Quality Control of Catalysts with Digital Image Processing

Catalysts are widely used as an important auxiliary material in the chemical and petrochemical industry. They are able to accelerate chemical reactions by lowering the activation energy, i.e. they change reaction kinetics. Thus, using catalysts in industrial processes has the advantage of higher productivity combined with less energy consumption.

CAMSIZER®

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Quality criteria of catalysts

In many processes a mixture of liquid or gaseous raw materials (e.g. petroleum) is transformed in a reactor with solid catalyst material. Depending on the desired reaction, catalysts of different materials such as ceramics, alumina, metal or alloys are used. To save costs, inexpensive substrates can be coated with the actual catalyst (e.g. platinum or molybdenum).

The active surface as well as the form of the catalysts is crucial for their efficiency. It is important that sufficient free volume is available for the reactants. Moreover, the counter pressure built up by the catalyst must not be too high. Spherical, comb-shaped or rod-shaped geometries have proven to be especially suitable. The rod-shaped catalysts have different shapes; their profiles can have an ellipsoid, trilobe or quadrulobe shape.

Quality Control

The following parameters are crucial for effective and controlled catalytic reactions with a high yield:

- A large active surface
- A large free volume in the reactor to have enough space for the mixing of the reactants
- A high and evenly distributed permeability in the reactor
- A defined ratio of active surface and reactant.

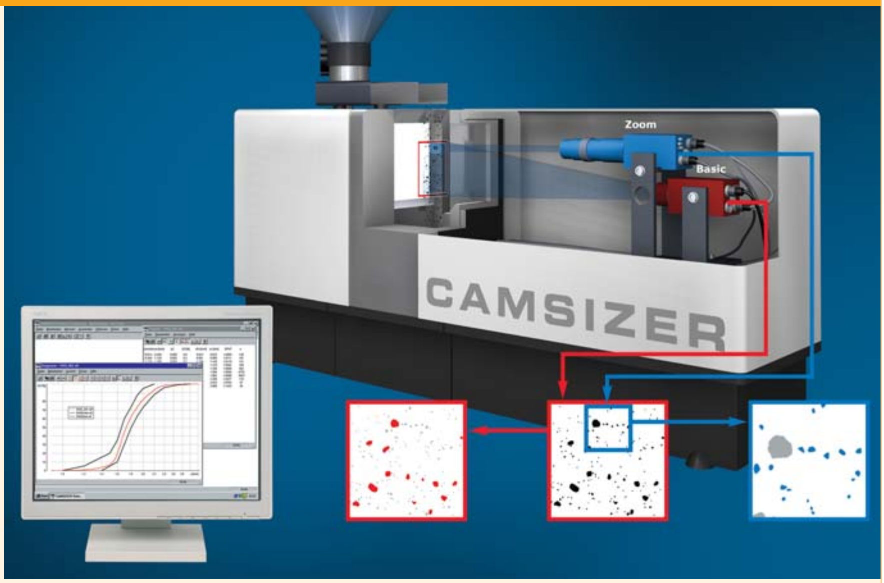
The analysis of size and shape plays an important role for the quality control of catalysts. Sieve analysis is often used for this, however, it only provides the required size information for spherical particles. Rods, for example, pass the mesh with a particular orientation which makes it impossible to clearly separate breadth and length values.

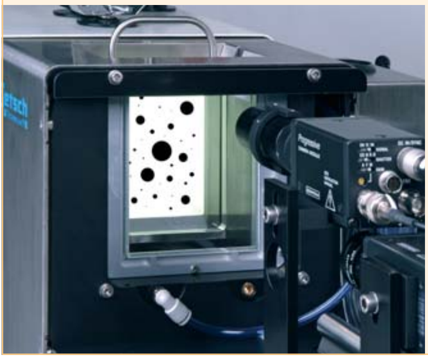
Another method is to measure catalysts with calipers which is time-consuming and hardly representative as it can only be done for small quantities.

The CAMSIZER System

Dynamic Image Analysis
With several hundred instruments in operation worldwide, **the CAMSIZER is the most successful tool for particle size and shape analysis of dry, flowable bulk goods with dynamic image analysis**. Due to its wide measuring range of **30 µm to 30 mm** and the excellent compatibility to sieve analysis, the CAMSIZER is a powerful alternative to traditional sieving.

The main areas where the CAMSIZER is used are quality control, research and production monitoring. In combination with solutions for the partial or complete measurement automation, continuous sample analysis can also be realized cost-effectively. The patented measuring setup of the CAMSIZER – two digital cameras as adaptive measuring unit – improves and optimizes particle analysis with digital image processing. Thus it is possible to cover the complete measuring range without any adjustments of the instrument.





Measurement Principle
The sample is fed in from the feed channel so that all particles fall through the measurement field. During the measurement procedure the two cameras (CCD) take over different jobs: the Basic Camera (CCD-B) records large particles, the Zoom Camera (CCD-Z) detects fine particles. The contact-free optical measurement is carried out in real time and simultaneously ascertains all the required information about particle size and shape. To make this possible in the context of an automated continuous analysis, an online version was developed which can be customized to the particular requirements.

The obtained results can be presented graphically and in tabular form as size fractions, frequency distribution or cumulative distribution. In addition, the CAMSIZER can determine the number of particles as well as the specific surface, the density, the shape parameters and also the transparency of the sample material. All parameters are ascertained with great precision and excellent reproducibility. The CAMSIZER software also allows the presentation of daily reports, trend analyses, mean value calculations and much more. In accordance with DIN 66165, a well structured, individually adjustable measurement protocol can be generated.

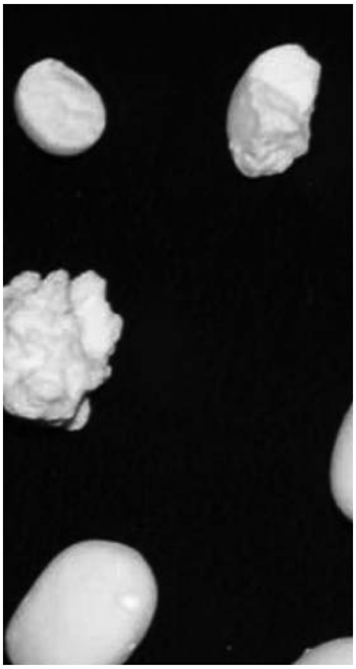
Geometry: quick and reliable detection

The benefits of using the CAMSIZER for the quality control of catalysts are quite obvious: A large quantity of particles can be measured in a very short time. **The resolution and reproducibility of the CAMSIZER measurements are much higher** than those of sieve analysis or measurements with calipers. The possibility to obtain other shape parameters apart from length and diameter is very important as these allow conclusions about the geometry of the catalyst. Thus it is possible to determine, for example, the quantity of defective particles.

The example of different catalysts will demonstrate how easily and reliably particle size and shape can be analyzed with the CAMSIZER. Many catalysts have a trilobe or quadrulobe geometry. The Camsizer can measure two different diameters for quadrulobe rods. It is able to detect the two diameters even if they are very similar, so that the two different geometries can be clearly separated.

The graphic shows the length distribution of the rods (flat curve) in the range of 1 to 6 mm. The breadth distribution is much narrower between 1 and 1.6 mm, showing two different values for the quadrulobe catalyst. The schematic presentation illustrates these differences.

The methodology is similar for spherical particles. The results for the diameters are consistent with the sieving results. However, the resolution of the CAMSIZER measurement is much higher. The shape parameters give information about the amount of defective particles; moreover, it can also be examined in how far the particles are ellipsoid or truly spherical. Such qualifications are based on the sphericity and symmetry of the sample. Figure 5 shows two curves with particles of a different sphericity. The green curve represents a good quality as the particles are mostly round. The red curve however shows lower sphericity values which is a sign for broken parts or angular particles (see snap shot).



Snap shot

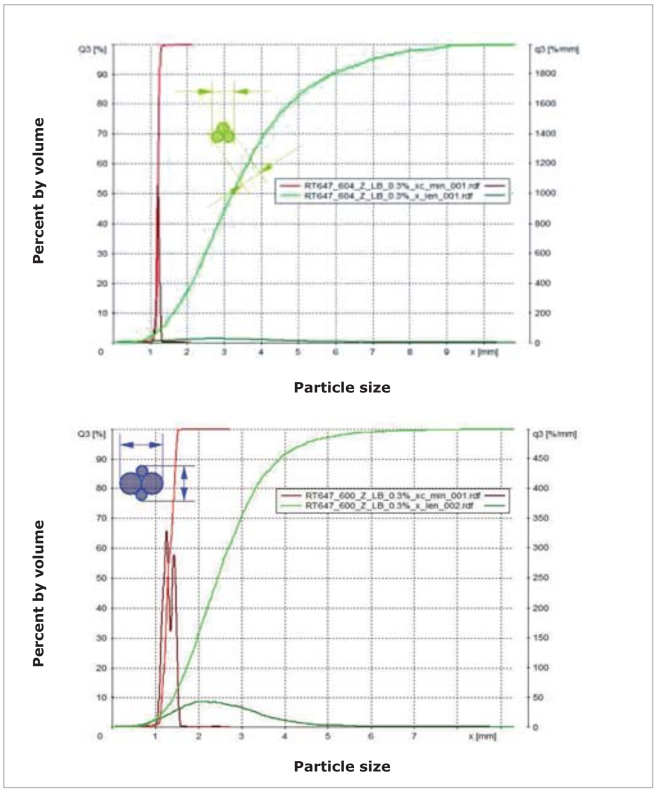


Fig. 3: Comparison of trilobe (left) and quadrulobe (right) catalysts in the CAMSIZER

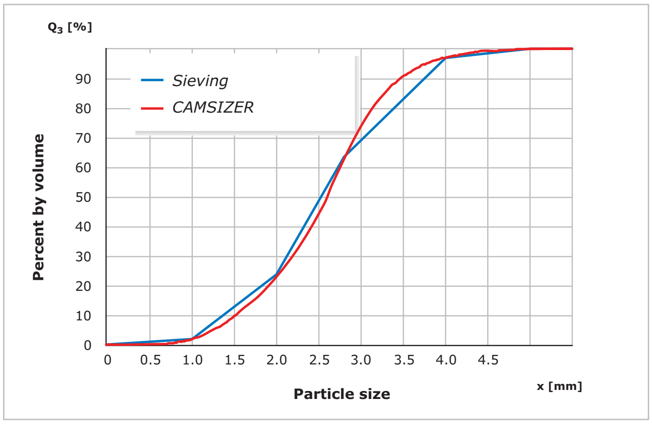


Fig. 4: Comparison sieve analysis and CAMSIZER

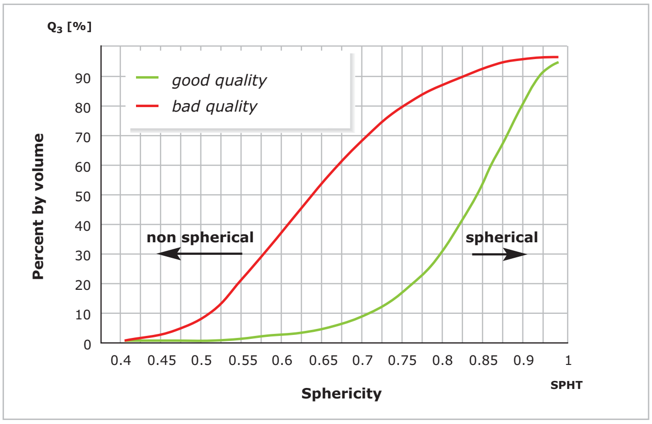


Fig. 5: Sphericity of qualitatively different samples