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► **To cite this version:**

Lucas Brivadis, Vincent Andrieu, E Chabanon, E. Gagnière, Nouredine Lebaz, et al.. Soft sensors for a batch crystallization process. Cristal 9, May 2019, Nancy, France. hal-02100158

**HAL Id: hal-02100158**

**<https://hal.science/hal-02100158v1>**

Submitted on 23 Jul 2019

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# Soft sensors for a batch crystallization process

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## Introduction

- **Context:** During a batch crystallization process, the online control and estimation of the **Particle Size Distribution (PSD)** is critically important for the industry.
- **Problem:** No online sensor can measure the PSD.
- **Objective:** To build an online soft sensor that estimates the PSD, based on either the **solid concentration** or the **FBRM® technology**.
- **What is new?** No use of a nucleation model.

## Model of the crystallization process

### Hypothesis:

- $[t_0, t_1]$ : Process duration.
- $[x_{\min}, x_{\max}]$ : **Spherical crystals** radius range.
- $z(t, \cdot)$ : PSD at time  $t$ .
- $G(t)$ : **Growth kinetic** (McCabe hypothesis).
- $u(t)$ : **Nucleation** at  $x_{\min}$ .

### Population Balance Equation:

$$\begin{cases} \partial_t z(t, x) = -G(t) \partial_x z(t, x) \\ z(t_0, x) = z_0(x) \\ z(t, x_{\min}) = u(t). \end{cases}$$

**Measurement :** The solid concentration (up to some constant):

$$y(t) = \int_{x_{\min}}^{x_{\max}} z(t, x) x^3 dx.$$

How to estimate the state  $z$  from the measurement  $y$  ?

## Luenberger original idea

**Two steps** strategy:

- 1 Estimate a function  $Tz$  of the state.
- 2 Invert this mapping  $T$ .

## Designing an online soft sensor (Luenberger-like observer)

**Step 1:** Estimation of  $Tz$ . Consider, for  $\lambda < 0$ ,

$$\dot{\zeta} = \lambda \zeta + y, \quad \zeta(t_0) = \zeta_0.$$

How to have  $(\zeta - Tz) \xrightarrow[+\infty]{} 0$  ? It is sufficient to satisfy

$$\frac{dTz}{dt} = \lambda Tz + y.$$

For the solution  $a$  of some PDE, one can choose

$$Tz(t) = \int_{x_{\min}}^{x_{\max}} a(t, x) z(t, x) dx.$$

**Step 2:** Inversion of  $T$  ?

## Tikhonov regularization method

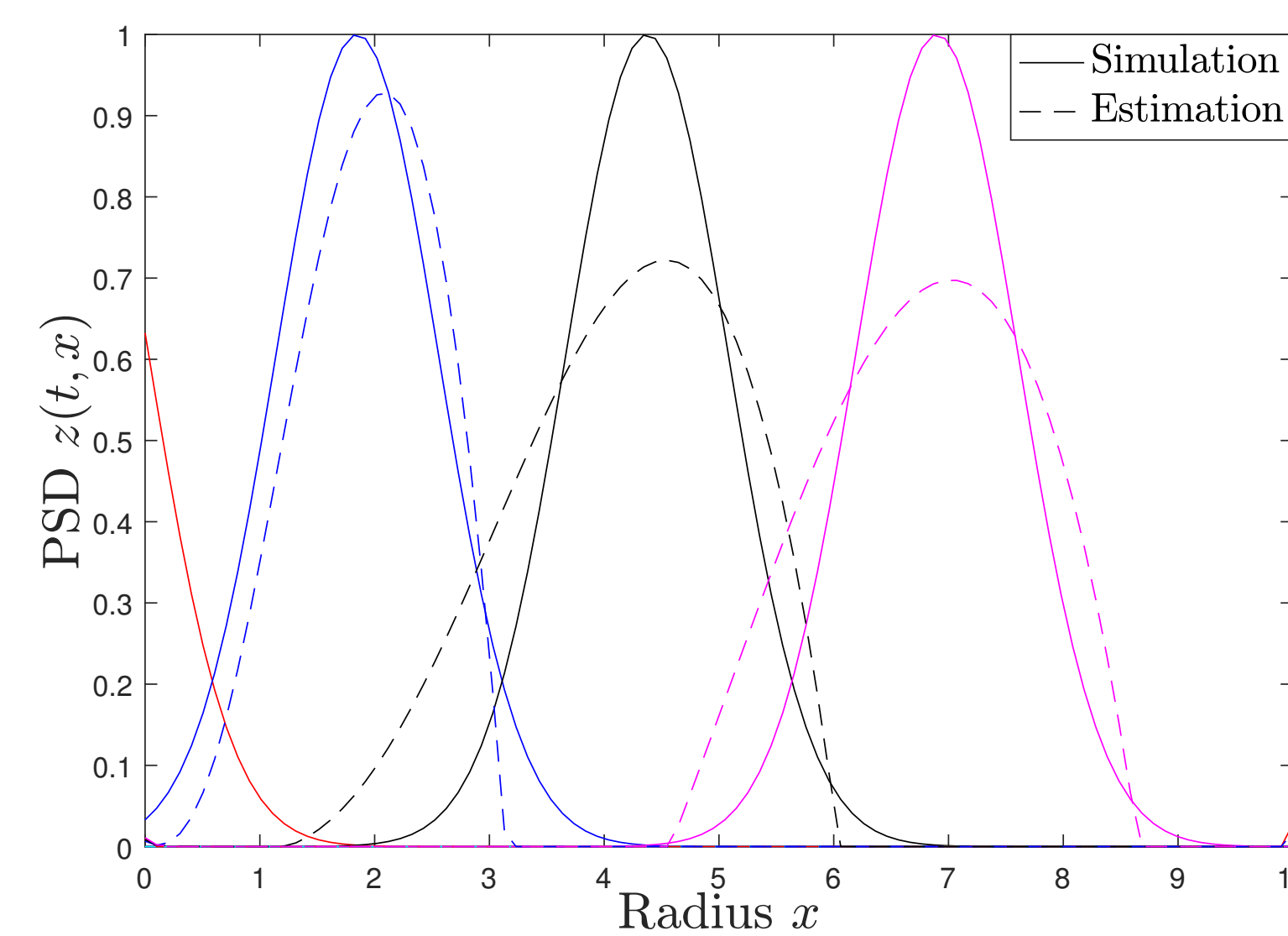
▪ **Problem:** Numerically estimate the solution of

$$\operatorname{argmin}_{\hat{z}(t)} \|T\hat{z}(t) - \zeta(t)\|_2^2$$

▪ **Idea:** Add a regularization term (**measure confidence**):

$$\operatorname{argmin}_{\hat{z}(t)} \|T\hat{z}(t) - \zeta(t)\|_2^2 + \delta^2 \|\hat{z}(t)\|_2^2$$

## From solid concentration to PSD



Soft sensor based on the solid concentration with  $\delta = 0.05$

### Limits of the soft sensor:

- Need an estimation of the growth kinetic  $G$ .
- Choice of  $\delta$ .
- Proof that  $y$  is not sufficient to determine  $z$  uniquely (**no injectivity**).
- Need a new physical sensor.

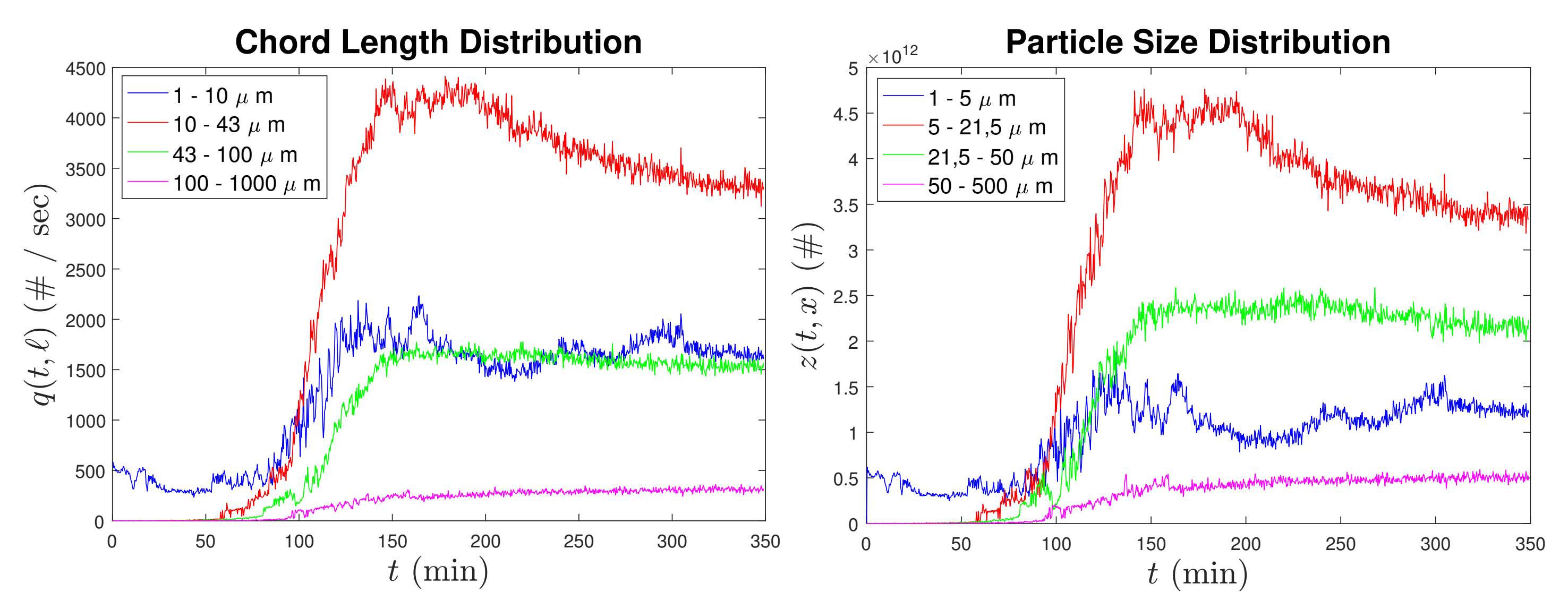
## Model of the FBRM® technology

The probe uses a focused laser beam that scans across the particles. We model the measure as the **Chord Length Distribution (CLD)**:

$$q(t, \ell) = C \int_{x_{\min}}^{x_{\max}} k(\ell, x) z(t, x) dx.$$

- $k(\ell, x)$  can be determined through a probabilistic analysis.
- $C$  is an unknown constant. The solid concentration can be used to estimate  $C$ .
- One can apply the Tikhonov method directly on  $q$ , without using a soft sensor.
- Proof that  $q$  is sufficient to determine  $z$  uniquely (**injectivity**).

## From CLD to PSD



## Conclusion

- We propose two new soft sensors for the online estimation of the PSD in a batch crystallization process.
- The first one is based only on the solid concentration. This method has been tested on simulations. For better results, we prove that a new sensor is required.
- The second one is based on a mathematical modeling of the FBRM® technology, and has been experimentally tested. It allows to reconstruct the PSD from the CLD, both numerically and theoretically.

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Acknowledgments:

This work has been funded via the project PEPS CNRS ELTaCriB Défi INFINITI.