Particle Size Analysis 1981

Edited by
N. STANLEY-WOOD and T. ALLEN

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Proceedings of the
Fourth Particle Size Analysis Conference,
Loughborough University of Technology,
21 – 24 September 1981

Organised by the Analytical Division of
The Royal Society of Chemistry

Co-sponsored by
German Institute of Chemical Engineers, (GVC), West Germany
The Fine Particle Society, Washington, DC, USA
STF – Ingenjorstbildning, Stockholm, Sweden

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A Wiley Heyden Publication

175 YEARS OF PUBLISHING 1807 1982

JOHN WILEY & SONS
Chichester · New York · Brisbane · Toronto · Singapore

British Library Cataloguing in Publication Data:

Particle Size Analysis Conference *(4th: 1981: Loughborough University of Technology)*
Particle size analysis.
1. Particle size determination—Congresses
I. Title II. Allen, T. III. Stanley-Wood, N.
IV. Royal Society of Chemistry. *Analytical Division*
620'.43 TR418.8

ISBN 0 471 26221 8

Printed in Great Britain by Mansell Ltd., Essex.

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FOREWORD

The Particle Size Analysis Group of the Analytical Division of the Royal Society of Chemistry is pleased to present these Proceedings of its Fourth International Conference on particle size analysis. It is believed that this volume will make an important contribution to the advancement of particle characterization as an analytical science, by helping to increase the understanding of the science upon which the analytical techniques are based; to further the application of untapped scientific principles to new methods of particle characterization; to improve established techniques in respect of accuracy, precision, speed and applicability; to make advances in the calibration and standardization of these techniques; and to promote the further education of those scientists, engineers, technicians and managers who are either directly involved in the practice of particle characterization, or who have to apply the results in their work.

The success of this conference can only be partly judged by these formal proceedings. The success was largely due to the efforts of a great many people whose help the Particle Size Analysis Group Committee now wish to acknowledge. First, we would like to thank all the authors and participants, especially the Session Chairmen, whose names appear under the title of each session, for their assistance in conducting the scientific programme at the meeting. We particularly with to recognize, with thanks, the tremendous efforts of the following members of the Local Organizing Committee who were responsible for the logistics of the meeting: Mr P.J. Lloyd, Mr C.R.G. Treasure, Mr R.E. Buxton, Mr R.H. Beresford and Mr B. Scarlett. We also wish to thank the management of the University of Technology, Loughborough who cooperated so effectively in supplying facilities. We are grateful to the firms who supported the exhibition of particle sizing equipment which was a very important part of the conference. Finally, we are deeply indebted to Dr T. Allen and Dr N.G. Stanley-Wood for their able assistance in soliciting and selecting papers before the meeting and in editing them for these proceedings.

Michael W.G. Burt

Chairman of the
Particle Size Analysis Group
and Conference Chairman

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Trends in Fineparticle Characterization Techniques

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Perhaps the first thing that one should comment on in a review lecture of this nature is the title. Over the years committees and editorial boards have struggled with the task of finding a name for the scientific study of finely divided materials. Thus when the Journal "Powder Technology" was established the editorial board spent two hours before coming up with a title "Powder Technology". At the time I strongly resisted this title since it would not obviously include aerosols and mists. It was with some satisfaction I noticed several years later that the editors of Powder Technology found it necessary to add an editorial indicating that it would accept papers on spray systems and other wet systems. At that time the subtitle 'An International Journal on the Science and Technology of Wet and Dry Particulate Systems' was added to the front cover of the journal. With regard to alternative terms I must admit to some personal animosity towards the term 'particulate' which I always think is more adjective than noun. When a society for promotion of the study of finely divided materials was established in the United States it took as its title The Fine Particle Society but failed to link the 'fine' and the 'particle' to make a compound noun. The scientific community is perverse in its attitude to compound nouns. It will happily live with terms such as 'photomicrograph' which have latin and greek roots strung together to form a long compound noun but will express horror at a noun such as 'fineparticle'. As will be set out in greater length elsewhere it can be shown that the term fineparticle has many advantages over particulate when it comes to structuring automated information searches through computer systems for information on finely divided material and therefore I intend to use the term 'fineparticle' throughout this review. [1 and 2].

The confusion attendant upon words such as 'particle size analysis' and 'particulate contamination' become particularly acute when one is studying problems associated with radiation hazards to uranium miners. Engineers become confused as one talks about alpha particles on inhaled particulates which are generated by particle emission on the disintegration of uranium atoms! The compound noun 'fineparticle' has not been abused or misunderstood so that one can invest in it a unique meaning which the writer proposes as follows:

> A fineparticle is a small unit of matter
> having a minimum dimension of approximately

> a thousand microns and whose behaviour is determined by a significant interaction of surface forces and the classical forces of traditional physics. Fineparticle science fades into colloidal science as minimum dimensions fall below approximately .1 micron.

Again, if one works with problems of dust inhalation the analysis of the chemical nature of the fineparticle as distinct from a description of the size and morphology of the fineparticle becomes an important distinction. Therefore, it is preferable to restrict the term 'fineparticle analysis' to a determination of the chemical structure of a fineparticle. One then uses the term 'fineparticle characterization' for techniques involving measuring size and shape.

The early techniques for characterizing fineparticles used in the period up to the late 1940's depended heavily on sieves, elutriators and microscopes. Standard specifications for characterization procedures prior to 1950 specified these types of devices [2]. However, the fineparticle community was quick to recognize that the electronics revolution which came after the conclusion of the Second World War offered great possibilities in the automated characterization of fineparticles. As a result of the utilization of advanced electronic techniques the first major conference on fineparticle characterization techniques took place at Nottingham [3]. In the period from 1954 up to 1974 the methodology improved rapidly with the introduction of instruments such as the Coulter Counter, other resista-zone counters and automated image analyzers. Groves has described the development of instruments during this period [4]. In this lecture we will essentially focus on developments which have occurred since the 1977 Symposium in Salford and will attempt to look at future trends in the coming decade [5]. We will discuss developments instrument by instrument. Because of the wealth of information available it will only be possible to mention highlights of recent trends.

PERMEABILITY - THE FORGOTTEN METHOD

One of the earliest techniques used for characterizing a fineparticle system was the permeability method. In this technique the powder under-study is compacted to form a plug which is then inserted into a pneumatic or liquid circuit [6]. The resistance offered to the flow of a fluid by the plug is then used to calculate the surface area of the powder. A major experimental problem encountered in this technique is the fabrication of a suitable plug with reproducible physical properties [7]. It has recently been demonstrated that many of the problems associated with the fabrication of the plug can be overcome by using isostatic compression of the powder plug by a hydraulically driven rubber membrane surrounding a cylindrical powder plug used in most methods for characterizing powders by permeability [8].

The hydraulic compression of the powder plug results in a system less sensitive to porosity fluctuation and also creates a plug which can be used in automated systems.

Many different measurement circuits have been used to measure the resistance to flow offered by a fabricated powder plug. When discussing the various permeability measurement procedures it is useful to refer to the analogous electrical circuits used to measure electrical resistance. Thus pneumatic analogues to the Wheatstone Bridge and other DC current circuits for measuring electrical resistance have been employed in various pneumatic circuits. In 1974 Stearn and co-workers described an AC analogue pneumatic system for measuring the fineness of fibres [9, 10]. The technique is directly applicable to powder systems but appears to have been overlooked in the fineparticle literature. In this system a plug of powder to be characterized is interposed between one air reservoir containing a loud speaker and another reservoir containing a microphone. The loud speaker diaphragm is driven at 50 Hz. and the microphone picks up the signal passing through the plug. The technique has been tested at Laurentian University for characterizing powders and it is hoped to publish a report in the near future [11].

Early in the days of permeametric techniques it was realized that the derivation of a surface area from the measured fluid flow resistance was an artifact and a great deal of study has gone into modelling the flow of fluids through the powder compact in order to improve the equations used to derive the calculated surface area from the plug resistance. The fact that the measured surface area is a derived characteristic has resulted in some scientists having an aversion to the method on philosophical grounds. However, the technique is inexpensive and is extremely useful in the cybernetic mode. In the cybernetic mode the system is used to detect differences between powders. Used in this way it can be very effective in the quality control laboratory. When using the cybernetic mode the machine is presented with various plugs of powder which represent the various levels of acceptability of product output. The measured performance and known characteristics of these plugs are stored in a memory system and used to check the performance of a new unknown sample. The machine can then decide the most probable description of the powder plug and its most valuable use.

HOLOGRAPHY

One of the earliest applications of holography was in the area of fineparticle characterization. In particular Thompson and co-workers [12] developed systems for looking at mists and such exotic systems as the exhausts from space rockets. Recently realtime holography of rapidly changing systems has been given a boost by studies reported by Trolinger and co-workers. They have studied the combustion of fluidized coal fineparticles in connection with the development of fluidized bed combustion of coal [13, 14]. Indeed the massive switch to the use of coal powders in the United States is giving tremendous impetus to the development of instrumentation for studying transient aerosol systems. For the same reasons there is increasing interest in the rheology of systems such as coal oil slurries [15].

LASER DOPPLER VELOCIMETRY

The characterization of fineparticles by studying the shift in the frequency of laser light bounced off of a moving fineparticle has developed at a tremendous pace. Whole books devoted to the subject have been published an it is not possible to give more than a general comment in this review lecture [16]. The tremendous interest being focused on the use of laser doppler velocimetry, the North American name for this technique, (known as laser doppler anemometry in Europe) arises from a growing realization that many transient aerosol systems, such as Sprays, Smokes and inhalable aerosols in the work environment, need to be studied in situ rather than by capturing them on filter papers for subsequent evaluation. The technical community can anticipate continual development and lowering of costs for such instrumentation in the next decade. Laser doppler shift measurements are also being used to study Brownian motion. In this situation, however, the amount of light energy involved is so low that one must be concerned with individual photons. As a consequence the technique is often referred to as laser-photon correlation spectroscopy. The nanometer equipment sold by the Coulter Electronics organization is based on this method and extensive development of the techniques have been carried out by Langley Ford and co-workers [17, 18, 19, 20].

IMAGE ANALYZERS

All of the major manufacturers of image analysis equipment based on television linescan logic have continued to upgrade and extend the logic operations of their instruments [2].

A particularly interesting innovation has been reported by Tracor Northern [21]. In this instrument a probe controlled by a computer searches a field of view along a scan line such as $\alpha \to \beta$. When it encounters a profile at a point such as A it measures the chord AB. It then returns to the mid point and creates a chord at right angles to AB and measures its magnitude. It then returns to the mid point of AB and explores the magnitude of the two chords at 45^o and 135^o to the direction of AB. It then repeats the process until 8 equally angularly spaced chords have been evaluated and it then moves along the direction AB or some other direction.

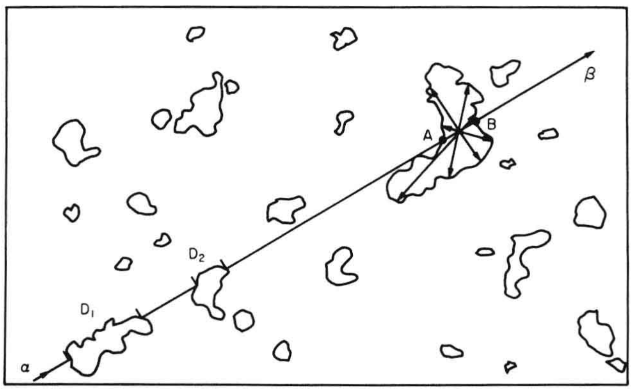

Figure 1. Search and explore image analysis system could revolutionize iconometrics in the next decade.

The average value of the 8 chords and their variation about their mean can be related to significant features of the fineparticle profile. Such a search and find procedure can also evaluate the sum of the various intercepts D_1 and D_2 along the scan line. This sum expressed as a fraction of the total scan length is the area coverage of the field of view [2]. Other workers in image analysis, or Iconometrics as the subject is often referred to, are placing emphasis on increased logic sophistication to recognize the pressure of fibres such as Asbestos present in a field of view containing many other irregular fineparticles [22].

SIGNATURE WAVEFORM CHARACTERIZATION OF FINEPARTICLE MORPHOLOGY

Early in the 1970's several workers started to use waveforms as a means of describing fineparticle shape. First Schwarz and Shane transformed a profile of a fineparticle into a waveform by plotting the magnitude of a vector rotating about the centroid of the circumscribing circle. This waveform could then be broken down into its components by Fourier analysis. A list of the harmonic components obviously constitutes a non-dimensional description of the shape of the profile. In essence the rotating vector was used to transform a two dimensional description of a profile into a one dimensional waveform which could then be described in non-dimensional terms via Fourier analysis. Beddow and co-workers have developed this method extensively using the centroid of the lamina representing the profile as the reference point. They have been able to establish that one can correlate the harmonics of the waveform generated in this manner with essential features of the morphology of the profile [23]. A complex waveform can be broken down into components other than simple harmonic waves. One technique which has been vigorously pursued by signal engineers in the electronic industry is to use Walsh functions. Digital square form signals in sequence can be regarded as constituting one of the simpler Walsh functions. A chess board has a two dimensional Walsh function. Since signal engineers are involved in the propagation of square wave pulses in digital signalling systems it is not surprising that they have found Walsh functions useful in their technology. As early as 1968 Meltzer and co-workers suggested that two dimensional Walsh functions may be useful in the description of the shape of leaves [24]. Meloy and Beddow have applied Walsh functions to the analysis of waveforms from fineparticle profiles but such a technique does not seem to offer any significant advantage over Fourier analysis of such waveforms.

Highly irregular waveforms are difficult to transform with simple vector transformations although such transformation can be achieved by using lists of tangents at various positions around the profile to constitute the waveform for subsequent analysis [25, 26]. Recently Kaye has shown that one can create a waveform which is characteristic of the three dimensional morphology of a convex fineparticle. In this waveform, described as the Cauchy waveform, the waveform is a list of the magnitude of projected areas against position in three dimensional space [2, 27]. Kaye has suggested that this can be used to characterize significant features of the morphology of a fineparticle in two different ways. In the so called systematic waveform the profile is manipulated about two independent orthogonal axes. This waveform then can be related to the morphology of the fineparticle. In an industrial situation the more useful waveform may be the so called Cauchy stochastic waveform. To generate this waveform the fineparticle to be characterized would be supported in such a manner that it can be rotated at random in the path of the beam of light being used to measure its projected area. For example in experiments proceeding at Laurentian University a fineparticle is maintained in a zone of a HIAC type counter with the use of an upward rising air jet. The different maximum dimensions of the area profile vector will appear with frequencies related to their relative dominance of the fineparticle profile. If this stochastic waveform is subjected to autocorrelation analysis the features of the autocorrelation performed with different Δ T's will yield features related to significant features of the fineparticle morphology [27, 28].

FRACTAL DESCRIPTION OF FINEPARTICLE PROFILES

In 1976 Mandelbrot published an exciting book entitled "Fractals - Form, Chance and Dimension" [29]. It was immediately recognized by several workers that the mathematics developed in this book were applicable to problems in fineparticle science. The first direct description of the use of dimensions intermediate between 1 and 2, the fractal dimension described by Mandelbrot to describe fineparticle profiles, was presented by the writer at the Conference on Fineparticle Characterization held at Salford in 1977 [30]. Since then publications by several workers have led to a rapid development of studies in this field [31, 32, 33, 34, 35].