# Particle Characterisation with Dynamic Image Analysis Determination of particle size and particle shape with a new measuring technique

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Sieve analysis and laser diffraction are long established methods for the determination of particle size distributions. Dynamic Image Analysis (DIA) is another particle analysis technology to measure particles > 1 micron, which has a number of advantages over these methods.

Retsch Technology's particle analysers CAMSIZER and CAMSIZER XT, which are based on DIA technology, evaluate images of the particles which provide considerably more information on the particles than, for example, a light scattering pattern produced by a laser diffraction particle analyser. These only allow for an indirect measurement of the particle size. The determination of parameters such as the length, width, or sphericity of particles is only possible by using image analysis.

This article deals with the following topics:

- A short explanation of the measuring principle of Dynamic Image Analysis
- Demonstration of the advantages of the measuring technique when analysing powders, granules or suspensions with practical application examples
- Description of the modular dispersion and sample feeding systems of the CAMSIZER XT

## The Principle of Dynamic Image Analysis

The functional principle of Dynamic Image Analysis is similar to that of a microscope: A camera takes enlarged digital pictures of particles which are then analysed by software that calculates the size and shape of every single particle in the picture.

There are two important differences between conventional microscopy and DIA:

Conventional microscopy for particle size analysis is a static method which means that the particles on the object plate don't move in relation to the optics. The Dynamic Image Analysis technology however, records each particle in movement. A stream of particles is generated, for example by gravity, air pressure or liquids, and passes the camera which takes pictures in a quick succession.

This leads to the second difference. Dynamic Image Analysis typically records more than 10,000 images per minute, whereas static analysis methods only allow for analysis of a few images.

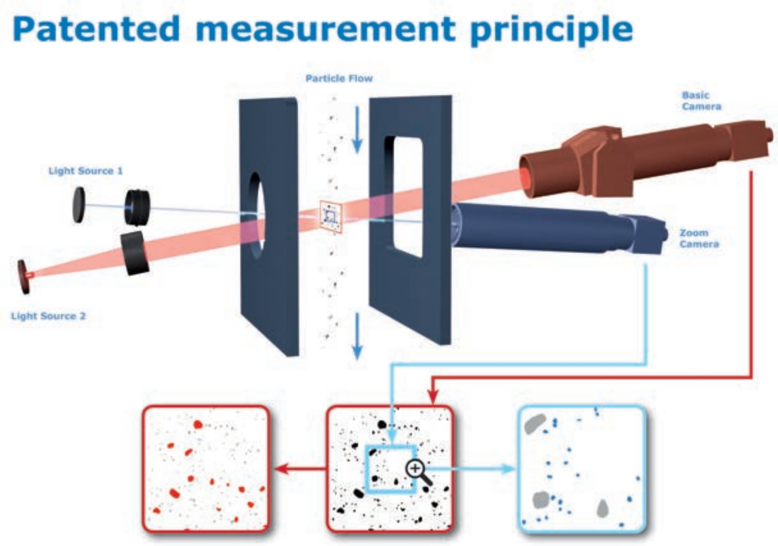


*Figure 1. Measurement principle Dynamic Image Analysis*

*Figure 1* shows the basic setup of the optics. A stream of particles moves through the measuring field. The particles are backlit while a camera on the opposite side takes images of the particles' silhouette. The software evaluates these images and very quickly determines the size distribution of the particles. A few hundred particles per image are evaluated in real time. Modern particle analysers such as Retsch Technology's CAMSIZER and CAMSIZER XT are equipped with two cameras working simultaneously, with different magnifications to cover a wide measuring range: one camera with high magnification is optimised for analysing the small particles, a second camera with a lower magnification but wide field of view allows to simultaneously analyse the larger particles with high detection efficiency. The CAMSIZER XT system records more than 275 images per second.

With Dynamic Image Analysis it is possible to analyse statistically relevant numbers of a few million particles very quickly. This method provides reproducible and statistically relevant results not only for the mean particle size but also for small quantities of undersized or oversized particles.

## Highest Resolution AND Exact Quantity

A mixture of 4 different monodisperse Latex particles was analysed with the CAMSIZER XT and a laser diffraction system. Thanks to the combination of the two megapixel cameras, the CAMSIZER XT was able to analyse particles smaller than 10 microns with considerably higher resolution than a laser diffraction analyser. Dynamic Image Analysis resolves the single components of 2.5 µm, 5 µm and 12 µm very well within the particle mixture. The mean sizes and proportions of the single components are precisely detected in the mixture. Although laser diffraction systems are able to measure the single components reliably, they fail to provide the total size distribution in high resolution. For example, a double peak of 10 µm and 12 µm cannot be resolved by a laser diffraction analyser.

With Dynamic Image Analysis every single particle is evaluated and assigned to a measuring class according to its size. This results in a high resolution which allows to differentiate between particles that are very close in size. Laser diffraction analysers measure the light scattering signals of the particles, thus determining the particle size indirectly. The size distribution of the particle collective is then back-calculated from the superimposed signals. The resolution provided by this method is substantially lower than that obtained by Dynamic Image Analysis.

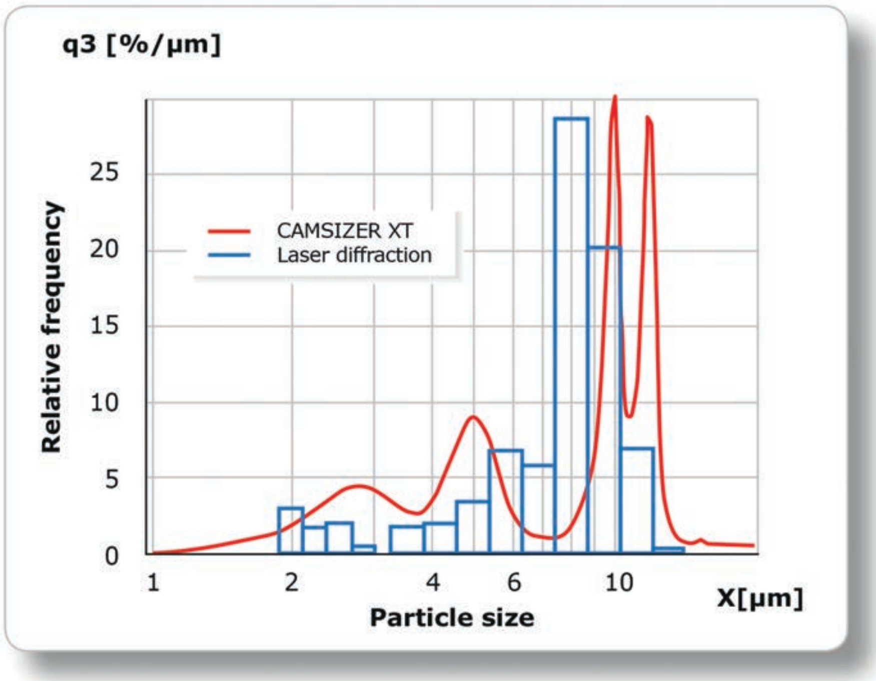


*Figure 2. Particle size distribution of a mixture of latex particles of 2.5 µm, 5 µm, 10 µm and 12 µm, measured with the CAMSIZER XT optical particle analyser and a laser diffraction system.*

## Particle Size: Exact Length and Breadth or just Equivalent Diameter?

Particle size analysis with the CAMSIZER XT records all the different particle characteristics. The example of cellulose fibres shows that the distributions with regards to different dimensions of the particles can be analysed simultaneously. The graphic shows the results in relation to the thickness, the area equivalent of the circle diameter, and the length of the fibres. Other measuring techniques, such as laser diffraction, only determine the equivalent diameter based on the assumption that the particle to be measured is spherical. By providing more detailed information on the particle, the CAMSIZER XT allows a much greater understanding of the particle.

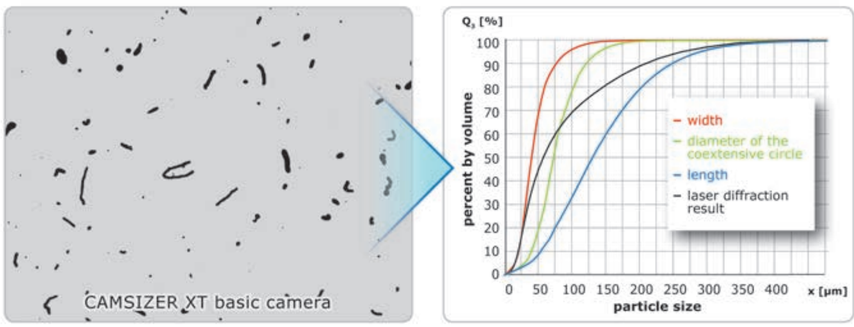


*Figure 3. Measuring cellulose fibres with CAMSIZER XT*

## Seeing is Believing: Analysis of Particle Shape

When measuring catalyst materials the amount of broken particles among the otherwise almost perfectly spherical particles is of interest. Broken particles can negatively influence the packing density of a catalyst and thus the flow characteristics in a chemical reactor. Non-round, broken particles can be easily detected with particle shape analysis. The width/length (b/l) aspect ratio or the symmetry parameters of broken particles differ considerably from those of spherical ones. The measurement sequence in *Figure 4* shows how an increasing amount of broken particles is reflected in the b/l diagram. The initial material is almost spherical with a b/l ratio of 0.95 (red curve). The increasing proportion of broken particles can be read off the amount at the threshold value 0.95.

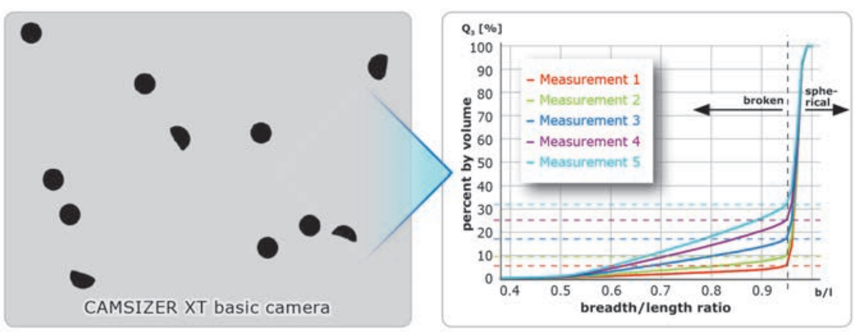


*Figure 4. Measuring catalyst materials with the CAMSIZER XT*

## Every Grain Counts: Detection of Agglomerates in the Production of Pharmaceutical Pellets

The production of pharmaceutical pellets is typically done by granulation, extrusion with subsequent spheronisation or coating. The desired result is a narrow and homogeneous particle size distribution of round particles. In the granulation and coating processes, the formation of agglomerates is an unwanted side effect. Agglomerates can have a negative impact on product properties; they can lead, to changes in the release rate of the active ingredients. Therefore, the amount of agglomerates is usually strictly controlled for each product batch. The CAMSIZER is able to detect percentages of agglomerates as low as 0.05%. Neither laser diffraction nor sieve analysis are suitable methods to reliably determination of such small percentages. Particle shape is also an important factor in this context.

Elongated particles, for example, can't be detected by either laser diffraction or sieve analysis.

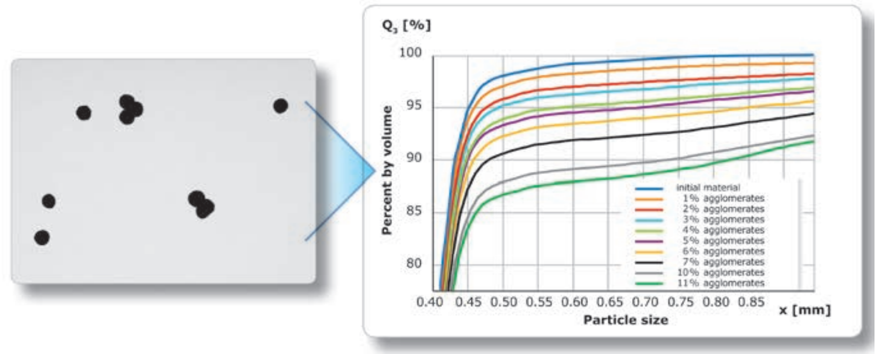


*Figure 5. The graphic shows the detection of oversized agglomerates by the CAMSIZER. Approx. 0.21 g of agglomerates, which is equal to about 10 particles, have been repeatedly added to a sample of 21 g to achieve mixtures in the range of 1% to 11%. The percentage of oversized particles at 0.7 mm exactly matches the calculated mixed percentages.*

## CAMSIZER XT: Dispersion Options and Modular Sample Feeding

The CAMSIZER XT is an advancement of the well-proven optical measurement system CAMSIZER for finer samples. In addition to the newly developed optics with a higher resolution it also features enhanced options for sample feeding. Fine particles tend to agglomerate which makes it difficult to detect the geometric dimensions of each individual particle. It is therefore beneficial to have various sample introduction options, to establish for each material, the best way to separate agglomerates without destroying the primary particles.

The CAMSIZER XT provides flexible solutions: The free fall X-fall cartridge, which provides the gentlest dispersion method for the material; the X-Jet air pressure dispersion cartridge with adjustable pressure and variable nozzle geometry; and finally, the X-Flow module in which particles are dispersed in liquids, optionally by an ultrasonic probe.

## Conclusion

Instead of 'calculating' particle size distributions from indirect size measurements, the DIA method provides precise, direct information from each particle about its length, width and shape. In addition to the calculated and averaged numbers for the size distribution, the images of the particles are available to view the sample size and shape, like in a microscope.

Dynamic Image Analysis provides faster and more accurate information than the established methods and is therefore ideally suited for particle characterisation in research and development and quality control typical sample analysis times are between 1 and 3 minutes.