



Particle Size and Particle Shape Analysis with Dynamic Image Analysis

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HORIBA

Precise Measurement of Particle Size and Shape with the CAMSIZER® X2

The quality control of fine powders can be substantially improved with the CAMSIZER X2. More precise and faster analysis of particle size and particle shape helps to improve product quality, reduce rejected lots and save on costs.

The CAMSIZER X2 is an advancement of the wellproven optical measurement system CAMSIZER for finer samples. The basic differences include higher resolution of the optical systems and improved options for the sample feeding. Fine particles tend to agglomerate which makes it difficult to detect the geometric dimensions of the primary particles. It is therefore beneficial to have various possibilities of feeding the sample into the measurement area. Thus it is possible to find the best way of dispersing agglomerates without destroying the primary particles for each material.

Here, the CAMSIZER X2 provides flexible solutions: from the free fall module, which is the gentlest method for feeding the material, to air pressure dispersion with adjustable pressure and variable nozzle geometry. Additionally, there is a wet module in which particles are dispersed in various liquids, with optional ultrasonic dispersion.



Key Features

- Extremely high resolution (0.8 µm/pixel)
- Measuring range from 0.8 µm to 8 mm without hardware adjustment
- Broad dynamic measurement range for broad distributions or multi-modal samples
- Reliable detection of smallest populations (< 0.1 %) of "oversized or undersized" particles
- Particle shape analysis (e.g. for the detection of agglomerates, broken particles, contaminations or rogue particles)
- Innovative optical design provides a resolution of 4.2 megapixel per camera

Ideal for:

- Pharmaceutical powders, granules and fine pellets
- Pulverized and granulated food
- Detergent powder and chemical raw materials
- Plastic powders (even with an electrostatic charge on the surface)
- Metal powders for additive manufacturing, MIM, solder powders
- Abrasives (medium-size & small grit)
- Fine sands & construction materials
- Fine plastic, glass, and carbon fibers

New Optical System

Bright light sources, fast cameras with high resolution, and particularly the new optical split view concept make the CAMSIZER X2 a superior analyzer. The patented split view optical design arranges the optical paths of the two cameras perpendicular to the direction of movement of the dispersed particles. The monochromatic light beams of the two pulsed LED light sources are focused and collimated to illuminate the detection area of each camera respectively (Basic and Zoom). Both light beams are combined with a prism so that they illuminate their respective, overlapping detection areas from the same direction which is orthogonal to the particle beam.

This new optical design provides an optimized illumination area, field of view, pulse length and pulse frequency for each camera. After the light beams have passed through the analysis zone, they are separated by a dichroic prism to be individually magnified and detected by one camera for each beam. With more than 300 images per second and 4.2 megapixel per image, the CAMSIZER X2 manages a 3.5 times higher data rate than the XT model while the software is capable of processing all images in real time. The new split view optical design is another highlight of the patented dual camera principle which is used in all CAMSIZER systems. One camera is optimized to analyze the small particles with high resolution in a small field of view. The other camera detects the big particles in a large field of view, providing excellent statistics.

High Precision Reference Object

Recalibrating the CAMSIZER X2 takes a matter of seconds with the help of a highly precise ($\pm 0.1 \mu m$) reticle, made by electron beam lithography which contains differently sized particles. Thus, the requirements of instrument calibration are fulfilled.

CAMSIZER® X2 Results

Evaluation and Documentation

HORIBA supplies the CAMSIZER X2 with a powerful, process-oriented control and evaluation software which offers flexible data export to the customer's LIMS. A major advantage is the evaluation of the results in real-time. Graphical representation of the results is available while the measurement is still in progress. At the same time the measurement process can be checked visually by observing the digital images. All particle images and parameters are evaluated directly during measurement and are also saved by the software module "Particle X-Plorer" in a database. Immediately after the measurement is finished, the results can be displayed in various report forms. By using the particle database, it is possible to clarify particular issues in detail at a later date, and even to recalculate the results.

Simple and Reliable Operation

The user can easily select individual measurement and evaluation parameters and save product-specific settings. These are known as standard operating procedures (SOP), and they simplify the change between different repetitive measuring tasks. The SOP can be protected against corruption through password protection. This ensures that the same instrument settings and output formats are always used with the highest degree of reliability and effectively eliminates operator induced errors.

A Wealth of Information

To meet individual user requirements, the measurement results are displayed as graphs, tables, characteristics, or particle images. Results obtained can be presented graphically and in tabular form as size fractions, frequency distribution or cumulative distribution. In addition, the CAMSIZER X2 software allows the presentation of daily reports, trend analyses, mean value calculations and much more. A clearly structured, individually configurable measuring protocol based on international standards is produced for each analysis. It is possible to store product specifications with regards to particle size and shape. If the measurement result deviates from these specifications, for example roundness of the sample does not match the specifications, the software automatically produces a warning.

Quality control during measurement Comparison of the measurement result with upper and lower specification limits

Trend analysis of production processes Up to 4 selectable parameters of the sample material can be continuously monitored

Particle Shape Applications

Depending on the application, the CAMSIZER X2 measures various areas, perimeters, and lengths of the particle projections, determining up to 50 different parameters per particle. Typical results obtained include:

- Chord length
- Straight length
- Feret diameter
- Martin diameter
- Aspect ratio (width/length)
- Convexity
- Roundness
- Symmetry
- Transparency
- Angularity

NEW: Particle X-Plorer with 3D Cloud

The new structure of the CAMSIZER X2 software not only allows for data evaluation in real-time but also for the storage of large amounts of image data in the Particle X-Plorer database. Particle images with noteworthy characteristics may be extracted and displayed at a later time. Similar image libraries are commonly used for documentation purposes in static image analysis (microscopy). This method allows for measurement of only a small number of particles. The CAMSIZER X2 particle database, however, handles millions of single particle images for each measurement.

3D Cloud: Graphic display of the 3 analysis parameters size, transparency, and aspect ratio for a mixture of road marking materials consisting of anti-skid aggregates and glass beads.

Particle size [mm]

In addition to the well-known two-dimensional display of results, the CAMSIZER X2 software supports three-dimensional graphics, i. e. three different parameters being displayed on the same graphic (3D cloud). The cloud makes it possible to detect differences between samples which are not visible in a two-dimensional display. Moreover, particle groups with specific characteristics may be identified and separately evaluated. Thus, it is possible to recalculate, for example, the size distribution of all round and transparent particles in a sample.

Electrodeposited copper

Water-atomized copper

All particle images and corresponding measurement parameters can be optionally stored in the Particle X-Plorer database. The entire measurement is additionally displayed in a diagram (right).

Modular Design for Optimized Measurement Conditions

The CAMSIZER X2's modular "X-Change" system offers three alternative dispersion methods, permitting the selection of the optimum method for each sample type:

- Air pressure dispersion
- Gravity dispersion
- Wet dispersion

The modules and plug-in cartridges are easily exchanged in one minute allowing for rapid switching between dispersion methods.

Sieve Analysis, Laser Diffraction, Static, or Dynamic Image Analysis?

A comparison of measurement techniques

| Performance Features | CAMSIZER X2 Dynamic Image Analysis | Sieve Analysis | Laser Diffraction | opt. Microscope Static Image Analysis |
|--|---|-------------------|----------------------|--|
| Wide dynamic measurement range | ++ | + | ++ | |
| Reproducibility and repeatability | ++ | + | | 6 |
| High resolution for narrow distributions | ++ | | | ++ |
| Particle shape analysis | ++ | | - | ++ |
| Direct measurement technique | ++ | ++ | | ++ |
| Compatibility of results with other techniques | \mathbf{e} | | 6 | e |
| Reliable detection of oversized grains | \bullet | ++ | | 6 |
| Robust hardware, easy operation for routine analysis | ++ | +++ | ++ | - |
| Analysis of individual particles | \bullet | | | ++ |
| High measurement speed, short measurement times | ++ | | + + | 6 |

Air Pressure Dispersion with "X-Jet"

The dispersion, that means the deagglomeration of the particles prior to passing through the measurement area, can be a crucial precondition for a proper measurement of the particles. Thanks to the flexible pressure adjustments of the "X-Jet" plug-in cartridge each material can be measured under optimum conditions.

Small particles have a relatively large surface area which increases the effects of adhesion forces or electrostatic charges, and can lead to aggregation of the particles. When passing through the nozzle, the sample material is dragged along by the compressed air and submitted to shear forces which break up agglomerates. These shear forces are enhanced by increasing pressure so that it is possible to separate even those particles which adhere tightly to each other. Too much pressure may be counter-productive: the shear forces may destroy the primary particles thus "grinding" the particles inside the nozzle.

Measuring range 0.8 μm – 5 mm

An optional cyclone is available for sample recovery. Although the air pressure dispersion accelerates the particles to up to 50 m/sec, the patented dual camera system ensures that wide particle size distributions, as well as narrow, monomodal samples below 10 microns, are analyzed accurately.

With the dynamic image analysis method, it is possible to detect agglomerates or broken particles by analyzing the particle shape quantitatively or simply by looking at individual particle images. If comminution of the particles is happening, it will be necessary to adjust the pressure. Other measurement methods like laser diffraction use similar dispersion principles but are not capable of providing information on particle shape. The sample is collected in a vacuum cleaner after the measurement.

Gravity Dispersion with "X-Fall"

Flowable, unagglomerated samples can be analyzed by using the "X-Fall" plug-in cartridge. In this mode, the particles fall from a chute through the field of view of the two cameras accelerated only by gravity. Thanks to the comparably low speed of the particles, the large field of view and the high frame rate, the detection efficiency is very high, even for large particles up to 8 mm. Only a few coarser particles in the sample are sufficient for reliable, reproducible detection. After the measurement, the sample materials fall into a collection box and are available for additional analyses without loss or contamination.

Benefits at a glance

- Ideal for free flowing materials
- Complete sample recovery
- Gentle, contact-free measurement

Wet Dispersion with the "X-Flow" Module

The wet module "X-Flow" analyzes samples in the range from 0.8 μ m to 1 mm in suspensions or emulsions. An advantage of this module is the small sample volume which is required. A low particle concentration in the dispersion medium, for example, 20 mg/l is already sufficient. It provides enough particles for a reproducible result in only 1 minute. Therefore, this module is highly suitable for applications involving pharmaceuticals or explosives. Here only a limited amount of sample material is available or advisable for safe operation. It can also be used for other "moist" samples including food and sand.

The measurement range of the "X-Flow" module starts at 0.8 μ m. The CAMSIZER X2 also analyzes particles up to 1 mm without difficulty, provided they are kept suspended in the dispersion medium. Depending on the maximum particle size in the sample, measurement cells of up to 4 mm may be used. Agglomerates can be further dispersed by an integrated ultrasonic probe.

For applications with organic solvents as the dispersion medium, the X-Flow module is equipped with a circulation system consisting of PTFE components and solvent-resistant seals.

Benefits at a glance

- Perfect for emulsions and suspensions
- Ideally suited for small sample volumes
- Resistant to organic solvents
- Integrated ultrasonic probe

LED

Key Features

Wide Dynamic Measuring Range

Coffee powder: The CAMSIZER X2 provides meaningful statistics which is particularly important for the detection of a small number of oversized particles. The graphic shows the comparison between three ground coffee samples which were ground quite differently. The red curve represents the narrow size distribution of a homogeneous sample while the green curve shows a much larger amount of over and undersized grains. The blue curve is a mixture, showing a significant number of coarse particles larger than 1 mm. An excessive amount of fines in the coffee powder could result in blocked filters. Too many coarse particles release less flavor into the beverage which makes the coffee taste less strong and aromatic.

Outstanding Repeatability and Reproducibility

Metal powder: Even the smallest amounts of undersized or oversized particles are reliably detected, regardless of the measurement time. Moreover, the shape of small particles, for example the aspect ratio of particles smaller than 10 microns, is analyzed more precisely thanks to the high resolution of the CAMSIZER X2.

The example shows a fine metal powder with a size distribution from 5 μ m to 20 μ m like it is used, for example, in additive manufacturing, as solder powder or in other powder metallurgical manufacturing processes.

Particle Shape Analysis

Catalysts: Non-spherical (broken) particles can be detected by looking at parameters such as aspect ratio (breadth divided by length, b/l) or symmetry. The graphic shows a series of measurements with increasing amounts of broken particles during a feeding process, leading to an increase of the broken fraction in the aspect ratio diagram.

The initial material is almost spherical with a b/l ratio of more than 0.95 (red curve). The increasing fraction of broken particles can be derived directly from the graph at the threshold value 0.95.

Size Results 100% Compatible with Sieve Analysis

Fluidized bed granules: Sieve analysis results may be compared directly with the data provided by CAMSIZER X2. The example shows the measurement of a granular mineral which is used as an additive to animal food. Thanks to the match between CAMSIZER X2 and sieve analysis results, product specifications can be directly compared even if they have been measured with different analytical techniques.

Comparison to laser diffraction and sieve analysis

Granulated beverage powder: Dynamic image analysis simultaneously determines the particle size distribution with regards to width (red), length (green) and mean diameter (blue). The orange line represents the result of a measurement carried out with a laser diffraction analyzer. This corresponds roughly with the mean diameter obtained by dynamic image analysis.

The laser diffraction curve shows a broader distribution. For the large particles around 0.5 mm the laser diffraction measurement shows oversized particles not visible in sieve analysis results. Measurement of particle width is identical to sieve analysis. Laser diffraction measurements are based on the assumption that all particles are spherical. Therefore, it is not possible to differenciate parameters such as "diameter" and "length". The curves shown are typical for samples containing non-round or longish particles.

Reliable detection of oversized particles

PMMA micro beads: The CAMSIZER X2 features the latest camera technology with high frame rates and excellent resolution to capture as many particles as possible in the shortest time. The graphic shows the comparison of two samples with different amounts of oversized particles. Sample 2 (red) contains more oversized particles at 20 microns than sample 1 (green). The CAMSIZER's detection efficiency for small amounts of oversized particles is 500 times higher than that of any laser particle analyzer.

Dry measurement of small particles

Micro-grit abrasive: The X-Jet module is designed for the reproducible and exact measurement of fine powders. The example shows a sample of a SiC micro-grit abrasive at the lower-end of the CAMSIZER X2 measuring range. The total size distribution lies between 1 μ m and 12 μ m. Thanks to a high depth of field, pulsed, ultra-strong light sources and short exposure times, even the rapidly flowing micron-sized particles are accurately detected. The exactly defined fineness of the grinding material ensures an optimum combination of abrasion efficiency and surface roughness.

Analysis of individual particles

Catalyst spheres: With the CAMSIZER X2 software, it is also possible to store and process images of individual particles. With one mouse click on the particle of interest all properties of this particle with respect to size and shape are displayed. Thus, the user can develop an intuitive understanding of the results. This is especially important for method development to visually verify threshold values determined from the curves. As an example, the software can detect agglomerated particles and uses them for the calculations or can also exclude them from the results.

| Technical Data CAMSIZER [®] X2 | | | | | |
|--|--|--|--|--|--|
| | Module "X-Dry" with | 10 µm to 8 mm | | | |
| Measuring ranges | "X-Fall" plug-in cartridge | | | | |
| | Module "X-Dry" with "X-Jet" plug-in cartridge | 0.8 µm to 5 mm | | | |
| | Module "X-Flow" | 0.8 µm to 1 mm | | | |
| Measurement principle | Dynamic Digital Image Analysis (ISO 13322-2) | | | | |
| Measurement time | approx. 1 to 3 min. (depends on required measurement statistics) | | | | |
| Number of cameras | 2 (measuring simultaneously) | | | | |
| Sample volume | <20 mg – 500 g (depends on sample type and measurement mode) | | | | |
| Measurement speed | >300 images/sec. | | | | |
| Size of analysis area | approx. 20 x 20 mm | | | | |
| Resolution | 0.8 µm per Pixel | | | | |
| | Particle size: Smallest diameter, length, mean diameter etc. | | | | |
| parameters | Particle shape: Aspect ratio (breadth to length), symmetry, sphericity, convexity etc. (ISO 9276-6) | | | | |
| Instrument data | Dimensions (H x W x D) | approx. 580 x 850 x 570 mm | | | |
| | Weight (without PC) | approx. 50 kg | | | |
| | Compressed air supply | approx. 6 - 8 bar | | | |
| | Compressed air consumption | approx. 25 - 140 l/min (depending on dispersion pressure selected) | | | |
| The CAMSIZER X2 is CE-certified and complies with the relevant guidelines and standards. The vacuum cleaner is included in the delivery scope of the X-Dry module. | | | | | |
| Options | Cyclone 21 CFR Part 11-compliant software IQ/OQ-documentation according to GLP/GMP | | | | |
| Accessories | Different hoppers and chute materials allow for adaptation of the CAMSIZER X2 to the requirements of the particular sample material, depending on flowability and sample volume | | | | |
| | | | | | |

CAMSIZER® X2 at a glance

The CAMSIZER X2 analyzes the particle size and shape of fine powders, emulsions and suspensions with particle sizes of 0.8 µm and higher with excellent repeatability.

Easy operation, short measurement times and flexible dispersion options for agglomerated particles are standard. This allows for routine use not only in R&D but also in quality control laboratories with high sample throughput.

Compared to other particle size analysis methods, such as laser diffraction, the CAMSIZER X2 is characterized by its direct image processing measurement principle. This allows for analysis of representative sample volumes, even with wide size distributions in a very short time.

The direct principle ensures a better understanding of the sample quality thanks to additional information (length, width, mean particle size and particle shape) and also a 500 times greater detection efficiency, e.g. for small quantities of oversized particles.

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