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Measurement and characterization of particles by acoustic methods —

Part 1: Concepts and procedures in ultrasonic attenuation spectroscopy

Mesurage et caractérisation des particules par des méthodes acoustiques —

Partie 1: Concepts et modes opératoires en spectroscopie d'atténuation ultrasonique

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20998-1 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 20998 consists of the following parts, under the general title *Measurement and characterization of particles by acoustic methods*:

— *Part 1: Concepts and procedures in ultrasonic attenuation spectroscopy*

The following parts are under preparation:

— *Part 2: Guidelines for linear theory*

— *Part 3: Guidelines for non-linear theory*

Introduction

It is well known that ultrasonic spectroscopy can be used to measure particle size distribution (PSD) in colloids, dispersions, and emulsions (see [6][7][8][9]). The basic concept is to measure the frequency-dependent attenuation or velocity of the ultrasound as it passes through the sample. This attenuation includes contributions due to scattering or absorption by particles in the sample, and the size distribution and concentration of dispersed material determines the attenuation spectrum (see [10][11][12]). Once this connection is established by empirical observation or by theoretical calculations, one can in principle estimate the PSD from the ultrasonic data. Ultrasonic techniques are useful for dynamic on-line measurements in concentrated slurries and emulsions. Traditionally, such measurements have been made off-line in a quality control laboratory, and constraints imposed by the instrumentation have required the use of diluted samples. By making in-process ultrasonic measurements at full concentration, one does not risk altering the dispersion state of the sample. In addition, dynamic processes (such as flocculation, dispersion, and comminution) can be observed directly in real time (see [13]). This data can be used in process control schemes to improve both the manufacturing process and the product performance.

ISO 20998 consists of three parts:

— Part 1 introduces the terminology, concepts and procedures for measuring ultrasonic attenuation spectra;

— Part 2 provides guidelines for determining particle size information from the measured spectra for cases where the spectrum is a linear function of the particle volume fraction;

— Part 3 addresses the determination of particle size for cases where the spectrum is not a linear function of volume fraction.

Measurement and characterization of particles by acoustic methods —

Part 1: Concepts and procedures in ultrasonic attenuation spectroscopy

1 Scope

This part of ISO 20998 describes ultrasonic methods for determining the size distributions of one or more material phases dispersed in a liquid. Colloids, dispersions, slurries and emulsions are within the scope of this part of ISO 20998. The typical particle size for such analysis ranges from 10 nm to 3 mm, although particles outside this range have also been successfully measured. Measurements can be made for concentrations of the dispersed phase ranging from 0,1 % by volume up to 50 % or more by volume, depending on the density contrast between the continuous and the dispersed phases. These methods can be used to monitor dynamic changes in the size distribution, including agglomeration or flocculation in concentrated systems.

2 Terms and definitions

For the purposes of this document, the following terms apply:

2.1
absorption
direct reduction of incident ultrasonic energy by means other than scattering

2.2
attenuation
extinction
total reduction of incident ultrasonic energy, including both scattering and absorption.

NOTE The recommended measurement unit is the decibel (dB), which is defined as 10 times the common (base 10) logarithm of the ratio of incident intensity to transmitted intensity, or equivalently 20 times the common logarithm of the ratio of incident amplitude to transmitted amplitude. The neper (Np) is a permitted alternative measurement unit based on the natural logarithm, rather than the common logarithm. The conversion factor is 1 Np = 8,686 dB.

2.3
attenuation coefficient
extinction coefficient
attenuation (extinction) per unit length of ultrasonic propagation through a material, measured in units of dB/cm or Np/cm.

NOTE Attenuation coefficients are sometimes scaled by frequency, or frequency-squared, to identify the dominant attenuation mechanism. For clarity, in this part of ISO 20998, only the attenuation per unit length (in dB/cm) is considered.

2.4
attenuation spectrum
attenuation coefficient measured as a function of frequency

2.5
bandwidth
range of frequencies contained in an ultrasonic signal, typically measured as the frequency difference between the -3 dB points on a spectrum analyser

2.6
broadband
characterized as having a bandwidth that is equal to at least half of the centre frequency

2.7
digitization
act of generating a digital (quantized) representation of a continuous signal

NOTE The number of bits determines the resolution (fidelity), and the sampling rate determines the bandwidth (Nyquist criterion).

2.8
excess attenuation
incremental attenuation caused by the presence of particles in the continuous phase

2.9
Fourier transform
mathematical transform that converts a time-varying signal into its frequency components, which is often implemented in computers as a Fast Fourier Transform (FFT) algorithm

2.10
interference
wave phenomenon of cancellation or enhancement observed when two or more waves overlap

2.11
intrinsic response
frequency-dependent response of the ultrasonic spectrometer itself

NOTE This is not to be confused with the intrinsic absorption of the sample component materials.

2.12
path length
distance traversed by the ultrasonic wave between the emitting transducer and the receiver

2.13
pulse
wave of sufficiently short duration to contain broadband Fourier components

2.14
reflection
return of an ultrasonic wave at an interface or surface

2.15
scattering
removal of ultrasonic energy from the incident wave by redirection

2.16
spectrum
frequency components of a signal, typically arranged as magnitude versus frequency

2.17
tone burst
short duration of a few cycles of a sinusoidal wave

NOTE Typically, a tone burst consists of 5 to 10 cycles of a sinusoidal wave.

2.18
transducer
device for generating ultrasound from an electrical signal or vice versa

NOTE Piezoelectric devices are commonly used for this purpose.

2.19
transmission
passage of ultrasound through a sample

2.20
transmission spectrum
transmission value measured as a function of frequency

2.21
transmission value
amplitude of an ultrasonic signal (or a component thereof) that has been transmitted through a sample, measured in volts or arbitrary units

2.22
ultrasound
high frequency (over 20 kHz) sound waves which propagate through fluids and solids

NOTE The range employed in particle characterization is typically 100 kHz to 100 MHz.

2.23
wave
fluctuation, e.g. pressure, shear or thermal, which propagates through a physical medium

2.24
waveform
shape of the wave when seen on an oscilloscope or digitized display

2.25
wavelength
length of a wave, determined by the distance between corresponding points on successive waves

3 Sampling and reference materials

3.1 Sampling considerations

3.1.1 Dry powders

It is necessary to disperse a dry powder in a liquid before measuring the ultrasonic attenuation spectrum. A representative sample of the powder shall be used in the preparation of the liquid dispersion. It is recommended that sampling procedures be carried out in accordance with ISO 14488. Dispersion of the powder should be carried out in accordance with ISO 14887.

3.1.2 Suspensions and slurries

The apparent particle size in flocculated or poorly-dispersed systems changes as a function of the applied shear stress. Therefore, unless floc size or quality of a suspension is to be measured, it is recommended that suspension and slurries be mixed thoroughly before a sample is withdrawn for ultrasonic analysis. The stability of the suspension impacts the results.

3.1.3 Emulsions

Many phenomena affect the homogeneity of emulsions, including creaming, droplet coalescence and phase separation. These changes affect the observed ultrasonic attenuation spectrum. If the initial droplet size distribution is to be measured for unstable emulsions, it is recommended that the sample be prepared immediately before the measurement.

3.2 Reference materials

3.2.1 Reference liquid

The use of a reference liquid is required in order to verify correct operation of the ultrasonic spectrometer itself. De-gassed clean water at ambient temperature has a relatively low attenuation coefficient (see [14]). Water is therefore recommended as a reference liquid for determining the intrinsic response of the spectrometer. A procedure for de-gassing water is given in IEC 62127-1.

3.2.2 Reference sample

The use of a reference sample is recommended to verify the correct estimation of particle size distribution from the observed attenuation spectrum, but no reference material has yet been identified for general use. The user should identify a well-characterized and stable material as a standard sample for monitoring variability in the size distribution results.

4 Methods

4.1 Principles

As ultrasound passes through a suspension, slurry, colloid or emulsion, it is scattered and absorbed by the discrete phase, with the result that the intensity of the transmitted sound is diminished. The attenuation coefficient is a function of ultrasonic frequency and depends on the composition and physical state of the particulate system. The measurement of the attenuation spectrum can be used to estimate the particle size distribution and concentration. The necessary apparatus is described in 4.2.

The total measured attenuation is due to the intrinsic absorption of the continuous liquid phase, the intrinsic absorption of the dispersed phase(s), thermal losses, viscous losses and scattering losses (see [6][7]). The relative importance of these loss mechanisms depends on the material system. A theoretical or empirical model of these mechanisms can be used to convert the observed data into an estimate of the particle size or particle size distribution. There is no single general procedure for determining particle size information from the attenuation spectrum. Different models and procedures are used depending on the application and nature of the sample, as described in ISO 20998-2.

The attenuation spectrum can be measured as long as the signal-to-noise ratio is adequate. However, an *a priori* theoretical model may not exist due to lack of knowledge about the dispersed or continuous phases. In cases where there is no suitable theoretical or empirical model available to describe the interaction of ultrasound with the system of interest, the attenuation spectrum can still be used to infer relative changes in particle size (see [13]).

A variety of techniques (see Annex A) have been used to measure ultrasonic spectra. Some of these methods have been implemented in laboratory instruments and some have been used in industrial applications. Ultrasonic spectroscopy has been used to measure particle size in a variety of material systems. Example applications are listed in Annex B.

4.2 Apparatus

4.2.1 General

As a minimum, the spectrometer consists of an excitation source, one or more ultrasonic transducers, a sample cell (or flow cell, in the case of in-process instruments), a preamplifier and a means for acquiring the signal. Each of these components shall be tailored to fit the particular needs of the implementation.

4.2.2 Excitation source

The excitation source produces the electrical signal that is converted by the transducer into ultrasonic waves. This circuit determines the frequency content of the resulting ultrasonic signal. This source may produce a continuous wave at a single frequency, a frequency sweep, a set of tone bursts (which may be at various frequencies), step pulses or broadband pulses. The frequency response (bandwidth) and electrical impedance of the source should be matched to those of the transducer. The output signal level can range from a few volts up to a few hundred volts, depending on the application and transducer type.

4.2.3 Transducers

Ultrasonic transducers convert the electrical signal from the excitation source into ultrasonic waves. The active element within the transducer is typically made of a piezoelectric material (such as barium titanate) or a piezoelectric polymer film (such as PVDF). When excited by an electrical signal, the piezoelectric element constricts and relaxes, sending a longitudinal compression wave through the facing material and into the dispersion. An acoustic delay line or buffer plate may be attached to the front of the transducer to protect it.

The construction of the transducer affects the frequency response. If the backing material heavily damps the vibration of the element, the natural resonance will be de-tuned, giving a broadband response.

In the through-transmission method (see Annex A) a second transducer is used to detect the transmitted ultrasonic waves and convert them into electrical signals. Due to the reciprocity theorem, the receiving characteristics of a transducer are the same as the emission characteristics. In a pulse-echo method, the same transducer is used to send and receive the ultrasonic pulses. In both arrangements, alignment of the transducers is important, and typically becomes critical at frequencies of the order of 10 MHz and higher.

Alignment of the transducers is achieved by pivoting them relative to each other, so that the bandwidth and signal strength of the ultrasonic signal are at a maximum. This action in effect aligns the directional patterns of emission and reception, which can be skewed with respect to the mechanical axis of the transducers. If the alignment is not correct, destructive interference at the edge of the receiver distorts the transmitted signal.

4.2.4 Sample cell

The sample cell is used to contain the dispersion and maintain the transducers in alignment with each other. This element is optional, as some instruments are designed as a probe that is inserted directly into a process vessel; in such cases, the probe body holds the transducers.

If a sample cell is used, the dispersion sample shall be circulated or stirred in order to prevent sedimentation. An exception to this requirement can be made in the case of stable suspensions where particles do not settle.

For in-process applications, if a flow cell design is used in which the process fluid (i.e. dispersion) flows through the cell, it is important to maintain an open bore all the way from inlet to outlet, in order to prevent plugging.

4.2.5 Preamplifier

The preamplifier is an optional element that boosts the relatively weak signal detected by the receiving transducer. Typically, this element will add 20 dB to 60 dB of gain to the signal. The bandwidth and input impedance of the preamplifier shall meet the needs of the transducer.