In-line Crystal Size Distribution analysis in industrial crystallisation processes by Ultrasonic Extinction

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Abstract

Ultrasonic Extinction is a relatively young method for the determination of particle (crystal) size distributions in high concentrated suspensions and allows the design of rugged instruments ideal for process application. After the description of technology and technical realisation, results of a successful installation will be presented to show that Ultrasonic Extinction is proven technology for continuous and batch crystallisation processes.

1. Introduction

In the last few years in-line particle size analysis has continued to reach more and more importance since this information is required for control and optimisation of chemical process (e.g. crystallisation)

Since a few years sensors based on Ultrasonic Extinction have been applied for in-line particle size analysis. As primary measurement information the frequency dependent Ultrasonic Extinction which can be converted by a mathematical algorithm into a particle size distribution, is used.

Based on Ultrasonic Extinction very robust sensors can be build, which also can cope with typical process pressures and temperatures. The use of Ultrasonic Extinction offers a variety of advantages compared to optical principles. The analysis can be carried out in opaque suspensions at high concentrations (typically up to 70% by volume.) The measured particle size distributions are only affected very little by the concentration, which may vary in a wide range. Contamination on the sensor components does not influence the results.

Using Ultrasonic the complete volume in the measuring zone is analysed so that the information about the suspension is gathered from the volume and not only from a small layer in front of a glass window as with optical instruments. Ultrasonic extinction is dependent on the acoustic properties of the product to be analysed. The acoustic properties can be determined by measurement quite easily and with high accuracy. Nevertheless the product must be known quite well regarding its consistency before particle size analysis can be performed.

2. Principle of Ultrasonic extinction

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

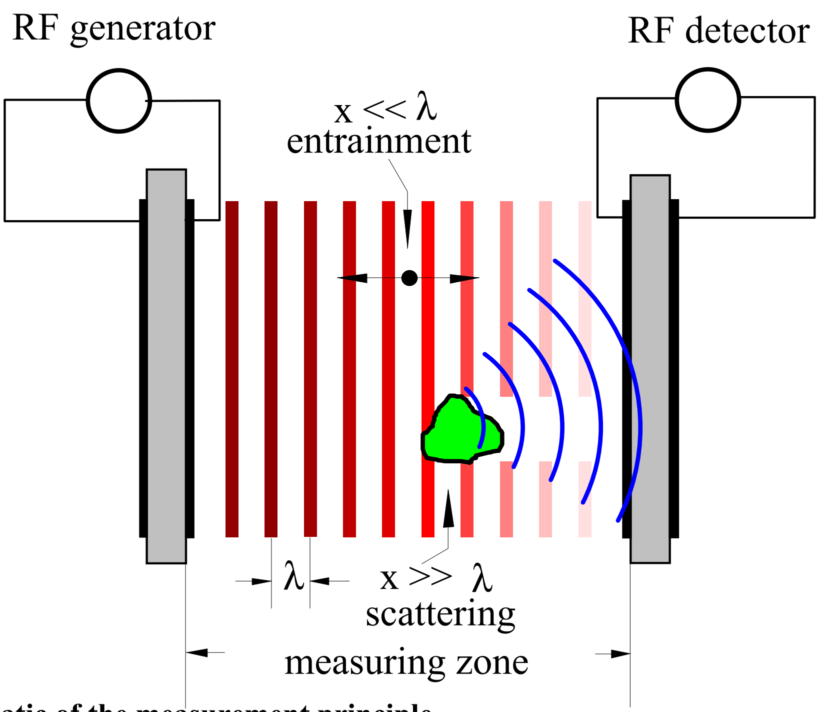

Fig.1: Schematic of the measurement principle

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.

3. Description of Ultrasonic Extinction of poly-disperse systems

The Ultrasonic Extinction of a suspension of mono-disperse particles can be described by the Lambert-Beer law according to Riebel.[1]

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

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:

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

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

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

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

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

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

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[1] Riebel U., Die Grundlage der Partikelgrößenanalyse mittels Ultraschallspektrometrie, Diss. Karlsruhe 1998

4. Realisation of the Measuring principle for in-line applications

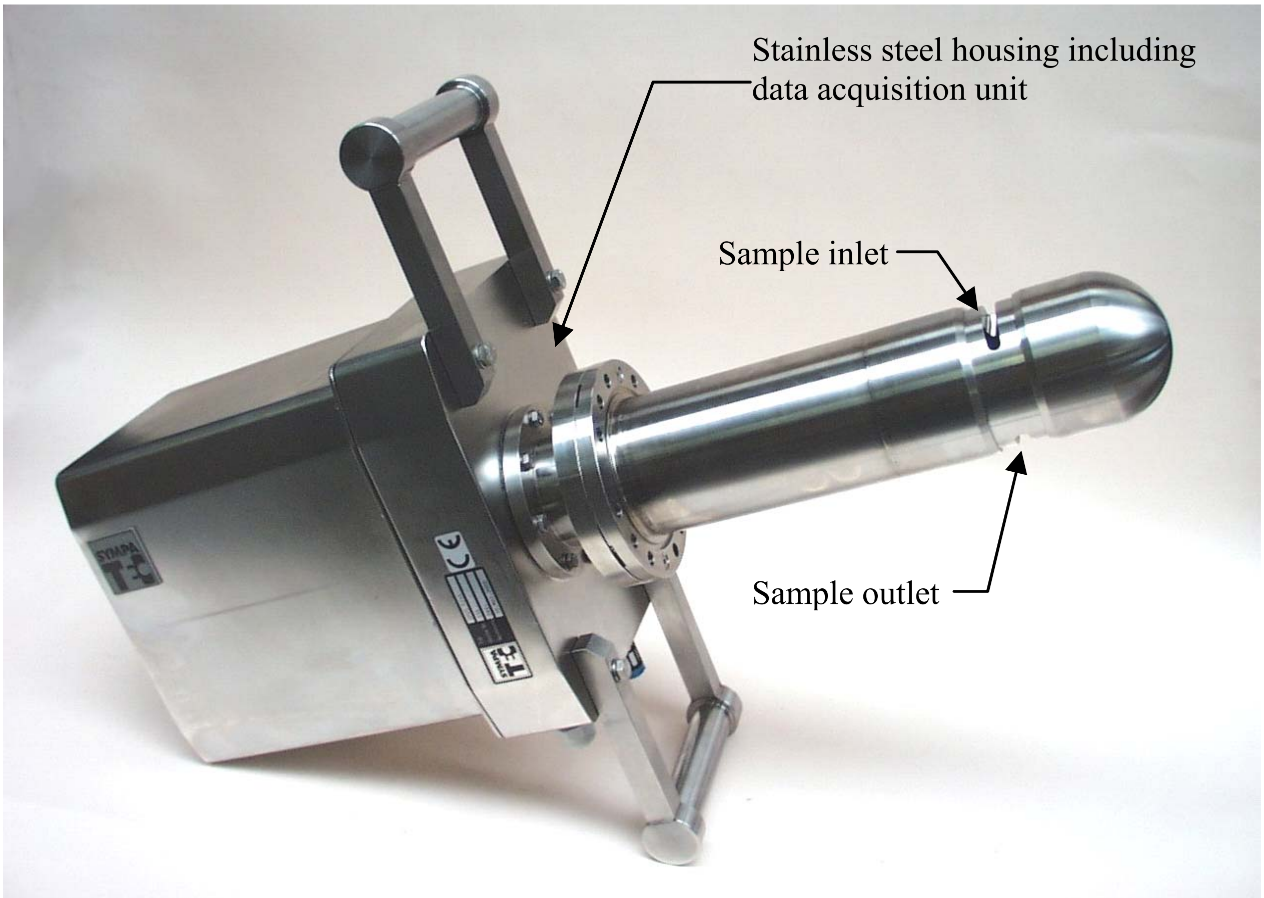

Fig.2: Technical realisation of Ultrasonic Extinction for in-line particle size analysis

Figure 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 prepared for fully automated real-time particle size analysis in process environment (Temperature 0 – 120°C, Pressure 0-40 bar, pH-value 1-14). 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 length 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 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 kind of crystallisation processes. [2]

[2] A.M. Neumann, Characterizing Industrial Crystallizers of Different Scale and Type, Diss. TU Delft 2001

5. OPUS Results from Crystallisation Processes

The following figure 3 presents some results over a period of one week time from a continuous ammonium sulphate crystallisation at ca. 115°C and 2 bars of pressure.

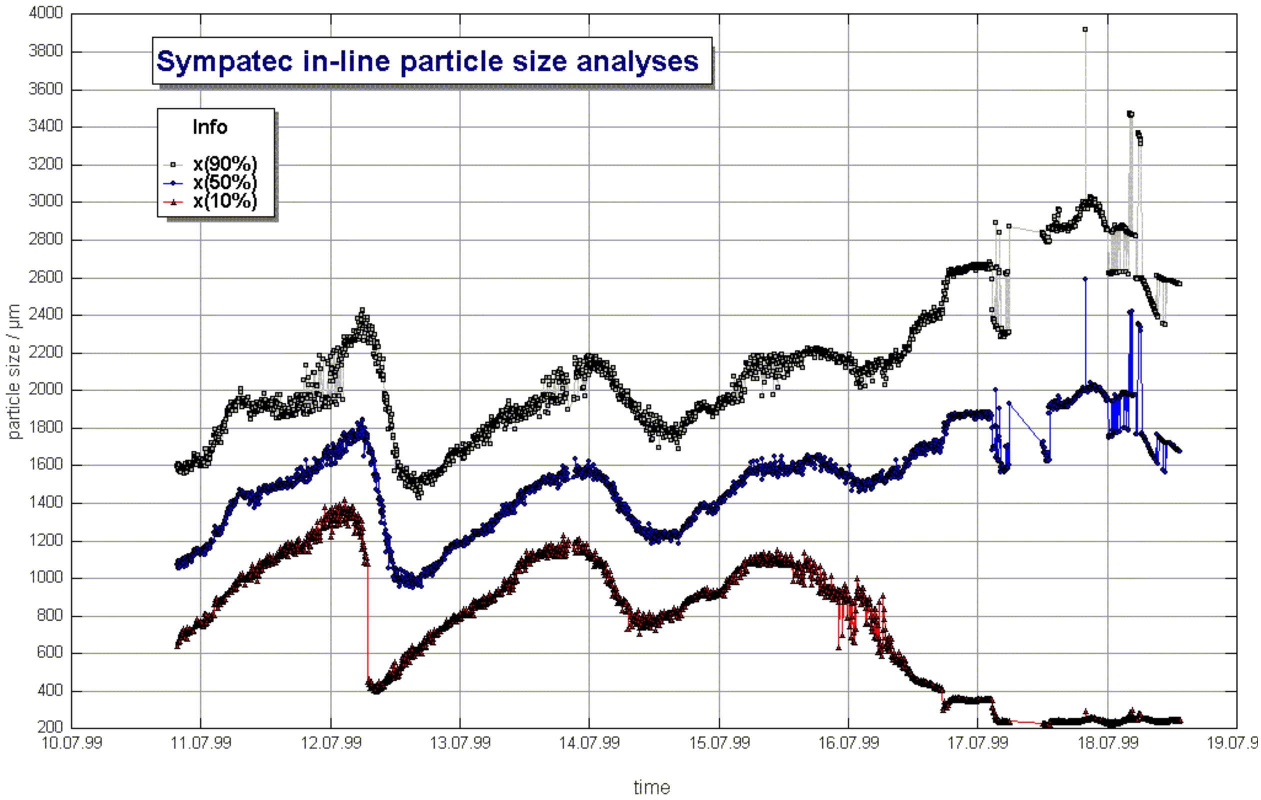

Fig.3. OPUS in-line results of an ammonium sulphate crystallisation

OPUS is installed in the re-circulation loop (Ø 1100mm) of the crystalliser and has a length of ca. 1400mm. Using the automated cleaning device the instrument is accessible every time it should be necessary. The known oscillation of continuously operated crystallisers is monitored. The decrease of the x_{10} value and the rapid increase of the x_{90} value at the end of the displayed period indicate a problem in the crystallisation process which has been monitored by OPUS. Since OPUS measures the crystal size distribution in real time, this information is available immediately for the control of the process. The point of installation of an in-line systems must be chosen carefully since it has to be assured that at this point a representative product flow is seen by the instrument.

6. Conclusion and Outlook

The presented ultrasonic based Sympatec OPUS system is best suited for in-line particle size analysis of crystallisation processes. Because of the ultrasonic specific properties the sensor is very robust and engineered to withstand the typical temperature and pressure in a crystallisation process without any problem. The finger probe design also makes installation easy for existing crystallisers.

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

Several installations show that OPUS is proved in the field of industrial crystallisation and processes can be controlled in real time.

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