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In- and Off-line Ultrasonic Extinction from 0.01 µm up to 3000 µm

1. Introduction

In 1988 Riebel [1] presented work which formed the basis for particle size analysis using ultrasonic extinction measurements in the range from 20 – 1000 µm. Ultrasonic extinction is mostly independent of the dispersion state of the suspensions. Measurements can be carried out on any type of liquid, at high solid concentrations and in a wide temperature range. These features predominate ultrasonic extinction for on line particle size measurements for suspensions and emulsions.

Based on Riebels work Sympatec introduced in 1990 a commercial instrument (OPUS®) for on line particle sizing. Results obtained with this instrument have been reported for example by Geers [3], and Witt [2]. In the last few years the technique has been rapidly improved. The measurement range covered by ultrasonic extinction measurements has been extended at the fine and the coarse end to now 0.01 µm to 3000 µm. The concentration may vary between 1 – 70 % Vol. Many experiences have been made with this instrument in laboratory scale and on-line in different processes.

The present flow-though cuvette solution has to be arranged in a bypass to the process. The growing demand of direct measurements inside a pipe or a vessel was answered by the development of a new sensor – called 'OPUS Probe'.

2. In-line particle sizing using ultrasonic extinction

2.1. Present flow through cuvette

Fig. 1 shows the typical set-up for the commonly used installation of an ultrasonic extinction-sensor for on-line measurements. This type of set-up has been realised for several installations in the past:

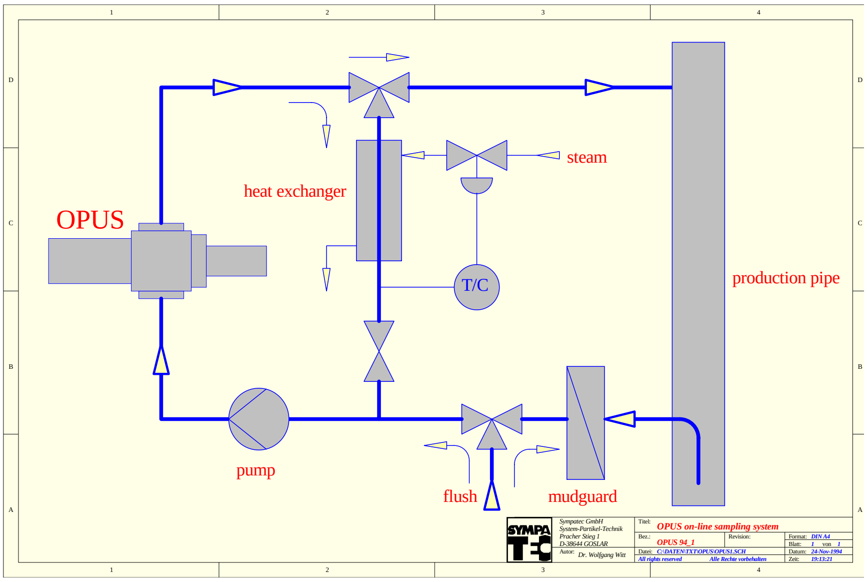

Fig. 1 Scheme of a typical OPUS on-line installation

The ultrasonic extinction sensor is realised as a flow-through cell. A pump is used in order to supply it with suspension. To achieve a proper temperature equilibrium between sensor and process a heat exchanger is needed. The sensor is coupled with the production pipe by two flanges and DN 25 mm pipes. The whole system has to be controlled by three valves.

A mudguard at the inlet of the suspension protects the pipe system and the sensor against big particle (typ. > 4 mm) which might occur in real processes. With the first valve it is possible to choose between sampling, flushing of the pipe system or flushing the mudguard. The two other valves form a loop with the heat exchanger, pump and the sensor inside. In the second position of these two valves the suspension passes the sensor and then gets back to the process pipe.

The access to the sensor (e.g. for service works) is very easy. For the background measurement, the sensor can be supplied with particle free liquid. For processes working at ambient temperatures there is no need for the heat exchanger.

The OPUS Sensor is connected with an electronic box that has to be installed nearby (max. cable length: 2 m) the OPUS-Sensor. The electronic box is connected with a computer (PC) by a fibre optical link. A programmable logic control (PLC) linked to the PC is used to control the valves.

With this type of set-up many successful experiences have been made in different processes (e.g. PVC-production, crystallisation processes).

2.2. A newly designed in-line sensor

Although the experiences with the OPUS flow through cell are generally positive, there is a growing demand of measuring directly inside the process pipe or a vessel in order to minimise the installation effort. To meet these requirements a new in-line sensor - called OPUS Probe - has been designed. Fig. 2 shows a photo of the new sensor while Fig. 3 shows a more detailed cross section:

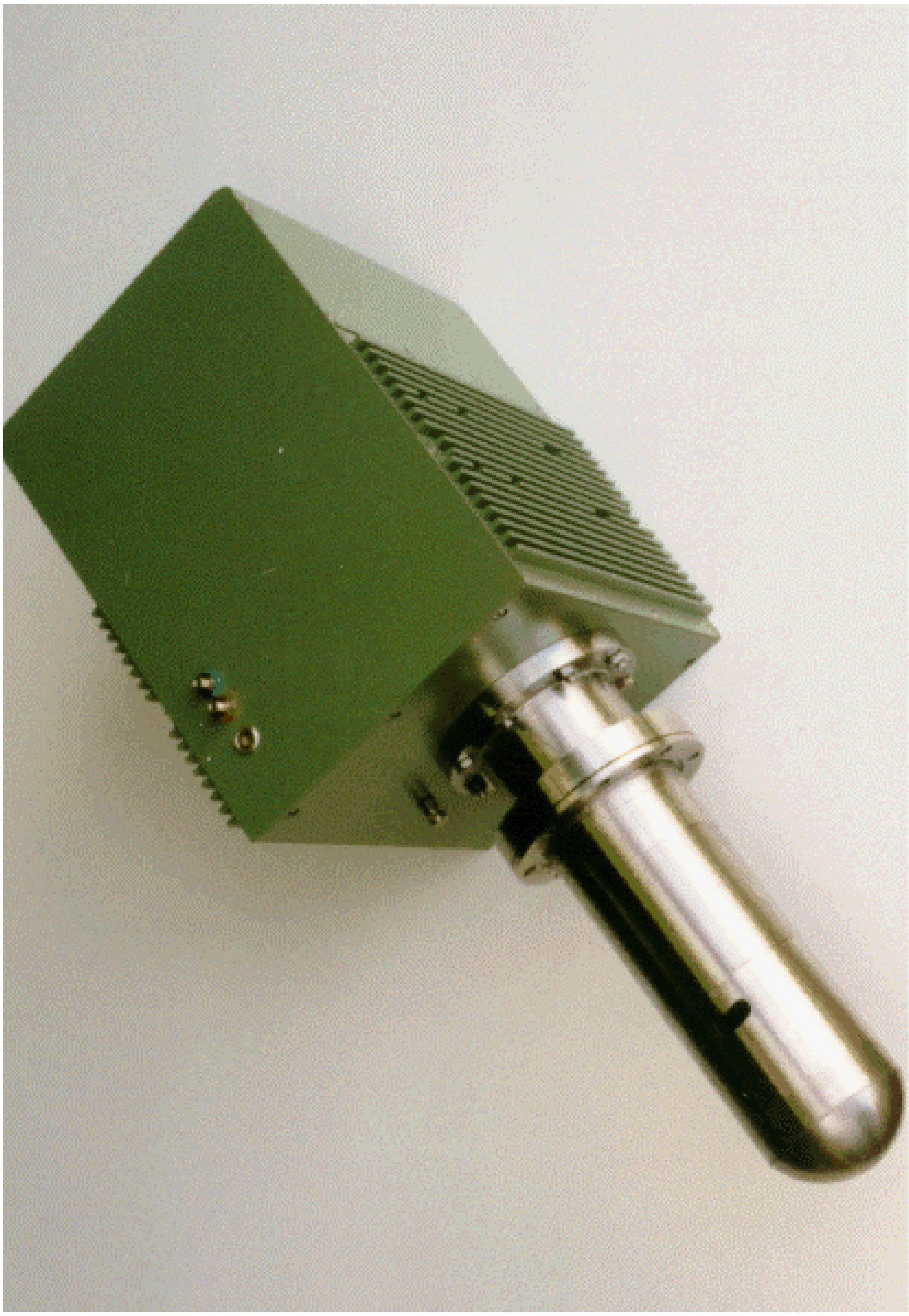

Fig. 2 Picture of the new In-line sensor (OPUS-Probe)

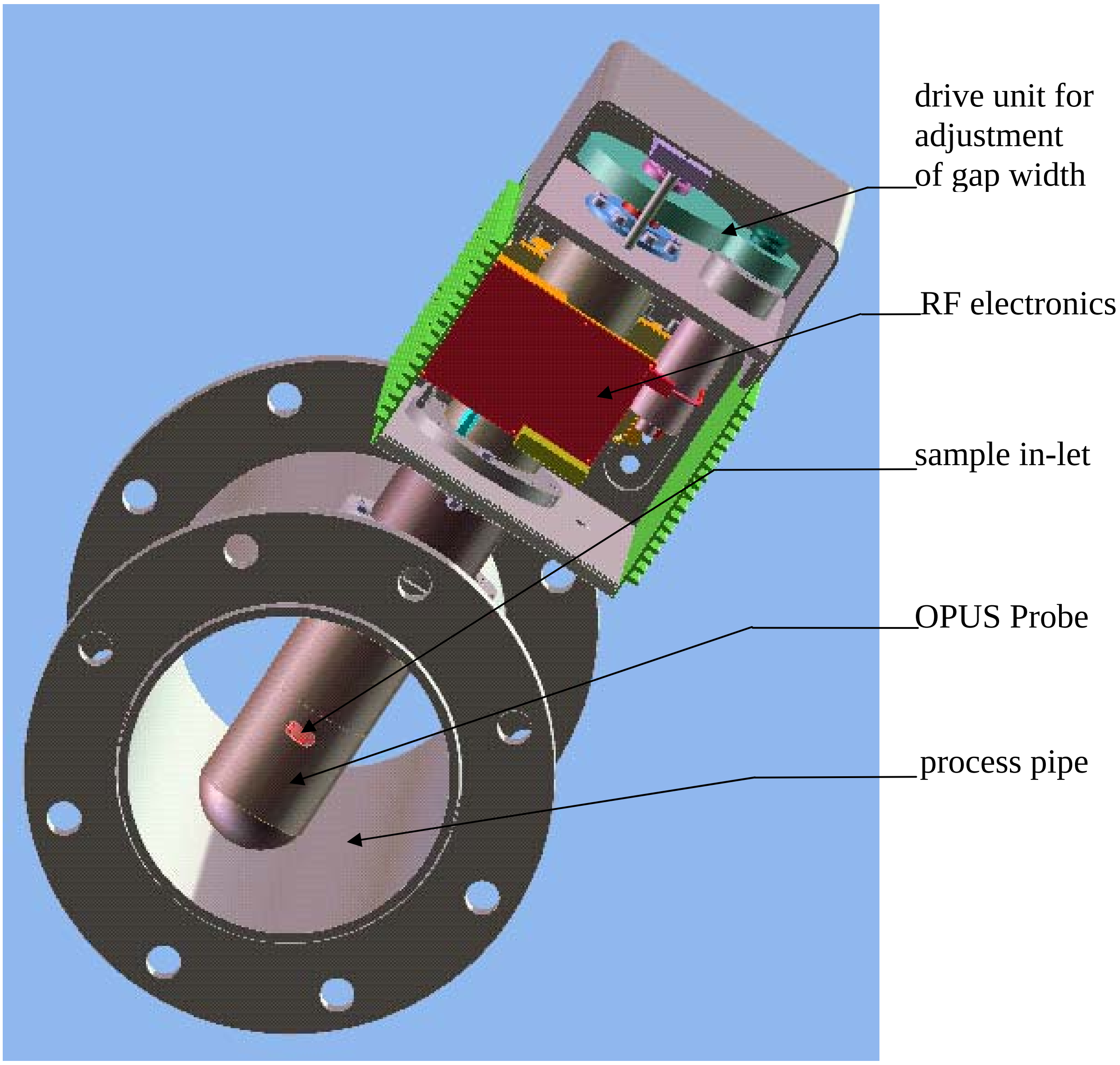

Fig. 3 Cross section of the OPUS Probe installed in a DN 200 pipe

The new ultrasonic extinction sensor is a “one flange solution”. It allows measurements to be carried out in-line inside a pipe or a vessel as well.

The part of the sensor which is inside the suspension is made of stainless steel. For the gaskets Kalrez® is used. The sensor can stand a maximum temperature of 120 °C. It is designed for a pressure range of 0 – 40 bar.

Fig. 3 shows the sensor without connection facilities. In order to obtain a representative suspension stream through the measurement zone it is possible to connect the ultrasonic extinction sensor with an in-line working pump and a rotating in-line sampling system (TWISTER®) [2] that scans over the whole cross section of the pipe. The flow rate of the in-line pump can be controlled and therefore, it gives the possibility to have an isokinetic, representative flow through the measurement zone. In addition the TWISTER® offers the possibility to supply the in-line sensor with particle free liquid, which is needed for the background measurement.

The RF-electronics and signal processing unit is directly connected to the sensor. The complete sensor is compact and requires a minimum of space. The housing for the electronics meets the requirements of the IP 55 standard. An explosion proof version will be available as an option.

The new sensor offers an automatic change of the gap width over the complete, acceptable pressure range of the sensor. The actual gap width can be measured by an integrated gap width sensor. Therefore, the sensor can be adapted during the measurement to the demands of the suspension (e.g. concentration) to be analysed. Due to this new feature the measurement resolution has been improved in the order of one magnitude.

In real processes strong vibrations are to be expected. In order to achieve a proper stability of the alignment and to get the possibility of a realignment, an auto-alignment function has been introduced to the new sensor. It guarantees that the precision of the transducer alignment is not affected by the gap adjustment or external influences. As a result the number of requested background measurements can be reduced to a minimum. The typical frequency is one background measurement per day.

The complete sensor is controlled by an external computer (PC) which is linked to the system either by a fibre optical link (maximum cable length 4 km) or a RS 485 interface. The computer will be installed in a control room offering the possibility to control the sensor with the data-bank based Sympatec WINDOX® software. In the standard set-up there is no need for manual interaction between the user and the system.

The measurement results are stored in the WINDOX® data base and can be accessed either directly from the measurement PC or via a network. The WINDOX® software gives the opportunity for trend analyses, reports, printouts, graphical presentations etc.. Characteristic values of the measured particle size distribution can be offered as 4 – 20 mA signals.

The OPUS Probe can currently be installed in pipes with a minimum size of DN 150. For larger pipe sizes or for the use of the OPUS Probe inside a vessel it is possible to extend the length of the sensor up to 2 metres.

3. Results

3.3. Laboratory measurements on emulsions

Measurements on a water/oil emulsion have been carried out using a test loop. An impact plate inside the flow system dispersed the oil with respect to the flow rate. Therefore, it was possible to change the droplet size distribution by altering the speed of the peristaltic pump used for the transport of the emulsion.

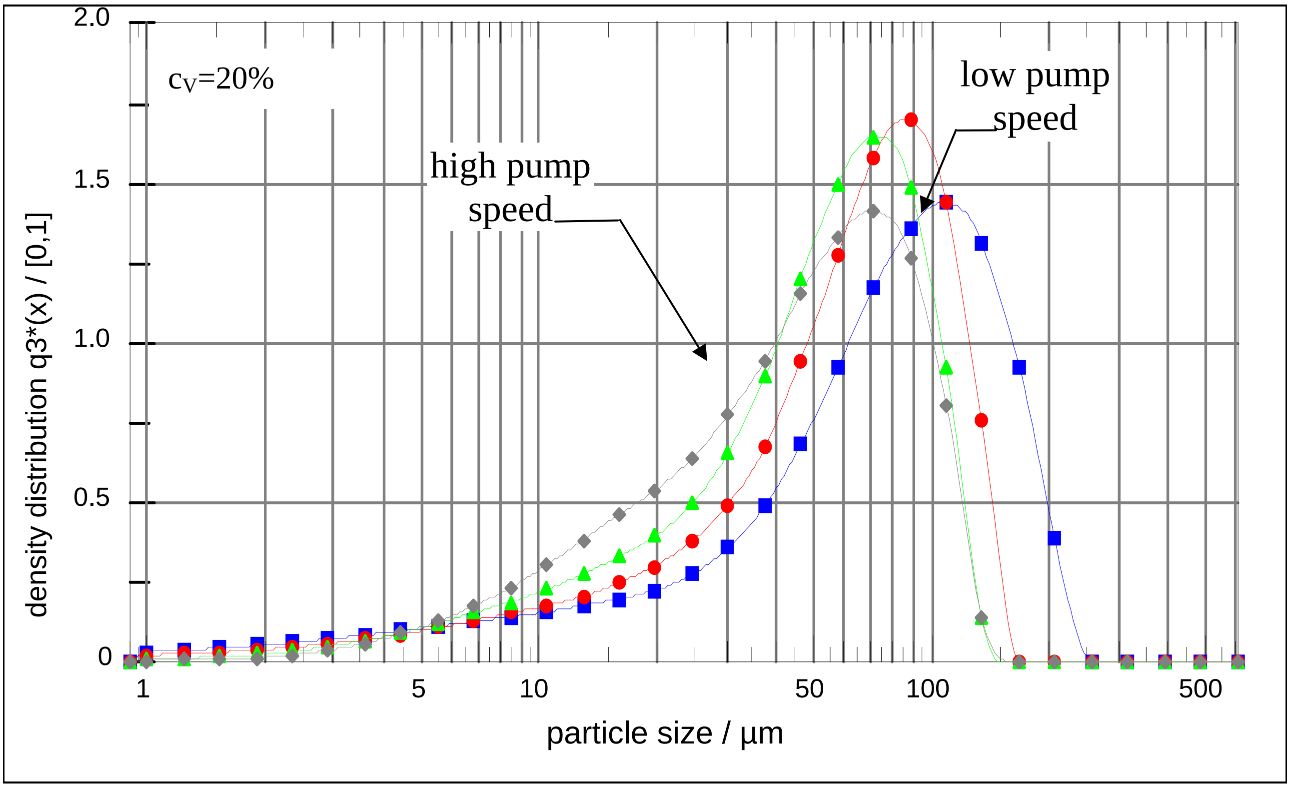

Fig. 4: Results obtained for a water/ oil emulsions at different dispersion energies

This in-line experiment in the laboratory scale shows the possibility to monitor a droplet size distribution with the OPUS system.

The density contrast between the two phases is very small. Therefore, it is no longer possible to find the extinction coefficients only as a function of the wavenumber (extinction function). The result is, that the matrices needed for the calculation of the droplet size distribution had to be calculated by a special algorithm based on the scattering theory for ultrasonic waves.

For these calculations material dependent data of the pure liquids are needed. It is possible to measure these data with the ultrasonic extinction sensor itself.

3.4. Measurements in the SUBMICRON Range

The measurement range of the OPUS system can be extended into the SUBMICRON range. As an example for this, measurements on commercial paint (Fig. 5) and paint pigments in water (Fig. 6)at different grinding steps are shown in the next diagram:

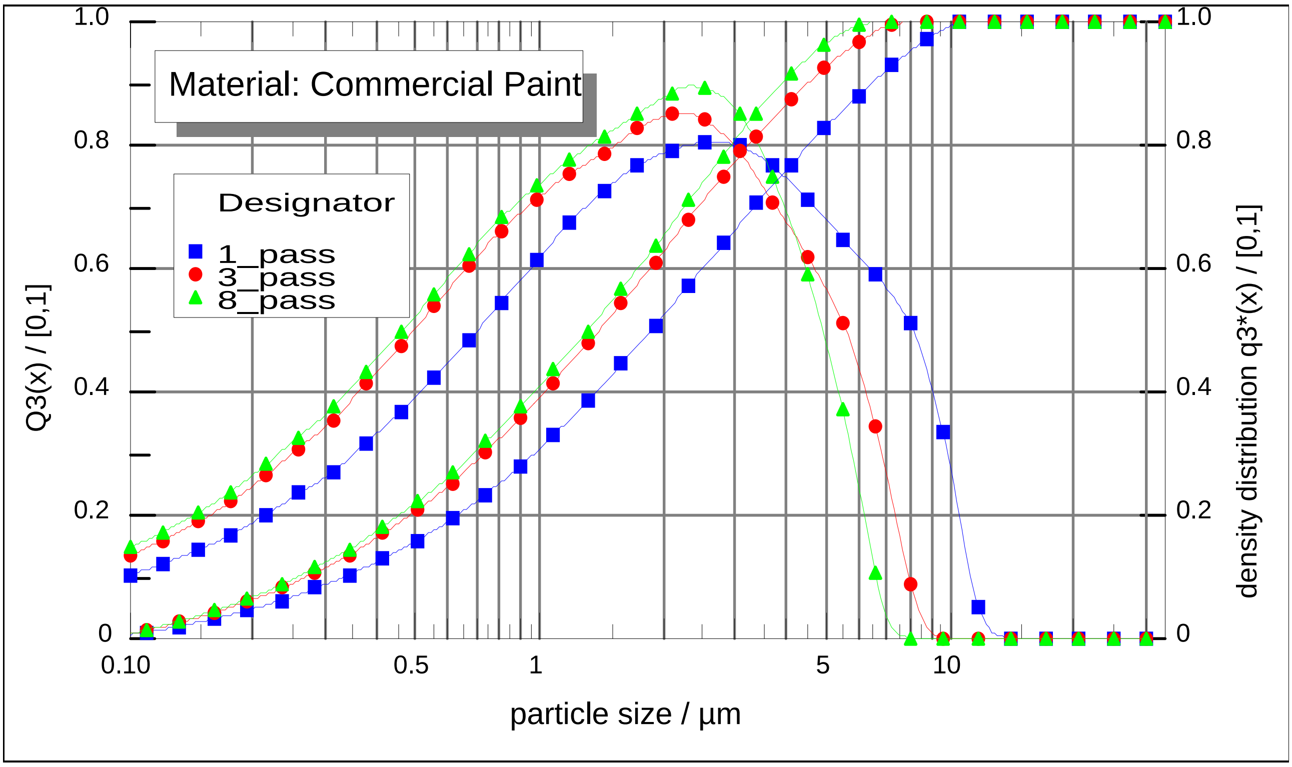

Fig. 5: Results for commercial paints at different grinding steps

The results show that it is possible to monitor the trend of the particle size distribution during a grinding process using the suspension with the original concentration. The measurements can be carried out under the conditions (concentration, temperature, pressure) given by the process. Therefore, the measurements will introduce a minimum of disturbance to the process to be monitored.

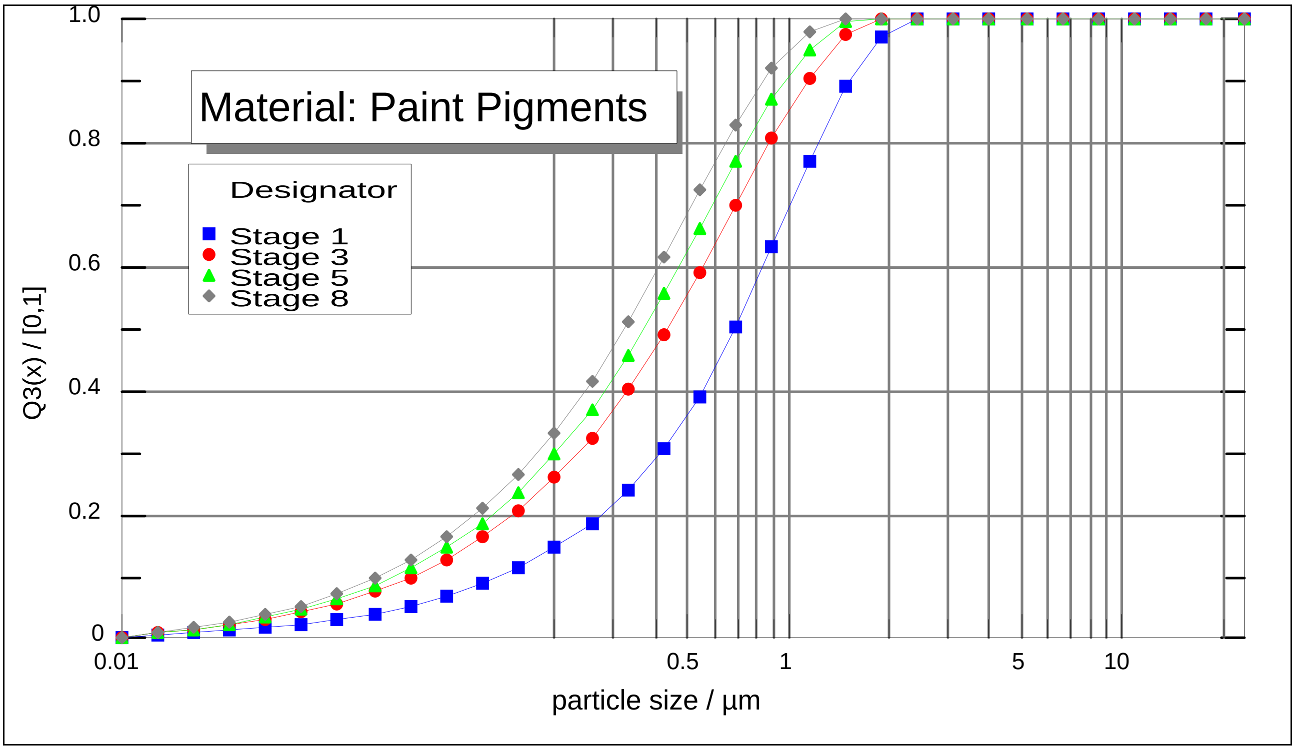

Fig. 6 : Results for paint pigments in water at different grinding steps

3.5. Temperature compensation

The OPUS sensor is designed for the temperature range of 0°C to 120 °C. The maximum temperature deviation between the background measurement and the measurements with particles usually should not be greater than ± 5 % of the temperature given in degree Celsius. The next example shows, that it is possible to extend the acceptable temperature deviation with a speed of sound correction.

The actual speed of sound can be measured with the OPUS sensor itself. Therefore, the influence of the temperature towards the measurement result can be suppressed by extending the measurement procedure.

The results show, that there is no dependence of the measured particle size distribution on the suspension temperature.

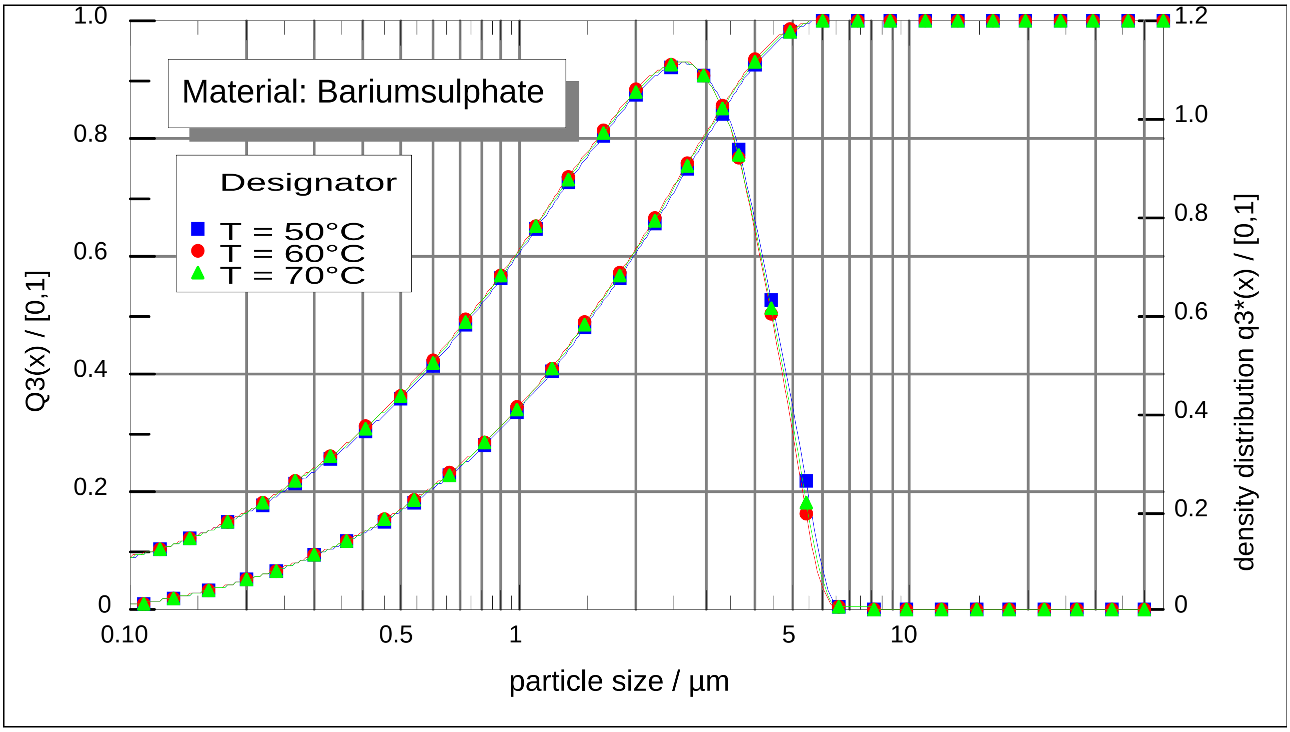

Fig. 7 Influence of temperature to the measured particle size distribution

3.6. Comparison with laser diffraction

In general the ultrasonic extinction measurement results agree quite well with particle size distributions obtained by laser diffraction analyses. Fig. 8 gives a comparison of results obtained with the two different measurement techniques.

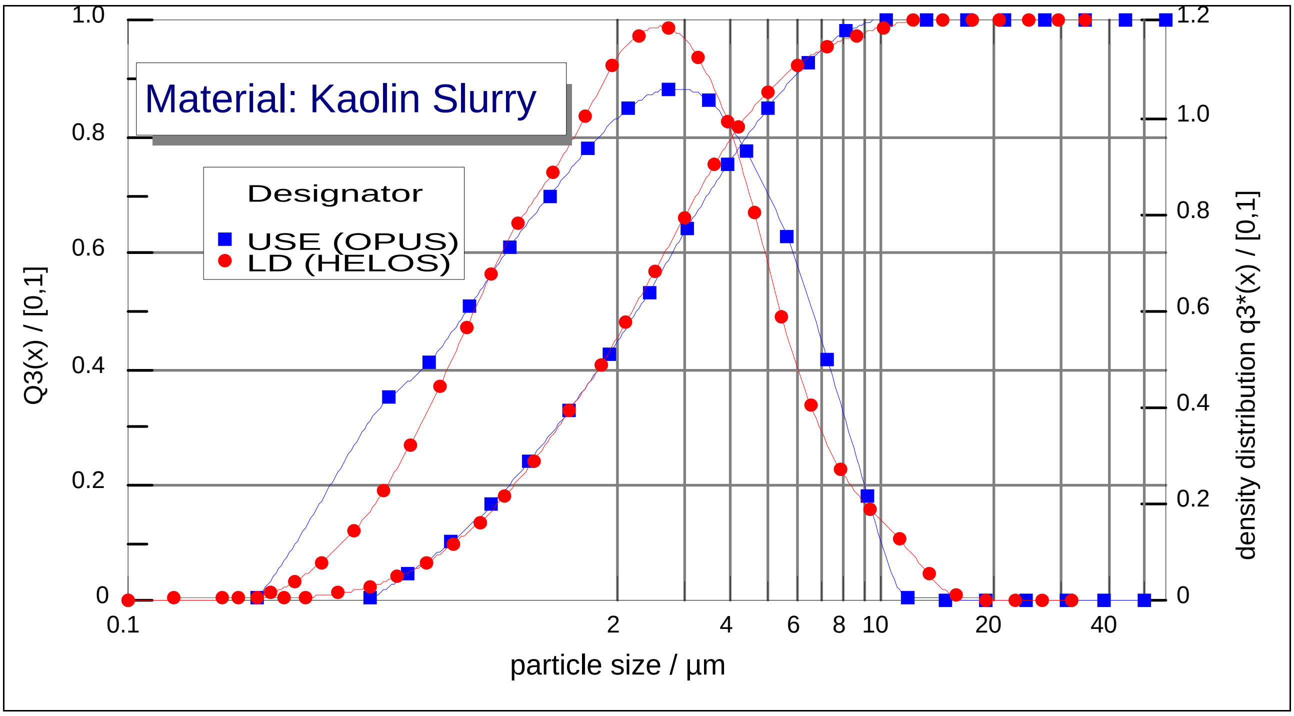

Fig. 8 Comparison of ultrasonic extinction results with laser diffraction measurements

The material was a kaolin slurry with a solids concentration 47 % Vol.. At this concentration the slurry can hardly be pumped but with the OPUS system it is possible to analyse the slurry at the original concentration. For the laser diffraction analyses the slurry had to be diluted.

Both measurement system give quite similar results. Deviations occur at the coarse end of the particle size distribution.

4. Conclusions

The ultrasonic extinction technique has undergone a rapid development in the last few years. A lot of experiences have been made with on-line working sensors in bypass to different processes and in the laboratory scale using a test loop. Based on these experiences the measurement range could be extended into the submicron range. The maximum detectable particle size increased also slightly. Further improvement has been made in the field of the acceptable solid concentration which now might vary in the range of 1.0 - 70 % Vol.. The influence of temperature fluctuations on the measured particle size distribution could be suppressed. This shows, that the OPUS flow through sensor is able to meet the requirements of different production conditions.

Although the present experiences with the flow through sensor are generally positive, there was still the demand to measure more directly inside the process. This demand has been answered by the development of the in-line sensor presented in this paper. It is able to meet any requirement towards a process measurement system.

5. References

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