WHITEPAPER

The Definitive Guide to Particle Sizing

Understanding what makes a particle sizing technique well-suited to a specific application

The need for particle size information is common to many industries, but the technique used to acquire it is not. This is no accident. For many decades, engineers have developed and refined technology to size particles – both solid and liquid – and there is now a broad selection to choose from, with different solutions uniquely well-suited to different applications. Alternative sizing techniques produce numerically dissimilar values, making it possible to generate any number of particle sizes for a given sample.

There are often specific reasons why a particle sizing technique enjoys popularity within a certain industry, or for a given application, that stem directly from its measurement principles and the features that these confer. From all the powerful and highly effective technology available, one or two solutions typically provide the most valuable insight, with certain technologies particularly complementary to one another. Choosing the most appropriate technique(s) makes it possible to robustly differentiate samples from one another with sensitivity and relevance, in any given situation.

This paper provides an introduction to particle sizing comparing six widely used techniques, explaining how they work and what this means in terms of strengths and limitations. The aim is to provide the reader with the understanding needed to make informed particle sizing choices, well-matched to their requirements.

What do you want to know?

The foundation of a sound choice of analytical instrumentation is a clear understanding of why the information is required. A good starting point for an assessment of particle sizing technology is therefore to consider what questions will application of the resulting data ideally answer. For example, is the requirement to:

- Rationalize bulk powder behavior in the process or to control performance attributes associated with individual particles such as solubility, reactivity, or bioavailability?
- Monitor and control a specific process – grinding/milling, precipitation, cleaning or recovery?
- Sensitively detect any change, rather than check the sample against a single number specification?
- Measure particle size in combination with other relevant variables such as particle concentration?

A more detailed analysis of these questions begins the process of understanding more about how particle sizing techniques vary in terms of their suitability for different applications.

In a bulk powder or suspension particles may be aggregated or agglomerated rather than present as individually discrete entities. The extent to which this occurs depends on the relative strength of inter-particular forces, which is influenced by particle size (along with other properties of the system) with finer particles more prone to agglomeration. When it comes to rationalizing in-process behavior measuring representative particle size data therefore often means measuring agglomerated particle size. On the other hand, if the goal is to investigate a property such as solubility then primary particle size – the size of individual dispersed particles - is a more relevant predictor of performance, particularly if any agglomerates disperse easily in solution. The particle sizing method applied needs to reflect this, typically via a preparatory dispersion step.

When it comes to comminution processes – grinding and milling – oversized particles indicate that processing is incomplete, while excessive fines, which may compromise downstream operations, are suggestive of over-processing and the associated inefficiencies of sub-optimal throughput and excessive energy consumption. This illustrates the potential importance of accurate sizing across the complete particle size distribution of the sample. For some applications it may be sufficient simply to determine that most of the sample is clustered around a certain particle size but for others the tails – coarse and fine – are equally critical. Fines may be particularly hazardous from the perspective of health and safety, presenting an inhalation risk, while just a few coarse particles can be highly problematic in an abrasive or pharmaceutical. Certain particle sizing techniques are particularly good for fines detection, some for the detection of coarse particles, while others offer high resolution across the size distribution.

In some instances particle sizing techniques are well-matched to specific processes because they are based on the same underlying scientific principles, a classic example being the use of sedimentation techniques to elucidate the performance of precipitation processes. Here process performance is dependent upon how the particle settles and sedimentation reports size on the basis of this behavior. A technique may also be prized for the additional information that comes along with particle size. For instance, the accurate measurement of particle count or concentration can be useful for cleaning or recovery processes, while the provision of shape data may be an important gain for materials where it has a critical impact on performance, such as metal powders for additive manufacturing and toners.

A family of complementary techniques

Armed with this understanding of some of the key ways in which particle sizing techniques vary we can assess the alternatives, looking at how they determine particle size and what this means in terms of the data generated. This section begins with dynamic image analysis – an intuitive, visual technique analogous to microscopy – before looking at four other techniques also applied to suspensions/samples dispersed in a liquid carrier, including two forms of light scattering: static and dynamic. It concludes with air permeability a traditional technique for dry particle sizing. While dynamic image analysis involves the visualization of particles the remaining five techniques all determine particle size by measuring a property related to it, generating values of particle diameter on the basis of spherical equivalence within the limitations and constraints of the technique.

When comparing these techniques it is vital to recognize that industrial samples are extraordinarily diverse with few, if any, consisting of perfectly spherical particles of homogeneous properties. Indeed, a common requirement is for particle size information for a blend of different materials. These are important practicalities to bear in mind when it comes to evaluating the implications associated with the assumptions underpinning each technique.

Dynamic image analysis (Particle Insight)

Basis of measurement: The determination of defined dimensions from individual particle images

Dynamic image analysis, as the name suggests is a visual technique, based on the capture of particle images (see figure 1). These images are silhouettes produced by transmitting light through a sample of suspended particles flowing through a thin flow cell. They are recorded by a high-resolution camera at a rate of thousands of images per second, making it possible to generate statistically significant particle size data in just a few minutes.

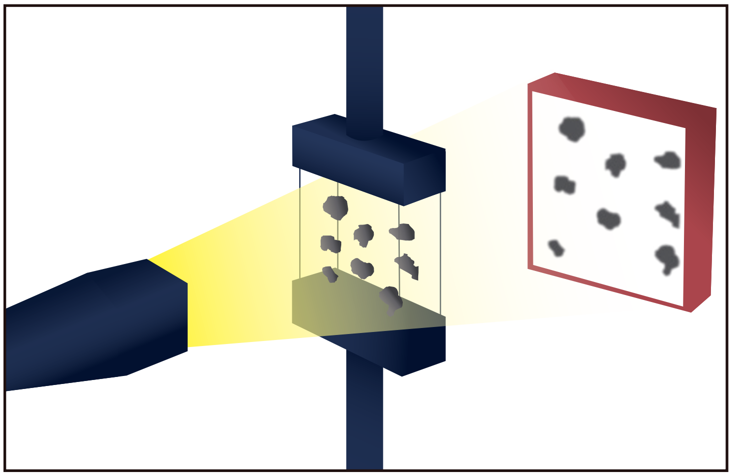


Figure 1: Dynamic image analysis captures 2D images of individual particles

With dynamic image analysis we are directly observing particles and measuring linear dimensions, without making any assumptions. However, there are a significant number of dimensions that can be measured, to produce multiple descriptors of particle size (see figure 2).

Figure 2: Martin's (top), Feret (middle), and Heywood (bottom) diameter are all equally valid descriptors of particle size.

Where:

Martin's diameter is the length of the chord that bisects the particle into two equal areas

Feret diameter is the distance between parallel lines tangential to the outline of the particle

Heywood diameter is the diameter of a circle with the same area as that of the projection of the particle.

Though equally valid, these diameters are numerically different - unless the sample is a population of perfect spheres - and it is easy to appreciate that they differ in terms of utility for a given application. Most obviously, if particles are passing through an orifice or restriction then the Feret diameter potentially has greatest significance, since it is providing insight on outer particle dimensions. Beyond these three diameters there are many other descriptors of size that can be determined from a 2D image, depending on the shape to which the particle is approximated, the particle shape model that is selected (see figure 3/Table 1).

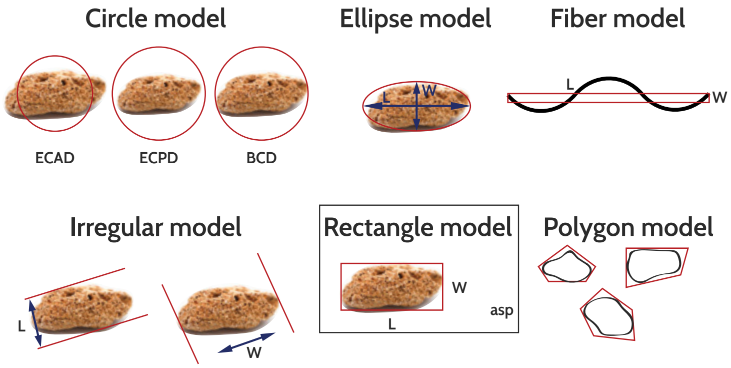


Figure 3: An array of particle size descriptors can be determined by dynamic image analysis, depending on the particle shape model selected.

Model	Descriptors Reported
Circle	Heywood diameter, equivalent perimeter diameter, bounding circle diameter, circularity, form factor, compactness.
Ellipse	Equivalent area diameter, bounding ellipse diameter, ellipsicity.
Rectangle	Bounding rectangle length, width, aspect ratio, rectangularity.
Polygon	Polygon order, convexity.
Fiber	Length, width, aspect ratio, curl.
Irregular	Feret length, width, aspect ratio, mean radius, smoothness.

Table 1: The size and shape parameters reported by dynamic image analysis depend on the selected particle shape model.

Looking at the images generated highlights the feasibility of determining metrics to describe particle shape, as well as size. This is a defining attraction of dynamic image analysis with commonly applied shape parameters including circularity, form factor, compactness, convexity, fiber length, width and aspect ratio, and smoothness (see table 1). With dynamic image analysis the user can select the particle shape model that is most appropriate to the sample and report particle size metrics on the basis of it. However, even a cursory examination of the particle images highlights the critical point that these are 2D images of 3D shapes; particle orientation is an important factor.

Depending on the design of dynamic image analyzer, particles may be circulating in a closed loop in which case they will be imaged multiple times in random orientations. This is a notable difference between dynamic imaging and static imaging methods/microscopy and is particularly important if you have flat, flake-like particles and the parameter of interest is, for example, flake thickness. Filtering the images to develop size distributions based solely on images that capture the dimension of interest is one way to address this issue and improve the relevance of the resulting data.

This brings us directly to the three primary limitations/ constraints associated with the technique which are that:

- The reported particle size distribution depends on the impact of particle orientation and the particle shape model applied
- Image clarity is a limited by the magnification power of the lens and the depth of the sample cell
- Concentration must be low enough to ensure that particles do not overlap during image capture.

These limitations are addressed to varying degrees by the design of individual instrumentation which determines, for example, the available magnification power, and through method development which determines the most suitable measurement concentration.

Why choose dynamic image analysis?

The capability to quantify shape, in addition to particle size, and to visually scrutinize a particle population is powerful motivation for the uptake of dynamic image analysis. Particle size and shape data, in combination, are especially useful when it comes to investigating characteristics such as powder flow properties, abrasion performance, particle packing, settling velocity, dispersion/homogeneity, compaction and contamination. Imaging data can also be extremely helpful when it comes to troubleshooting an alternative particle sizing method that is influenced by shape, making dynamic image analysis highly complementary to, for example, laser diffraction.

Dynamic image analysis is unaffected by particle properties so can be successfully applied to multicomponent blends and since the technique is light-based it can differentiate and quantify particle opacity. Both size and shape distributions are number-based making the technique extremely sensitive to small changes in the amount of very fine or coarse particles present.

X-ray sedimentation (SediGraph® III Plus)

Basis of measurement: Spherical diameter equivalence on the basis of sedimentation/ settling velocity (Stokes Diameter)

In the case of X-ray sedimentation, particle size is determined from measurements of terminal settling velocity with Stokes Law (see equation 1) describing the relationship between the two parameters.

$$V_t = \frac{f_a d^2}{18\eta} (\rho - \rho_L)$$

Equation 1: Stokes law relates particle diameter (d) to terminal settling velocity (V_t). [Where f_a is the accelerating force e.g. gravity, centrifugal, η is the viscosity of the suspension medium, ρ is particle density and ρ_L is liquid density.]

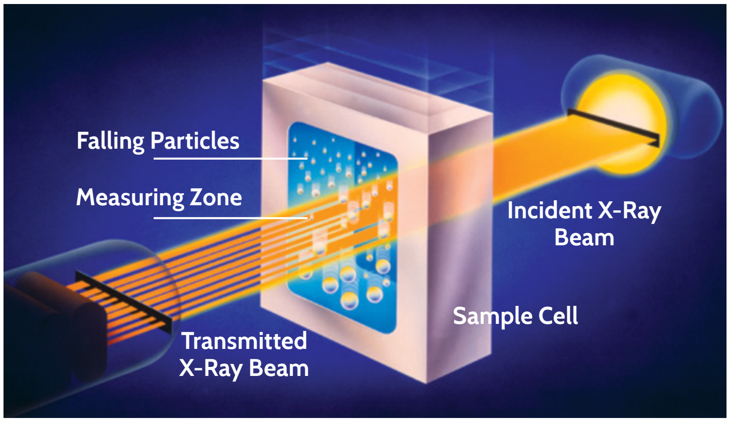


Figure 4: In X-ray sedimentation settling velocity is measured via photon absorption to determine particle size

Figure 4 illustrates how settling velocity is measured in an X-ray sedimentation analysis. A dispersed sample is pumped through the sample cell to allow measurement of the attenuated X-ray beam, the absorption associated with the dispersed sample. Agitation is then ceased allowing the suspended particles to settle. Monitoring X-ray intensity throughout the settling process detects progressively finer particles falling through the measurement cell, allowing the determination of settling velocities for the particle population, from measurements of time and distance. These velocities are converted into particle size data by applying Stokes law, with the relative mass concentration in each size class determined from the photon absorption data using the Beer-Lambert-Bouguer law. This law relates the extent of X-ray attenuation to the properties of the sample, the mass concentration of particles within in.

To understand the limitations of X-ray sedimentation we need to examine the assumptions that underpin Stokes Law. These are that:

- Particles are spherical
- There is no interaction between particles and/or between particles and the walls of the container (sample cell) i.e. the particles exhibit independent, unhindered settling
- Particles all have the same density (and greater density than the dispersant)
- Fluid flow around the particles is laminar.

For non-spherical particles X-ray sedimentation reports the diameter of a sphere of the same material that has the same settling velocity as the particle. This is referred to as the **Stokes Diameter** or equivalent spherical diameter by sedimentation.

The shape of a particle impacts the drag force acting on it, irregularity increasing drag and reducing settling velocity, thereby causing a shift towards a smaller reported equivalent size distribution. With respect to density, Stokes Law shows clearly that less dense particles will settle more slowly, if all other system variables remain the same. Lower density particles will therefore be reported as having a smaller particle size distribution than equally sized particles of greater density.

Why choose X-ray sedimentation?

X-ray sedimentation is underpinned by two well-understood physical phenomena – sedimentation and photon absorption – both of which are described by relatively simple laws. This makes data interpretation fast and straightforward, with no reliance on optical properties or extensive modelling for size determination; the only additional data requirements are for density (particle and fluid) and viscosity (fluid).

The technique is well-established and covers an industrially useful dynamic range, of 0.1 to 300µm, with Stokes law universally applicable. In addition, this is one of the few techniques that will account for the mass percentage of material in the sample that is outside the range of sizes analyzed. X-ray sedimentation systems are a faster and automated alternative to the Andreasen pipette method but report equivalent data with Stokes diameter values especially relevant to applications where settling or sedimentation behavior is of interest e.g. in soil and mineral analysis.

Electrical Sensing Zone (Elzone® II)

Basis of measurement: Spherical diameter equivalence on the basis of electrical resistance (Coulter diameter or Electrical Sensing Zone (ESZ) diameter)

A schematic of the apparatus required for particle sizing via the Electrical Sensing Zone technique or Coulter Principle is shown in figure 5.

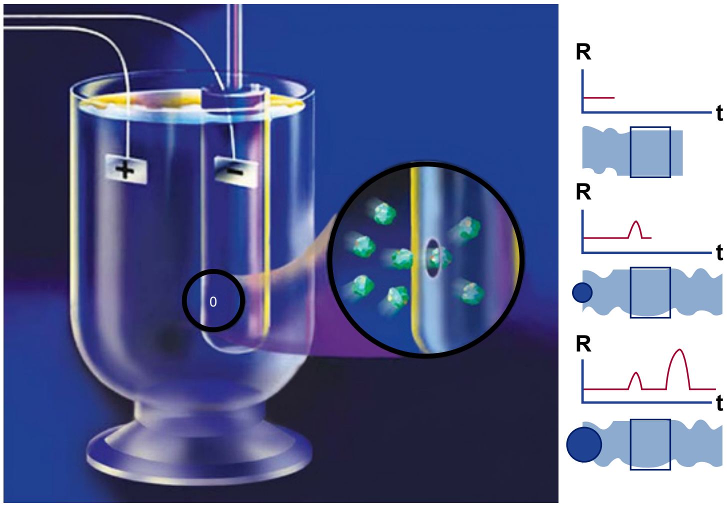


Figure 5: Non-conducting particles passing through the orifice of an electrical sensing zone instrument reduce its electrical resistance causing an electrical pulse that correlates with particle size.

Sample is dispersed at low concentration in a compatible electrolyte solution, aqueous or organic, and placed in a sample cup. Current flows through the apparatus from an electrode in the sample cup, through the orifice in a closed end glass dip tube, to a second electrode. Sample - particles and electrolyte – is also drawn through the orifice by application of a partial vacuum. The electrical resistance of the orifice is defined by equation 2.

$$R_i = \frac{\rho L}{A}$$

Equation 2: The end-to-end resistance of a linear conductor of constant cross-section (Ri) is a function of the geometry of the orifice [Where L = length, A = cross-sectional area, and ρ = the resistivity of the electrolyte.]

When there is only electrolyte in the orifice, resistance is at a minimum (see top figure on the right of figure 5). A spherical non-conducting particle swept through the orifice displaces a volume of electrolyte equal to that of the particle effectively reducing the cross-sectional area (A) of the orifice and increasing electrical resistance (Ri). This triggers a voltage spike across the electrodes (see figure 5), the amplitude of which is proportional to particle volume. For non-spherical particles, particle size is defined as the diameter of a sphere of the same material that gives the same change in electrical resistance as that of the measured particle. This is the **equivalent spherical Electrical Sensing Zone diameter** or **Coulter diamete**r.

In an electrical sensing zone measurement particle shape affects the transit time of the particle through the orifice (see figure 6), with longer particles giving rise to wider pulse widths. Since individual pulses are recorded for each particle, this digital signature of a sample can be interrogated to provide some assessment of particle shape, to differentiate agglomerates and dispersed particles, for example. Counting the pulses and sorting them by amplitude produces a number-based particle size distribution for the sample.

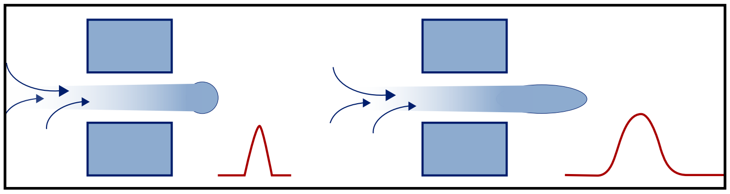


Figure 6: Particle shape directly impacts the shape of signal pulse generated in electrical sensing zone measurements.

The Coulter Principle is only rigorously applicable if the:

- Particles in the sample are non-conductive
- Orifice diameter is suitable for the size of particles (particle diameter should lie between 2 and 70% of the orifice diameter)
- Concentration of particles is low enough to essentially eliminate the possibility of multiple particles passing through the orifice together
- The length of the longest particle is less than that of the orifice.

Orifice geometry is typically optimized to measure across a particle size range of ~0.5 to 250 µm, via the use of a series of interchangeable orifice tubes while concentration limitations are addressed as part of method development.

Why choose Electrical Sensing Zone sizing?

The Electrical Sensing Zone technique is fast, providing real-time measurements of exceptional resolution. A number-based technique it offers high sensitivity to fines and over-sized particles but because measurements are based on the volume of liquid displaced, these distributions are easily converted to volume-based data with no mathematical modelling. Furthermore, particle size measurements are essentially unaffected by the physical properties of the sample, providing that particles have no accessible porosity, since this leads to undersizing. Particle size distribution data can be generated for samples containing particles with mixed or varying physical properties, with different or unknown optical properties, densities, colors and shapes. This is a useful attribute for many applications and ensures robust measurement in the face of process variability.

This is a well-established technique (1963) and the associated technology has been refined extensively to maximize its inherent attractions for industrial applications. For example, using a precision metering pump to draw dispersion through the orifice makes it possible to convert particle counts into number of particles per unit volume of liquid to generate solids concentration data. These can be particularly useful for settling or dissolution studies.

Static light scattering (Saturn DigiSizer® II)

Basis of measurement: Spherical diameter equivalence on the basis of light scattering (Spherical scattering diameter or Mie diameter)

Studies of the physics of light have long since established that light interacting with a particle is scattered – diffracted, reflected or refracted (see figure 7). Mathematical models describing this behavior underpin two related but markedly different particle sizing methods: static light scattering, commonly known as laser diffraction, and dynamic light scattering. Let's start by looking at static light scattering.

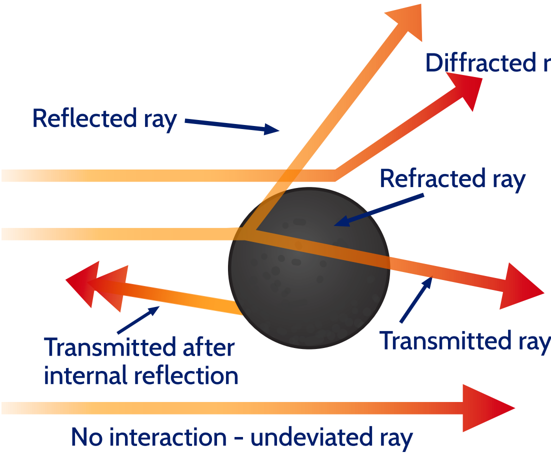


Figure 7: Light interacting with a particle is subject to scattering – diffraction, refraction or reflection.

Monochromatic, coherent light interacting with a spherical particle is scattered with specific relative intensities (to the incident beam) at specific angles, producing a pattern that is dependent on particle size, in accordance with Mie theory (1908 - see Equation 3).

$$I(\Theta)= \frac{I_o [i_1(\Theta) + i_2(\Theta)]}{2k^2\alpha^2}$$

Equation 3: Scattered light intensity at angle Θ ($I(\Theta)$) is a function of incident light intensity (I_o), wavenumber (k) and the distance between the scattering particle and the detector (α). [Where k is defined as $2\pi/\lambda_i$, λ_i is the wavelength of the incident light, and $i_1(\Theta)$ and $i_2(\Theta)$ are scattering functions.]

And the scattering functions themselves are a function of:

$$\frac{2\pi R n_m}{\lambda_i}$$

[Where: R is the radius of the spherical particle and n_m is the refractive index of the suspension medium.]

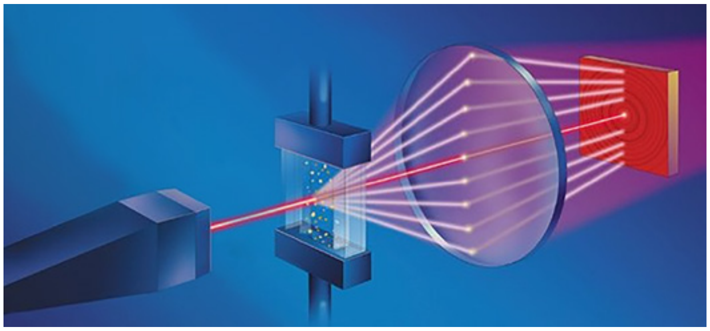


Figure 8: In a static light scattering analysis the sample is illuminated by a source of monochromatic light and particle size is then determined from the detected scattering pattern.

In a static light scattering analysis, the sample is illuminated by a source of light and scattered light intensity is then detected as a function of angle to the incident beam. Mie theory allows us to calculate the size distribution of a population of spheres that would give rise to a specific scattering pattern, with light of a certain wavelength, in a suspension of known refractive index. By mathematically 'comparing' these theoretical populations with the measured scattering pattern it is therefore possible to determine a particle size distribution, the amount (volume) of particles of each size present, that best represents the sample.

The particle size produced by static light scattering is therefore the diameter of a sphere of the same material that produces a scattering pattern that best fits the observed scattering pattern. This is the **equivalent spherical scattering diameter** or **Mie diameter.**

Mie theory is only rigorously applicable if:

- Particles are spherical and isotropic, scattering equally however orientated
- The concentration of the sample is low enough to prevent multiple scattering, the interaction of light with more than one particle prior to detection
- All particles have the same refractive index
- The real refractive index of the suspending medium is known

Certain of these issues, such as concentration, can be addressed via method development and optical properties are readily accessible for many systems. However, the data fitting process associated with static light scattering is relatively complex. This is not a particle-by-particle sizing method, rather the distribution in its entirety is determined by comparison to a theoretical volume-based distribution, with any deviation from the assumptions associated with Mie theory directly impacting the integrity of the reported data. If the sample contains particles with different refractive indexes, for example, the reported distribution will still be a population of spheres of identical refractive index and skewed with respect to size values by the fitting process. Similarly, if particles are irregularly shaped, and therefore anisotropic, or porous, the the reported particle size distribution can be distorted.

Why choose static light scattering?

Static light scattering is a fast, mature technique with a very broad dynamic range - from tens of nanometers to millimeters, depending on instrument configuration. As a result, it is suitable for the vast majority of industrial particle sizing applications. Traditionally a photo diode array is used for detection of the scattered light pattern, but state-of-the-art systems are configured with a charge coupled device (CCD) to significantly boost resolution. The performance of such systems is particularly differentiated with respect to the detection of fines and coarse particles, with much greater resolution achieved at the extremes of the particle size distribution. By combining exemplary repeatability, reproducibility, and sensitivity these systems are able to detect minute differences in particle size distribution that standard laser diffraction analyzers are unable to, elucidating complex behaviors and boosting troubleshooting capabilities.

Dynamic light scattering (Zetasizer)

Basis of measurement: Spherical diameter equivalence on the basis of diffusion speed (Hydrodynamic diameter)

In dynamic light scattering, measurements of light intensity fluctuation are used to determine the speed at which fine particles move when suspended in a liquid, as they exhibit random Brownian motion. The velocity of Brownian motion is defined by the translational diffusion coefficient and related to particle size via the Stokes-Einstein relationship (see equation 4).

$$D = \frac{k_B T}{3\pi\eta_0 d}$$

Equation 4: The Stokes-Einstein relationship shows that translational diffusion coefficient (D) is inversely proportional to the hydrodynamic diameter of a particle (d) [Where k_B is Boltzmann's constant, T is absolute temperature and η_0 is the viscosity of the suspending liquid.]

Size	Brownian Motion	Scattering Fluctuation
Small	Fast	Rapid
Large	Slow	Moderate

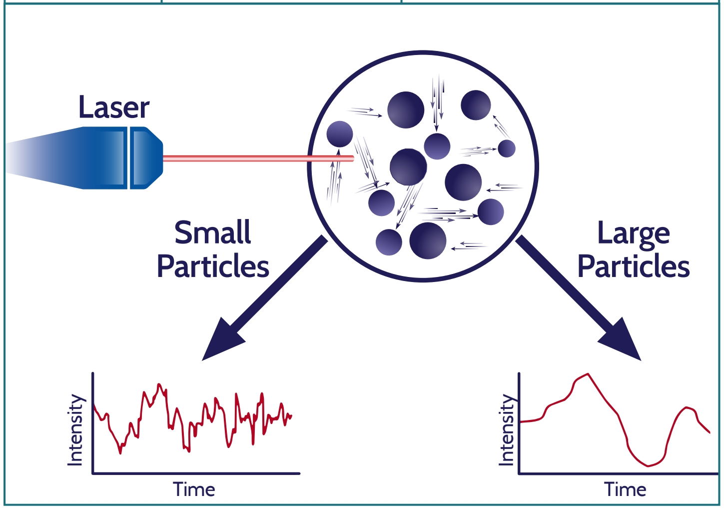


Figure 9: The size of particles in a sample can be determined from fluctuations in the scattered light pattern they produce as a result of Brownian motion.

The speed of Brownian motion is determined by detecting short-term fluctuations (measured over a timescale of microseconds) in scattered light intensity, produced by illuminating the sample with a source of coherent monochromatic light. Measurements are made using a photon counting device and then subject to correlation, a data processing technique that involves determination of the time autocorrelation function of the scattered intensity in accordance with equation 5.

$$G(\tau) = \frac{I(t_0)^* \, I(t_0 + \tau)}{I(t_\infty)^2}$$

Equation 5: The time autocorrelation function G(τ) is a function of intensity at time 0 ($I(t_0)$), intensity measured after a short time delay ($I(t_0 + \tau)$) and intensity after an infinite time ($I(t_\infty)$), in reality after just a couple seconds, because of the timescale of analysis.

The autocorrelation function is determined for multiple time delays, from just a few nanoseconds, through to several milliseconds, to produce a correlation coefficient or autocorrelation plot (see figure 10). In simple terms this plot shows how quickly the similarity of the signal decays, with respect to itself, as a function of the time delay associated with the comparison [1]. Loss of correlation occurs more quickly for samples exhibiting light intensity fluctuations over a shorter timescale i.e. those containing smaller, faster moving particles. From the autocorrelation plot it is possible to assess the quality of the measurement (the signal to noise ratio), and to determine both particle size and polydispersity (see figure 10).

Within the Stokes-Einstein relationship there is an assumption of sphericity. For non-spherical particles, the size reported is the diameter of a sphere of the same material that diffuses at the same speed as the particle being measured, the hydrodynamic diameter.

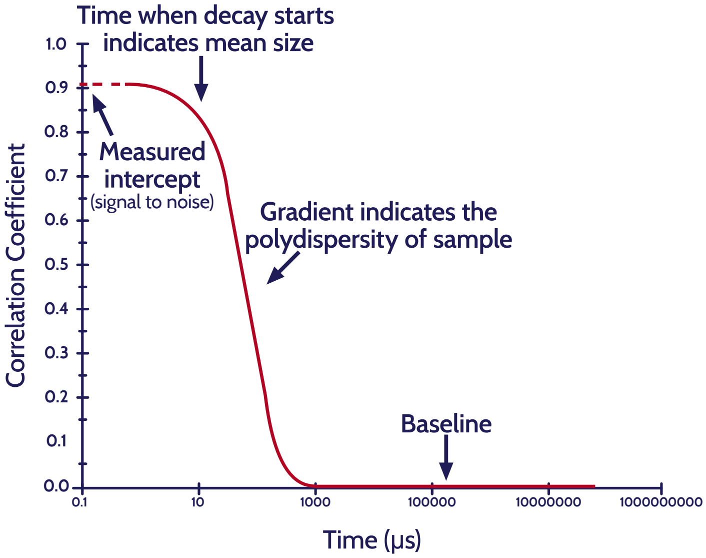


Figure 10: The correlation coefficient curve shows how quickly loss of correlation occurs in the detected light intensity fluctuation pattern with smaller particles losing correlation more quickly than larger ones.

The principles underpinning dynamic light scattering give rise to the following limitations:

- Particles must be moving solely as a result of Brownian motion, rather than settling
- The suspension must be stable i.e. with respect to the degree of dispersion
- The viscosity of the suspension must not be significantly different from that of the pure base fluid

With respect to physical properties it is only necessary to know the refractive index and the viscosity of the suspending liquid.

Why choose dynamic light scattering?

The defining attraction of dynamic light scattering is its dynamic range which extends from below 1nm to 10 μm making the technique highly complementary to the others outlined here for industrial particle sizing. Rapid, repeatable and reproducible, dynamic light scattering is a versatile technique for measuring materials in the nano-region, an area of growing interest in many industries. Furthermore, sample size requirements are minimal. This can be particularly useful in research and/or when dealing with novel materials.

Air permeability (MIC SAS)

Basis of measurement: Average particle diameter on the basis of specific surface area

Air permeability is a dry particle sizing technique with a sizing range that is highly complementary to sieving, as evidenced from the name of the instrument most traditionally associated with it: The Fisher 95 Sub-Sieve Sizer.

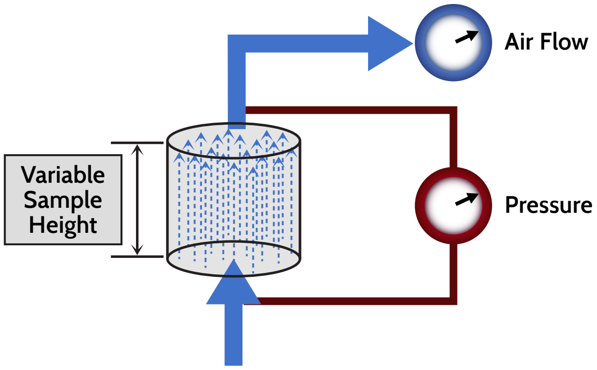


Figure 11: Specific surface area by can be determined by measuring the pressure drop across a powder bed at a defined air flow rate.

In an air permeability measurement, a compact bed of powder of known height is loaded into the measurement cylinder between two pressure tappings. The pressure drop associated with a specific air flow rate through the bed is measured. The Kozeny-Carman equation describes the relationship between the specific surface area of the sample, pressure drop across and air flow rate (see equation 6).

$$S = \frac{7d}{\rho\,(1 - \varepsilon)} \sqrt{\frac{\varepsilon^3 \pi \Delta P}{l \eta Q}}$$

Equation 6: The Kozeny-Carman equation correlates the specific surface area of the sample (S) with pressure drop across the powder bed (ΔP) and air flow rate through the bed (Q). [Where d is the diameter of the cylinder, ρ is sample particle density, ε is volume porosity of the bed, l is cylinder length and η is air dynamic viscosity]

In practice there are two ways a measurement can be carried out. Either a certain pressure drop can be maintained/targeted by varying flow rate, or pressure drop can be measured for a defined flow rate. Either way it is the specific surface area of the sample that is calculated, and this can only be converted into an averaged particle size. Unlike the other particle sizing techniques described here air permeability provides a single averaged data point for the sample with no insight into particle size distribution (see equation 7).

$$d_p = \frac{6}{S\rho}$$

Equation 7: An average particle diameter (dp) can be inferred from specific surface area (S) and particle density (ρ).

Alongside the limitation of not being able to provide distribution data, air permeability has the following constraints:

- The primary data reported are specific surface area though these are converted into particle size values
- The surface area measurement does not take account of sub-surface structures such as pores and cavities
- There is an assumption that the bed consists of uniform spherical particles.

Why choose air permeability?

Air permeability is a quick, simple, traditional technique for measurement in the 0.5 to 75 µm size range where dry sieving becomes impractical. It requires minimal sample preparation and is specifically referenced in many ISO and ASTM standards. Analysis is dry and non-destructive, and particles are static during measurement. Air permeability is particularly useful for relatively dense particles with minimal accessible porosity, metal powders being a prime example. Advances in instrumentation have made air permeability a much easier to use, highly automated technique while generating data that are highly consistent with traditional 'Fisher numbers', the benchmark for particle sizing in a number of industries.

Comparing particle size data

With a clearer understanding of the different particle sizing techniques in place it is useful to observe how they perform, for samples with different characteristics. The following studies provide useful insight in this regard.

Study 1 – Glass spheres

Glass spheres exemplify the type of sample for which techniques produce the most closely similar data. These particles are relatively smooth, non-porous, regularly shaped and almost perfectly spherical. Particle size distributions for glass bead samples measured by electrical sensing zone, static light scattering, dynamic light scattering, and X-ray sedimentation are shown below. There is close agreement between all the data sets with the exception of the X-ray sedimentation results which indicate a slightly broader size distribution. This is attributable to a density distribution within the sample.

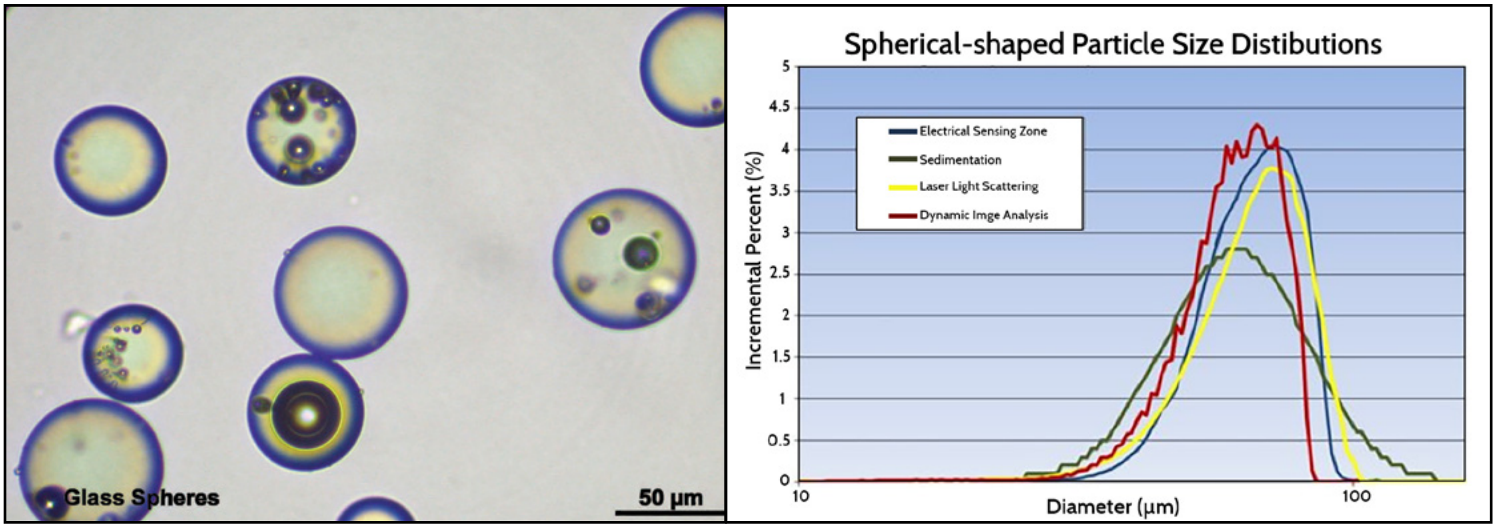


Figure 12: Particle size data for glass beads shows relatively close agreement between the techniques, a density distribution within the sample broadening the distribution observed with sedimentation.

Study 2 – Garnet

Analyses of garnet highlight the impact of measuring somewhat irregular particles, a more industrially relevant situation. Here static and dynamic light scattering produce particle size distributions centered on a larger particle size that the other two techniques. These particles scatter anisotropically, and orientation is likely to be a factor increasing measured particle size. On the other hand, in sedimentation analysis, the irregular shape has a dragging effect, leading to a slower terminal velocity and relatively small reported particle size distribution.

The electrical sensing zone method determines size based on volume displaced which means that reported particle size distributions tend to be small. Electrolyte solution fills the pores and cavities of an irregular particle generating a diameter associated with the skeletal volume. This contrasts with the diameter reported by alternative methods such as light scattering which tends to be more influenced by the larger dimensions of the particle, and essentially more reflective of an envelope volume.

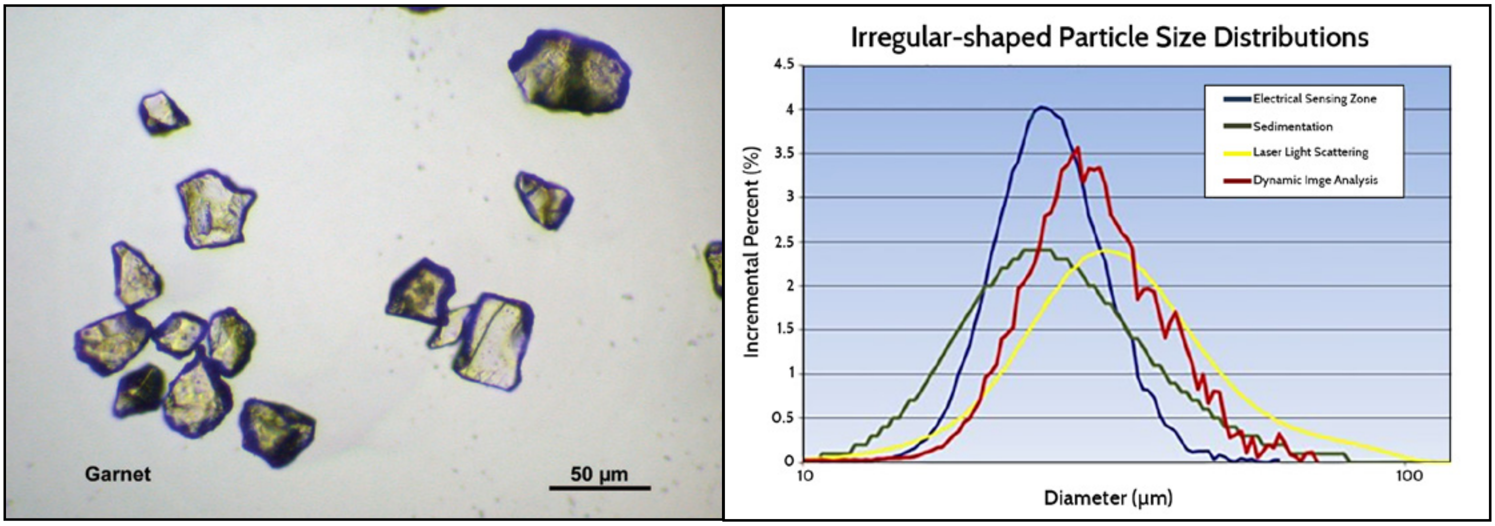


Figure 13: Irregularity of particle shape leads to divergence in the particle size data; anisotropic scattering impacts light scattering results while sedimentation data are impacted by the increased drag associated with particle shape.

Study 3 – Wollastonite

Differences between the results reported by the alternative particle sizing techniques are particularly exacerbated when it comes to highly irregular, needle-shaped particles as found in this wollastonite sample. As with garnet, both light scattering techniques report a larger particle size than the two alternative techniques and with static light scattering a multi-modal distribution is observed. As these particles tumble through the flow path of the instrument light can scatter from very differently sized facets of the particle – the length of the rod or the end, which has a much smaller dimension. A range of spherical size models is required to fit the scattering pattern of this high aspect ratio of sample and the result is the multi-model distribution observed.

The breadth of the sedimentation distribution is associated with alignment of the particles which settle in the manner of least resistance, with long particles having an elongated surface causing a drag effect as they settle. The electrical sensing zone method, as with garnet, produces a particle size distribution centered on a relatively fine particle size because the skeletal volume of the rods is converted to a solid spherical volume, on the basis of volume displaced.

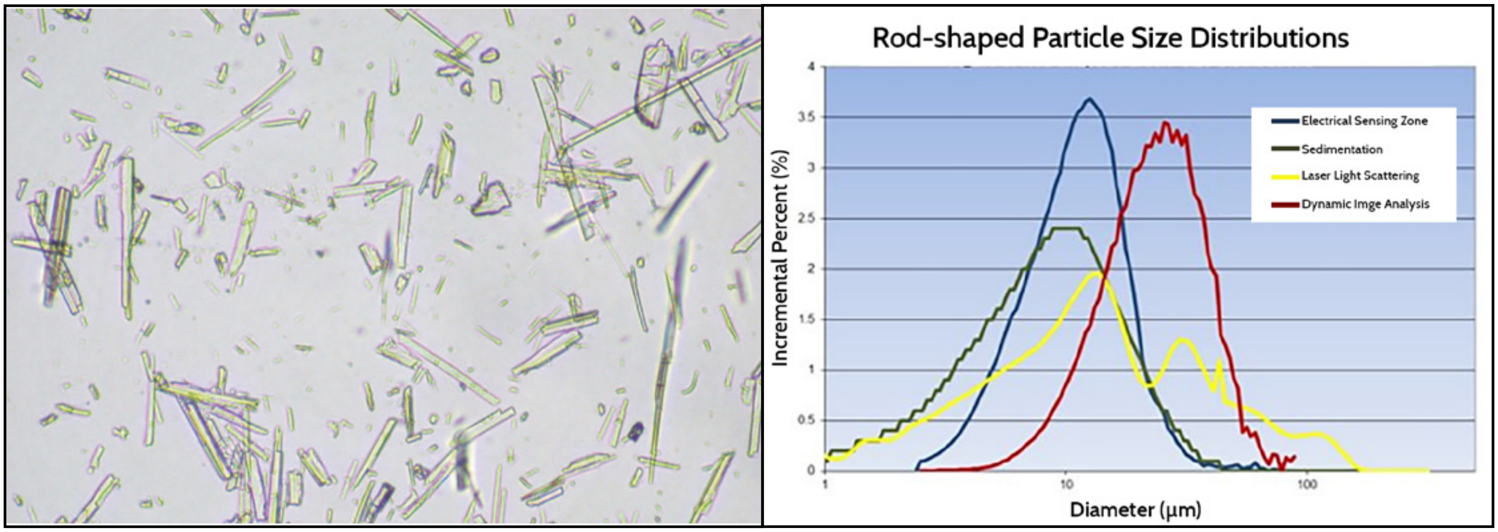


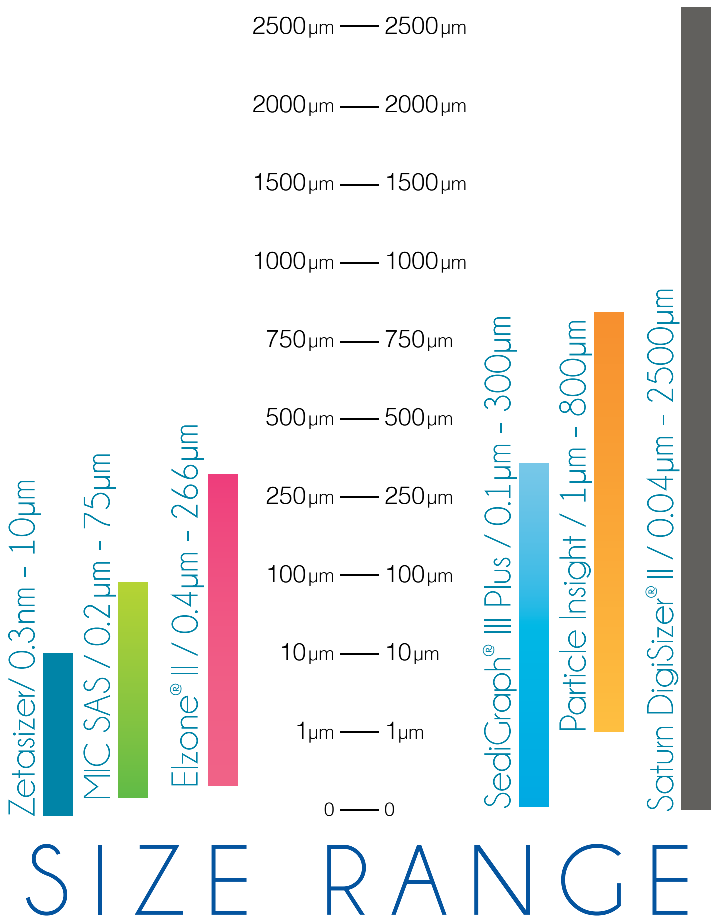
Figure 14: The impact of particle shape is particularly pronounced with high aspect ratio or needle shaped particles, with static light scattering reporting a multi-model distribution because of the impact of random orientation.

Concluding thoughts

When it comes to choosing a particle sizing technique it is vital to recognize that there is no definitive 'right' technique, no one descriptor of particle size. Rather there are a number of techniques each of which produces a different numerical measure of particle size, based on spherical equivalence via the measurement of a specific property. Only in relatively limited cases will these results agree, a crucial point to be aware of when it comes to comparing or reporting data. Including the method of measurement is good practice when quoting particle size data, along with shape data if available.

There is an array of excellent technology to choose from to meet particle sizing applications, the maturity of many techniques resulting in highly advanced, easy-to-use instrumentation. Understanding how a technique works and what this means in terms of the resulting data is essential, whether simply applying an industry accepted method or looking for fresh insight to solve a problem. Bringing a complementary technique into the mix can be just what is needed to progress, the use of dynamic image analysis to provide shape data to elucidate particle performance or analytical failure being a classical example.

Selecting the right particle sizing solution(s) for your application is the key to repeatable, reproducible data that sensitively differentiate samples in a way that is relevant to your work, to access optimal, cost effective support.



Technique	Application	Originally Developed for	Defining attraction(s)
Dynamic Image Analysis	Any organic or inorganic powders or aqueous bases suspensions where particles are larger than 1 micron	Shape analysis	Accelerated microscopy; provides statistically significant shape data, alongside size information
Sedimentation	Inorganic material in high concentration; typically soils, minerals, and fine metal powders	High concentration slurries	Automated measurement generating data equivalent to Andreasson pipette method; accounts for mass percent of particles outside of size range analyzed
Static Light Scattering (Laser Diffraction)	Any organic or inorganic powders or aqueous based suspensions	Fast analysis for a quick check of size distribution	Rapid generation of a particle size distribution, broad dynamic range
Electric Sensing Zone	Small concentrations of organic or inorganic particles, or aqueous based suspensions	Particle counting or concentration	Simultaneous measurement of particle number/ concentration; ability to handle samples with mixed properties
Air Permeability	High density powders, such as powdered metals	Fast analysis of powdered metals	Automated measurement generating data equivalent to Fisher Sub-Sieve Sizer
Particle suspension analysis for sub-micron range	Any nano-suspension of particles or emulsions smaller than 1 micron. Typically includes Zeta Potential	Particle suspension analyzer for sub-micron range	Sizing into the nano range; minimal sample volume requirements.

References:

[1] 'Dynamic light scattering – An introduction in 30 minutes' Technical Note available to view at: http://particulatesystems.com/wp-content/uploads/2018/09/Dynamic_Light_Scattering_An_Intro_in_30_Minutes.pdf

[2] 'The Effect of Particle Shape on Particle Size Measurement' Available to view at: https://www.micromeritics.com/Repository/Files/The_Effects_of_Particle_Shape_on_Measured_Particle_Size_2011.pdf