Proc. Eurosensors XXVI, September 9-12, 2012, Kraków, Poland

Particle characterization in highly concentrated suspensions by ultrasound scattering method

S. Wöckel a, *, U. Hempel a, R. Weser b, B. Wessely b, J. Auge c

a Institut für Automation und Kommunikation e.V., Werner-Heisenberg-Str. 1, 39106 Magdeburg, Germany
b Institut für Verfahrenstechnik und Umwelttechnik, TU Dresden, Dresden, Germany
c University of Applied Sciences Magdeburg-Stendal, 39011 Magdeburg, Germany

Abstract

A new method for inline characterization of particles in high concentrated suspensions by ultrasonic scattering is described by extracting the statistical features of scattered sound waves that correlate with the particle size and concentration. To provide a high spatial resolution and range - in case of media with high attenuation - ultrasonic sensors with a high bandwidth and intensity are used in combination with signal coding techniques [1]. The effects of particle dependent attenuation and multiple reflections are investigated by processing the signals in the frequency and time domain. Further, a spatial dependent attenuation coefficient is calculated that is equivalent to the values gained by conventional transmission methods.

© 2012 The Authors. Published by Elsevier Ltd. Selection and/or peer-review under responsibility of the Symposium Cracoviense Sp. z.o.o. Open access under CC BY-NC-ND license.

Keywords: ultrasound scattering; particle characterization; suspension

1. Motivation

The inline monitoring of suspensions with a high concentration of particles needs the characterization of different parameter like size, distribution and concentration which are recommended for quality control in industrial processes. Especially in case of high particle concentration the applicability of common optical methods is limited to laboratory applications. In contrast, ultrasonic extinction methods can provide a benefit concerning inline measurements. The processing of the ultrasonic transmission and extinction

* Corresponding author. Tel.: +49 391 9901 430; fax: +49 391 9901 591.
E-mail address: sebastian.woeckel@ifak.eu.
enables the calculation of a damping coefficient that correlates with the particle system (suspension). It contains information on particle dimension, distribution and concentration. Due to the increased ultrasonic attenuation caused by high particle content and corresponding multiple reflections the conventional transmission methods require a small measuring gap. Those small gaps are vulnerable for blockage and do not provide reliability or inline-capability. Alternatively, this article presents a new method for overcoming this problem by using ultrasonic scattering techniques applying at least one transducer in pulse-echo-(reflection)-mode and analysing the transmitted and the reflected signals that contain the information on the particle system. Commercially existing devices [2, 3] which work in reflection mode are limited to low particle contents and assume single particle scattering. The novelty of the presented new approach is mainly its applicability for suspensions with a high particle concentration. Corresponding industrial applications for such a system are the process monitoring of paints, conditioning of mineral or clearing sludge and saw suspension for semiconductor production.

2. Pulse-echo operation mode for direct back-scattering

2.1. Measurement setup,- standard deviation and equivalent attenuation

The presented approach uses the scattered (reflected) instead of the transmitted ultrasonic signal to characterize suspensions with high particle content. To provide a high spatial resolution and range - in case of media with high attenuation - ultrasonic sensors with a large bandwidth and intensity are used. The effects of particle dependent attenuation and multiple reflections are investigated by processing the signals in the frequency and time domain.

![Figure 1: Schematic for the acquisition of the backscattering of ultrasonic waves at particle systems (suspensions) with pulse-echo method](image)

Figure 1 shows a schematic of the measurement setup. In this contribution the results of direct back scattering for classical pulse-echo operation with one transducer including a delay line are discussed. The delay line is used to provide far field conditions, to separate the excitation and the scattering signal and to protect the active surface from the abrasive particle medium. Since the experimental conditions are important for analysis of particle scattering, the reflection at the inner surface of the delay line window is used for calibration of temperature influence of the media and the electronic. Further, the temperature of the suspension is measured and the particle concentration is controlled with common laboratory equipment. To provide a homogeneous distribution of the particles a special stirring and flow control must be established. The measurements are done with an ultrasonic transducer (centre frequency: 4.5 MHz, -6dB-bandwidth: 3 MHz; Olympus) and square impulse excitation (US-Pulser/Receiver UT340; Utex Scientific Instruments, Inc.). The pulse-echo operation mode delivers a voltage signal (equivalent to pressure amplitude \( p \)) dependent of travel time \( t \) containing the reflection at the periphery and the direct back scattering.
of the particles. First and multiple reflections at the window dominate the echo signal (Fig. 1). A reduced signal to noise ratio and the small amplitude in case of high attenuation preclude the identification and separation of single particle reflections for characterization purposes. Hence, in place of the absolute echo amplitude, the standard deviation $\sigma$ of moving particles for a number of $N$ single measurements was processed instead (Fig. 2), since this information is not influenced by stationary reflections from the peripheries (Fig. 1). By passing the suspension, the echo signals as well as its standard deviation exponentially decrease with range (and time $t$) due to attenuation (Fig. 2). This attenuation depends on particle size and concentration [1], [4].

Fig. 2: Standard deviation $\sigma$ of $N = 200$ single ultrasonic scattering signals ($f = 4.5$ MHz) displayed over the travel time (penetration depth) within the suspension ($\text{SiO}_2$: particle size $d < 20 \, \mu\text{m}$; $\rho = 2.35 \, \text{g/cm}^3$; particle content 30 Ma.-%).

2.2. Correlation of statistical feature to particle concentration

The emitted sound waves ($f = 4.5$ MHz) have a wavelength of approximately $330 \, \mu\text{m}$ in the suspension, which is one scale above the size of the particles ($x_{50} = 12 \, \mu\text{m}$). Accordingly, the particle scattering is weak [4]. In accordance with the following measurements for different concentration the reflection amplitude and especially its standard deviation can be processed even in case of such weak scattering.

Fig. 3: Standard deviation in logarithmic scale for rising particle concentration
In the course of the measurements the concentration was increased up to mass content of 50 Ma.-%. Fig. 3 shows the standard deviation in logarithmic scale over time for different concentrations. Since a correlation of the standard deviation to the particle concentration is postulated the descent of the curves is equivalent to their time dependent exponential attenuation (e.g.: 0.5 dB/µs in case of 10 Ma.-%). This decrease changes with rising concentration.

By the prove of example the validity of the correlation to an equivalent attenuation calculated by the standard deviation of the scattering signal is shown. Table 1 exemplifies the results of the water suspension with SiO₂-Particles (density: 2,35 g/cm³, size < 20 µm). The equivalent scattering attenuation and the transmission attenuation which was additionally verified by a conventional transmission spectrometer [5] show a direct correlation (Tab. 1) and allow for the determination of particle concentration. To compare the equivalent values with the reference values the time dependent attenuation [dB/µs] was transformed to spatial domain by using the corresponding sound velocities (1450…1500 m/s) that change with concentration. Finally, a normalization to the centre frequency (4.5 MHz) was applied.

Table 1: Ultrasonic attenuation coefficients calculated from the backscattered signals in comparison to conventional attenuation values gained with a reference ultrasonic transmission spectrometer (DT1200). Different values for SiO₂-suspensions with rising mass content [Ma.-]% in distilled water are shown.

<table>
<thead>
<tr>
<th>Solid content SiO₂ [Ma.-%]</th>
<th>Attenuation of back-scattering [dB/µs]</th>
<th>Attenuation of back-scattering [dB/cm/MHz]</th>
<th>Transmission attenuation Reference: spectrometer (DT1200) [dB/cm/MHz]</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0,5</td>
<td>0,75</td>
<td>0,63</td>
</tr>
<tr>
<td>20</td>
<td>0,96</td>
<td>1,44</td>
<td>1,36</td>
</tr>
<tr>
<td>30</td>
<td>1,43</td>
<td>2,16</td>
<td>2,12</td>
</tr>
<tr>
<td>40</td>
<td>1,9</td>
<td>2,89</td>
<td>2,84</td>
</tr>
<tr>
<td>50</td>
<td>2,16</td>
<td>3,31</td>
<td>3,48</td>
</tr>
</tbody>
</table>

3. Conclusion

Concluding, the novelty of the presented approach is the extraction of the particle-concentration-dependent attenuation out of the statistical features of the backscattered (180° reflection) ultrasonic signal. The reflection configuration and the simple probe design (Fig. 1) overcome the measurement gap as a major drawback of conventional transmission configuration - normally limited in its inline applicability - and opens up for a variety of industrial applications dealing with high concentrated suspensions. The ultrasonic scattering attenuation coefficients in comparison to conventional transmission attenuation values show a direct coherence over 99%. Thus, assuming a constant particle size, the concentration can be quantified with this method already. In case of changing particle size additional parameter like the absolute value of the scattering amplitude near the windows surface (Fig. 3) need to be included. Studies show that this amplitude depends on concentration and the particle size. Hence, both parameters can be used to characterize the particle system.

References