Population Balance Models for Granulation Process Design

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Acknowledgments

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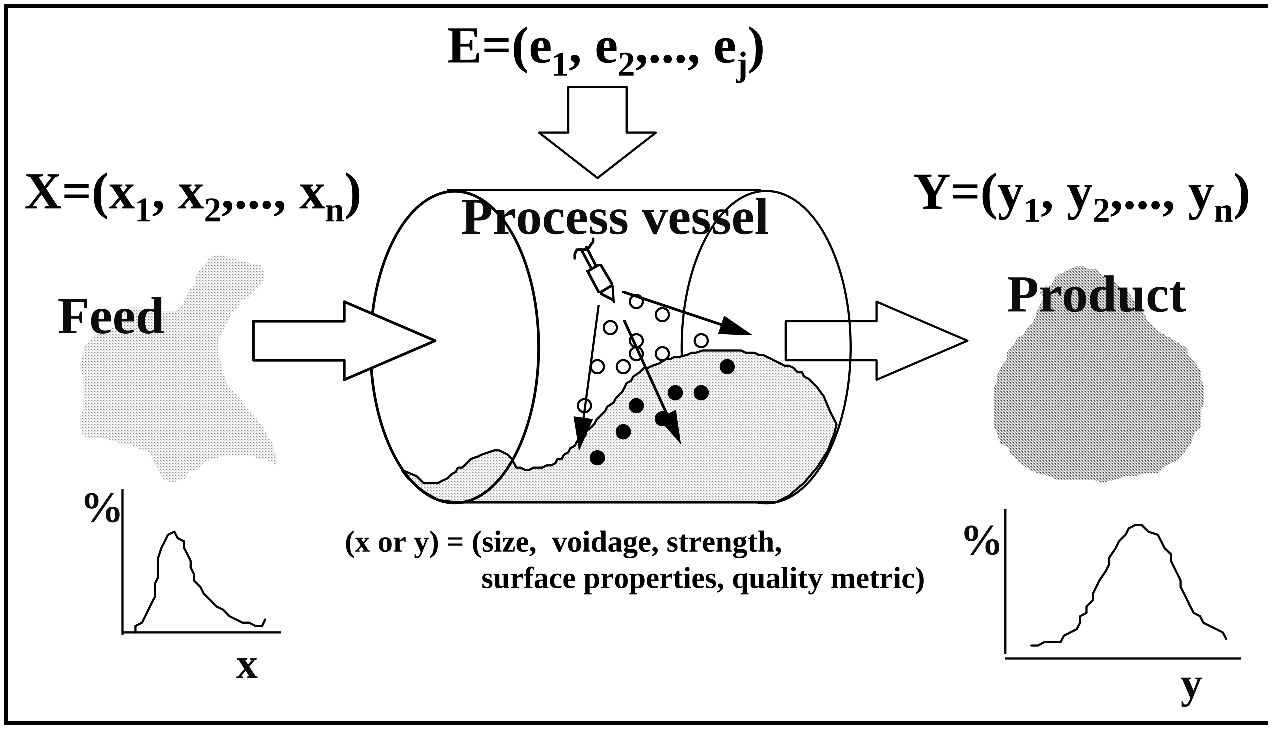
  - Charles Emmanuel
  - Frank Doyle
  - Ian Cameron
  - Fu Yang Wang
  - Carl Wassgren
- Current and former students
  - Anthony Adetayo
  - Lian Liu
  - Simon Iveson
  - Karen Hapgood
  - Hans Wildeboer
  - Ben Freireich
  - Jonathan Poon
  - Rohit Ramchandran

Outline

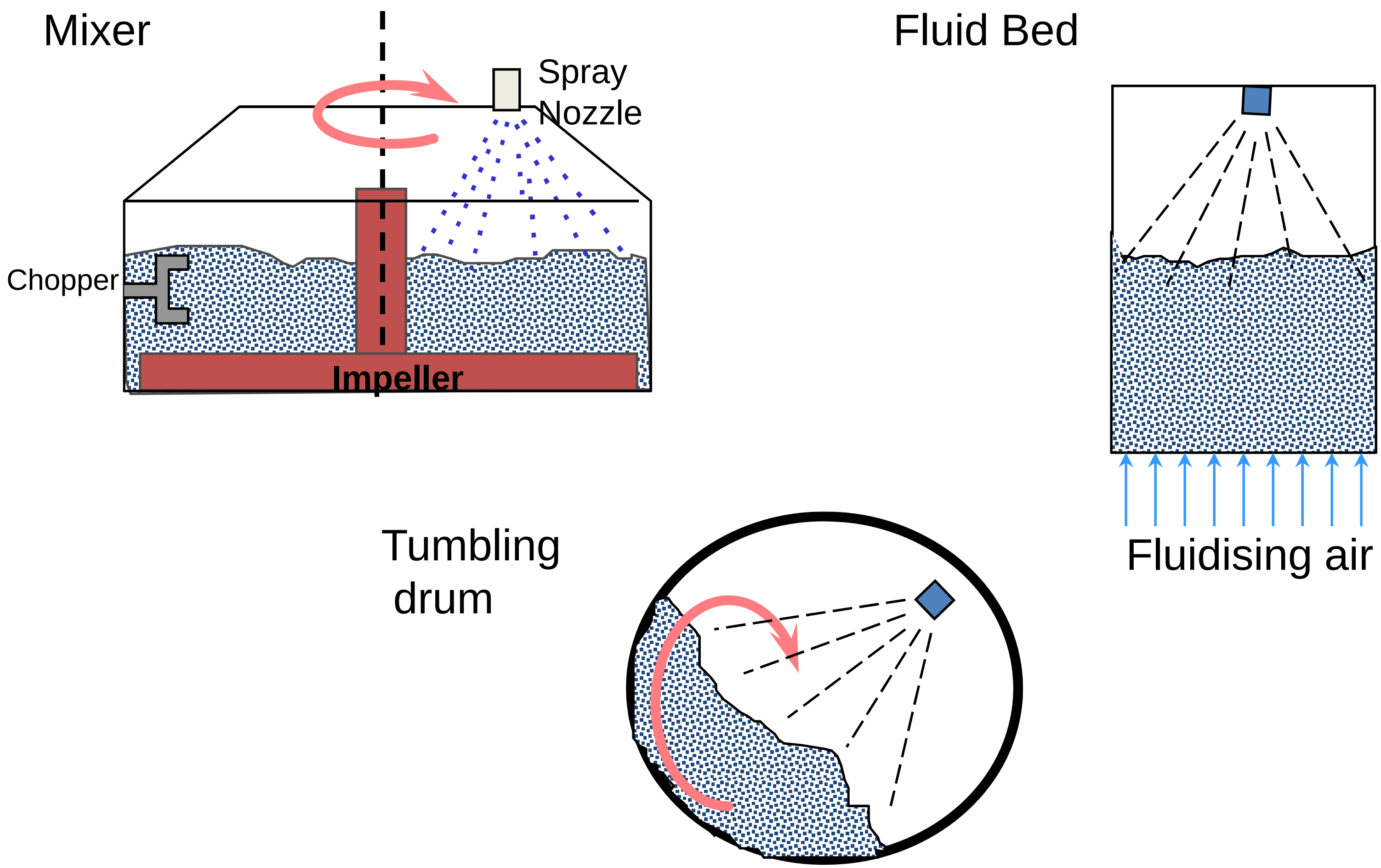
- Introduction to granulation processes
- Modelling coalescence
  - A little bit of history
  - Multidimensional PBM
- Modelling nucleation and layering

Size enlargement by granulation



- liquid binder used to form interparticle bonds
- agitation of “wet mass” to promote liquid binder dispersion and granule growth
- fluid beds, tumbling drums, mixers

Granulation equipment



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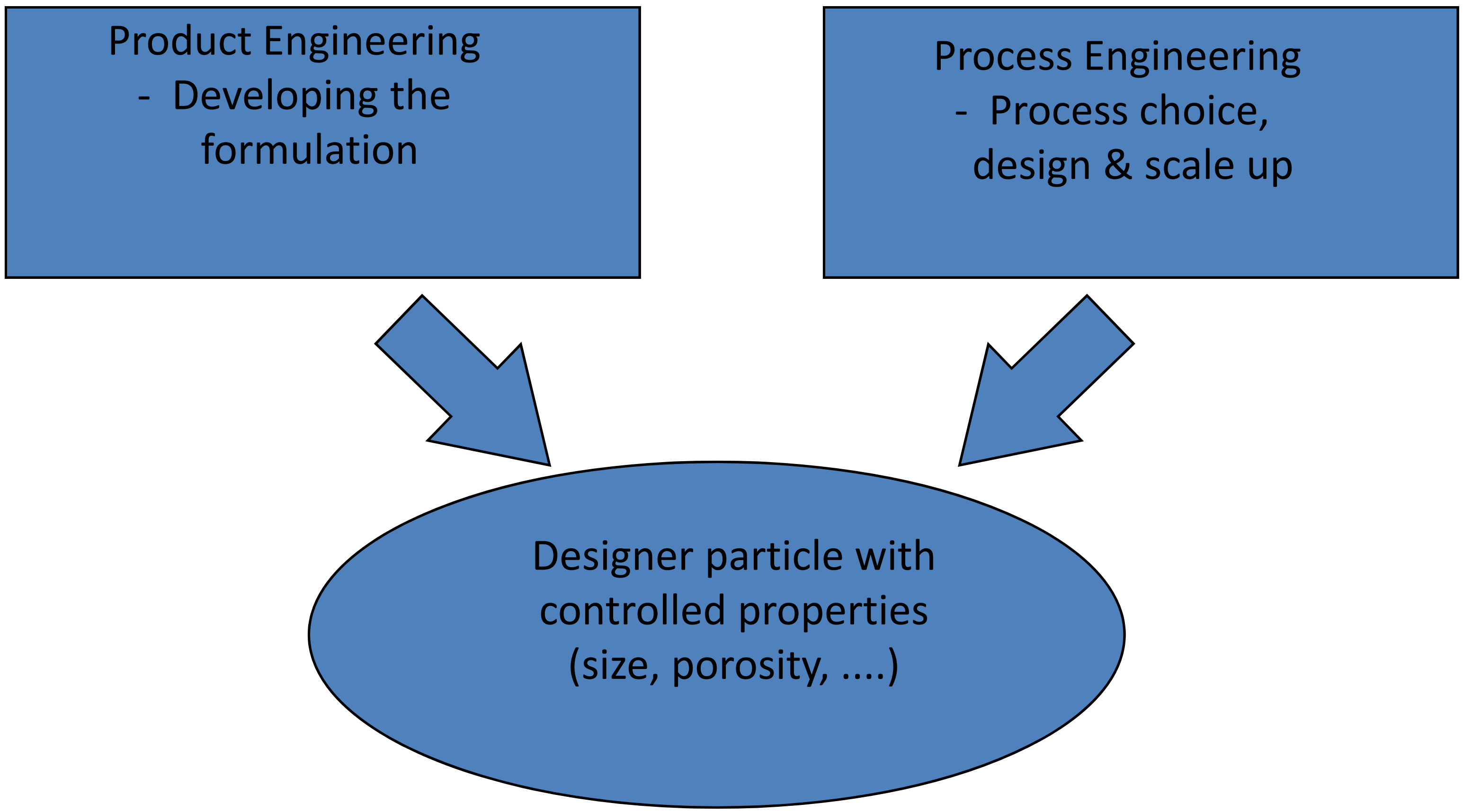
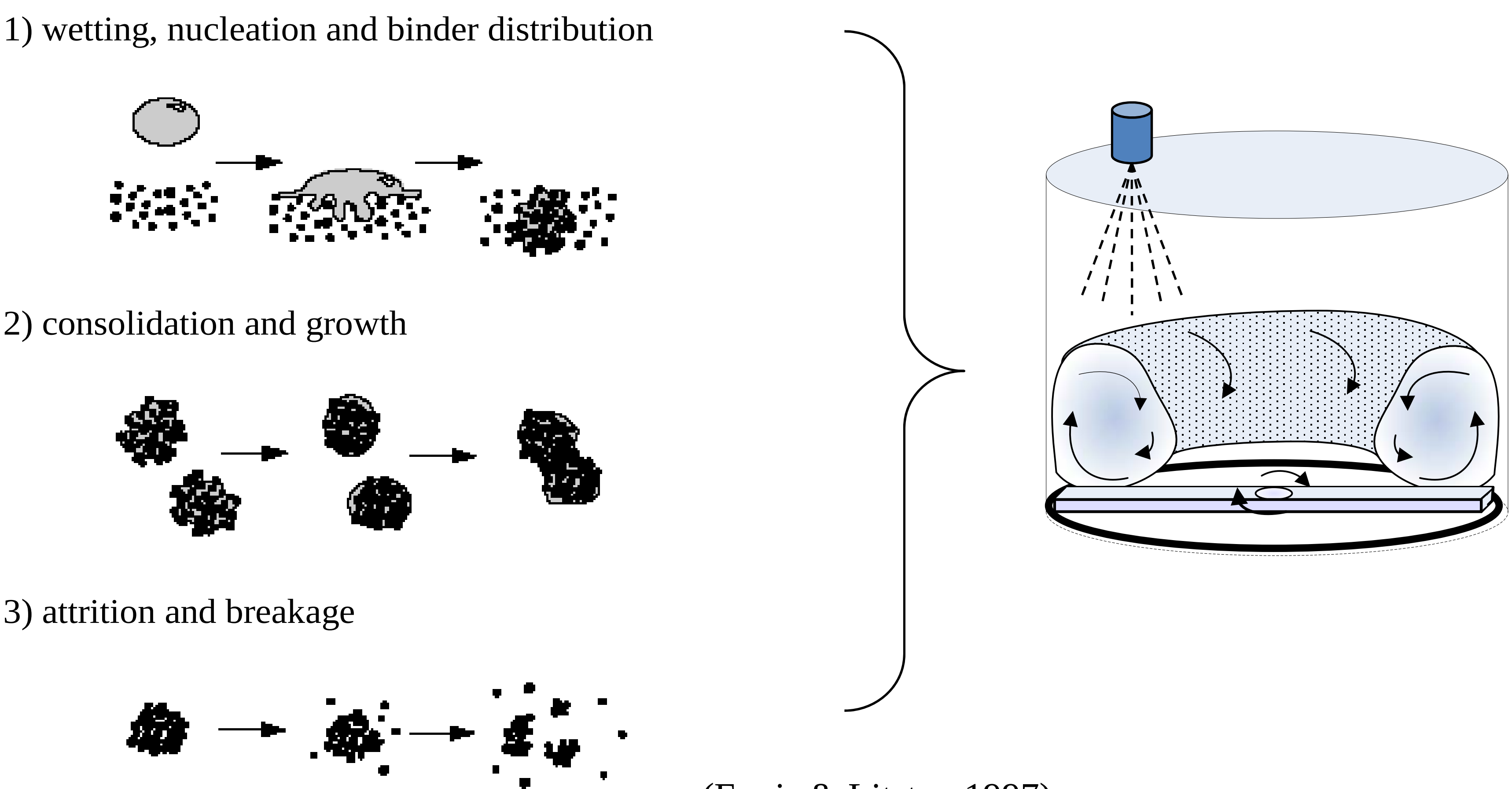
Powder flow in a mixer granulator

Particle Design Principles

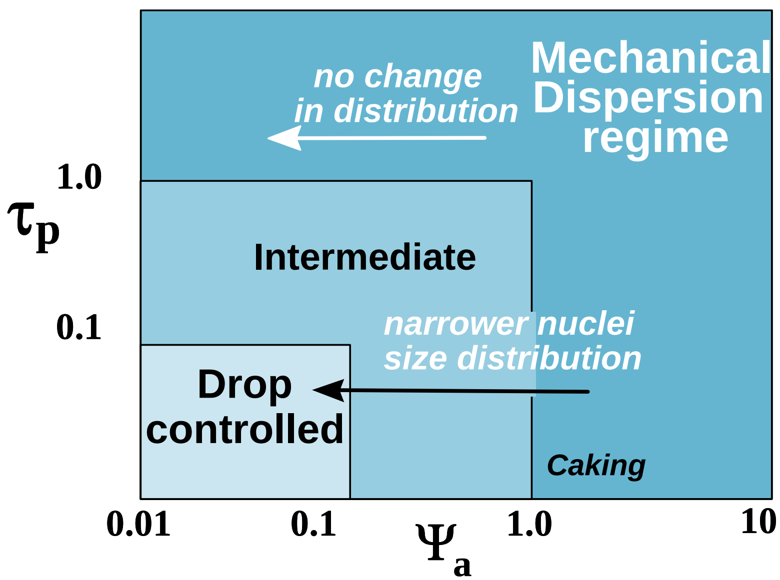


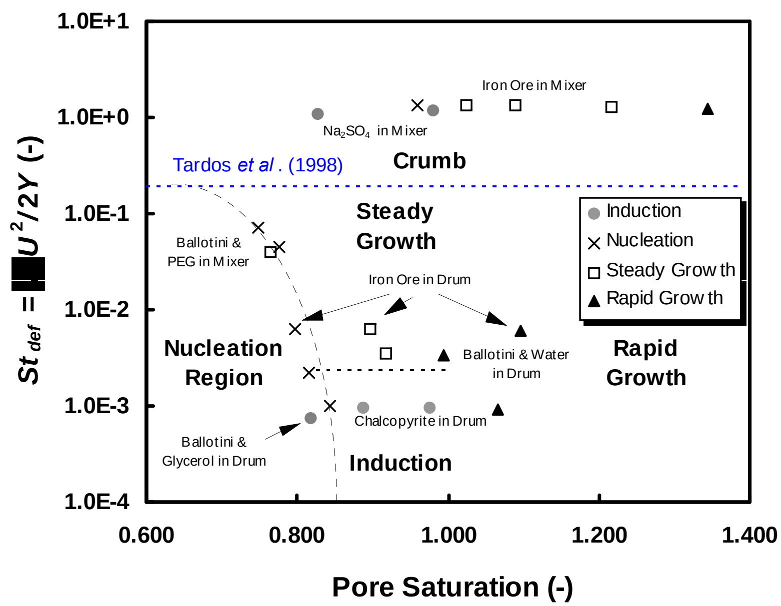
Granulation Rate Processes



(Ennis & Litster, 1997)

Granulation rate processes





Controlling groups are known for nucleation and growth.
Regime maps are available.

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Current status for design

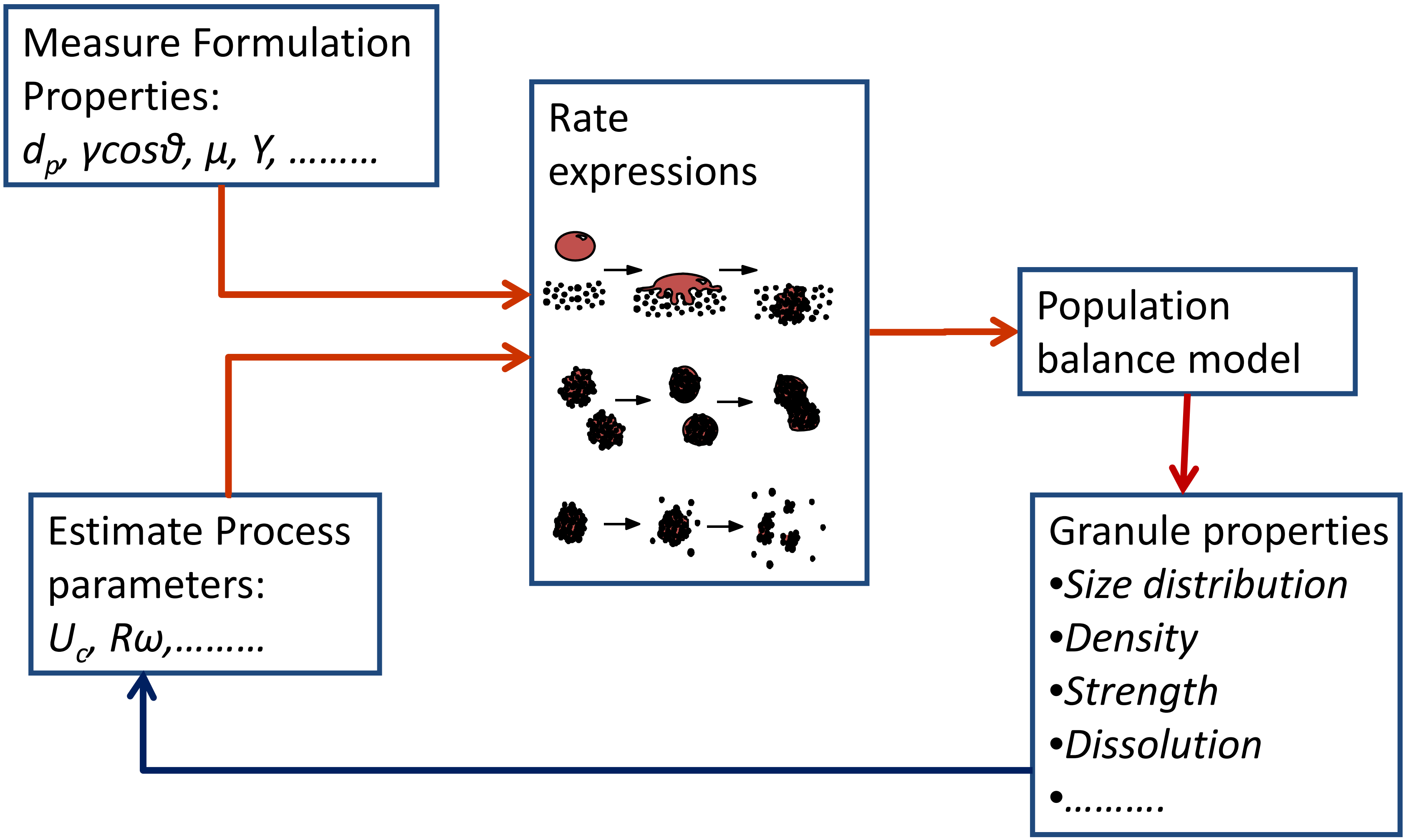
Measure Formulation Properties:
d_p, $\gamma cos\theta$, μ, Y, ...

Estimate Process parameters:
U_c, $R\omega$,.........

Calculate important dimensionless groups:
ψ_α, t_p, St_{def}, S, ...

- Formulation design
- Process choice
- Rational scale up
- Trouble shooting

An Engineering Design Approach



Meso scale modelling of granulation processes

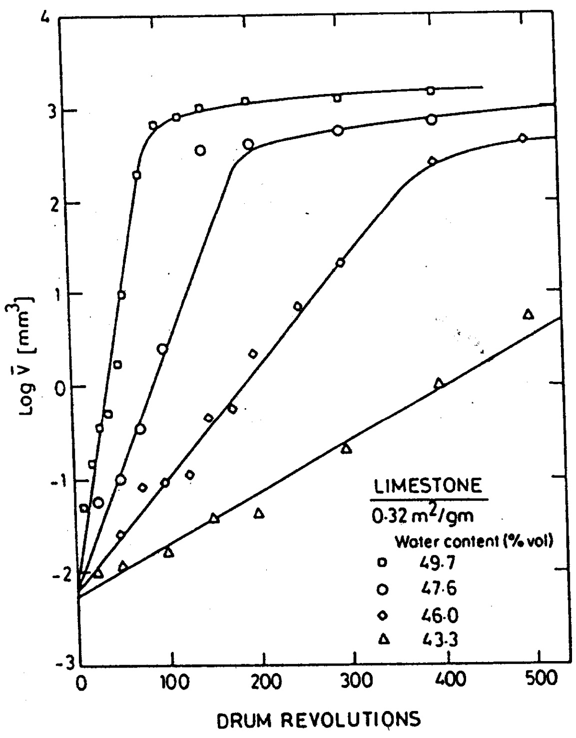
- Population balance models can track track product attributes:

$$\frac{\partial Vn(x,t)}{\partial t} = \dot{Q}_{in}\, n_{in}(x) - \dot{Q}_{ex}\, n_{ex}(x) - V\,\frac{\partial Gn(x,t)}{\partial x} + V\,\dot{b}(x) - V\,\dot{d}(x)$$

$$\Downarrow$$

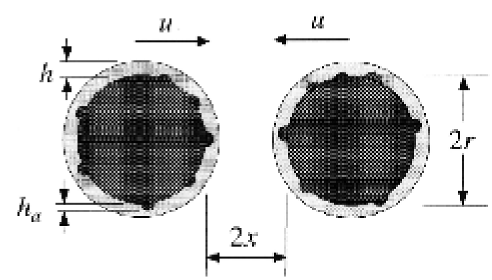
$$\frac{\partial Vn(v,t)}{\partial t} = \dot{Q}_{in} n_{in}(v) - \dot{Q}_{ex} n_{ex}(v) - V\,\frac{\partial (G^{*} - A^{*})n(v,t)}{\partial v}$$
$$+V(\dot{b}(v)_{nuc} + \dot{b}(v)_{coal} + \dot{b}(v)_{br} - \dot{d}(v)_{coal} - \dot{d}(v)_{br})$$

A little bit of history



Early work by Kapur, Sastry and Fuerstenau focused on coalescence

Ennis *et al*. (1991) first to focus on effect of granule properties



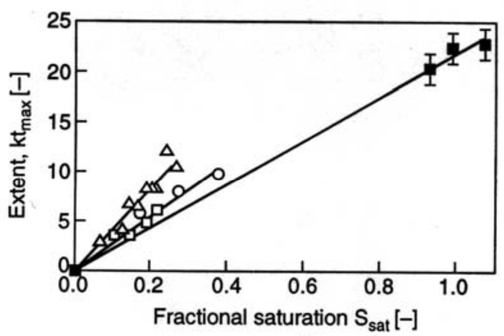
$$St_v < St^* \Rightarrow \text{granules will coalesce}$$

$$St_v = \frac{4\rho_g U_c d_g}{9\mu}$$

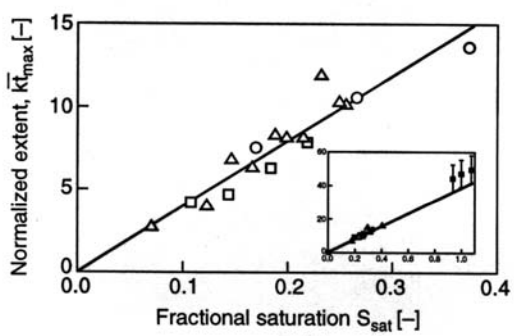
$$St^* = \left(1 + \frac{1}{e_r}\right) \ln\left(\frac{h_b}{h_a}\right)$$

A little bit of history

Adetayo, A.A., J.D. Litster, S.E. Pratsinis and B.J. Ennis, *Powder Technol.*, **82,** 37 (1995)



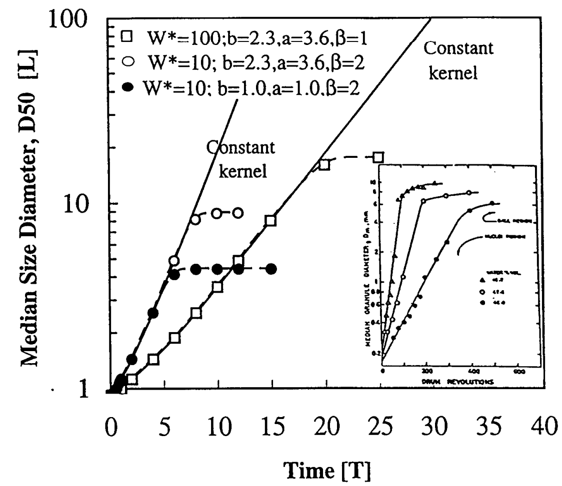
(a)

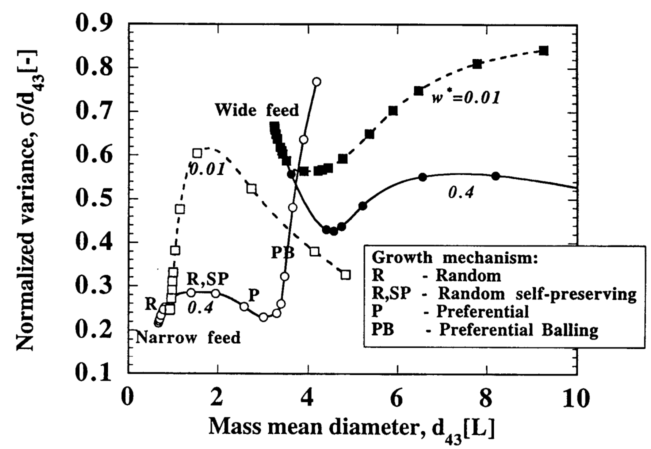


(b)

$$kt_{max} = 6\ln\left(\frac{St*9\mu}{8\rho_g u_c d_0}\right) \propto \ln\left(\frac{\mu}{\rho_g u_c d_0}\right)$$

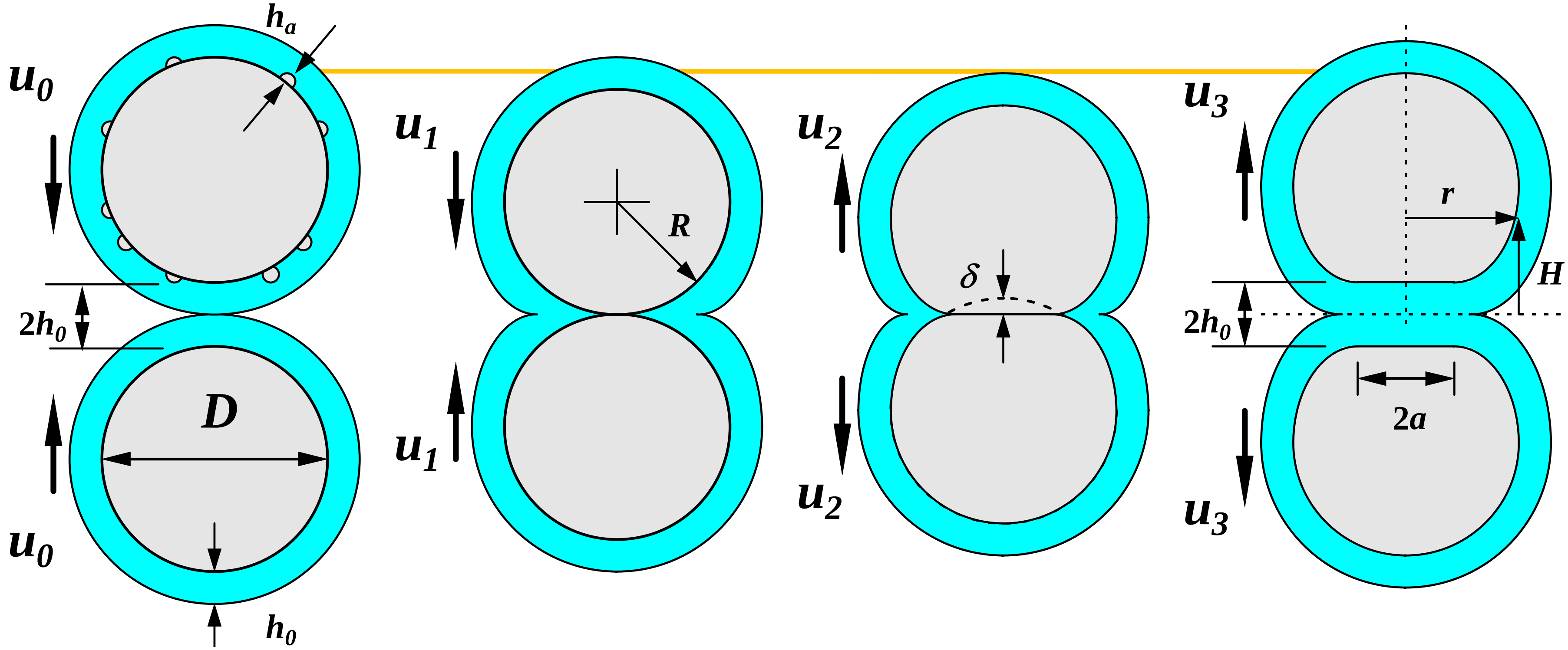
Adetayo and Ennis, 1998





$$\beta(u,v) = \begin{cases} k_o, w < w^* \\ 0, w > w^* \end{cases} \quad where \quad w = \frac{(uv)^a}{(u+v)^b}$$

Modelling Coalescence



Type I:

$$St_v < \ln\left(\frac{h_0}{h_a}\right)$$

Type II:

$$\left(\frac{Y_d}{E^*}\right)^{1/2}\left(St_{def}\right)^{-9/8} < \frac{0.172}{St_{vis}}\left(\frac{\tilde{D}}{h_0}\right)^2\left[1 - St_{vis}\ln\left(\frac{h_0}{h_a}\right)\right]^{5/4}\left(\frac{h_0^2}{h_a^2} - 1\right)$$

Liu et al., *AIChE J*, **46**, 529-539 (2000)

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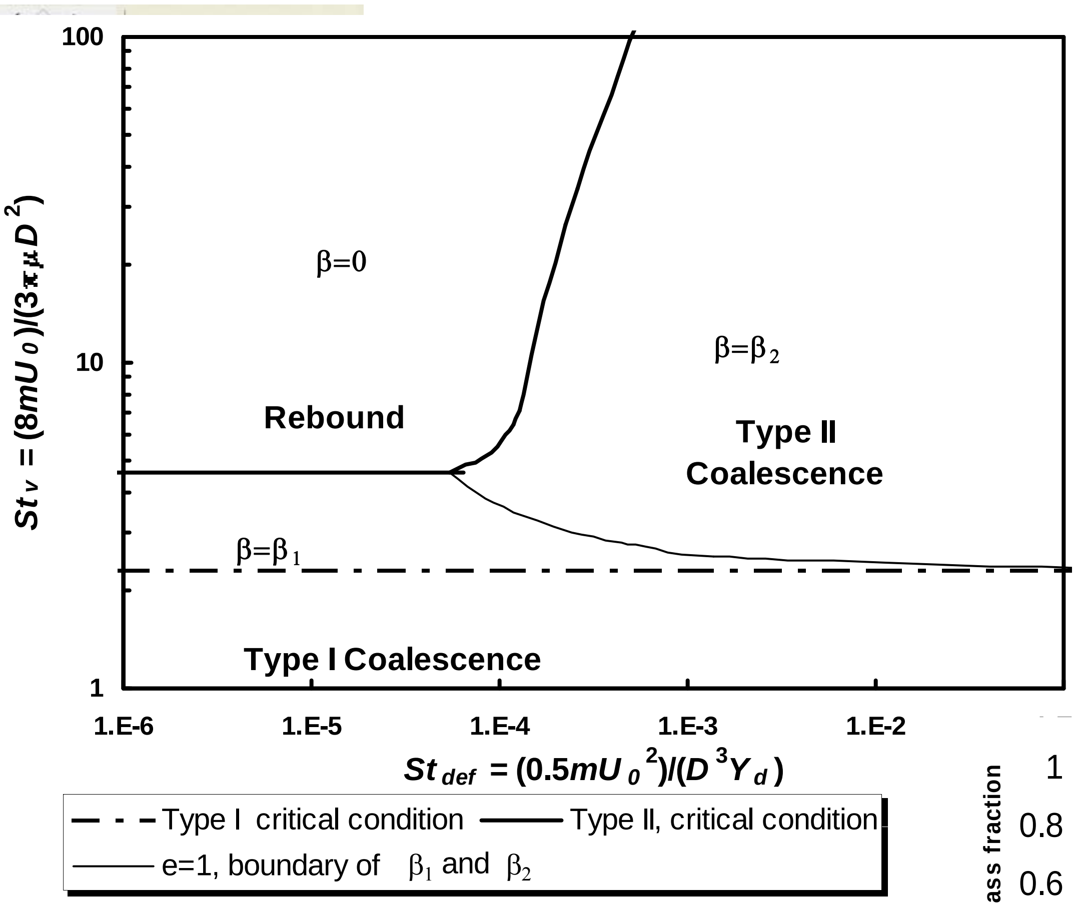
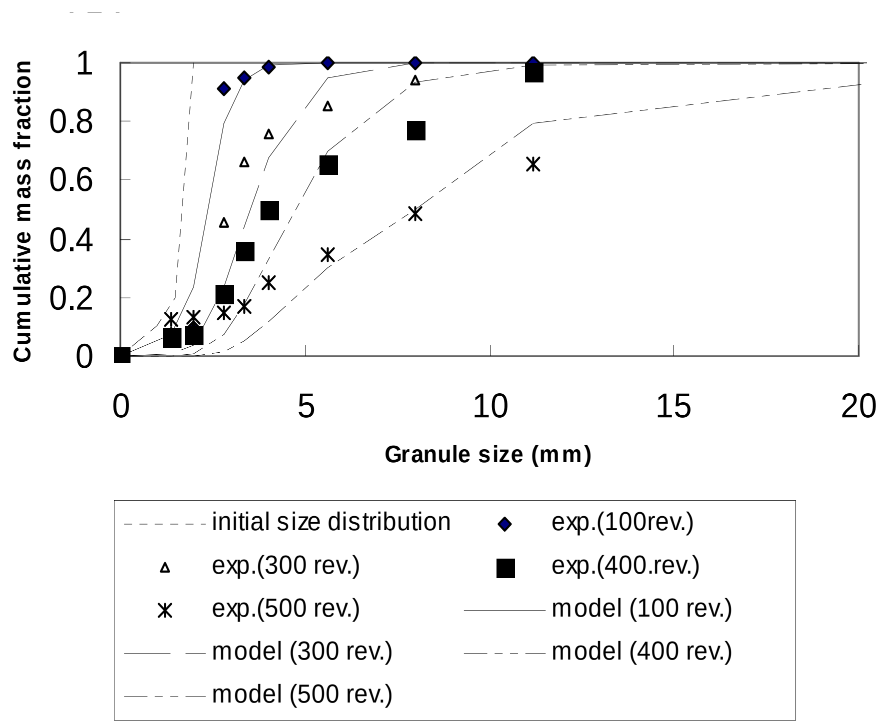




Liu & Litster, 2002

Multidimensional PB models

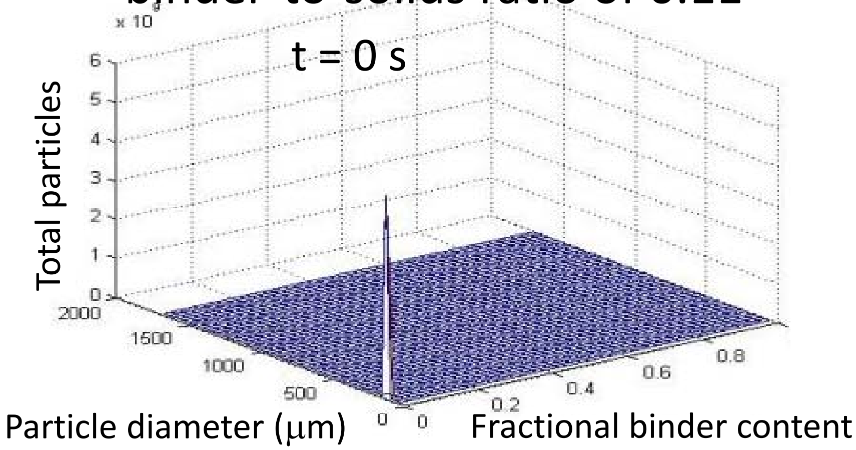
- Tracks the distribution n(s,l,g)

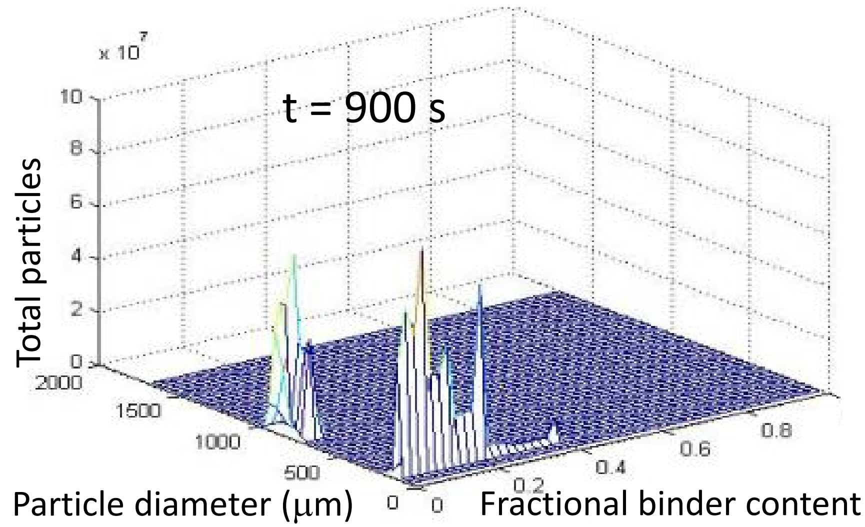
$$\frac{\partial Vn(s,l,g,t)}{\partial t}+\frac{\partial VG_s n(s,l,g,t)}{\partial s}+\frac{\partial VG_l n(s,l,g,t)}{\partial l}+\frac{\partial VG_g n(s,l,g,t)}{\partial g}$$
$$=\dot{Q}_{in}n_{in}(s,l,g,t)-\dot{Q}_{ex}n_{ex}(s,l,g,t)+V(\dot{b}(s,l,g,t)_{nuc}+\dot{b}(s,l,g,t)_{coal}$$
$$+\dot{b}(s,l,g,t)_{br}-\dot{d}(s,l,g,t)_{coal}-\dot{d}(s,l,g,t)_{br})$$

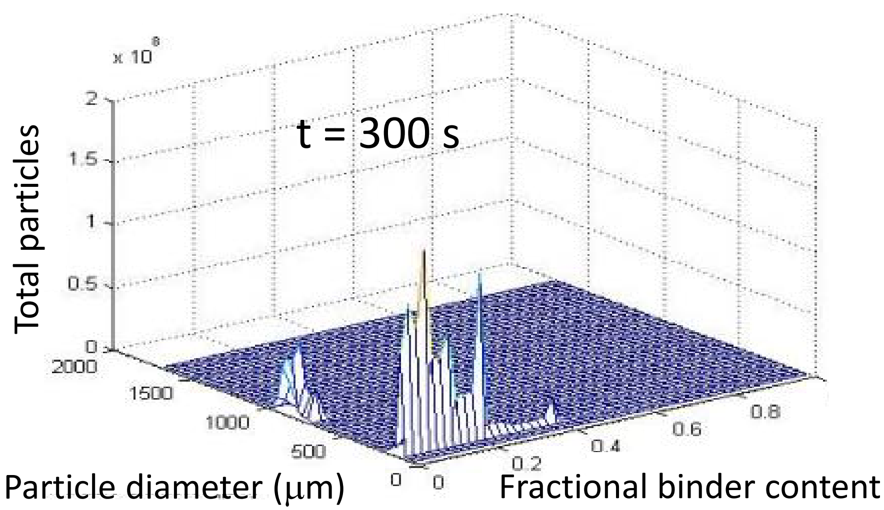
- Challenge for efficient numerical solution
- Challenge for experimental validation

Evolution of Two-Dimensional Distribution

- Simulation result
  - binder-to-solids ratio of 0.11







Poon et al., *Chem Eng Sci*, **63**, 1315-1329 (2008)

Ramchandran et al., (2008)

Particulate Systems (C-SOPS)

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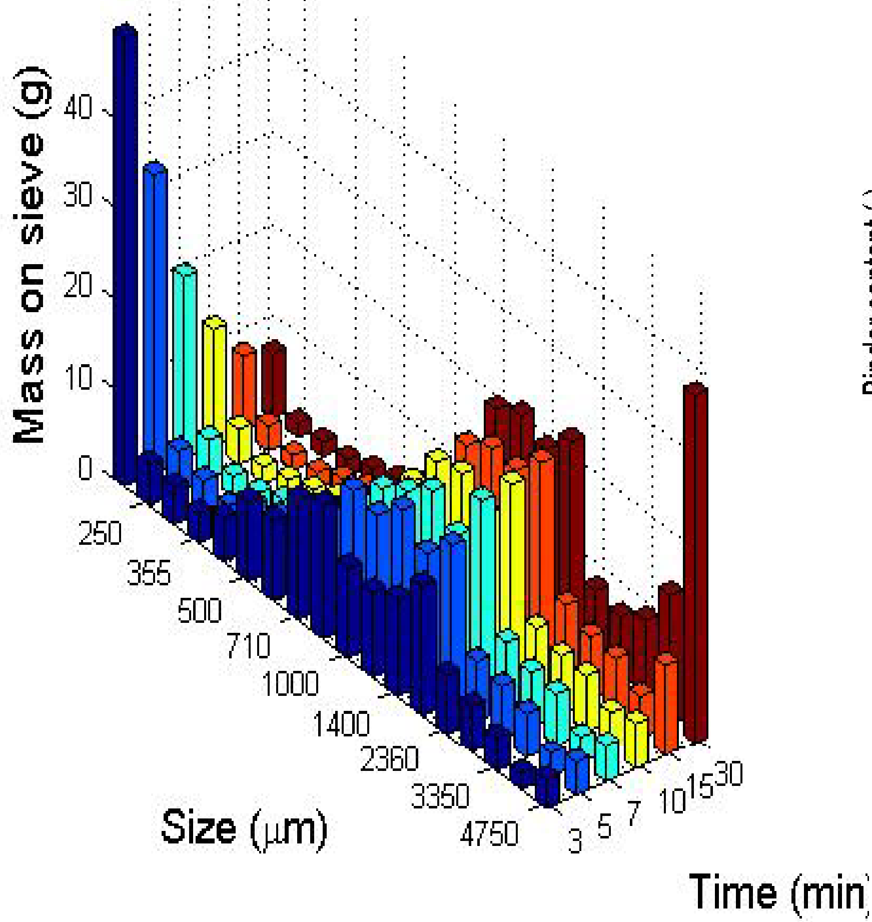
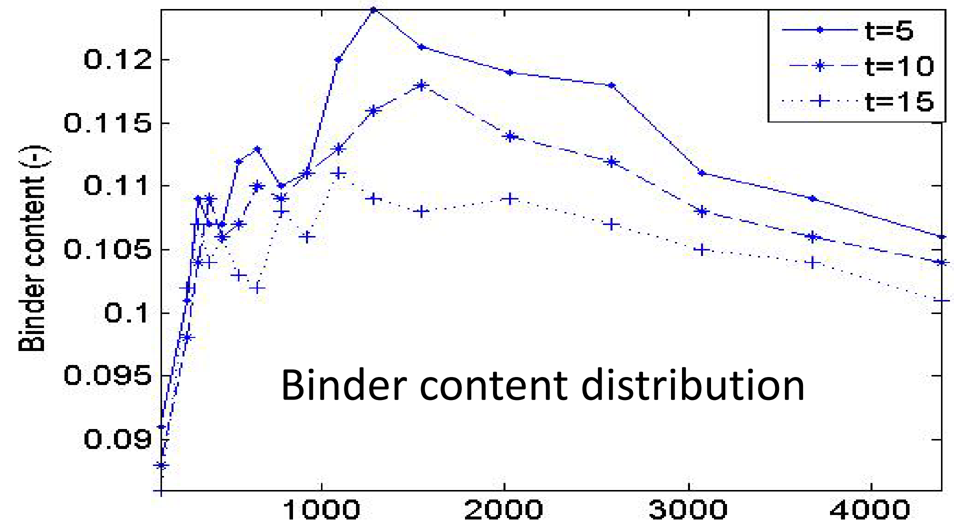
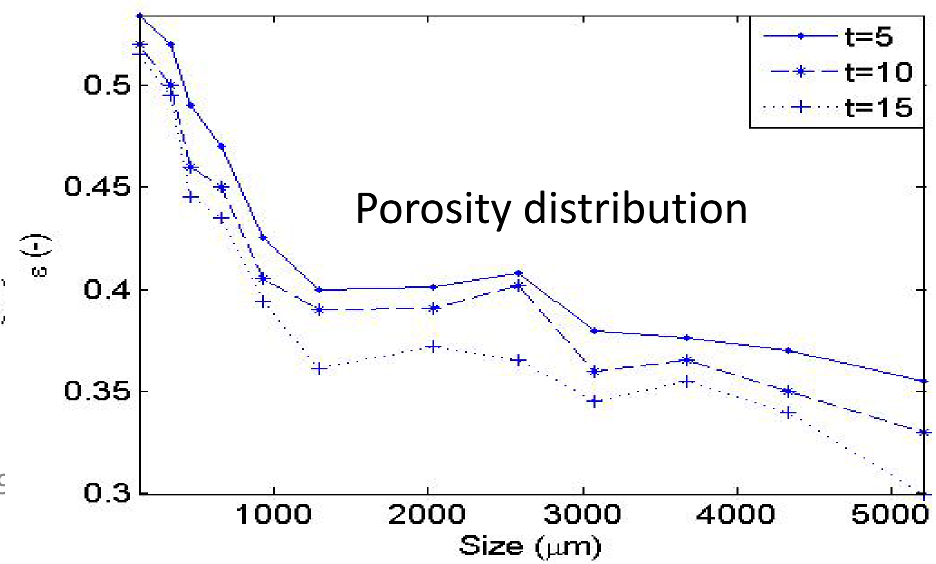
Batch Granulation Results

- clear evidence of multi-dimensional heterogeneity



Granule size distribution

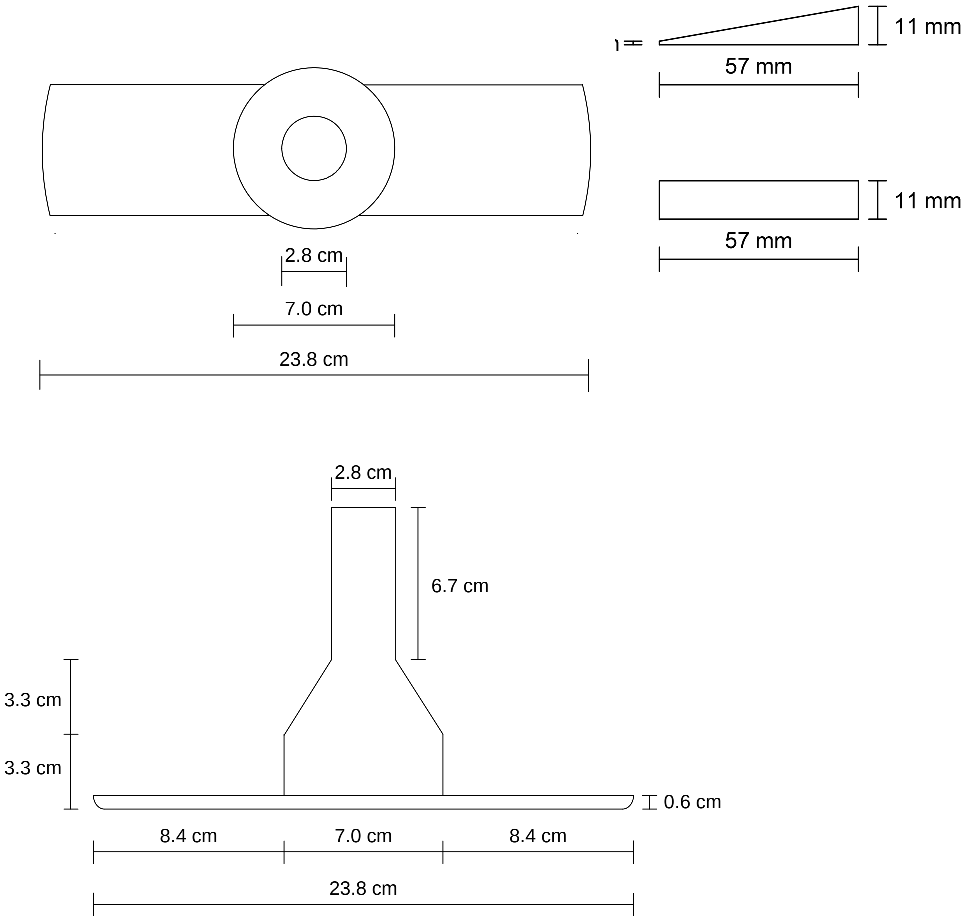




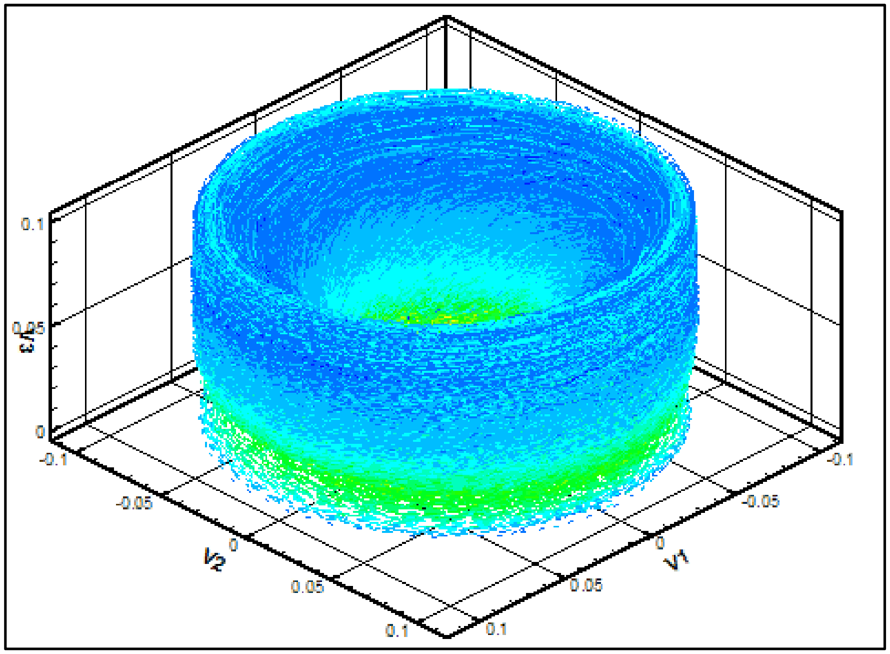
binder-to-solid ratio = 0.125
drum load = 1.75 kg

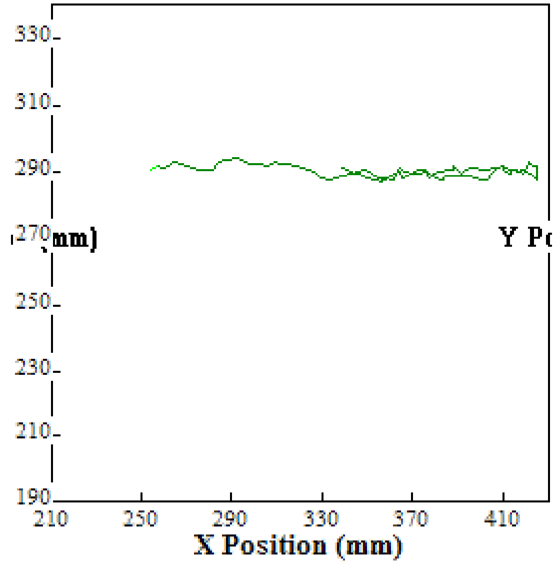
- Multidimensional PB approaches:
  - allow incorporation of more physics in the rate expressions
  - Track property distributions of interest to us
- But powder flow and particle interactions still represented in a crude, lumped parameter format

The impact of mixing and powder flow

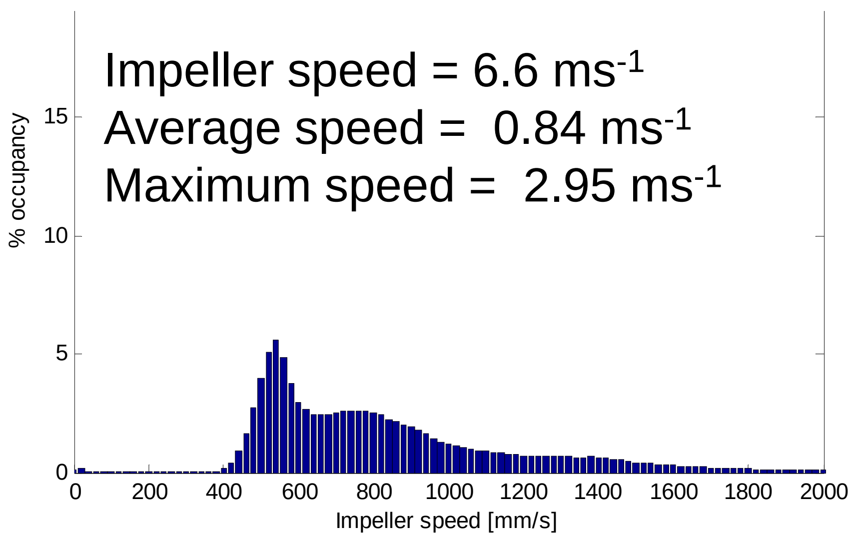


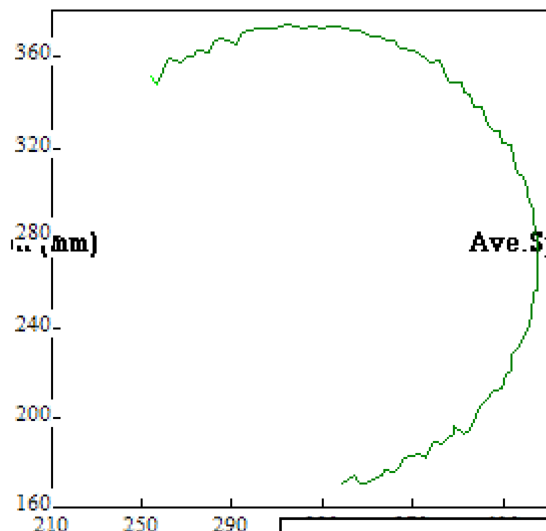
PEPT results (600 rpm)





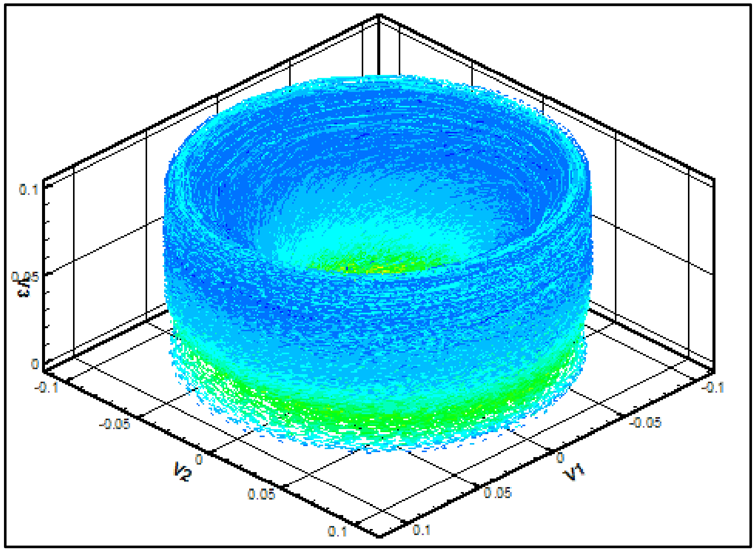
Impeller speed = 6.6 ms^{-1}
Average speed = 0.84 ms^{-1}
Maximum speed = 2.95 ms^{-1}

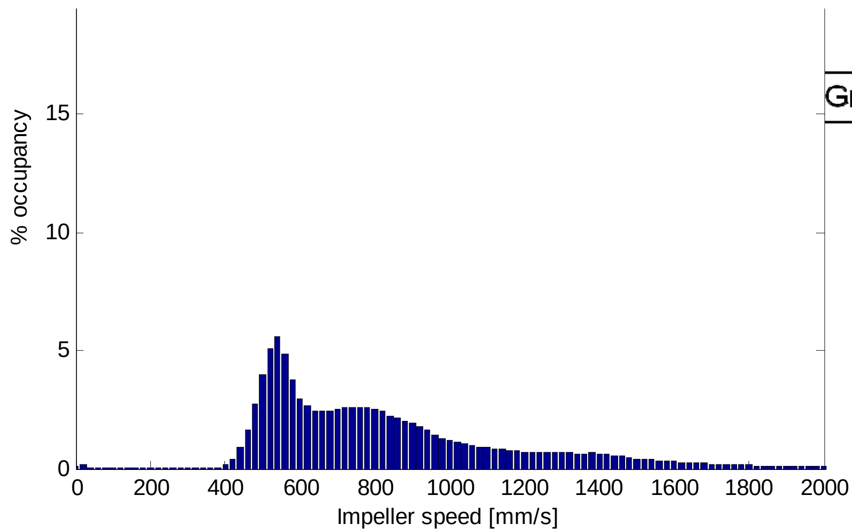


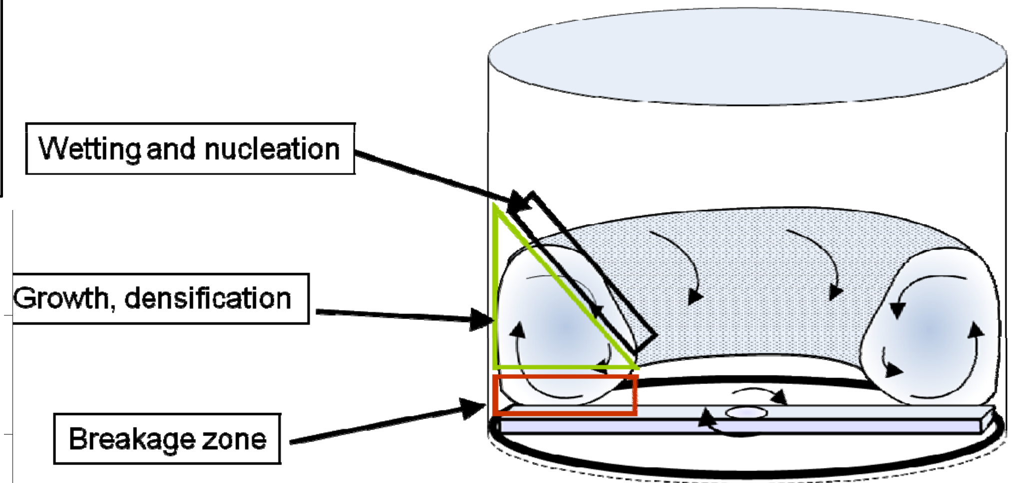


Anh Tran, PhD (2008)

Regions for granulation rate processes







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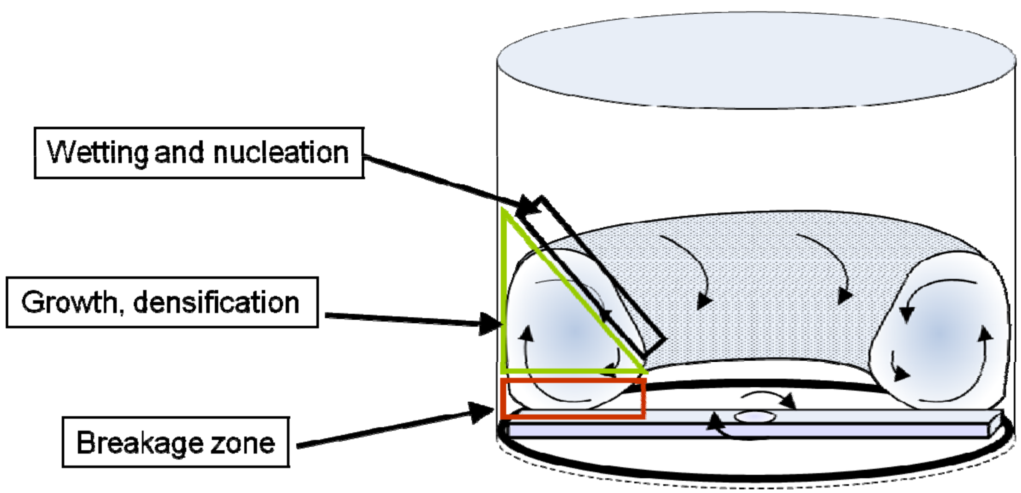
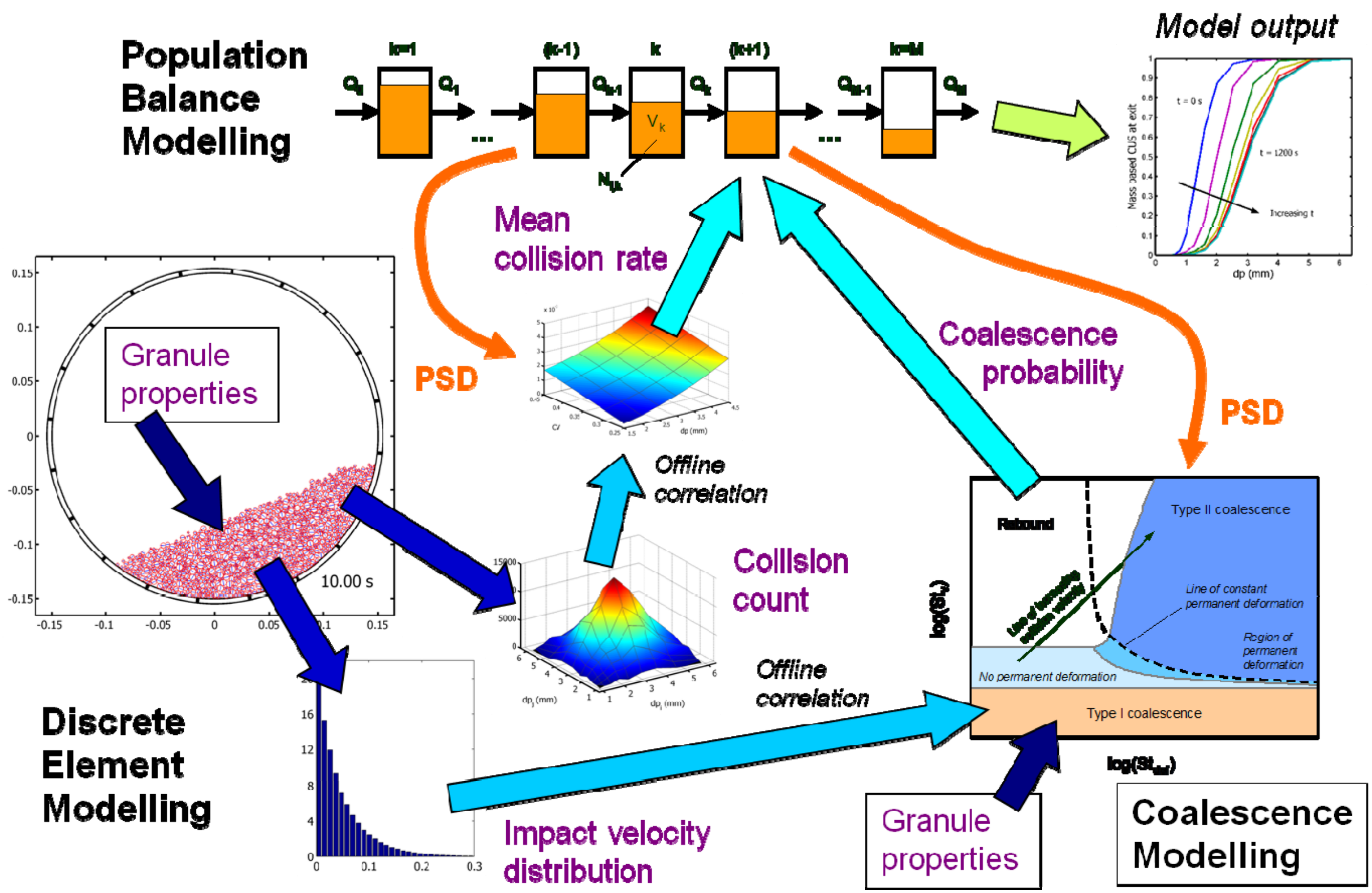
PEPT derived powder flow data

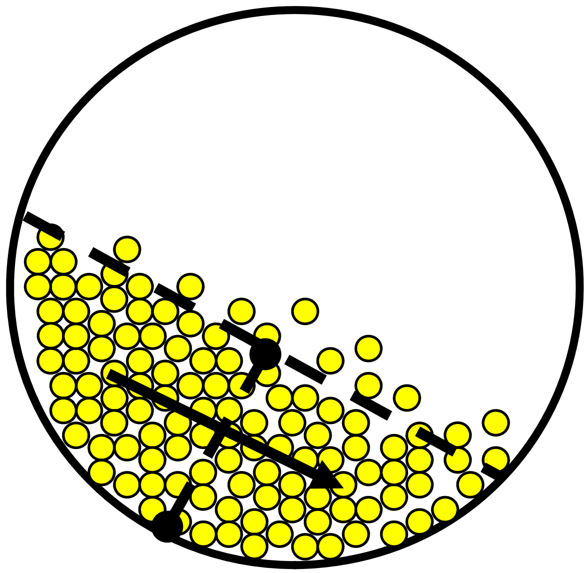
- Data required for models
  - Occupancy ✓
  - Velocity field ✓
  - Fluxes ✓
  - Stresses ✗
  - Collision frequencies ✗
  - Predictive ✗

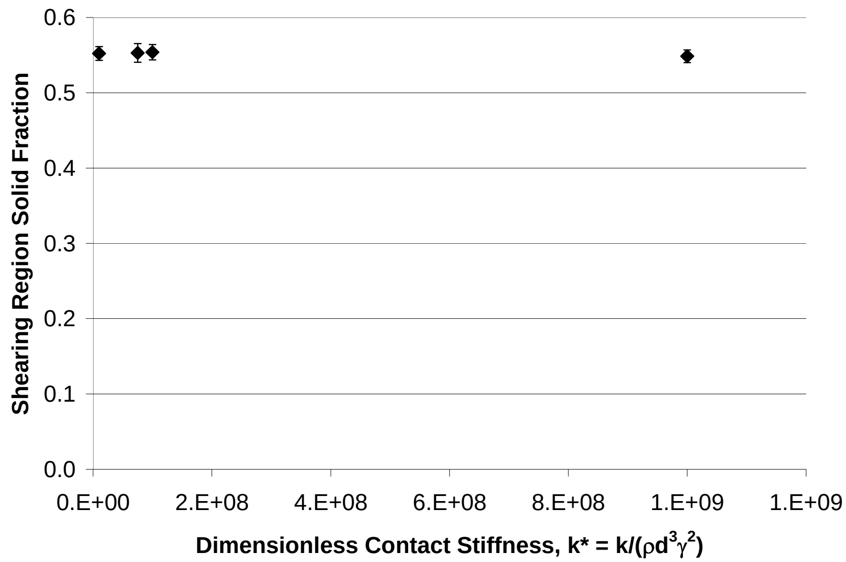


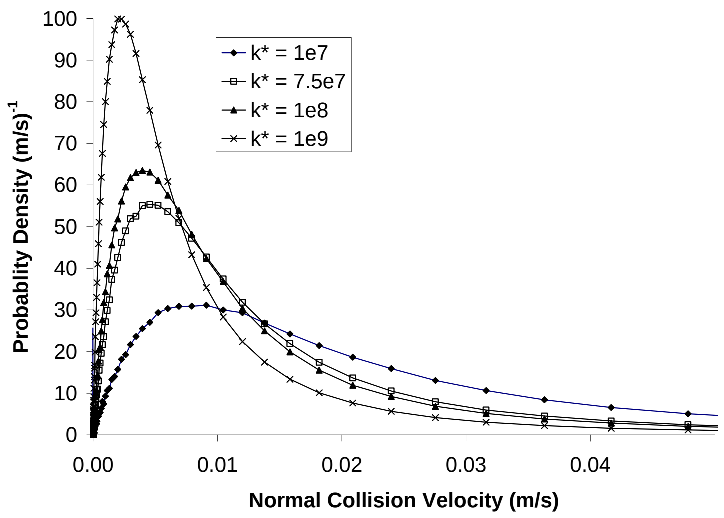
Multiscale modelling



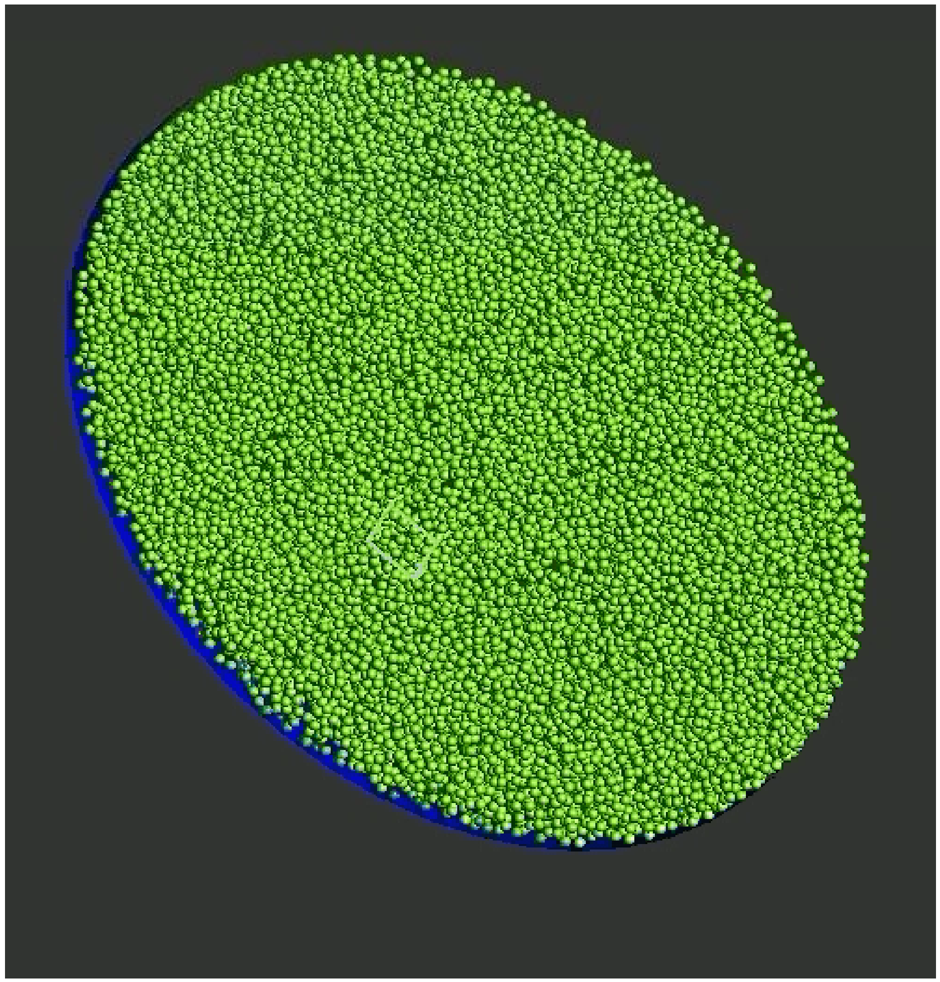
Parameter sensitivity

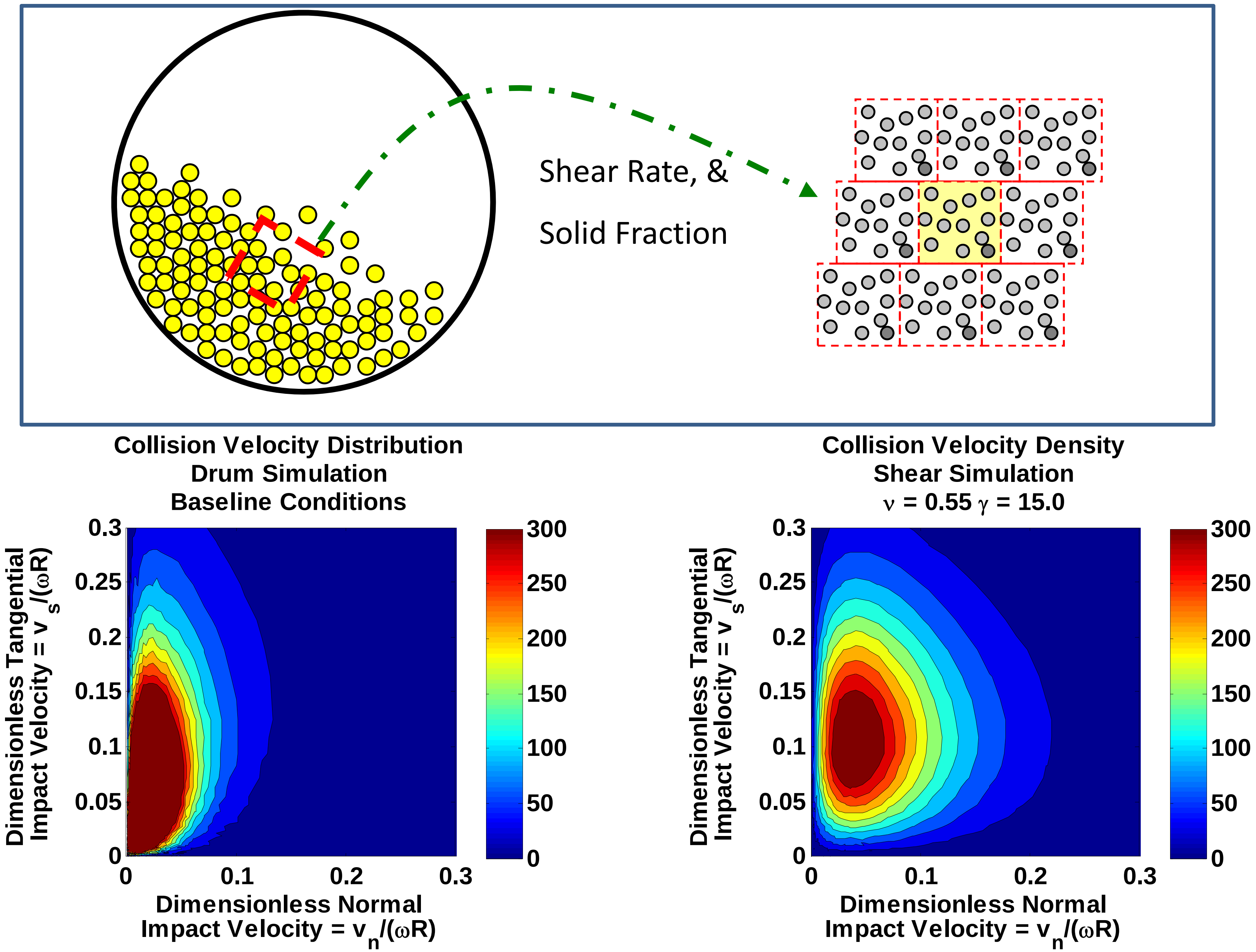




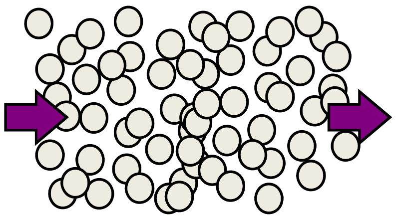


Ben Freireich (2008)




Shear Rate, &
Solid Fraction
Collision Velocity Distribution
Drum Simulation
Baseline Conditions
Dimensionless Tangential
Impact Velocity = v_s/(ωR)
Dimensionless Normal
Impact Velocity = v_n/(ωR)
Collision Velocity Density
Shear Simulation
ν = 0.55 γ = 15.0
Dimensionless Tangential
Impact Velocity = v_s/(ωR)
Dimensionless Normal
Impact Velocity = v_n/(ωR)

Dimensionless Spray Flux



- Area of drops compared to area sprayed, at a flowrate *V* and a powder flux *A and drop size* d_d

$$\Psi_a = \frac{3\dot{V}}{2\dot{A}d_d}$$

- Low spray flux means a narrow nuclei distribution as most drops are well separated.
- Fraction of nuclei formed by 2 or more drops

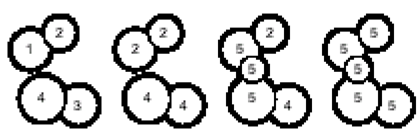
$$f_{multi} = 1 - \exp(-4\psi_a)$$

Modelling Nucleation

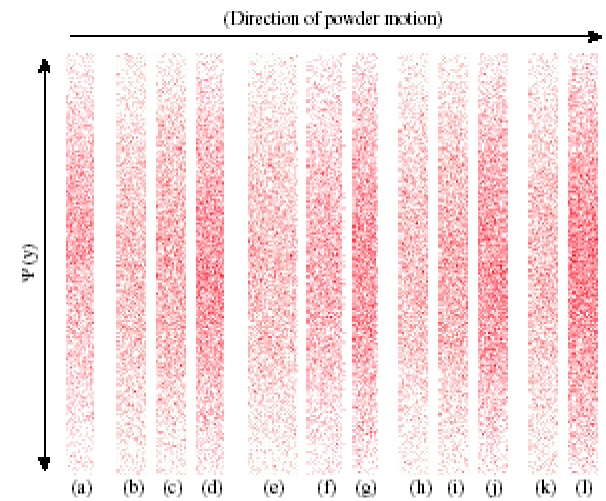
- Purely drop controlled:

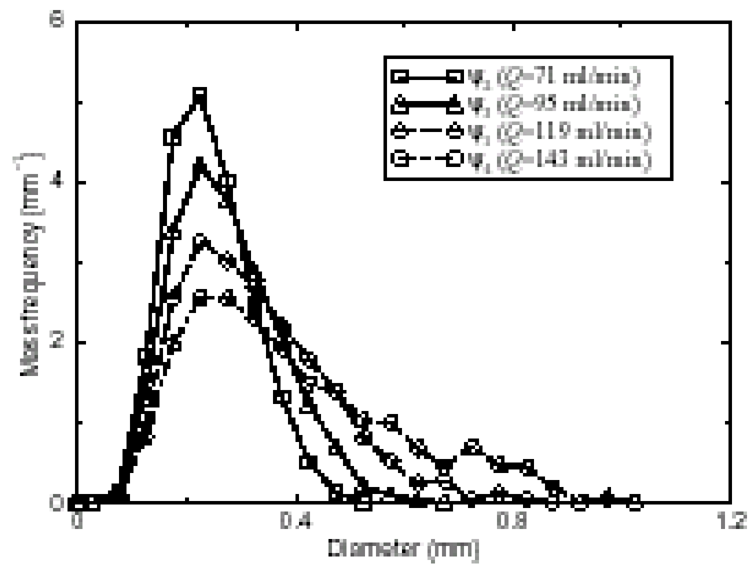
$$\dot{b}(v)_{nuc} = S n_s (s \varepsilon v)$$

- Extension to allow for non-uniform (real) sprays and some drop overlap using Monte Carlo simulation



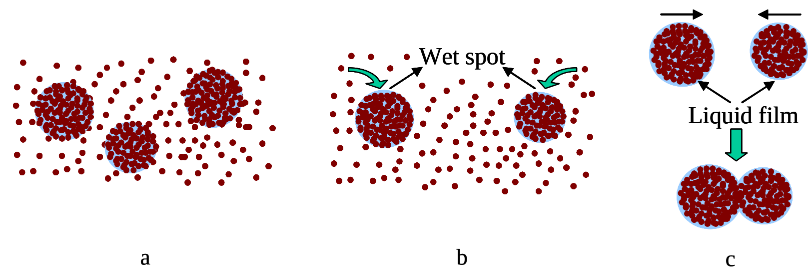
Wildeboer, 2002





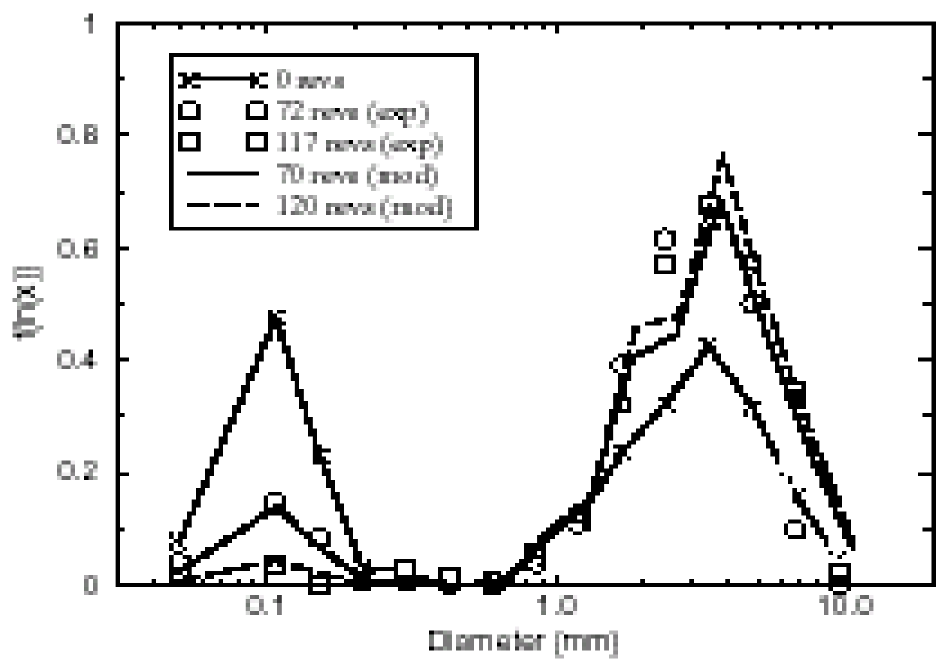
Modelling consolidation and layering

- Consolidating granules pick up fines (layering) as liquid is squeezed to the surface
- When fines are used up, coalescence may start



$$G(v) = -k_{con} v \frac{\varepsilon - \varepsilon_{\min}}{\varepsilon}$$

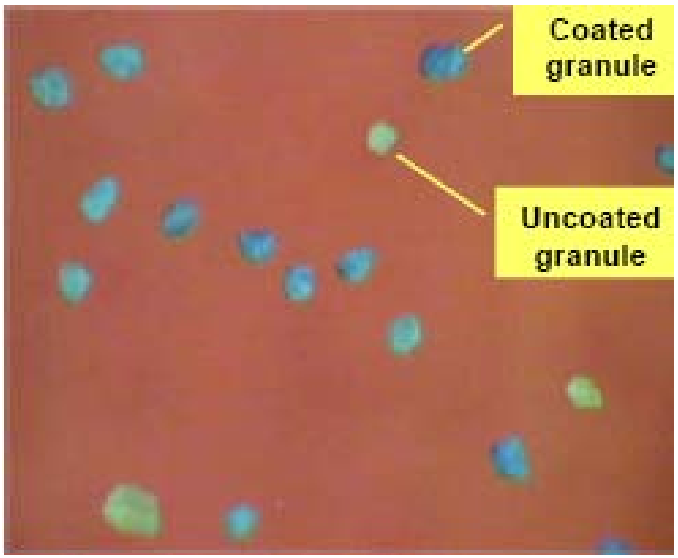
$$k_{con} = f(St_{def})$$

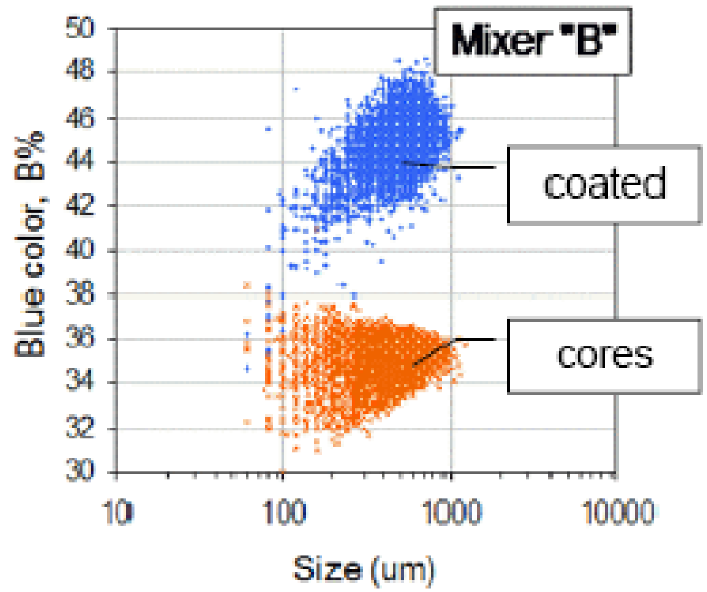


- Predict induction time in ba systems

Wildeboer, 2002

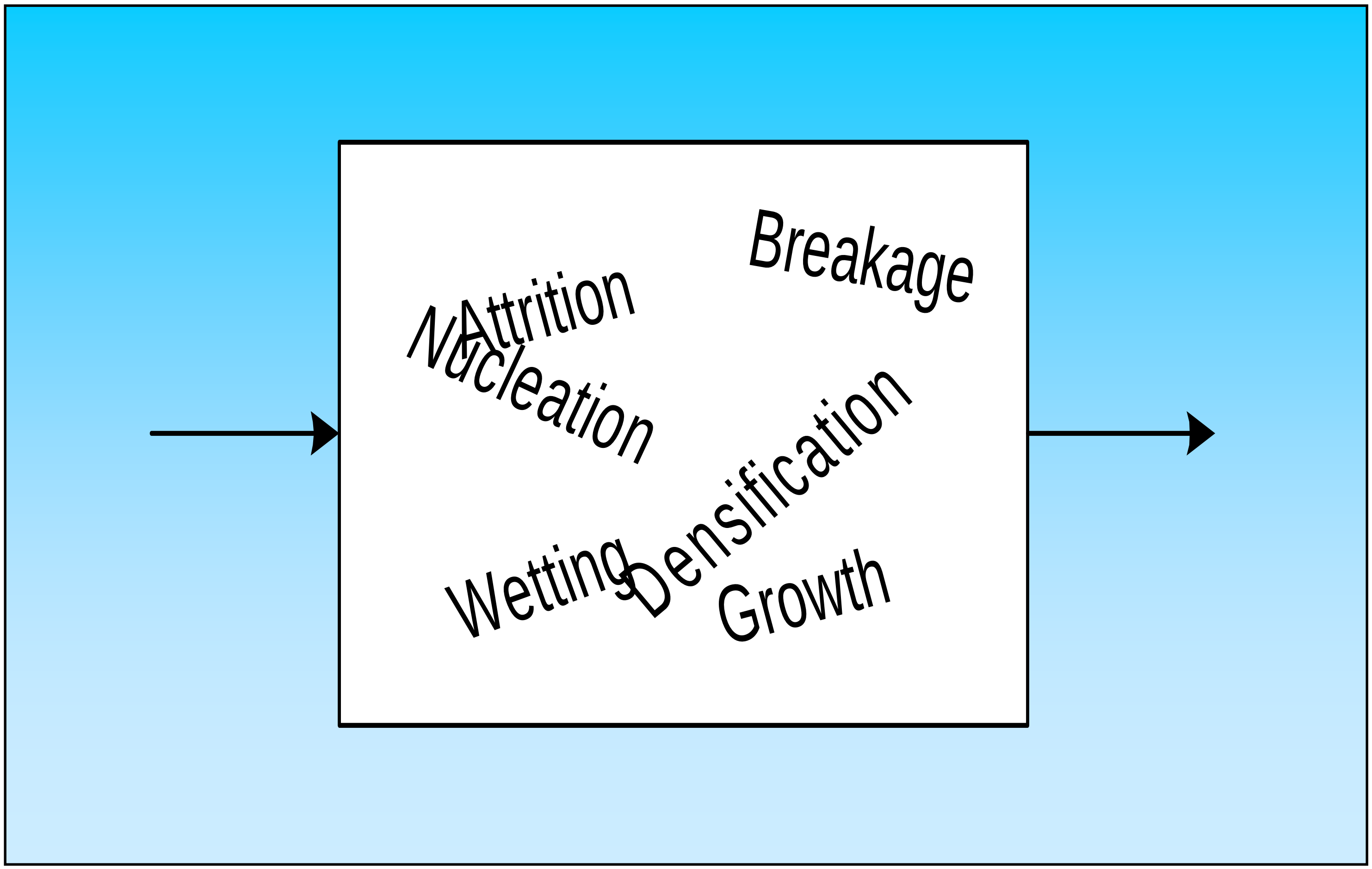
2-D distribution for layering



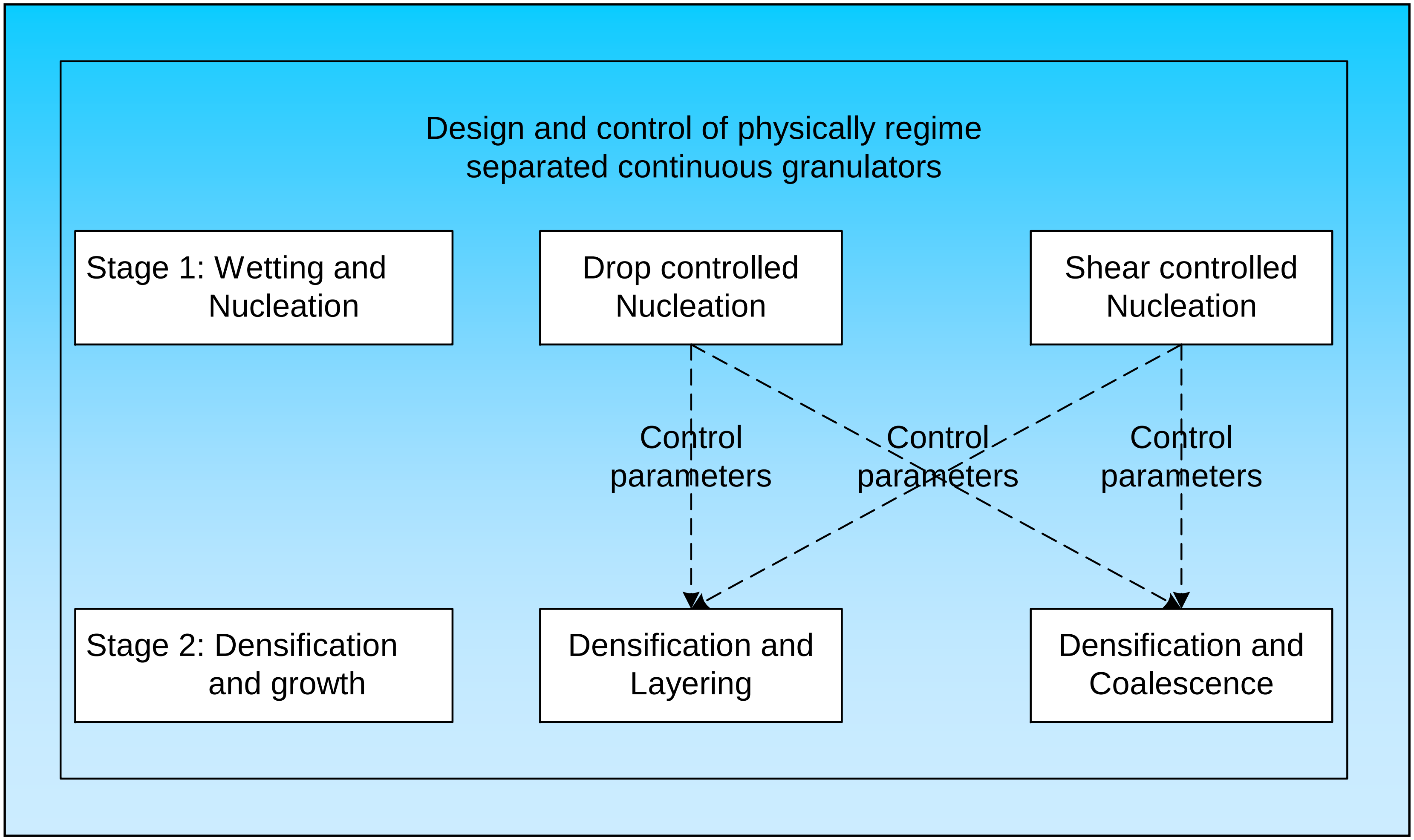


Mort et al., 2008

And now for something completely different: From this…….



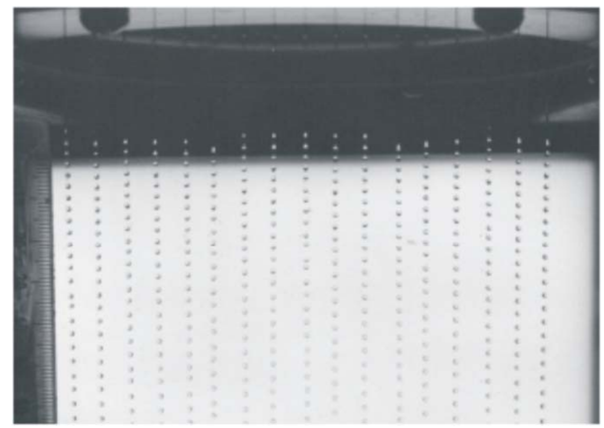
……. to this!

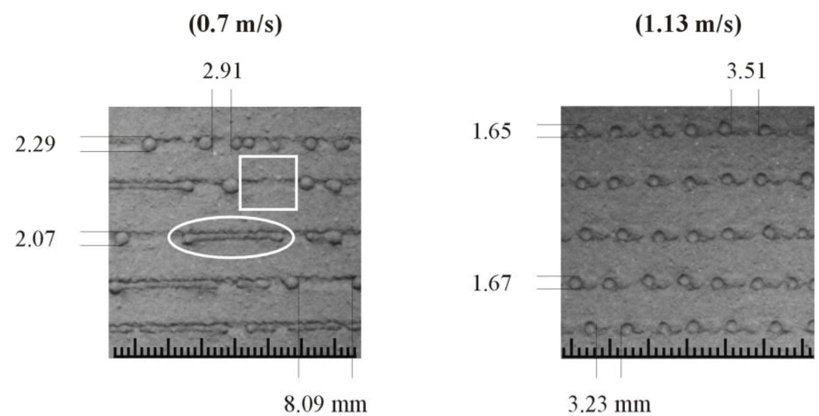


Wildeboer et al., *Powder Tech*, **171**, 96=205 (2007)

Regime separated granulators – Hans Wildeboer

- New nucleation device to give nearly monosized nuclei granules
  - Acoustic controlled showerhead over a thin bed of powder on a conveyor belt
  - Effectiveness depends on drop-drop and drop powder interactions at the powder surface
  - Extremely narrow size distributions possible





Summary