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PARTICLE SIZE MEASUREMENT TECHNIQUES: A REVIEW OF METHODS AND APPLICATIONS

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Abstract. A number of particle sizing techniques are available to measure solid particles median size of nano-scale to centimeter. The techniques cover a wide range of applications in industries where the particle size information is an important parameter in manufacturing process. The particle size measurement (in terms of application) can be categorized into two groups, offline application and on-line measurement. On-line particle size measurement in pneumatic transporter is challenging area in terms of measurement condition, where particles flow homogeneity, humidity, velocity and concentration profile are directly affects the measurement results. All these parameters make difficult the instrumentation design. This paper highlights some of the important instrumentation in particle sizing based on their measurement principle. The measurement methods include physical methods, laser scattering based methods, Image based methods and electrical sensing zone method. In addition, the paper highlights recent innovative on-line measurement techniques used in particle size measurement.

Keyword: Sedimentation; sieving; laser scattering; phase-Doppler; morphology; coulter count; resonance frequency; optical tomography; electrostatic sensor

1.0 INTRODUCTION

Particles size is an important parameter in many industries such as coal fired power plants, pharmaceutical, chemical and food stuff production process. Measurement and control of size parameter effectively improves productivity, product quality and process efficiency in such industries. For example, one of the important parameter in efficiency and quality control of combustion in coal fired power station furnaces is size of pulverized coal particles. Due to that reason,

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development of a reliable, cost effective and high accuracy measurement system is a great interest to both the researchers and engineers.

Particle sizing methods cover a wide range of instrumentation techniques, among them image and light scattering methods in terms of real applications in laboratories are dominant techniques [1]. But these techniques have limitation for industrial in-line application due to harsh and dusty nature of such industries; dust contaminates the probes window and increases the output reading error. Also image and light scattering methods have sensitivity limitation to depth of particle flow. To on-line particle size measurement, in recent years other innovative methods have been developed including resonant measurement based particle sizing [2], CCD Imaging tomography [3], particle sizing using electrical charge analysis [4], analysis of acoustic emission signal [5] and Image based particle sizing [6].

A favorable particle size measurement should be without any obstructive effects on particles flow on the conveyor, also the harsh condition of industrial environment does not affect the quality of measurement and measuring devices. Cost effective instrumentation with reasonable accuracy are other important parameter to choose a correct size measuring device. Installing a proper measurement system depend on factors such as nature of particles, mass flow rate, material type, particles size, velocity, moisture and homogeneity profile of particles flow.

2.0 PHYSICAL PARTICLE SIZING METHODS

Physical particle sizing methods are categorized into two main methods, sedimentation and sieving techniques [7]. Sedimentation of particle in a fluid is one of the oldest methods to extract particles size distribution. Stoke's law is the basic rule in this measurement system that used to determine particle size distribution by measuring the required time for particles to transfer a known distance in a liquid with known viscosity and density. Sedimentation methods can be either in gravitational sedimentation method [8] or centrifugal sedimentation [9], Figure 1 shows a commercial version of this technique. Gravitational sedimentation is limited for particles with small size because particles analysis time for small particles is too long, for example particles less than 0.1 micron will never settle in gravitational sedimentation method. Centrifugal method covers a wider range of particles size with smaller settling time. Stoke's law for centrifugal sedimentation can be given as:

$$D = \{(18\eta \ln(R_f / R_0)) / ((\rho_p - \rho_f) / \omega^2 t)\}^{0.5} \quad (1)$$

where D is the particles diameter (cm), η is the fluid viscosity (poise), R_f is the final radius of the rotation(cm), R_0 is the initial radius of rotation (cm), ρ_p is particle density (g/ml), ρ_f is the fluid density (g/ml), ω is the rotational velocity (radians/sec), t is the time required to sediment from R_f to R_0 (sec). The normal range for sedimentation method particle sizing is between 0.01 and 50 micron [10, 11]. The centrifugal apparatus design improvement affects the speed, classification performance and output results [12, 13]. Sedimentation technique could by applied in the area of ceramics, metal powders, soil science, cosmetics, pigments, catalysts, construction materials and abrasives industries.

Figure 1 SediGraph III 5120, a commercial version of particle size measurement. Measures particles range from 0.10 to 300 μm equivalent spherical diameter using stokes law [14]

Sieving is other major technique in physical particle sizing methods. Sieving method is the simplest possible method that uses sieve aperture to sort particles into categories solely on the basis of size, independently from other factors such as particles density, surface, etc [15]. Two common methods in sieving are stacked method which uses a sieve with decreasing the mesh size, and reverse method that uses multiple sieving steps with each step a single sieve. Sieving method can sort particle >40 micron [16]. Wet sieving applying the pressure on fluid materials is another sieving method which is able to sort the particle <40 micron [17].

3.0 LASER BASED PARTICLES SIZING METHODS

Figure 2 shows the schematic and perspective of laser scattering based particle sizing arrangement. From the picture, in physical principles a laser beam after passing a beam expander is scattered by particles in Sample Flow Cell. Scattered light passes through a Fourier transform lens and shape a diffraction pattern of concentric rings at the detector plane [18]. Figure 3, is the commercial version of laser diffraction method with components description. The laser based instruments applicable in wide range of particles measurement include cement, polymers, toners, powder metal, mineral production, pharmaceuticals and food stuff. Techniques for analysis of scattered light can be categorized in two groups, amplitude dependent techniques and amplitude independents techniques.

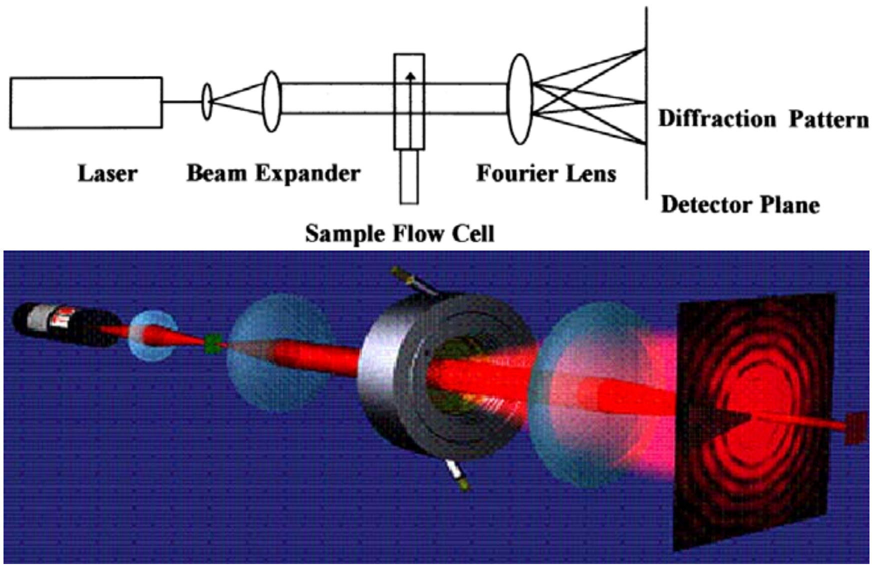

Figure 2 Basic optical arrangement for size analysis [18]

Amplitude dependent techniques are based on measuring the intensity of scattered light, and amplitude independent techniques considered other features of scattered light such as phase difference of scattered light at different spatial location [19]. Absolute intensity and Dual beam sizing techniques are among the amplitude dependent methods, while Phase-Doppler analysis, Project grids, and Visibility are amplitude independent methods [1].

Absolute intensity ratio operates by measuring absolute magnitude of scattered light. The basic idea of this method is the large particle causes the power of scattered light to be decreased and vice versa. This technique is suitable for particles ranging between 0.5 and 1000 micron with the accuracy of 5-15% [20, 21].

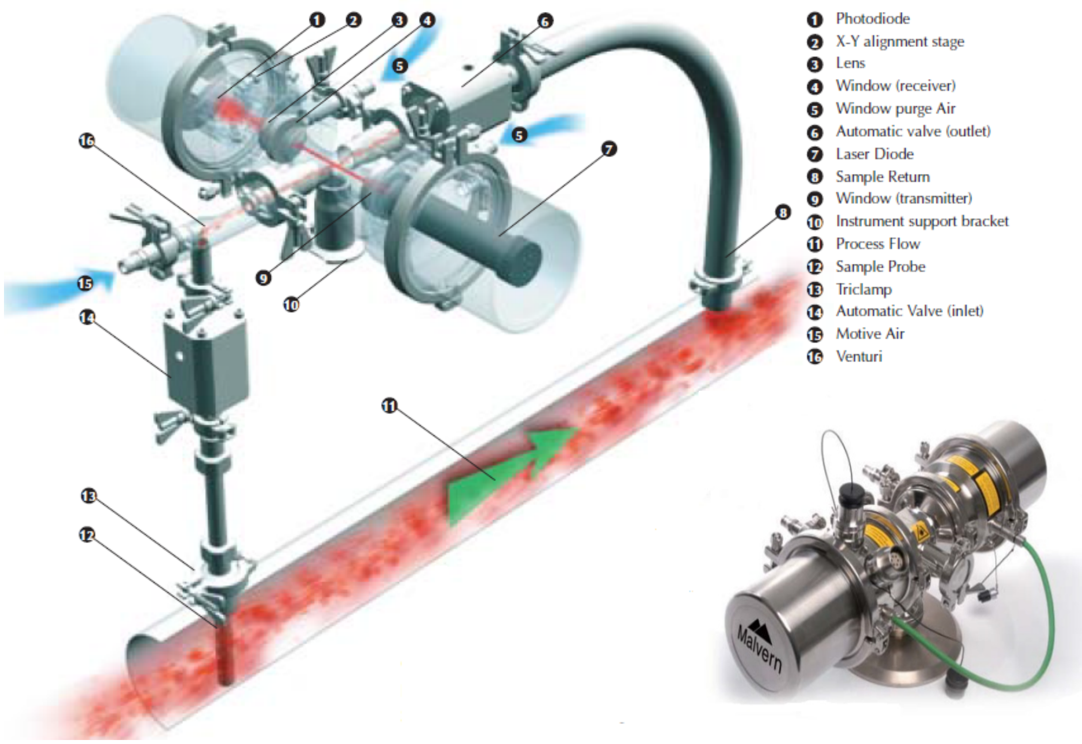

Figure 3 Insitec X a production by Malvern Company, able to online measurement of particles in range of 0.1 to 2500µm using light scattering method [22]

Dual beam sizing system uses two beams with different wavelength; one beam has larger diameter than other one. The beams are illuminated in way that the smaller diameter beam surrounded by larger diameter beam in a coaxially form. The intensity of larger diameter beam is used for particle size analysis and center beam is used as trigger for measuring system to insure the particle positioned in the center axis [23]. The size range and accuracy of this method are the same as the absolute intensity.

Phase-Doppler technique measures the phase difference between incident beam and received scattered beam; and it is widely applied in particle size and velocity measurement [24, 25]. This method has the highest accuracy among the laser base method and which is better than 5% and the measuring range for this method is between 0.5 and 10000 micron.

Project grid technique in light scattering method utilizes a grating in front of receiving detector in a way that any grating correspond a unique particle size. When the particle passes the gratings, the particle size is correspond to the grating that no longer shows a plateau of intensity [1]. Shadow Doppler technique is the newer version of project grid technique where the shadow of the particle is imaged with a photo diode array arranged in detector plane, and size information is extracted by analysis of temporal signal on diode array. This technique can be used for both velocity and particle size measurement [26, 27]. Project grid technique has low accuracy, which is more than 15% and its particle size measurement is 0.5 to 1000 micron.

Visibility technique uses the maximum and minimum scattered light intensity from a particle in the fringe pattern. The fringe pattern is produced by crossed laser beam in sample particle [28]. The frequency of scattered light has direct relation with particle velocity. The Visibility concept V can be defined as follow [1]:

$$V = \frac{(I_{\max} - \mathrm{I}_{\min})}{(I_{\max} + I_{\min})} \tag{2}$$

The visibility V has direct relation with the particle size [1]. This method can support particle sizing in the range of 0.5-1000 micron with accuracy of 5-15%.

4.0 IMAGE BASED PARTICLES SIZING METHODS

To measure particle size using image based measurement techniques, photography equipment and image processing algorithms are applied to find estimated diameter of particle. As it depicted in Figure 4, in this method a 2D image is captured from a 3D particle and size analysis is calculated by comparing the 2D image with the equaling circle diameter to get the approximate size of the particle. Morphology G3, as it depicted in Figure 5 is a commercial version of image based particle sizing. In terms of application, image based particle size measurement has the highest precision and can offer exact shape analysis. But the method is expensive and takes a long analysis time.

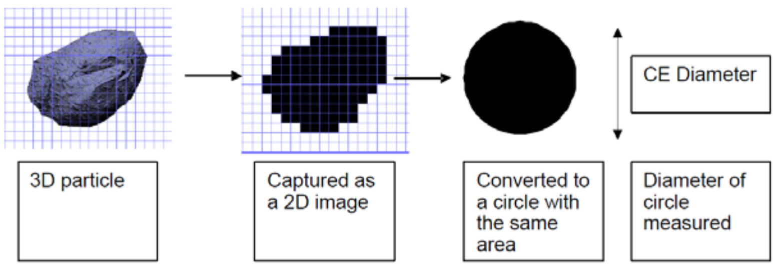

Figure 4 Image based particle sizing procedure

Rinaldi and Rossi developed an atomic analysis technique to classify and morphology carbides under 250nm [29] , Francus used image analysis method to measure grain-size variation in thin section of soft clastic sedimentation with dimension of 7-50 micron [30], Shen et al [31] applied image particle sizing method to measure size and velocity of falling particles. They used an image processing algorithm to measure the particle size and a velocimetry tacking algorithm based on fuzzy logic to measure the velocity [31]. Digital imaging using CCD camera is an innovative method in imaging area that used by Carter *et al.* [32] to measure particle size in pneumatic conveyors in the range of 100 to 2000 micron. In this method they applied a thin laser sheet to visualize the depth particles flow, and the results are compared to laser diffraction in-line instrumentation. The image based size measurement system is found to have a better repeatability than laser diffraction method [33, 34].

Figure 5 Morphology G3 a production by Malvern Company, based on Image analysis method [35]

5.0 ESZ METHODS

Coulter count or electrical sensing zone method is introduced by Coulter in 1950s. According to basic principle of this method, particles are dispersed in electrolyte solution. When a particle passes through the electrical sensing zone aperture, it displaces electrolyte according to its value, causing an impedance change between the electrodes. The basic setup of ESZ and its commercial version are shown in Figure 6.

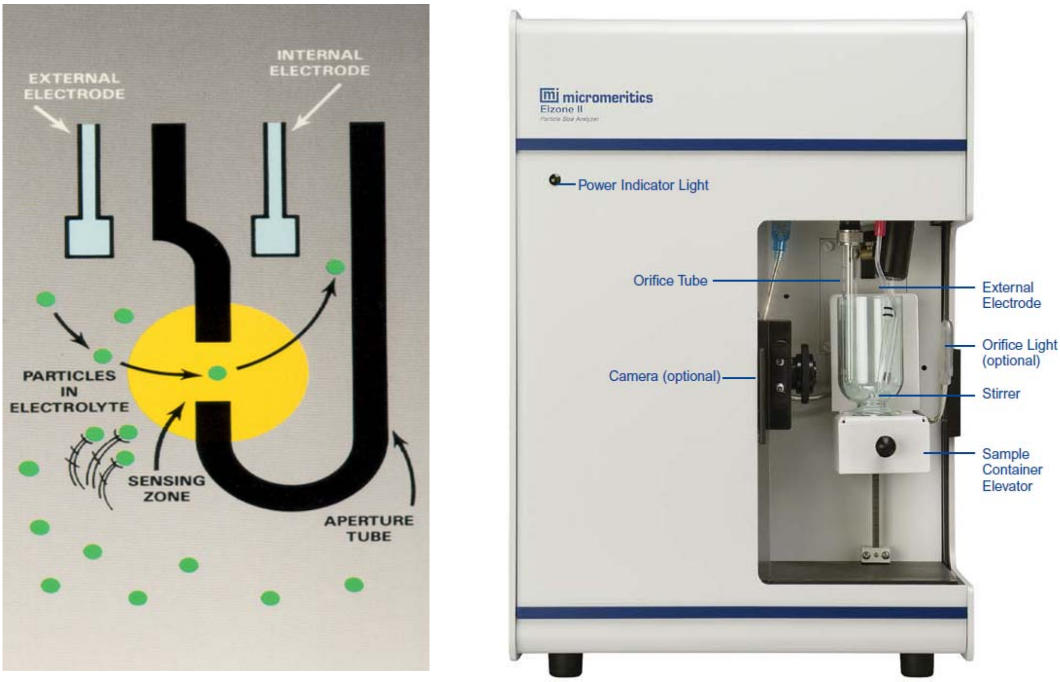

Figure 6 Electric sensing zone basic set up and its commercial version, Elzone II 5390 by Micromeritics Company [36]

The intensity of this change is proportional to the particles size [37]. Design of Coulter count apparatus depend on particles type and diameter, a micro machine Coulter instrument can measures particles <40 micron [38]. Also Sowebry et al. used Nano technology where he designed a Nano-scale aperture for sensing zone [39]. ESZ instrument cover a wide range of application such as in biotech research to measurement the size of platelets, blood cells, bacteria, plant cells, yeast, mammalian, pollen, spores, in toner and inks, emulsions and abrasives.

6.0 OTHER METHOD

Analysis of resonant frequency is another way in particle size measurement. In this method, a structure such as a rod is installed inside the flow pipe. Colliding particles to the rod causes it start to vibration in its natural frequency. Different size particles produce different resonant frequency, analysis of peak frequency could extract particle size information [2].

Optical tomography based on CCD linear image sensor particle sizing is proposed by Idroas. In this method, four CCD sensors are applied to detect density of diffracted light which illuminated from a light source to particle. By an image reconstruction algorithm, particle size could be measured [3].

Analysis of acoustic emission signal generated by particle's impact to an obstacle is an innovative size measurement method for particle size between 1 and 10 mm in a flow conveyor. In this method, particles impact to an obstacle produces pressure wave which spread in the air as acoustic signal and captured by a sensitive microphone, and spread in obstacle body as vibration and capture by piezoelectric sensor. Bigger particles size impact increased the vibration time and frequency [5].

Using electrostatic sensor to measure the particle size is presented by Zhang and Yan. In this method, natural electrostatic charge on particles flow is detected by an electrostatic sensor. The magnitude of detected charge from particles flow is proportional to particles median size. This method can support particle size measurement between 100 and 1000 micron with the accuracy of 15% [4].

7.0 DISCUSSION AND CONCLUSION

The described methods in this review can be divided into two: methods mostly utilized for academic purpose and real applied methods in manufacturing industrial application. In addition to fulfill customer requirements, instrumentation manufacturers mostly look for measuring methods, techniques and algorithms that depress the cost price and improve the reliability, accuracy and quality of the instrument. For example, size measurement using tomography system, electrostatic sensor and the techniques in laser scattering method, except for Phase-Doppler techniques and intensity measurement are not under instrument's manufacturer interests because they are not economy. In the case of electrostatic sensor, it is the cheapest available method but it does not have a good accuracy. Table 1 includes a summary of discussed methods in particle sizing.

Table 1 Summary of particle sizing methods

Method	Physical		Laser based					Image based	Electrical Sensing Zone	Others			
Technique	Sedimentation	Sieving	Absolute Intensity	Dual Beam	Phase-Doppler	Project grids	Visibility	Photography	Coulter Count	Resonant Frequency Analysis	Optical Tomography	Acoustic Analysis	Electrical Charge detection
Measurement Range (µm)	0.01-300	>40	0.5-1000	0.5-1000	0.5-10000	0.5-1000	0.5-1000	<0.25	<40	30-150	14	1000-10000	100-1000
Relative Error (%)	NA	NA	5-15	5-15	<5	>15	5-15	<5	NA	NA	NA	NA	15
In-Process	No	No	Yes	No	Some	No	No	Some	No	Yes	No	Yes	Yes
Lab	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	No	No

Selecting a suitable measurement system for an application depends on the nature of sample, application environment, on-line measurement or laboratory application and to its price. For instance, in size measurement of biotech material, the ESZ instrument is quite suitable. If both size measurement and shape analysis are required, then using the expensive microscopy image tool is unavoidable. Most of the commercial particle size instruments are designed for industrial laboratory application that required sample from production line and analyzed in laboratory. In some cases, such as coal-fired furnaces or metallic powder, industrial on-line size measurement in pneumatic transporter is necessary. Harsh environment, dusty nature, unsteady velocity and inhomogeneous particles mass profile of pneumatic transporter made the on-line measurement a challenging research area. The commercial versions of size measurement instrument mostly employed laser scattering methods. Laser based techniques have a limited sensing range to depth of flow, susceptible for window contamination in dusty nature environment that decreases the accuracy and it works well only with spherical particles. However other proposed methods for on-line size measurement have low accuracy problem such as electrostatic sensing method or they are obstructive such as acoustic and vibration analysis based size measurement. Therefore, instrument manufacturers still are working on improving the laser scattering based method and its apparatus design.

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