Dynamic Image Analysis of Powdered Milk (and other Spray Dried Materials)

Instrument: CAMSIZER X2

Introduction: granulation of particles by spray drying

Dairy products like milk, cream, or even yogurt can be evaporated to dryness in order to produce a powder which has increased shelf life and is easier to handle. These powders are then conveniently used to produce delicacies like chocolate, pastries, or ice cream. Powdered milk is produced by spray-drying which is a well-established method in food technology but also in the chemical or pharmaceutical industry.
For spray drying, the milk is first pasteurized, and then transferred to a spray tower where it is dispersed through tiny nozzles (atomizers) into 200°C hot air. The droplets immediately loose most of the residual water and the water content is reduced from 87,5% to 3 % in the process. The result is a fine powder. The particle size distribution usually lies in a range between 20 µm and 500 µm; its size affects properties like dissolution, reconstitution, or flowability and is therefore a quality relevant feature.
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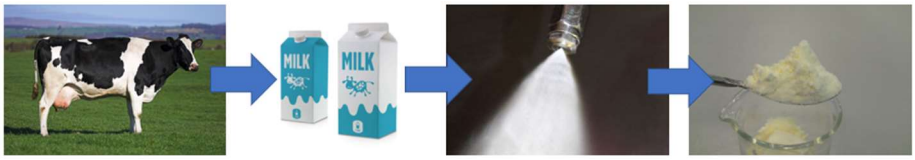


Fig. 1: *Powdered milk and cream are produced by spray drying. The powder agglomerates due to its high fat content.*

Advantages of Dynamic Image Analysis

Particle size analysis of powdered dairy products is a challenge! Sieving is not ideal as the particles readily form agglomerates due to the greasy nature of the material. Even air jet sieving does not always improve the situation as it may brake large particles and makes small particles form agglomerates. Since wet analysis is not an option either, effective dry dispersion is required. The dry dispersion system X-Jet of the CAMSIZER X2 features a Venturi nozzle with adjustable dispersion pressure. This should be selected carefully to ensure that the particles are effectively separated without breaking the filigree structures of the spray-dried powder. In a series of tests we ascertained that a rather low dispersion pressure of 30 kPa was sufficient.
Once proper dispersion is achieved, image evaluation with the CAMSIZER X2 is the optimum choice to measure the size distribution of the powdered milk. Particle images are captured as shadow projections and evaluated in real time. The measurement result is based on the examination of 300 images per second which equates to millions of individual particles in a 2-3 minutes analysis cycle. Length and width are determined simultaneously, along with various parameters defining particle geometry, like aspect ratio, sphericity, roundness and many more.

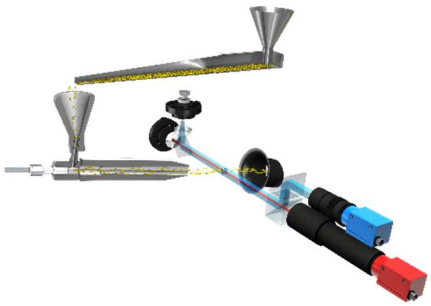


Fig. 2: *The X-Jet dry dispersion module of the CAMSIZER X2 guarantees effective, yet gentle dispersion*

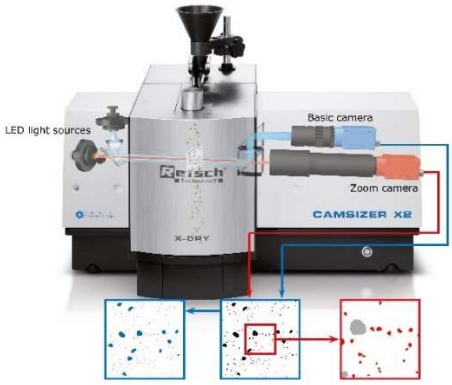


Fig. 3: *CAMSIZER X2 dynamic image analyser with dual camera technology*

Example 1: Powdered Milk

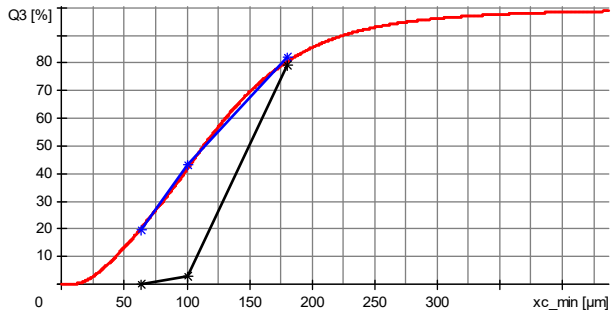


***Fig. 4**: Particle Size distribution of powdered milk. CAMSIZER X2 (**red**), air jet sieving (blue) and vibratory sieving (**black**).*

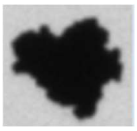
The particle size distribution of milk powder usually lies between 20 µm and 500 µm. Figure 4 shows the result of a CAMSIZER X2 measurement together with the corresponding sieve analysis results. CAMSIZER X2 and air jet sieving produce identical results. The milk powder has a relatively low fat content and is as easily deagglomerated by air jet sieving as by the X-Jet module of CAMSIZER X2. Consequently, the results are identical. Note that the result obtained by vibratory sieving is different. The 200 µm value is approximately the same for all three techniques, at 100 µm however, the percentage passing the sieve is much too low. The particles agglomerate on the sieve and block the apertures. Hence, vibratory sieving is not suitable for the analysis of powdered milk!

The CAMSIZER X2 result is based on the analysis of millions of individual particles. Images may be saved during the measurement and displayed in a particle database. Figure 5 shows some exemplary images together with the relevant size and shape parameters. The CAMSIZER X2 has two cameras with different magnifications (BASIC camera and ZOOM camera), simultaneously analysing large and small particles. Figure 5a shows BASIC images, figure 5b shows ZOOM camera images.

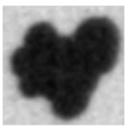
Width: 235 µm,
Length: 342 µm,
SPHT=0,600

Width: 276 µm,
Length: 332 µm,
SPHT=0,633

Width: 204 µm,
Length: 265 µm,
SPHT=0,612

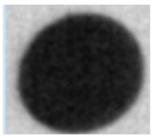


Width: 300 µm,
Length: 377 µm,
SPHT=0,613

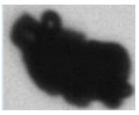


Width: 65 µm,
Length: 81 µm,
SPHT=0,723

Width: 84 µm,
Length: 124 µm,
SPHT=0,841



Width: 89 µm,
Length: 98 µm,
SPHT=0,960



Width: 51 µm,
Length: 96 µm,
SPHT=0,690

***Fig. 5a**: Powdered milk, large particles (BASIC Camera).* ***Fig. 5b**: Powdered milk, small particles (ZOOM Camera).*

Figure 5 clearly shows that the particle shape ranges from very round particles, representing the original morphology of the milk droplets, to very irregular structures. The parameter sphericity (SPHT) is displayed for every particle in Figure 6. SPHT is calculated from the ratio of area to perimeter of the particle projection. SPHT equals 1 for a perfect circle and is lower for particles with irregular shape. Fig. 6 shows the sphericity distribution of four samples of powdered milk.

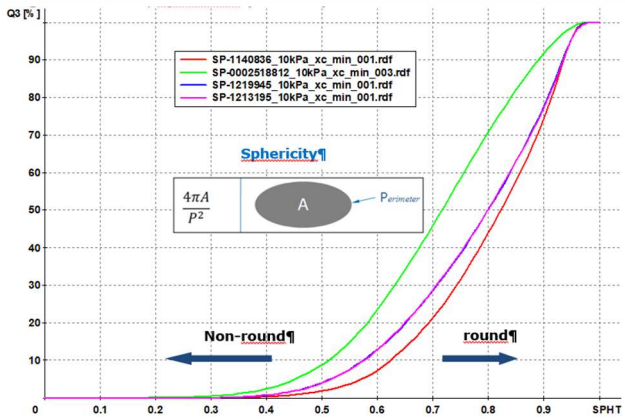


Fig. 6: *Particle shape of 4 milk powder samples. Q3 distribution of SPHT.*

Powdered Cream – Advantage CAMSIZER X2 over Sieve Analysis

The particle size distribution of powdered cream is especially difficult to obtain. Due to the high fat content, the product readily agglomerates. Vibratory sieving is not an option, as the particles do not separate from each other and the apertures of the test sieves get blocked very easily. Even air jet sieving, which is usually very effective in terms of deagglomeration, fails in this case. Figure 7 shows a 63 µm sieve with powdered cream sample after 1 minute of air jet sieving. The sample sticks to the lid and to the frame. Large agglomerates which have formed in the process remain on the test sieve. A meaningful analysis is not possible.

Fig. 7*: Powdered cream on a 63 µm sieve after one minute air jet sieving.*

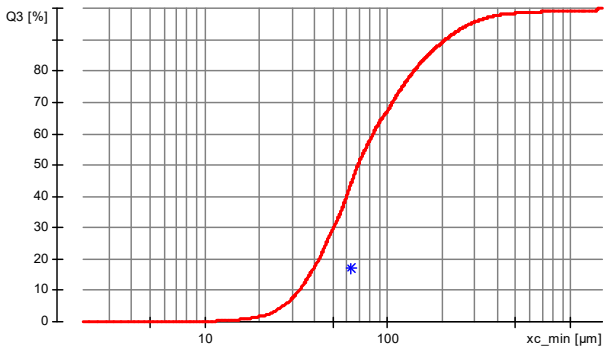


Fig. 8*: Particle size distribution of a powdered cream sample, measured with CAMSIZER X2 (**red**) and air jet sieving (**blue ***).*

The X-Jet dry dispersion unit of the CAMSIZER X2, however, is capable of deagglomerating the sample. The powder is constantly and homogeneously conveyed to the instrument with a vibratory feeder. Inside the dispersion nozzle particles are separated from each other, accelerated in the air flow and detected by the camera system in flight. The resulting size distribution is therefore reliable, meaningful and comprehensive (Fig. 8).

Summary

The CAMSIZER X2 is suitable for the particle size and shape analysis of spray dried powders. Even easily agglomerating materials like powdered cream can be effectively dispersed and measured within a few minutes. Compared to sieve analysis, the dispersion is more reliable and the dispersion pressure can be adjusted to the sample properties. Cleaning is reduced to a minimum.

CAMSIZER X2 -Benefits at a glance

- Analysis time 2-3 minutes
- Measuring range 0.8 µm – 8 mm (CAMSIZER X2)
- Automated analysis
- Wet and dry analysis
- Results comparable to sieve analysis
- Higher resolution than sieving or laser diffraction
- High sample throughput
- Excellent reproducibility
- Excellent statistics
- Objective, independent of operator
- Highest sensitivity for oversize and undersize particles
- Shape analysis: length and diameter of particles
- Low maintenance, robust design
- Full compliance with 21CFR part 11

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