From Optimization of industrial Crystallizers to Design of New Equipment using Advanced Modelling Tools

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R&D Process Engineering
Solvay

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Who are we?

A major global player in Chemicals with compelling strengths

Created by Ernest Solvay in 1863, Solvay is a **Global** company, with historical anchorage in Europe, and headquartered in Brussels.

Our strengths

- **90% of sales** in businesses among the **top 3 global leaders**
- A **balanced portfolio of activities**, directed **at growth regions**
- A culture of **sustainability, innovation and operational excellence**

€9.9 bn
NET SALES

€1,663 bn
Adjusted REBITDA

117
INDUSTRIAL SITES

15
MAJOR R&I

29,400
EMPLOYEES
55 COUNTRIES

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Investments, energy and quality: The key drivers of process improvements

- **Reduce the investments costs**
  - Increase of the existing equipment capacity ("debottlenecking")
  - intensification of new equipment
  - reuse of existing devices ("retrofitting")

- **Reduce the energy consumption**

➔ better control of heat transfers, continuous process

- **Best Product**

gCRYSTAL: an opportunity to develop advanced crystallization models

Why modelling crystallization?

- Major impact on most of the processes involving solid
- Complex operation

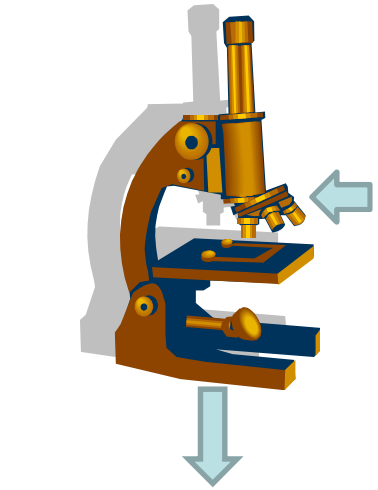
Why using gCRYSTAL?

- To avoid building a full “in-house” population balance code

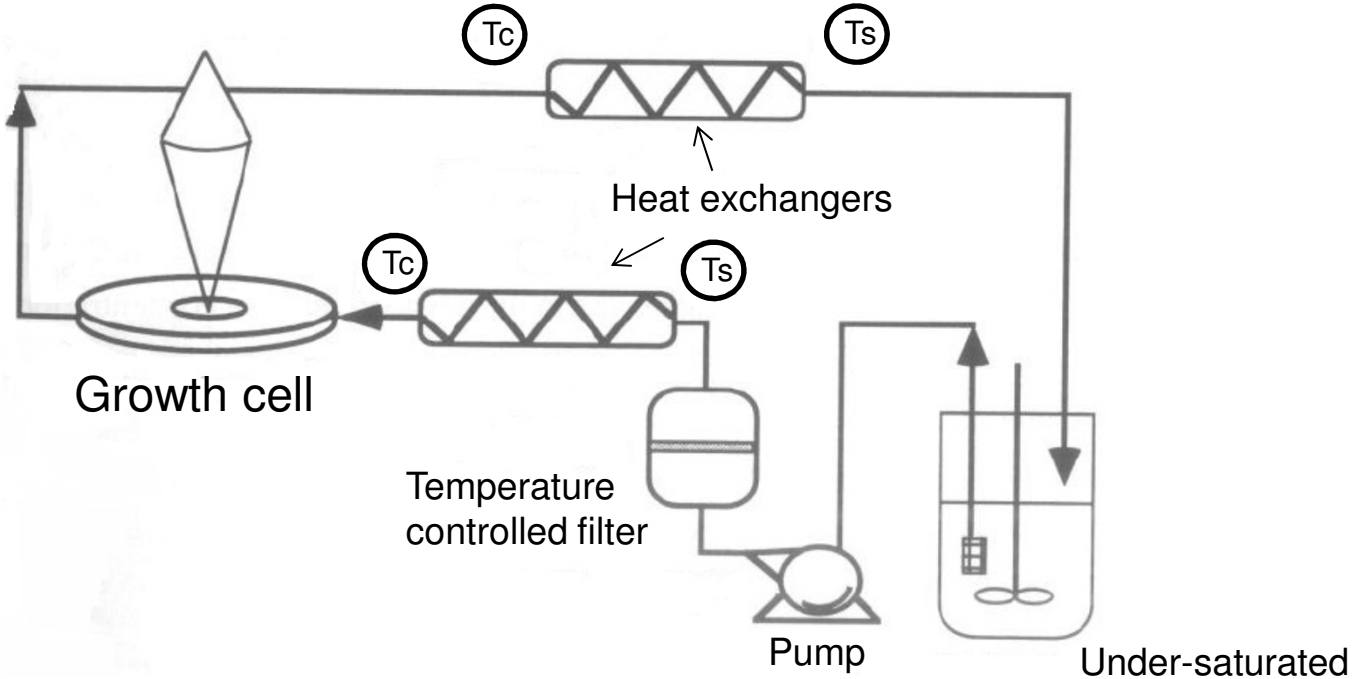
Outline

- Building a batch crystallization model using gCRYSTAL
  - Data acquisition
  - Model & Kinetic parameters estimation
  - Kinetic model development
- Optimizing an industrial crystallizer capacity and energy consumption
- Towards the Design of New Crystallizers

A direct measurement of the growth rate kinetic



Camera



Growth cell scheme

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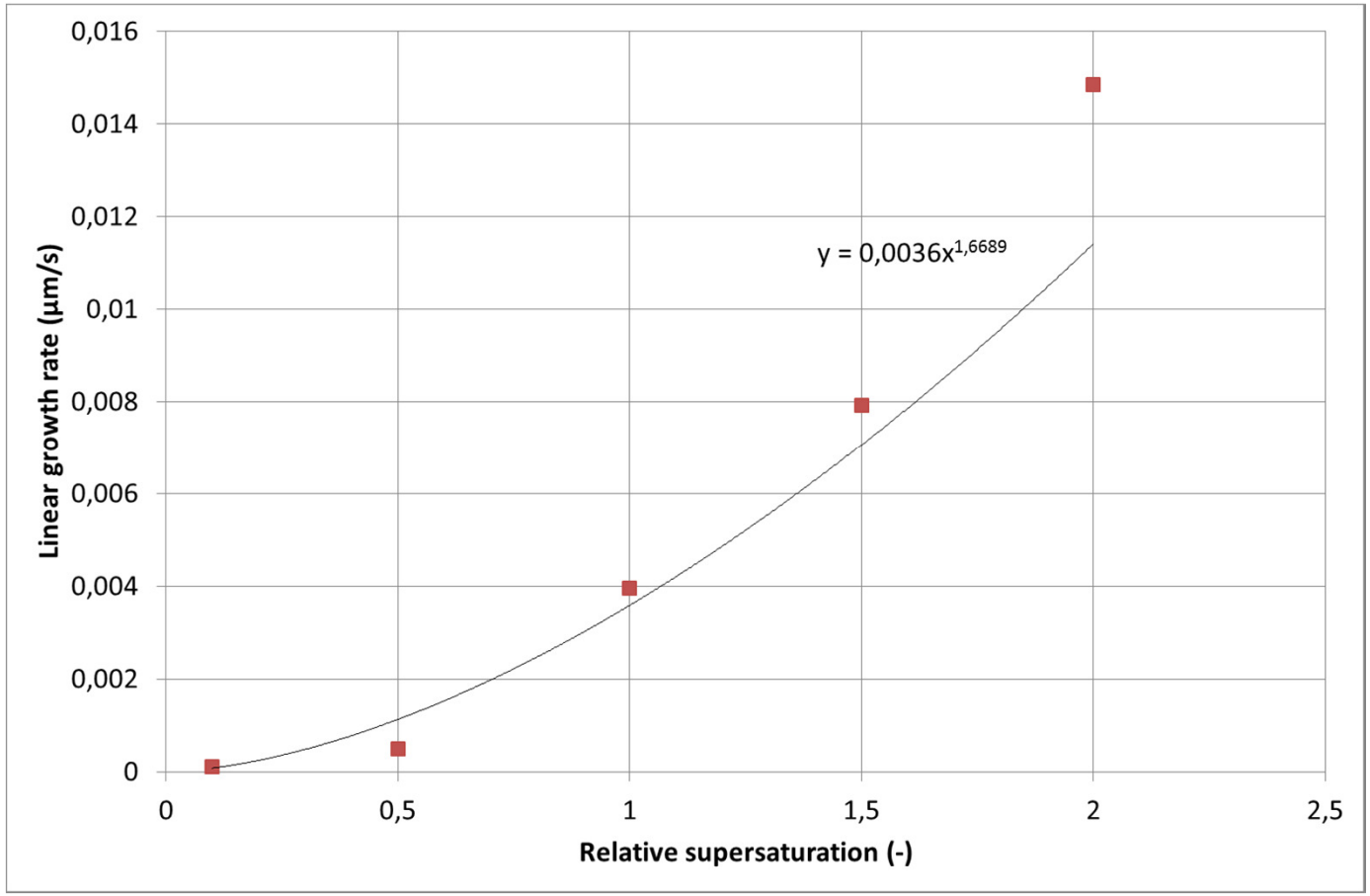
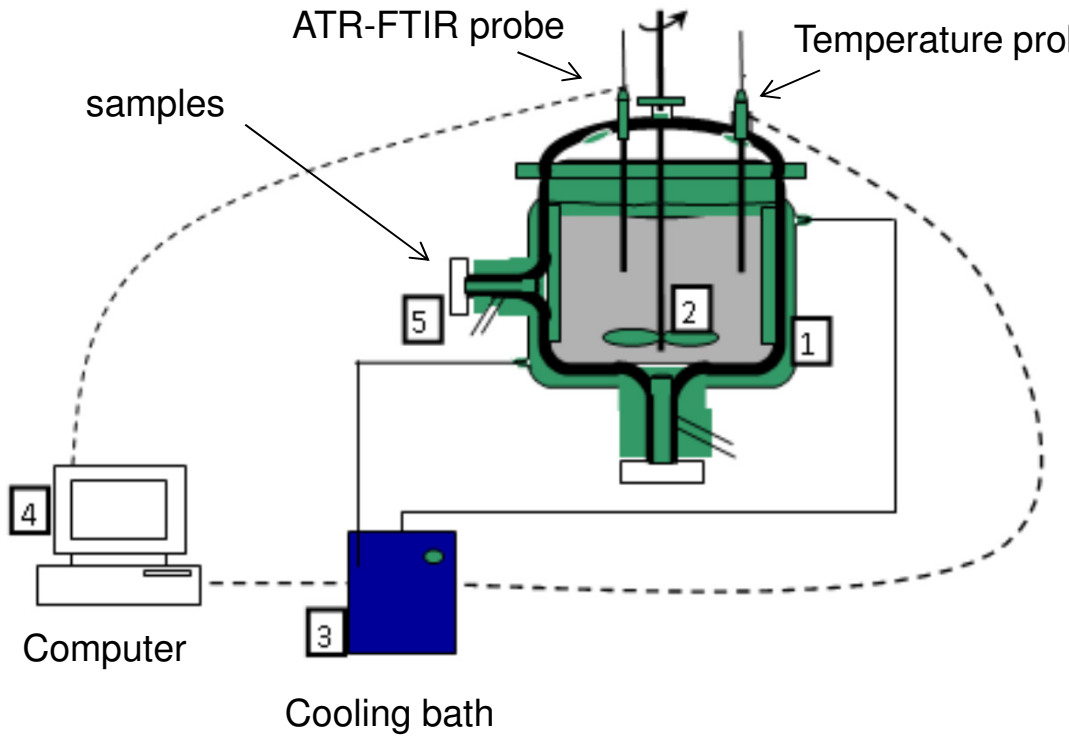
Growth rate function of the supersaturation



Power law kinetic

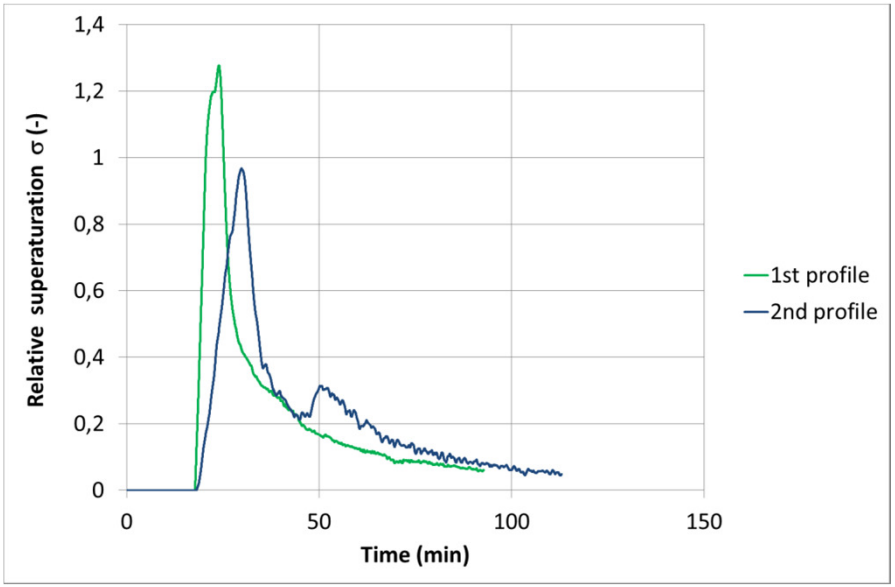
Monitoring temperature, concentration and crystal size during a batch cryst.



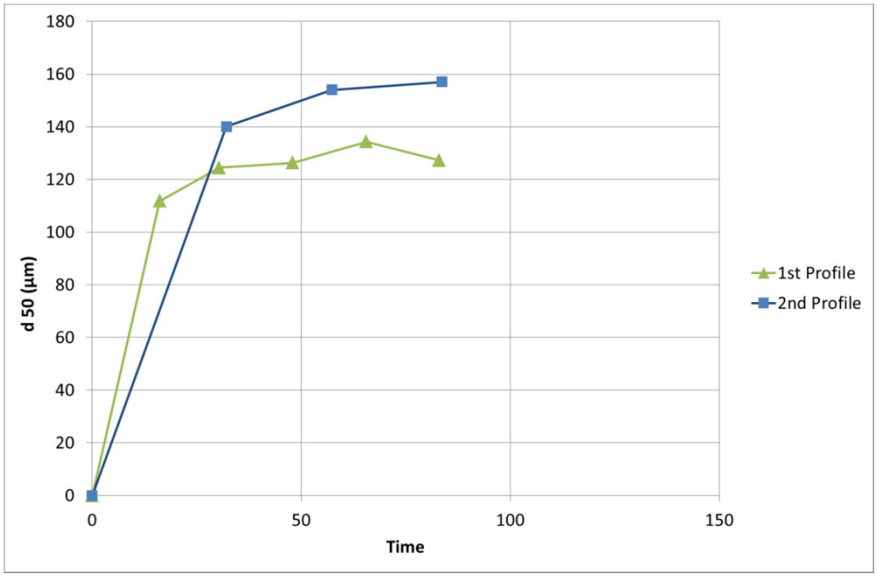
1- MSMPR
2- Mixell TT impeller
3- Cooling bath
4- Computer
5- Sampling

Effects of changing the temperature profile

Supersaturation profiles



Crystal size



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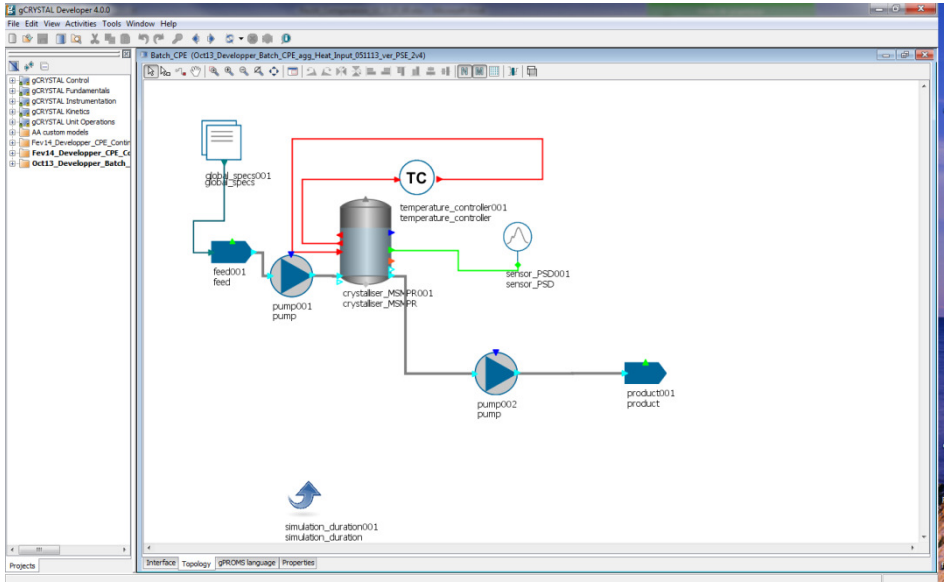
  - heat exchange
  - modelling approach of the scaling mechanism
- Towards the Design of New Crystallizers

gCRYSTAL model: a fast way to simulate a batch crystallization

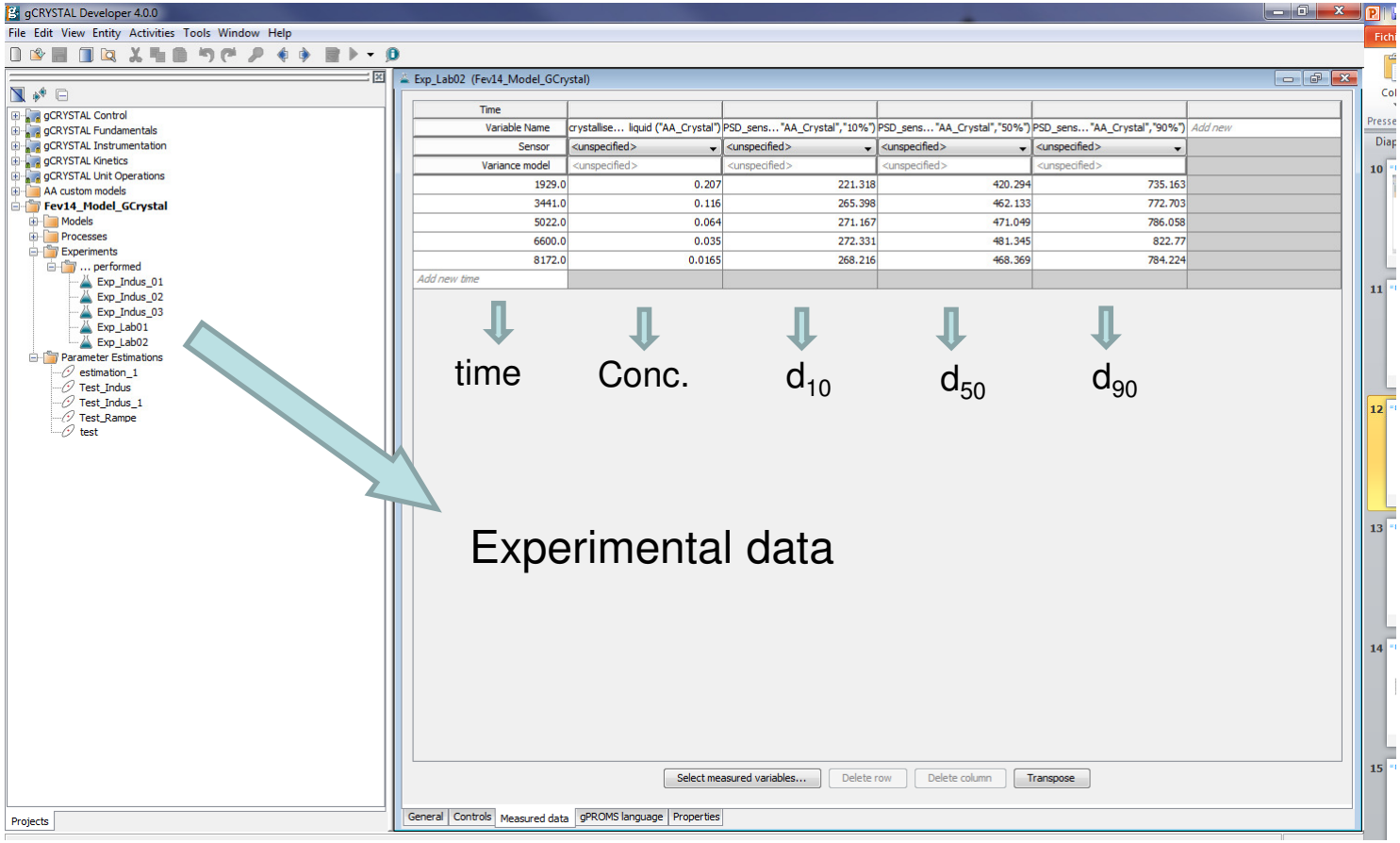


"Bulid-in" kinetics from gCRYSTAL were used

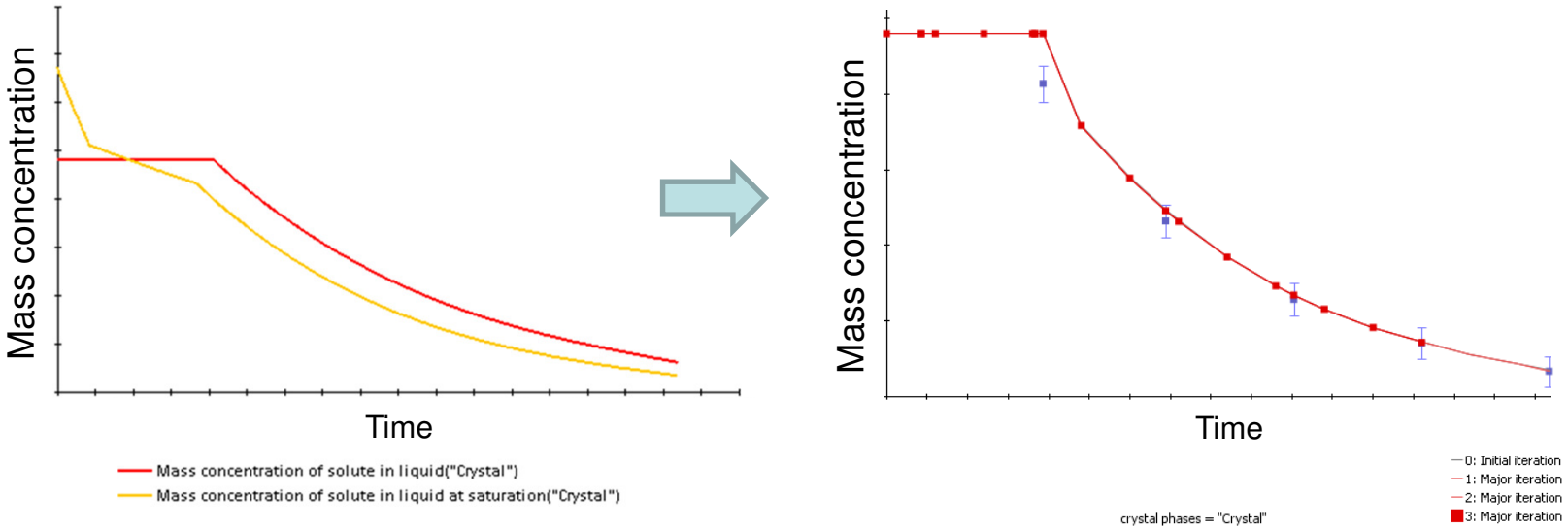
1ary nucleation $$r_{N1} = \boldsymbol{A_n} \cdot e^{-\frac{\boldsymbol{B_n}}{\ln(C/C^*)}}$$

Linear growth rate $$G = \boldsymbol{k_c} \cdot \frac{2 \cdot M_\omega}{\rho} \left(\frac{C - C^*}{C^*}\right)^{\boldsymbol{j_c}}$$

Entering the experimental data



Good estimation of the kinetc parameters using the optimization feature



Good match between simulated and experimental concentration profiles for different cooling rate

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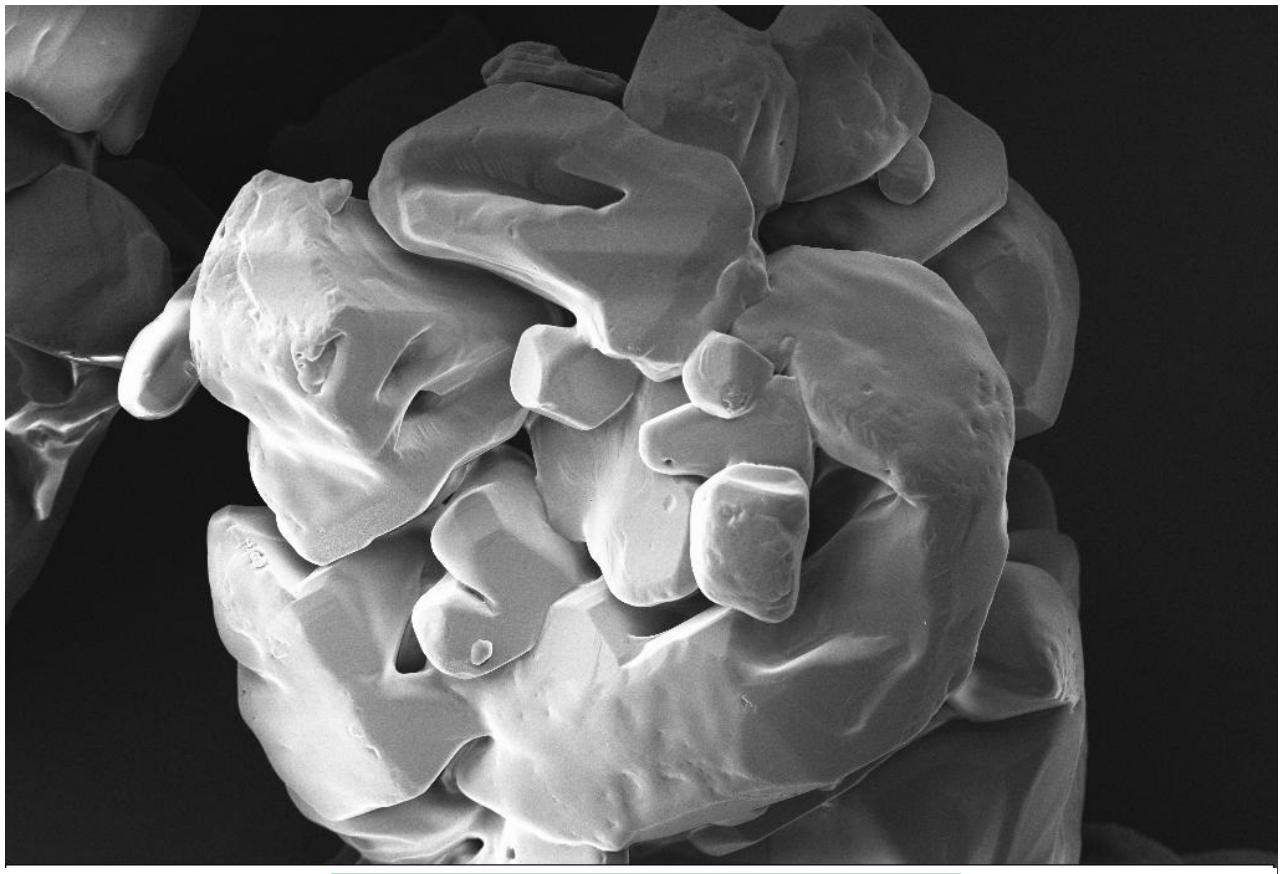
Crystal Habit



Crystals look like aggregates

Agglomeration model

Addition of an Aggregation kinetics (Mumtaz)

Fitting parameter

Agglomeration kernel $$\beta_{agg} = \left(\sqrt{\frac{8\pi\varepsilon}{15\nu}} \cdot \bar{d}_{3.0}^{3} \right) \cdot \frac{(A_{50}G)/(\varepsilon\rho\bar{d}_{3.0}^{2})}{1+(A_{50}G)/(\varepsilon\rho\bar{d}_{3.0}^{2})}$$

Collision rate Agglomeration efficiency

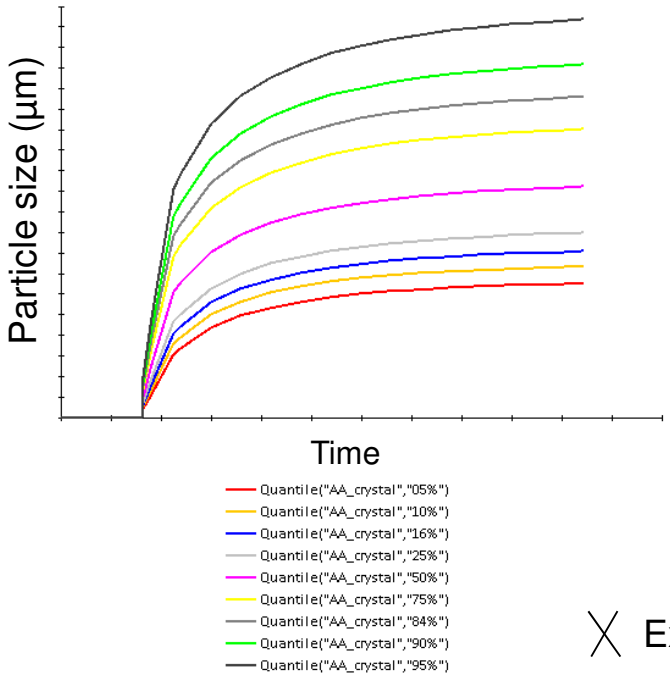
L	length of the line of contact between two collided crystals
σ^{*}	apparent yield strength
G	linear growth rate of crystals
ε	energy dissipation rate
ρ	liquid density
$\bar{d}_{3,0}$	3,0 mean particle size

Particle size prediction with the new model

Good match !!

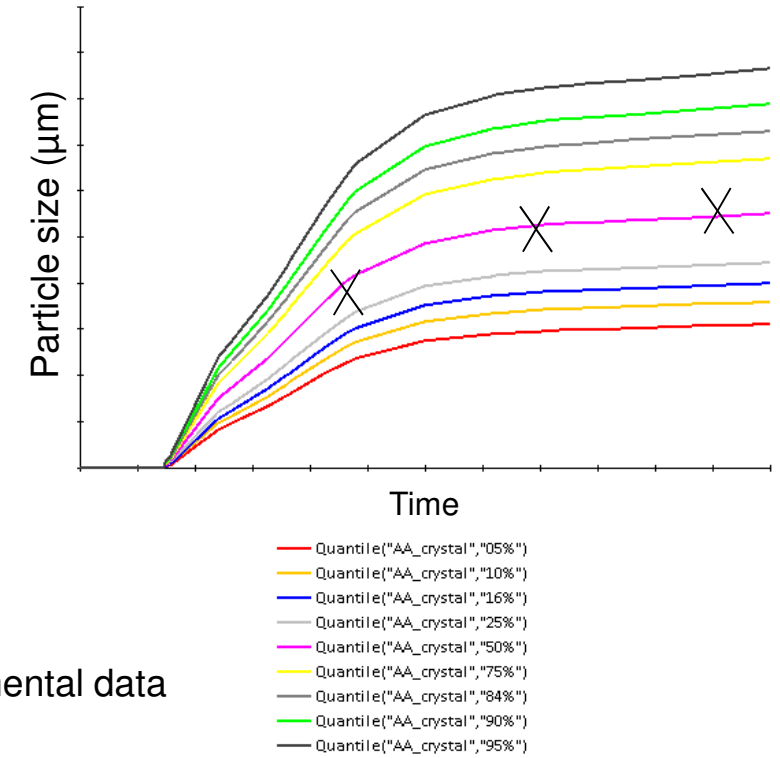
Without agglomeration

50 µm



With agglomeration

300 µm



Experimental data

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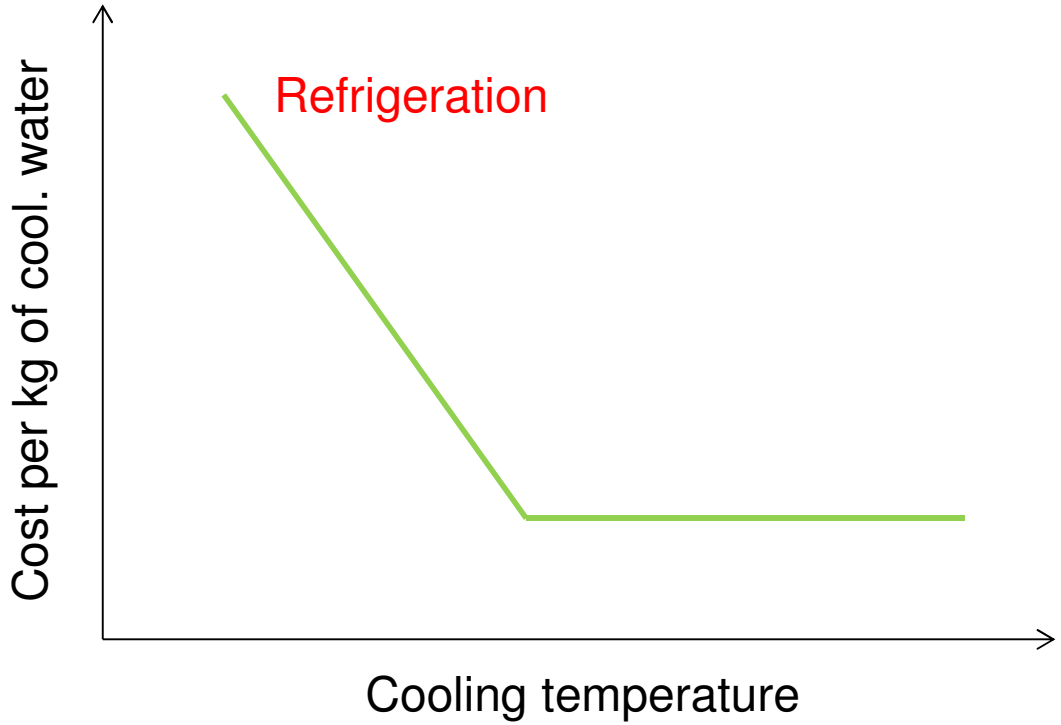
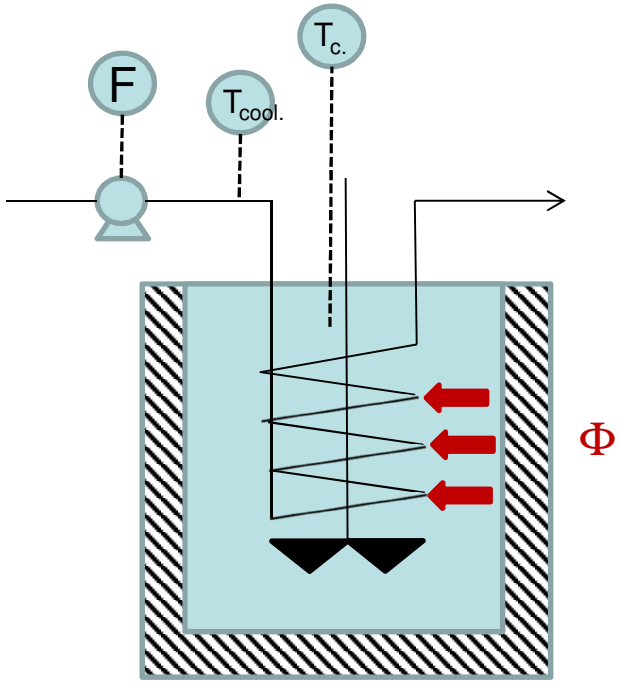
Energy cost for cooling crystallization



The temperature of the cooling water has to be controlled for better economical results

How to control the heat exchange?

$$\Phi = U \cdot A \cdot \left(T_{cryst.} - T_{cool.}\right)$$

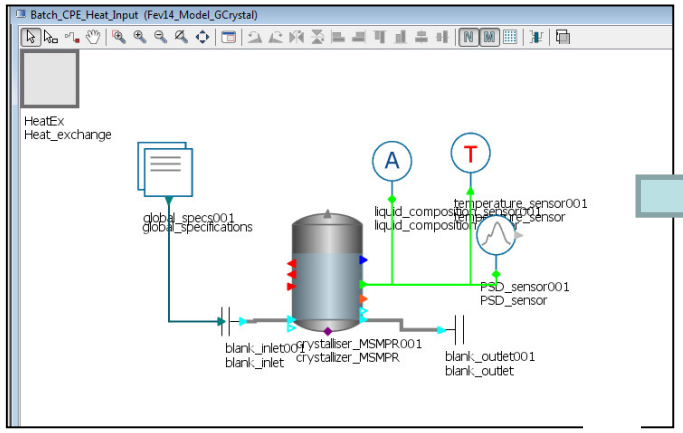


Modeling of a heat controlled crystallizer

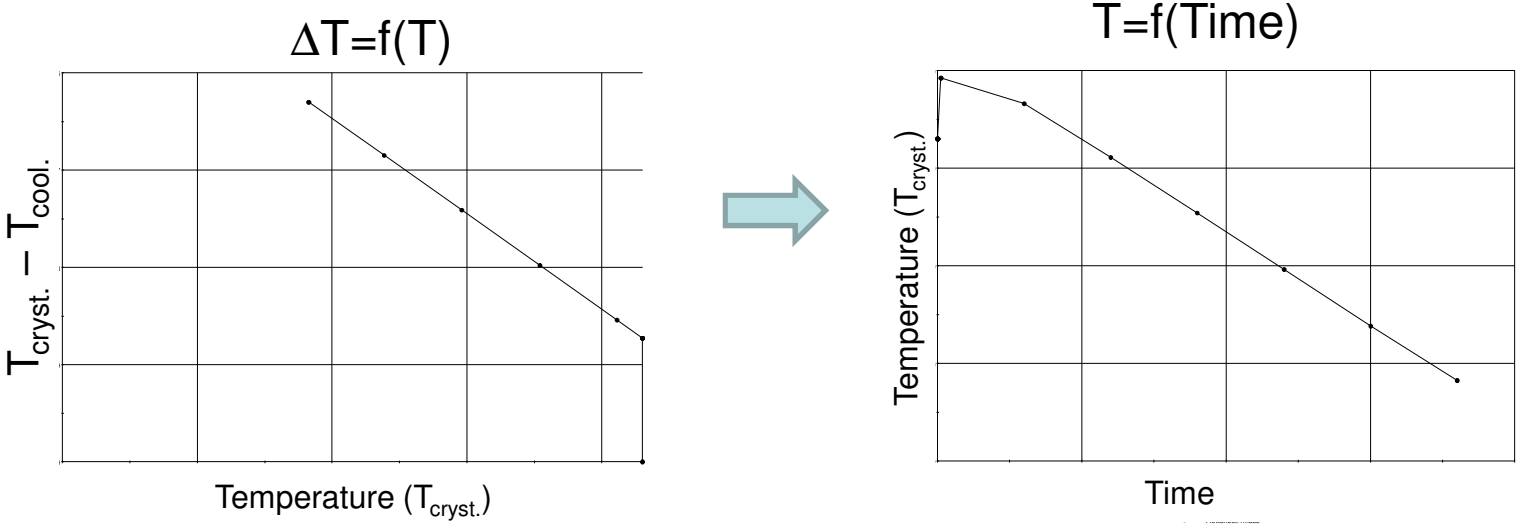
T_{cryst}	T_{cryst}-T_{cool}
T1	ΔT1
T2	ΔT2
T3	ΔT3
…	…

Coupling heat balance and population balance

gCRYSTAL developper version



Instead of being directly controled by a T° profile, the crystallization is controlled by the **heat output profile**



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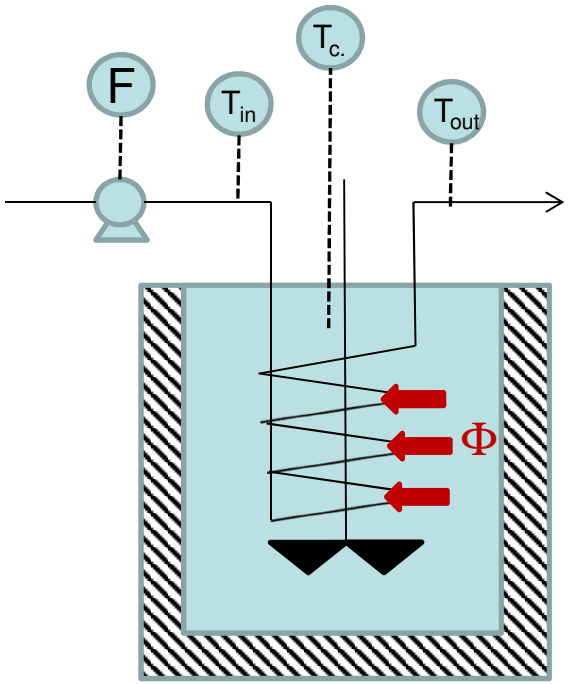
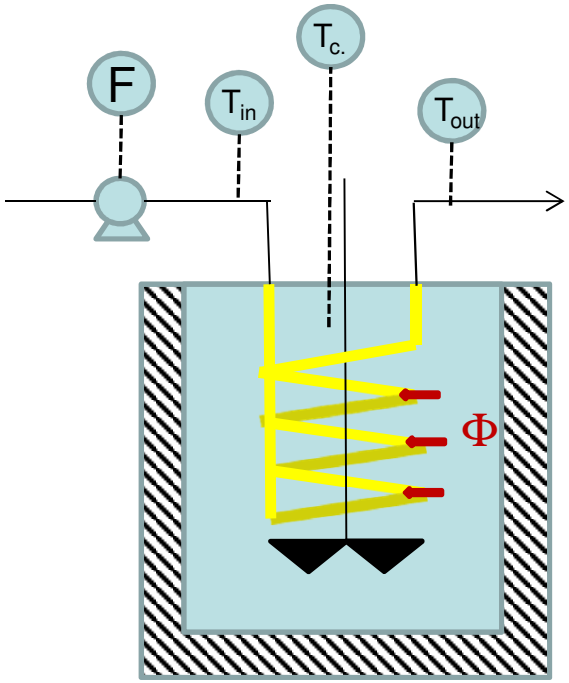
What is scaling?

Initial T°



Beginning of crystallization

Final T°

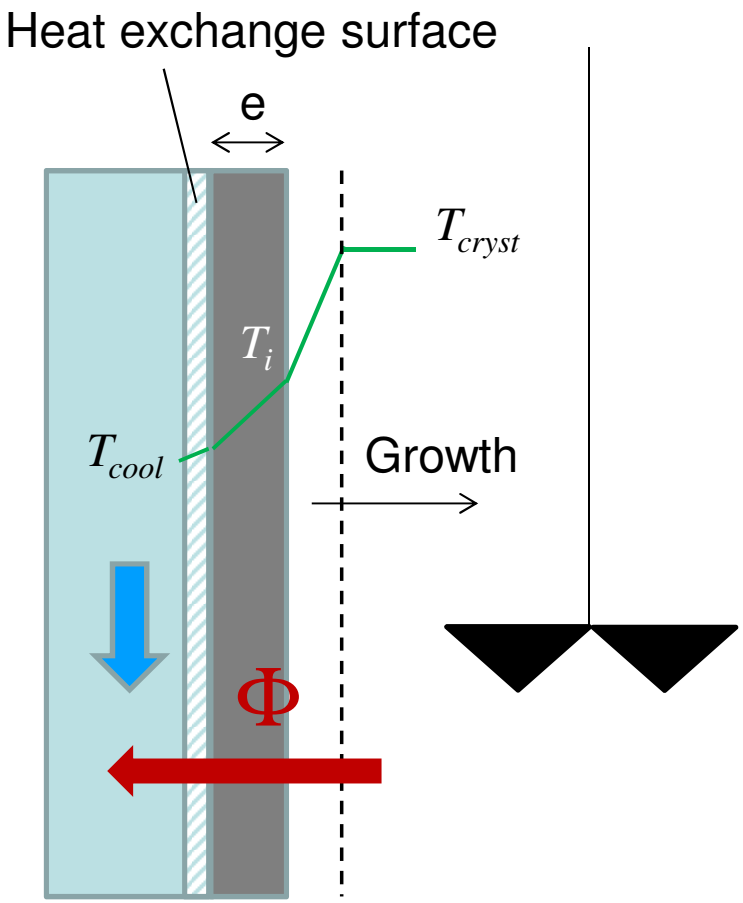


End of crystallization

$$\Phi = \boldsymbol{U} \cdot A \cdot \Delta T$$

Scaling affects the heat exchange coefficient

How to model scaling?



$$U(t) = \frac{U_0}{1 + f \cdot e(t)}$$

U:	heat exchange coefficient
U_0:	Initial heat exchange coefficient
e:	Scaling thickness
f:	Thermal conductivity factor

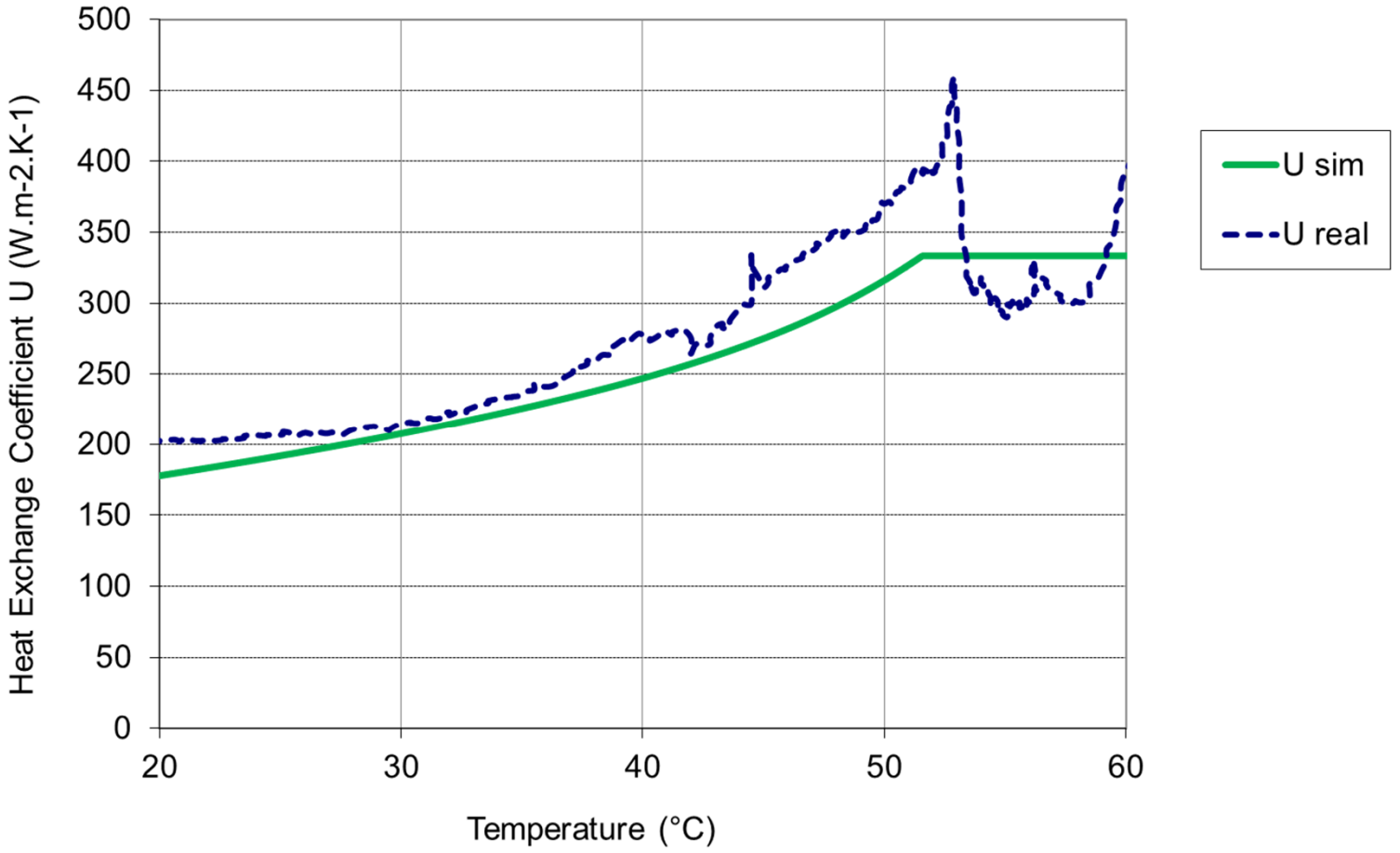
$$e(t)=f(\sigma_s, \sigma_c)$$

With local supersat. σ_c

Implementation in gCRYSTAL Developer version

Evolution of the heat exchange coefficient

Scaling mechanism



Good match between simulated and real Heat Exchange Coefficient

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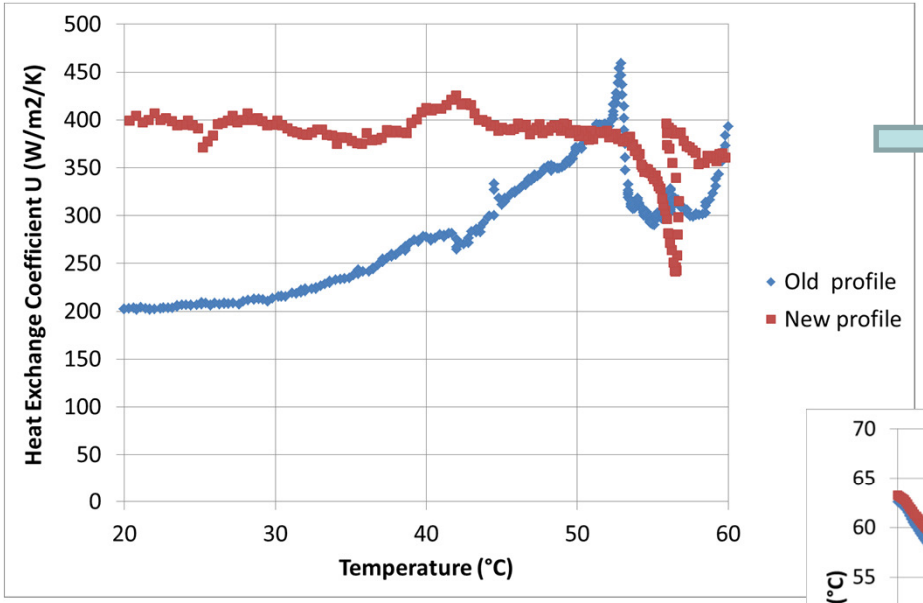
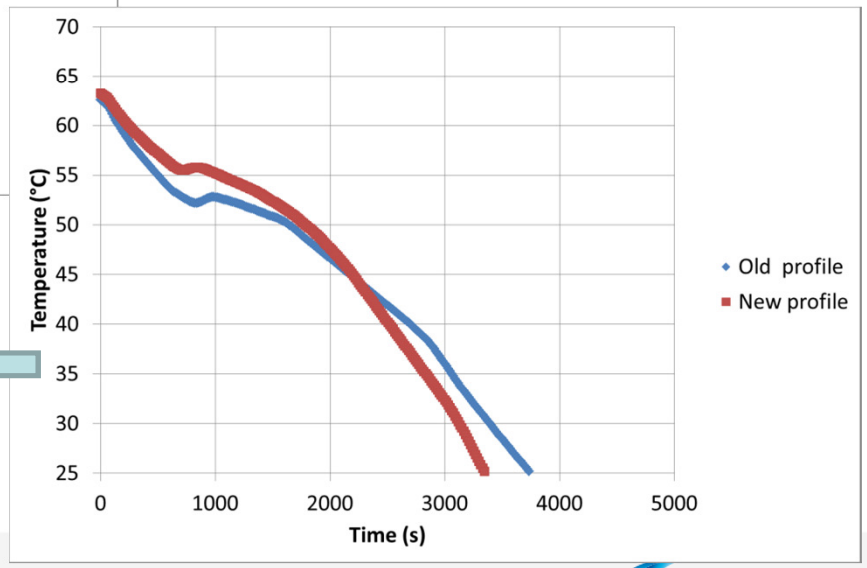
New cooling profile to avoid scaling

Stability of the heat exchange coefficient !



- No risk of residues in the crystallizer
- Decrease the energy cost by 15%

Decrease of the operation duration !!



Higher productivity 10%

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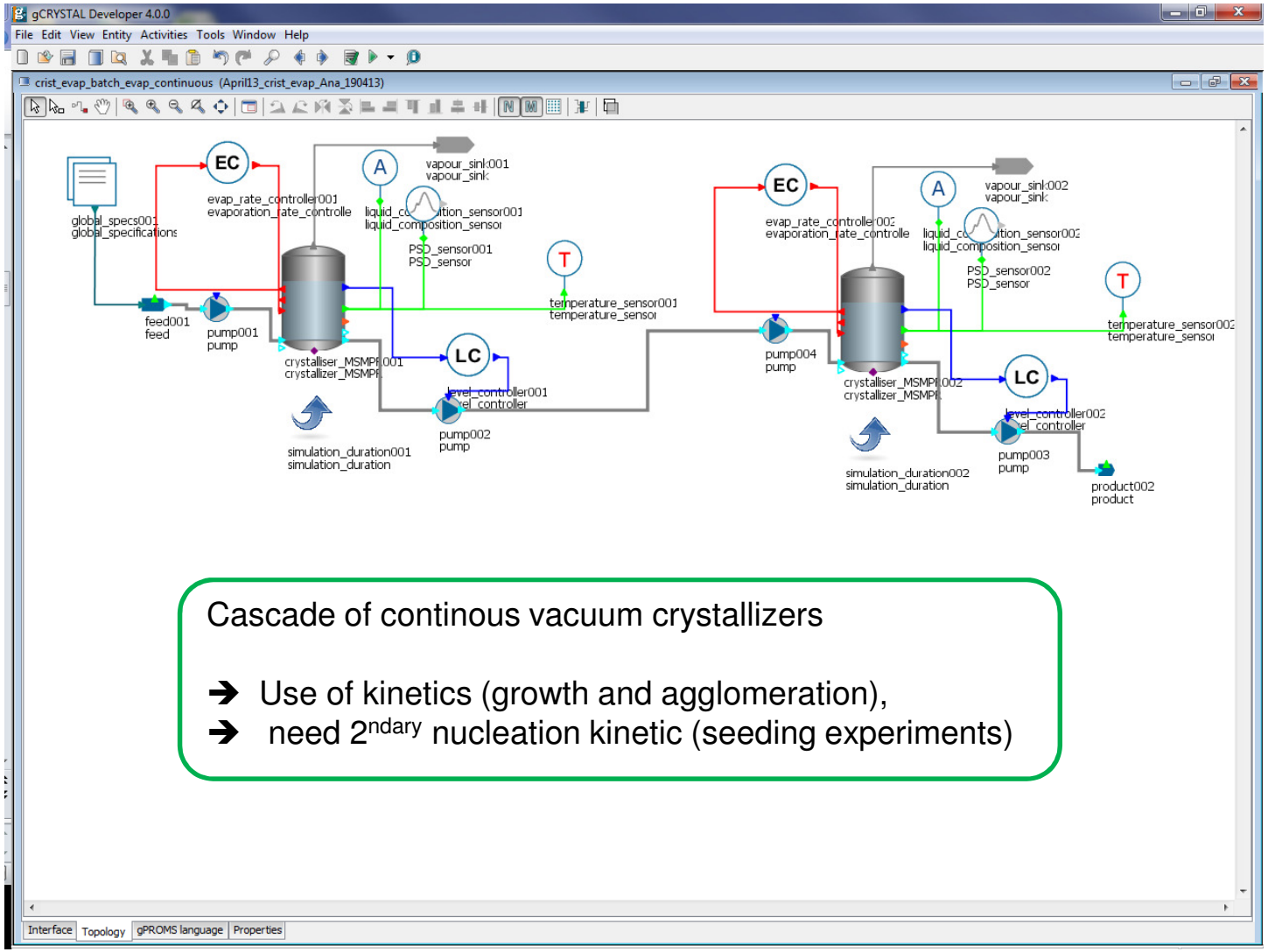
Prospects: New crystallizer configurations



Cascade of continous vacuum crystallizers

➔ Use of kinetics (growth and agglomeration),
➔ need 2ndary nucleation kinetic (seeding experiments)

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Conclusions

- Thanks to a simple “Step-by-Step” approach we managed to simulate the evolution with time of the supersaturation and of the DTC for a batch cooling crystallization
- The kinetic laws available in gCRYSTAL were sufficient to produce a satisfying model
- Using the developer version of gCRYSTAL , a specific heat exchange model was added to the initial one.
- This allowed us to simulate the effect of the scaling mechanism and later to optimize the cooling profile. The production capacity was increased by 10% and 15% of the electricity consumption was saved.

Aknowledgments

Hassan Mumtaz, PhD, Senior Consultant at PSE, Solids Business

P. Carvin, Solvay - Expert in solid processes

E. Chateigner, Solvay - Process Engineering Technician

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Crystallization mechanisms

Kinetic models considered in a 1st step

"Bulid-in" kinetics from gCRYSTAL were used

1ary nucleation $$r_{N1} = \boldsymbol{A_n} \cdot e^{-\frac{\boldsymbol{B_n}}{\ln(c/c^*)}}$$

Linear growth rate $$G = \boldsymbol{k_c} \cdot \frac{2 \cdot M_\omega}{\rho} \left(\frac{c - c^*}{c^*}\right)^{\boldsymbol{j_c}}$$

A_n:	Pre-exponential factor of the primary nucleation	[s-1]
B_n:	Exponential factor of the primary nucleation	[-]
c:	Solute concentration	[kg.kg^{-1}]
c^*:	Solubility in the crystallization medium	[kg.kg^{-1}]
k_c:	Crystal growth factor	
j_c:	Crystal growth supersaturation exponent	
n:	Distribution function	[m^{-1}]
M_w:	Molar mass of the crystals	[kg.mol^{-1}]
t:	Time	[s]
L:	Crystals characteristic size	[m]
G:	Crystal growth rate	[m.s^{-1}]
R_{NI}:	Distribution of the primary nucleation rate	[m^{-1}.s^{-1}]

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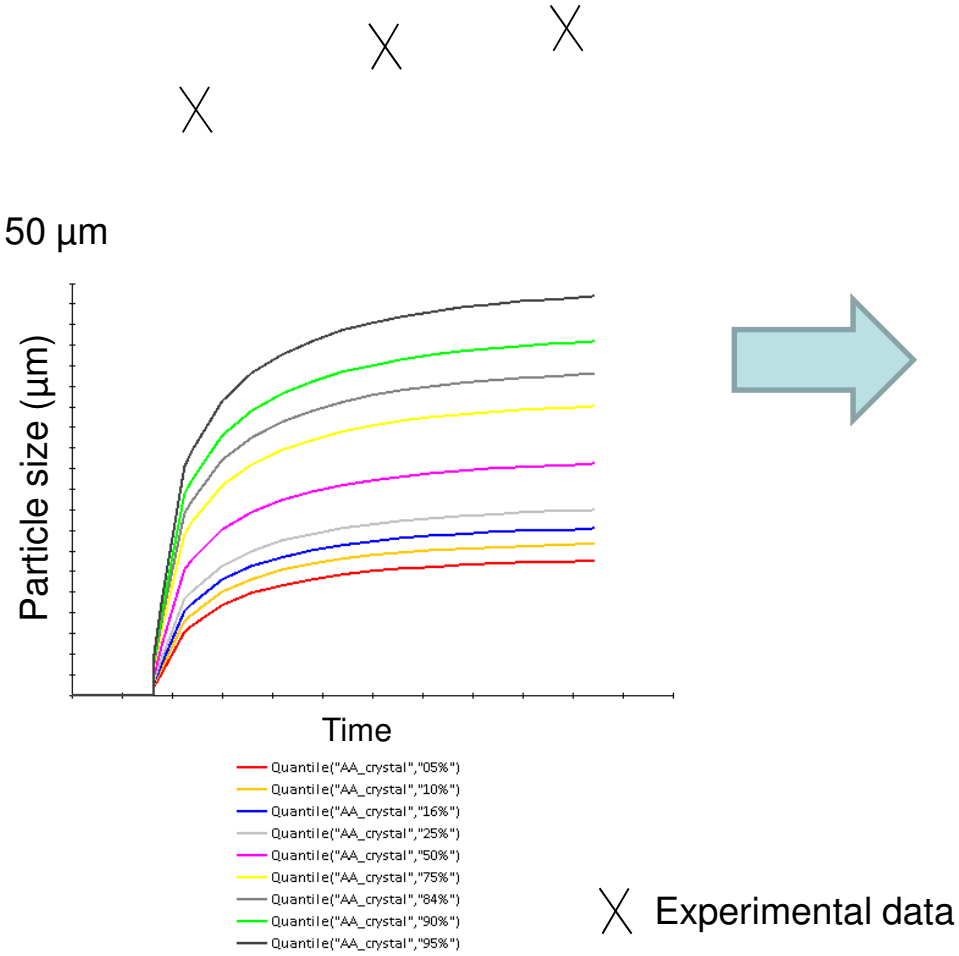
Fitting the experimental results

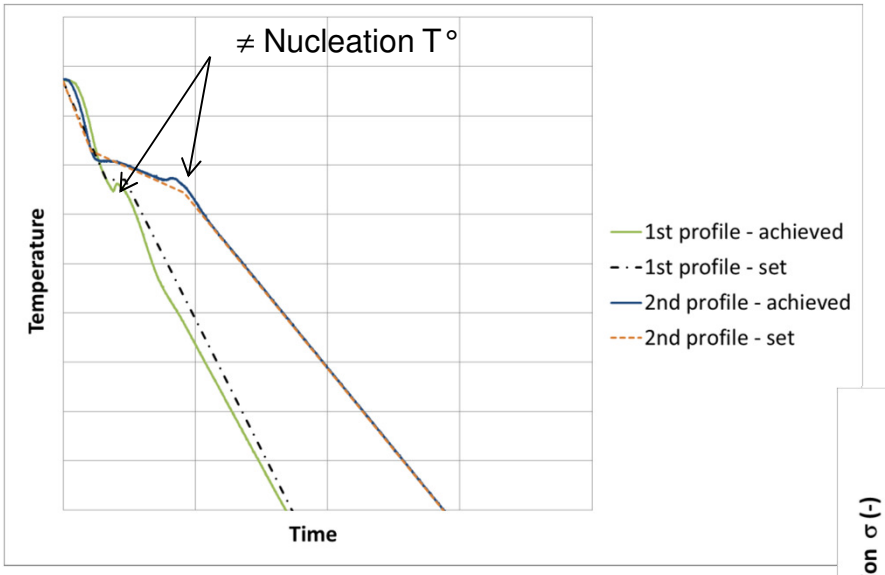


No matching !

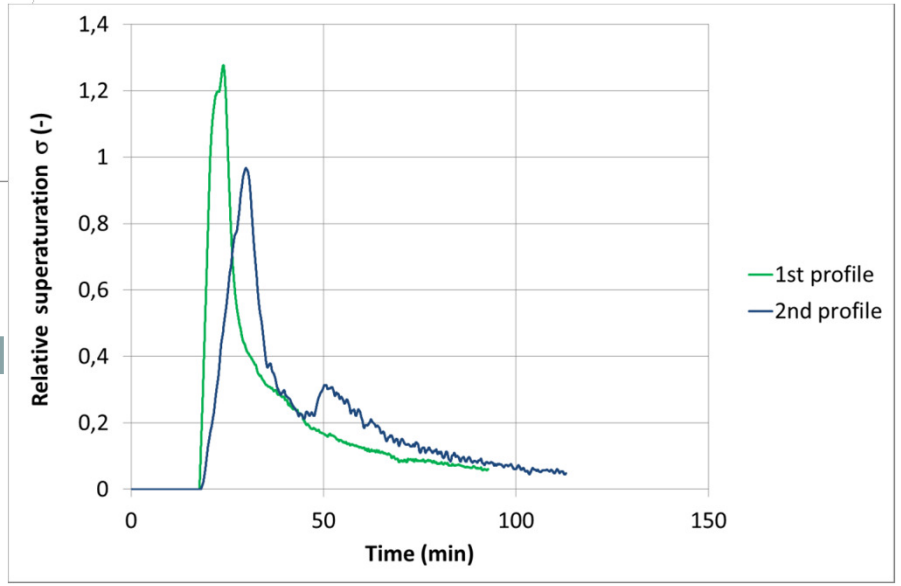
Experimental data

Effects of changing the temperature profile

Temperature profiles

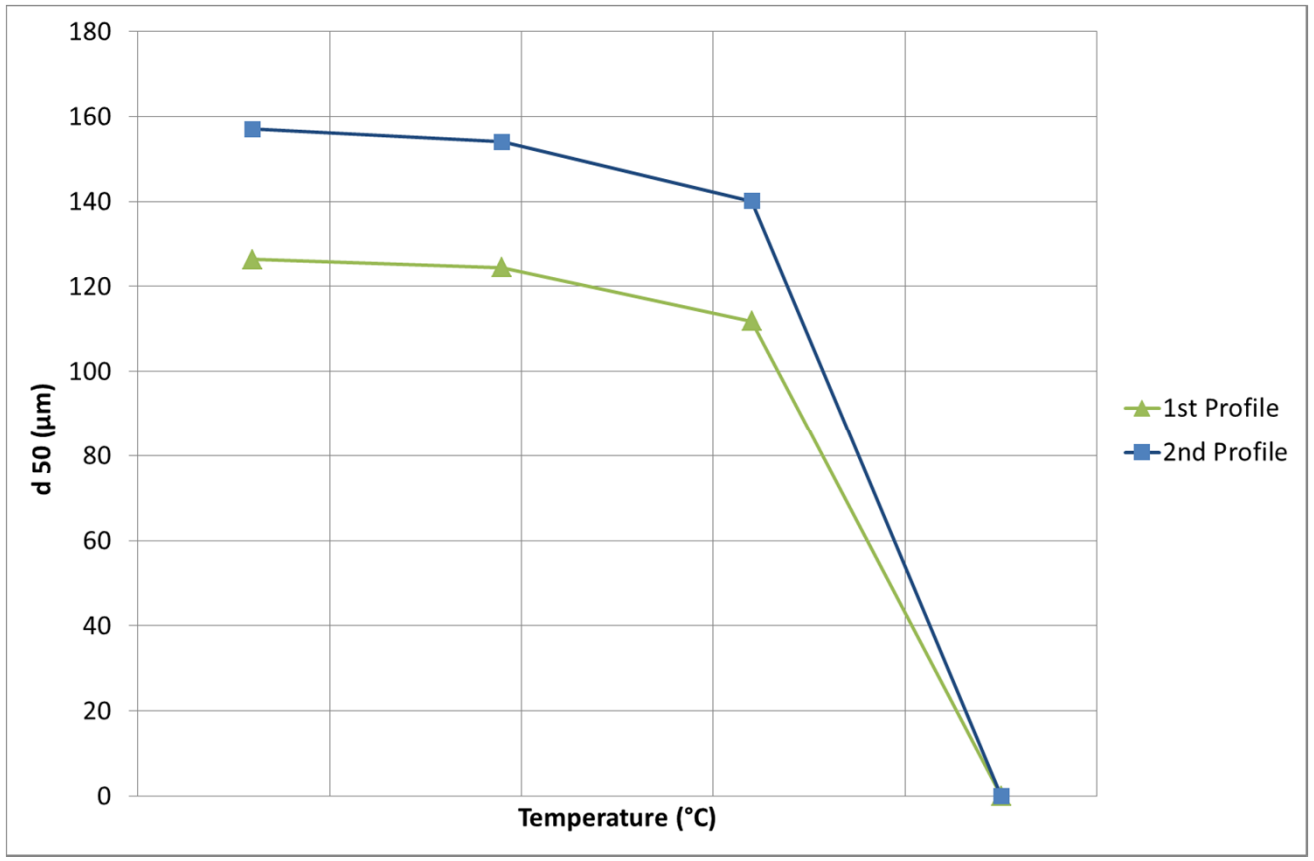


Supersaturation profiles



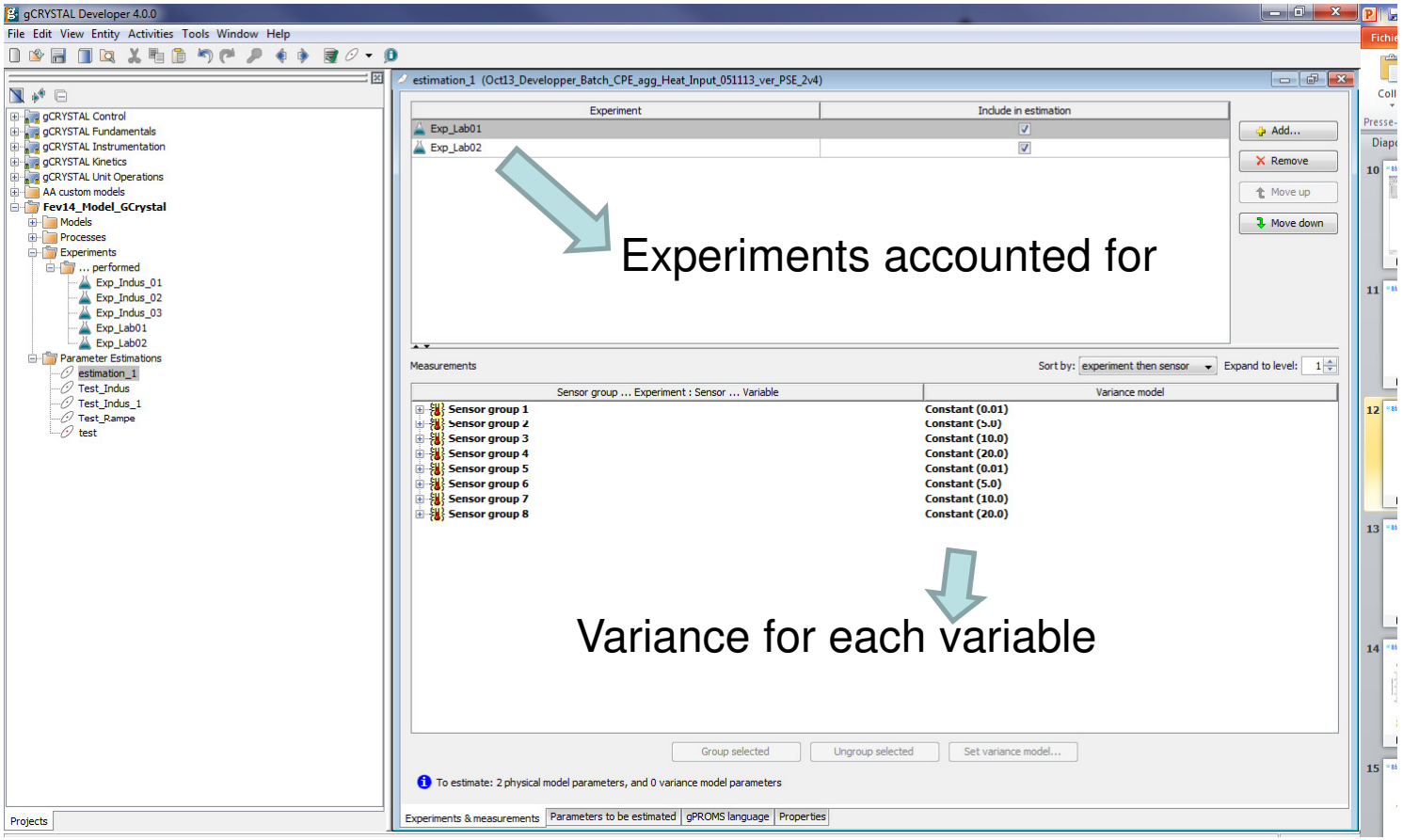
Different crystallization intensities

Experimental results: Evolution of the particle size



Decrease of the crystal size with the increase of the cooling rate

Setting-up the parameter estimation



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