How to measure size and shape of extrudates by Dynamic Image Analysis

Instrument: CAMSIZER P4

Extrudates

This application note is about the size and shape measurement of extrudates but can also be applied to the analysis of other elongated, cylindrical or rod-shaped particles.
Extrusion is a common process in many industries which allows the mass production of uniform bodies with a well-defined cross-section. This is achieved by squeezing a ductile mass through a die and cutting the string of material into pieces by rotating blades, followed by solidifying the particles, e. g. by cooling or sintering.
Examples for extruded materials are:

- Catalysts
- Ceramics
- Wood pellets
- Plastics
- Pharmaceuticals
- Foodstuff
- Animal Feed
- Abrasives

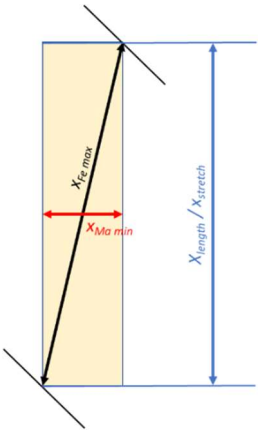
When examining the size of extrudates, the width is supposed to be uniform as it is determined by the die. In reality however, apertures may be partially blocked or particles may stick together. Shrinkage or expansion of the pieces after extrusion may also lead to undesired cross-sections of the extrudates.
The length distribution analysis is also required to judge the quality of the product. For example, catalysts are often used in reactors in the chemical or petrochemical industry. These “catalysts" consist of an extruded ceramic carrier with a metal coating which is the actual catalyst. The precise knowledge of the size distribution allows to optimize reaction rates, pressure and flow conditions. The ideal catalyst carrier provides a large active surface, enough interstitial space for proper mixing of the reactants and a high and evenly distributed permeability.
Particle analysis is required to give further information beyond the analysis of particle length. Automated image analysis systems like the CAMSIZER P4 accurately determine the amount of dust, broken, bended, fused or deformed particles as well as inaccurate cutting of the edges which indicates that the blades are wearing off.

***Fig. 1**: The process of extrusion (left) creates elongated particles: e. g. sugar sprinkles, catalyst carriers or animal feed (from left to right).*

Length and width definitions

Length and width are not measurable for many standard particle size analyzers, which usually only provide an “equivalent diameter”. For example, laser diffraction analysers, are relating all size data to the properties of a spherical model particle.
Also sieving is a less suitable method for characterizing elongated samples because the particles tend to align in a way that lets them pass an aperture with their smallest projection area, so sieving basically only measures the particle diameter. Particles with a low aspect ratio (width divided by length) however, are not likely to pass the sieve apertures vertically. Sometimes horizontal (planar) sieving is applied for elongated material, trying to prevent the particle from vertically passing the sieve and thus obtain information on the particle length. However, the actual movement of the particles during sieving is hardly predictable and so this method has not proven to be very reliable in practice. Furthermore, sieve analysis provides poor resolution due to the limitation of available sieve sizes.
Elongated particles, especially extrudates, are often measured one-by-one with a caliper. Although this is very accurate for individual pieces, the disadvantages of this method are obvious. Even well-trained personnel will need a lot of time to measure a small number of particles. This is not only tedious work; the result is also not meaningful because it is not based on enough data points to be representative for the

entire sample. Besides calipers, there is only one technique that provides accurate length measurement and that is image analysis. It provides direct width and length information based on different size definitions. Fig. 3 outlines definitions for particle width and length for 2-D projections of oriented elongated particles.



$$x_{length} = \sqrt{x_{Fe\ max}^2 - x_{Ma\ min}^2}$$

$$x_{stretch} = \frac{A}{x_{Ma\ min}}$$

$$A = Area$$

***Fig. 2**: Definitions of particle length and width*

The width of such particles is most conveniently defined as the smallest area bisector ($x_{Ma\ min}$, smallest Martin diameter). This will give the correct value, even if the extrudate is arcuate. Particle length may be defined as the maximum Feret diameter $x_{Fe\ max}$ which is the longest distance between two parallel lines touching the particle projection. This is a bigger value than the distance between the basal planes of a cylindrical particle, which is what a caliper measurement would yield. The length of extrudates is therefore most commonly described by the parameters x_{length} or $x_{stretch}$, which are obtained by applying Pythagoras' theorem respectively calculated from the particle area.

Further size definitions are available. The above-mentioned definitions represent only the most typical parameters.

Dynamic Image Analysis with the CAMSIZER P4

The CAMSIZER P4 dynamic digital image analyzer determines particle size and shape in a range from 20 µm to 30 mm. The particles under investigation are conveyed by a vibratory chute into the measurement zone where they pass in front of a planar light source in free fall. The resulting shadow projections are captured by a camera system and evaluated in real time. This allows the simultaneous measurement of the length and width distribution and of the particle shape. The huge advantage of this arrangement is the vast amount of sample that can be processed in only a few minutes. During measurement the particles are in random orientation, but for a correct measurement of the particle length, they must be detected in the ideal orientation with the longitudinal extension parallel to the focal plane.

Alignment of the particles for analysis in the CAMSIZER P4 can be achieved by using a motorized guidance sheet. This device forces the particles to pass through a gap between two metal brackets which aligns them into the desired orientation. One of these brackets is mobile and features flexible flaps to improve alignment and prevent blockage.

The operation of the motorized guidance sheet is demonstrated in a 3-minute video which can be found in the "downloads" section of our homepage www.retsch-technology.com.

This setup has proven to be very effective for alignment of elongated particles and the following measurement example will illustrate this.

***Fig. 3:** The CAMSIZER P4 dynamic image analyzer.*

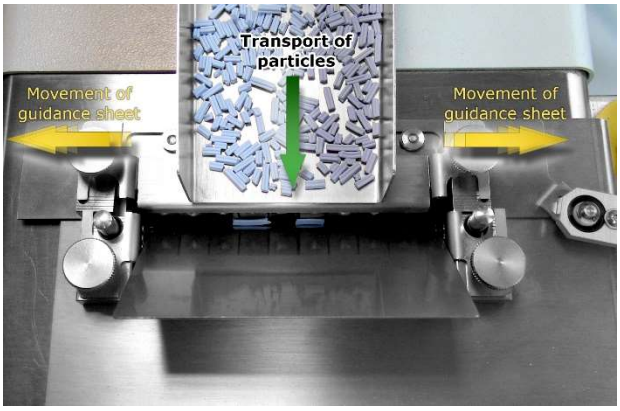


***Fig. 4:** Motorized guidance sheet for the CAMSIZER P4. Note that particles are aligned in the gap and enter the measurement zone in perfect orientation.*

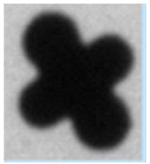
Measurement example: Extrudates

In this example we present the results of the particle size analysis of extruded catalyst support rods. The length distribution ranges from 2 mm to 14 mm, the width is 2 mm with a cross-shaped basal plate.

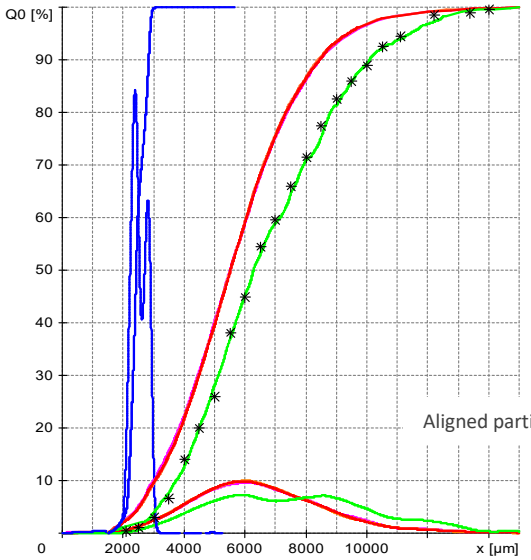
In free fall, the probability to measure the correct orientation is low. Even if one particle was captured several times, the probability to capture it at least once in the desired orientation would be too small. Only a proper mechanical alignment will lead to correct results. The motorized guidance sheet aligns the particles as they pass through the measurement zone of the CAMSIZER P4. This is proven by caliper measurement of 200 hand-picked particles and comparing this result to the CAMSIZER P4 analysis of exactly the same 200 particles. (Fig. 6). The two results are identical.

Fig. 5: *Extruded catalyst sample. Advantage CAMSIZER P4: 250 ml analyzed in 3 minutes, Caliper: 200 pieces in 1 hour*

Fig. 6 shows the CAMSIZER P4 measurement result for 250 ml catalyst extrudate. The analysis time was three minutes, whereas the manual measurement of the 200 pieces took about one hour. The CAMIZER P4 measurement is 20 times faster and evaluates 20 times more particles (Fig. 5), that means the overall gain is more than 400x!
Note that the average length of the 250ml sample is smaller than the average length of the 200 pieces, since operators tend to pick bigger particles which are easier to measure. The repeatability of the CAMIZER P4 results is excellent, the curves from three consecutive measurements overlap perfectly. The width distribution is slightly bimodal, because the two bars of the cross-shaped basal plane are different in size.



Cross-Section:
Bimodal width



Aligned particle: perfect length measurement

Fig. 6: *Three consecutive measurements of 250 ml catalyst extrudate with the CAMSIZER P4. Size definition is x_{length} (**red** /**orange** /**purple** - curves overlapping). The repeatability is excellent. Width measurement $x_{Ma\ min}$ (**blue**) is bimodal because the basal plate is cross-shaped with two bars of different size. CAMSIZER P4 length measurement of 200 pieces (**green**) is slightly larger than the entire sample due to sampling error. The result of the caliper measurement of these 200 pieces (**black** *) matches perfectly with CAMSIZER P4.*

An additional benefit of digital image analysis is that the particle shape can be determined. In this case, shape analysis allows to identify defects such as fused, broken, or curved particles. The percentage of defective particles can reliably be measured. Fig. 7 shows some examples of undesired extrudate particles.

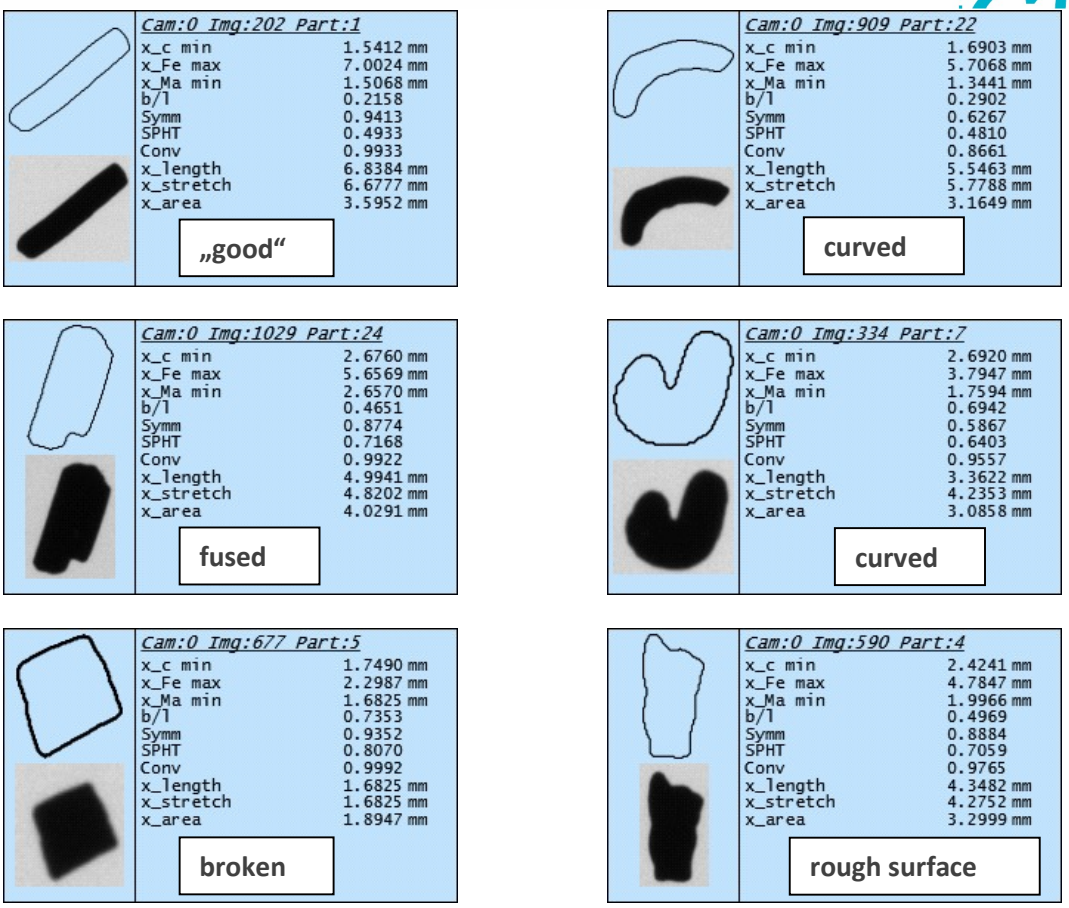


***Fig. 7**: Different kinds of defective extrudates are reliably identified by the CAMSIZER software.*

Summary

The CAMSIZER P4 is ideal for the measurement of elongated particles like extrudates, plastic pellets, wood chips or rice grains. Retsch Technology offers hardware solutions to achieve perfect orientation of the particles for the analysis. The results are perfectly comparable with caliper measurements but show better repeatability, less sampling errors and are obtained significantly faster and fully automatically.

CAMSIZER P4 -Benefits at a glance

- Analysis time 2-3 minutes
- Measuring range 20 µm – 30mm
- Automated analysis
- Results comparable to sieve analysis
- Results comparable to caliper measurement
- High sample throughput
- Excellent reproducibility
- Objective, independent of operator
- Shape analysis: length and diameter of particles
- Low maintenance, robust design
- Full compliance with 21CFR part 11

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