POWDER TECHNOLOGY SERIES

# PARTIC

DIMENTATION METHODS

CLASSIFICATION

Claus Bernhardt



## **Particle Size Analysis**

## Classification and sedimentation methods

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#### **Particle Size Analysis**

To my wife Ariane

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## Preface to the English edition

Today more than ever, experts concerned with the preparation, processing and analysis of disperse systems need methods of measurement which provide information on the quantitative distribution of the characteristics of particle size and particle shape. They have to select from a wide range of methods and devices from which it is by no means easy to make a choice. This situation is confirmed and aggravated by the huge amount of related publications, the subjects of which extend from theoretical fundamentals to the development and improvement of devices and techniques, to experience gathered in using these methods and to special systems of particles.

The beginnings of scientific particle size analysis reach back to the middle of the nineteenth century. In this period, they were mostly determined by the needs and development trends of other branches of science such as soil science and physical chemistry. Only at the beginning of the twentieth century did particle size analysis become an independent discipline, proved by the books of von Hahn ([H24] 1928) and Geßner ([G14] 1931). This was followed by the monographs of Herdan ([H18] 1953), Rose ([R33] 1953) and Cadle ([C25] 1955), as well as Orr and Dallavalle ([O6] 1959), Batel ([B19] 1960, 1964, 1971), Irani and Callis ([I1] 1963), Lauer ([L7] 1963) and lastly Allen ([A2] 1968, 1975, 1981) and Kouzov ([K17] 1971, 1974, 1987).

In the last two decades, it appears to have become a common conviction that the considerable technological progress made in this field requires monographs dedicated to the subdisciplines of particle size analysis. By its restriction to classification and sedimentation methods, this monograph is intended to promote a deeper understanding of their theoretical fundamentals, which are scattered over many branches of science. On this basis, an attempt is made to illustrate technical problems of measurement and their effect on the interpretation of results. In so far the book presupposes the reader to have a certain degree of experience in the field of granulometric analysis. At the same time, it is intended for the novice who is advised to look behind appearances for the working principle and its controlling influences.

If the concepts of granulometry and granulometric analysis are used in the text, this is not only a tribute to German usage but also a reverence for my

teacher Professor Ernst-Joachim Ivers to whom I still owe many insights 20 years after the end of his working life.

This English edition is not an unedited translation of the German edition of 1990. The text has been substantially revised in some chapters, taking into account the literature published in the mean time.

I wish to thank Dr.-Ing. H. Finken, Freiberg, who has prepared the translation from German into English with deep scientific understanding and in close contact with the author.

I also wish to express my gratitude to Chapman & Hall for their support to this project without which the English edition could not have been published.

> Dr.-Ing. habil. C. Bernhardt Freiberg

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# Position, tasks and structure of particle size analysis

Today the concept of **particle size analysis** is that of a special field of particle measurement technology, which in turn is part of particulate technology. This classification has developed over the last 20 years; it is the result of a scientific integration process taking place in many industrialized countries of the world.

In recent years, the meaning and mutual connection of the related concepts as well as the tasks of the scientific disciplines designated by them have been the subject of intensive discussion which, however, has not led to a generally accepted terminology.

The concept of particulate technology generally refers to the industrial processing of disperse material systems, and all states of aggregation are permitted both for the disperse and the continuous phase. Table 1.1 gives the most usual designations of the various combinations. The individual elements of the disperse systems are called particles; depending on their state of aggregation, they are called grains, droplets or bubbles.

It is evident that the disperse systems mentioned are very different in terms of their industrial importance. First place is doubtless occupied by the solid disperse systems, which very often are the subject or product of processes of conversion. Schubert [S79][S82] gives some examples:

- · preparation and processing of solid mineral materials including solid fuels;
- · utilization of secondary materials;
- manufacture of primary construction materials;
- many processes in the chemical industry;
- processes in the ceramics and glass industry;
- industrial processes in agriculture and the food industry;
- conservation of the biosphere.

Without doubt, the most important are the mechanical processes in which the solid disperse systems above are subjected to conversion. These are listed in Table 1.2.

Particle measurement technology by no means views its task only in the determination of the dispersion state of such systems but also deals with the

Table 1.1 Disperse systems

	Disperse phase (particles)			
Dispersion medium (continuous phase)	Solid (grain)	Liquid (droplet)	Gaseous (bubble)	
Solid	Conglomerate	Solid emulsion	Porous system	
Liquid	Suspension Slurry Slip Paste	Emulsion	Bubble system Foam	
Gaseous	Fume Aerosol Dust Powder Bulk material	Aerosol	=	

**Table 1.2** Mechanical processes for the conversion of solid disperse systems (according to [S11])

Objective	Processes	Examples
Change of mixing	Separation processes	Classification, sorting, filtration
state	Mixing Processes	Manufacture of suspensions and aerosols
Change of dispersion	Comminution Processes	Grinding
state	Agglomeration Processes	Pelleting, briquetting

technological characteristics resulting from the disperse properties. Leschonski [L44] names three basic tasks of particle measurement technology:

- 1. Characterization of a disperse solid by measurement of:
  - (a) particle size distribution;
  - (b) mean particle size;
  - (c) surface;
  - (d) shape;
  - (e) density;
  - (f) strength;

- (g) solubility;
- (h) adhesive forces.
- 2. Characterization of a two-phase flow by measurement of:
  - (a) spatial and temporal distribution of the solid;
  - (b) mean solid concentration:
  - (c) solid mass and volume flows;
  - (d) spatial and temporal distribution of particle velocity;
  - (e) mean particle velocity.
- 3. Characterization of a bulk material by measurement of:
  - (a) yield locuses;
  - (b) packing structures;
  - (c) pore size distribution;
  - (d) permeability;
  - (e) adhesive forces;
  - (f) capillary pressure;
  - (g) strength.

Ivers [18] proposed the designation granulometry or particle size analysis for the measurement of the characteristics 1(a)-(d) of solid disperse systems; this designation was synonymous with grain size analysis, granulometric analysis, etc. for a long time. On the basis of this proposal, Heidenreich later decisively expanded the concept of 'granulometry' in two directions [H54] [H55] [H56]:

- 1. All disperse systems as shown in Table 1.1 are included; measurement is carried out to characterize the granulometric or dispersion state before, during or after conversion processes.
- 2. A second task is to reveal the connections between the dispersion state and further material properties. Heidenreich [H54] sees these connections, which are usually called characteristic functions following a proposal by Rumpf [R32], in relation to:
  - (a) properties concerning the behaviour of the disperse substances during conversion (e.g. separability, mixability, flow characteristics, wettability, strength);
  - (b) properties determining the behaviour of disperse substances as products (e.g. effectiveness of grinding agents, setting of cements, lung permeability of dusts):
  - (c) properties of products of which disperse substances are essential components (e.g. flow behaviour and taste of chocolate, optical and strength properties of paper, strength of concrete).

Particle size analysis, i.e. measurements characterizing the granulometric or dispersion state, thus serves to obtain information on the size or shape of particles. The latter are called granulometric characteristics. The result of measurement is either frequency distributions or mean or integral characteristics of various types (see Chapter 8) which characterize the disperse system.

Therefore, the determination of granulometric frequency distributions of the particle size requires the measurement of parameters which are directly or indirectly derived from the granulometric characteristic 'particle size', and a suitable type of frequency of quantitative assessment.

Unlike mathematical statistics which employs only the number for frequency assessment, the following types of quantities are permitted in particle size analysis:

- number N
- size d
- area A
- volume V
- mass m.

For instance, measurable physical particle properties derived from the granulometric characteristic of particle size are:

- 1. Geometric parameters:
  - (a) length;
  - (b) area;
  - (c) volume.
- 2. Mass.
- 3. Sedimentation rate.
- 4. Field disturbance effects:
  - (a) in the electrical field;
  - (b) in the electromagnetic field (extinction, scattering, diffraction);
  - (c) in the fluid-dynamic field.

Particle sizes have for a long time been calculated from these measured particle characteristics by means of the so-called **spherical equivalence principle**: in place of the particle size x, the diameter of a sphere is given which produces the same measurement effect as the particle in question. Hence, the same value of the particle size x will always be measured for a particle on the basis of the above criteria only if it is spherical. If the shape of a particle is different from a sphere, different sizes will be measured by different methods. Therefore, a difference is made between surface, volume, sedimentation velocity or resistance, equivalent sphere diameters or particle sizes. Leschonski [L45] [A6] gives an inequality for some of these diameters:

$$x_{ps} > x_{pm} = x_A > x_V > x_W \tag{1.1}$$

where  $x_{ps}$  is the diameter of the sphere of equal projection area as the particle in stable position,  $x_{pm}$  the diameter of the sphere of equal projection area as the particle in mean position,  $x_A$  the diameter of sphere of equal surface area,  $x_V$  the diameter of the sphere of equal volume and  $x_W$  the diameter of the sphere of equal sedimentation velocity.

For instance, if the shape factor termed sphericity by Wadell [W4] is used

to describe the deviation from the spherical form

$$\psi_{w_2} = (x_V/x_A)^2 \le 1 \tag{1.2}$$

one obtains [L45]

$$x_{\mathbf{W}} = x_{A} \psi_{\mathbf{Wa}}^{3/4} = x_{\mathbf{pm}} \psi_{\mathbf{Wa}}^{3/4} = x_{V} \psi_{\mathbf{Wa}}^{1/4}. \tag{1.3}$$

Two conclusions can be made on this basis:

- 1. The use of different physical principles for particle size analysis leads to different results if the particle shape is not spherical.
- 2. The use of spherical substances is required to explain the causes producing the differences between results.

Finally, to be able to measure the granulometric state of a disperse substance, it has to be adapted to the selected method and the aim of measurement with respect to quantity, concentration and the degree of dispersion. For the quantity and concentration of the disperse substance, the principles of sampling and sample splitting are valid which provide the basis for the determination of the minimal amounts required. These, however, are not always in agreement with the potential and limits of the measuring devices. In the case where the sample size used by the device is smaller than that prescribed by the condition of representativity, additional errors have to be expected.

The selection of the correct or necessary degree of dispersion for the measurement is a problem which frequently cannot be satisfactorily solved. Particularly with fine and superfine materials, the disperse state is increasingly influenced by physico-chemical boundary surface phenomena and thus by the forces of interaction between particles. Wettability, destruction of agglomerates and the stability of the disperse system are problems which have to be solved prior to measurement. On the other hand, it is frequently difficult even to formulate the final objective of the measurement, for instance during the disagglomeration of solids in liquids. Moreover, there are some methods for which the spatial and temporal constancy of the dispersion degree, concentration, etc. of the disperse material subjected to measurement has to be ensured.

From the above, the immense influence of the phenomena of material behaviour on the measurement process becomes clear; more often than not, such phenomena are not sufficiently considered.

A particle size analysis would not be complete without the processing, evaluation and representation of the primary measurement data as dependent on the aim of measurement. In many cases, data have to be represented, densified, summarized or even adapted to the range of experience of the analyst. Computer side-effects, intended or unintended loss of information, erroneous smoothing of data, etc. are some examples which should be taken into account and which cannot be easily excluded at the current high level of automatic data processing.

The structure of the equipment used for particle size analysis derives from the range of tasks to be solved and the physical effects used for this purpose.

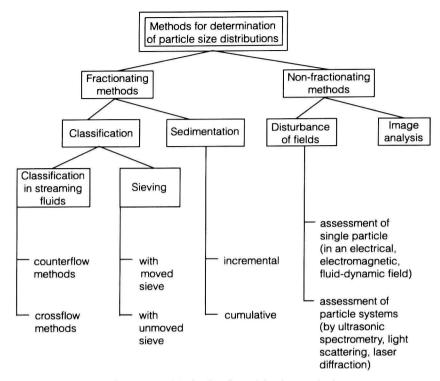


Figure 1.1 Methods of particle size analysis.

In this context, the overview given in Fig. 1.1 refers only to methods allowing the determination of particle size distributions. On the whole, a distinction has to be made between fractionating and non-fractionating methods. Non-fractionating methods comprise image analysis and all methods based on field disturbances. The latter either use effects allowing the measurement of individual particles in electrical, electromagnetic or fluid-dynamic fields, or assess collectives of particles by methods such as ultrasound spectroscopy, light scattering or laser diffraction. In this field particularly, there have in recent years been major breakthroughs which are essentially due to the increased use of computers for data acquisition and processing.

This monograph is dedicated to the other branch of particle size analysis, i.e. the fractionating methods of Fig. 1.1 comprising classification and sedimentation. Apart from other reasons, the combination of these two groups is based on the fact that some sedimentation methods, namely all decanting methods, make use of the principle of classification so that they should properly be defined as classification methods. In addition, all sedimentation processes, and especially the cumulative two-layer methods, produce states which only lack product separation to be described as genuine classification processes. Another

common feature of the fractionating methods is the overwhelming use of the quantity types volume or mass; in aerosol measurement and field flow fractioning, however, other approaches are used as well (e.g. quantity type number or area).

Finally, it is of decisive importance that the particle characteristics sieve aperture size and drag diameter are technologically relevant parameters. Thus, these methods are especially suitable for the characterization of products in the manufacture of which these characteristics are used. Leschonski [L44] extended this to the requirement to use measurement techniques 'the particle characteristic of which is in direct connection with the product property to be assessed. In this case, it is highly probable that the product property measured by this technique correlates with the measurement data of particle size analysis.

The selection of a certain measurement device on the world market requires the consideration and comparison of further aspects. Sresty and Venkateswar [\$78] name 10 selection criteria:

- 1. Objective
- 2. Required information
- 3. Type of dispersion medium
- 4. Range of particle sizes
- 5. Error
- 6. Number of samples
- 7. Mass of sample
- 8. Material properties of the sample
- 9. Time requirement
- 10. Purchase price and operation cost.

Information on most of these aspects can be obtained in overviews critically reviewing the current state of the art; some of these are [A2] [A30] [K60] [L11] [L44] [L45] [H54] [H55] [H56] [S78] [S80] [S81] [N21].