APPLICATION NOTE

MICROTRAC MRB

Using Dynamic Image Analysis as an Alternative to Sieving for the Quality Control of Sugar

Instruments: CAMSIZER P4 / CAMSIZER X2

Sieve Analysis of Sugar

Sieve analysis according to ICUMSA Method GS2/9-37 is the standard for particle sizing of crystal sugar. Officially, all labs follow these guidelines, and comparable results should be seen in round robin tests. On a closer look however, each lab follows procedures that deviate slightly from those outlined in the ICUMSA Method. As a result, the size distributions measured by different labs vary remarkably. In this article we will discuss the typical deviations that can be found in the daily operation and propose a new analysis method which provides more reproducible and reliable results. The typical deviations from the perfect sieving process may be divided into three sections.

- Systematic errors
- Hardware related errors
- Operating Errors

Table 1: Examples for the maximum batch and permitted sieve oversize for 200 mm ø sieves

Mesh size in µm	Max. batch in cm^3	Max. permitted sieve oversize in cm^3
25	14	7
45	20	10
63	26	13
125	38	19
250	58	29
500	88	44
1000	126	63
2000	220	110
4000	346	173
8000	566	283

Systematic Errors

- Contaminated sieves: When the mesh wires are coated with sugar, the effective mesh size becomes smaller than the original mesh size of the sieve fabric. The proposed cleaning method recommended by ICUMSA using warm water is time-consuming (drying process), thus not applied very often. As a result, the effective mesh width becomes smaller with time if the sieve is not cleaned appropriately.
- Damaged sieves: Sometimes sieves are damaged, for example by brushing. The quick and harsh brushing procedure, which is applied instead of cleaning in warm water, results in the effective mesh size becoming too large. It is therefore strongly recommended to clean sieves with mesh sizes <500 µm not with a brush or hair pencil, but with warm water or in an ultrasonic bath.
- Inefficient sample splitting: Many labs use exactly 100 g for sieve analysis, so that no percent calculation is required. On the one hand, it is impossible to split a sample in a representative way so that the total sample is exactly 100 g. Thus the sample aliquot used for size analysis is not representative, and due to segregation effects, the size distribution of the 100 g might not be identical to the size distribution of the total bulk sample that was provided for the measurement.
- Overloading of sieves: On the other hand, a batch of 100 g of sample can overload the sieves for smaller crystal sizes. Compare DIN 66165 for the maximum amount of material allowed on each sieve after analysis (Table 1). With 100 g of sample the required sieving time to achieve a complete separation will be extremely long (10 to 15 minutes). The proposed sample amount of 80 to 100 g is only recommended for some medium size distributions. For large-sized sugar products (e. g. rock candy) the number of particles will not be sufficient, 100 g just means a few particles and the result is therefore inconclusive. For small-sized sugar products (fractions below 500 µm), the amount of 100 g is usually too much and thus could block the sieve mesh.
- Inadequate sieving time (5 minutes or less): Users want to keep the analysis time as short as possible which frequently leads to inadequate sieving times. As a result, the fractions are not completely separated.
- Reduced/insufficient number of sieves: According to the ICUMSA Method, not more than 30% of the total sample should be retained on any sieve. However, many labs are mainly interested in oversized and undersized fractions, so they reduce the number of sieves in order to be able to handle the sieve stack quickly and save time. As a result, for example 45% of sample material may be retained on the “undersize” sieve, which is then completely overloaded (Fig. 1).

Figure 1 compares an incorrectly performed sieve measurement with a result provided by the optical particle analyzer CAMSIZER® based on Dynamic Image Analysis (ISO 13322-2). Due to insufficient sieving the data point for the 630 µm sieve shows incomplete separation of remaining coarse and passing fine particles.

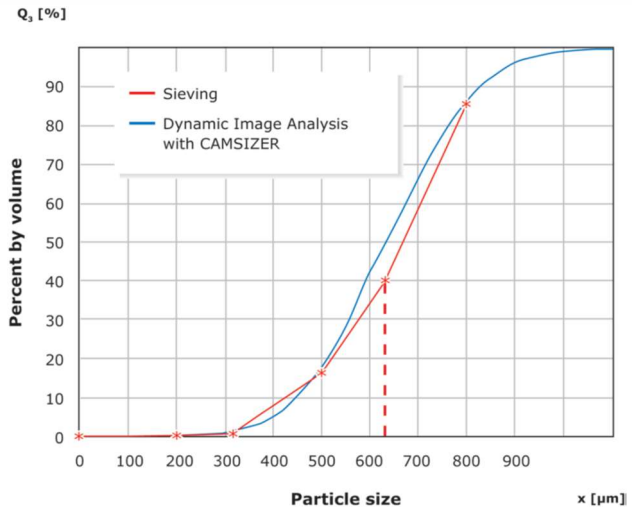


Fig. 1: *Comparison of an incorrectly performed sieve measurement (* red asterisks) with a result provided by the optical particle analyzer CAMSIZER® based on Dynamic Image Analysis (blue curve).*

More than 45% of the total sample is retained on this sieve. By reducing the sample amount (50 g instead of 150 g, as used in this case), increasing the sieving time, or adding a 710 µm sieve between 630 µm and 800 µm this problem can be avoided.

Hardware-related issues

Beside the systematic errors related to the sample handling and sieving equipment, there are also issues related to instrumentation itself:

- Uncalibrated sieves.
- Uncalibrated sieve shakers: The amplitude of the sieve shaker has a strong influence on the result. If it is too low or too high this will result in a degraded sifting quality.
- Uncalibrated balances or balances with low resolution.
- Worn out or broken sieves (Fig. 2)

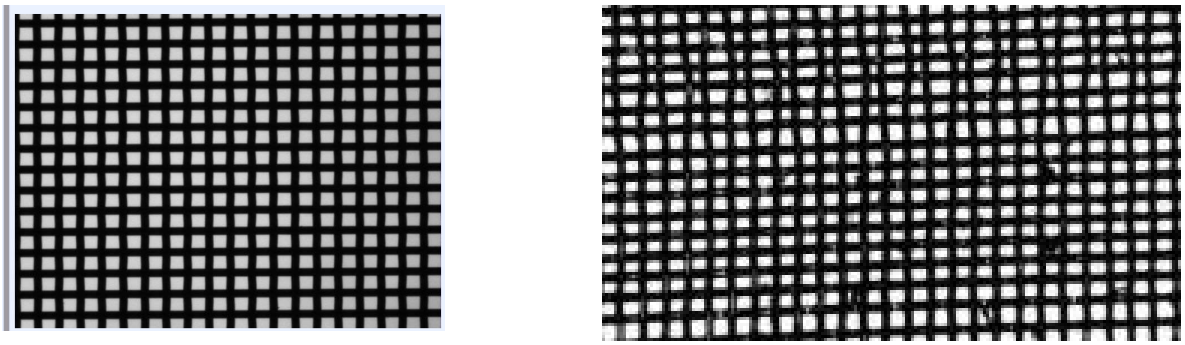


Fig 2: *New sieve (left) and worn-out sieve cloth (right). The apertures of the new test sieve are uniform and almost square. Sieves are optically measured (calibrated) before shipment to check whether they meet the requirements of ISO 3310-1 or ASTM E11.*
In the right screen fabric, the worn-out apertures allow particles larger than the nominal mesh size to pass.

Operating errors

Last but not least, a surprisingly high number of unsystematic, human mistakes might occur during the sieving process, such as mixing up numbers when writing down the mass of each fraction, calculating errors when converting mass in percentage, mixing up of sieves (smaller sieve on top of the larger one), or handling errors related to the weighing process (tare/ cleaning of balance). For more guidelines and tips related to the sieving process, see DIN 66165 (overloading of sieves), ISO 3310 (calibration of sieves), or the sieve analysis Expert Guide “Taking a close look at sieve analysis” published by Retsch GmbH (www.retsch.com).

Dynamic Image Analysis

Dynamic Image Analysis (DIA) is an established method in the measuring range 1 µm to 30 mm. This method is superior to sieve analysis in its resolution, precision and reproducibility and delivers additional detailed particle shape information. MICROTRAC offers two particle analyzers, the CAMSIZER P4 and the CAMSIZER X2 (Fig. 3), which both use Dynamic Image Analysis (DIA) to measure the size and shape of

particles in a range from 30 µm to 30 mm (CAMSIZER P4) and 0.8 µm to 8 mm (CAMSIZER X2). Together, the analyzers cover the complete size range of sugar products, from 3 µm icing sugar to 30 mm rock candy. In contrast to sieve analysis, the method of DIA allows to measure the crystal size distribution of sugar with better reproducibility, less manpower and less maintenance and "handling" issues. The results generated by both methods may however be identical, if the above mentioned issues are avoided. Thus the DIA can substitute the sieving process without the need to define new product specifications.

Fig. 3: *Microtrac dynamic image analysis instruments CAMSIZER P4 and CAMSIZER X2. The P4 model has a measurement range of 20 µm – 30 mm and is suitable for dry pourable material. The X2 model is optimized for finer particles from 0.8 µm to 8 mm and masures particle size in an air-flow. Both are suitable for sugar analysis.*

For many applications, the exact quantification of the amount of oversized and undersized material is as important as the analysis of the average particle size. The example in Figure 4 shows that DIA delivers identical results also in this respect. Due to the higher degree of automation of the DIA, the inherent possibilities to produce errors are strongly reduced. Pre-defined Standard Operating Procedures (SOPs) are executed by the computer, resulting in identical results independent of the operator. In addition, the DIA allows for a significant reduction of workload compared to sieve analysis. For example, the measurement time for 30 samples performed by sieve analysis amounts to approx. 7.5 h. With DIA, 30 samples can be measured in less than 1.5 h.

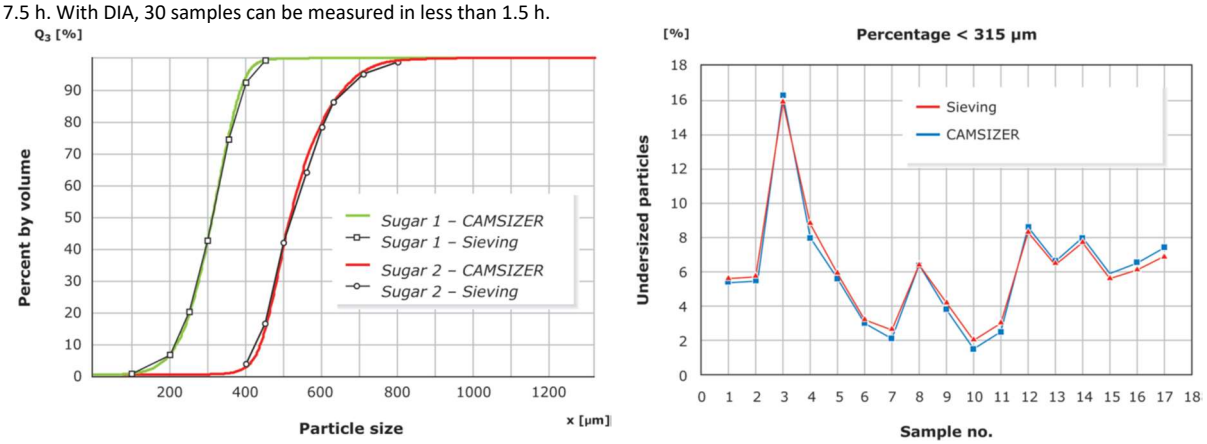
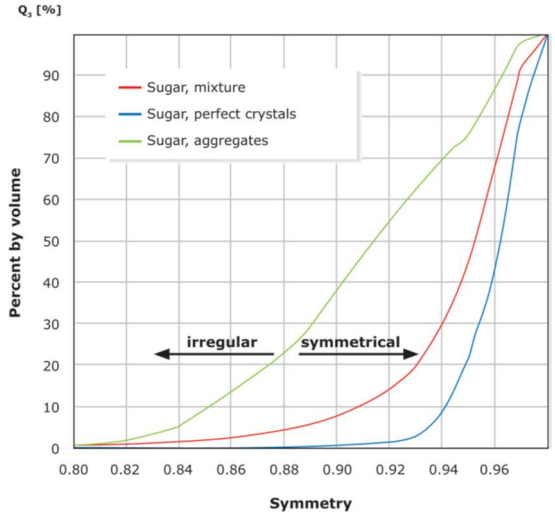


Fig. 4: *The CAMSIZER result correlate almost perfectly with data from correctly performed sieve analysis (left side). Thanks to short analysis time, a constant monitoring of product quality is possible. The right chart shows the percentage of particles < 315 µm in 17 consecutive measurements. The trend analysis shows how product quality is changing over time.*

Particle Shape Analysis

In addition to particle size analysis, the CAMSIZER P4 and CAMSIZER X2 also allow for measurement of the shape. For example, for monocrystalline sugar products, which are popular in Asia, the amount of single or poly crystals can be easily determined (Fig. 5).



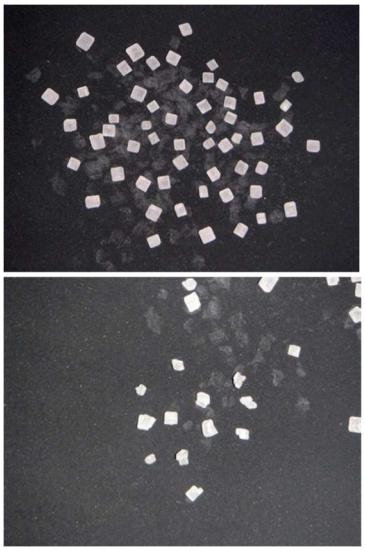


Fig. 5: *CAMSIZER shape analysis for three different sugar samples. The symmetry of the perfect crystals is high (blue curve, upper photo on the right). The aggregatres (green curve, lower photo) have lower symmetry, indicated by the curve being shifted to the left. A mixture of both samples (red curve) shows intermediate values.*

CAMSIZER benefits

In summary, the advantages of Dynamic Image Analysis are:

- Wide, dynamic measuring range;
- Excellent compatibility to sieve analysis acc. to ICUMSA;
- Reliable detection of smallest amounts of “undersize” and “oversize”;
- Dispersion options: air pressure, gravity, liquid;
- Short measurement times (1–3 minutes);
- Excellent reproducibility and repeatability;
- High resolution, especially for small size distributions;
- Significant reduction of workload compared to sieving;
- An AutoSampler is available for CAMSIZER P4 to further reduce workload (Fig. 6).

Fig. 6: *CAMSIZER P4 with AutoSampler. The Sampler aumatically feeds new sample to the instrument as soon as one analysis is finished. Measurement parameters are selected according to the barcodes on the beakers.*

For further information please contact us at:
www.microtrac.com