

PARTICLE SIZE DISTRIBUTION IN FOOD POWDERS

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Contents

- 1. Introduction
- 2. Methods for Particle Size Measurement
 - 2.1. Size Characterization Methods
 - 2.1.1. Direct Methods
 - 2.1.2. Indirect Methods
 - 2.1.3. Scanning Methods
 - 2.2. Size Ranges
- 3. Particle Size Distribution
 - 3.1. Particle Size Distribution Functions
 - 3.2. Measures of Central Tendency
- 4. Applications in Food Powder Field
 - 4.1. Attrition Evaluation
 - 4.2. Mathematical Treatment of Distribution Functions for Further Information
 - 4.3. Evaluation of Grain Hardness
- 5. Conclusion
- Glossary
- Bibliography
- Biographical Sketches

Summary

Particle size measurement and the determination of particle size distribution are essential to anyone dealing with particulate systems. Particles have different shapes and are involved in different processes. In this way, not only is choosing an appropriate method to measure particle size challenging, but also how to define size and shape. Once the appropriate definition of size and the technique to measure it have been determined, different approaches exist that can fit the data into a given distribution function.

1. Introduction

The characterization of a particulate system is usually made through the analysis of particle size and its distribution. Particle size measurement is most often required as a simple quality control technique, a method used to investigate changes in research and design process, and/or as a requirement in patent or product specification applications.

Indeed, particle size is typically a primary variable in all experimental tests and is essential to anyone dealing with particulate systems. The measurement of particle size distribution is one of the most widely used methods in the industry because of its importance in unit operations, such as mixing, extruding, and pneumatic handling of powdered materials (see *Food Process Engineering*).

Foods are frequently in the form of fine particles during processing and marketing. The bulk density, compressibility, and flowability of a food powder are highly dependent on particle size and size distribution (see *Physical Properties of Food Powders*). Segregation will happen in a free-flowing powder mixture because of the differences in particle size. Size distribution is also one of the factors affecting the flowability of powders. For quality control or system property description purposes, the need to determine the particle size distribution of food powders becomes apparent, including proper descriptors of size distribution that are essential in the analysis of handling, processing, and functionality of many food powders.

2. Methods for Particle Size Measurement

Technologies from various fields that are based on different principles are available to characterize particle size distribution; extensive reviews can be found on the subject. Thus, the major challenge in particle size determination is in selecting the best technology available and acquiring an understanding of the basic principles behind it, rather than creating a new technology.

As has been pointed out, ‘the assignment of a particle “size” to an irregularly shaped particle is philosophically problematic’. Except for spherical particles, there is no unique size parameter available to describe irregular shaped fine particles. Particles have different shapes and are involved in different processes during their manufacture and use. Therefore, the individual size descriptor (diameter) and the average characteristics of a given powder may vary depending on the objectives. As seen in Table 1, many definitions of diameter exist based on different characteristics.

d_v	Volume	Diameter of Sphere (DS) having the same volume
d_s	Surface	DS having the same surface
d_{sv}	Surface/volume	DS having the same surface/volume ratio
d_d	Drag	DS having the same resistant to motion
d_f	Free falling	DS having the same free falling speed
d_{st}	Stokes	DS having the same free falling speed in a laminar flow
d_a	Projected area	D of a circle with same projected area (stable)
d_p	Projected area	Idem but on a random position
d_a	Sieve	Width of the minimum square
d_f	Feret	Distance between parallel tangents
d_m	Martin	Chord on a fixed direction

Table 1. Criteria for determining the diameter of a particle.

[Adapted from: Allen T. (1997). *Particle Size Measurement*. London: Chapman and Hall Ltd.]

In addition, the average particle diameter may vary depending on the objectives. Assuming there are 10 particles with diameters ranging from 1 to 10, the arithmetic mean of this group is 5.5, the diameter of the particle with average surface area is 6.2, and the diameter of the particle of average volume is 6.71. As the particle area and volume are proportional to the square and cube of the diameter, respectively, the weighting factor to calculate the average diameter will depend on which characteristic is being described. Other characteristics, such as weight and inertia, are often considered in calculating the average particle diameter.

Because only a very small part of the particulate material in question is subjected to the determination procedure, it is essential that this part be representative of the total material in order to generalize desired information from the test results about the size distribution or other physical properties of the material (i.e., an unbiased sample of the statistical universe is needed).

2.1. Size Characterization Methods

Methods to characterize particle size are classified as direct and indirect. Among the direct methods, optical and image analysis are of major interest. Several indirect methods will be described in this review; these can be classified as sieving, sedimentation, fluid classification, and scanning.

2.1.1. Direct Methods

Direct methods are often referred to as absolute methods since particles are observed and measured “directly”. They allow not only size measurement but also morphology, texture, and mass and fractal dimension determination. Image analysis and optical methods are the most common direct methods.

Optical microscopy methods are used for particles ranging from 3 to 150 μm , the minimum size depending on the wavelength used and the refractive characteristics of the particles and suspension media. Particles less than the lower limit appear as diffuse circles and individual particles are difficult to distinguish from groups of particles. Thus, smaller particles tend to be over estimated in size.

Images seen in the microscope are projected areas of particles that tend to present the maximum area to the observer. Usually the area measured tends to be larger than that using other methods, because smaller dimensions of the particles tend to be neglected.

The measured diameters are normally Martin’s diameter, Feret’s diameter, or the projected area diameter (Figure 1). Feret’s diameter tends to be the largest of the three. The projected area is preferred since it considers two dimensions.

Transmission electron microscopy (TEM) and Scanning electron microscopy (SEM) are used to measure very small sizes. TEM typically ranges from 0.001 to 0.5 μm .

Image analysis techniques had virtually eliminated the manual methods. Systems for image analysis use scanning devices for converting images into digital information, which then can be processed. Algorithms, used by image analysis software, are calibrated with objects of known dimensions.

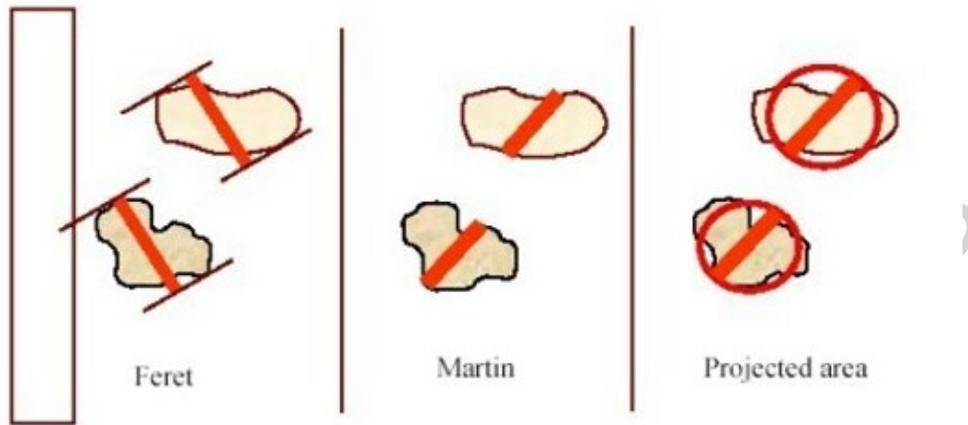


Figure 1. Feret, Martin, and Projected area diameters.

Surface texture, fractal dimension characterization, angularity, and roundness can be determined through image analysis. The major drawback in automatic image analysis systems is the difficulty in differentiating between clusters or “false aggregates” and single particles, mainly when analyzing particles with irregular shape. Erosion-dilation techniques are often used to solve this problem, although an overestimation of the number of fines may occur.

2.1.2. Indirect Methods

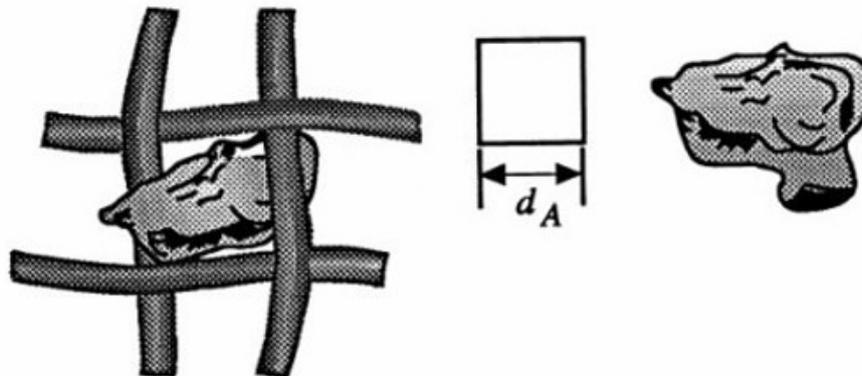


Figure 2. Particle size determination through sieve diameter.

[From: Allen T. (1997). *Particle Size Measurement*. London: Chapman and Hall Ltd.]

Sieving is the oldest method for particle size characterization. As seen in Figure 2, fractionation by sieving is a function of two dimensions. Sieve diameter (d_a) is defined as the minimum square aperture through which a particle can pass.

Different sieve progressions exist to characterize the size distribution of a population of particles. In 1867, Rittinger proposed a $\sqrt{2}$ progression of aperture size based on 75 μm . Modern standards are based on a fourth root of two progressions. USA standard sieves are described by the American Society for Testing and Materials (ASTM) as nominal apertures and permissible variations.

The basic process in sieving involves stacking the sieves in ascending order of aperture and placing the powder on the top sieve at the beginning and recording the weight of the particles retained on each sieve at the end of sieving.

A closed pan is placed at the bottom of the stack to collect the fines and a lid is put on the top to prevent loss of powder.

Manual and mechanical methods for sieving exist as well as dry and wet sieving systems. In the sonic shifter method, two motions are combined: 1) a vertical oscillating column of air generated by a diaphragm and 2) a repetitive mechanical pulse.

The oscillating vertical air sets the sample in periodic vertical motion, which reduces sieve blinding and breaks down aggregates, producing very little abrasion and thus particle breakage (Figure 3).

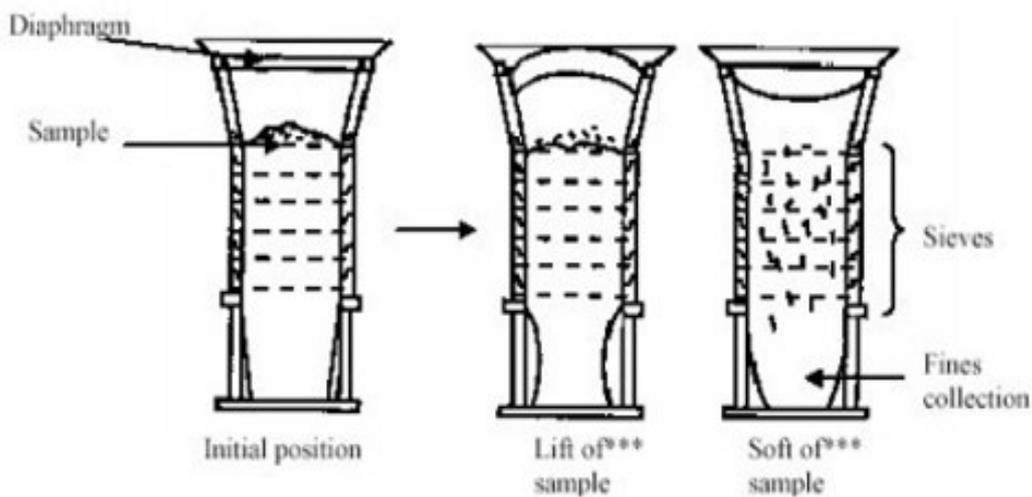


Figure 3. The sonic sifter.

In the air jet sieving system (Figure 4), air is drawn upwards through a sieve from a rotating slip so the powder is fluidized. Vacuum is applied to the bottom of the sieve removing fine particles to a collecting device. The action is very gentle, so this method is suitable for brittle and fragile powders.

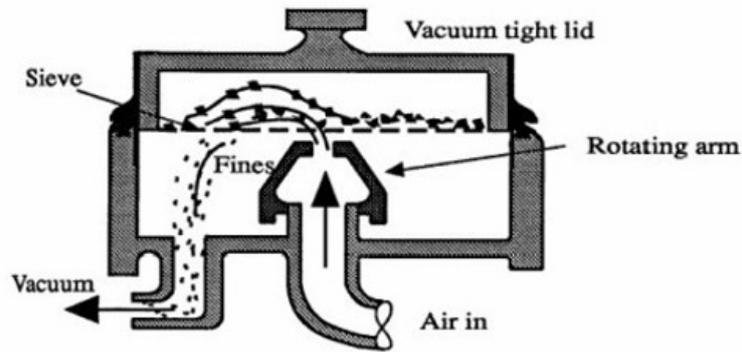


Figure 4. Air jet sieving.
(Allen, 1997)

An example of wet sieving is the felvation method: this technique uses an elutriation process, with sieves acting as stops for the powder suspension. The powder is suspended and allowed to flow in a pulsing mode through the different units. Each felvation unit contains a sieve of successive size (Figure 5).

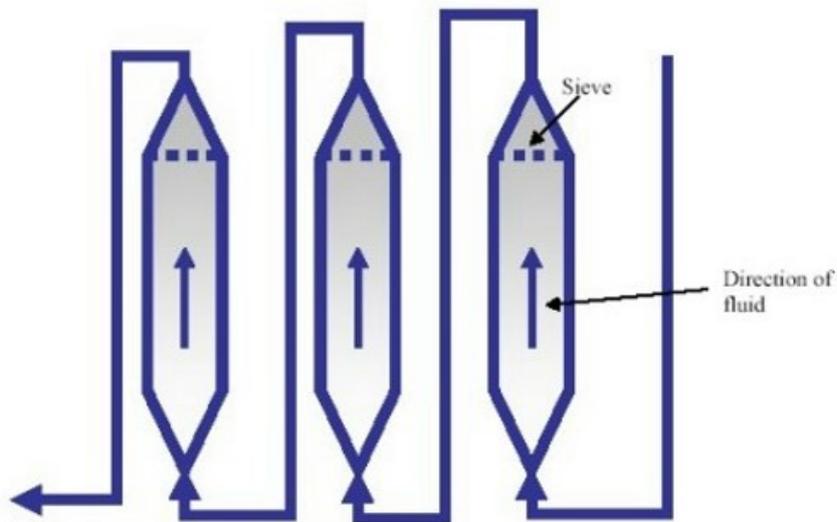


Figure 5. Felvation sieving.

Automatic systems have also been developed. An automatic sonic shifter has a system of sieves that are automatically weighed after sieving. Other principles such as ultrasound have been applied to sieving systems in order to maintain particles in constant motion during the sieving process.

Sedimentation methods are used to determine the Stokes diameter and are based on how a particle travels in a viscous fluid. The Stokes diameter of a particle is equivalent to the diameter of a sphere having the same density and settling speed as the particle under laminar flow.

The behavior of a spherical particle moving in a viscous fluid under the gravitational

force is described as follows:

$$W - B - F_D = m \cdot \frac{du}{dt} \quad (1)$$

where W is the weight of the particle, B is the buoyancy force, F_D is the drag force, m is the mass of the particle, u is the velocity, and t is the time (Figure 6). Equation (1) can also be expressed in terms of gravitational acceleration (g):

$$m \cdot g - m' \cdot g - F_D = m \cdot \frac{du}{dt} \quad (2)$$

where m' is the mass of the fluid occupied by the particle.

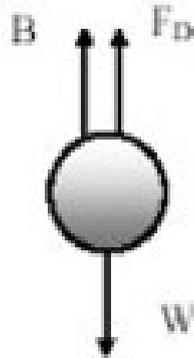


Figure 6. Forces acting on a particle falling in a viscous fluid. (B) Buoyancy, (F_D) Drag force, (W) Weight.

Small particles rapidly reach constant velocity ($\frac{du}{dt} = 0$), then $F_D = mg - m'g$.

Solving for the drag force and expressing Equation (2) in terms of the density of the particle (ρ_s) and fluid (ρ_f):

$$F_D = \frac{\pi}{6} \cdot (\rho_s - \rho_f) \cdot g \cdot D^3 \quad (3)$$

where D is the diameter of the particle.

The drag force can be expressed in terms of the drag coefficient (C_D):

$$F_D = \frac{C_D}{(\text{area})(\text{dynamic pressure})} = C_D \cdot \frac{\pi \cdot D^2}{4} \cdot \frac{\rho_f \cdot u^2}{2} \quad (4)$$

Insert F_D into Equation (3) and solving for C_D :

$$C_D = \frac{4}{3} \cdot \frac{(\rho_s - \rho_f)}{\rho_f} \cdot \frac{g \cdot D}{u^2} \quad (5)$$

At very low Re numbers (laminar flow), a linear relation between R_e and C_D is observed:

$$C_D = \frac{24}{R_e} \quad (6)$$

where

$$R_e = \frac{\rho_f \cdot u \cdot D}{\eta} \quad (7)$$

Insert Equations (6) and (7) into Equation (5), and solving for the particles Stokes diameter:

$$D = \sqrt{\frac{18 \cdot \eta \cdot u_{St}}{(\rho_s - \rho_f) \cdot g}} \quad \text{Stokes diameter} \quad (8)$$

Several assumptions are made in the derivation of this equation: particle is spherical, terminal velocity is reached, the fluid is homogeneous, and no interaction among particles exists. Careful procedure should be followed when irregularly shaped particles are measured with sedimentation methods.

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Biographical Sketches

Gustavo V. Barbosa-Cánovas received his B.S. in Mechanical Engineering at the University of Uruguay and his M.S. and Ph.D. in Food Engineering at the University of Massachusetts-Amherst. He then worked as an Assistant Professor at the University of Puerto Rico from 1985-1990, during which he was granted two National Science Foundation (NSF) awards for research productivity. Following this, he went to Washington State University (WSU) where he is now Professor of Food Engineering and Director of the Center for Thermal Processing of Food (CNPF). Dr Barbosa-Cánovas chaired the Organizing Committee for the 1997 and 1999 Conference on Food Engineering (CoFE). In addition, he is an Editor of the journal *Food Science and Technology International* published by SAGE, the journal *Innovative Food Science and Emerging Technologies* published by Elsevier Science, and the Food Engineering Theme in the Encyclopedia of Life Support Systems (EOLSS) to be published by UNESCO. Dr Barbosa-Cánovas is the Editor in Chief of the Food Engineering Book Series published by Kluwer Academic and Plenum Publishers (KAPP) as well as the Food Preservation Technology Book Series published by CRC Press. He has chaired and organized several technical sessions at the American Institute of Chemical Engineers (AIChE) and Institute of Food Technologists (ITF) annual meetings, edited 12 books on Food Engineering topics, and authored, among others, *Dehydration of Foods* (Chapman and Hall), *Nonthermal Preservation of Foods* (Marcel Dekker), *Food Engineering Laboratory Manual* (Technomic) and *Engineering Properties of Biological Materials* (ASAE). Dr Barbosa-Cánovas is also part of the editorial board for four technical journals, including the *Journal of Food Engineering*, *Journal of Food Process Engineering*, *Journal of Food Science Technology* (LWT), and the *International Journal of Physical Properties of Foods*. He is International Consultant for the United Nations' Food Agriculture Organization (FAO), Associate Researcher for the United Nations' PEDECIBA (a special program to develop basic sciences), and a consultant for several major food companies in the United States.

Hong Helen Yan comes from Chengdu, P.R. China. She completed her M.S. and Ph.D. in Food Engineering in the Department of Biological Systems Engineering at Washington State University, Pullman, Washington, and received a M.S. and B.S. in Mechanical Engineering from Chengdu University of Science and Technology, Chengdu, P.R. China. Her research interests are focused in a number of areas, related to the physical properties of food powders, including particle size characterization, attrition and segregation tendency and reduction, compaction and compression evaluation, and flowability improvement. She has published several research papers in peer-reviewed journals, and co-authored two other textbook chapters.

Federico Harte received a B.S. in Agricultural Engineering from the University of Uruguay in 1996. He is a Ph.D. candidate in the Food Engineering program at Washington State University and Initial Professor in Food Technology for the College of Agricultural Engineering at the University of Uruguay.