Tackling the inverse problem in multidimensional granulation modelling

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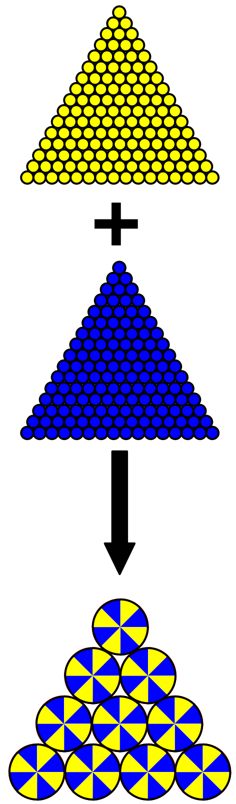
Wet granulation – A start

- Products



- “Lump” small particles into bigger entities
  - Improve **handling** (storage, transport, safety,...)
  - Creation of **micro mixtures** (segregation, distribution of active components)
- **Here:** Model the process with multidimensional population balance model

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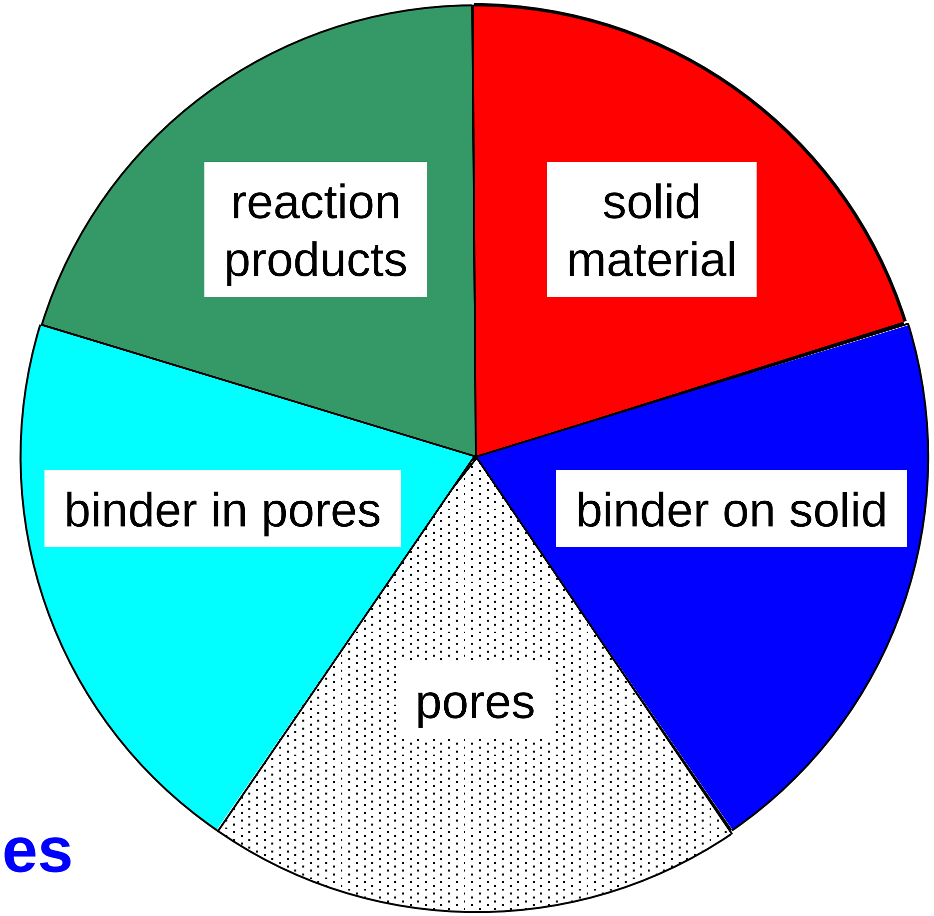
Model – Particle description

- composition of a granule
  - ➢ solid material
  - ➢ binder on solid
  - ➢ pores
  - ➢ binder in pores
  - ➢ reaction products
- particle shape: sphere

→ **5 independent variables** for particle description



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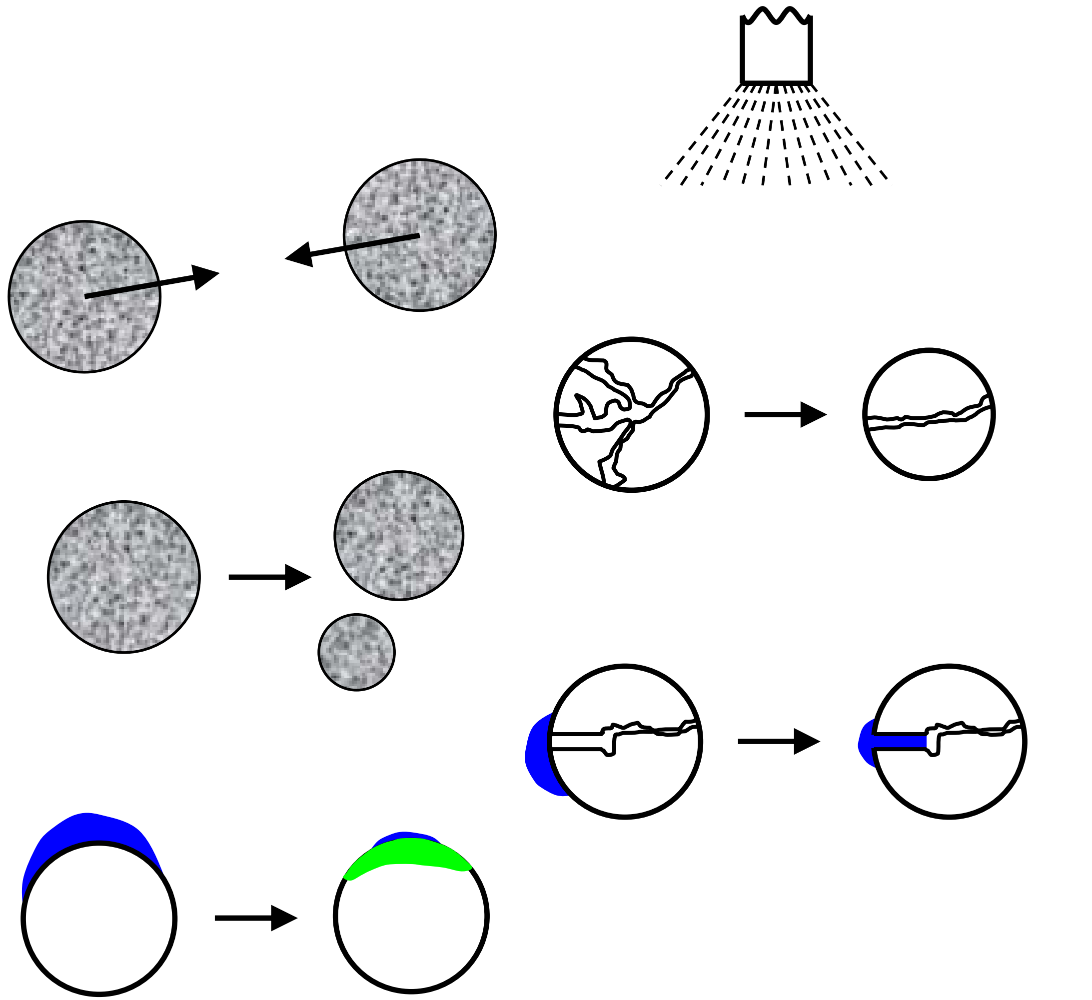
Model - Transformations

- Addition of binder
- Coalescence
- Compaction
- Breakage
- Penetration
- Reaction



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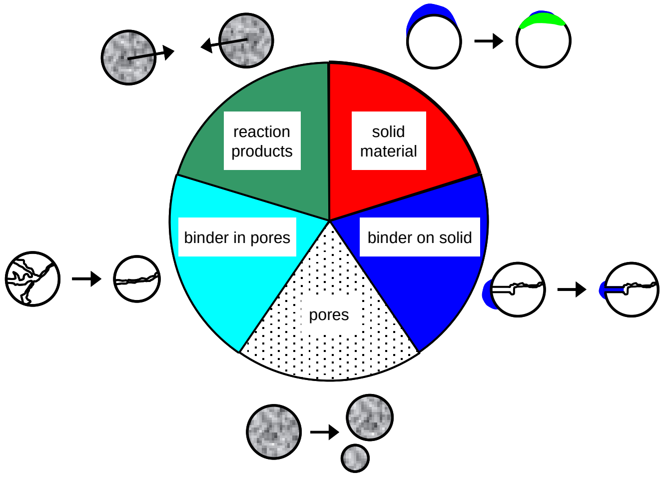
Using the model

- So what? – Run simulations!

Not quite!



$x_1 \ldots x_K$

S

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solid particles	s_o	$\mathrm{m^3}$
	ρ_{s_o}	$\mathrm{kg/m^3}$
liquid droplets	$V_{\mathrm{droplet}} = l_e$	$\mathrm{m^3}$
	η_l	Pa s
	ρ_{l_e}	$\mathrm{kg/m^3}$
Mixer-granulator operating parameters		
	n_{impeller}	$\mathrm{s^{-1}}$
	r_{impeller}	m
	$\widetilde{u}_{\mathrm{col}}$	-
	$\widetilde{u}_{\mathrm{imp}}$	-
Breakage		
	$\widehat{k}_{\mathrm{att}}$	$\mathrm{s\,m^{-5}}$
	s_r^*	-
	a	-
	b	-
	$v_{\mathrm{frag,min}}$	$\mathrm{m^3}$
	$\nu_{\mathrm{max,I}}$	-
	$\nu_{\mathrm{min,max}}$	-
	α	-
	β	-
	$\nu_{\mathrm{max,II}}$	-
Chemical Reaction		
	C	-
	$k_{\mathrm{reac,e}}$	m/s
	$k_{\mathrm{reac,i}}$	m/s
Coalescence		
	e_{s_o}	-
	e_{s_r}	-
	h_a	m
	$\widehat{K}_0$	$\mathrm{m^3}$
Compaction		
	k_{porred}	s/m
	$\varepsilon_{\mathrm{min}}$	-
Penetration		
	$\widehat{k}_{\mathrm{pen}}$	$\mathrm{kg^{1/2}\,s^{-3/2}\,m^{-7/2}}$

→ Establish *x*'s through expe

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Unknown parameters

- unknown parameters:
  - – coalescence rate constant $K_0 = ?$
  - – compaction rate constant $k_{comp} = ?$
  - – breakage rate constant $k_{att} = ?$
  - – reaction rate constant $k_{reac} = ?$

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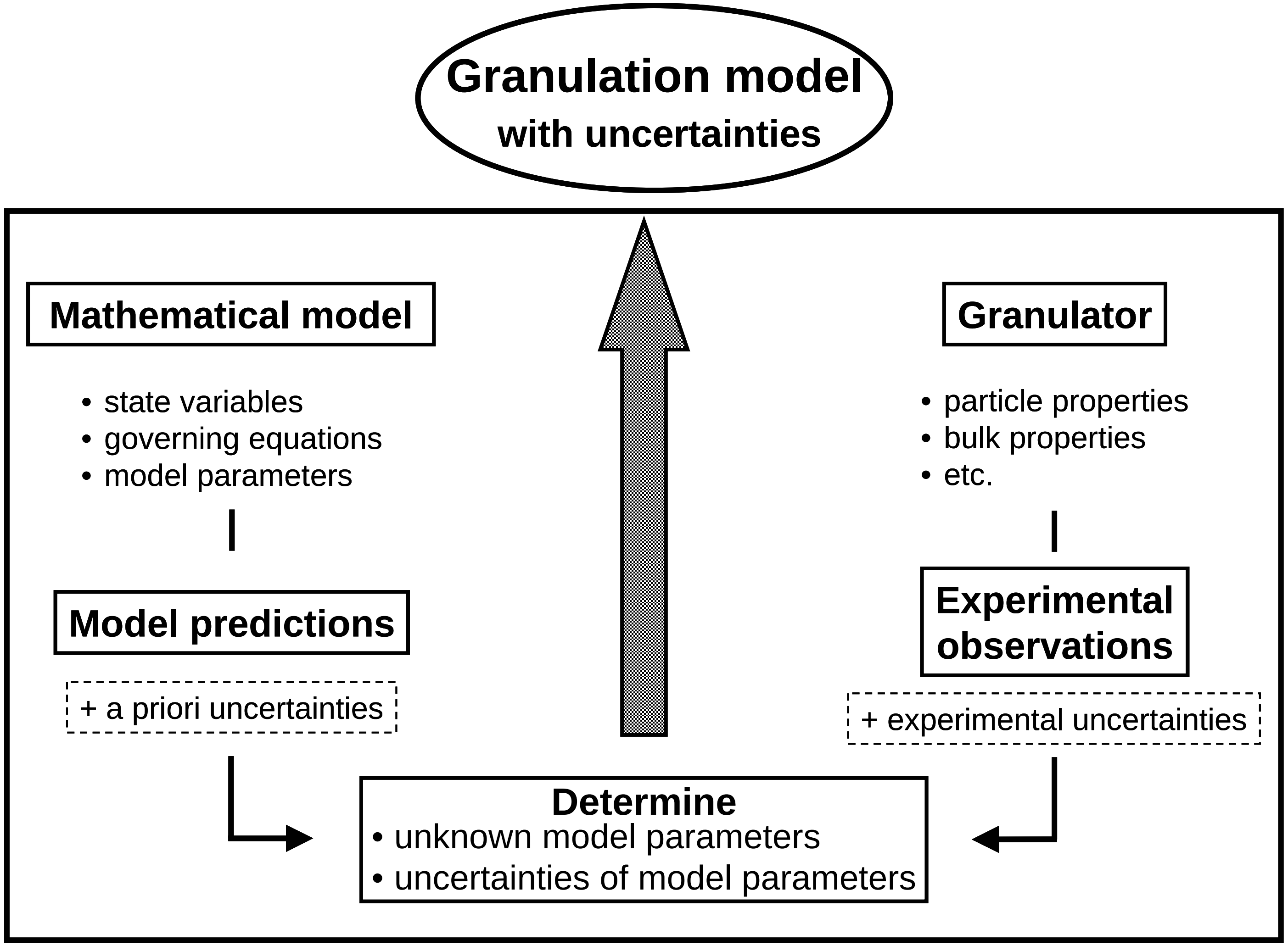
The idea



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Theory

- Experimental data

$$\eta^{\text{exp}} = \eta_0^{\text{exp}} \pm \sigma^{\text{exp}}$$

- Model response with paramaters $\boldsymbol{x}$

$$\eta = \eta(\boldsymbol{x}) \qquad \text{with} \qquad \boldsymbol{x} = (x_1, \ldots, x_K)$$

- and $x = x_0 + c\xi$ with uncertainty factor *c* and standard normally distributed ξ

For a simple linear model, *K*=1

$$\eta(x) = A + Bx \qquad \eta(x, c, \xi) = A + B(x_0 + c\xi)$$

$$\mu(x_0) = E[\eta(x_0, c, \xi)] = A + Bx_0 \qquad \sigma(c) = \sqrt{\mathrm{Var}(\eta(x_0, c, \xi))} = \sqrt{B^2 c^2}$$

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Theory II

- Optimal values for parameters and associated uncertainties

$$(\boldsymbol{x}_0^*, \boldsymbol{c}^*) = \underset{\boldsymbol{x}_0, \boldsymbol{c}}{\text{argmin}} \{\Phi(\boldsymbol{x}_0, \boldsymbol{c})\}$$

- with objective function

$$\Phi(\mathbf{x}_0, \mathbf{c}) = \sum_{i=1}^{N} \left([\eta_{0,i}^{\exp} - \mu_i(\mathbf{x_0}, \mathbf{c})]^2 + [\sigma_i^{\exp} - \sigma_i(\mathbf{x_0}, \mathbf{c})]^2 \right)$$

- and constraints

$$x_{0,k,\text{low}} \le x_{0,k} \le x_{0,k,\text{up}} \qquad (k = 1, \ldots, K)$$

$$0 \le c \le c^{(0)}$$

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Get on with it

- Problem: Running the optimisation on the complex model is computationally expensive

→ find substitute: **Response surfaces**

= approximation of process behaviour

$$\eta(x) = y_{sim}(x) + \varepsilon$$

- How to use?

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Response surfaces

- approximate process (model) behaviour locally
- e.g., 1st order response surface

$$\eta(x) = \beta_0 + \sum_{k=1}^{K} \beta_k x_k$$

with $\eta(x) = \text{response surface}$

$K = \text{number of variables}$

$\beta_0, \beta_k = \text{parameters of surface}$

- β_k : process sensitivities with respect to x_k

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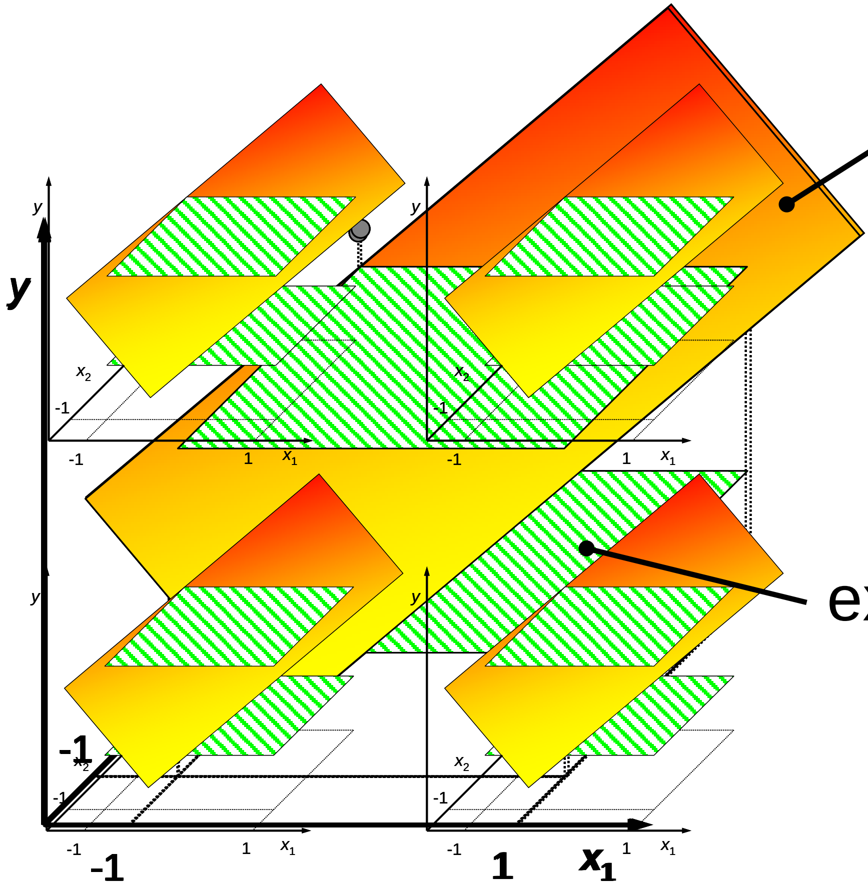
Methodology in short

scenario 1

scenario 2



response surface

experiment 1

scenario 3

scenario 4

→ parameters: K, k_0, k_{reac}

$$\eta(x) = \beta_0 + \sum_{k=1}^{K} \beta_k x_k$$

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Example

- experimental data from Simmons et al. (2006)
  - bench scale granulation of non-pareils (sugar) with PEG4000/water binder
  - run at 900 and 1200 rpm impeller speed
  - examine amount of agglomerates
  - 4 sampling times

→ 8 experimental data sets

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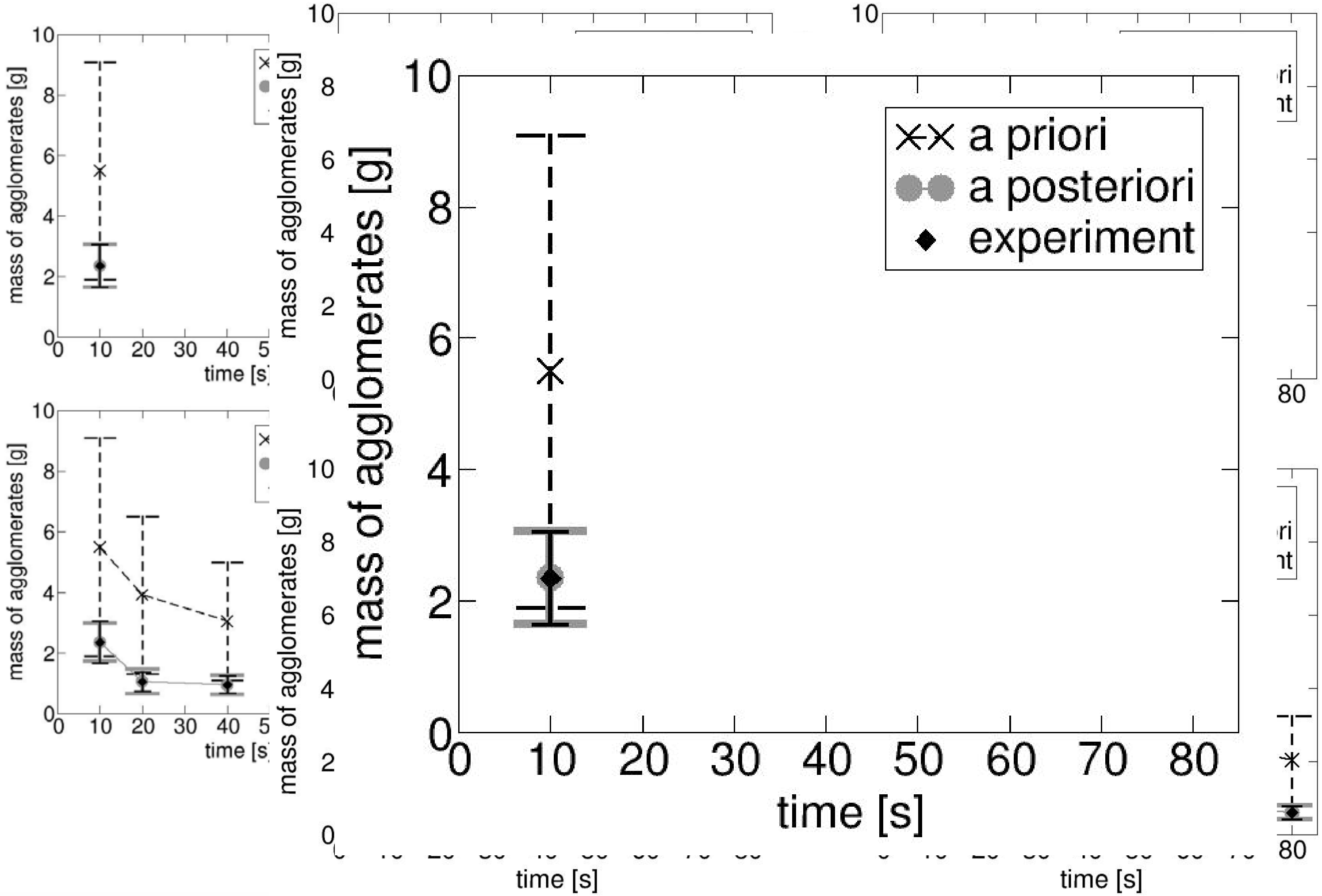
Example – 900 rpm data



uncertainties

)) $\cdot 10^{-10}$ m^3

)4 s/m

$\cdot 10^{7}$ s/m^5

$\cdot 10^{-9}$ m/s

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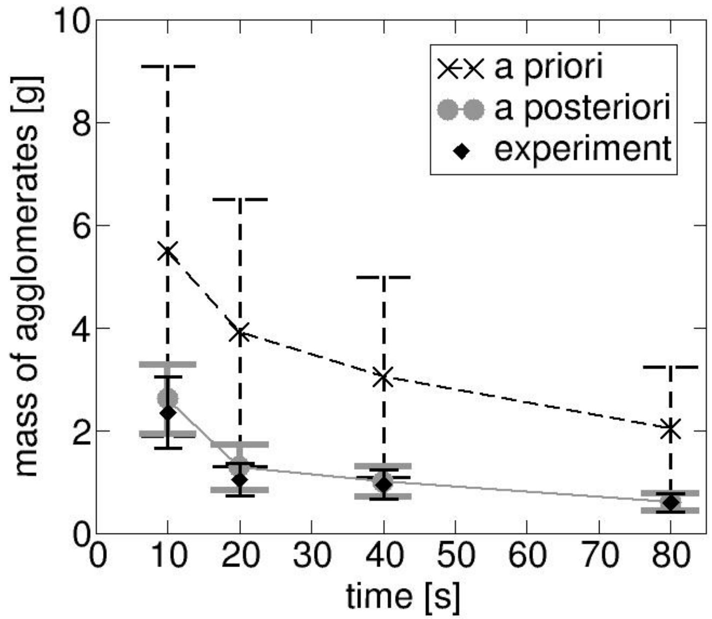
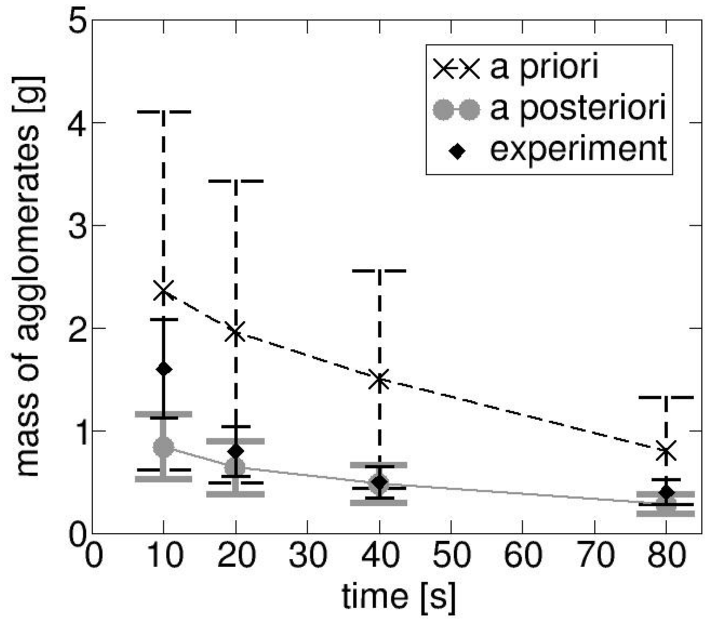
Example – all data



900 rpm



1200 rpm

Parameters and uncertainties using all 8 sets

$K_0 = (1.23 \pm 0.00) \cdot 10^{-10}\ \mathrm{m}^3$, $k_{comp} = 0.20 \pm 0.05$ s/m

$k_{att} = (4.9 \pm 0.0) \cdot 10^{7}\ \mathrm{s/m}^5$, $k_{reac} = (4.000 \pm 0.003) \cdot 10^{-9}$ m/s

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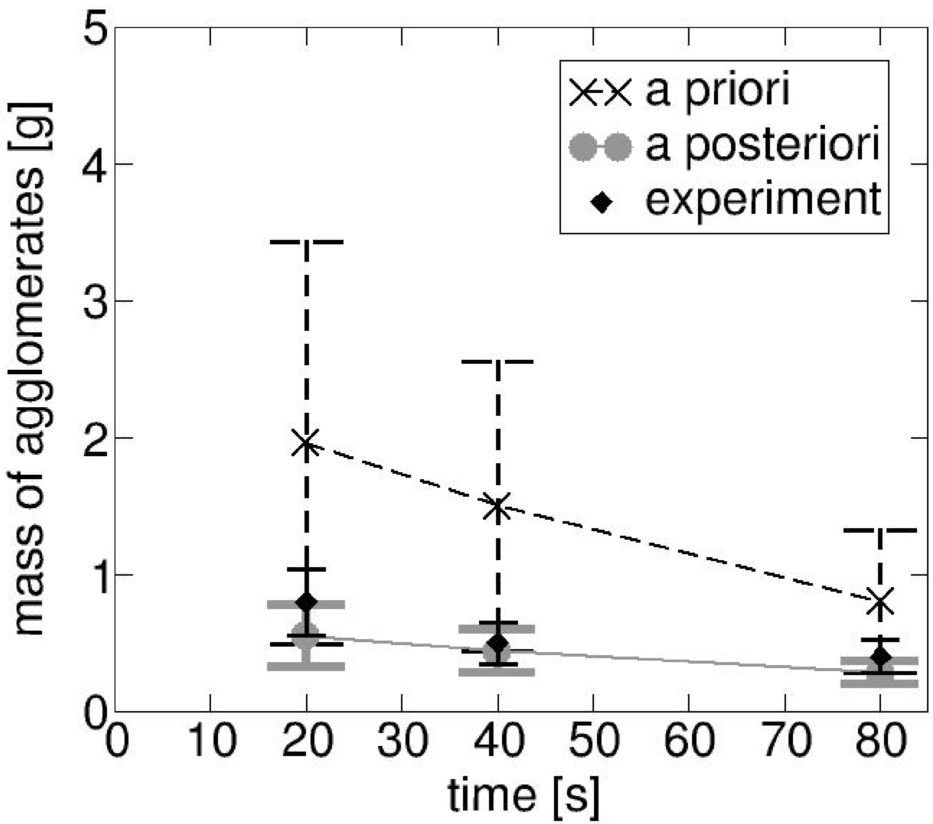
All data – a closer look



→ revised set of parameters

→ exclude from procedure

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Summary

- Population balance model for granulation
  - 5 dimensional particle space
  - several subprocesses
- Approach for parameter and uncertainty estimation using experimental data
- Falsification of models

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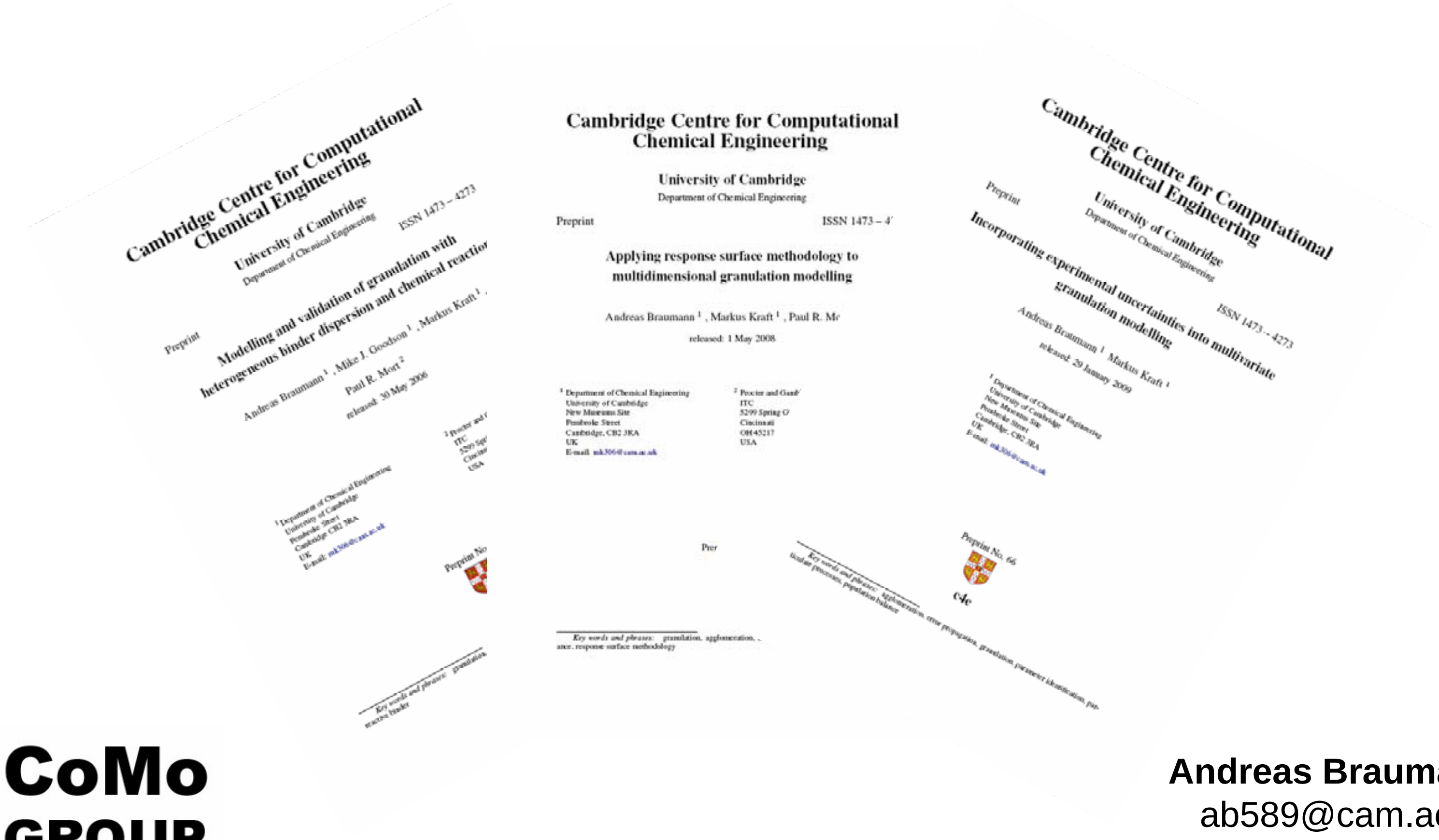
Further information

CoMo Group preprints

http://como.cheng.cam.ac.uk/index.php?Page=Preprints



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