MICROTRAC
PARTICLE CHARACTERIZATION

THREE-DIMENSIONAL IMAGE ANALYSIS OF EXTRUDED CATALYST RODS

Context

In chemistry, catalysis refers to a process in which the reaction kinetics are influenced in such a way that reactions are accelerated, run in a certain direction or under special conditions. In the chemical industry, catalysts are used for many processes, as the energy required can be significantly reduced.

In heterogeneous catalysis, the catalyst and the substances involved in the chemical reaction are separate physical phases, usually in different states of aggregation. For technical purposes, the catalyst is usually in solid form, while the reaction partners are liquid or gaseous substances. During catalysis, some of the reaction partners involved briefly form a chemical bond with the surface of the catalyst, which facilitates or accelerates the reaction with other substances or allows it to take place at a lower energy level. In the process, the catalyst does not form permanent bonds with other substances involved, so that ideally it is not consumed. Important catalyst materials are zeolites, ceramics, or metal surfaces. The greater the surface area of the catalyst and its porosity, the more efficient the catalysis.

Many solid catalysts used in industry are in the form of pellets or granules for ease of handling. These may be spherical particles or extrudates. These extrudates are produced by forcing a pasty mass through a die, which can have holes of different sizes and shapes. The emerging strings are cut and harden in further processing steps, resulting in hard-brittle rod-shaped granulates. These are placed in suitable reactors through which the substances that are to participate in the catalytic reaction flow. Extruded catalysts are often used in the chemical industry, particularly in petrochemicals, to enable processes such as the production of fuels, plastics, and other materials. The extrusion process allows for precise shaping and high surface area, which improves the effectiveness of the catalyst.

Fig.1: Left: Production of extrudates; Right: Typical extruded catalysts

Size Measurement of extruded catalyst

The rod-shaped extruded catalysts considered here typically have a diameter of the base surface of 1-2 mm and a length of 2-6 mm. The size and shape of the base surface is predetermined by the die of the extruder and is relatively constant. Nevertheless, measuring the width of the catalysts is of interest because the dies wear out over time or can become clogged, which can be detected early by a change in the diameter of the granules. The base surfaces are rarely round; often they have a cross-shaped ("quadrilobes") or a three-lobed cross-section ("trilobes"). This serves to enlarge and optimize the surface. Even more important for assessing the quality of catalysts is the length distribution. A sieve analysis is out of the question here, because as soon as the granules straighten up a little, the sieve analysis determines the width and not the length of the particles. So all that remains is the laborious measurement of a few particles with a caliper, which is usually not representative of the total quantity due to the small sample size.

Static image analysis (so-called flat-bed scanners) is occasionally used. Here, the catalysts are spread out on a surface, photographed from above and ideally measured by software. However, the preparative effort here is very high and the number of particles is similarly low as in the measurement with a caliper.

The best alternative to the methods mentioned is measurement with dynamic image analysis.

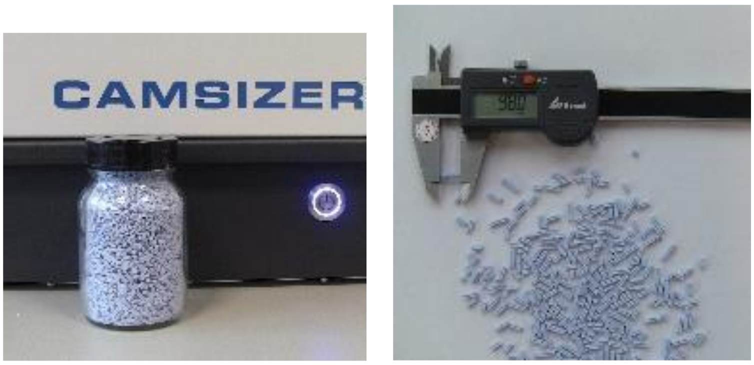


***Fig.2:** A sample of extruded catalysts. The CAMSIZER 3D image analyzer measures 250 ml in 3 minutes, manual measurement with the caliper analyzes approx. 200 particles per hour.*

In this particle characterization method, the sample falls between an area light source and a camera system so that the shadow projections of the individual particles are recorded. The measurement of these shadow projections provides a variety of size and shape values for each detected object. The orientation of the particles is random. For the elongated extrudates, this means that the width is easily measurable on all particles.
The situation is different when it comes to length measurement. Only if the longitudinal axis is exactly in the image plane, an exact length determination is possible. However, it is likely that most of the particles are oriented differently and therefore appear "too short". Efforts have therefore been made in the past to align elongated particles with suitable guiding devices ("motorised guidance sheet”) so that they show their true length when measured. This works sufficiently well for extrudates whose aspect ratio is less than 1:3, but for shorter rods, the reliability decreases. These guide devices had to be aligned very precisely on the measuring device, and mean a not inconsiderable maintenance.

Fig.3: *Top: CAMSIZER measuring principle; Bottom: alignment of particles with the "guidance sheet".*

The latest generation of particle measuring devices of the CAMSIZER series, the CAMSIZER 3D offers a completely new method for three-dimensional particle measurement. Each particle is recorded not just once, but up to thirty times as it moves past the camera system. In doing so, it shows different orientations (projections), of which the one with the greatest value for the particle length corresponds to the true length. The 3D measurement is superior to the 2D analysis and makes the guidance sheet obsolete.

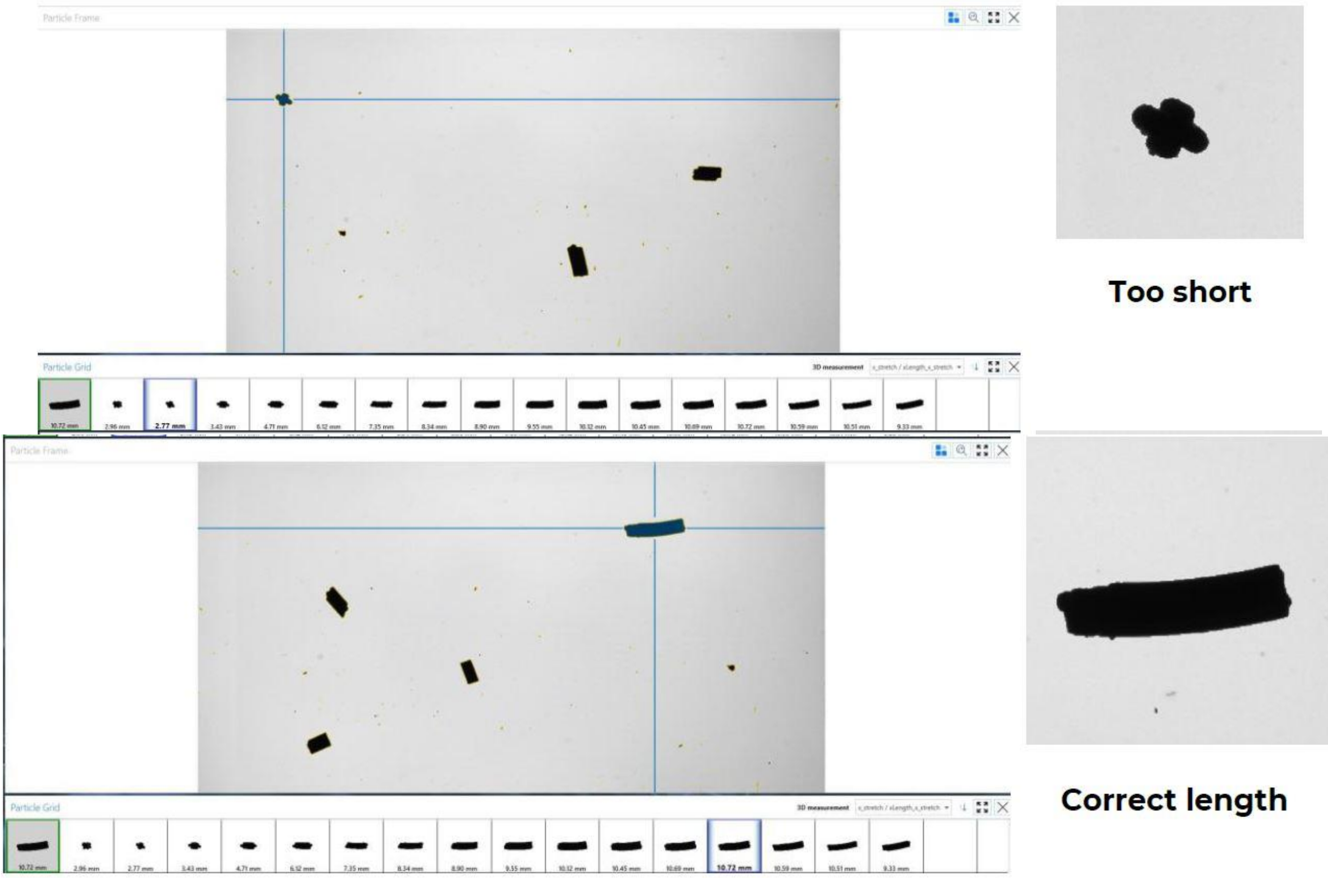


Fig. 4: *Three-dimensional analysis of extrudate particles. The direction of fall in the picture is from left to right. Several individual images are taken of each particle, which is displayed in the "3D track" below the overall image. The projection with the correct length is used.*

Difference between 2D and 3D measurement

In the following example, the length distribution of a sample of extruded catalysts was determined in three different ways using CAMSIZER 3D:

- Measurement in 2D mode, in which images are taken of each particle in random orientation.
- Measurement in 2D mode using a motorized guidance sheet to align the particles in the image plane.
- Measurement in 3D mode, in which several images of each particle are taken in different orientations. The projection that provides the greatest value for the length measurement is used for the evaluation.

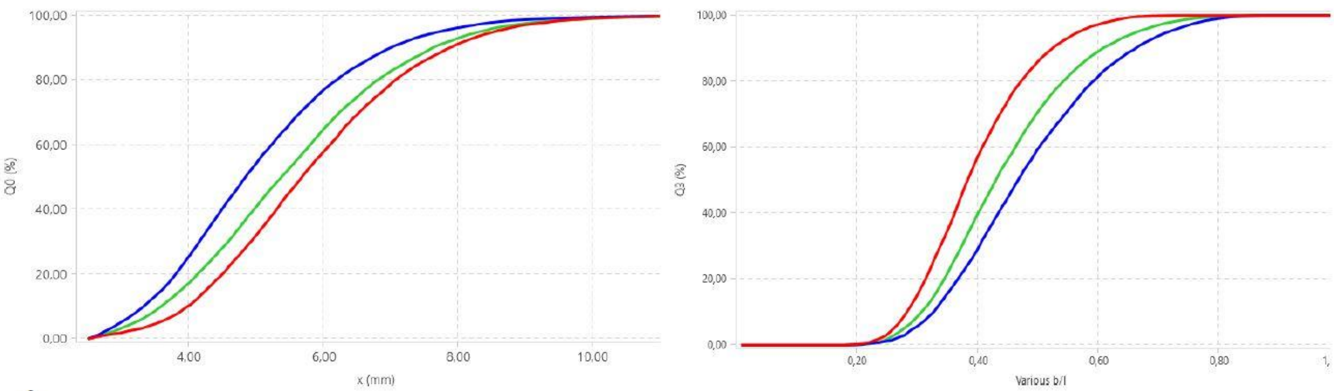


Fig. 5: *Comparison of 2D length measurement without guidance sheet (random orientation,* ***blue****), 2D measurement with guidance sheet (****green****) and 3D measurement (****red****).*

The left chart shows the length distribution, the right one shows the aspect ratio. The greatest length is measured in 3D mode, because here the particles are recorded in their optimal orientation. The 2D measurement with guidance sheet is slightly shorter. The alignment works, but is not as good as in 3D measurement, especially for short extrudates. In the case of 2D measurement in random orientation, the desired length is not measured correctly. Consequently, the aspect ratio is also the lowest in 3D measurement.

Verification of the length measurements

To verify the results of the 3D measurement method, the length of 50 rod-shaped catalyst pellets was determined with the caliper. Subsequently, the same 50 pellets were analyzed with the CAMSIZER 3D. The results are shown in the figure as a cumulative distribution. It is important that the distribution is number based (Q0). In image analysis, various size parameters can be determined simultaneously, such as length, width, and thickness of the particles. A separate distribution is calculated for each size definition. The length of the particles can also be determined in several ways:

- $X_{Fe\ max}$: maximum Feret diameter. This is the maximum distance of parallel tangents applied to the particle projection. In the case of an extrudate particle oriented in the image plane, which shows a rectangular projection, this corresponds to the diagonal. Thus, the measured length would be greater than the distance of the base surfaces of the cylindrical particle, which is measured with the caliper.
- $X_{Fe\ rec}$: The Feret diameter that is perpendicular to the measured particle width. In the case of straight, non-curved extrudate particles, this corresponds to the measurement with the caliper.
- Stretched length $x_{stretch}$: Length is calculated from the area of the particle projection and the width.
- Calculated length x_{length}: Length is calculated from x_{Fe} max and the particle width via the Pythagoras equation.

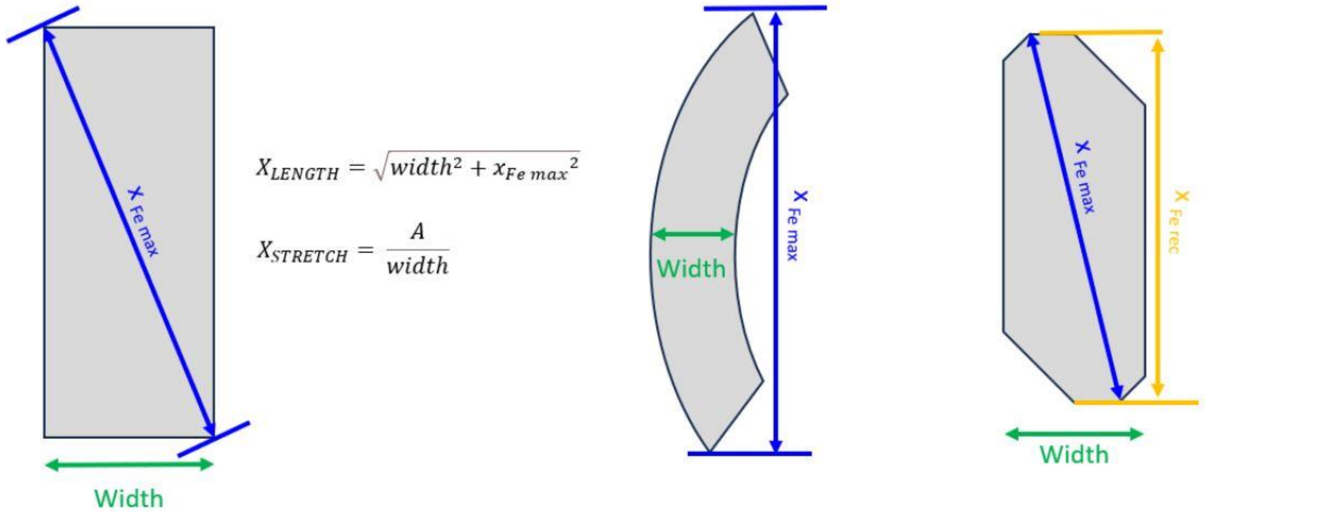


Fig.6: *Particle length measurement. $X_{Fe\ max}$ is the maximum tangent distance. X_{length} and $X_{stretch}$ are calculated from width and X_{Fe} max and $x_{Fe\ max}$, respectively, calculated from width and area (**A**). These length definitions correspond to the measurement with the caliper.*

Which length parameter gives the best results also depends on the application and the characteristics of the sample being examined. Extrudates can be cut or broken, with the former having straight, parallel base surfaces and the latter having uneven, non-parallel base surfaces. In the case of cut extrudates, the "stretched length" gives good results, in the case of broken extrudates, x_{Fe} rec is often the parameter that gives the best results. In the example in the figure, the length definition $x_{stretch}$ provides the best match with the hand measurement.

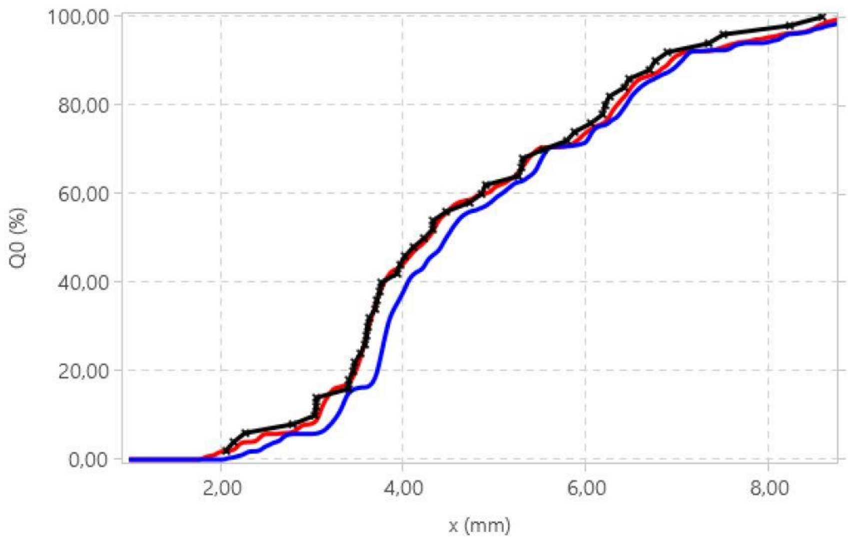


Fig.7: *Measurement of 50 rod-shaped catalysts with the caliper (**black**) and 3D analysis with the CAMSIZER. $X_{stretch}$ (**red**) provides a good agreement with the hand measurement, $x_{Fe\ rec}$ (**blue**) is slightly larger.*

The importance of sampling

In the traditional analysis with the caliper, only a few particles are considered, usually 50 - 100 pieces. These are not representative of the overall sample, because users tend to pick out the long particles for measurement, firstly because they are more conspicuous, and secondly because they are more convenient to measure. The result of the manual measurement is therefore often larger than that of the image analysis, in which a large, representative sample is measured. The example shows that the measurement of the total sample gives a shorter mean length than the measurement of the 50 hand-picked particles.

Image analysis is therefore much more objective and accurate than manual measurement. The time savings are also significant: while manual measurement takes 30-60 minutes, depending on the sample size, the CAMSIZER can analyze thousands of particles within only 2-5 minutes of measurement time.

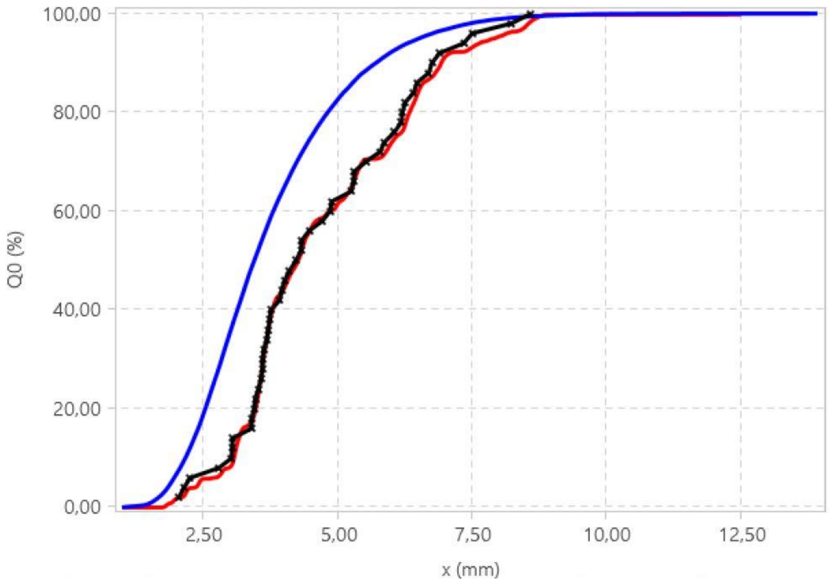
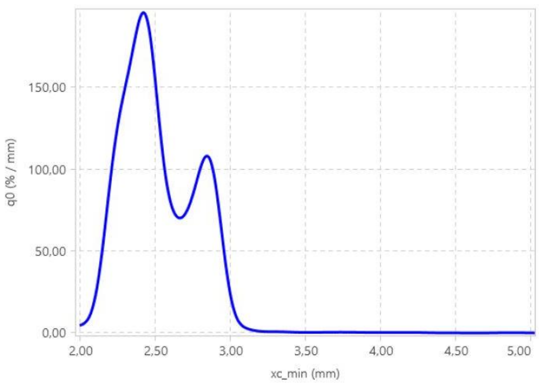


Fig.8: *Comparison of the CAMSIZER 3D measurement of 50 extrudate particles (**red**) with manual measurement (**black**) and the complete sample (**blue**). The sampling error causes the distributions to differ greatly. The 50 particles examined are not representative of the total amount.*

Width measurement

Width measurement of extrudates is already possible in 2D mode without any problems because this is independent of the orientation of the particles. It can be measured in the top view of the base surface as well as in the orientation of the longitudinal axis in the image plane, and with all orientations in between. The measurement example shows the width measurement of a catalyst with a cross-shaped base ("quadrolobe"). The beams of the cross have different lengths, resulting in a "double peak" of the width distribution, with the two maxima corresponding to the size of the two beams of the cross.



Extrudate pellet with cross-shaped base plate.

Fig.9*: When measuring width, the two bars of different lengths of the cross-shaped base surface show up as a double peak.*

Summary

Measuring the size of catalysts is important to understand and optimize their effectiveness and efficiency in chemical processes. The size of a catalyst particle has a significant influence on its surface area and thus on its catalytic activity. The larger the surface area, the more reaction molecules can be adsorbed on the surface, which increases the reaction rate. Measuring the size of catalysts is therefore an important parameter in the quality control of catalysts and in the optimization of chemical processes to ensure that the catalysts have optimal activity and efficiency. Packing density and flow behavior in the reactor can be predicted by means of size measurement. In the case of used catalytic converters, size and shape measurement can be used to determine whether they are suitable for reuse.

More about CAMSIZER 3D and DIA (Dynamic Image Analysis)

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