HORIBA Scientific

Application Note

Particle Size and Shape Analysis of Catalysts using Dynamic Image Analysis

AN162

Hydrotreating catalysts come in different sizes and shapes depending on the application and manufacturer. Common shapes include spheres, pellets, cylinders, and 3 or 4 lobe (trilobe & quadralobe) shaped pieces. The size and shape of the catalyst pieces are important specifications that require quantification. Older techniques including calipers and sieves are now being replaced by dynamic image analysis using the CAMSIZER. The CAMSIZER provides quick, accurate definitions of all physical dimensions important to catalyst manufacturers and end users.

Introduction

Catalysts are a chemical species that increase the rate of a chemical reaction by providing an alternative reaction pathway to the reaction product, typically by lowering the activation energy. A catalyst is different than a chemical reagent in that it participates in but is not consumed by the reaction. Catalysts are involved in more than 80% of all processes in the chemical industry. In most of these processes, a mixture of chemicals (liquid or gaseous) is induced into a reactor filled with catalyst material. The reaction vessel is put to a certain pressure and temperature to start the reaction. The acceleration effect arises mainly from absorption and activation phenomena at the surface of the catalyst.

For effective catalytic reactions with a high yield it is important to have:

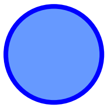
- Large surface area of the catalysts
- Enough free volume in the reactor in order to achieve a high throughput - the more volume is occupied by the catalyst, the less reactant can be introduced to the reactor
- The ratio of catalyst surface and reactant volume is crucial for controlling the reaction kinetics
- High permeability within the reactor in order to have a high and constant flow and good mixing of the reactants

For elongated catalysts, size and shape distribution provides useful information on how the aims mentioned above can be achieved. The size and shape of the catalyst pieces is a trade off between the desire to minimize pore diffusion effects in the catalyst particles (requiring small sizes), and pressure drop across the reactor (requiring large particle sizes).

Size analysis has to provide information on width as well as length and length distribution of the particles and to show the amount of dust in the sample. Shape analysis needs to give results on various dimensions as well as symmetry.

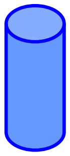
Taking all these parameters into account, it is possible to predict the reaction behavior of the catalysts in a certain process. Except for spherical catalysts, these ambitious demands can never be fulfilled with a sieve analysis. Measuring single rods with a caliper gives precise results but this method is time consuming and still it is only possible to measure a very small quantity of particles.

Spherical catalysts



Spherical catalysts can be measured with the CAMSIZER quite easily without special efforts. Using $x_{c\ min}$ or $x_{Ma\ min}$ the results should be in good agreement with sieve analysis. With shape parameters like symmetry it is possible to detect broken or defective particles. Aspect ratio (b/l) or sphericity can control the roundness of the beads.

Elongated catalysts/cylinders



It is possible to define the length and width of cylindrical catalysts several ways using the CAMSIZER.

The length can be defined using $x_{Fe\ max}$, x_{length} or $x_{stretch}$, depending on the preference of the user. $x_{Fe\ max}$ displays the longest dimension (diagonal) of the particle projection, x_{length} and $x_{stretch}$ deliver the true height of the orientated extrudates – see Figure 1 below.

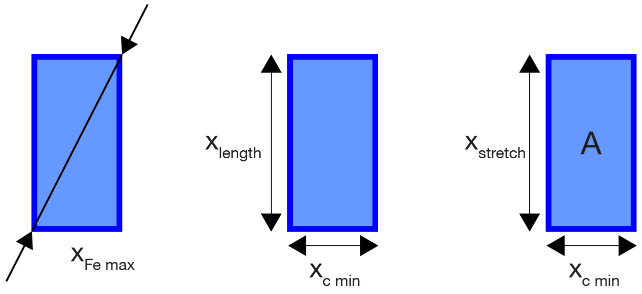


Figure 1

$$x_{length} = \sqrt{(x_{Fe\max})^2 - (x_{c\min})^2}$$

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

The width for straight extrudates can be defined as $x_{c\ min}$ or $x_{Ma\ min}$. The width measurement for bended extrudates is better defined by $x_{Ma\ min}$ as seen below in Figure 2.

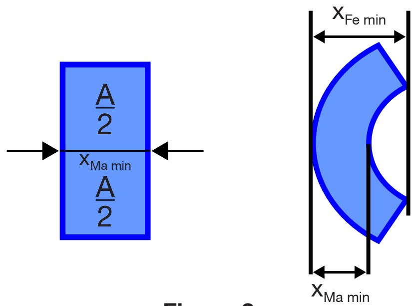


Figure 2

Special cross sections:

Many catalyst extrudates have a trilobe or quadralobe cross section (Figure 3). Using the $x_{c\ min}$ or $x_{Ma\ min}$ size definition it is possible to distinguish between the two different diameters of the quadralobe extrudates. They are reflected in the two maxima of the bimodal $x_{c\ min}$ or $x_{Ma\ min}$ distribution (Figure 4). For a trilobal cross section $x_{c\ min}$ delivers a width distribution of a mean value from all projections (Figure 5).

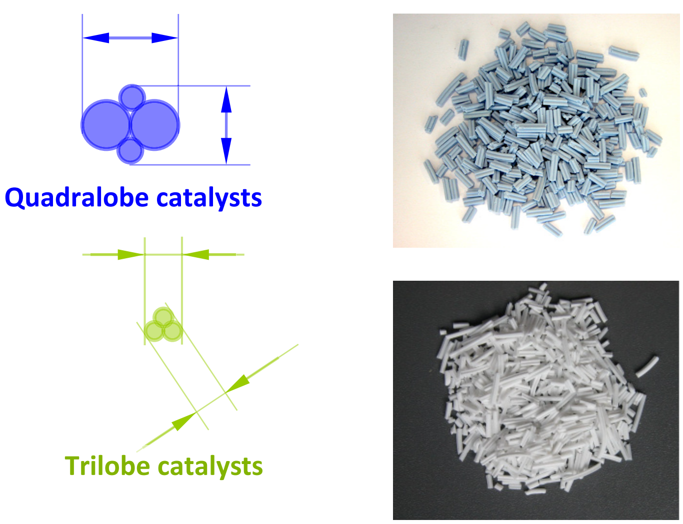


Figure 3

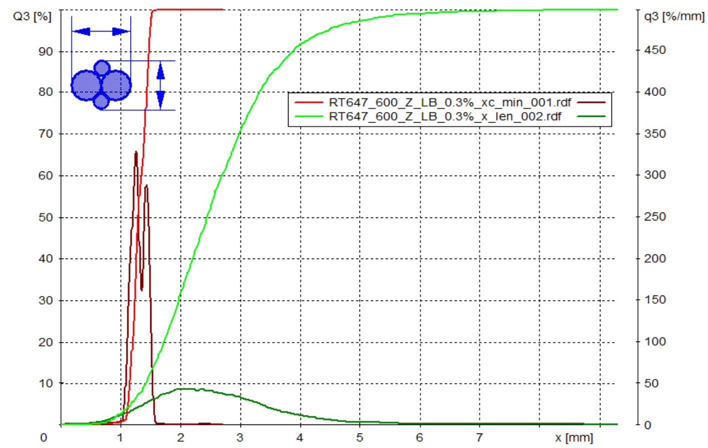


Figure 4

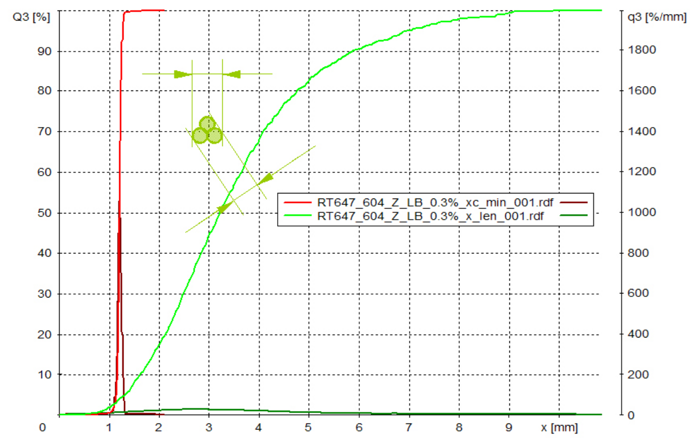


Figure 5

Note: In both graphs the shorter green distribution shows the x_{length} length distribution. The taller peaks in maroon show the $x_{c\ min}$ width distributions, including the two different values for the quadralobe shape.

Measurement advice

When measuring extrudate catalysts it is recommended to use a flexible guidance sheet for proper orientation to the cameras. Setting the “Part of image” function to “Basic 1/3” assures particle detection before the onset of random orientation. Overlapping particles can be discarded from result calculations by selecting “Ignore conv < 0.96~0.97”. Try a slow and a fast measurement and compare “Q_3 over Conv” to find a suitable value for the threshold. $x_{Ma\ min}$ is better suited for width measurement than $x_{c\ min}$ for bended extrudates.

Conclusions

Using the CAMSIZER dynamic image analyzer provides direct measurement of the length and width distributions for extruded catalysts, as well as supplying images of all particles analyzed. Compared to sieving or manual measurement with callipers, the CAMSIZER provides a reduction in analysis time, workload, and manpower costs. Since a large number of particles are analyzed the CAMSIZER generates statistically valid results that are highly reproducible. Additional size and shape parameters are available for custom shaped catalysts or applications. Many catalyst manufacturers have successfully switched from older techniques to the CAMSIZER and are experiencing the benefits of reduced expense and improved data quality.

Figure 6: CAMSIZER