International Journal of Processing and Post Harvest Technology
A REVIEW
Volume 3 | Issue 2 | December, 2012 | 315-319

Recent techniques for particle size analysis of food powder

■ RANJEET SINGH

SUMMARY : Particle size information is a measure for the quality of processed food products. In this paper, an attempt has been made to review various techniques and methods used for particle size analysis of food powers. Various methods for particle size measurement *viz.*, based on separation, counting and ensemble methods are being used. Each measurement techniques produce a different answer because it is measuring a different dimension of particle. We have discussed some of the relative features, advantage and disadvantages of different methods employed for particle size analysis.

KEY **W**ORDS : Particle size analysis, Sieve analysis, Sedimentation, Electro-zone sensing, Microscopy laser diffraction

How to cite this paper : Singh, Ranjeet (2012). Recent techniques for particle size analysis of food powder. *Internat. J. Proc. & Post Harvest Technol.*, **3** (2) : 315-319.

Research chronicle : Received : 13.07.2012; **Accepted :** 20.10.2012

The importance of information on particle size analysis in food industry and allied sector has grown up sharply during the last decade. An understanding of the characteristics of masses of particle solid is necessary in designing processes and equipments for processing. Processed products-can be characterized by a range of parameters, such as composition, particle size, shape, and surface area. To optimize processing and meet the needs of a processed end product, its most significant characteristics must be specified and tightly controlled. By controlling the distribution of the desired particle attributes, produce better flow characteristics or packing density will be controled and enhance the properties of the final product. The ability to accurately analyze and control particle size will help to design the dissolution rate of a drug and the hydration rate and texture of food product. It also predicts material-handling properties such as flowability, filter blockage, and dusting tendency, and, in doing so, to better design the process equipment.

Earlier, the standard screens were used to measure the size of particles in the range between about 3 and 0.0015 inches (76 mm and 38 mm). Each screen is identified in meshes per inch. The characteristics of one common series, Tyler standard screen are based on the opening of the 200-mesh screen (McCabe *et al.*, 1993). In making an analysis, the sample is placed on the top screen and the stack shaken for 20 min. The particles retained on each screen are removed and weighed, and the mass of the individual screen increments is converted to mass fraction or mass percentages of the total sample. The approach is more appropriate for granules compared to powders. Particle size estimation of powders by sieve analysis method has an inbuilt problem of sticking sample particles on the sieve, thereby not permitting the small particle to pass through the sieve opening. However, the approach continued in the absence of better system. But these methods are not much accurate and could not deal with particle size distribution over a wide range of particle size. Although the effect of particles interacting with light was described early in last century, the idea of measuring particle size with physics principle could be realized only after reliable laser based system developed provided the source of monochromatic light and the microcomputers are powerful enough to calculate the particle size distribution. In parallel, the instrument based on laser diffraction (LD) was developed and had become the standard technique for particle size analysis in food and allied industry (Leschonski *et al.*,1984).

The first instrument by mean of laser diffraction for particle size analysis (PSA) of powders covered a measuring ranges from coarser than 1 micron unto 200 microns. In the mid-eighties a different attitude towards the particle size analysis entered the global market with capable dry dispenser

AUTHOR FOR CORRESPONDENCE

RANJEET SINGH, Agro-Produce Processing Division, Central Institute of Agricultural Engineering, Nabi Bagh, BHOPAL (M.P.)INDIA
Email : razi100@rediffmail.com

(Leschonski *et al.*, 1984). This new introduction revolutionized the particle size analysis of powders in the food industry. This technology grew the field of application. Today the particle size system options are growing. The latest particle measuring systems are completely supported and adjusted from data-base controlled software and peripherals. On line and in-line applications for particle size analysis are ready today (Rothele and Witt, 1998)

Modern particle analysis can be based on many theories and models. Fraunhofer model (Anonymous, 2000) is one of the simplest theories used. This model can predict the scattering pattern of light that is created when an opaque and solid disc of a certain known size is passed through a laser beam. The results (*i.e* particle size distribution) thus, obtained are volume based and expressed in the terms of equivalent spheres and the analyses was done on the basis of derivation of distribution parameters.

Particle sizing methods can be separated into three different classes

Separation methods :

An outside force/process is used for the separation of particles based on the size. The quantities of separated different sizes are determined.

Counting methods :

Individual particles are measured and counts of similar size particles are placed in to 'size bins' to construct a distribution.

Ensemble methods :

All particles in the samples are measured at the same time. Size distribution data is extracted from combined signal for all particles.

The details of various methods are shown in flow diagram below (Fig. 1).

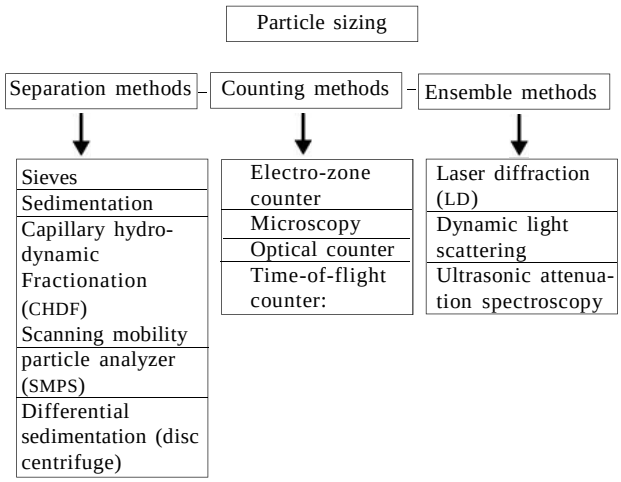

Fig. 1: Particle sizing methods

Methods of particle size measurement :

Sieves :

It is an old, but cheap and readily usable technique for large particles, such as those found in some food processing applications. It allows separation into some size bands if required. Using this technique it is not possible to measure sprays or emulsions and dry food powders below 38 mm. Cohesive and agglomerated materials, such as milk powder, are also difficult to measure and materials like food pulp particles are impossible. The longer the measurement times the smaller the answer, as particles orient themselves to fall through the sieve. A true weight distribution is not produced as the method relies on measuring the second smallest dimension of the particle. This method can also produce strange results with rod-like materials such as food fibrEs. It is a low-resolution method and usually only four to five size classes are provided. This method is cumbersome and time taking (McCabe *et al*, 1993). The details of the various particle size analysis methods are summarized in Table1.

Sedimentation :

This is the traditional method uses equipment as simple as the Andreason pipette or as complex as centrifuges and X-rays. However, as the density of the material is needed, it is no good for emulsions where the material does not settle, or for very dense material that settles too quickly. Temperatures also require close monitoring in order to control viscosity. A 1^0C change in temperature will produce a 2 per cent change in viscosity (Clarke, 2006). Other disadvantages include slowness of measurement, which makes repeat measurements tedious. Irregularly shaped particles, such as disc-shaped particle, take even longer to settle due to their increased in drag as compared with equivalent spherical particles. The technique also has a limited range, with particular difficulties below 2 mm and above 50 mm (Burgess *et al.*, 2004).

Capillary hydro-dynamic fractionation (CHDF) :

This technique is based on the Brownian motion. As the particle move down the capillary defuse across the capillary bore due to Brownian motion (Beckman *et al.*, 2005). Over time, each of the particles resides at all possible distances from the center of the capillary. Large particles reach the end of the capillary first, whereas small particles reach the end last. An optical/ultrasonic detector at the end of the capillary measures the concentration of particles as they exit the capillary in order to deduce the particle size.

Scanning mobility particle analyzer (SMPS) :

This technique is based on the principle of a charges particle in an electric field. The particle flow is injected at the outside edge of the Differential Mobility Analyzer (DMA). Particle of a given mobility exit through a sample slit at the top

Table 1: Comparative study of various particle sizing techniques

Class	Methods	Advantage	Disadvantages
Separation	Sieves	It is an old, but cheap and readily usable for large particles Separation in size bands	Time taking Agglomeration lead to error Not reliable technique. Only for large solid particles. Not suitable for particle less than 38μm
	Sedimentation	Broad dynamic range Very good resolution with small particles Performance can be easily analyzed with standards. Moderately dependent on particle geometry	Complicated algorithm for size separation History of mechanical and maintenance problems Relatively low run time Separation in rolling and sliding mode lead to error
	Capillary hydro-dynamic fractionation (CHDF)	Relatively fast analysis time Minimum of information is needed about the optical characteristics Performance can be analyzed with standards.	Aqueous emulsifier medium only Very poor resolution Capillary plugging is common problem Non spherical particle may not be correctly measured.
	Scanning mobility particle analyzer (SMPS)	Most widely method used for aerosols Onsite particle size measure	Can measure only aerosol particles usually from 2.5-1000nm size
	Differential sedimentation (Disc centrifuge)	High resolution Dynamic range up-to 1000 Size results may be corrected. Measure low density particles.	Max dynamic range is 75 when disc speed is fixed. Non spherical particles are reported as smaller than correct. Analysis time is long for small particles. Absolute weight accuracy is dependent on optical properties and size.
Counting	Electro-zone counter	Broad range of size (0.5-300μm) Simple and easy to calibrate Quick analysis time. Gives repeatable results Resolution comparable to LALLS	Dynamic size range is limited. Need conductive fluids Need electrically insulated sample Stray oversize particles lead to error below 1-2 μm particles. Lower limit resolution is poor.
	Microscopy	Evaluate range of shape and size	Unreliable results Long analysis time Representative sample critical Agglomeration of particle lead to misleading results Slow, expensive and need expertise.
	Optical counter	Broad range of size (0.5-300μm) Simple and easy to calibrate Quick analysis time. Gives repeatable results Resolution comparable to LALLS	Dynamic size range is limited. Need conductive fluids Need electrically insulated sample Stray oversize particles lead to error below 1-2 μm particles. Lower
	Time-of-flight counter	Works with dry powder Easy calibration with known standards Range 0.2 to 700 microns Fast analysis time (1 min)	Particle suspension is difficult Particle below 0.2 microns can't be measured Unknown error due to non-spherical particles
Ensemble	Laser diffraction (LD)	Simple and fast data collection. Broad range (0.01-900μm) For solid and liquid measurement Non-destructive User friendly	Low resolution power Dependent on optical parameter. Particles with different optical properties cannot be measured. Scattering problems
	Dynamic light scattering	Minimum information needed. Differently mixture of material can be analyzed. Small measurable particle size Tiny sample needed. Fast and simple analysis Non destructive	Extremely low resolution Problem of small particle in large sample
	Ultrasonic attenuation spectroscopy	Able to measure turbid suspension Technique relatively easy to implement	Extremely low resolution Needed intense data for analysis Restricted to online applications in industry

of DMA, while all other particles exit with exhaust flow (Crowder *et al.,* 2002). The size of the particle is determined by the particle concentration, charge, voltage and flow within the DMA.

Differential sedimentation (disc centrifuge) :

This technique is based on the principle of both strokes law and a light scattering. The disc centrifuge is a hollow, optically clear disc with a central opening on one side. The disc rotates with high speed ranging from 900-24,000 RPM. A dilute sample (<1% solid content) is injected into the center of the disc. The time for particle to reach the detector beam versus beam intensity is converted to a size distribution using both strokes law and Mie theory of light scattering.

Electro-zone counter :

This technique was originally developed for sizing blood cells. For industrial food materials it has many drawbacks. It is difficult to measure emulsions and impossible to measure sprays. Dry powders require suspension. Measurement must take place in an electrolyte, which creates difficulties for organic materials, and the method requires calibration standards that are expensive and change size in distilled water and electrolyte. It is slow for materials of relatively wide particle size and it is not easy to measure particles below 2 mm. Porous particles and dense materials pose additional problems.

Microscopy :

This is an excellent technique that allows direct examination of the particles in question, and one that is relatively cheap. However, it is not suitable as a quality control technique. Also, as relatively few particles are examined, there is an error of unrepresentative sampling and if weight distribution is measured results are magnified. During measurement, missing one 10 mm particle has the same effect as missing one thousand 1 mm particles. Sample preparation for electron microscopy is tedious and time consuming, and for manual methods relatively fewer particles are examined.

Optical counter :

The light counter is very much the optical equivalent of the electrozone counter. Particles are forced through a counting chamber, where a focused laser beam is partially blocked as the particle passes. The reduction in light intensity reaching a detector is related to the optical cross section of the particle, and this is converted to the particle size (Haskell, 1998).

Time-of-flight counter :

The technique is based on the principle that particles accelerate in the air flow according to size. Smaller particle accelerate more rapidly than larger particles. This technique is targeted for dry powders. An air stream is containing particles is drawn through a fine nozzle into a partial vacuum, producing a supersonic shock of air (Shekunov, 2004). The accelerated particles then pass two focused laser beams. The first laser beam detects each particle and starts a time of flight clock, while arrival at the second laser beam stops the clock.

Laser diffraction (LD) :

Laser diffraction is becoming the preferred standard in many food industries for quality control. It offers a wide dynamic range and is very flexible. For example, it is possible to measure the output for a spray nozzle to get correct droplet size of milk powder, something that has led to its wide application in the food industries. Dry powders can be measured directly and liquid suspensions and emulsions can be measured in a re-circulating cell (Singh, 2006). This gives high reproducibility for the determination of primary particle size. LD in non-destructive and non-intrusive and a volume distribution are generated which is equal to the weight distribution where density is constant, making it of direct relevance to food application. Other benefits are rapidity, time saving; repeatability for reliable results and high resolution (Shekunov *et al.,* 2003). There is no need to calibrate against a standard, but equipment performance can be easily verified.

Dynamic light scattering :

In dynamic light scattering, also known as quasi-elastic light scattering, the Brownian motion of sub-micron particles is measured as a function of time (Garmise, 2006). A laser beam is scattered by particle in suspension. The diffusion of particles causes rapid fluctuations in scattering intensity around a mean value of certain angle (10-150 degree).

Ultrasonic attenuation spectroscopy :

This technique is based on the principle that plane sound waves moving through a particle suspension are attenuated in a predictable manner according to the size and concentration of the particle in the suspension, the spacing of the transmitter and receiver and other physical parameters (Swaminathan and Kildsig, 2002). Attenuation of an ultrasonic wave passing through a suspension may be modeled given a set of mechanical, thermo-dynamical and transport properties describing both the continuous and particulate media. The relationship between spectral data and particle size is illustrated by considering attenuation curves (Shiraishi *et al.,* 1995).

Conclusion :

Particle size analysis of food powders assumes greater importance as it is directly linked with processed product quality. Numbers of methods are used for particle analysis and sizing of food powders like sieve analysis, sedimentation, microscopy and electro zone sensing etc. but, these methods are not much accurate and could not deal with particle size

distribution over a wider range of particle size. This problem is accurately dealt with particle size analyzer based on laser diffraction. Laser diffraction (LD) is becoming the preferred standard in food and allied industries for characterization and quality control. It offers a wide dynamic range and is very flexible. Ease in operation and better results output are the advantages, which makes it ahead of other particle size analysis techniques. Finally, it is clear that as more particle sizing methods become validated and accepted by the food industry, they will be included in food regulatory guidelines, thus enabling more consistent application of these techniques in food and allied industry.

LITERATURE CITED

Anonymous (2000). Sympatee PM-Tour 2000 documentation, Sympatee GmbH, System-Partikal-Technik, Clausthal, GERMANY.

Beckman, A., Shan, D., Ali, A., Dai, W., Ward-Smith, S. and Goldenberg, M. (2005). Micrometer-scale particle sizing by laser diffraction: critical impact of the imaginary component of refractive index. *Pharm. Res.*, **22**:518-522

Burgess, D.J., Duffy, E., Etzler, F. and Hickey, A.J. (2004). Particle size analysis: AAPS workshop report, cosponsored by the Food and Drug Administration and the United States Pharmacopoeia. *AAPS J.*, **6**:1-12.

Clarke, J. (2006). Inspired by design: evaluating novel particle production techniques. Proceedings of the Conference on Respiratory Drug Delivery, Boca Raton, FL, **10**:287-295.

Crowder, T.M., Rosati, J.A., Schroeter, J.D., Hichey, A.J. and Martonen, T.B. (2002). Fundamental effects of particle morphology on lung delivery: predictions of Stokes law and the particular relevance to dry powder inhaler formulation and development. *Pharm. Res.*, **19**:239-245.

Garmise, R.J., Mar, K., Crowder, T.M., Hwang, C.R., Ferriter, M., Huang, J., Mikszta, J. A.,. Sullivan, V. J and Hickey, A.J. (2006). Formulation of a dry powder influenza vaccine for nasal delivery. *AAPS Harm Sci.Tech.*, **7**:E1-E7.

Haskell, R.J. (1998). Characterization of submicron systems via optical methods. *J. Pharm. Sci.* **87**:125-128.

Heuer, M. and Leschoneski, K. (1985). Results obtained from a new instrument for the measurement of particle size distribution patterns. *Part. Charact*, **4**: 7-13

Leschonski, K., Rothele, S. and Menzel, U. (1984). A special feeder for diffraction pattern analysis of dry powder. *Part. Charact*., **1**: 161-166

McCabe, W. L., Smith, J.C. and Harriott, P. (1993). *Unit operations of chemical engineering*. (5th Ed.) McGraw-Hill International publication, SINGAPORE.

Rothele, S. and Witt, W. (1998). In-line laser diffraction with innovation sampling; Proc.7. Europ. Symposium *Part. Charact*. pp.611-624.

Shekunov, B.Y., Feeley, J.C., Chow, L., Tong, H.H.Y. and York, P. (2003). Aerosolisation behaviour of micronised and supercritically-processed powders. *J. Aerosol Sci.*, **34**:553-568.

Shekunov, B.Y. (2004). Production of powders for respiratory drug delivery. In: P. York, U. B. Kompella, and B. Y. Shekunov (eds.), Supercritical fluid technology for drug product development. New York: Marcel Decker. *Drugs Pharm. Sci.*, **138**:247-282.

Shiraishi, T., Sano, A., Kondo, S., Yuasa, H. and Kanaya, Y. (1995). Studies on the granulation process of granules for tableting with a high speed mixer. II. Influence of particle size of active substance on granulation. *Chem. Pharm. Bull.*, **43**: 654-659.

Singh, R. (2006). Particle size analysis of tomato powder, *J. Food Process. & Preser.*, **30**: 87-98.

Swaminathan, V. and Kildsig, D.O. (2002). Polydisperse powder mixtures: effect of particle size and shape on mixture stability. *Drug Dev. Ind. Pharm.*, **28**:41-48.