Ultrasonic Extinction for In-line Measurement of Particle Size and Concentration of Suspensions and Emulsions

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ABSTRACT

Ultrasonic Extinction is a well suitable method for the determination of particle size distributions (PSD) in highly concentrated suspensions and emulsion. It allows for the design of rugged instruments which are ideal for process applications. Today, for irregular shaped particles > 10 µm no satisfying theory is available for the description of the ultrasonic extinction. A new calibration procedure was developed, which overcomes this limitation. Some selected industrial applications demonstrate the ability of this technology for particle size analysis under extreme conditions .

Keywords In-line, Particle Size Analysis, High Concentration, Ultrasonic Extinction

1 INTRODUCTION

For many industrial processes particle size distribution and concentration are important parameters. There is a continuously growing interest in in-line measurements of both quantities in order to improve the control and efficiency of the processes. In a plant the measurement device has to withstand pressure and temperature inside the pipes or vessels . Chemical aggressive liquids must not affect the sensor. The requirements of hazardous areas have to be covered.

Ultrasonic Extinction (UE) Spectroscopy is a technique for particle size analysis in the range of 0.01 µm to 3000 µm. It allows for the analysis of suspensions and emulsions at high solids concentrations (e.g. up to 70 % vol.). Ultrasonic extinction devices measure the frequency dependent UE. As a result, the particle size distribution and the particle concentration can be obtained.

Based on UE very robust sensors can be build, so that they withstand high pressures (e.g. 40 bar) and high temperatures (e.g. 120 °C). A stainless steel housing combined with high quality sealings (e.g. KALREZ™) gives high chemical resistance to the sensor. By using ultrasonic waves opaque suspensions and emulsions can be analysed without any restrictions. No dilution of the suspension or emulsion is required, so he properties of the sample are not be changed by the analysis.

2 PRINCIPLE OF ULTRASONIC EXTINCTION

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

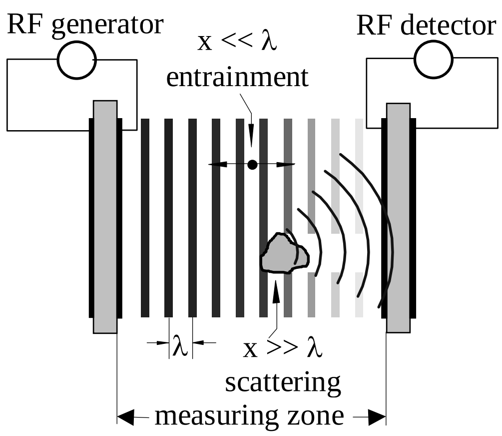

Fig.1: Schematic of the measurement principle

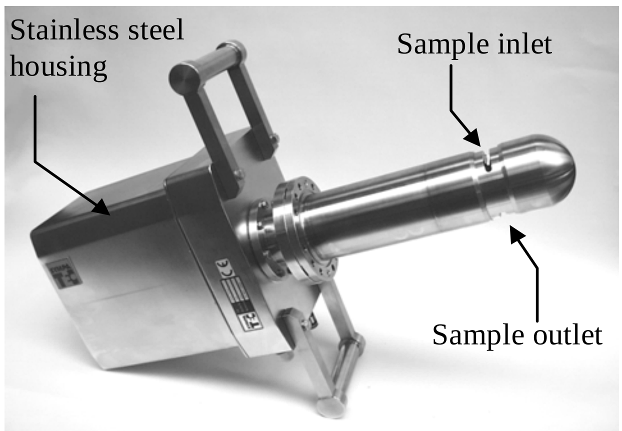

Fig.2: Technical realisation of Ultrasonic Extinction for in-line particle size analysis (Sympatec OPUS)

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. This interaction results in an attenuation of the intensity of the ultrasonic waves. After passing the measuring zone the ultrasonic waves are received by an ultrasonic detector and are reconverted into an electrical signal. The extinction of the ultrasonic waves is calculated from the ratio of the signal amplitudes of the generator and the detector side.

The UE of a suspension of mono-disperse particles with the diameter x can be described by the Lambert-Beer's law according to Riebel (1988).

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

The extinction $-\ln(I/I_0)$ at a given frequency f_i is linear dependent on the thickness of the suspension layer Δl, the projection area-concentration C_{PF} and the related extinction cross section K. In a poly-disperse system the extinction of single particles overlays:

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

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

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

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

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

This system of equations is numerically unstable and must be solved by suitable algorithms. The key for the calculation of the particle size distribution is the knowledge of the related extinction cross section K as a function of the dimensionless wave number σ as defined in equation (5)

$$\sigma = \frac{\pi \cdot x}{\lambda} = \frac{\pi \cdot \mathrm{x} \cdot \mathrm{f}}{\mathrm{c}} . \tag{5}$$

The wave number includes the ratio of the particle size and the ultrasonic wavelength λ within the liquid. The wavelength is calculated from the ratio of the frequency f and the speed of sound c of the liquid.

There are several models, see e.g. Harker et. al. (1991), to calculate the extinction cross section for small wave numbers (σ < 0.1). These models describe the viscous and thermal losses due to the partial entrainment of the particles by the ultrasonic wave, whilst scattering is neglected. For larger wave numbers scattering becomes more and more relevant. Fig. 3 and Fig. 4 show the extinction cross section of glass beads and quartz sand measured by Riebel (1988) compared with results obtained by the scattering theory (Faran, 1951):

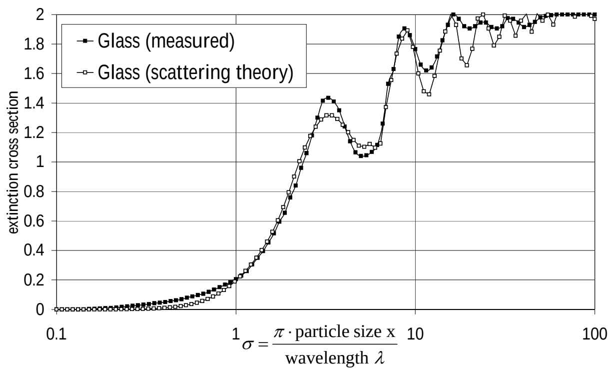

Fig. 3: Extinction function for glass beads

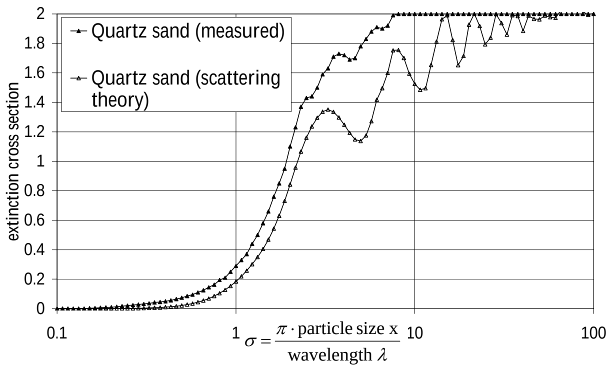

Fig. 4: Extinction function for quartz sand

The acoustic properties of glass and quartz are nearly identical. Therefore, the only significant difference between the two samples is the particle shape. For the spherical glass particles the extinction function calculated by scattering theory matches the measured result quiet well.

In contrast, there is a great deviation between the calculated and measured extinction function for quartz sand. The scattering theory assumes spherical particles and therefore can not be used for the irregular shaped quartz particles (particle shape: rounded cubes).

But the extinction function for arbitrary shaped particles can be evaluated empirically: Firstly, the particle size distribution is measured with a suitable particle sizing method (e.g. laser diffraction). Secondly, the result is mathematically combined with the measured ultrasonic extinction spectrum of the same sample. With this kind of calibration procedure the extinction function can be evaluated within the wave number limits given by the minimum and maximum particle size, and the frequency limits of the measurement.

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 sensor and can be adapted to nearly all kind of process pipes or vessels using a standard DN 100 flange. It is designed for a fully automated real-time particle size analysis in a process environment. A version for hazardous areas (Zone 1 ambient, Zone 0 in the measuring zone) is available as an option .

As the OPUS probe provides different lengths from 330 to 3.500 mm (measured from flange to tip) the instrument can be applied to a variety of processes. For smaller pipe diameters special adapters are offered covering process pipe diameters down to DN 10. As an option an automatic cleaning system (Docking-Positioner & Cleaner) can move the complete sensor out of the process pipe or vessel for cleaning purpose, inspection or maintenance without the need to shut down the process. OPUS has been applied successfully to various kinds of processes.

3.1 Technical Data of the UE Sensor OPUS

➢	measuring range:	0.01 – 3,000 µm	➢ easy adaptation by large variety of adapters from DN 10 - 200 and different probe lengths from 330 – 3500 mm
➢	particle concentration: (pure solid)	1 – 70 % Vol.	➢ easy access (e.g. via Docking Positioner & Cleaner)
➢	temperature range:	0 – 120 °C	➢ highly resistant due to the applied materials in contact with the product like stainless steel, PTFE, Sigradur ®
➢	pressure range:	0 – 40 bar	
➢	pH-range:	1 – 14	
➢	high viscosities		

Table 1.: Technical specifications of the OPUS sensor

3.1 Application of the UE Sensor in a Crystalliser

The following figures demonstrate the abilities of a measurement device based on ultrasonic extinction.

Fig.5a: On line installation

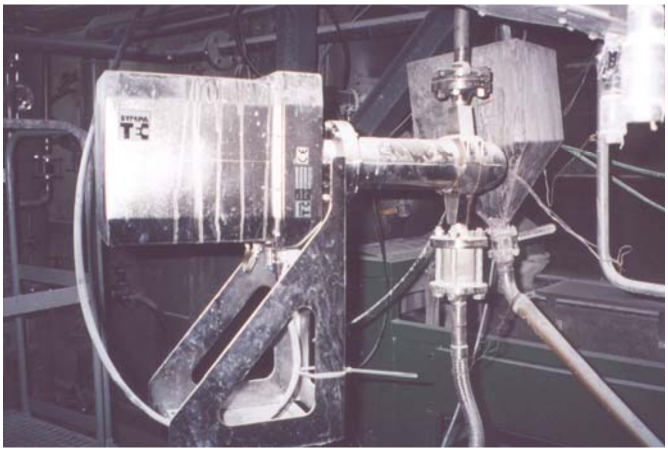

Fig.5b: On line installation

Fig 5 shows the installation of an UE measurement device within a plant used for the continuous crystallisation of a heavy metal salt. The temperature of the suspension is about 100 °C. The pressure is about 1.5 bar inside the pipe system. The measurement system has been installed as bypass to a circulation pipe.

4 RESULTS

Fig. 6 shows the results obtained during this continuous crystallisation of a heavy metal salt.

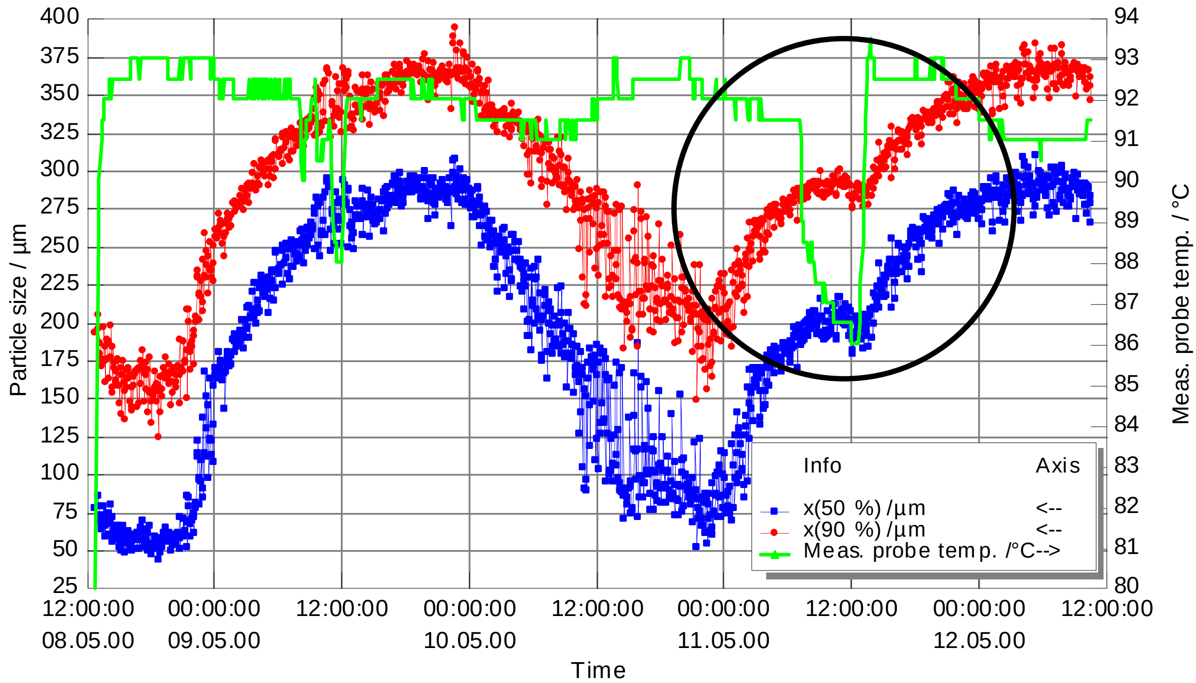

Fig. 6: Typical results measured on-line

It is well known that the crystal size distribution oscillates in continuous crystallisers. This can be observed with the ultrasonic extinction measurement device in real time. A change of the temperature (see circle) leads to a significant change of the particle size distribution.

The measured particle size data have been used to model the crystalliser. The model for the crystalliser offers in combination with the in line measurement of the particle size distribution the possibility of direct control of the particle size distribution within the crystalliser.

5 CONCLUSIONS

UE based measurement system is well suited for in-line particle size analysis of suspensions and emulsions. Using ultrasound the sensors can be designed very robust and withstand process environments with typical high temperatures and pressures easily. A finger probe design also simplifies the installation in existing plants. It also can be used in pilot plants or lab applications.

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

For the calculation of the particle size distribution the knowledge of the related extinction cross section of the particles is essential. For small and for large, non-absorbing, spherical particles suitable theories are available for the calculation of the related extinction cross section. An empirical approach for the determination of the extinction function widens the range of application towards large, absorbing and irregular shaped particles.

UE based instruments have proven their capability in several installations in a wide variety of industrial processes, and their suitability providing the required information for a better understanding of the processes behaviour. The real time monitoring of particle size distributions often allows for control of the production as an elementary part of the quality management.

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