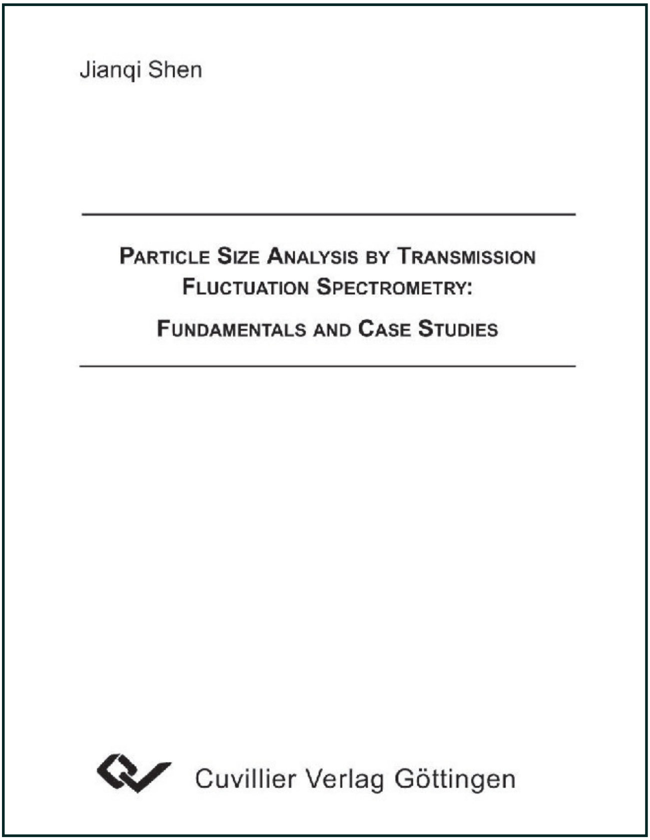
Jianqi Shen (Autor)

Particle Size Analysis by Transmission Fluctuation Spectrometry Fundamentals and Case Studies



Jianqi Shen

PARTICLE SIZE ANALYSIS BY TRANSMISSION
FLUCTUATION SPECTROMETRY:
FUNDAMENTALS AND CASE STUDIES

Cuvillier Verlag Göttingen

https://cuvillier.de/de/shop/publications/3100

1 INTRODUCTION

Particle size distribution and concentration are known to be critical parameters of particulate materials in the operation of industrial process such as wet milling, suspension crystallization, suspension polymerization and hydraulic conveying: They control the filterability, flowability, friability and dryability of the particulate materials. And they are also very important in the end-use applications of the materials [1-3]. For example, it determines the setting time of cement, the hiding powder of pigments, the activity of chemical catalysts, the efficacy of drugs and the taste of chocolate.

A number of theories and instruments have been developed for getting particle characteristics, most of those are expensive and complicated to use. The measurements, in the past, were usually achieved by manual sampling followed by sample preparation (such as drying, mixing, crushing and dividing) and off-line laboratory analysis. However this procedure is often subject to significant sampling errors and, most importantly, the measurements are too slow for control purposes. By contrast, on-line analysis can provide rapid and accurate measurement in real time and thus opening up new possibilities for improved quality control and optimization of product yield. As a result, there has been a rapid increase in the industrial application of on-line analysis instrumentation over the past few decades [4].

The light scattering methods are among the most effective approaches for the measurements of particle sizes and distributions. Indeed they have several advantages over other methods, such as electron microscopy and sieving, because high-quality measurements can be performed in situ and in real time.

Optical techniques can provide either direct information on particle distributions when measurements are carried out on single particles one at a time or indirect information when the techniques are used to study systems containing many particles. The latter case is the most convenient because measurements can be performed simultaneously on a large number of particles and because of simple experimental setups. Indirect optical characterization is primarily achieved by measuring (1) the angular distribution of the light scattered by the particles or (2) the extinction of light through the particle system.

The extinction technique is very simple in terms of measurement principle and very convenient with regard to the optical arrangement. Traditional extinction measurements are based on Bouger-Lambert-Beer's law [5,6], describing the attenuation of the intensity of a beam passing through a particle suspension where the beam diameter is much larger than the particle size. Based on a quasicontinuum approach, the BLBL does not account for the discrete nature of the particles, hence it cannot apply to the case in which particle size is close to or larger than the beam diameter. In 1985, Gregory [7,8] published his paper, establishing a technique of extinction measurement by a narrow beam. By using the average and the standard deviation of the fluctuating extinction value, the average particle size and the particle concentration are determined. Unfortunately, this approach is suitable only for narrow sized particle systems, as only an average value of the particle diameter can be obtained so that signals from broader size distributions cannot be interpreted properly. This technique was, later, developed by Wessely and Ripperger et al [9,10]. With an additional variation of the size of the measuring light beam, the particle size distribution (PSD) can be determined.

Recently, the transmission fluctuation spectrometry (TFS) was developed, by Riebel, Kräuter and Breitenstein [11-17], as a new method of particle size analysis. Also based on the statistical fluctuations of the transmission signal, this method uses a more sophisticated mathematical treatment of the transmission signal in order to extract the full information on the particle size distribution and particle concentration.

The basic idea of the TFS came from the case of a disperse system which is irradiated by a circular beam of light [11]. The probability of the beam to pass through the particle system without interaction with a particle is found to be a function of the beam-to-particle diameter ratio. The function is shifting with the particle concentration, while the shape of the function remains virtually independent of the particle concentration. This idea was developed in Kräuter's thesis [14], where the transmission fluctuations were expressed in terms of the expectancy of the transmission square (ETS). By varying the beam diameter, the transmission fluctuation spectra for variable spatial averaging of the transmission fluctuation signals were obtained both experimentally and with simulations at different particle concentrations. An empirical expression of the ETS was obtained.

The TFS was also developed into the case where the particle suspension is irradiated by an infinitesimally narrow beam [14,15]. An analytical solution for the description of the transmission fluctuations in terms of the expectancy of the transmission square was provided for the case of a variation of the temporal resolution of the signal capture. The spectrum was obtained with a gliding average over variable time intervals. Unfortunately, this is difficult to realize in practice, especially with small particles, due to the widening of narrow beams because of diffraction.

Thus a more general approach was developed [16,17], applying to variable combination of temporal averaging and spatial averaging of the transmission fluctuations. This new theory allows to describe the transmission fluctuation behavior of beams with a circular cross section and uniform beam intensity distribution. So far, the theory is based on a layer model of the suspension and it is restricted to low particle concentrations and geometric ray propagation of the radiation. The experimental implementation of such conditions is difficult.

First, the theory is restricted to a circular beam with a uniform intensity distribution. This would require a complicated optical arrangement. From the viewpoint of practical applications, a laser beam in Gaussian mode may be a more direct choice.

Secondly, temporal averaging of the transmission signal in the time domain is more or less time-consuming, which is not ideal for real-time measurements. Since the solution is obtained as a spectrum, it is possible to do temporal averaging with signal filters at various cutoff frequencies instead of the gliding average over various time intervals. The analog signal analysis in the frequency domain, which is easy to realize with electronics, allows the real time measurement.

The objective of this thesis is to extend the theory to the case of Gaussian beams of variable diameter and to signal processing in the frequency domain. Considering the real applications where the particle concentrations are high, the theory is validated to concentrated suspensions. Effects of particle-particle interactions such as the monolayer structure and particle overlapping are studied with simulations and measurements.

Chapter 2 gives a review of the transmission fluctuation spectrometry, based on a layer model, including the special cases of temporal averaging of the transmission fluctuations within an infinitesimally narrow beam, spatial averaging of transmission fluctuations with a variable beam diameter and the combined temporal and spatial averaging in the time domain and within a circular uniform beam. The transmission fluctuations are expressed in terms of the expectancy of the transmission square (ETS).

In chapter 3, the TFS theory is extended to any combinations of temporal averaging in the frequency domain (or in the time domain) and spatial averaging within a Gaussian beam (or a circular uniform beam). The transition function of the expectancy of the transmission square is factorized into parts, describing the characteristics of temporal averaging, spatial averaging, particle size and shape, and the monolayer structure. High concentration effects from the monolayer structure are discussed in detail.

Measurements on dilute to moderately concentrated particle suspensions are given in chapter 4 using a simple optical arrangement with a focussed Gaussian beam and an electronic signal processing system, consisting of an array of 12 parallel channels with different cutoff frequencies. The modified Chahine inversion algorithm [18] is employed to extract the particle size distributions and the particle concentrations from the measurements.

Effects from steric interactions between particles in concentrated suspensions, including the monolayer structure and particle overlapping, are studied in chapter 5 theoretically and in chapter 6 by simulations. An empirical correction of the high concentration effects is obtained, which is employed in the inversion algorithm, in chapter 7, to achieve a correct evaluation of the particle size distributions and particle concentrations for concentrated suspensions.

The last chapter gives some general conclusions, concerning the future development of transmission fluctuation spectrometry and the applications.