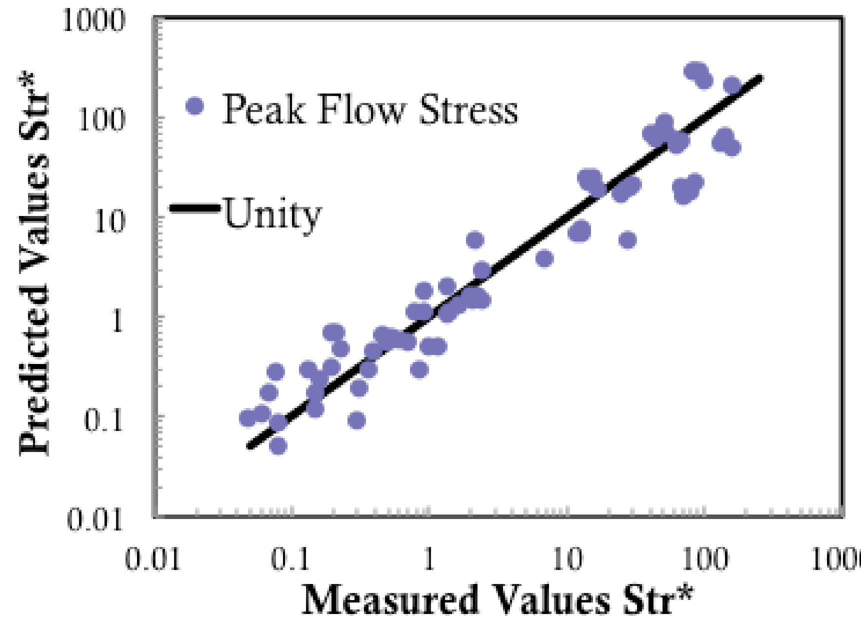
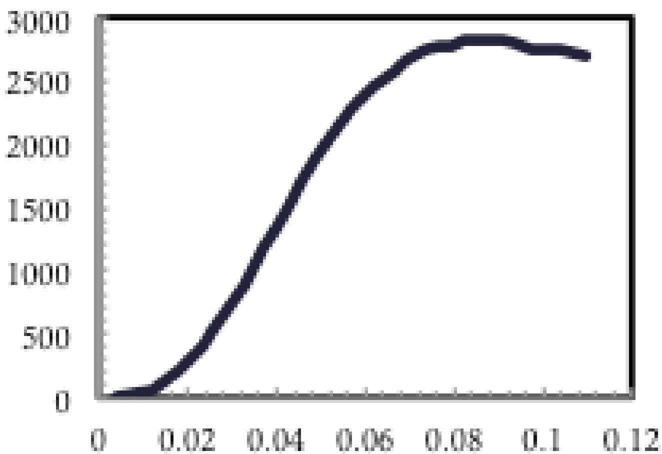
Modeling induction growth







$$Str^* = 0.001 \pm .002 \left[Ca^a e^b s^c \right]$$

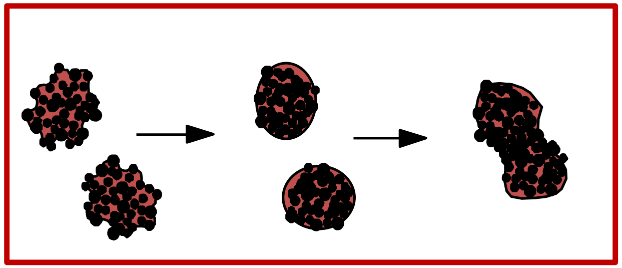
a: 0.29 ± 0.12

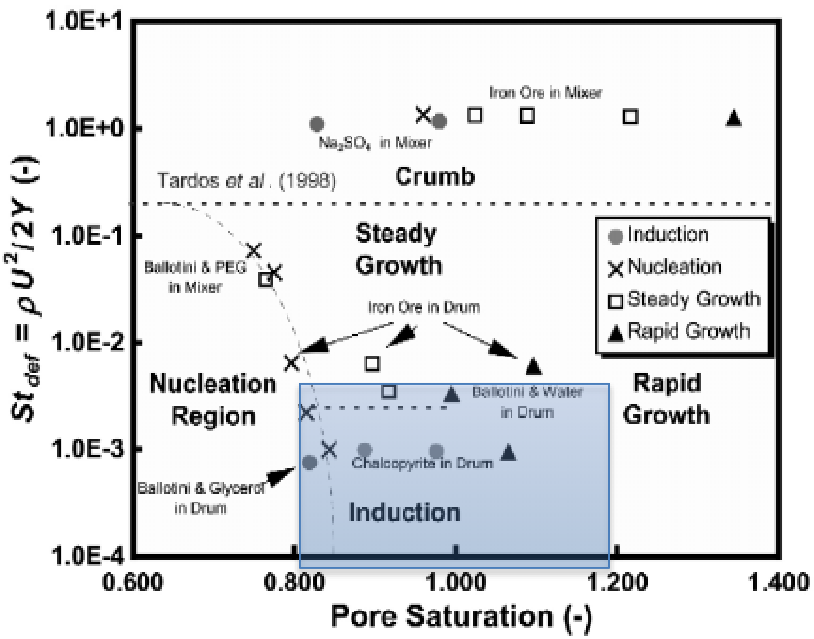
b: -12.6 ± 1.5

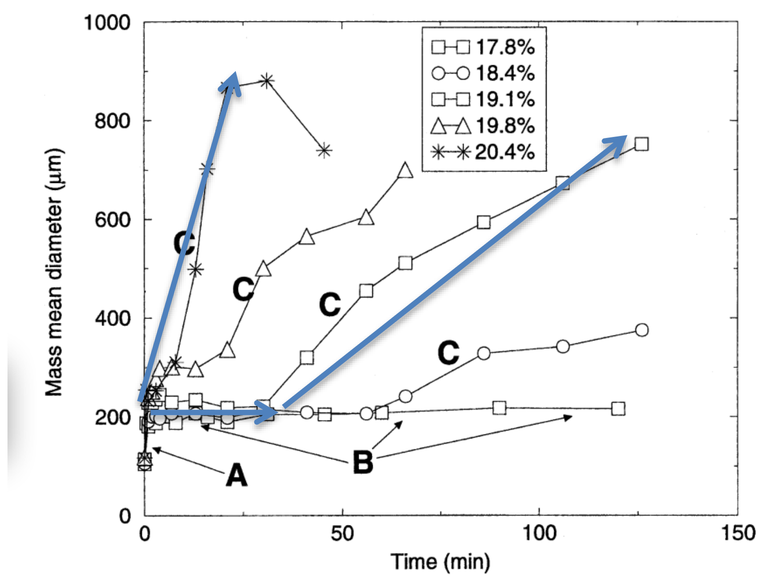
c: - 4.4 ± 0.96

Modeling induction growth

Consolidation and Growth

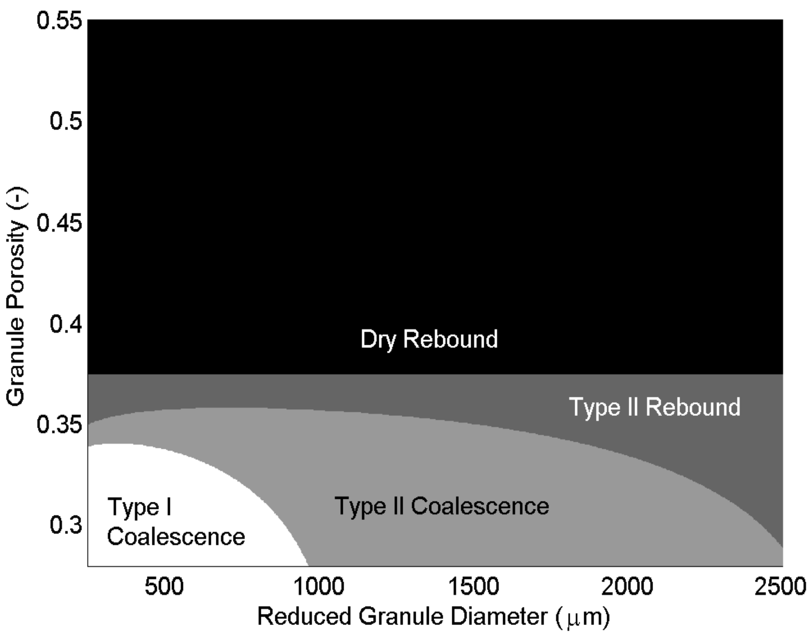




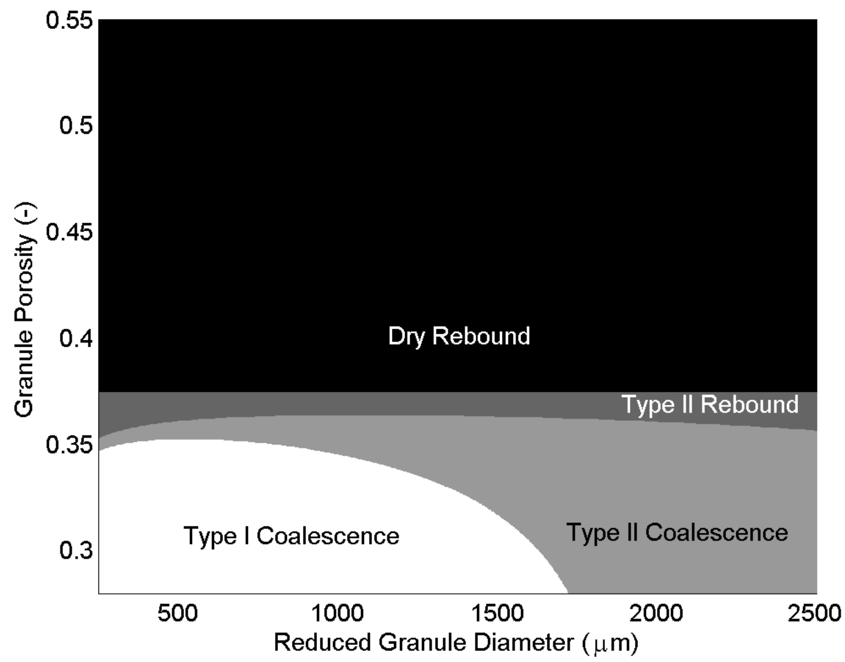


Dan Pohlman

Coalescence regime predictions



w = 0.152; S= 0.6;* U_c*= 0.6 ms*$^{-1}$



w = 0.152; S= 0.6;* U_c*= 0.4 ms*$^{-1}$

Regime Map Approach

Measure Formulation Properties:
d_p, $\gamma\cos\theta$, μ, Y…

Estimate Process Parameters at different conditions:
U_c, $R\omega$…

↓

Calculate important dimensionless groups:
Ψ_a, t_p, St_{def}, S…

↓

Locate the regime of operation on nucleation and growth regime maps that are functions of dimensionless groups

↓

Design and perform laboratory scale experiments regarding to regime maps

↓

Determine the exact borders of different regimes on the regime map for the given formulation and determine optimum design space

↓

Scale-up by keeping the dimensionless numbers constant

Quantitative Engineering Approaches

What do we know?	How do we design experiments and scale ?	Implications
Nothing except parameters we can vary →	Statistical Experimental Design →	Lots of experiments at all scales
Controlling mechanisms →	• Careful formulation and process characterization • Designing experiments based on **dimensionless groups and regime maps** →	• Reduced experiments at all scales • Use dimensionless groups to scale up
Fully predictive model →	• Careful formulation and process characterization • Design min. number of experiments to validate and fine tune the model →	• Least number of experiments • Pilot/full scale model validation and parameter estimation

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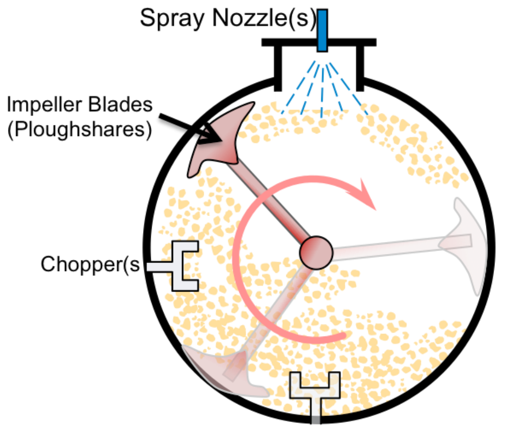
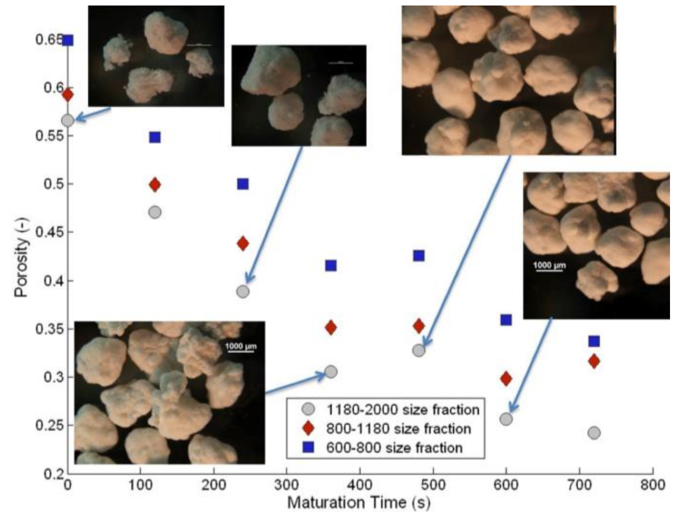
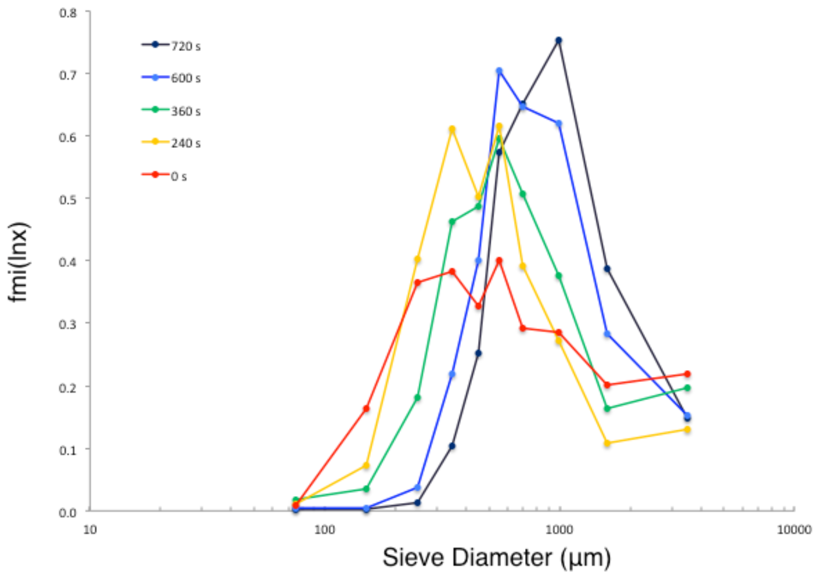
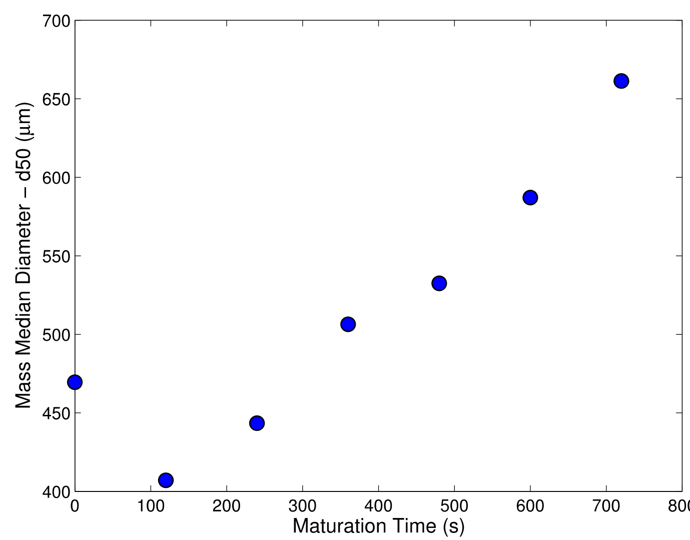
Need to capture complex processes



Horizontal high-shear mixer

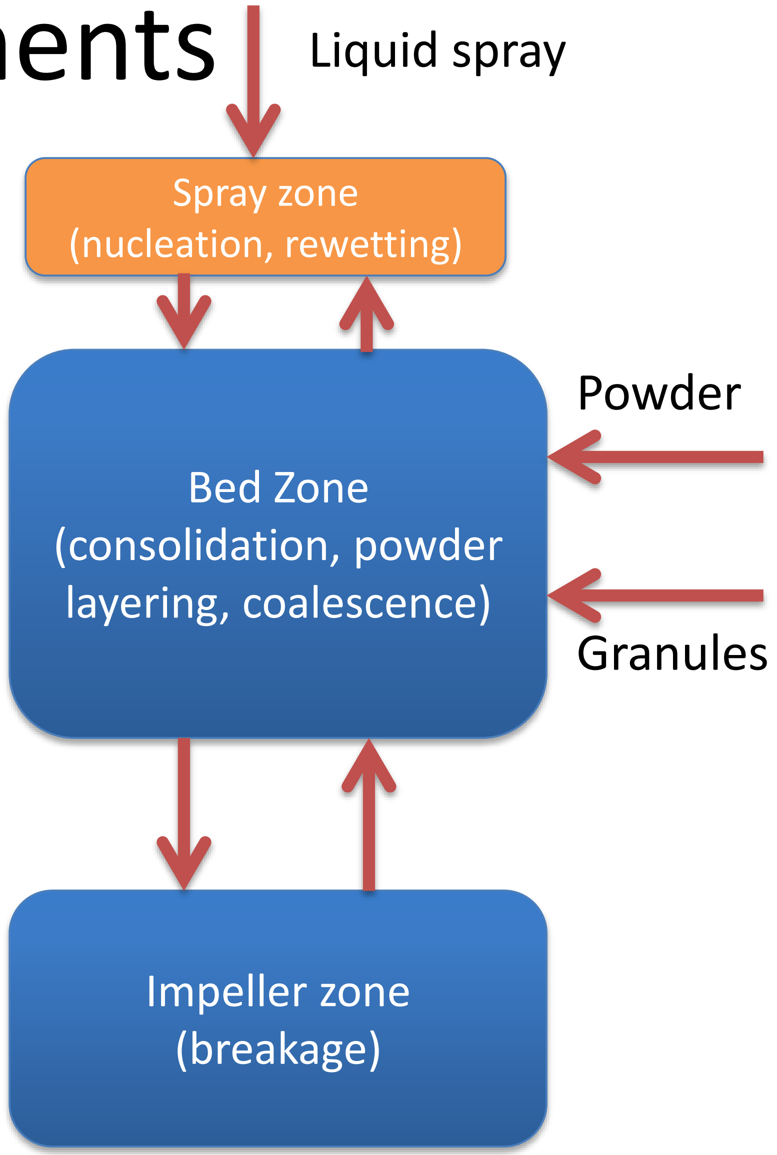






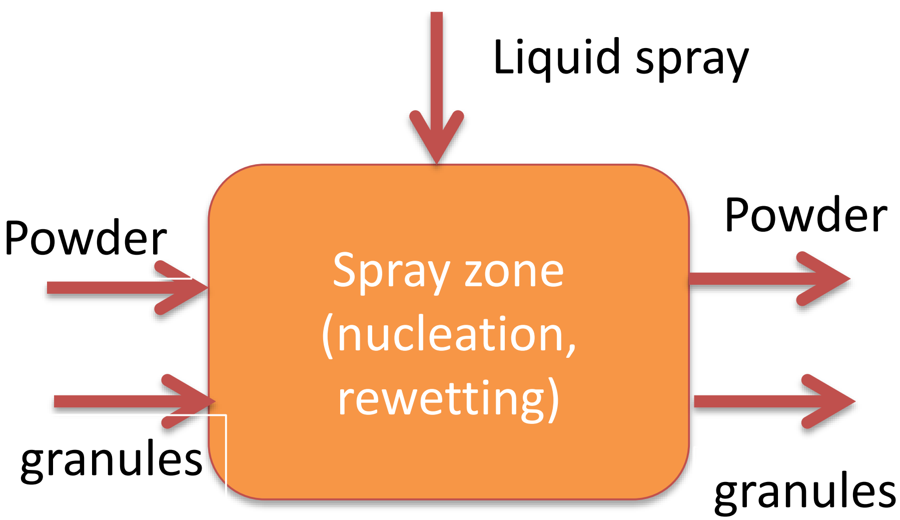
Key elements

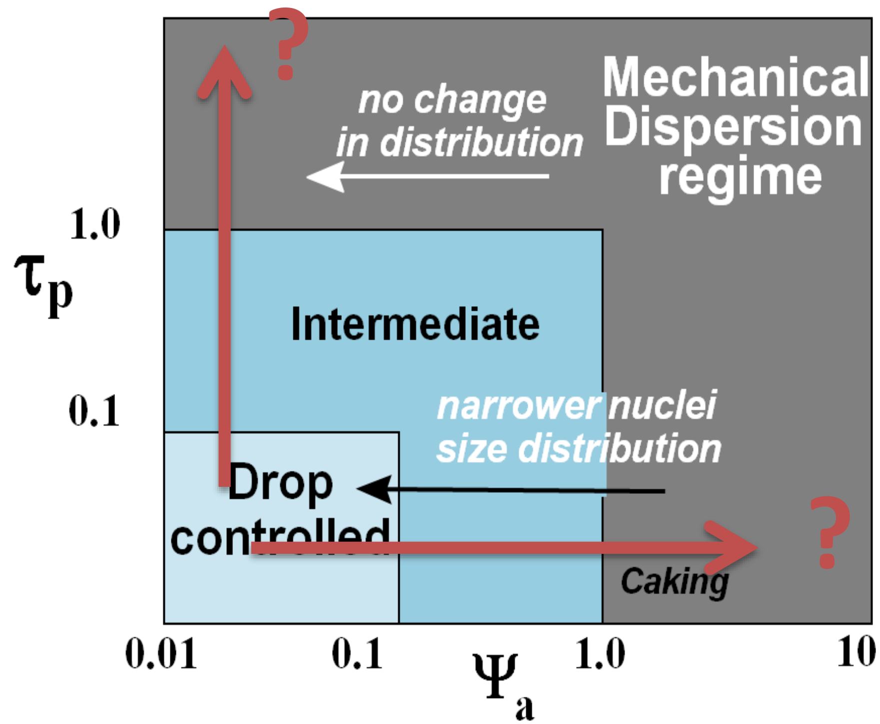
- Nuclei and rewetting generator
- Zone for consolidation, powder layering and coalescence
- Zone for breakage
- Continuous powder and liquid phases
- 3D granule phase v_s v_l v_g



Spray zone

- Small zone with geometry set by spray geometry





Spray zone model

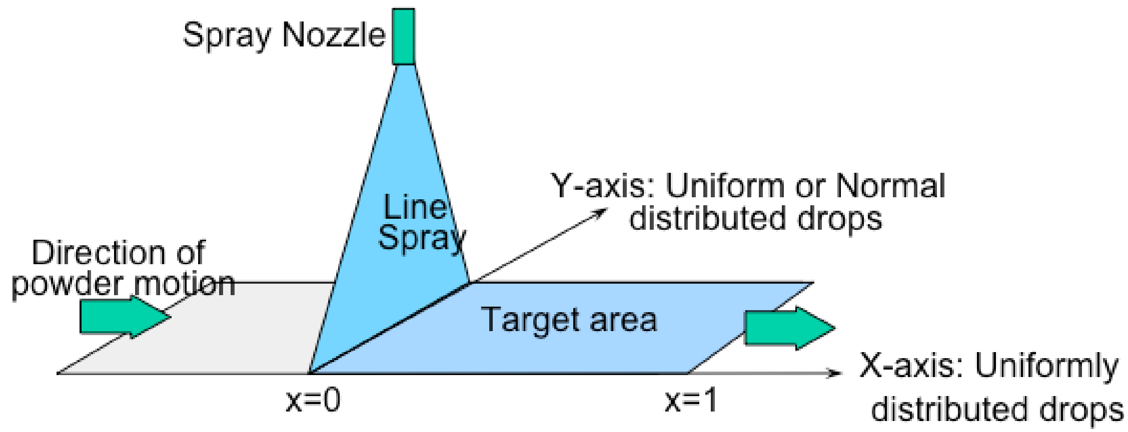
- Key relationships

$$y = \frac{3\dot{Q}_{spray}}{2W u d_{3,2drop}}$$

- Nucleus structure

$$v_s = \left[\frac{1 - e_{nuc}}{e_{nuc}} \cdot \frac{1}{S_{nuc}} \right] v_l$$

$$v_g = \left[\frac{1 - S_{nuc}}{S_{nuc}} \right] v_l$$



- Ratio of nuclei to layering

$$\dot{Q}_{nuc} = \left(1 - f_g\right)\dot{Q}_{spray}$$

$$\dot{Q}_{layer} = f_g \dot{Q}_{spray}$$

Spray zone model

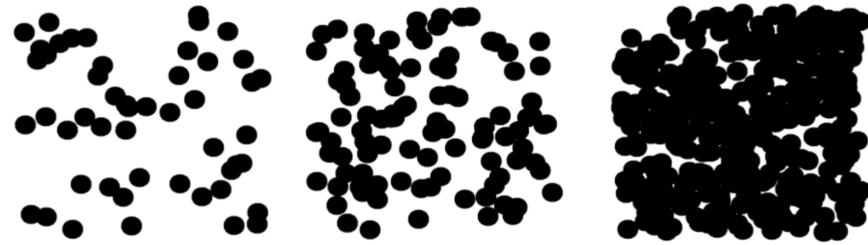
- Nucleation model

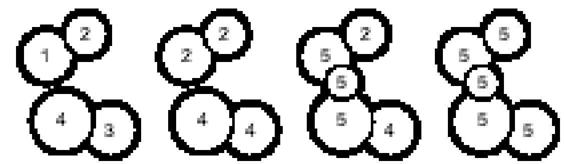
$$V\dot{b}_{nuc}\left(v_s, v_l, v_g\right) = \dot{Q}_{nuc} f[n_d(v_l), \psi]$$

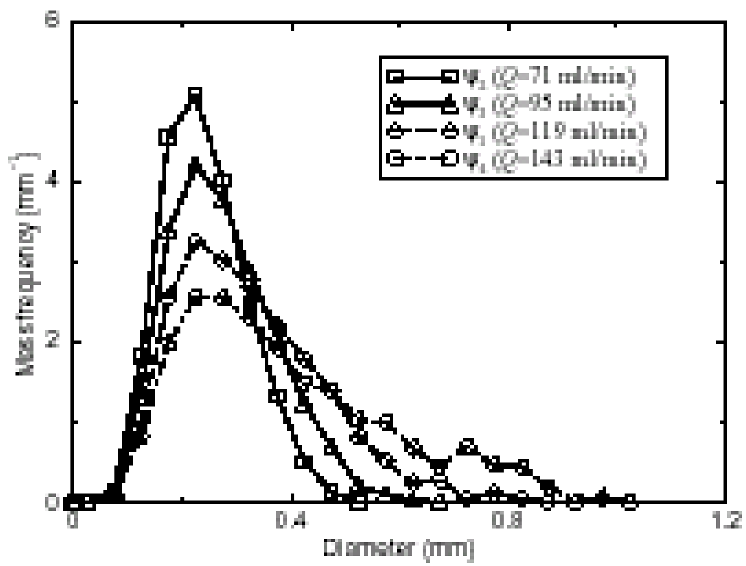
- f approaches Kn_d as ψ approaches 0 (drop controlled nucleation) but broadens the size distribution as ψ increases. Poisson distribution suggests:

$$\left(\bar{v}_{1,0}\right)_{nuc} = 4\psi \left[\frac{1}{e_{nuc} S_{nuc}}\right] \left(\bar{v}_{1,0}\right)_{drop}$$

- Distribution keeps the same mode but becomes more skewed as 4ψ increases.

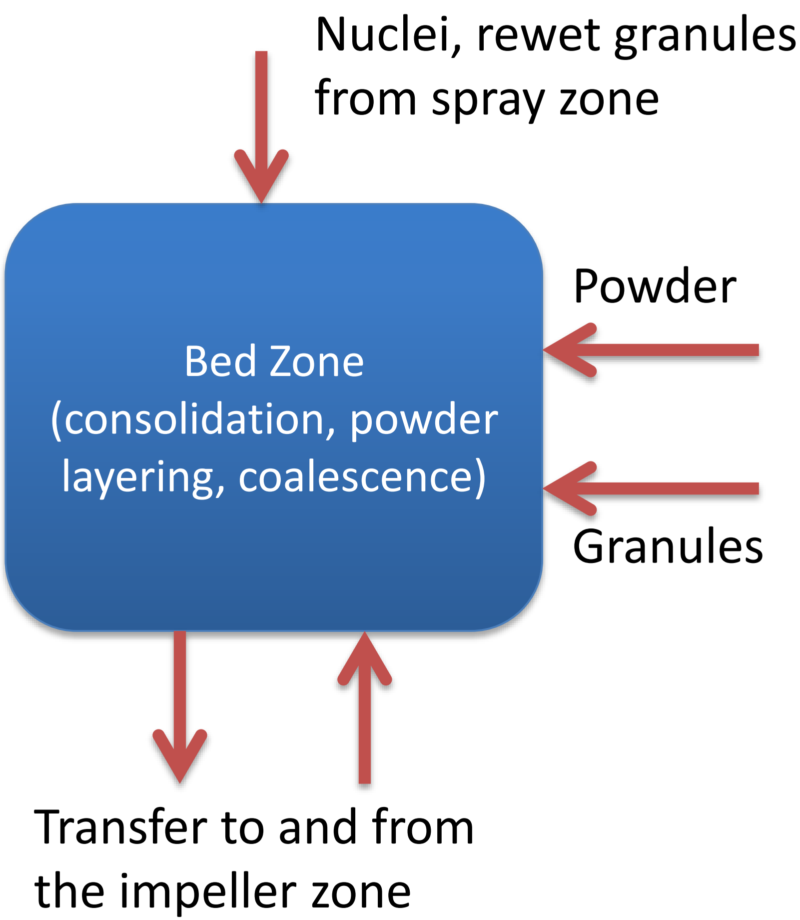






Bed Zone Model

- Consolidation modeled as first order process
- Powder layering occurs if
  - Granules are surface wet
  - Powder is still available
  - Directly linked to consolidation rate
- Coalescence only occurs if all the powder used up
  - Predict induction time
  - Surface wet granules
  - Granule deformation



Bed Zone Model

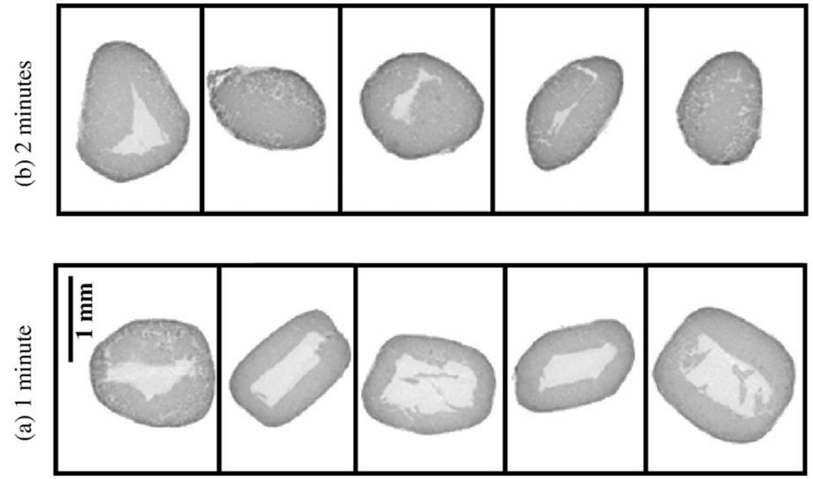
$$\frac{\partial V n_b(v_p, v_s)}{\partial t} + \frac{\partial V n_b(v_p, v_s) G_s}{\partial v_s} = \dot{Q}_{i-b} n_i - \dot{Q}_{b-i} n_b + \dot{Q}_{paste} n_p$$

$$G_s \propto \frac{d\varepsilon}{dt} = -k_c(\varepsilon - \varepsilon_{min})$$

$$k_c = f\left(St_{def}\right)$$

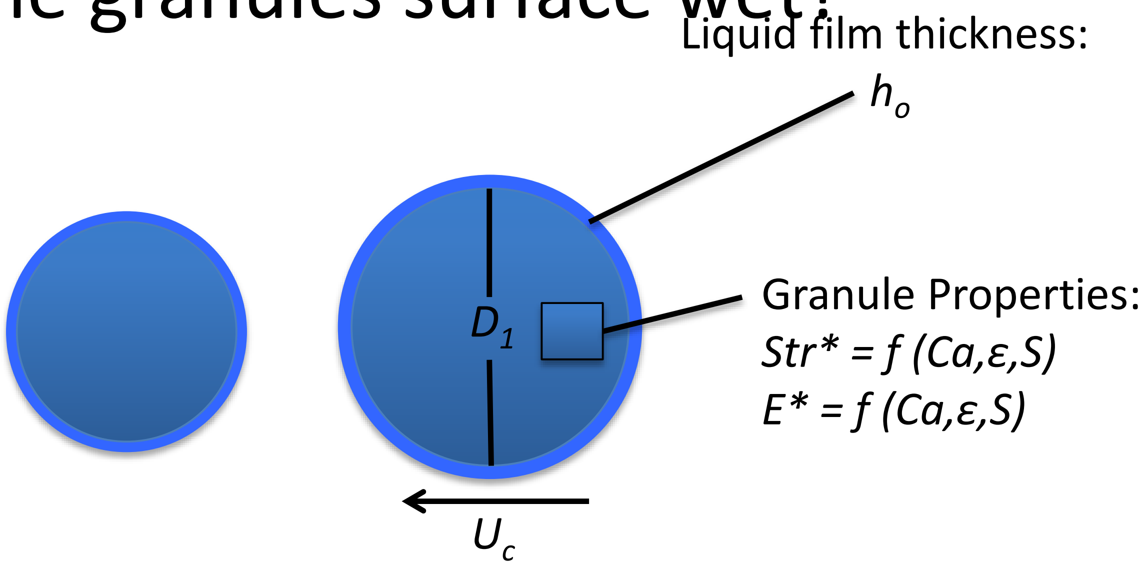
$$G_g = \frac{dv_g}{dt} = -k\,\frac{(1-\varepsilon_{\min})(v_s + v_l + v_g)}{v_s}\left[v_l + v_g - \frac{v_s(\varepsilon_{\min})}{1-\varepsilon_{\min}}\right]$$

- Based on models from either Iveson, or Hounslow
- Either squeezing paste to the surface, or removing air from the granule



Bed Zone Model

- Are the granules surface wet?



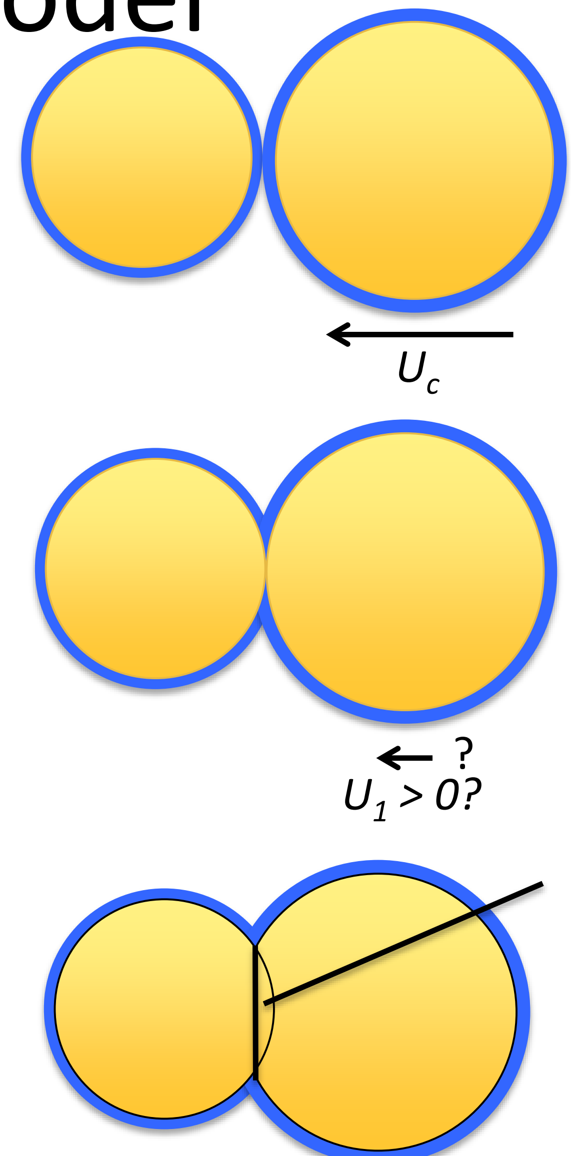
$$h_o = \begin{cases} D\dfrac{\varepsilon(S - S^*)}{\rho_l} & S > S^* \\ 0 & S < S^* \end{cases}$$

Bed Zone Model

- Coalescence

$$b = \begin{cases} k_I \; if \; St_v < St^* & type \; I \\ k_{II} \; if \; f(St_v, St_{def}) < 0 & type \; II \\ 0 \; if \; neither & \end{cases}$$

  – Other models in the literature where surface wet granules are required (Cryer, Biggs and Hounslow, …)

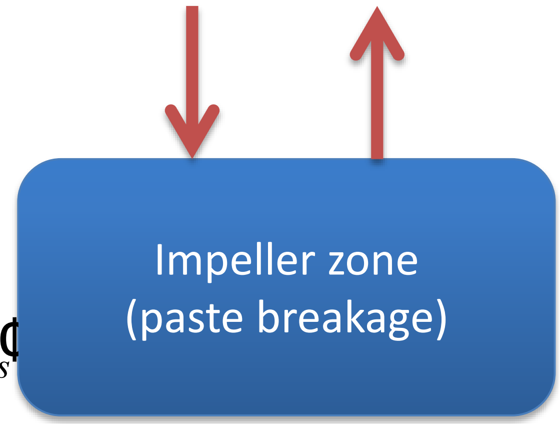


Impeller zone model

$$0 = \dot{Q}_{i-b} n_b - \dot{Q}_{b-i} n_i + V_i[\dot{b}_{br} - \dot{d}_{br}]$$

$$\dot{d}_{br} = S_{br} n_i(v_p, v_s)$$

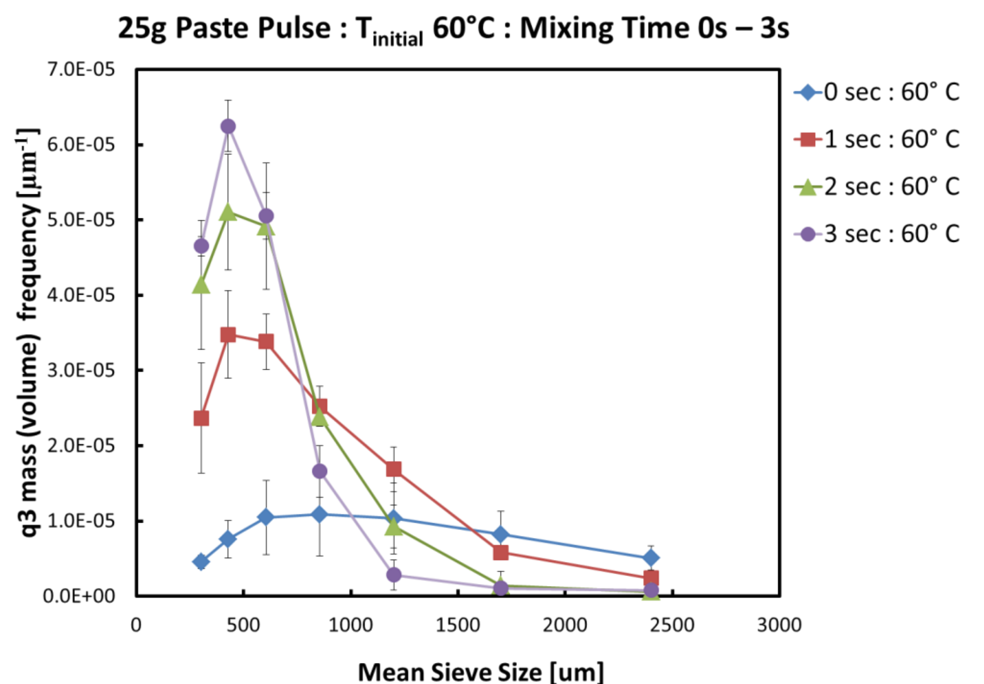
$$\dot{b}_{br} = \int_{v_p}^{\infty}\int_{v_s}^{\infty} \varphi(v'_p, v'_s, v_p, v_s) S_{br} n_i(v'_p, v'_s)\, dv'_p\, dv'_s$$



$$S_{br} = f(St_{def}, v_p)$$

Paste rheology

Impact energy or
Shear field



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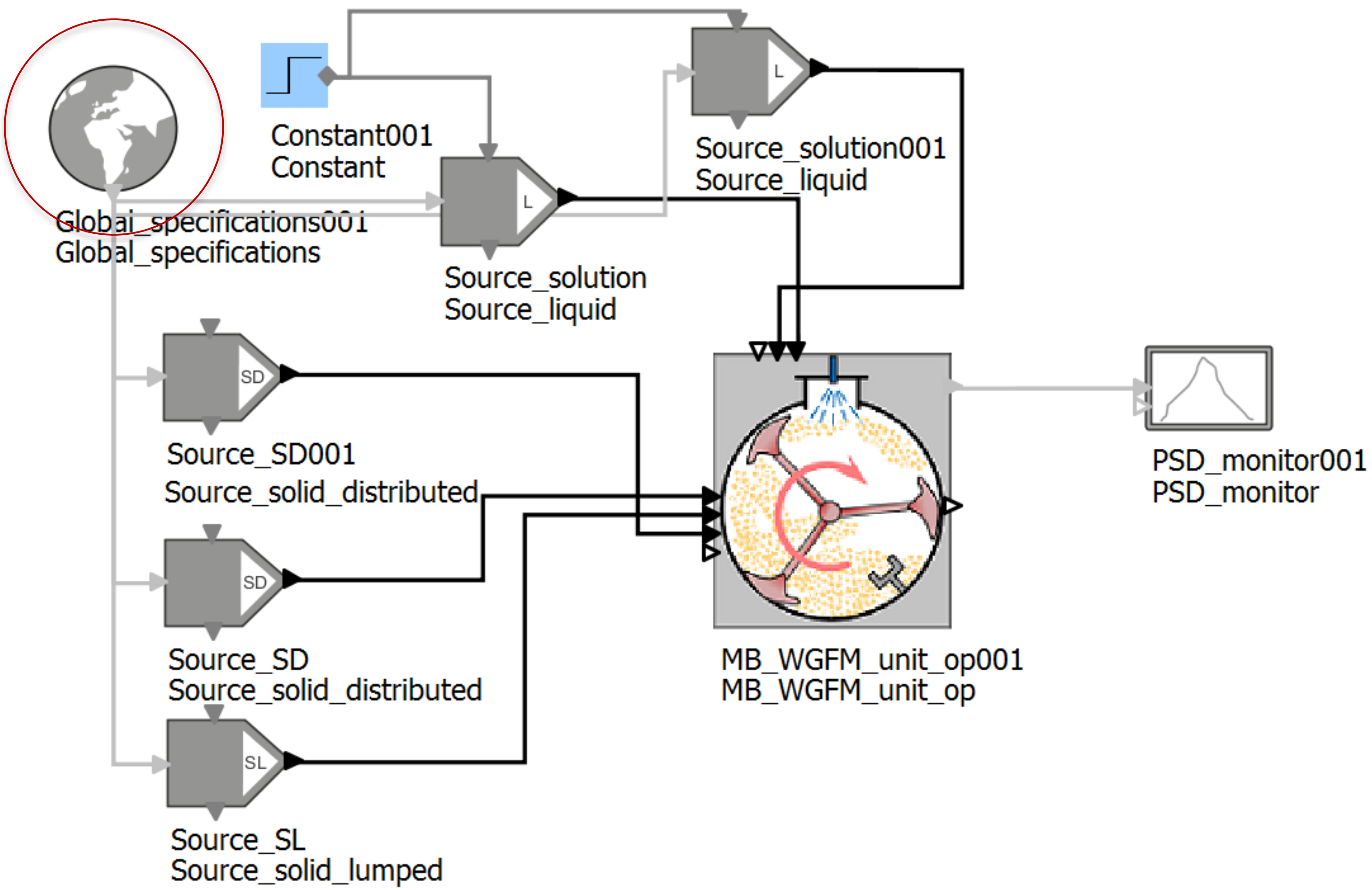
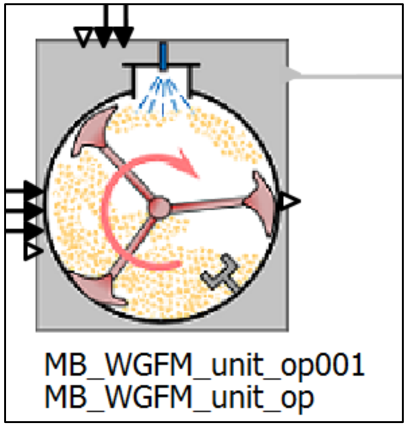
Implementation in gSOLIDS

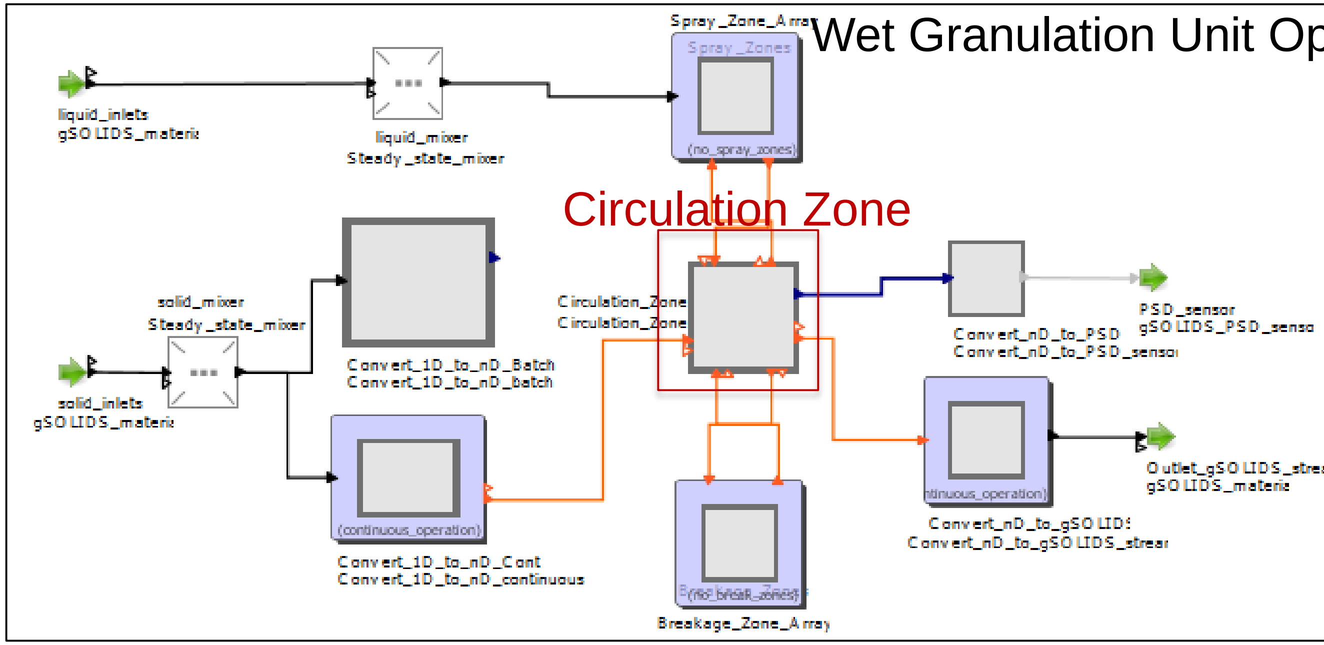
- Key issues
  - Capture key physics in the rate process models
  - Needs to be at least a 2D population balance
  - Compartment model to capture different zones in granulator
- University-PSE collaboration
  - Jim Litster spent 1 month at PSE in September 2013
  - Dan Pohlman spent a six month internship at PSE in 2014
  - Validation is underway for case study from Novozymes

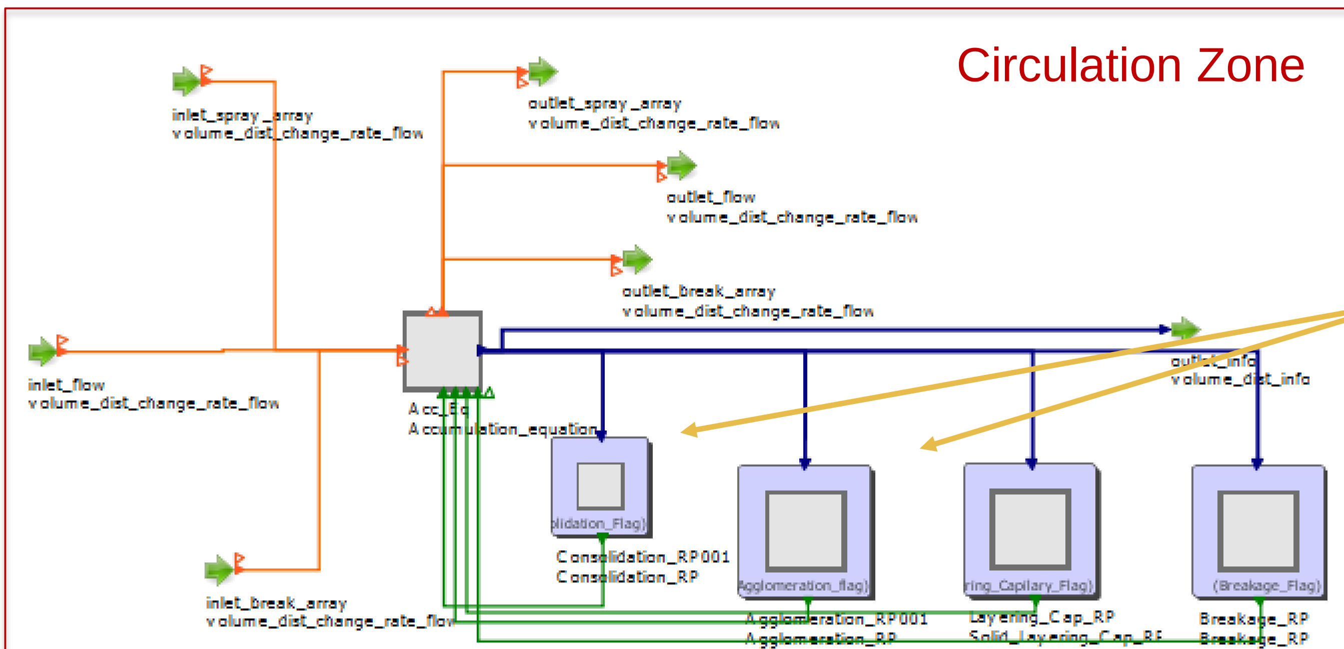
High Shear Wet Granulation Flowsheet



gPROMS Granulation Model

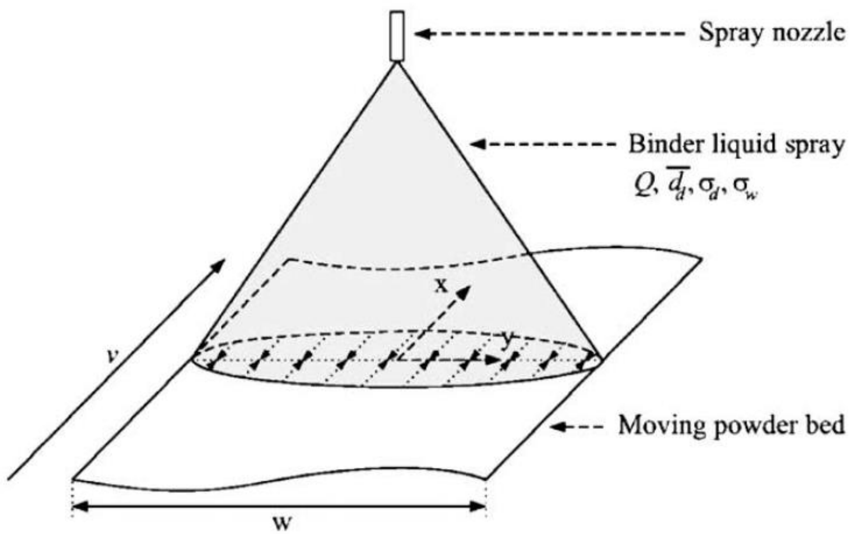


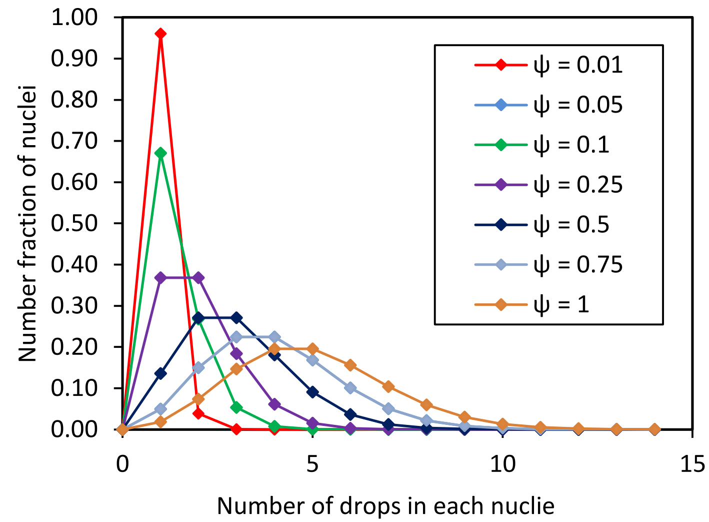


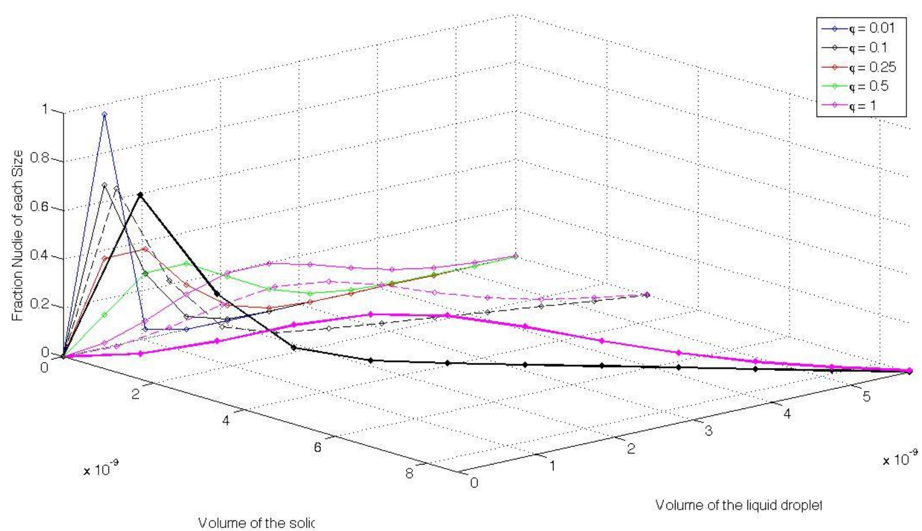


On / Off
Rate Processes

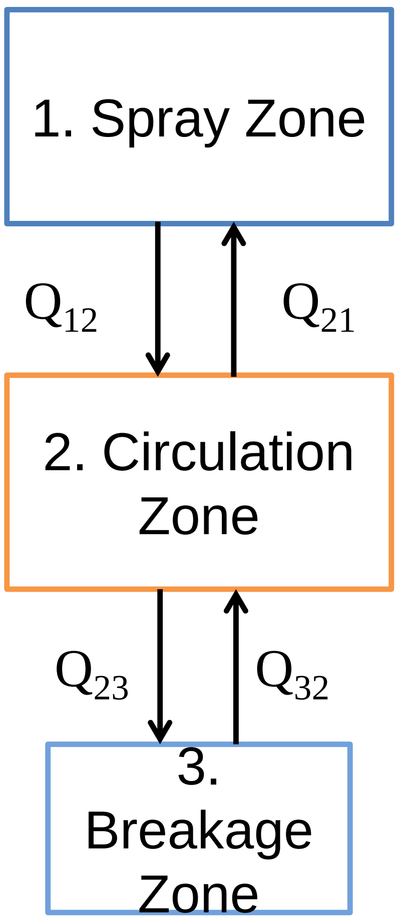
Spray Zone Model

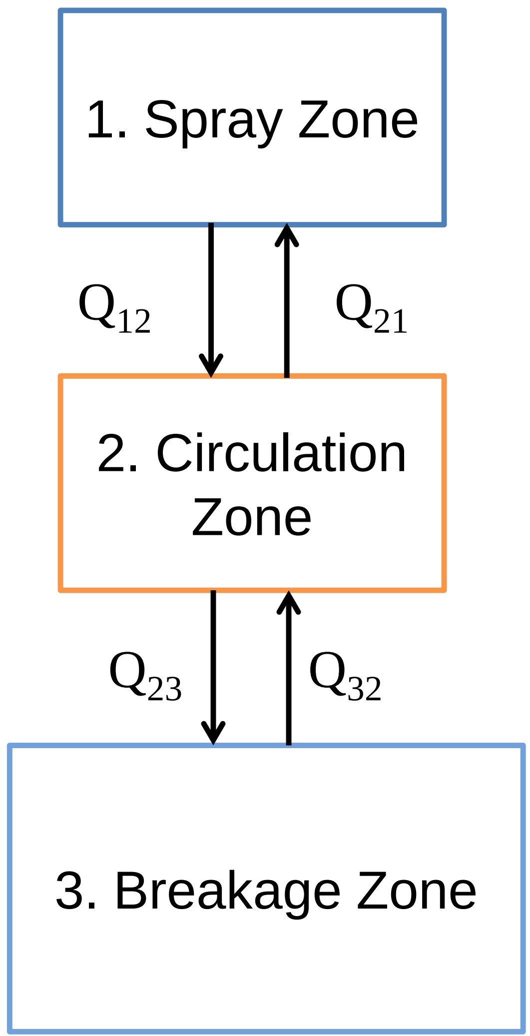


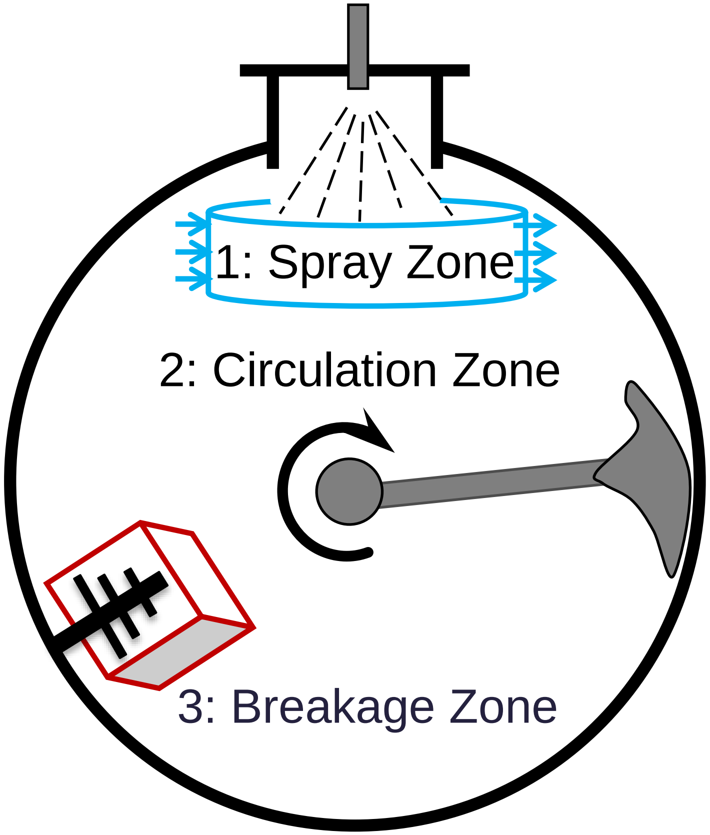




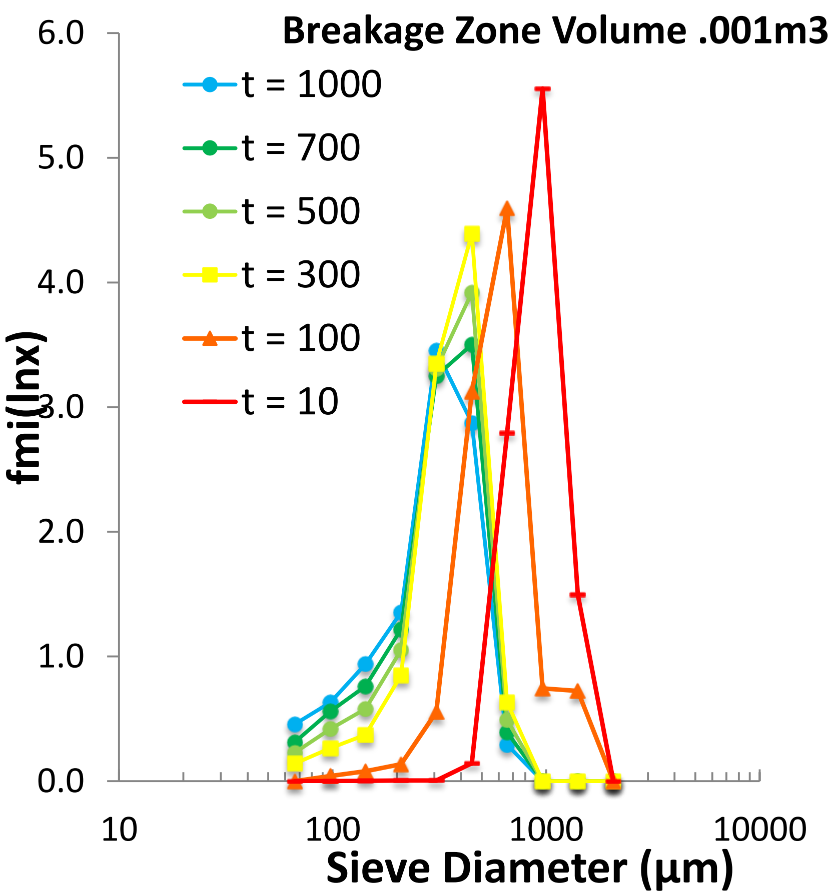
Compartment model - Breakage

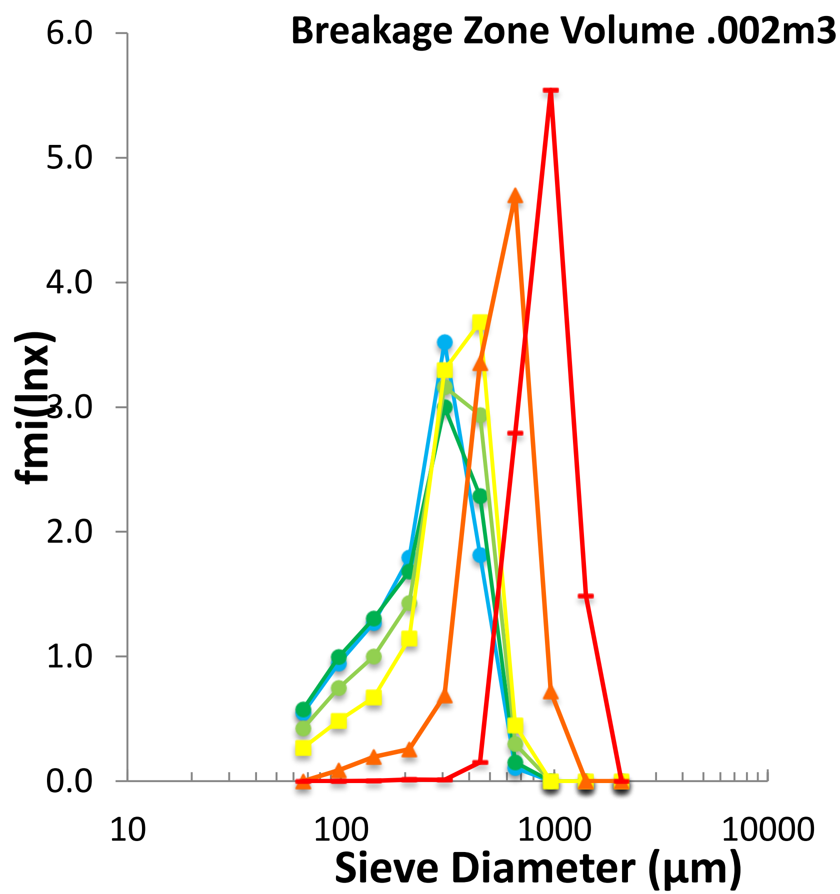




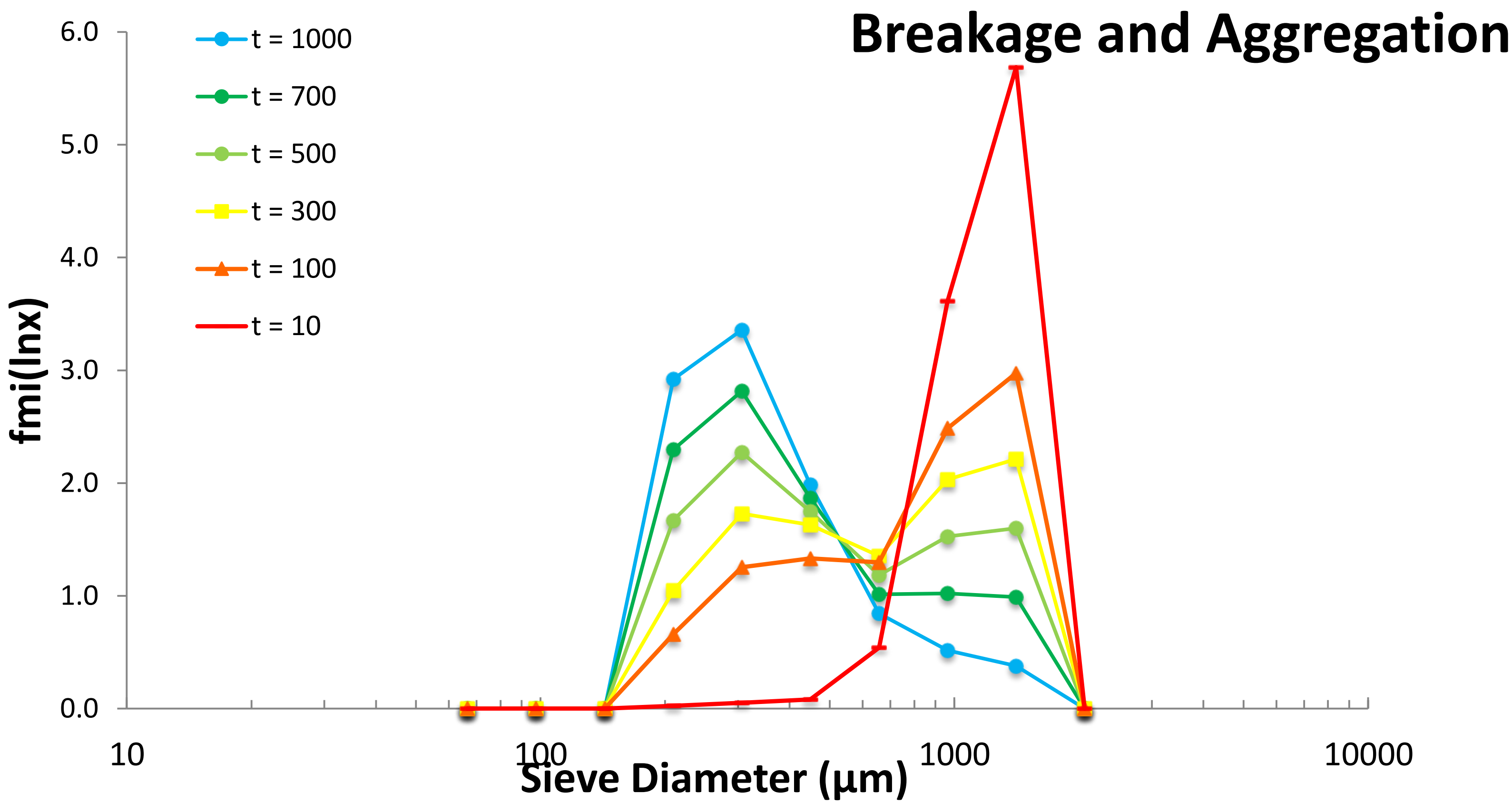


Compartment Model - Breakage

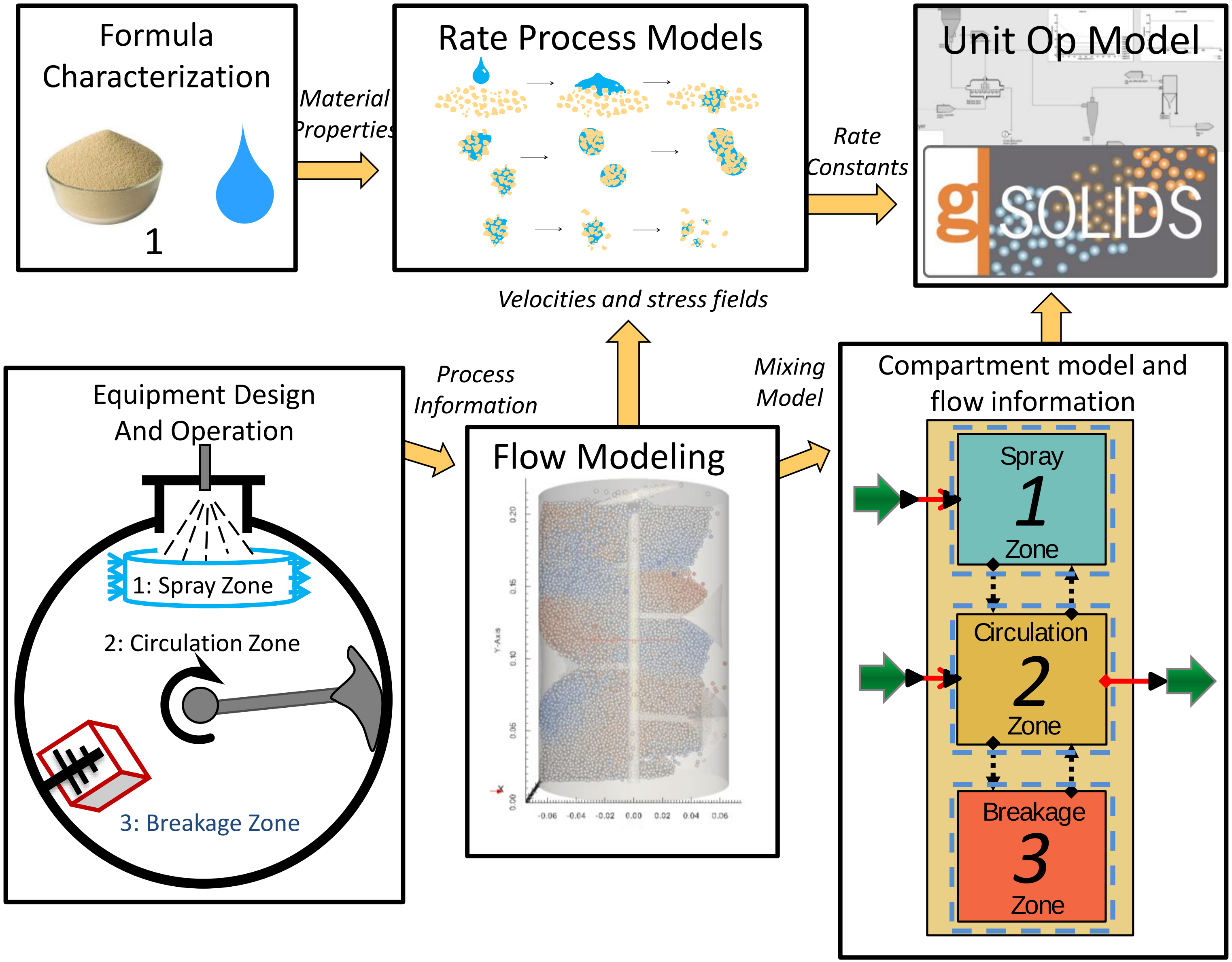




Model Results Breakage and Aggregation



Approach to Granulation



Summary

- A granulation process model needs to capture enough of the underlying physics to be useful for design and scaling
  - Key rate processes and key dimensionless groups
  - Compartment model for the granulator
  - More than 1-D population balance is needed
- Model is implemented in gSolids and validation against a complex industrial case study is underway.

Acknowledgments

- Colleagues at PSE: Sean Bermingham, Dana Barasso, Jianfeng Li, David Slade, …..
- Colleagues at Sheffield: Agba Salman, Mike Hounslow, Rachel Smith
- Colleagues at Novozymes: Poul Bach, Mark Bollinger
- Funding support from EPSRC, Novozymes, PSE

Back up slides

Bed Zone Model

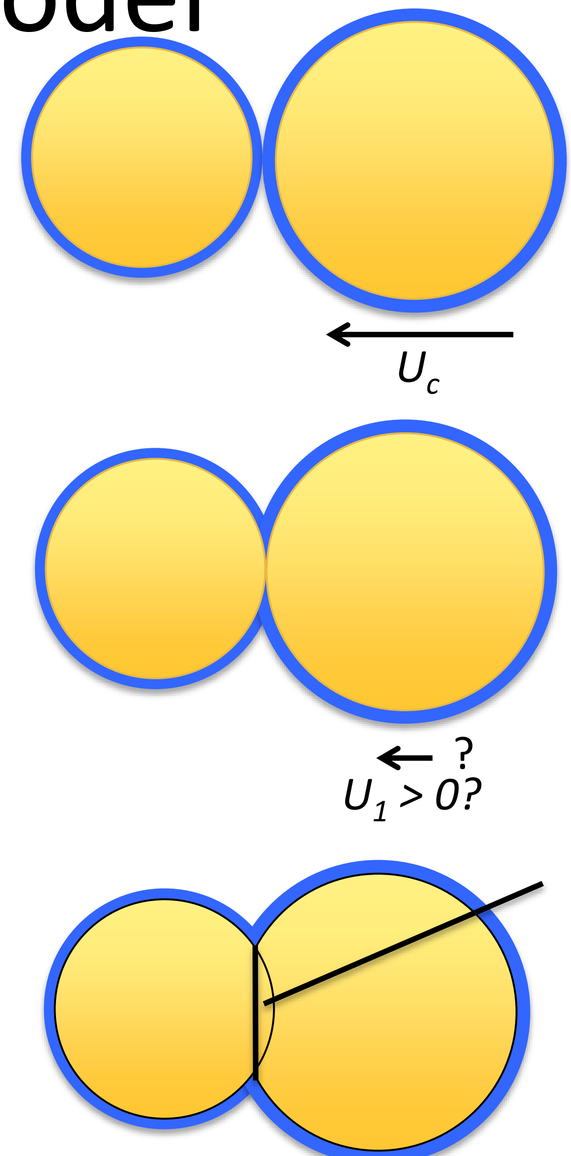
- Coalescence
  - Much more likely if granule is surface wet
  - Type I (Ennis)

    If $St_v < \ln(h_o/h_a)$
  - Type II requires deformation

$$\left(\frac{Y_d}{E^*}\right)^{\frac{1}{2}} (St_{def})^{-\frac{9}{8}} < \frac{.172}{St_v}\left(\frac{\tilde{D}}{h_o}\right)^2 \left[1 - \frac{1}{St_v} ln\left(\frac{h_o}{h_a}\right)\right]^{\frac{5}{4}}$$

$$\times \left[\left(\frac{{h_o}^2}{{h_a}^2} - 1\right) + \frac{2h_o}{\delta''}\left(\frac{h_o}{h_a} - 1\right) + \frac{2{h_o}^2}{(\delta'')^2} ln\left(\frac{h_o}{h_a}\right)\right]$$

$$\left[1 - 7.36\left(\frac{Y_d}{E^*}\right)(St_{def})^{-\frac{1}{4}}\left(1 - \frac{1}{St_v} ln\left(\frac{h_o}{h_a}\right)\right)^{-\frac{1}{2}}\right]^2$$



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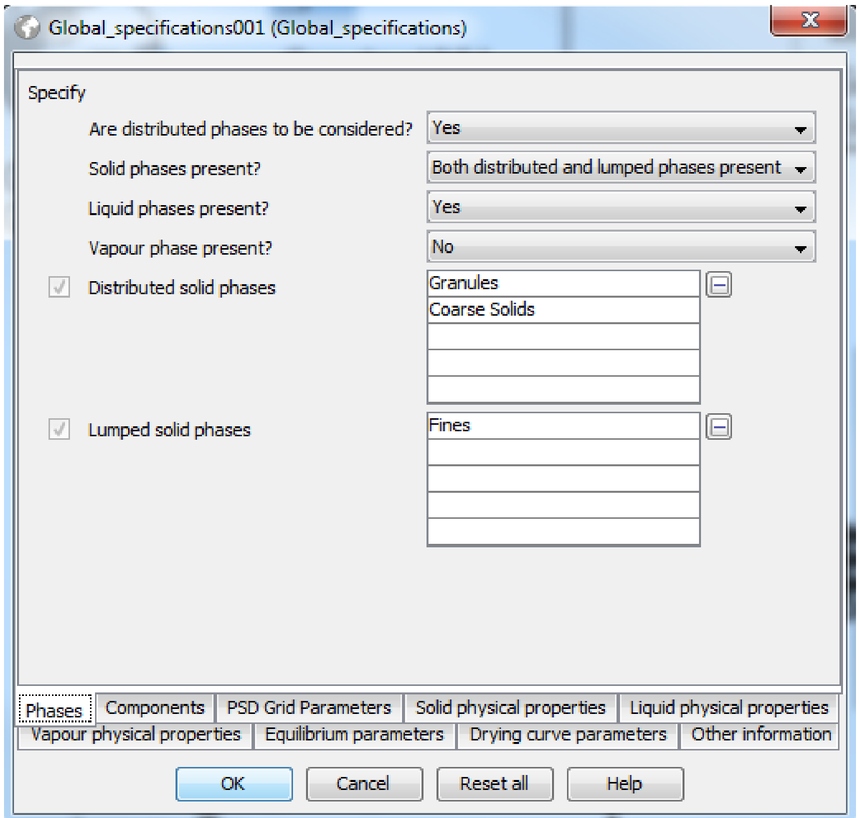
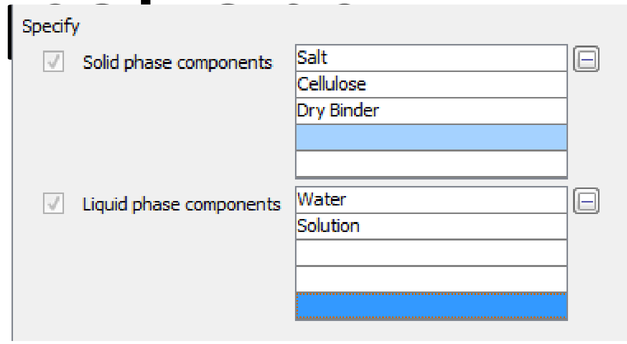
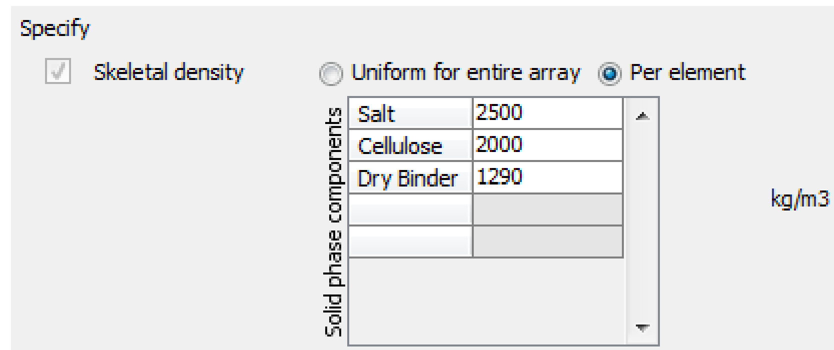
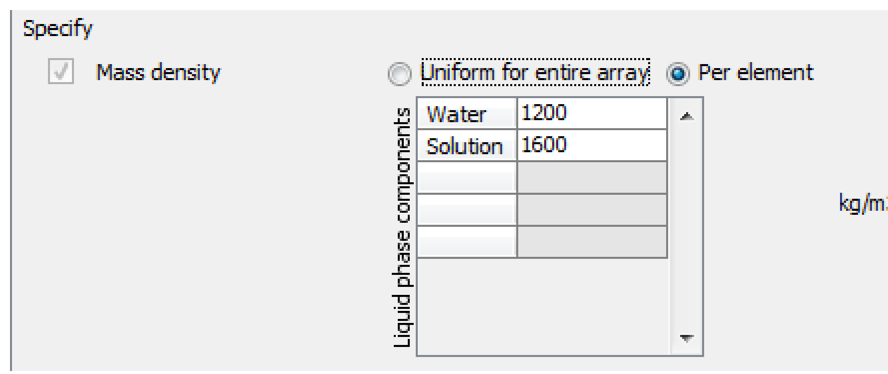
Global Specifications

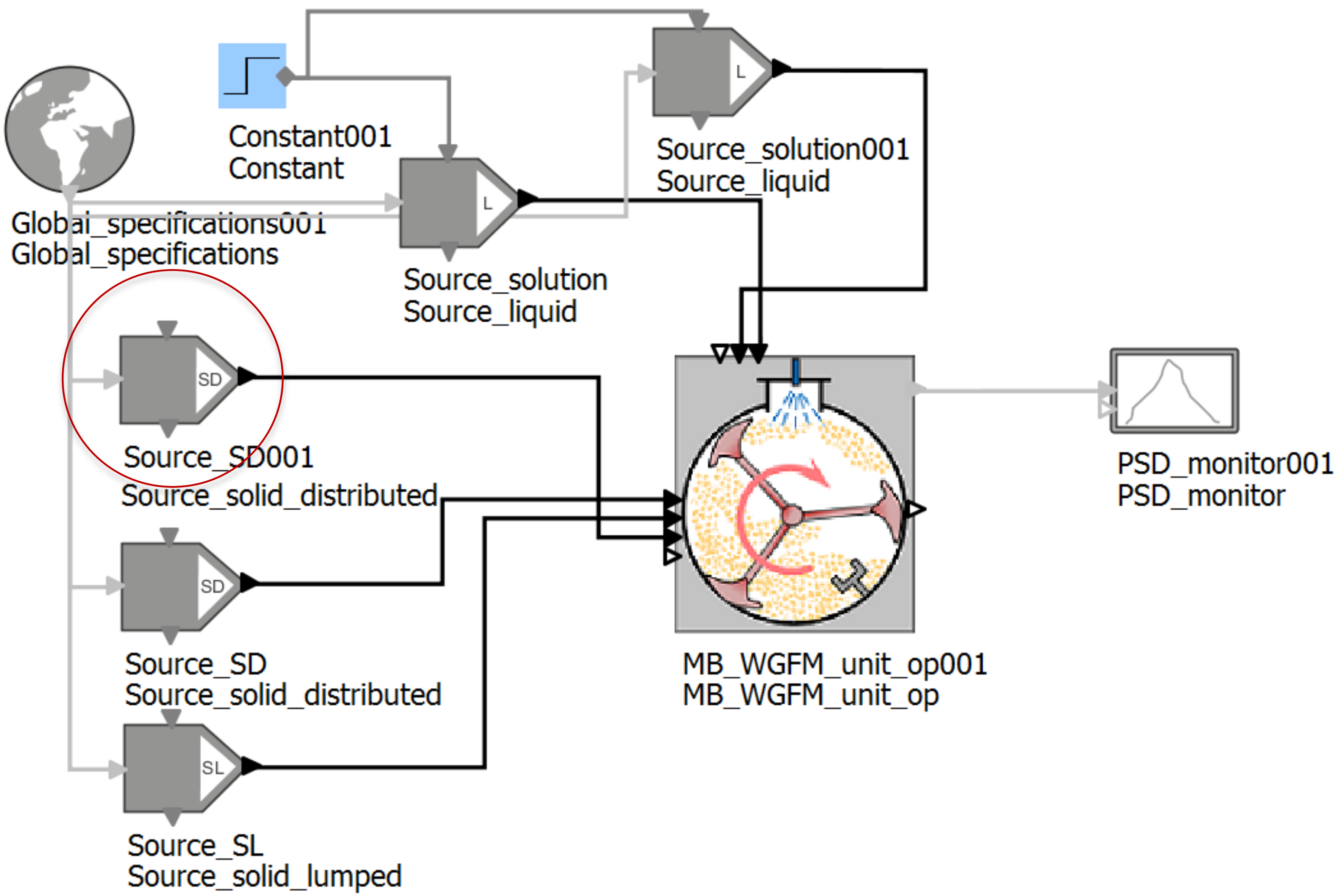


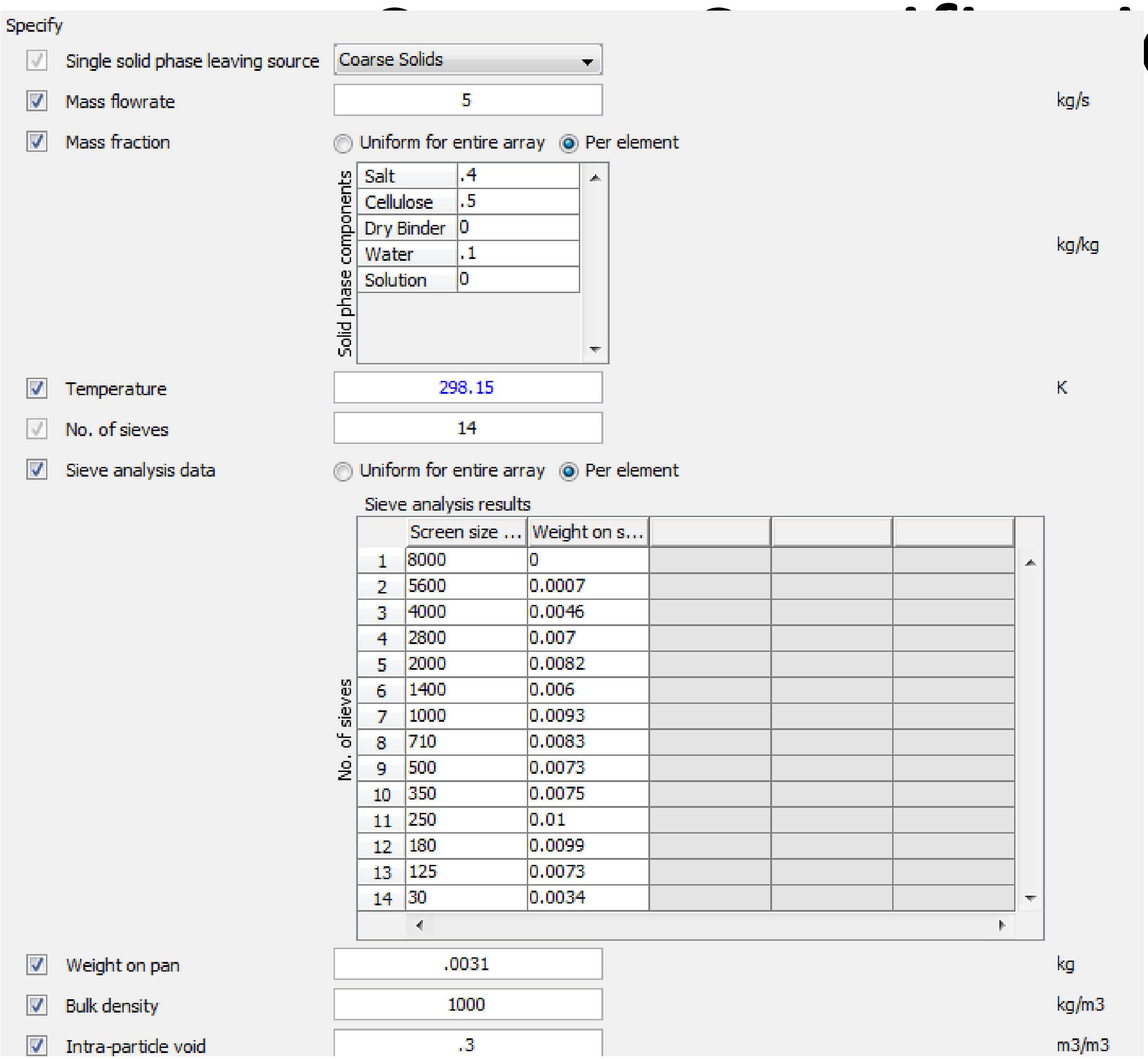






High Shear Wet Granulation Flowsheet





Specify

- Single solid phase leaving source: Coarse Solids
- Mass flowrate: 5 kg/s
- Mass fraction: Uniform for entire array / Per element

Solid phase components (kg/kg)

Salt	.4
Cellulose	.5
Dry Binder	0
Water	.1
Solution	0

- Temperature: 298.15 K
- No. of sieves: 14
- Sieve analysis data: Uniform for entire array / Per element

Sieve analysis results

No. of sieves	Screen size ...	Weight on s...
1	8000	0
2	5600	0.0007
3	4000	0.0046
4	2800	0.007
5	2000	0.0082
6	1400	0.006
7	1000	0.0093
8	710	0.0083
9	500	0.0073
10	350	0.0075
11	250	0.01
12	180	0.0099
13	125	0.0073
14	30	0.0034

- Weight on pan: .0031 kg
- Bulk density: 1000 kg/m3
- Intra-particle void: .3 m3/m3

ons

Input for the composition of a source stream

Input of the size distribution by sieve size

Bed Zone Model

- Overall PB model

$$\frac{\partial V n_b G_g}{\partial v_g} + \frac{\partial V n_b G_s}{\partial v_s}$$

$$= \dot{Q}_{i-b} n_i - \dot{Q}_{b-i} n_b + \dot{Q}_{s-b} n_s - \dot{Q}_{b-s} n_s + V_b \dot{b}_{coal} - V_b \dot{d}_{coal}$$

$$\dot{b}_{coal} = \frac{1}{2} \int_0^{v_s} \int_0^{v_l} \int_0^{v_g} \beta n_b(v_s', v_l', v_g') n(v_s - v_s', v_l - v_l', v_g - v_g') dv_s' dv_l' dv_g'$$

$$\dot{d}_{coal} = \int_0^{\infty} \int_0^{\infty} \int_0^{\infty} \beta n(v_s, v_l, v_g) n(v_s', v_l', v_g') dv_s' dv_l' dv_g'$$