"In-line Crystal size analysis with a highly adaptable and industrially approved sensor based on ultrasonic Extinction"

Paper published on

15^{th} International Symposium on Industrial Crystallization

15 - 18 September 2002
Sorrento / Italy

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In-line Crystal size analysis with a highly adaptable and industrially appoved sensor based on ultrasonic Extinction

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Ultrasonic Extinction is a relatively new method for the determination of particle or crystal size distributions (CSD) in highly concentrated suspensions and requires the design of rugged instruments ideal for process application. Following the description of the technology and technical realisation, results of successful installations will be presented to show that Ultrasonic Extinction is a proven technology for determination of CDS in crystallization processes.

1. INTRODUCTION

The continuously growing demands for higher product quality and production efficiency require the extension of processing methods to their technical limitations. But where are these limitations? In order to answer this question reliable and precise instrument for the determination of the product and process quality are required. In the field of crystallization one of the elementary information requirements about this quality is the CSD that has to be monitored in order to reach a better understanding of the process and to optimise the final product quality. That includes not only the CSD at the end of the line but also the monitoring of nucleation, growing and dynamic behaviour of crystals even during the process, where a single-minded control of the crystallization regarding the desired CSD can be taken, is required.

Present methods for crystal size analysis (CSA) are mostly based on time consuming lab analyses, where samples have to be taken out of the process and conditioned to the requirements of the measuring principles. Any treatment of samples, like dilution or drying, significantly influences the CSD of the sample. Further, the sampling sequence is limited to the capacity of the operator, which usually leads to poor sampling rates.

Thus, a reliable and suitable control of crystallization processes, its dynamic and stability should fulfil the following demands:

- measuring directly in the crystallizer or re-circulation pipe / no extraction of samples
- measuring in original conditions / no manipulation of CSD
- high measuring frequency / high timely resolution
- high measuring volume / best statistics
- high information depth / best monitoring

The crystals should not be required to adapt to the measuring device, but the analytical instrument has to adapt to the process, the in-line equipment has to operate under process conditions like

- high crystal concentrations
- change of crystal concentrations
- high temperatures
- changing of temperatures
- broad crystal size range
- change of viscosity
- easy to install
- easy to access
- mechanically and chemically resistant
- various pressures

The following pages introduce the well established in-line particle size analyser OPUS (**O**nline **P**article Size Analysis with **U**ltrasonic **S**pectrometry) that is designed to operate under the described conditions as it is not based on optical principles.

2. PRINCIPLE OF ULTRASONIC EXTINCTION

The design of an instrument for the determination of frequency dependent Ultrasonic Extinction is schematically presented in Fig. 1.

An electrical high frequency generator is connected to a piezoelectric ultrasonic transducer. The generated ultrasonic waves are coupled into the suspension and interact with the suspended particles. After passing the measuring zone the ultrasonic waves are received by an ultrasonic detector and converted into an electrical signal. The extinction of the ultrasonic waves is calculated from the ratio of the signal amplitudes on the generator and detector side

The Ultrasonic Extinction of a suspension of mono-disperse particles can be described by the Lambert-Beer law according to Riebel (1988).

$$-\ln\left(\frac{I}{I_0}\right)_{f_i} = \Delta l \cdot C_{PF} \cdot K(f_i, x) \qquad (1)$$

The Extinction $-\ln(I/I_0)$ at a give frequency f_i is linear dependent on the thickness of the suspension layer Δl, the projection area-concentration C_{PF} and the extinction coefficient K.

In a poly-disperse system the extinction of single particles overlays:

$$-\ln\left(\frac{I}{I_0}\right)_{f_i} = \Delta l \cdot C_{PF} \cdot \int_{x_{\min}}^{x_{\max}} K(f_i, x) \cdot q_2(x) dx \qquad (2)$$

The integral in equation 2 can be substituted by a sum as a first approach:

$$-\ln\left(\frac{I}{I_0}\right)_{f_i} \cong \Delta l \cdot C_{PF} \cdot \sum_j K(f_i, x_j) \cdot q_2(x_j) \Delta x \qquad (3)$$

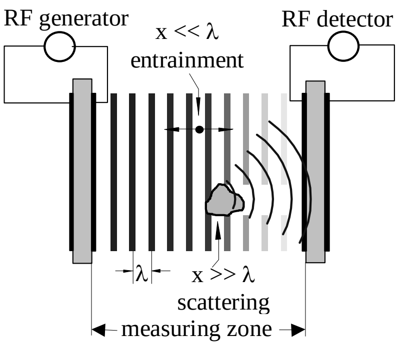

Fig.1: Schematic of the measurement principle

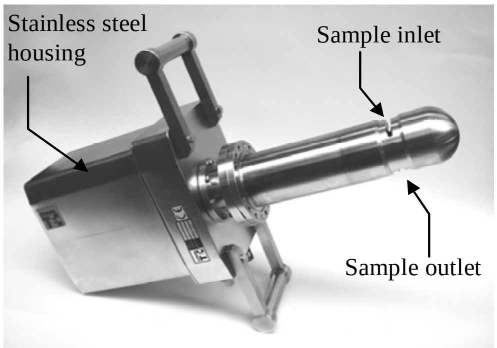

Fig.2: Technical realisation of Ultrasonic Extinction for in-line particle size analysis

If now extinction measurements are performed at various frequencies, this results in a linear equation system:

$$\begin{pmatrix} m(f_1) \\ \vdots \\ m(f_j) \end{pmatrix} = \Delta l \cdot C_{PF} \cdot \begin{pmatrix} K_{1,1} & \cdots & K_{1,j} \\ \vdots & \ddots & \vdots \\ K_{i,1} & \cdots & K_{i,j} \end{pmatrix} \cdot \begin{pmatrix} q_{21} \cdot \Delta x_1 \\ \vdots \\ q_{2i} \cdot \Delta x_i \end{pmatrix} \qquad (4)$$

This equation system is numerically unstable and must be solved by suitable algorithms.

3. TECHNICAL REALISATION OF THE MEASURING PRINCIPLE FOR IN-LINE APPLICATIONS

Fig. 2 presents the Sympatec OPUS System. The instrument is designed as a finger probe and can be adapted to nearly all kind of process pipes or vessels using a DN 100 flange. OPUS is designed for fully automated real-time particle size analysis in process environment. As an option OPUS is available in an explosion proof version as well (Zone 1 ambient, Zone 0 in the measuring zone).

Since OPUS is available in different lengths from 330 to 3.500mm (measured from flange to tip) the instrument can be applied to a variety of processes. For smaller pipe diameters adapters covering process pipes down to DN10 are available. Using an automatic cleaning system (Docking-Positioner & Cleaner) the probe can be moved out of the process pipe or vessel for cleaning, inspection or maintenance without the need to shut down the process. OPUS has been applied successfully to different kinds of crystallization processes (Neumann, 2001).

Hence, OPUS operates under process conditions and fulfils the above listed requirements like

- high crystal concentration 1 – 70 % Vol. (pure solid)
- high temperatures between 0 – 120 °C
- broad measuring range 0.01 – 3000 µm
- easy to access (e.g. Docking Positioner & Cleaner)
- high viscosities
- pressure from 0 – 40 bar

- easy adaptation by large variety of adapters from DN 10 - 200 and different probe lengths from 330 – 3500 mm
- pH-values between 1 – 14
- highly resistant due to the applied materials in contact with the product like stainless steel, Teflon ®, Sigradur ®

4. OPUS RESULTS FROM CRYSTALLIZATION PROCESSES

4.1 Pilot plant application for Research & Development

Fig. 3 shows an installation example in a pilot plant for R & D purposes, where different cooling ramps of a mineral oxidizer crystallization had been tested with respect to the required final CSD. Here, OPUS is mounted in a Stand-By-Rack and installed in a re-circulation loop with a FT 15 adapter. After going through a certain cooling ramp the suspension is circulated through the sensor where CSA are performed. CSD results obtained from several cooling ramps are presented in figure 4.

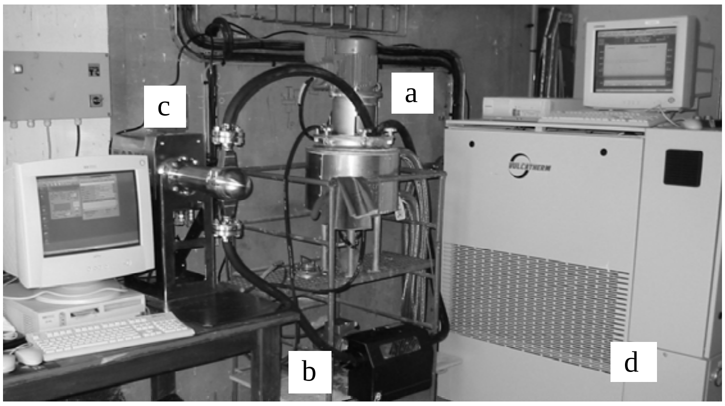

Fig. 3: Pilot plant installation of OPUS for R&D purposes of mineral oxidizer. a) Crystallizer (14 l) with stirrer, b) Peristaltic pump, c) OPUS, d) Heat control

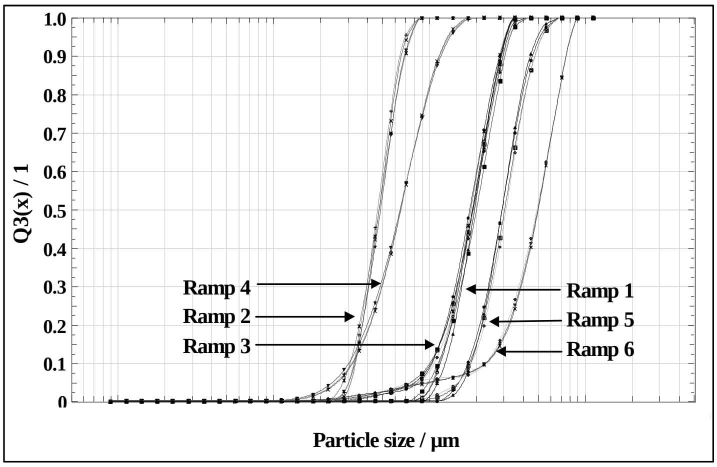

Fig. 4: OPUS results (cumulative distribution $Q_3(x)$) in pilot plant.

4.2 Process control in industrial environment

Another installation example is presented in Fig. 5, where OPUS is installed in a re-circulation loop of a 1700 l detergent crystallizer via a FT 25 adapter. Here, OPUS monitors the dynamic crystallization behaviour for different kinds of products as e.g. sodium perborate or sodium percarbonate during continuous and batch production.

Fig. 5: OPUS installation in industrial environment. a) Crystallizer (1700 l), b) OPUS, c) Re-circulation flow

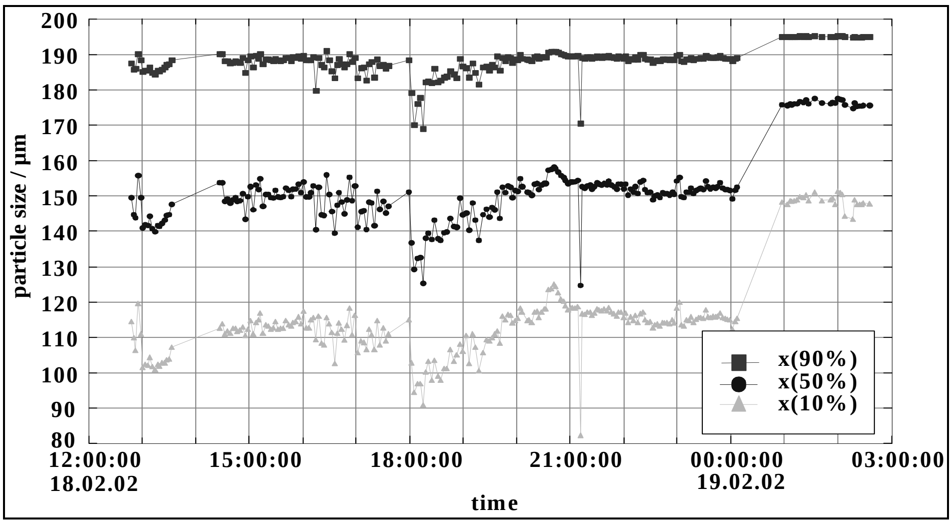

Fig 6: OPUS results from process control of detergent crystals.

Results obtained from this installation (typ. conc. 40 % vol.) are shown in Fig. 6 as characteristic values (x_{10}, x_{50}, x_{90}) vs. time. The diagram shows a stable behaviour of the CSD during a continuously crystallization until 17:00. Here, the CSD drops significantly, most probably caused by a too high re-circulation flow of fine seeds. The real time monitoring of this effect allows the operator to react immediately. Thus, the process could be stabilized again very soon. After 00:00 the product had been changed.

5. CONCLUSION AND OUTLOOK

The presented ultrasonic based Sympatec OPUS system is best suited for in-line particle size analysis of crystallization processes. Because of the ultrasonic specific properties the sensor is very robustly engineered to withstand the typical temperatures and pressures in process environments without any problem. The finger probe design also makes installation easy for existing crystallizers, not only in processes, but also in pilot plants and lab application.

Since the measurements are carried out at original solid concentrations, dilution of the crystal suspension is not required.

Several installations have proven OPUS capability for the field of industrial crystallization and its performance to provide the required information for a better understanding of the processes behaviour. The real time monitoring of crystal size distributions even allows control of the production itself as an elementary part of the quality management.

In the future it is to be expected that in-line particle size analysis will provide the necessary information for the modelling of industrial crystallization processes. Based on improved models automatic process control will be possible which will lead to a better quality of the user's product.

Symbols

I	Intensity of the received sound wave	[J m^2 s^{-1}]
I_0	Intensity of the introduce sound wave	[J m^2 s^{-1}]
f	frequency	[Hz]
Δl	thickness of suspension layer	[m]
C_{PF}	projection area-concentration	[m^{-1}]
K	Extinction coefficient	[-]
x	crystal size diameter	[m]
q_2	projection area density distribution	[m^{-1}]
m	measured extinction	[-]

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