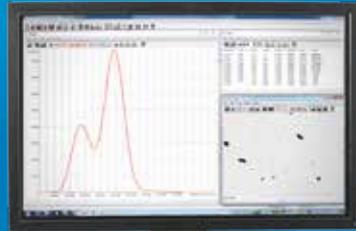




Sieve Shaker  
AS 200 control



Optical Particle Analyzer  
CAMSIZER<sup>®</sup> XT



Laser Diffraction Particle  
Size Analyzer  
LA-960

**Retsch Technology GmbH**  
Retsch-Allee 1-5  
42781 Haan, Germany

Phone +49 21 04/23 33-300  
Fax +49 21 04/23 33-399

E-Mail [technology@retsch.de](mailto:technology@retsch.de)  
Internet [www.retsch-technology.de](http://www.retsch-technology.de)

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## Different measuring techniques provide different results – but what is the truth?

The most common methods to determine the particle size are dynamic digital image analysis (DIA), static laser light scattering (SLS, also called laser diffraction) and sieve analysis. This article presents the advantages and drawbacks of each technique, their comparability among each other as well as detailed application examples.

Each method covers a characteristic size range within which measurement is possible, as shown in figure 1, and which partly overlap. The three methods presented here, for example, all measure particles in a range from 1 µm to 3 mm. However, the results for measuring the same sample can vary considerably.

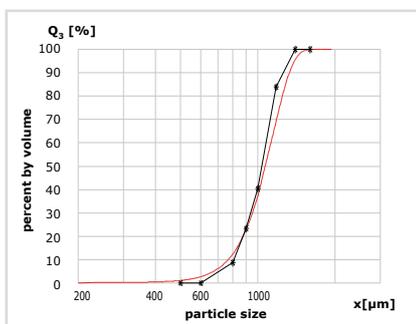
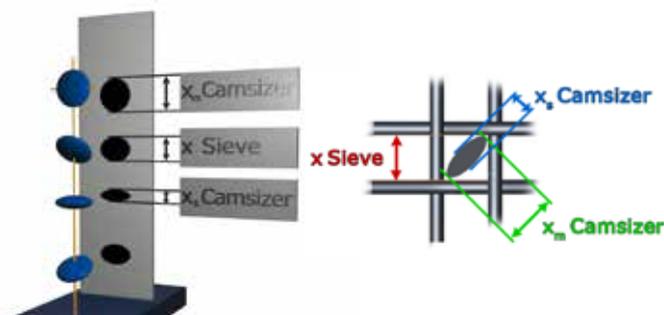
**This article will help to interpret the informative value and significance of particle analysis results and to decide which method is best suited for a particular application.** The analyzers used for the measurements presented in this article are sieve shakers (Retsch), image analysis systems CAMSIZER<sup>®</sup> P4 and CAMSIZER<sup>®</sup> XT (Retsch Technology) and laser granulometer Horiba LA-960.

Fig. 1: Measuring ranges of different methods

	Particle size			
	1 nm	1 µm	1 mm	10 cm
<b>Sieve analysis (dry)</b>			20 µm	30 mm
<b>Digital image processing</b>		20 µm	CAMSIZER <sup>®</sup> P4	30 mm
		1 µm	CAMSIZER <sup>®</sup> XT	3 mm
<b>Light scattering</b>	10 nm	LA-960	5 mm	

## Sieve analysis: committed to tradition

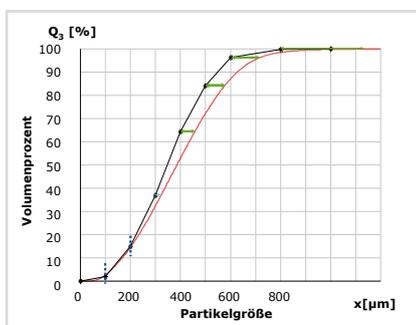
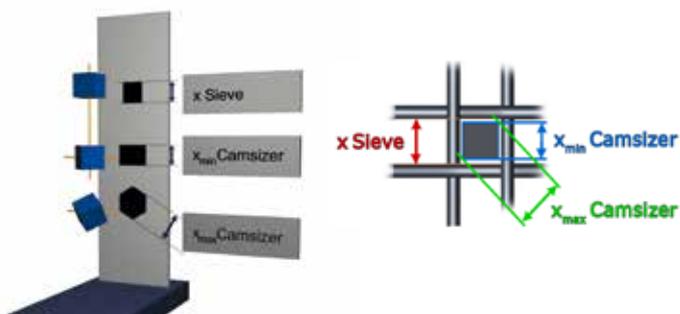
Sieve analysis still is the traditional and most commonly used method for particle size determination. A sieve stack consists of a number of sieves with increasing aperture sizes stacked upon each other and the sample is placed on the uppermost sieve. The stack is clamped to a sieve shaker and set into vibration for a period of time. As a result, the particles are distributed to the sieves in the stack (fractions) according to their size. Ideally, the particles pass the smallest possible sieve aperture with their smallest projection surface. Taking cubic particles as a model, this corresponds to the edge length of the cube. For lenticular particles, the size determined by sieve analysis would be a value between the thickness and the diameter of the lense, as the particle is oriented diagonally towards the sieve aperture (fig. 2 and 3). **Hence, sieve analysis is a technique which measures particles in their preferred orientation with a tendency to determine mostly the particle breadth.**



Sieve analysis is carried out up to a point where the sample mass on the respective sieves no longer changes (= constant mass). Each sieve is weighed and the volume of each fraction is calculated in percent by weight, providing a mass-related distribution. **The resolution of sieve analysis is limited by the number of obtainable**

Fig. 2: Model of a measurement of lenticular particles with sieve analysis and DIA. The lense falls diagonally through the smallest possible sieve aperture. DIA "sees" the lense larger or smaller, depending on its orientation. This results in different particle size distributions: the red curve shows the DIA result, the black curve represents sieve analysis results.

**size fractions.** A standard sieve stack consists of a maximum of 8 sieves which means that the particle size distribution is based on only 8 data points. Automation of the procedure is hardly possible which makes it rather time-consuming. The single steps of sieve analysis are: initial weighing, 5 – 10 minutes sieving, back weighing, and cleaning of the sieves. The most common sources of error are: overloading of the sieves (blocking of sieve apertures, too coarse results); old, worn or damaged sieves (too fine results); or errors in data transfer. It should also be taken into account that the aperture sizes of new standard-compliant sieves are also subject to certain tolerances. The average real aperture size of a 1 mm sieve, for example, is permitted to deviate about  $\pm 30 \mu\text{m}$ , for a  $100 \mu\text{m}$  sieve it is  $\pm 5 \mu\text{m}$  (i.e. the average real aperture size lies between 95 and  $105 \mu\text{m}$ ). However, this is just the mean value which means that some of the apertures can be even larger. With sufficient sieving time the particles find the largest apertures which results in particles larger than the nominal aperture size of the sieve passing through the sieve.



Thus the sieve becomes effectively larger than the nominal aperture size indicates. These tolerances are particularly apparent in sieve analysis results of spherical samples or samples with narrow particle size distributions, as can be clearly seen in figure 4 which shows the measurement results of a sample of glass beads.

Fig. 3: Model of a measurement of cubic particles with sieve analysis and DIA. Sieve analysis determines the edge length whereas DIA measures the edge length or, depending on the orientation of the particle, a higher value (max. edge length \* square root of 2 of the hexagonal projection), but never smaller than the value obtained by sieve analysis. Red curve = measurement with DIA, black curve = sieve analysis.

In addition to the dry sieving procedure with wire mesh sieves described in this article, there are other special methods such as wet sieving or air jet sieving.

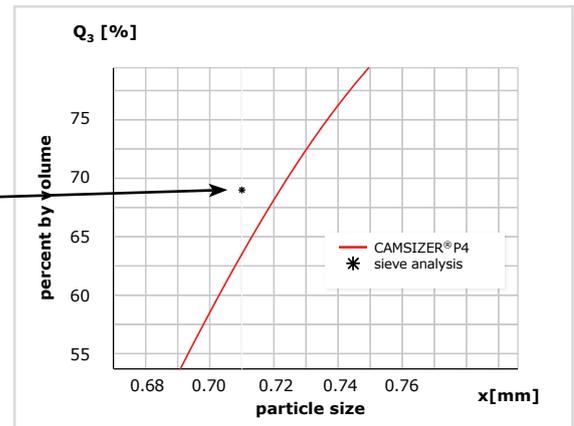
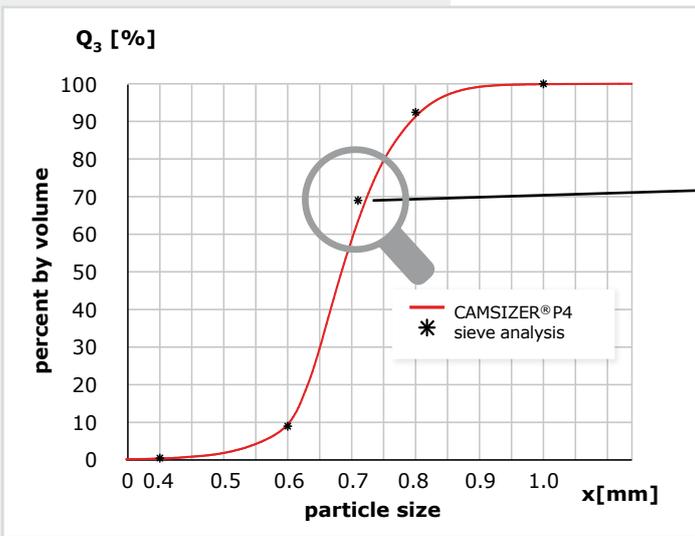


Fig. 4: Measurement of a glass bead sample with DIA (CAMSIZER® P4, red) and sieve analysis (\* black). The measurements can only be compared at the points which represent the sieve fractions. There is excellent agreement between the results.

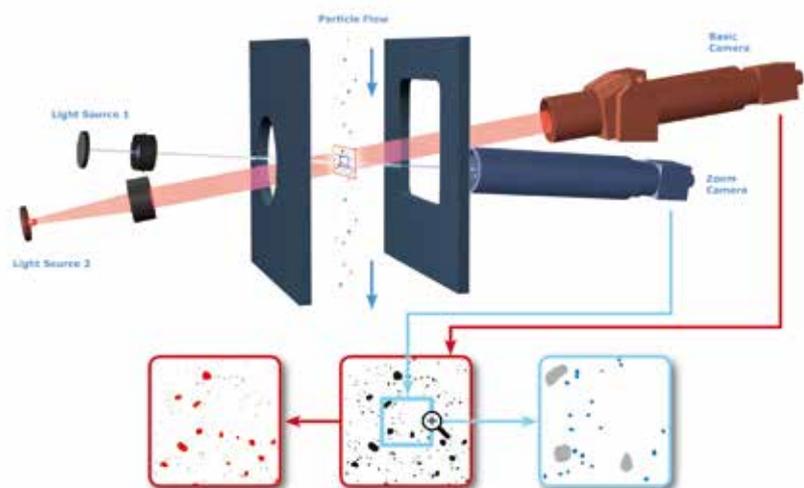
When taking a closer look at the data at 710 µm, it becomes apparent that the deviation of the Q<sub>3</sub>(x) value is 6% which seems quite a lot at first sight. However, the deviation in size is only 13 µm and is thus within the tolerance of a 710 µm sieve which is ±25 µm. As the cumulative curve is very steep at this point, even a small difference in size has a strong impact on the Q<sub>3</sub>(x) value.

## Dynamic Digital Image Analysis (DIA): What you see is what you get

There are two techniques of dynamic image analysis. Static DIA is basically a microscope which measures the sample placed on an object plate step by step. Although the quality of the images is very good and the optical resolution quite high, this technique has some decisive drawbacks with regards to representing particle size distributions: the measuring range is limited, the procedure is rather time-consuming and the quantity of analyzed particles is often not sufficient to obtain a statistically sound statement about the entire sample. Consequently, we will only discuss dynamic image analysis in this article. **This involves a large amount of particles which pass a camera system in a relatively short time (2 to 5 minutes) and are analyzed moving in real time.** Figure 5 shows a schematic of this measurement principle as implemented in the CAMSIZER® XT. The system measures free falling particles as well as suspensions and also features dispersion by air pressure for those particles which tend to agglomerate. **Modern DIA systems analyze more than 100 images per second in real time, detecting millions of individual particles within only a few minutes.** This performance is based on fast cameras, bright light sources, short exposure times and powerful software.

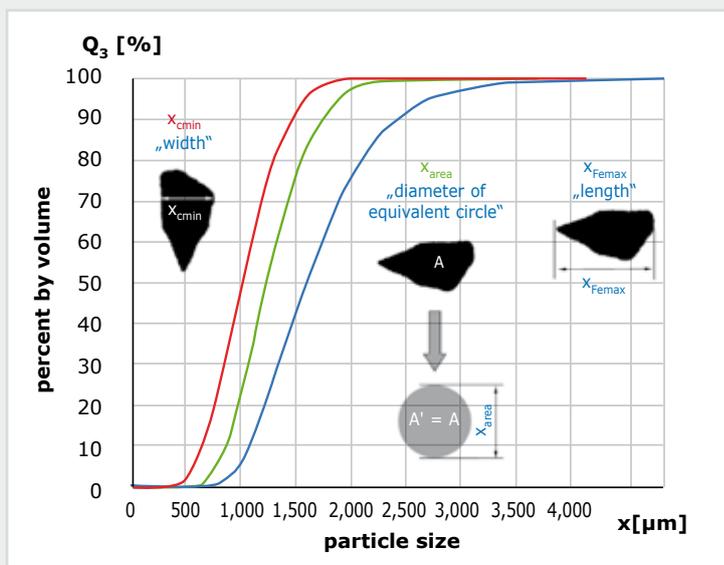
Fig. 5: The image analysis system of the CAMSIZER® XT. The CAMSIZER® series uses patented dual camera technology with different resolutions to realize an extremely wide measuring range. The cameras capture the projections of the particles.

## Patented Measurement Principle



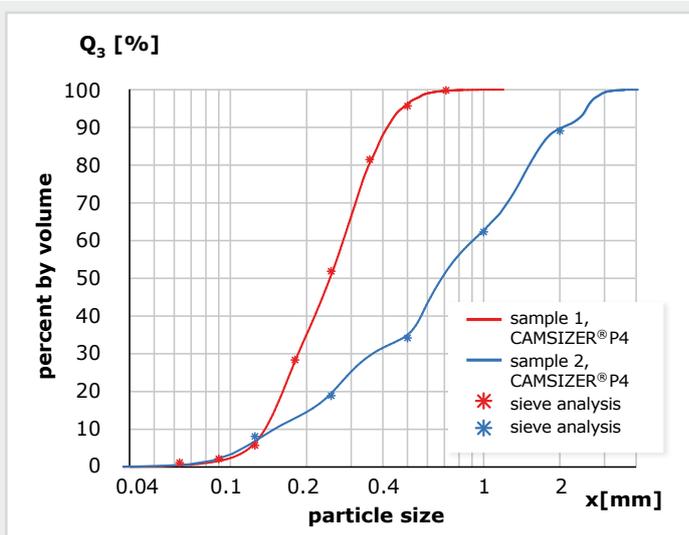
In contrast to sieve analysis DIA measures the particles in a completely random orientation. **A variety of size as well as shape parameters are determined based on the particle images.** Typical size parameters are, for example, breadth, length and diameter of equivalent circle (see fig. 6). Parameters to describe the particle shape include sphericity, symmetry, convexity and aspect ratio. **An essential characteristic of DIA is the extremely high detection sensitivity for oversized grains.** The CAMSIZER® P4, for example, is designed to detect every single particle of a sample; the model CAMSIZER® XT has a detection limit of 0.1% for oversized particles. The resolution of DIA systems is also unbeatable: smallest size differences within the micrometer range are reliably detected and multimodal distributions are dissolved without fail.

Abb. 6: The DIA uses various size definitions to determine the particle size distribution. Consequently, one measurement can produce several distributions. In this example, the red curve is based on the measurement of particle width; the blue one represents particle length. The parameter X-area stands for the diameter of equivalent circle which is defined as the particle size. It depends on the original question which results are finally relevant. When examining fibres or extrudates, the length parameters are of interest; width is more important if comparison to sieve analysis is required.



If DIA is compared to sieve analysis, the particle “breadth” is the common parameter. However, when measuring irregularly shaped particles there are still systematic differences in the obtained results due to the fact that DIA measures the particles in random orientation. Figures 2 and 3 illustrate for special particle shapes how the differences in the particle size measurements occur and how they can be interpreted. The differences in particle size distributions are systematic for each defined particle shape. **The CAMSIZER® software features algorithms which allow for compensation of these differences so that the DIA results correlate to almost 100% to those obtained by sieve analysis** (fig. 7). This procedure is frequently applied in particle size analysis applications for quality control because in a globalized market many products are analyzed by different laboratories with different measuring techniques, creating a need for comparability.

Fig. 7: Example for the excellent agreement between measurement results of two sand samples obtained by DIA (red and blue curve) and sieve analysis (\*).



## Laser diffraction: Spheres and collectives

**With static laser light analysis, also called laser diffraction, particle sizes are measured indirectly by detecting intensity distributions of laser light scattered by particles at different angles.** Figure 8 shows the set up of a modern laser granulometer such as Horiba's LA-960. This technique is based on the phenomenon that light is scattered by particles and the correlation between intensity distribution and particle sizes are well-known. Simply put, large particles scatter the light to small angles while small particles produce larger angles. Whereas large particles produce rather sharp intensity distributions with distinctive maxima and minima at defined angles, the light scattering pattern of small particles becomes more and more diffuse and the overall intensity decreases. It is particularly difficult to measure differently sized particles in a polydisperse sample as the individual light scattering signals of the particles superimpose each other.

Optics Design LA-960

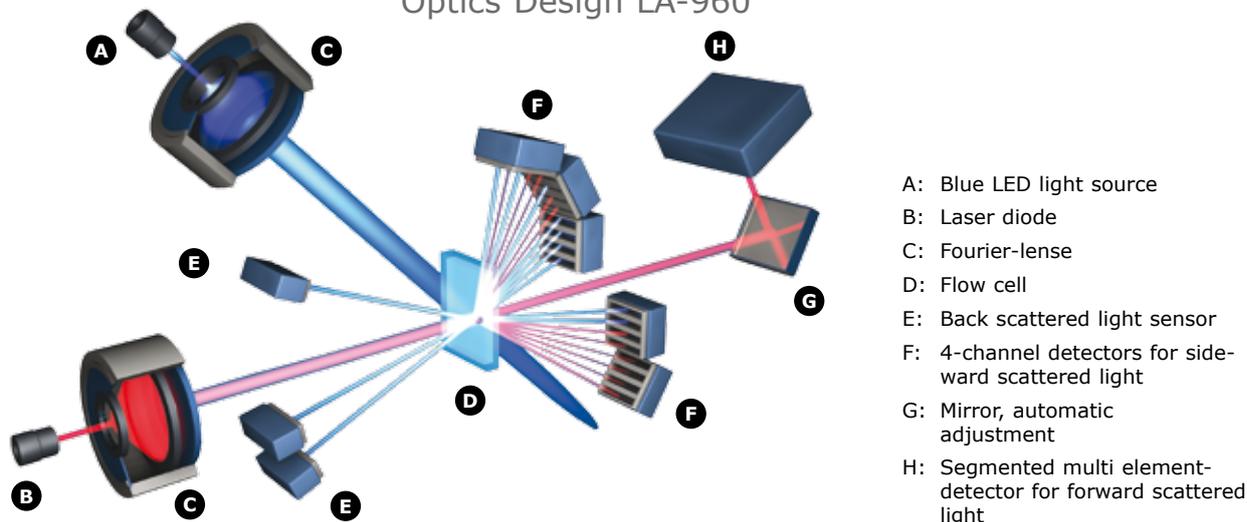


Fig. 8: The laser light scattering spectrometer Horiba LA-960 uses two light sources and 93 measurement channels to record the scattered light pattern over a wide angle. It is possible to analyze suspensions, emulsions as well as dry powders in a measuring range from 0.01  $\mu\text{m}$  to 5,000  $\mu\text{m}$ .

Static laser light scattering (SLS) is an indirect method which calculates particle size distributions on the basis of superimposed scattered light patterns caused by a whole collective of particles. Moreover, it is essential to know the optical characteristics of the material (refraction index) for small particles to obtain reliable results. SLS is based on the assumption that all particles are spherical so that shape analysis is not possible. **A big advantage of SLS is the enormously wide measuring range; none of the other techniques presented here is able to detect particles smaller than 1 micron.** Analyses with SLS are easy to carry out and they can be automated to a large extent. **A drawback of this method is the relatively poor resolution.** Even the latest analyzer generation cannot detect oversized fractions if the volume is below 2 Vol%. To dissolve multimodal distributions the size of the two components involved needs to differ at least by factor 3. More than three different components in a mixture are basically not detectable. Figure 9 shows the example of a mixture of polystyrene-latex standard particles. In contrast to SLS, dynamic image analysis is capable of detecting exactly four different particle sizes whereas the laser diffraction analyzer cannot accurately dissolve the 10  $\mu\text{m}$  and 12  $\mu\text{m}$  particles.

Fig. 9: Measurement of a mixture of four particle standards (2.5  $\mu\text{m}$  - 5  $\mu\text{m}$  - 10  $\mu\text{m}$  - 12  $\mu\text{m}$ ). While DIA is able to distinguish the four components (red), laser diffraction identifies only three components.

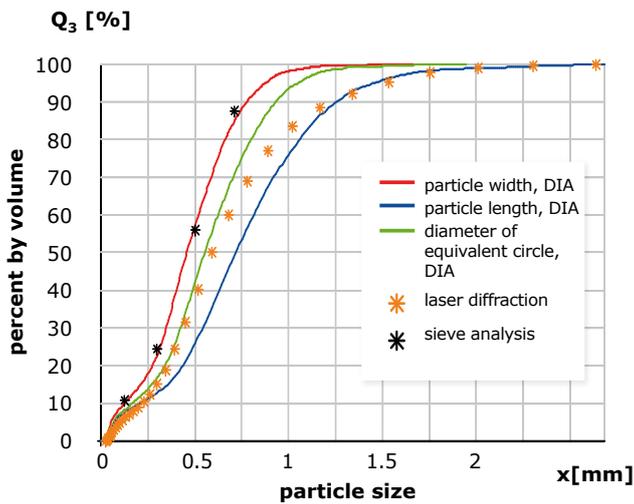
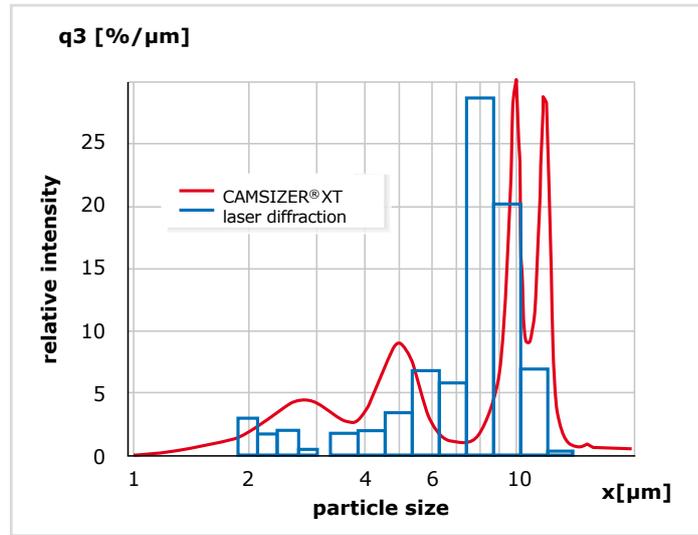
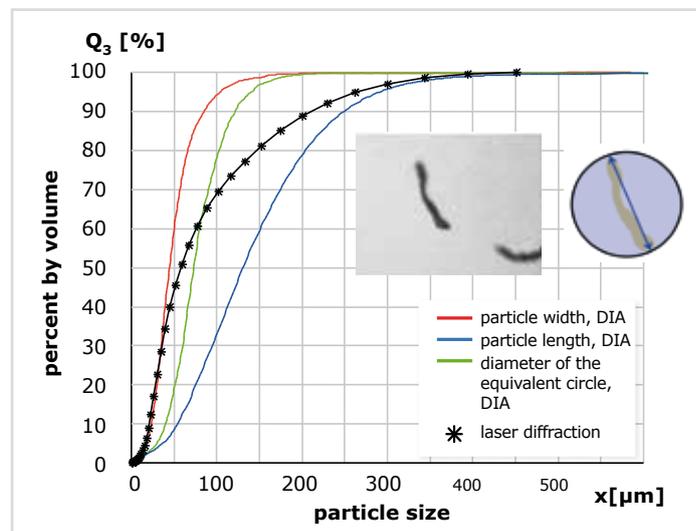


Fig. 10: Measurement of ground coffee with different methods. DIA, particle width (red); DIA, particle length (blue); DIA, diameter of the equivalent circle (green); laser diffraction (orange \*); sieve analyses (black \*).

Figure 10 illustrates the comparability between SLS, DIA and sieve analysis using the example of ground coffee. Sieve analysis provides the finest results; the measurement of particle breadth with the CAMSIZER<sup>®</sup> XT comes quite close to this. There is no comparability between sieve analysis and laser diffraction; the result obtained with SLS corresponds roughly to the X-area parameter (diameter of equivalent circle). The various particle dimensions which are measured are all attributed to spherically shaped particles. Therefore, SLS always provides wider size distributions than image analysis.

This becomes even more apparent in figure 11 which shows a measurement comparison of cellulose fibers. Whereas DIA distinguishes between the thickness and length of the fibers, SLS is not able to do so. The measurement curve of laser diffraction first runs parallel to the width measurement of DIA (red) and then approaches the "fibre length" (blue).

Fig. 11: Measurement of cellulose fibres. The CAMSIZER<sup>®</sup> XT (image analysis) measures particle breadth (red), particle length (blue), and X-area (green). The SLS measurement (\*) is a mixture of breadth and length and shows a continuous transition. DIA can distinguish between breadth and length.



## Conclusion

**Of all the methods presented in this article, dynamic digital image analysis is the only technique capable of providing exact information about the particle size while also considering their shape.** This means that it is possible to follow the results obtained by other methods and make them comparable, if required. Thanks to the direct measurement of particles, the information content and resolution of DIA are significantly superior to laser diffraction and sieve analysis.

The advantages of sieve analysis are the traditionally wide usage and the relatively cost-efficient equipment.

Particle size analysis by laser diffraction is the only method allowing for measurement in a size range below 1 micron.

The advantages and disadvantages of the respective method are summarized in tables 1 and 2.

Table 1: Comparison sieve analysis and dynamic digital image analysis

	Sieve Analysis	Image Analysis
Measuring range	10 µm – 63 mm	CAMSIZER® P4: 20 µm – 30 mm CAMSIZER® XT: 1 µm – 3 mm
Particle shape analysis	no	yes
Detection of oversized grains	each particle	CAMSIZER® P4: each particle CAMSIZER® XT: <0.1% Vol.
Resolution	poor	very high
Dissolution of multimodalities	poor	excellent
Repeatability and lab-to-lab comparison	limited	very good
Comparability of methods	identical results possible	
Handling	simple, time-consuming, error-prone	simple, objective, fast

Table 2: Comparison laser diffraction and dynamic digital image analysis

	Laser Diffraction	Image Analysis
Measuring range	10 nm – 5 mm	> 1 µm
Particle shape analysis	no	yes
Detection of oversized grains	> 2%	CAMSIZER® P4: each particle CAMSIZER® XT: <0.1% Vol.
Resolution	good in the micron range, getting worse with increasing particle size	excellent over the whole measuring range
Dissolution of multimodalities	limited (starting with factor 3 for mixtures)	significantly better size resolution
Comparability to sieve analysis	poor	identical results possible
information content	equivalent diameter, spherical model, indirect method	real particle dimensions, direct length