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Correlation between sieve analysis and image analysis made easy

Dynamic Image Analysis (DIA) has become a widely used method for routine analysis of particle size and particle shape in many industries. In this white paper we explain how traditional sieve analysis can successfully be replaced by DIA. The results produced by both techniques can be made to match so that product specifications based on sieve analysis remain unchanged. Users of image analysis benefit from reduced workload, higher sample throughput and more detailed results.

Sieve analysis is still a standard method for routine determination of particle size distributions of powders and granulates. On the one hand, it is cost-efficient and apparently easily executed, but on the other hand, it is also prone to error and flawed by various inaccuracies. The time required for one analysis may add up to 15 -30 minutes, including weighing, sieving process and cleaning. The information gained is limited as the number of data points is defined by the number of available test sieves. In contrast, DIA delivers a high-resolution measurement result within 2 - 3 minutes which contains additional information on particle shape and is generated automatically.

Not surprisingly, some systematic differences can be observed in the results generated by different measurement techniques. In this article, these systematic differences are discussed for DIA and sieve analysis using application examples with various materials and particle shapes. Finally, we present possible solutions how to overcome these deviations and how to establish a robust and reliable correlation of DIA and sieve data.

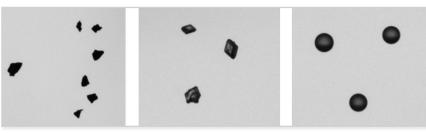


Measurement principles of DIA and sieve analysis

The measurement principle of dynamic image analysis is quite simple: a strong LED light source illuminates a flow of particles and a camera system captures the particle images as shadow projections. The images are transferred to a PC and a powerful evaluation software processes the data and generates size and shape distributions. Fig. 1 shows two state-of-the-art image analyzers, CAMSIZER P4 and CAMSIZER X2 from Retsch Technology which record and evaluate 60 or up to 320 images per second respectively. The result generated by these analyzers is based on the size and shape data from hundreds of thousands or even millions of individual particles (depending on particle size and sample amount).







CAMSIZER X2 (right) from Retsch Technology. The P4 model is suitable for fast analysis of pourable bulk solids in a size range from 20 µm to 30 mm. The X2 model is optimized for fine powders in a size range from 0.8 µm to 8 mm.

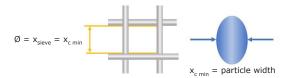
Fig 1 above: Two high-end DIA particle analyzers: CAMSIZER P4 (left) and

Fig. 1 below: Example images from CAMSIZER measurements: activated carbon (left), sugar crystals (middle), and expandable polystyrene beads (right).

An unambigious definition of particle size only exists for spherical particles. For all other shapes, the size may be derived from different physical dimensions. Fig. 2 shows different size definitions for the 2D projection of an angular particle: width $(x_{c\, min},$ smallest chord), length $(x_{Fe\, max},$ maximum length), and equal area diameter (x_{area}) . Depending on which definition is selected, different results are achieved. Each distribution is correct but provides information on different sample properties. Users who wish to correlate image analysis with sieve results will use the size definition $x_{c\, min}$ because particles will pass a sieve mesh preferentially with their smallest projection area, which corresponds to their width (Fig 2).

 Q_3 [%] 100 90 "width" 80 "diameter of length percent by volume 70 equivalent circle 60 50 40 30 20 10 0 500 1,000 1,500 2,000 2,500 3,000 3,500 4,000 $x[\mu m]$ particle size

Fig. 2: Size definitions used in dynamic image analysis. The parameter $x_{\rm c \, min}$ (particle width) provides best correlation with sieve results.



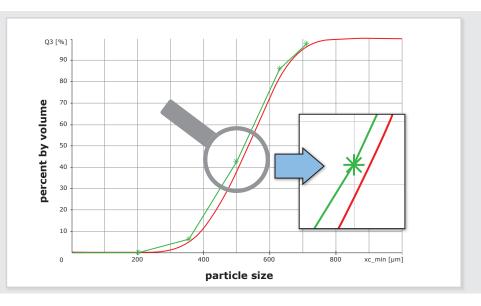
DIA and sieve analysis of spherical particles

Spherical particles are obviously the simplest example for the comparison of different sizing techniques: there is only one possible particle diameter, regardless of its orientation and one would not expect any notable deviations. Examples for relatively round particles are pellets generated in granulation and coating processes, glass beads, EPS particles, but also fine metal powders. The measurement example in Fig. 3, however, shows a divergence between the CAMSIZER result (size definition $\mathbf{x}_{\text{c min}}$) and the sieve result: the latter is apparently finer.

To understand this, it is necessary to examine the test sieves more closely. Each analytical test sieve is manufactured and inspected according to the standard ISO 3310-1. This standard defines how much the real apertures of a test sieve may deviate from the nominal aperture size. Mean aperture size and standard deviation are examined individually for both directions of the wire mesh (warp and weft) as well as the maximum allowed opening. The resulting tolerance for a test sieve with 500 μm nominal aperture size is $\pm 16.2~\mu m$ for the mean real aperture and the maximum allowed aperture is 580.5 $\mu m!$ As a consequence, every ISO 3310-1 compliant test sieve will usually feature a significant number of openings larger than the nominal size, even if the mean aperture is close to the nominal size. Thus, large particles, that should be retained, can pass the sieve and will be classified as finer than they really are. Hence, the sieve result is finer than that of CAMSIZER analysis which determines the accurate size of the spherical particles. The magnitude of this deviation depends on how much the individual sieves deviate from the nominal size.

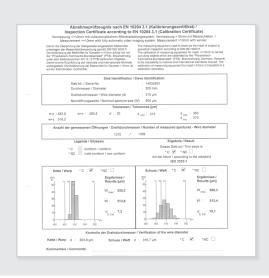


Fig. 3 Sieve analysis (green *) and CAMSIZER P4 result (red) for round particles, cumulative distribution Q_3 . The offset is only a few micrometers and well within the tolerance of the test sieves. As the distribution is very narrow (sharp increase of the Q_3 curve), a small deviation in size of only 15 μ m results in a large difference in Q_3 of almost 5%.



This information can be obtained from the calibration certificate of each test sieve which is available on request from the manufacturer (Fig. 4). To achieve correlation between DIA and sieve analysis, it is recommended to consider the real aperture size from the calibration certificate. Alternatively, a constant factor can be established for each size to compensate the effect of sieve tolerance. It is, however, a common observation that sieve results change notably when one test sieve is exchanged for another one of the same nominal size. As a consequence, the correlation factor to DIA is only valid if the sieves are not changed.

Fig. 4 Excerpt from a calibration certificate of a 500 μm analytical test sieve. The mean real aperture is 513.8 μm and 513.4 μm respectively. The maximum aperture is 558.3 μm and 530.3 μm respectively. This sieve complies with ISO 3310-1 but allows round particles as big as 530 μm to pass.



DIA and sieve analysis of non-spherical particles

Some systematic differences between DIA and sieve analysis arise from the shape of the particles.

Angular Particles

Sieve analysis determines the edge length of the cubes and is therefore a measurement technique that determines particle size in a preferred orientation. During the sieving process the particles have many opportunities to compare themselves with different apertures in different orientations. For cubical-shaped model particles it can be observed that during the sieving process they will pass the smallest possible aperture with their smallest projection area (Fig. 5). This is not the case with DIA where particles are captured in absolutely random orientations.

For some of these random 2D-orientations, $x_{c \, min}$ yields the same numerical result as sieve analysis (e.g. with the cube face pointing towards the camera). In many cases, the $x_{c \, min}$ of the random 2D particle projection will produce a larger numerical value than sieve analysis. The largest possible value is reached when the corner of the cube is pointing towards the camera. Then, the 2D projection is a hexagon with an $x_{c \, min}$ equal to the edge length (d) times square root of two:

$$x_{c min} = d \cdot \sqrt{2}$$

The DIA system can measure a cube up to 1.414 times larger than sieve analysis, but never smaller!

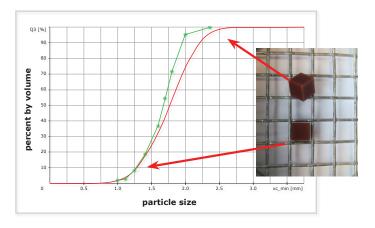


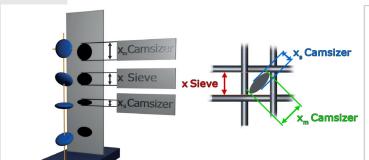
Fig. 5 Sieve analysis (green *) and CAMSIZER P4 result (red) for angular, approximately cubicalshaped particles. Cumulative distributions Q₃.

For this reason, the correlation between DIA and sieve analysis for real angular particles is usually good for the fine fraction of the distribution because here the small projections are recorded; in the coarse fraction the comparability is worse as this represents the larger projection areas.

Flat, flaky and lenticular particles

Flaky or lenticular particles will also pass the smallest possible aperture with their smallest projection area. They will, however, orientate diagonally in the square holes of the sieve, so that the numerical value obtained from sieving is a value between the thickness and the diameter of the particle (Fig.6). In random orientation, the measured $\mathbf{x}_{\text{c min}}$ value lies between thickness and diameter of the particle, i. e. the result can be larger or smaller than that obtained by sieving. For real samples this will cause the cumulative curves to intersect, which is very typical for flat particles. The distribution measured by image analysis will always be wider than the distribution obtained from sieve analysis.





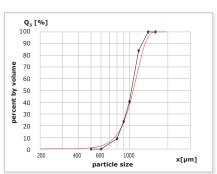


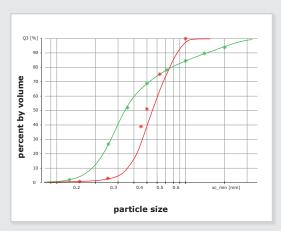
Fig. 6 Sieve analysis (black *) and CAMSIZER P4 result (red) for flattened particles, cumulative distribution Q₃. Particles are passing the apertures diagonally, the CAMSIZER can measure a smaller or larger size depending on the orientation of the particle during detection. The resulting distribution is therefore wider than the sieve analysis result.

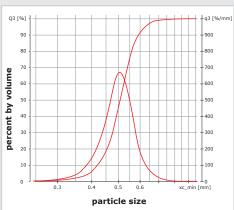
Influence of distribution width and sieve correlation

From the above observations it is clear that a simple "shape factor" that will shift the entire distribution by a constant factor, can never be sufficient to achieve complete compliance between sieve analysis and DIA. A more promising approach is using different factors that depend on the ${\bf Q}_3$ value. This method is applicable for all samples with the same shape and width of distribution. If the distribution width is changing, however, this correlation technique will fail as well as the deviations between DIA and sieving also change with the distribution width. Consider a sample of lenticular particles as in Fig. 6. A real sample will contain lenses of different sizes and due to orientation effects, some of the small ones will be detected as "too large", and some of the large ones will be detected as "too small" by the image analyzer. For a wide distribution with lenses of many different sizes, these deviations will cancel each other out and the overall correlation will become very good. On the other hand, if all lenses have identical thickness and diameter, the systematic differences between the two techniques become apparent. Thus, for narrow distribution, a stronger correlation factor is required than for wide distributions.

In practice, users who deal with wide distributions often don't need to apply any correlation mechanism. For all other cases, it is possible to achieve a robust correlation that is valid for all particles with a particular shape but is independent of width of distribution. The idea is to let the image analyzer measure a test sample of a specific material where the deviation from sieving is highest, and that would be a very narrow distribution. From this result the algorithm will learn the fundamental difference for this material, independent from distribution width and can apply this to any other sample with similar particle shape. This training sample is obtained by sieve analysis; the narrower the fraction, the better the correlation algorithm (e. g. 600 μm – 630 μm or 1.12 mm – 1.18 mm, this will produce particles that are all the same size in terms of sieving).







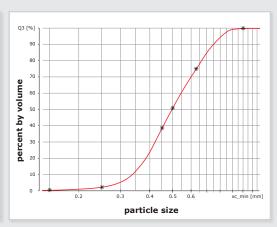
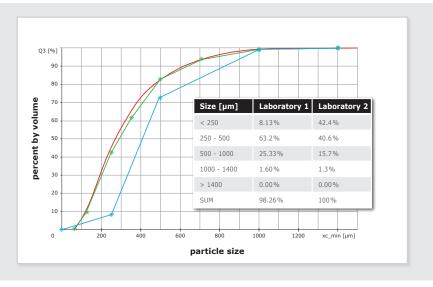


Fig. 7 leftz: Size analysis of two sand samples, one with a wide distribution (Sample 1, green) and one with a narrow distribution (Sample 2, red). CAMSIZER P4 results and corresponding sieve data as *. The wide distribution is in much better agreement with sieving, the red curve shows the typical deviations. Middle: CAMSIZER P4 analysis of the fraction 450 μ m – 500 μ m of sample 2. This fraction is suitable as a training sample to find a correlation function. If this correlation is then applied to the measurement result of sample 2, DIA and sieve analysis match perfectly (right).

Fig. 8: Example of a faulty sieve analysis. Results of a fine sand sample: CAMSIZER P4 (red). Sieve results from two different laboratories: Lab 1 (blue *), Lab 2 (green *). The result of Lab 1 is significantly coarser than those of Lab 2 and of DIA. Too much initial sample weight leads to overloading of the 250 μm and 500 µm sieves and small particles don't have the opportunity to pass. Furthermore, the fractions do not add up to 100% for the analysis of Lab 1 (loss of sample!). The sieve result of Lab 2 is correct and in good agreement with DIA.



With the procedure described above, the user of a CAMSIZER system can find a robust sieve correlation in three simple steps: 1. CAMSIZER analysis, 2. sieve analysis, 3. CAMSIZER analysis of a narrow fraction. The basic requirement for successful sieve correlation is, however, that the sieve data is correct. It is not possible to match DIA data with faulty sieve analysis results, so always make sure that the test sieves used are clean and in good condition. Damaged or worn out sieves must be replaced. The sieving process has to be long enough to give all particles the opportunity to pass, i. e. the sieving has to be continued until the mass of sample on any sieve does not change with prolonged sieving time. Another frequent error in sieve analysis is overloading. If too much sample is placed on one sieve, apertures will be blocked, preventing small particles from passing. The smaller the particle size, the less sample is allowed.



Some users always take 100 g of sample because then gram equals percent and the calculation is much easier. For many fine samples 100 g is already too much, plus there is always the danger of making a sampling error when a particular mass is used. It is better to reduce the sample amount with a sample splitter and use one aliquot for the analysis.

Conclusion

Dynamic Image Analysis is a highly precise and reliable technique for the characterization of particle size and shape of bulk solids. Compared to traditional sieve analysis it offers a reduction of workload and higher throughput plus much additional information on the material at hand. Thanks to sophisticated material-specific correlation functions that can easily be established by the user it is possible to achieve results that match sieve analysis very accurately and reliably. When interpreting the results, the limitations and inaccuracies of sieve analysis have to be considered.

CAMSIZER Dual Camera Technology

