SOLVAY
asking more from chemistry®

Multiscale approach for continuous crystallizer revamp

APMF - Apr 2016

We are a world leader
in the chemical industry

26,000
employees

119
industrial sites

15
major
R&I centers

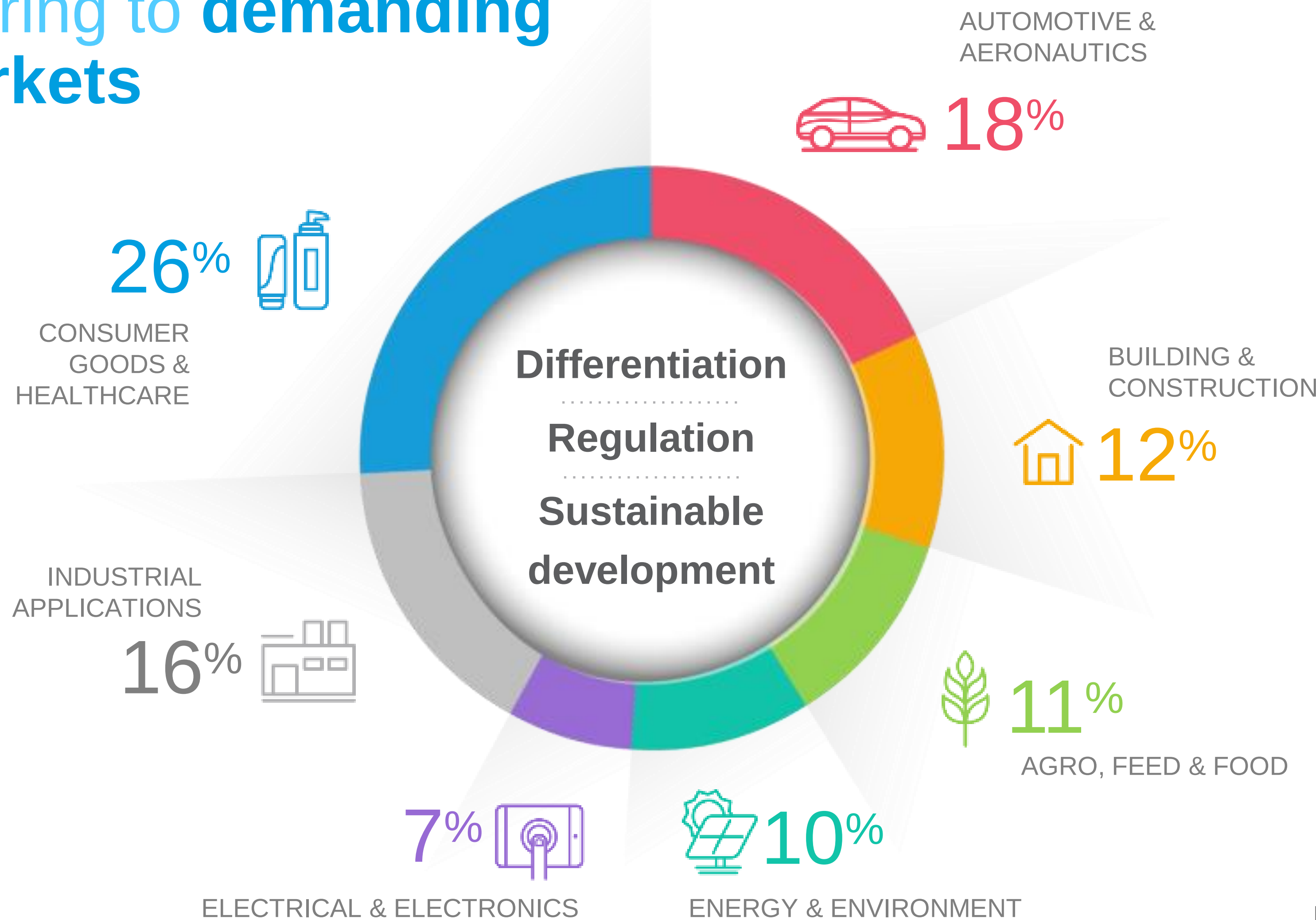
52
countries

€ 10,213
million of net sales

€ 1,783
million of REBITDA

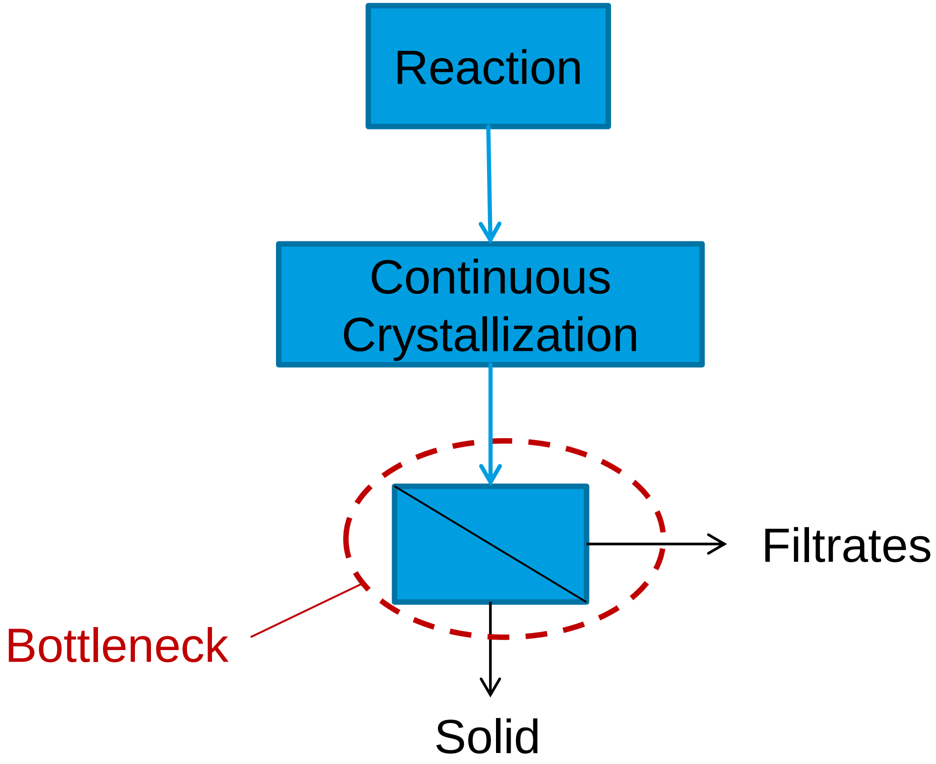
2014 figures

We adapt our product offering to **demanding markets**



Distribution of 2014 net sales

Limitation of the plant production capacity



Objective: Production increase

with limited Investments

- Modification of the current separation system

➔ tests of different operating conditions and filtration medium: No success ☹

➔ A new liquid / solid separation technology has been found, but it is expensive and there are still some technical risks

Improvement of the feed quality
➔ Work on the crystallization operation !

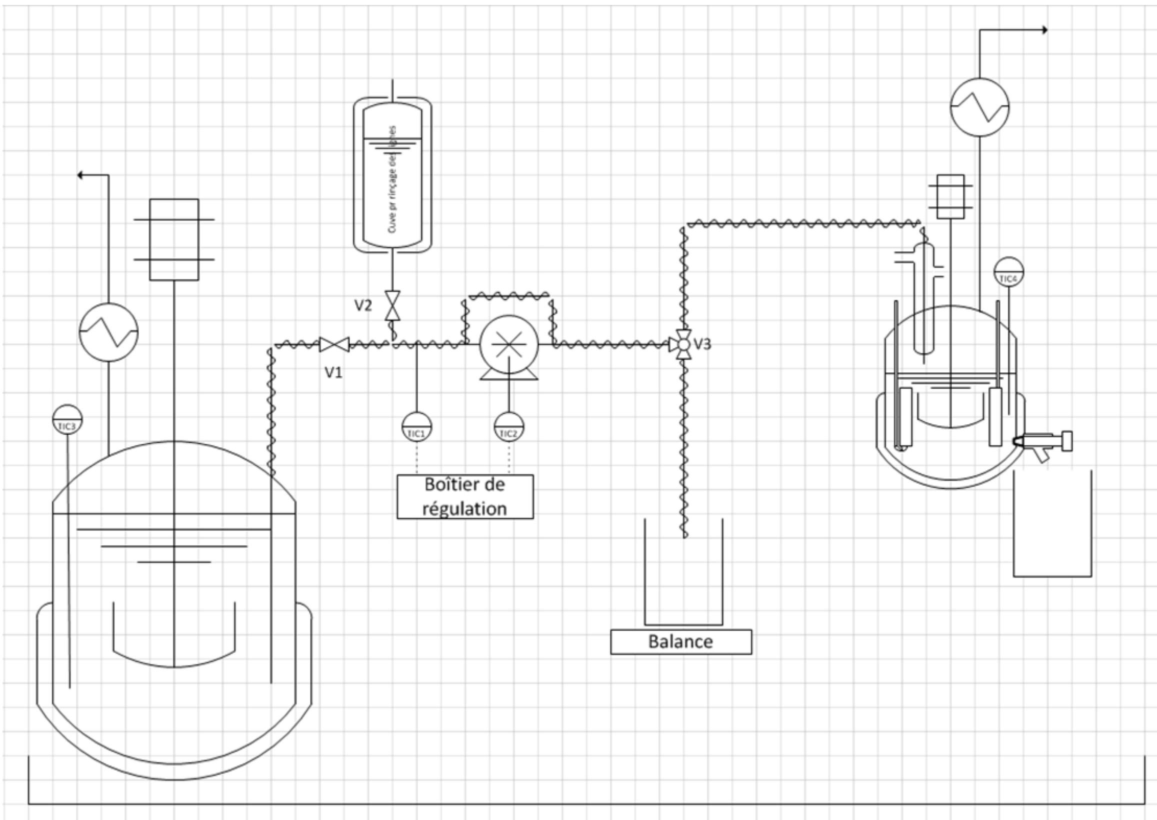
Controling Crystal's filter-ability

Multiscale approach

1. Experiments at lab scale
2. Cystallization modelling within gCRYSTAL – Lab scale
3. Simulation of the industrial crystallizer
4. Input of the CFD / gCRYSTAL Multizonal
5. Optimization of the crystallizer design and operating parameters

Lab scale experiments

Pilote-scale MSMPR - continuously operated crystallizer

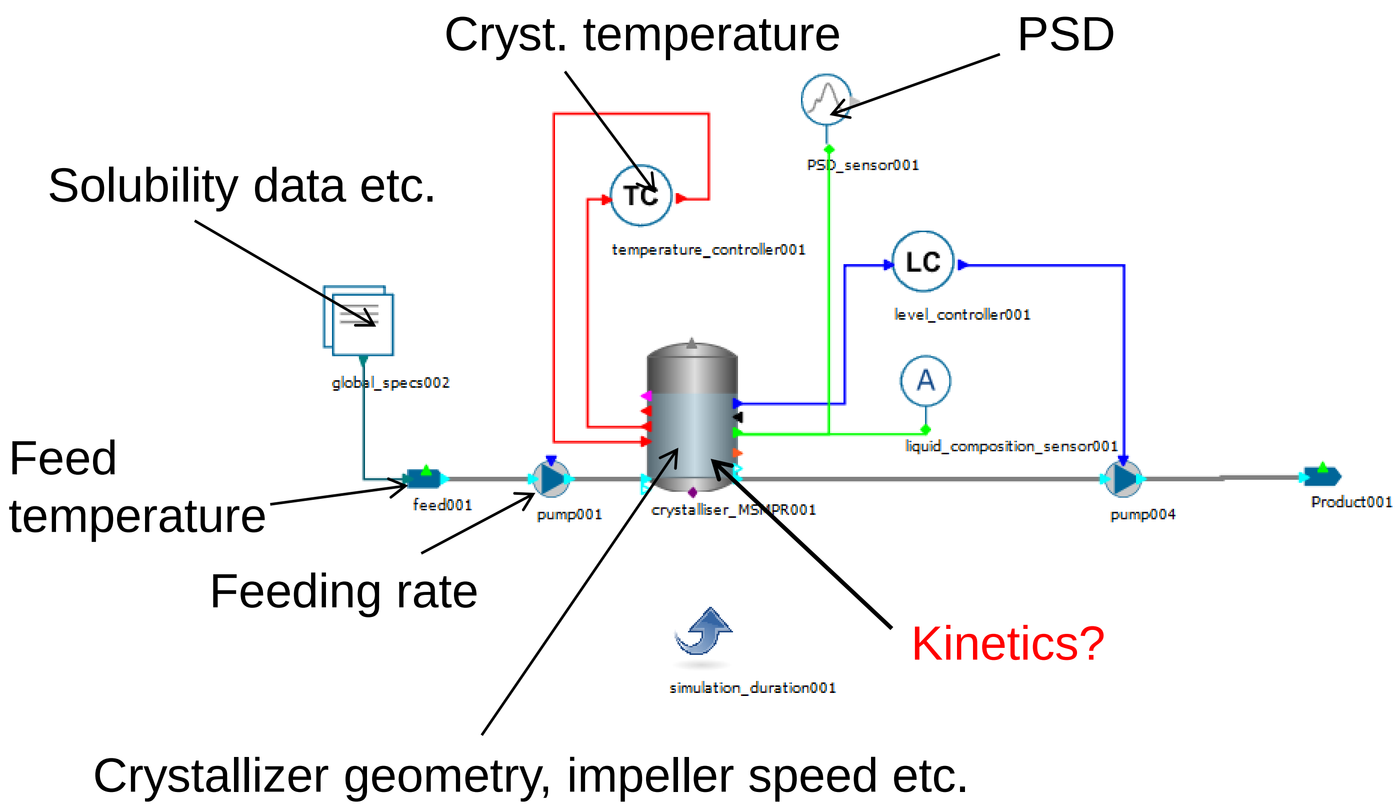


Parameters:

- $T_{cryst.}$
- $T_{feed.}$
- τ_{res}
- C_{solid}
- ω

Modelling continuous crystallization

Lab scale

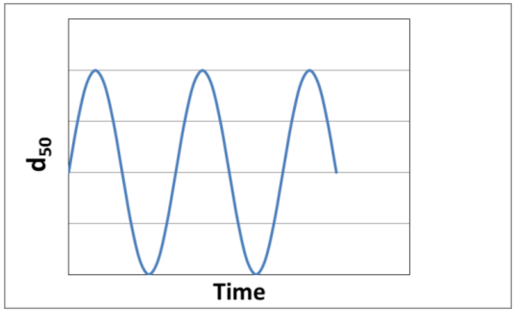


Crystallization kinetics

2^{ndary} nucleation

1 - Activated secondary nucleation

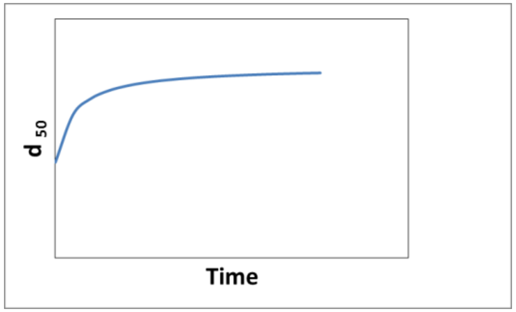
$$J_{sec} = A_t \left[\exp\left(\ln k_s\right) \frac{D_{AB}}{d_m^4} \exp\left(-\pi \left(K \ln\left(\frac{c_c}{c^*}\right)\right)^2 \frac{1}{\nu \ln S_a} \right) \right]$$



Periodic regime

2 - Attrition

$$J_{sec\,ci} = \exp\left(\ln k_{n\_ci}\right) \sigma^{n_{ci}} \frac{N_Q}{N_P} k_v \rho_c \varepsilon \int_{L_{min\_ci}}^{\infty} n \; L^3 \, dL$$



Stable regime

Crystallization kinetics

Growth & Agglomeration

1 – Crystal growth

power law kinetic

$$G = k_g \exp\left(\frac{-E_{A,g}}{RT}\right)\sigma^g$$

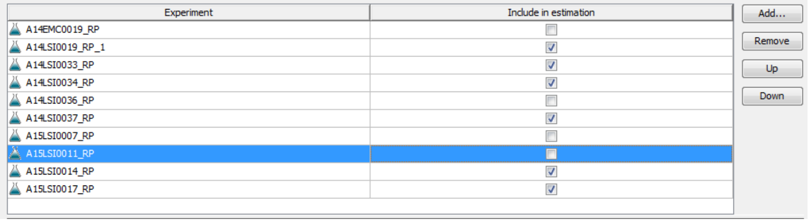
2 – Agglomeration

not in the initial model, because its effect can be difficult to distinguish from crystal growth

Parameter estimation

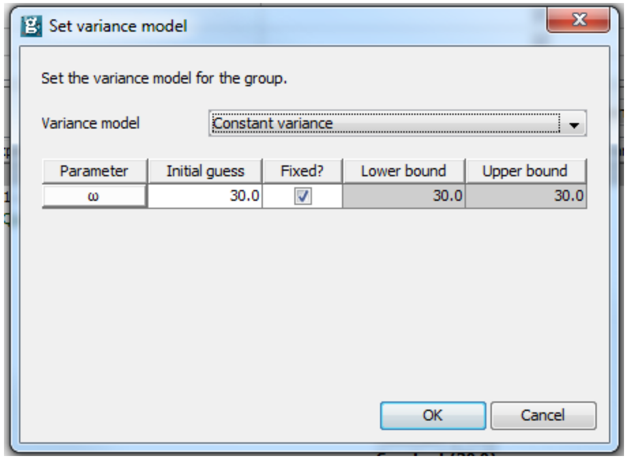
set-up

Choosing the experiments



Experiment	Include in estimation
A14EMC0019_RP	☐
A14LSI0019_RP_1	☑
A14LSI0033_RP	☑
A14LSI0034_RP	☑
A14LSI0036_RP	☐
A14LSI0037_RP	☑
A15LSI0007_RP	☐
A15LSI0011_RP	☐
A15LSI0014_RP	☑
A15LSI0017_RP	☑

Error in each measurement



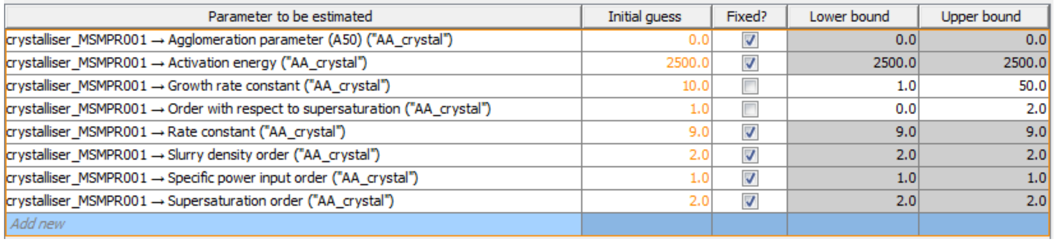
Set variance model

Set the variance model for the group.

Variance model: Constant variance

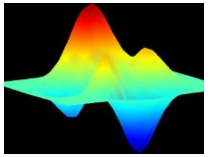
Parameter	Initial guess	Fixed?	Lower bound	Upper bound
ω	30.0	☑	30.0	30.0

Kinetic parameters I.C. & boundaries



Parameter to be estimated	Initial guess	Fixed?	Lower bound	Upper bound
crystalliser_MSMPR001 → Agglomeration parameter (A50) ("AA_crystal")	0.0	☑	0.0	0.0
crystalliser_MSMPR001 → Activation energy ("AA_crystal")	2500.0	☑	2500.0	2500.0
crystalliser_MSMPR001 → Growth rate constant ("AA_crystal")	10.0	☐	1.0	50.0
crystalliser_MSMPR001 → Order with respect to supersaturation ("AA_crystal")	1.0	☐	0.0	2.0
crystalliser_MSMPR001 → Rate constant ("AA_crystal")	9.0	☑	9.0	9.0
crystalliser_MSMPR001 → Slurry density order ("AA_crystal")	2.0	☑	2.0	2.0
crystalliser_MSMPR001 → Specific power input order ("AA_crystal")	1.0	☑	1.0	1.0
crystalliser_MSMPR001 → Supersaturation order ("AA_crystal")	2.0	☑	2.0	2.0
Add new				

Running the optimization routine



1. Experiments | **2. Lab. model** | 3. Indus. model | 4. CFD Input | 5. Optimization

Simulations vs Experiments

Good Fit at lab scale

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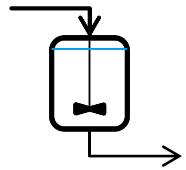
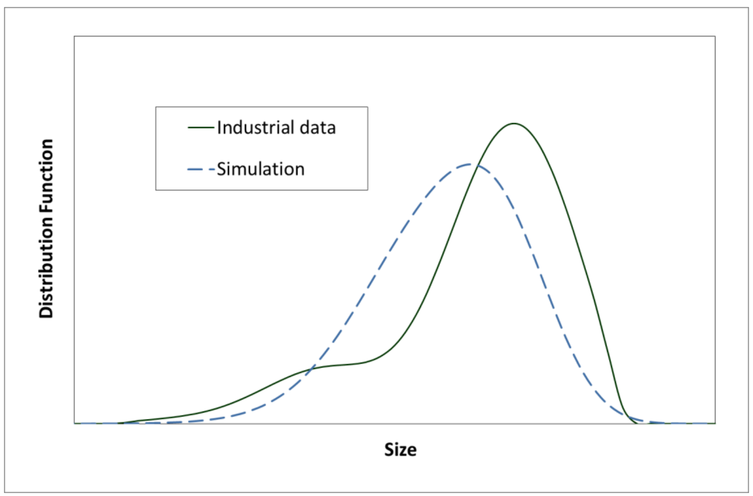
Simulation of the industrial crystallizer
real operating conditions



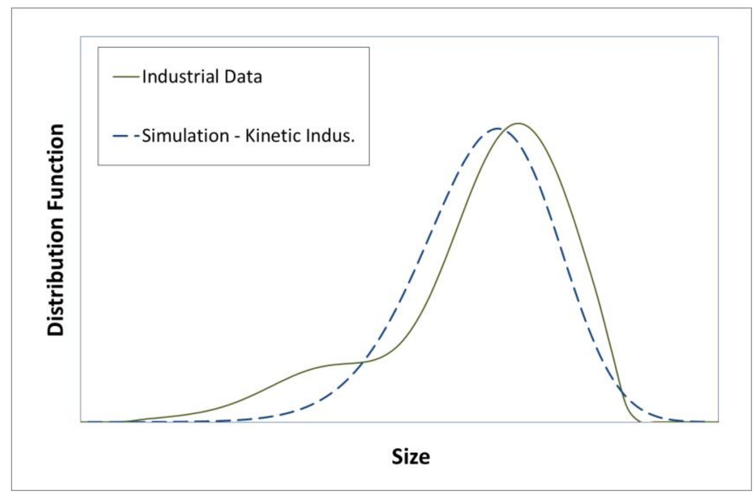
- Real dimensions, operating conditions etc.
- Kinetics from lab data

Simulation with Lab kinetics



Bad fit !

Identification of industrial kinetics



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Test of new operating conditions

Discrepency between simulation and industrial test

Modification of the temperature conditions

Test at industrial scale

Bad fit with the simulation and
No real Improvement !

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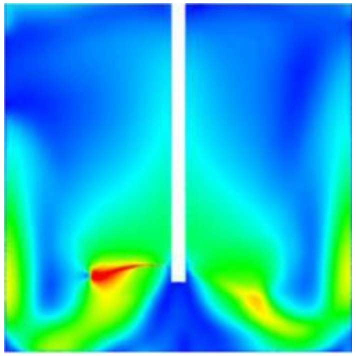
Using CFD simulation in g-CRYSTAL

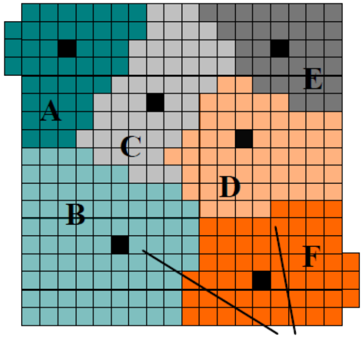
g-CRYSTAL Multizonal

CFD simulation

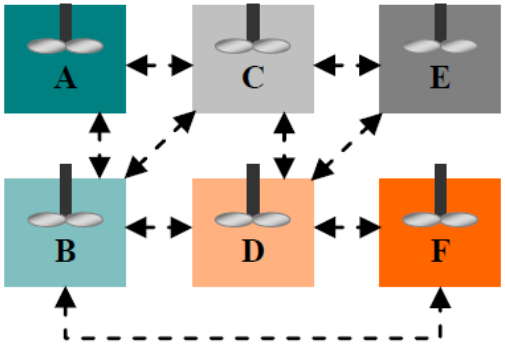


1 crystallization stage

Choosing the « Zones »

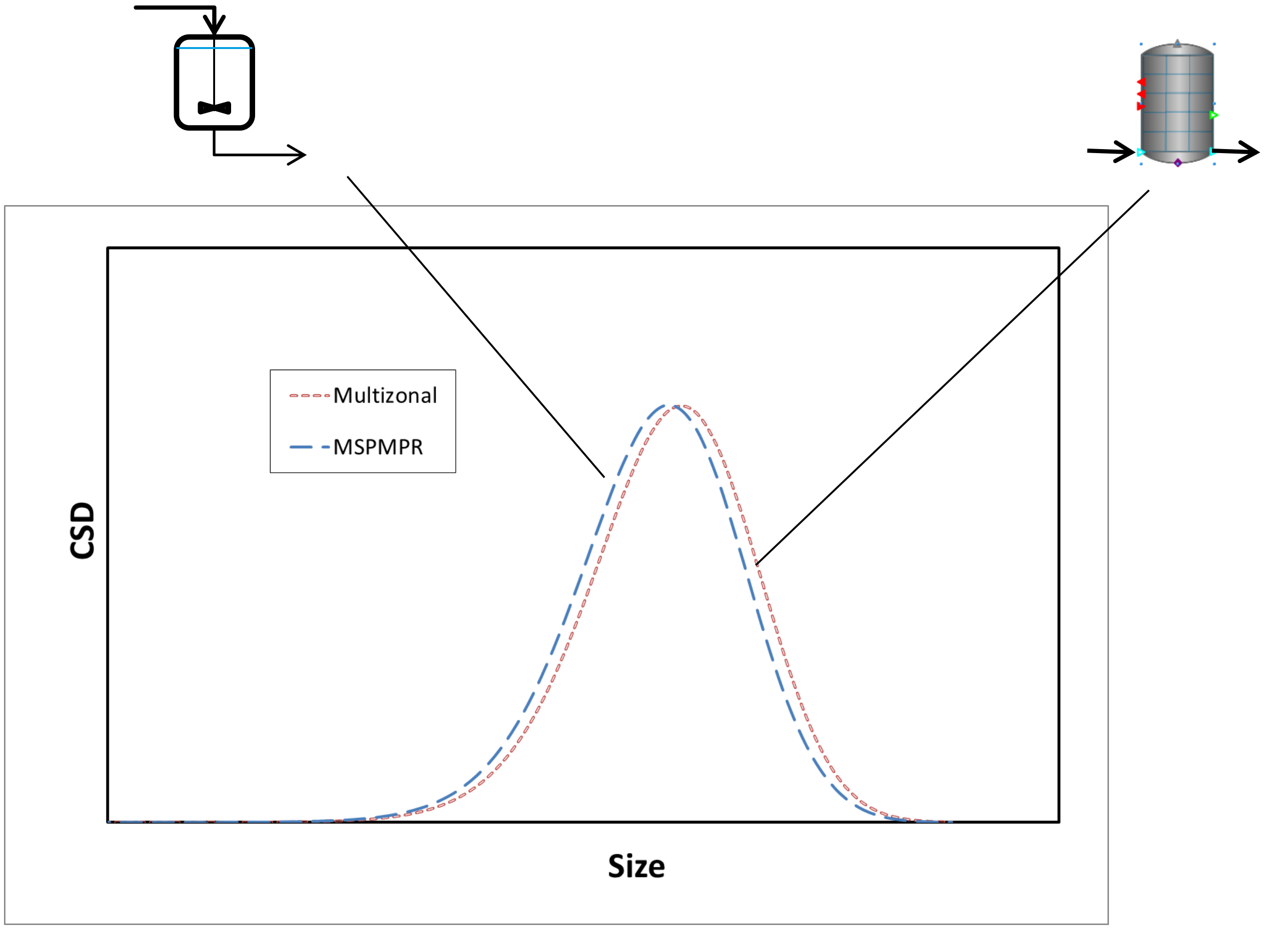


Extraction of CFD data



MSMPR vs Multizonal

Small difference with the simple model



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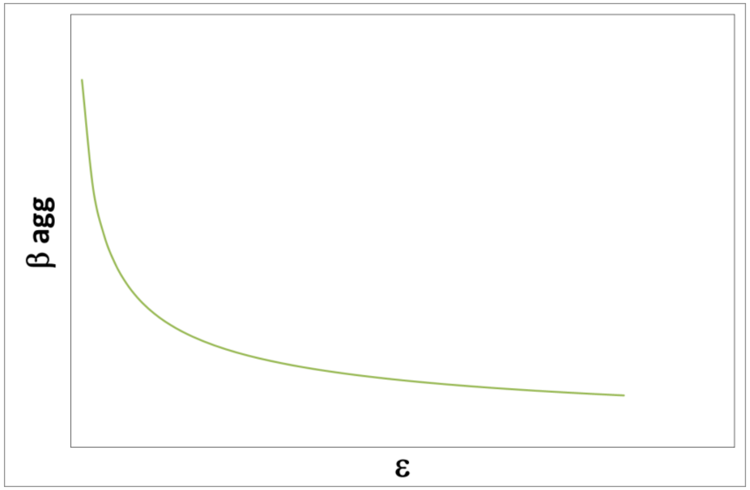
Model improvement

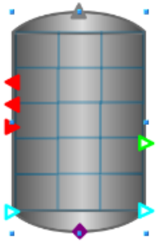
Agglomeration mechanism

Mumtaz Agglomeration kernel

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

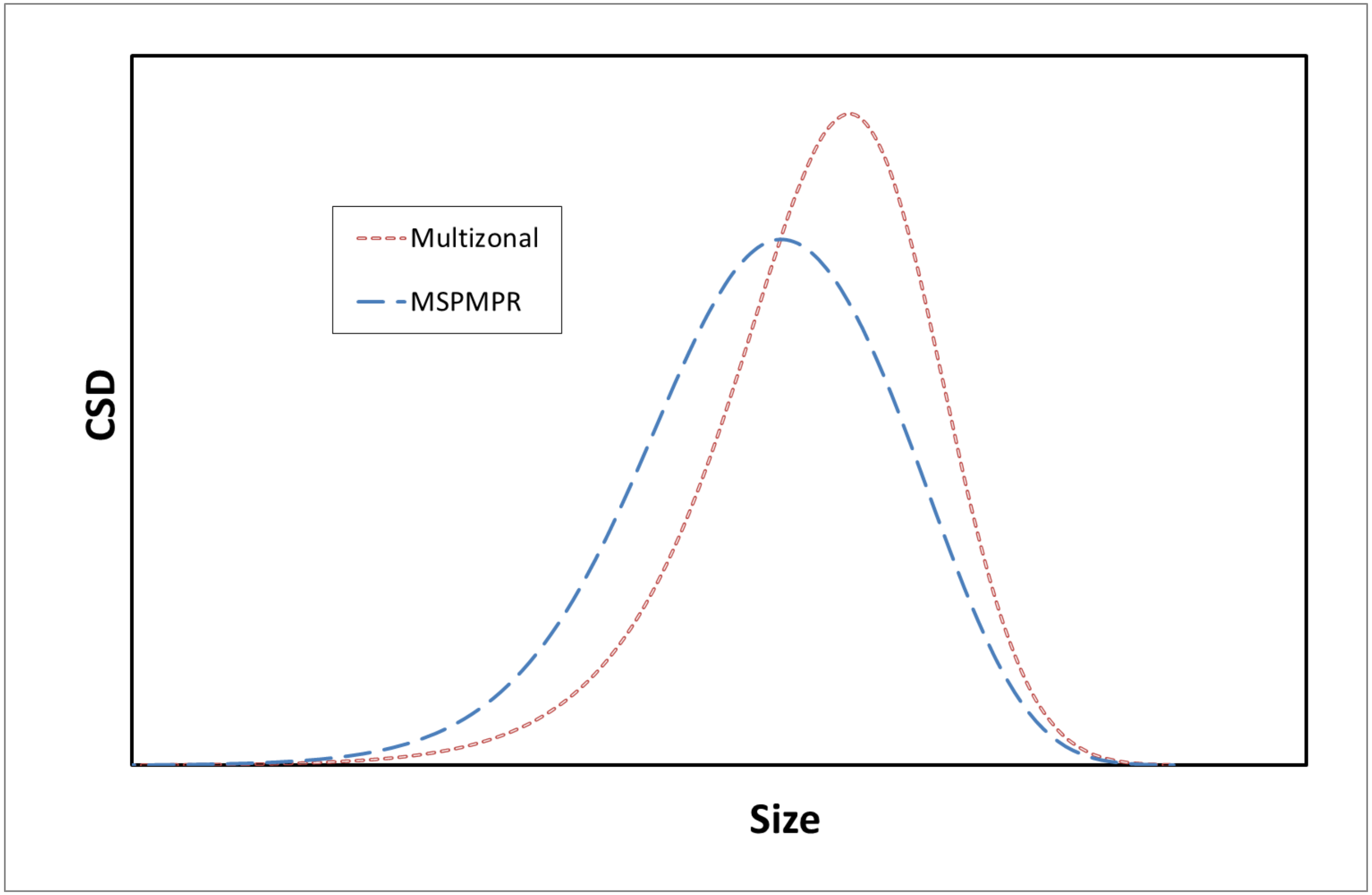


Non linear behaviour



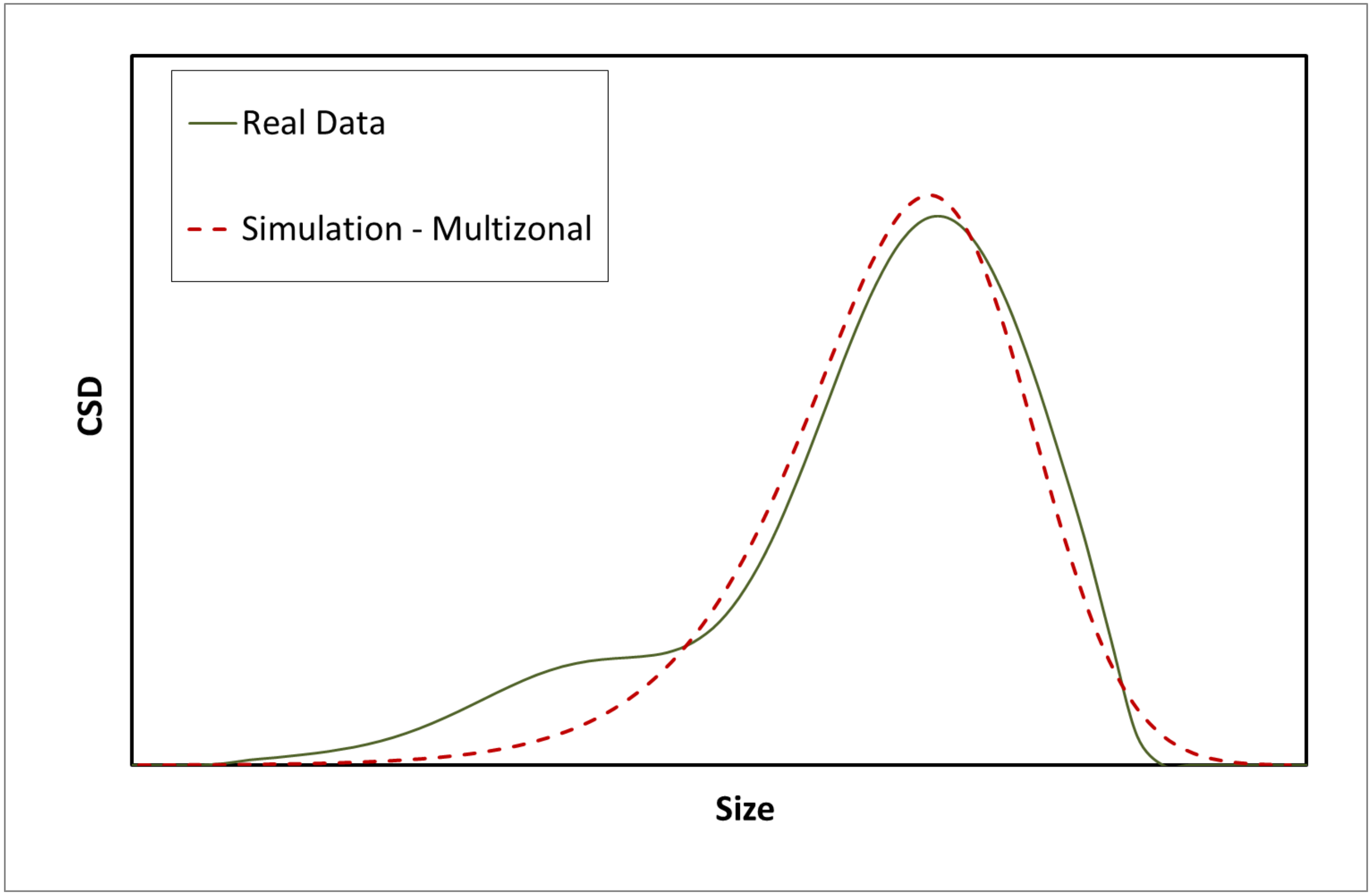
Effect of the hydrodynamic

Industrial scale



1. Experiments 2. Lab. model 3. Indus. model **4. CFD Input** 5. Optimization

Better fit with the Multizonal Crystallizer
Industrial Scale



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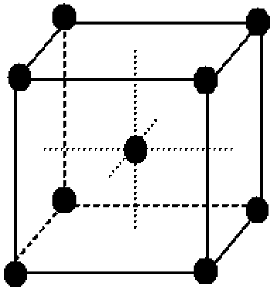
Designing Crystallizer Modifications

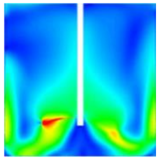
DOE Approach

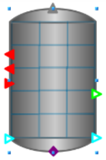
1.
- Inlet position
- Impeller geometry
- Average Power Input **ε**

→ 2^3 DOE

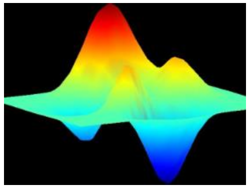


2. Monophasic CFD Simulations → Multizonal Simulations





3. Analysis of the DOE → Best set of modifications



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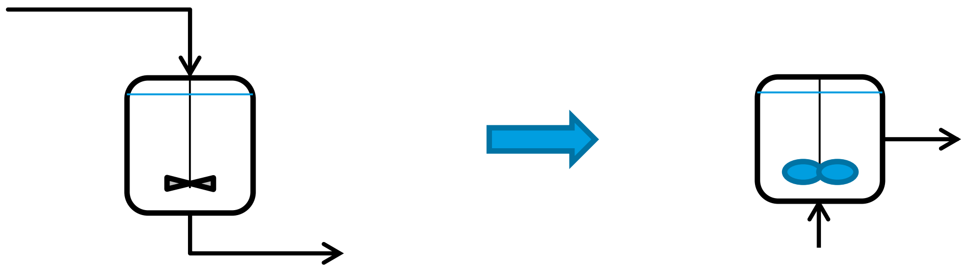
SOLVAY asking more from chemistry®

Capacity increase after modifications

At low investment



+

ε

Narrower CSD

Higher filter-ability

We achieved a 25% increase in filtration capacity

Conclusions

- CFD + Population Balance Modeling is a very efficient tool for
  - Optimizing existing crystallizer ➔ reduce experimental costs (time and money !)
  - Designing New equipments ➔ Allow the implementation of breaktrough technologies

&
Future Work

- A purification model can be extracted from the population balance

$$C_i = f(r_{N2}, G, Agg)$$

⬇

optimization with the same model

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Thank You for your Attention !