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PARTICLE CHARACTERIZATION

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TOP 10 ERRORS IN PARTICLE ANALYSIS AND HOW TO AVOID THEM

Particle analysis is an integral part of the quality control of bulk materials and is routinely performed in numerous laboratories. The methods used have often been established for years and are hardly ever questioned. Nevertheless, the procedure should be critically reviewed from time to time because a whole series of sources of error can negatively influence the results of a particle analysis. This article is intended to provide food for thought to make methods for particle characterization more reliable and accurate.

1. SAMPLING

When sampling inhomogeneous bulk materials, it must be ensured that the properties of the laboratory sample taken correspond to those of the total quantity. In this case, one speaks of representative sampling. This can be complicated by the fact that materials separate by size during handling (segregation). During transport, for example, small particles move down the interstitial spaces due to vibration and collect at the bottom of the container. In bulk cones, one usually observes a concentration of the small particles inside the cone. Therefore, sampling at a single location can hardly be representative. Subsamples are frequently taken from several locations and mixed together to counteract the effect of segregation. Suitable aids such as sampling lances can further improve the situation.

2. SAMPLE DIVISION

The sample amount available for particle analysis is usually too large for the measuring instruments used. In many cases, the quantity must be further reduced in the laboratory. Poor or unperformed sample division is one of the main sources of error in particle analysis, especially for materials with wide size distributions. Random sampling produces subsamples with different particle distributions, which can be seen from the poor reproducibility of the measurement results (Fig. 1, left). The use of sample dividers can remedy this situation. Even a simple sample splitter leads to significantly improved reproducibility when several subsamples are analyzed. The best dividing results are achieved by automatic rotating sample dividers such as the Retsch PT 100 (Fig. 1, right). The sample material used is a standard sand with a particle size between 63 µm and 4000 µm. The blue and black * indicate the reference values.

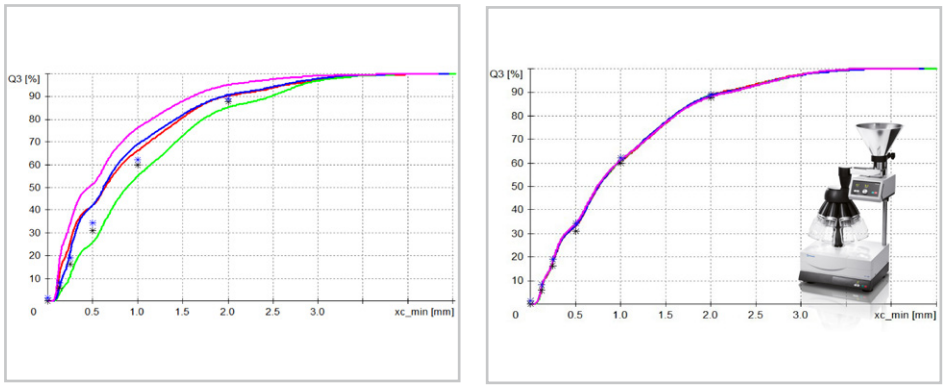


*Fig. 1 (left): Random sampling. Four measurements with the CAMSIZER P4 image analyzer (red / blue / violet / green) provide four different results. None is within the expected range (black and blue *).*
Fig. 1 (right): Sample division with rotating sample divider provides four identical and correct results.

3. DISPERSION

Dispersion is the separation of particles to make them accessible to measurement. Particles that stick together due to different attracting forces are called agglomerates. It is usually desirable to break up these agglomerates before measurement. However, it may also be of interest to create agglomerates in a targeted manner (granulation). In this case, care should be taken with dispersion so as not to destroy the structures to be measured. For dry measurements, dispersion is usually done in a compressed air stream. Figure 5 shows an example of dry measurements with the CAMSIZER X2 at different dispersion pressures. In the first example (Fig. 3a), as the pressure increases, the result becomes finer and finer until it stabilizes at 150kPa and above. 150kPa would therefore be the optimum dispersion pressure for this sample. In general, "as much as necessary and as little as possible" applies when selecting the dispersion pressure. For most powdered materials, 20-30 kPa is already sufficient for complete dispersion. In the second measurement example (Fig. 3b), the dispersion becomes increasingly finer from a pressure of 100 kPa which suggests that the particles are ground. Pourable samples can even be analyzed in free fall.

Agglomerates can also occur in suspensions. This can often be prevented by selecting a suitable dispersing medium (carrier fluid). Agglomerates that are still present in the suspension can be broken up by using ultrasound. Most modern particle sizers have powerful ultrasonic probes built in, so that sample preparation is done entirely inside the instrument.
In general, the larger the particles, the higher the probability of error in sampling and sample splitting. With finer particles, the susceptibility to error is more likely to occur during dispersion.

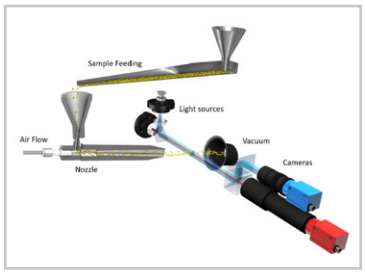
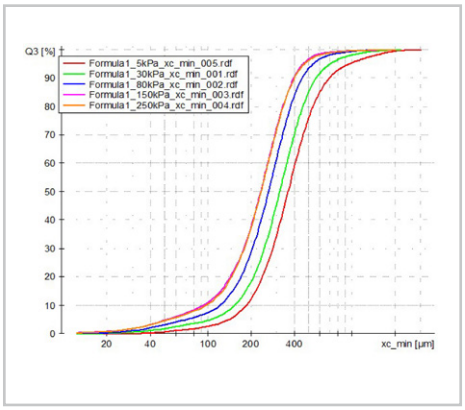


Fig. 2: The dry dispersion module of the CAMSIZER X2



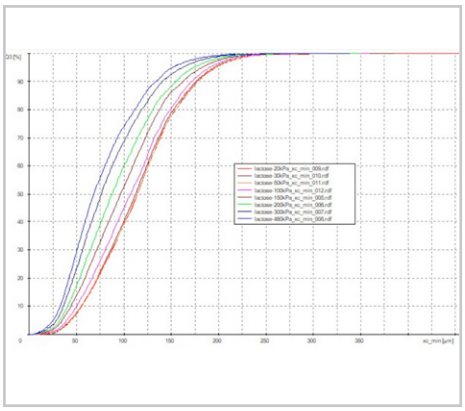


Fig. 3a: The result becomes finer with increasing pressure. 5 kPa (red), 30 kPa (green), 80 kPa (blue), 150 kPa (violet) and 250 kPa (orange). No change can be detected from 150 kPa to 250 kPa. Sample: milk powder.
Fig. 3b: Measurements at 20 to 50 kPa yield identical results, from 100 kPa the result becomes finer, indicating progressive destruction of the particles. 20kPa (red), 30 kPa (brown), 50 kPa (orange), 100kPa (violet), 100 kPa (purple), 150 kPa (gray), 200 kPa (green), 300 kPa (dark green) and 460 kPa (blue).

4. SIZE DEFINITION

Strictly speaking, particle size is only unambiguously defined for spherical structures, namely as the diameter of this very sphere. For non-spherical particles, different measured values can be obtained, depending on the orientation and the measuring technique used. In the example in Fig. 4, the sphere and Lego brick fit through a 16 mm sieve, while they are retained by a 14 mm sieve. For sieve analysis, both objects are the same size, they have the same "equivalent diameter" of 14-16 mm, it is not possible to be more precise with sieve analysis. When measuring with the caliper, smaller or larger values are obtained, depending on the orientation.

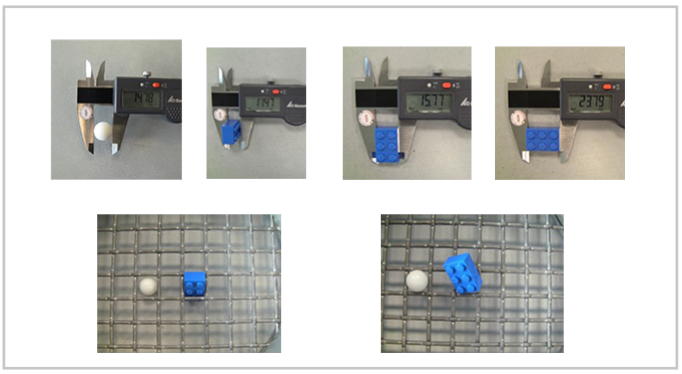


Fig. 4: Particle size also depends on the shape and the measuring equipment used!

Even more advanced particle measurement methods use different "size models". In sieve analysis, particles ideally orient themselves so that their smallest projected area fits through the smallest possible mesh. Sieve analysis thus tends to determine particle width. In imaging techniques (e.g. as used by CAMSIZER), different size definitions are accessible. Size distributions can be reported separately for length and width.

In laser diffraction, all diffraction signals are evaluated as if they were generated by ideally spherical model particles. In contrast to image analysis, the particle shape cannot be determined. Furthermore, laser diffraction evaluates a signal generated by a particle collective with particles of different sizes. Calculation of the size distribution is therefore indirect. Nevertheless, laser diffraction is an established technique thanks to its great versatility and wide measurement range from a few nanometers to the low millimeter range. Fig. 5 shows the

result of the size measurement of a sample of coffee powder with sieving, CAMSIZER image analysis and laser diffraction.

From the considerations described above, it follows that different methods for particle measurement inevitably produce different results. While laser diffraction and sieve analysis are difficult to correlate, the results of sieve analysis and image analysis are often very close, since imaging techniques can determine particle width and sieve analysis tends to be a width measurement.

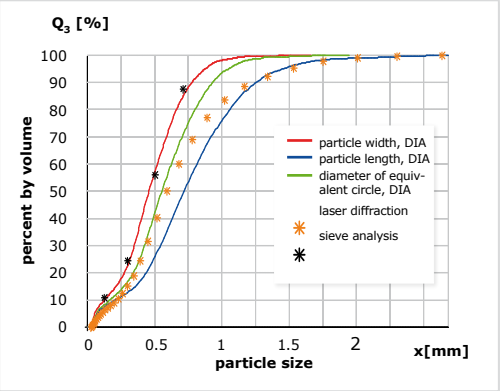


*Fig. 5: Particle size distributions of a sample of coffee powder determined with sieve analysis (black *), laser diffraction (orange *) and dynamic image analysis. Image analysis provides three results based on particle width (red), particle length (blue) or circle equivalent diameter (green). The definition "width" fits well with sieve analysis, laser diffraction tends to correspond to circle equivalent diameter.*

5. INCORRECT SAMPLE AMOUNT

Using too much or too little material can negatively influence the measurement result. In laser diffraction, too high a particle concentration can lead to multiple scattering, and if too little sample is used, the signal-to-noise ratio is poor. However, modern laser analyzers indicate the ideal concentration for measurement and warn users as soon as the amount is too high or too low. In image analysis, you can't actually use too much sample. If too little sample is analyzed, the result will be unreliable and poorly repeatable due to the small number of detections. Since the required amount of particle detections depends on the size of the particles and even more on the distribution width, it is difficult to make general recommendations here. Repeatability tests are helpful, especially looking at the "rough end" of the distribution. Repeatability can be improved by using more sample. In dynamic image analysis with CAMSIZER instruments, enough particles are detected in 2-5 minutes under normal conditions to obtain a reliable measurement result.

The strongest influence of sample quantity is in sieve analysis: one of the most common errors here are overloaded sieves. If too much sample volume is used, particles can get stuck in the meshes and block them. Small particles then no longer fall through the blocked sieve and the measured size distribution is "too coarse".
In sieve analysis, the sample weight must be adjusted to the particle size, the sieve stack used and the density. The simple way of always using 100 grams usually leads to a dead end, because 100 grams can be too much or too little. In no case is a representative sample division given when weighing 100 g.

6. UNDERESTIMATING TOLERANCES

Every measuring instrument shows certain systematic uncertainties and tolerances which must be taken into account when interpreting the results. This will be illustrated here using the example of sieve analysis. Test sieves made of wire cloth are manufactured according to the standards DIN ISO 3310-1 or ASTM E11. These standards specify how the real mesh size of each sieve is to be tested. Each test sieve is inspected by an optical method before delivery and a specified number of meshes are measured. The mean value of the measured opening width must be within prescribed tolerances around the nominal mesh size. For a sieve of nominal mesh size 500 µm, the mean value of the real mesh size must lie within an interval of +/- 16.2 µm. A sieve conforming to the standard can therefore have an average opening width of 483.8 µm to 516.2 µm. It is important to note that these are average values; some openings can be even larger and thus allow particles of a corresponding size to pass through the sieve. Therefore, the standard also defines the maximum permissible aperture size for each sieve size. Calibration certificates are available for each sieve which contain information on the real mesh sizes and their statistical distribution.

7. OVERESTIMATING SENSITIVITY

A frequent issue in particle analysis is the detection of oversize particles, i. e. a small amount of particles that are larger than major part of the distribution. Here, the sensitivity of the measurement method plays a decisive role. Imaging methods offer the advantage that each particle detected represents a "measurement incident" and is thus also shown in the result. This means, for example, that the CAMSIZER X2 can detect oversize particle contents of less than 0.02 %. Laser diffraction is a "collective measurement method", i. e. a scattered light signal is evaluated that is generated by all particles simultaneously. The contributions of the individual particle sizes are superimposed and an iterative procedure is used to calculate the size distribution. If the amount of oversize particles is small, the contribution of these particles is not sufficient (signal/noise ratio) to show up in the result. For a reliable detection of oversize particles with laser diffraction, the contribution should be >2 %. Microtrac's SYNC laser diffraction analyzer offers much better detection capabilities for oversize particles, as the SYNC has a built-in camera that detects oversize particles with a high probability of detection.

8. WRONG DENSITY DISTRIBUTION

Particle size distributions can be represented graphically in several ways, with the particle size always on the x-axis. Intuitively easy to access is the histogram representation, where the bar width corresponds to the lower and upper limit of the measurement class and the height corresponds to the amount of particles in the respective size interval. These size intervals are often determined by the performance and resolution of the measurement system used. While a sieve stack of 8 sieves results in 9 size classes (the sieve bottom counts), image analyzers provide several thousand measurement classes, and laser diffraction analyzers 64-150 classes, depending on the detector configuration. More information content is provided here by the cumulative curve, which shows the summation of the quantities in each measurement class. This produces a curve which rises continuously from 0% to 100%. For each x-value (size), the quantity of particles smaller than x can be read from the cumulative curve. In addition, the cumulative curve directly shows percentiles, such as the d50 value (median).

Popular with many users is the representation as distribution density, often succinctly and incorrectly referred to as a "Gaussian curve". The distribution density is the first derivative of the cumulative curve. Where the cumulative curve rises steeply, the density distribution has a maximum; where the cumulative curve is flat, the density distribution has a minimum. It is important here that a true density distribution shows the slope of the cumulative curve. Thus, the quantity in the measurement class must be divided by the class width. The accuracy of the density distribution increases with the number of measurement classes. The procedure of connecting the bars of the histogram by a "balancing curve" does not provide a den-

sity distribution. Due to the low information content and the error-proneness of the density distribution, it should be dispensed with in favor of a cumulative distribution.

9. TYPES OF DISTRIBUTION (NUMBER, VOLUME, INTENSITY)

Particle analysis results are usually given as a percentage, either as a percentage per measurement class or as a proportion greater or smaller than a certain size x. However, these percentages can have very different meanings. It makes a huge difference whether these values refer to mass, volume or number. Which type of distribution is present depends strongly on the measuring system used. In sieve analysis, the weights of the sample in each fraction are determined by backweighing and are then converted into mass percentages. These are identical to a volume-based distribution, provided there are no density differences between particles of different sizes. Other methods, such as hand measurement with a caliper, provide number-based distributions based on the number of particles in each measurement class. The difference between number-based and mass/volume-based distributions lies in the fact that for volume distributions, large particles have a stronger weighting, while for number distributions, small particles are weighted stronger.

Laser diffraction relates all signals to a sphere of equal effect and thus provides volume-based distributions. Since a collective signal and not individual incidents are evaluated here, laser diffraction cannot determine number distributions. The situation is different for single particle measurement methods, such as image analysis. Here, the measurement data are primarily distributed based on number. While microscopic methods (static image analysis) often work with number distributions, it is common practice in dynamic image analysis to convert to volume distributions. Since image analysis covers different size definitions, it is reliably possible to carry out this conversion with a suitable volume model (usually a prolate rotational ellipsoid). This makes image analysis data comparable to sieve data or laser diffraction. Converting laser diffraction results to number distributions is also possible, but since only a simple spherical model is available here, this is less accurate, and the volume distribution should be used whenever possible.

Dynamic light scattering represents a special case. Here particle sizes are weighted according to their contribution to the total scattering intensity. This leads to large particles being very strongly represented in the result, because the scattering intensity increases with size by a factor of 106, which means that a 100 nm particle scatters a million times more photons than a 10 nm particle. In DLS, it is common to convert distributions to "volume-based", but care must be taken when interpreting the results to determine which distribution type was used.

5 mm **10 mm** **15 mm** **40 mm**

Size	Weight	P_3	Number	P_0
5 mm	190 g	25%	490	85,5%
10 mm	190 g	25%	64	11,2%
15 mm	190 g	25%	18	3,1%
40 mm	190 g	25%	1	0,2%
Gesamt	760 g	100%	573	100%

Fig. 6/Table 1: Difference between number- and mass-based distribution using the example of four different grinding ball sizes. In the volume- or mass-related distribution (p3), all fractions are present in equal proportions at 25%. Since the number decreases with increasing particle size, the number-related proportions (p0) are higher in those of the small grinding balls.

10. WORKING WITHOUT SOPS

As with all other analytical methods, a uniform, standardized procedure is also a prerequisite for consistent and meaningful measurement results in particle measurement. Such Standard Operating Procedures (SOPs) always guarantee the same, defined measurement processes and work steps. A prerequisite is that all instrument settings are stored by the software and can be retrieved. However, an SOP comprises more than just instrument settings. Specifications for sampling, sample division, sample preparation and evaluation should also be precisely specified here. It is advisable to create work instructions that are as precise as possible to guarantee consistent quality of the measurement results.

CONCLUSION

Various methods are used for particle analysis, the most common being laser diffraction, dynamic image analysis and sieve analysis. Successful analysis and meaningful results can only be achieved if preparatory steps such as sampling, sample division and sample preparation are carried out correctly. The selection of the appropriate method for the sample material and a meaningful evaluation of the measurement data finally lead to a successful particle analysis.

Microtrac MRB is one of the leading suppliers of particle measurement technology from the fields of laser diffraction and dynamic light scattering as well as static and dynamic image analysis and offers the complete portfolio for particle characterization from a single source.

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