Measuring the Particle Shape of Micronized Powders

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What we'll talk about

- Importance of particle shape
- Dynamic image analysis principles
- Case studies
- Q&A

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Starting point

- Somewhat familiar with particle technology
- Particle sizes over 1-5 microns and up to many millimeters
- Stay current with free webinars
  - Particle size essentials

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Featured technologies

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- **LA-950**
  Laser Diffraction
- **SZ-100**
  Dynamic Light Scattering & Zeta Potential
- **CAMSIZER & CAMSIZER XT**
  Dynamic Image Analysis
- **PSA300**
  Static Image Analysis
- **SA-9600**
  Flowing Gas BET Surface Area

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CAMSIZER XT: Fine Image Analysis

- High resolution size & shape
- Extends lower detection range
- Cohesive & free-flowing powders, suspensions
- 1 micron – 3 millimeter

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What we'll talk about

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Why is Shape Important?

- Most (all?) particles are not perfectly behaving spheres
- Shape can influence almost everything
  - Particle processes, making products
  - Product performance
  - Making measurements
- So in other words, shape can affect almost everything

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Particle Processes

- Powder flow; spheres flow easily, needles do not
- Liquid flow; increased aspect ratio will increase viscosity
- Powder mixing; blend time may change with shape
- Also VERY associated with size, hard sometimes to separate size and shape

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Powder Flow

- Understand that spheres flow more easily than needles
- How to quantify? Need to first know something about powder flow testing
- Won't go into great detail in today's talk
- Just show results including particle shape

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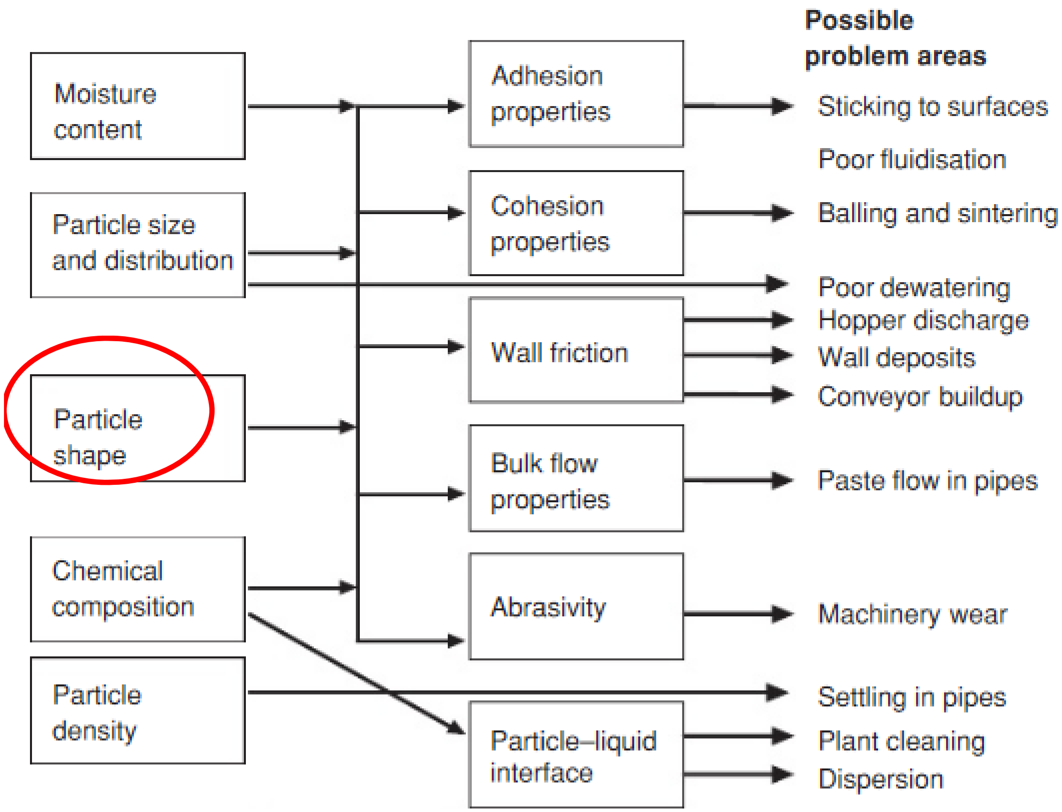
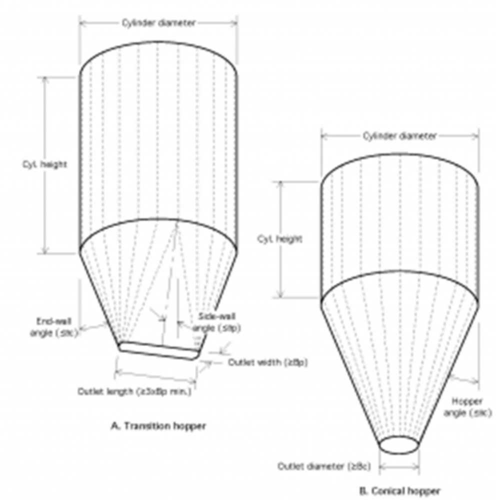
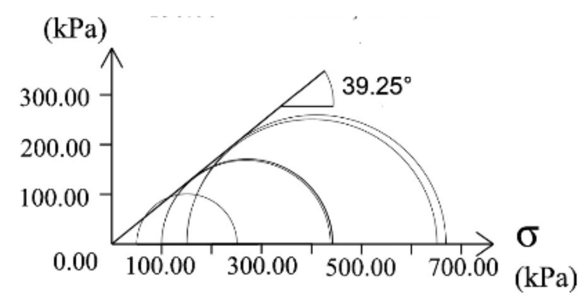
Powder Flow*





Θc =hopper angle

BC = outlet diameter



Angle of internal friction

* **Bulk Solids Handling**
Equipment Selection and Operation

Edited by

Don McGlinchey
Reader
Centre for Industrial Bulk Solids Handling
Glasgow Caledonian University
UK

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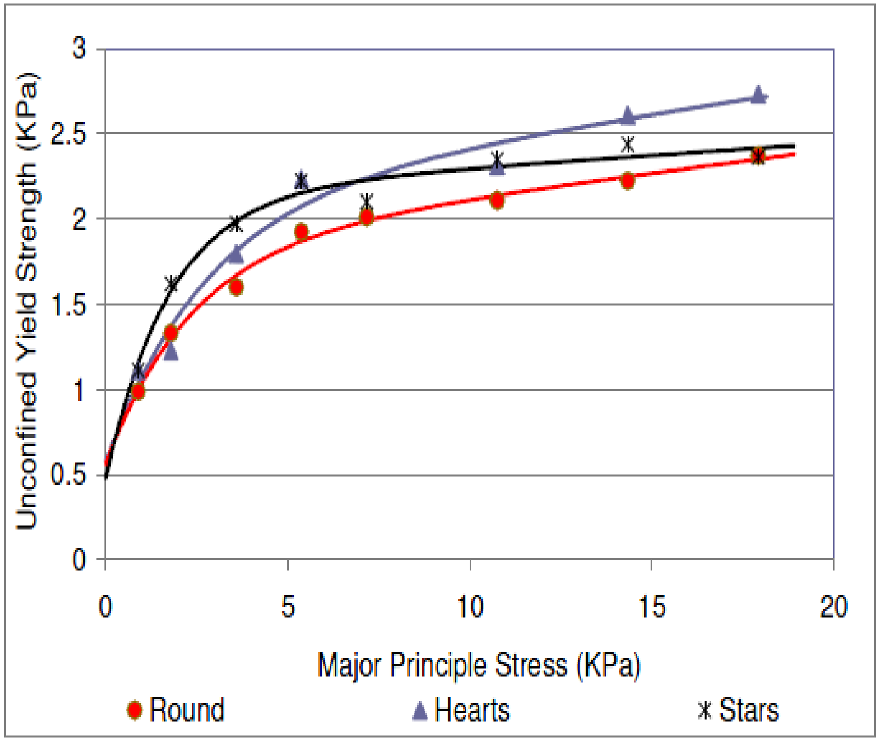
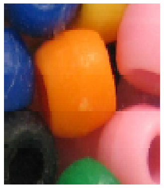
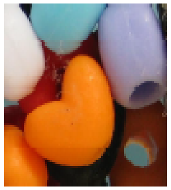
Powder Flow

- Unconfined Yield Strength
- Major principle stress that causes an unconfined bulk material to fail in shear
- Directly proportional to arching & formation of rat holes
- Influences by # contact points

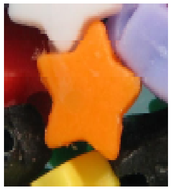




Round



Heart



Star

From: Johansen, Effect of Particle Shape on Unconfined Yield Strength, Material Flow Solutions,Inc.

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Powder Flow*

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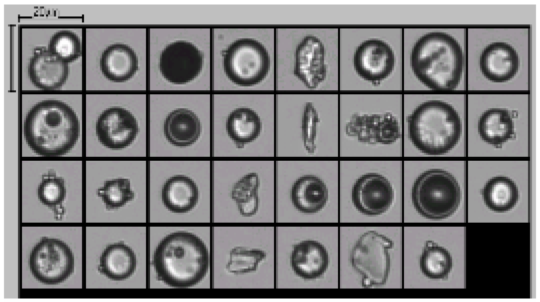
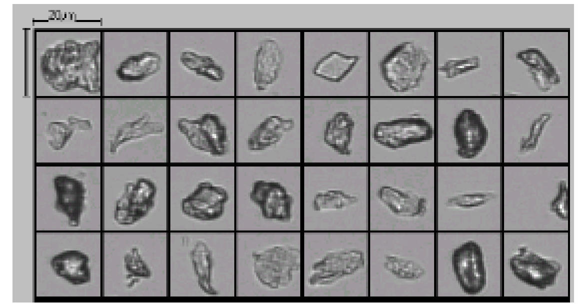


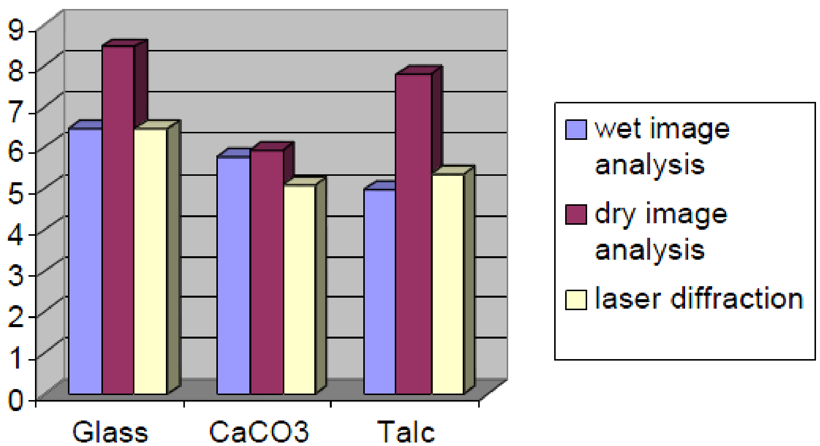
Glass spheres

$CaCo_3$

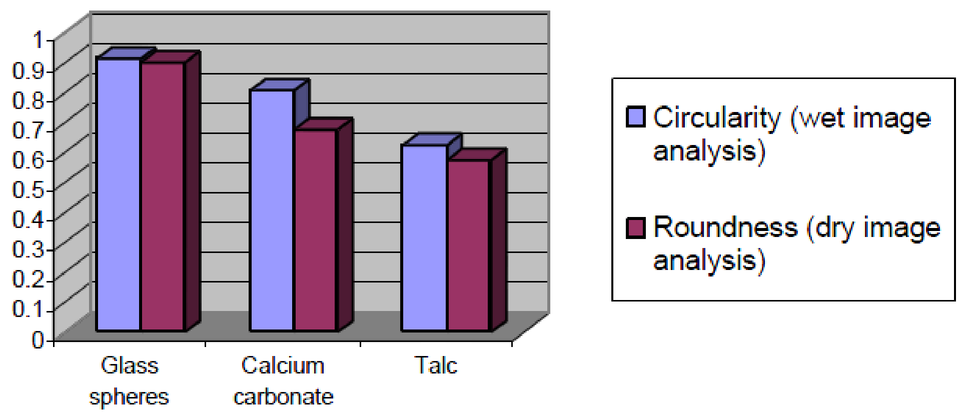


Talc

Similar size ~ 5 µm



But different shape



*Bumiller, et. al., A Preliminary Investigation Concerning the Effect of Particle Shape on a Powder's Flow Properties, Proceeding from WCPT4, July 2002

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Powder Flow

	Talc	Calcium Carbonate	Glass Spheres
BC (ft.)	0.4	0.6	0.1
Bulk density range [pcf] β	 13 to 43 0.16	 35 to 75 0.11	 53 to 84 0.06
θc [deg]	4*	12	23
Critical flow rate [tph]	0.3	9.1	2.8
δ [deg]	35	38	36

*Flow questionable along any sloping hopper surface

BC: minimum outlet diameter to prevent arching
Θc: hopper wall angle (from verticle) to achieve mass flow
Critical flow rate: predicted flow from outlet
δ : effective angle of internal friction

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Powder Flow*

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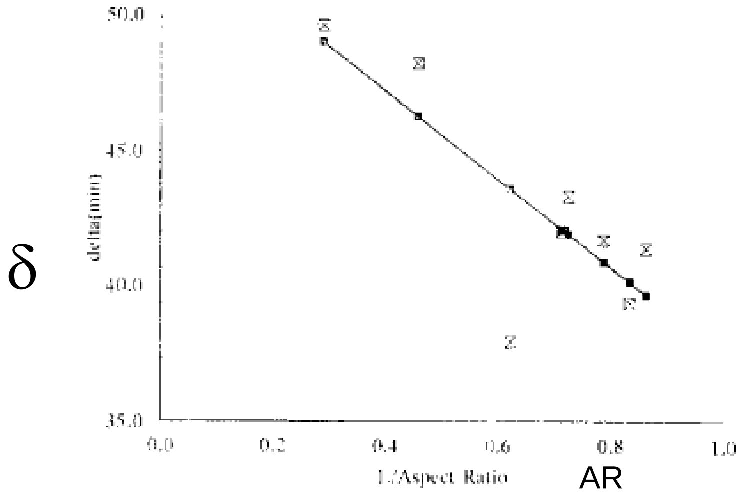
Powder	d_{vs} [μm]	Shape (BP)	AR	SF
Pregelatinized starch	103.2	Angular	1.38 ± 0.26	7.54 ± 0.35
Paracetamol	537.6	Angular	1.61 ± 0.65	7.38 ± 0.53
Calcium carbonate	4.6	Cubic	1.20 ± 0.20	7.66 ± 0.36
Potassium chloride	481.1	Cubic	1.27 ± 0.29	7.70 ± 0.56
Maize starch	49.2	Round	1.16 ± 0.12	3.86 ± 0.18
Microfine cellulose	363.3	Round	1.40 ± 0.36	4.48 ± 0.78
Microcryst. cellulose	107.7	Rod shaped	2.19 ± 0.99	7.16 ± 0.43
Acetylsalicylic acid	721.7	Needle shaped	3.47 ± 1.17	7.45 ± 0.39



$$SF = Co + \frac{P}{l} \times \frac{A}{\frac{S \times S_p}{2}} - \frac{A}{\frac{\pi}{4} S_p^2} \times \frac{A}{S \times S_p}$$

$$\delta = 0.347 \cdot e^{AR} - 2.434 \cdot \ln d_{vs} + 59.336$$

Fig. 4. The influence of particle shape on the angle of internal friction at optimum magnesium stearate concentration. ■, Estimated values; ☒, experimental values.

*Podczeck & Miah, The influence of particle size and shape on the angle of internal friction and the flow factor of unlubricated and lubricated powders, International Journal of Pharmaceutics 144 (1996) 187 194

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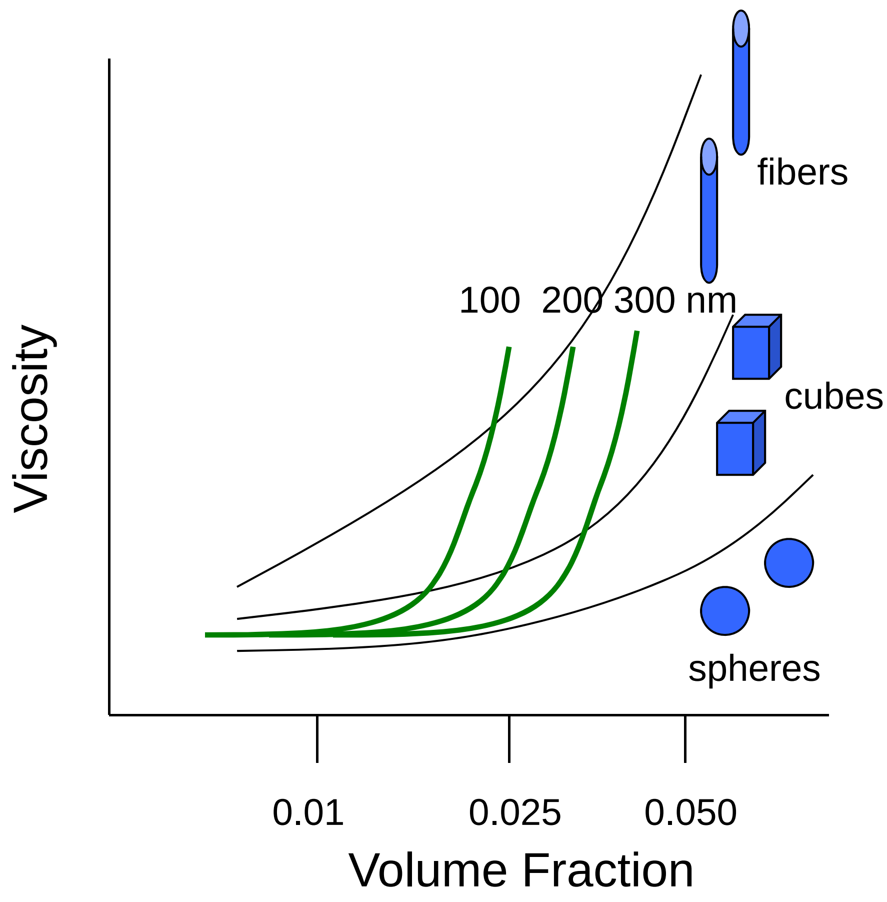
Rheology/Viscosity

- Complex relationship between size/shape and rheology
- More spherical shape = lower viscosity
- Small particle size = higher viscosity
- Wider particle size distribution = lower viscosity



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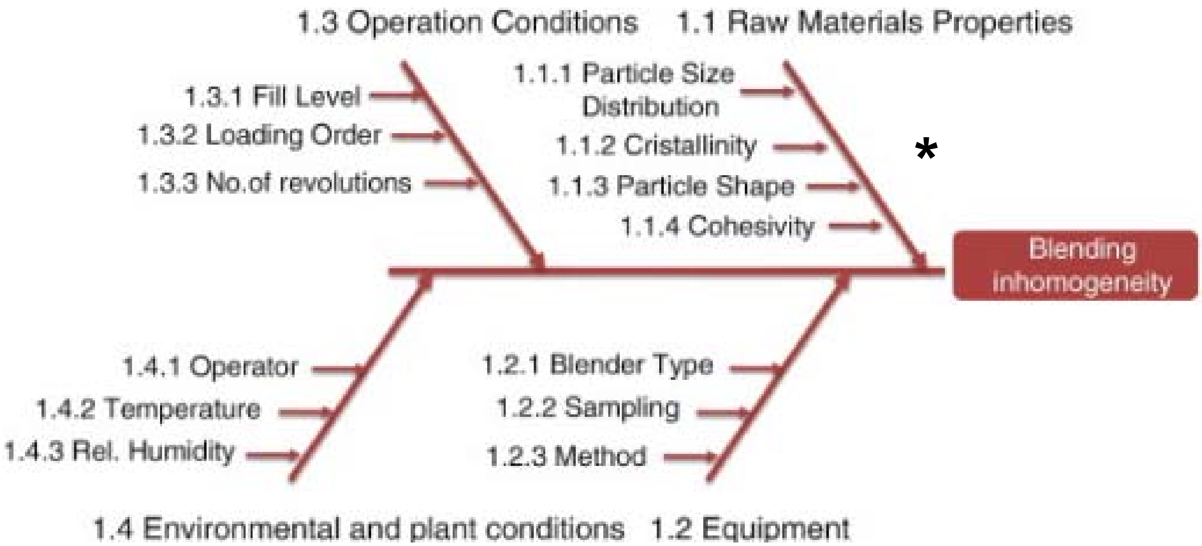
Mixing

- Shape affects mobility and therefore causes segregation
- Smaller effect than size
- Shape differences could still be substantial



*Koller et. al.,Continuous quantitative monitoring of powder mixing dynamics by near-infrared spectroscopy Powder Technology, Volume 205, Issues 1–3, 10 January 2011, Pages 87–96

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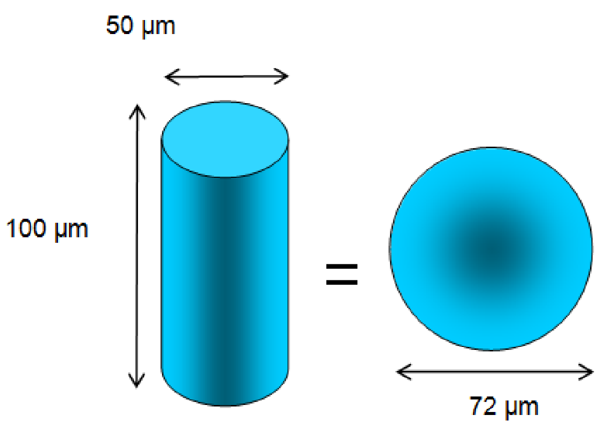
Shape Effects Size Measurements

- Inherent effect since light scattering instruments report equivalent spherical diameter
- Sieve vs. laser diffraction vs. image analysis
- Consider cylinder vs. sphere

$V = \pi r^2 h$



$V = \frac{4\pi r^3}{3}$

Sieve = 50 mm
Laser = 72 mm
IA = full description

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Modeling/Predicting Differences

- In particle size; choose a technique, influence the answer
- Well explained in this paper

PDA Journal
of Pharmaceutical Science and Technology

PDA
Parenteral Drug Association

Theoretical Aspects of Particulate Matter Monitoring by Microscopic and Instrumental Methods†

Hans G. Schroeder* and Patrick P. Deluca

Presented at the Spring Meeting of the Parenteral Drug Association in Philadelphia, June 1977

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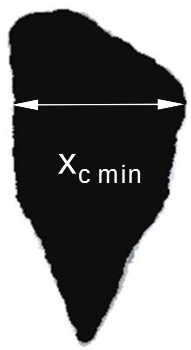
Defining Size

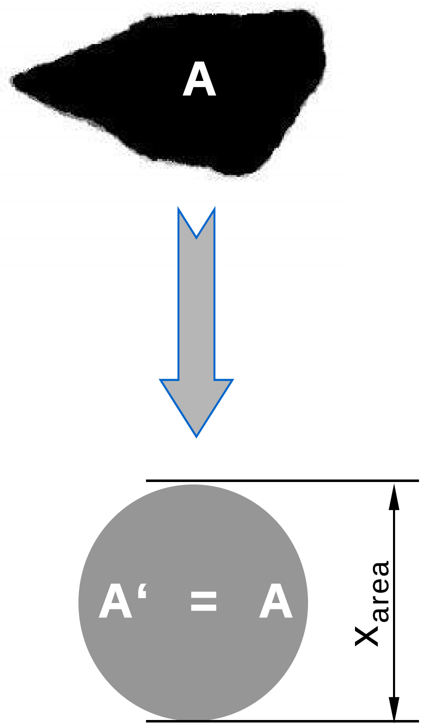
$X_{c\,min}$

"width"



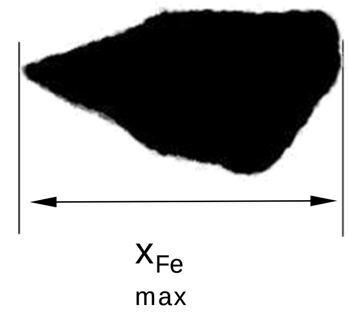
X_{area}

"diameter over projection surface"



$X_{Fe\,max}$

"length"



CAMSIZER results are compatible with sieve analysis

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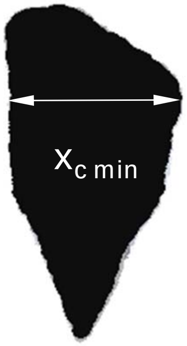
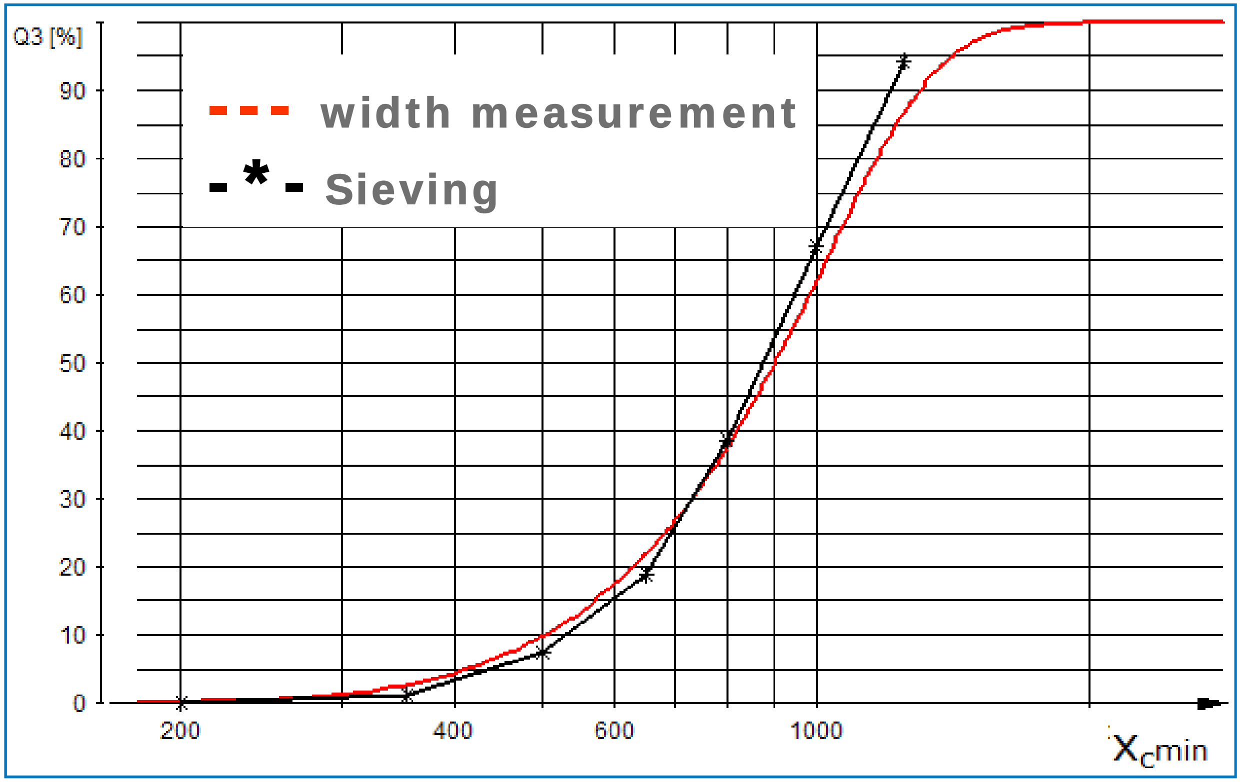
Width ⇔ Sieving

$X_{c\,min}$

"width"





comparison

CAMSIZER-measurement $x_{c\,min}$ (**red**) and sieving * (**black**)

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Want to Measure Shape?

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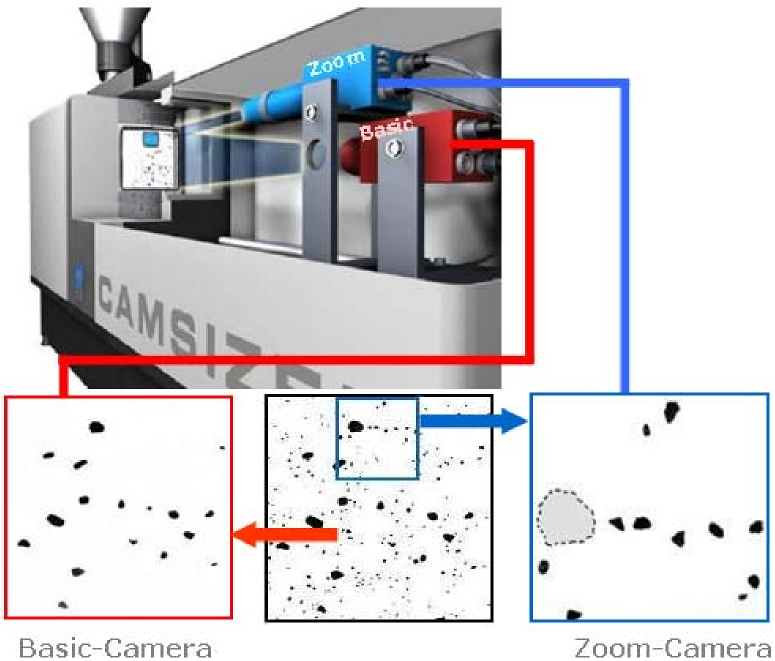
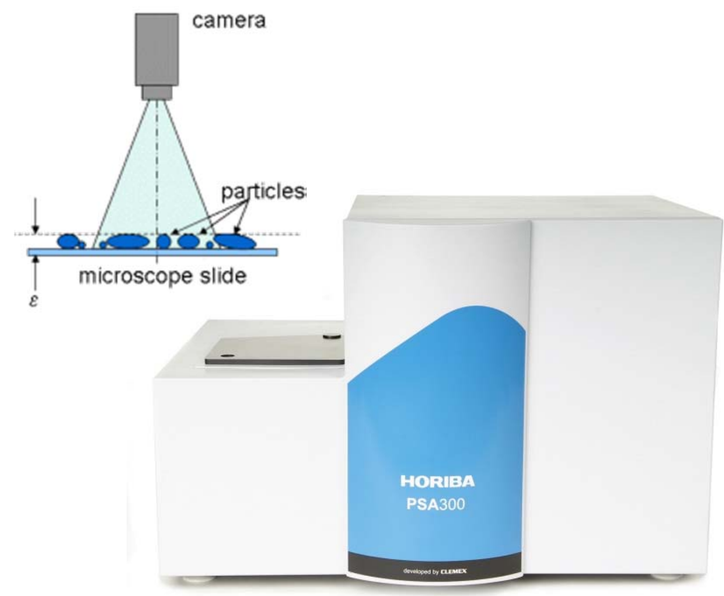
- Use image analysis !
- Direct measurement of size + shape
- Get not only size but also shape distribution

CAMSIZER



PSA300



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Summary

- Particle shape a critical physical parameter
- Affects the product, the process and lab measurements
- Understand how shape affects your business
- Understand how shape influences your measurements

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What we'll talk about

- Importance of particle shape
- **Dynamic image analysis principles**
- Examples
- Q&A

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Image Analysis: Two Approaches

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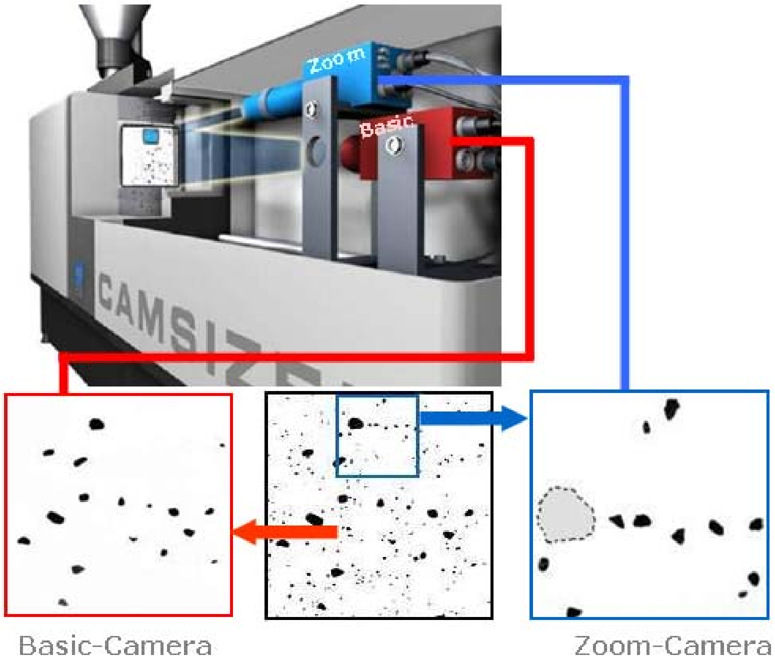
Static:
particles fixed on slide,
stage moves slide

0.5 – 1000 um
2000 um w/1.25 objective

Dynamic:
particles flow past camera



1 – 3000 um

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Static or Dynamic Image Analysis?

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- Dynamic
  - Broad size distributions (since it is easier to obtain data from a lot of particles)
  - Samples that flow easily (since they must be dropped in front of camera)
  - Powders, pellets, granules
- Static
  - Samples that are more difficult to disperse (there are more methods for dispersing the samples)
  - Samples that are more delicate
  - Pastes, sticky particles, suspensions
  - Precious samples

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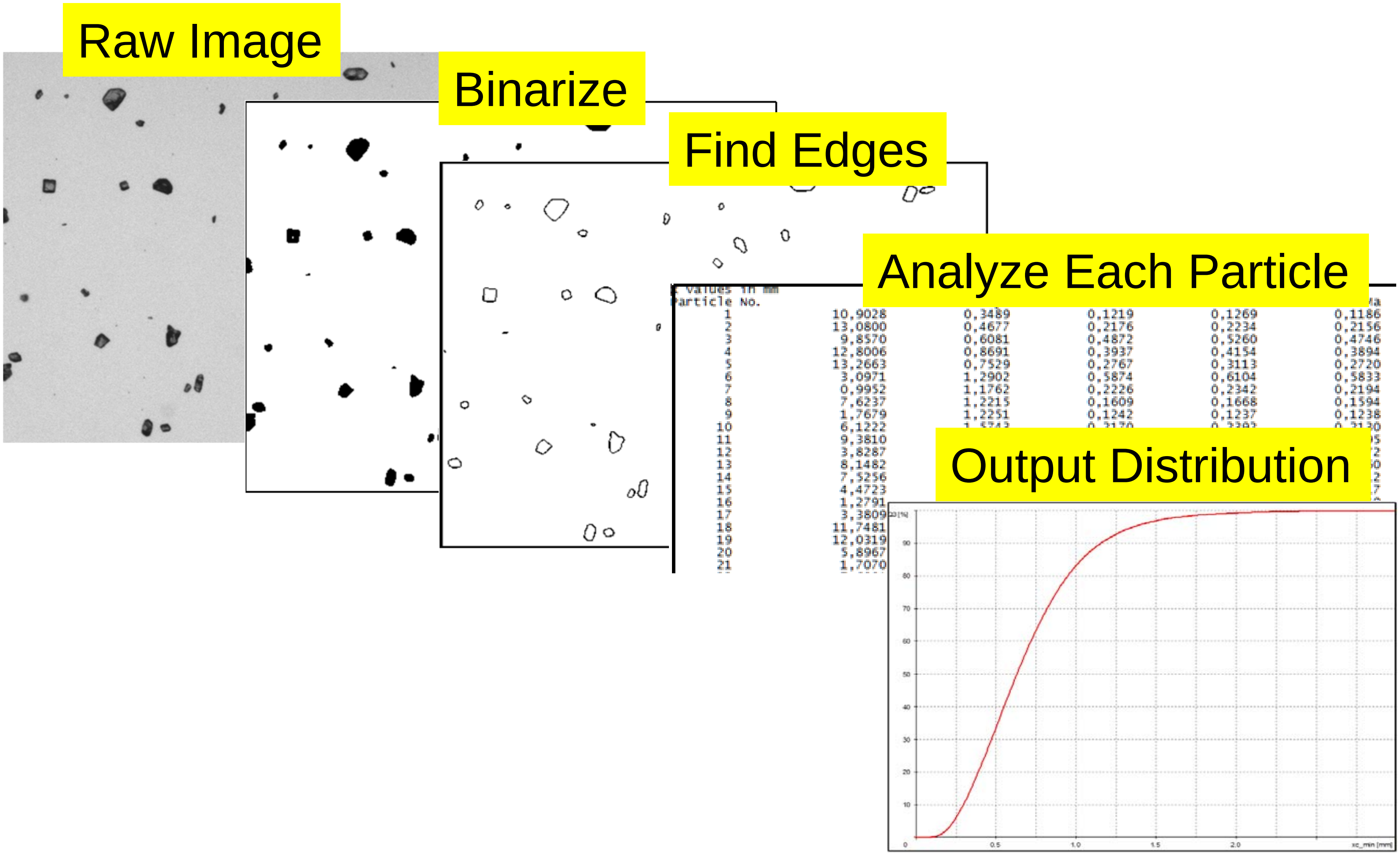
Data Evaluation



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Dynamic Image Analysis: Moving Particles

Use gravity, or, better, vacuum (from a compressed air supply and venturi) in order to draw particles through instrument. Vacuum helps keep the windows clean.

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Why Dynamic Image Analysis?

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- Robust measurement….the interaction between the instrument and the particle is optical, so there is no wear and change in calibration.
- High resolution size distribution results
- Fast

Also, these are all reasons to use Dynamic Image Analysis instead of sieves.

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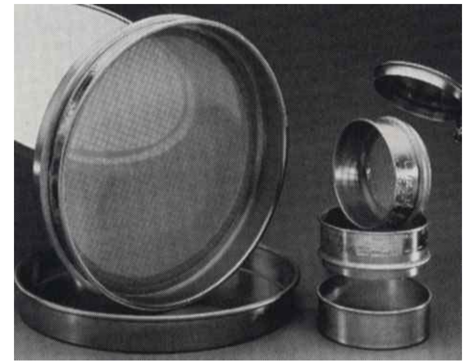
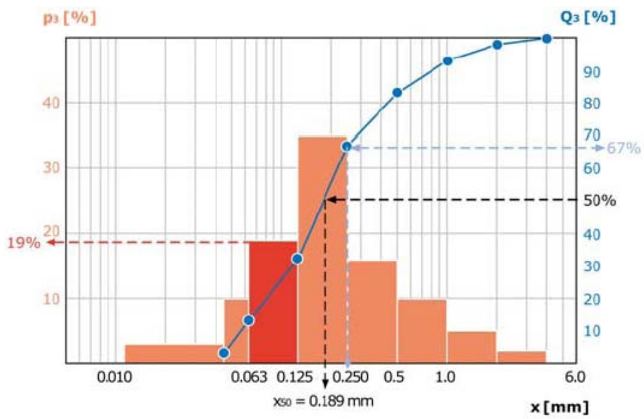
Sieves

- Solid particles 20 μm – 125 mm
- Low equipment cost
- Some automation/calculation available
- Difficult to tell when sieve results are “drifting” due to wear
- Results depend on nature of “shaking” leading to operator to operator variations in results.
- Limited information





Size class [mm]			p_3 [%]	Q_3 [%]
	<	0.045	3.0	3.0
0.045	-	0.063	10.0	13.0
0.063	**-**	**0.125**	**19.0**	**32.0**
0.125	-	0.250	35.0	67.0
0.250	-	0.500	16.0	83.0
0.500	-	1.000	10.0	93.0
1.000	-	2.000	5.0	98.0
2.000	-	4.000	2.0	100.0
> 4.000			0.0	100.0

x_{50} = 0.189 mm

More information available through www.retsch.com

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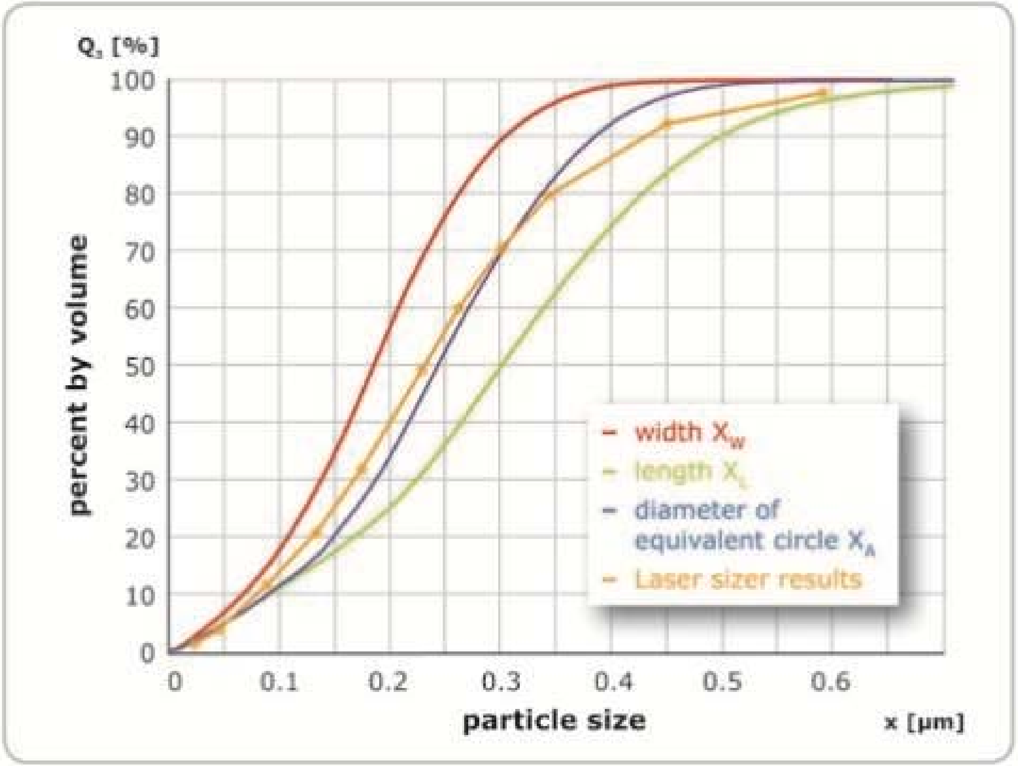
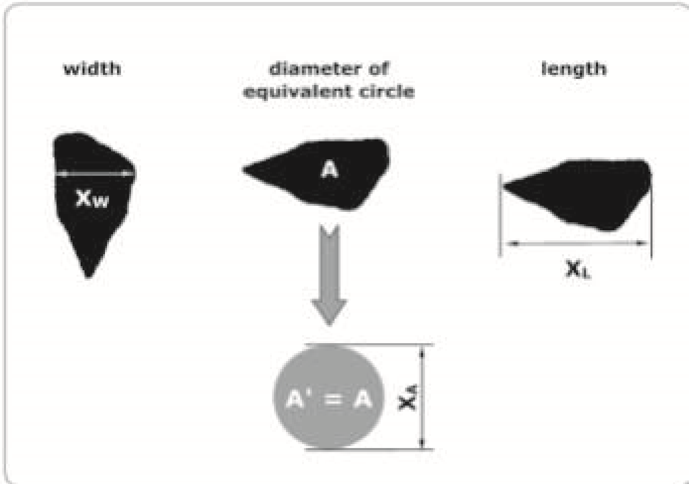
Advantage of Image Analysis

Better size analysis due to understanding of particle shape: Length, Width, Average Diameter





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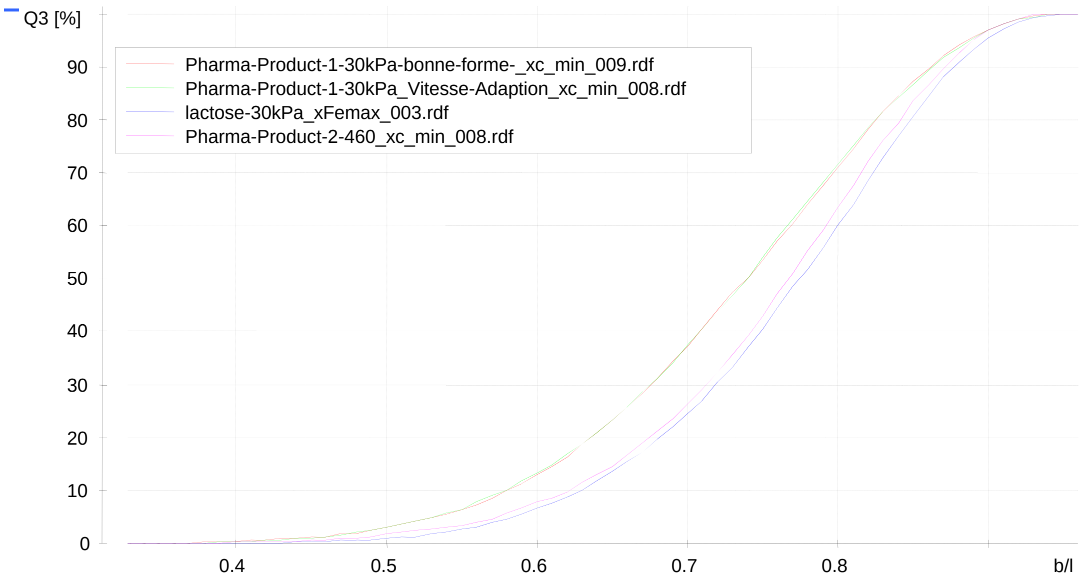
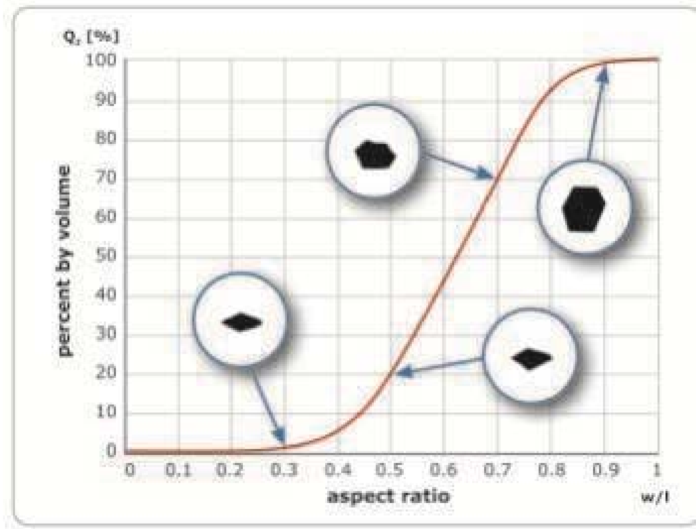
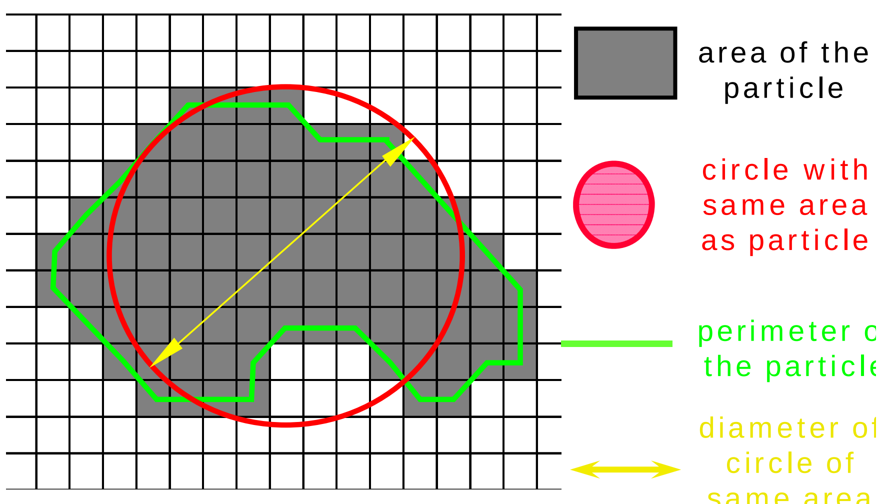
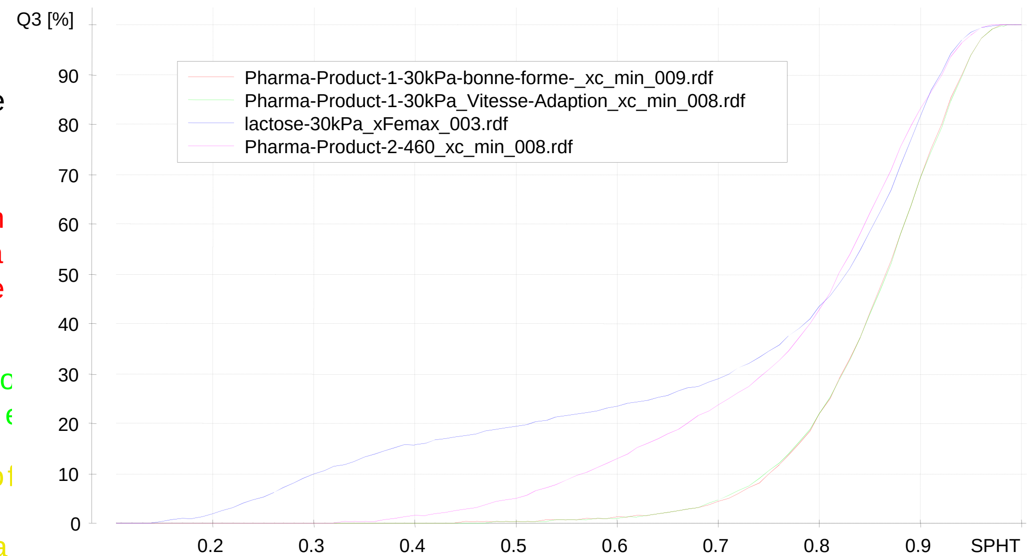
Shape Analysis with CAMSIZER XT









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X-Jet Dry Powder Dispersion

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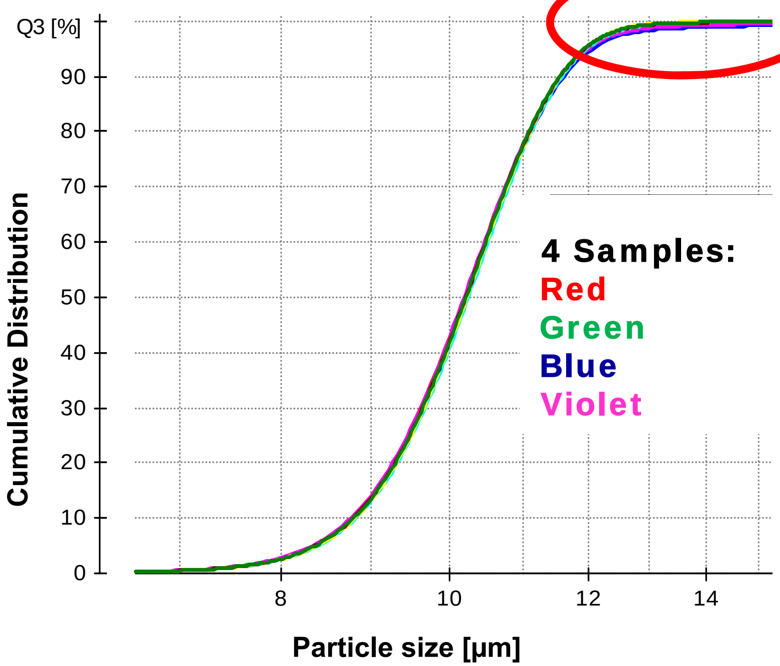
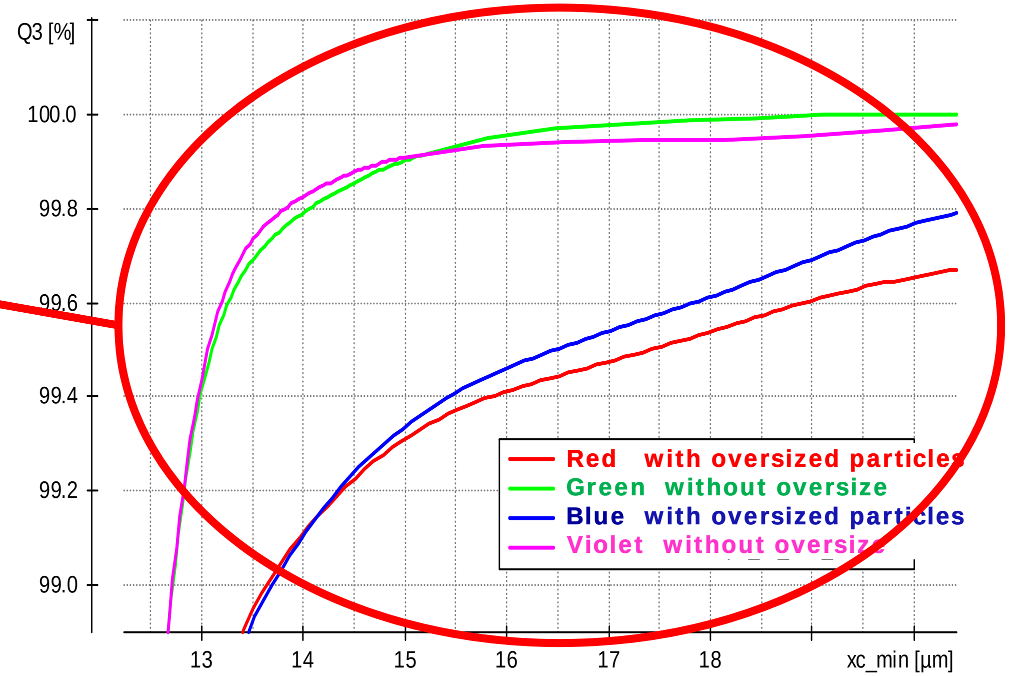
Detection of oversized particles





Δ ~ approx 0.5% Vol.

Sample: PMMA Beads

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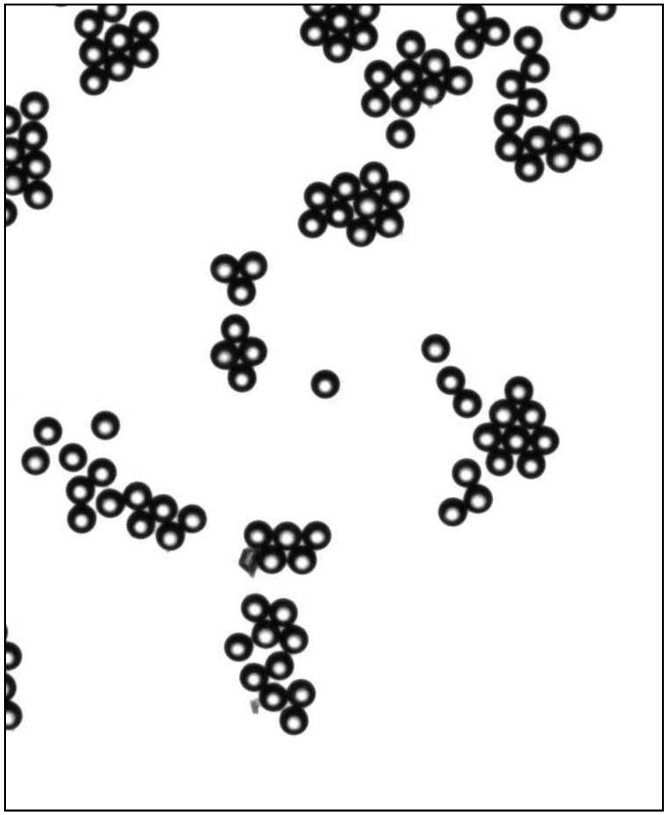
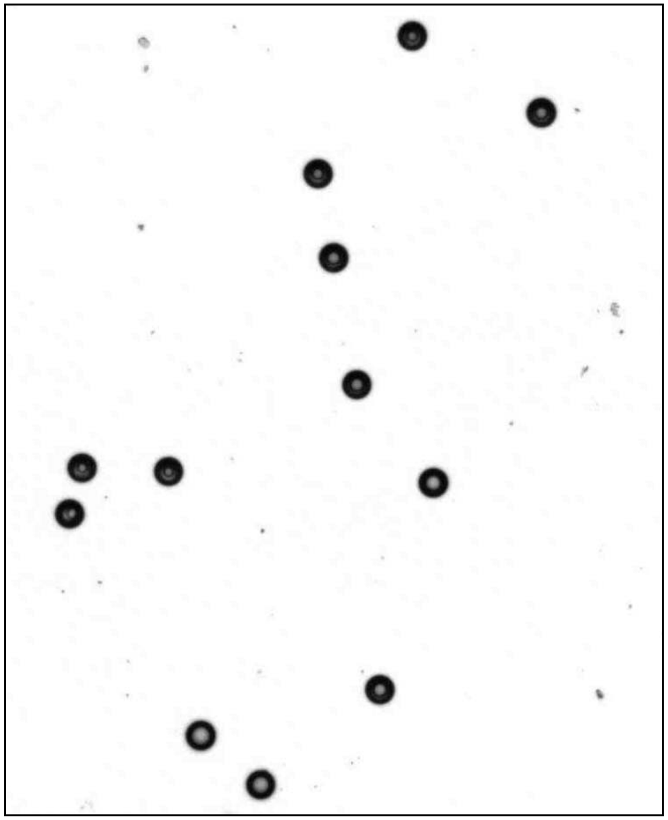
Dispersing a Sample

Want to spread particles out so that they don’t touch.

No



Yes



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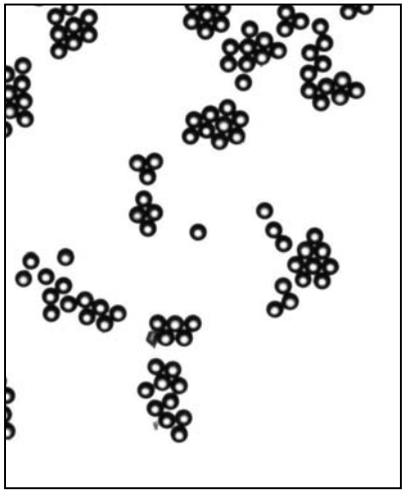
Control feed rate.

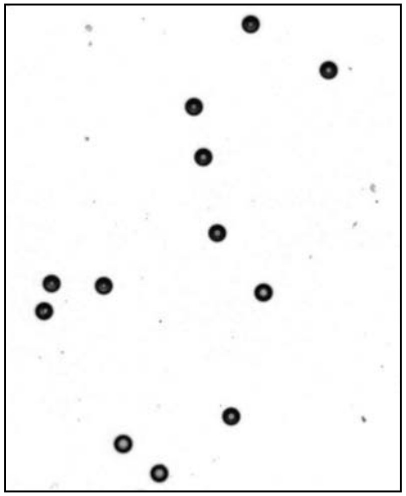
Want to spread particles out so that they don't touch.

Use % of field of view that is covered in order to control feed rate. Try 1% at first.

Feeding Too fast



Good



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Measurement Error Sources

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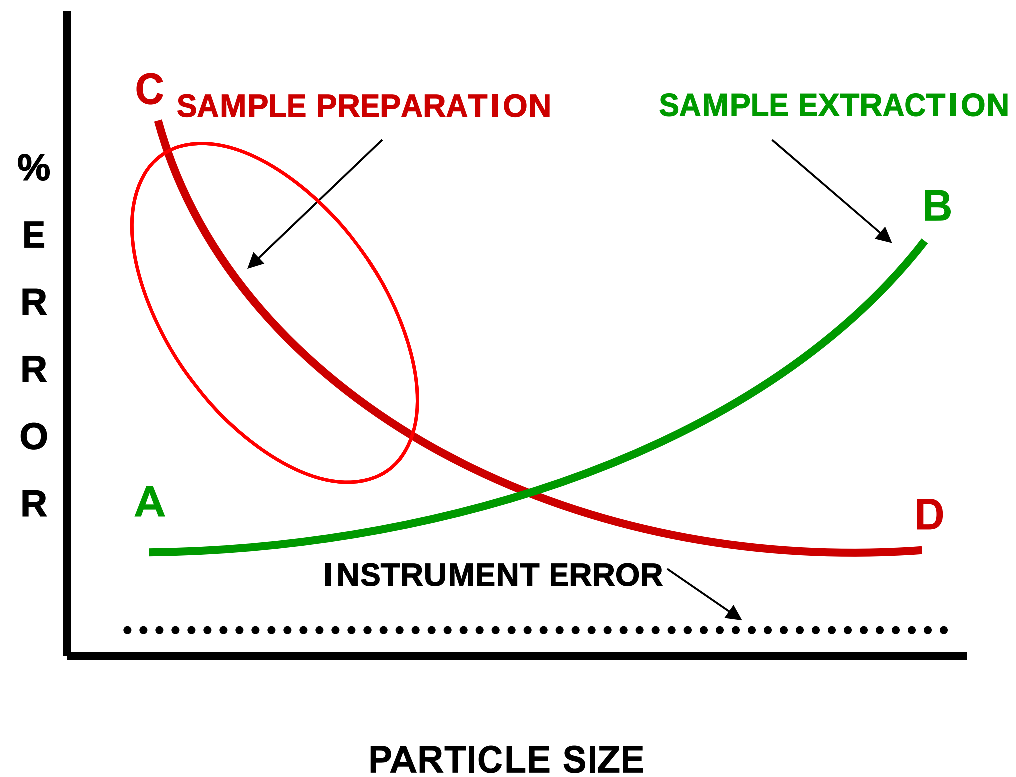
SMALL PARTICLES

- POTENTIALLY SMALL EXTRACTION ERRORS (A)
- POTENTIALLY LARGE SAMPLE PREP ERRORS (C)

LARGE PARTICLES

- POTENTIALLY LARGE EXTRACTION ERRORS (B)
- POTENTIALLY SMALL SAMPLE PREP ERRORS (D)



INSTRUMENT ERROR IS SMALL AND RELATIVELY CONSTANT

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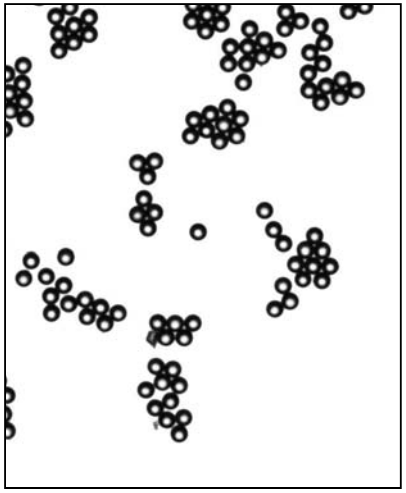
Micronized Powders

Want to spread particles out so that they don't touch.

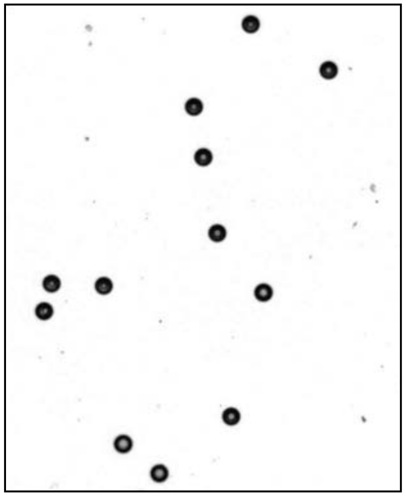
Small and sticky particles will stay clumped together. Need to use energy to separate particles. That means <u>air dispersion</u> or liquid dispersion.

~~**Feeding Too fast**~~

Not enough dispersion energy



Good



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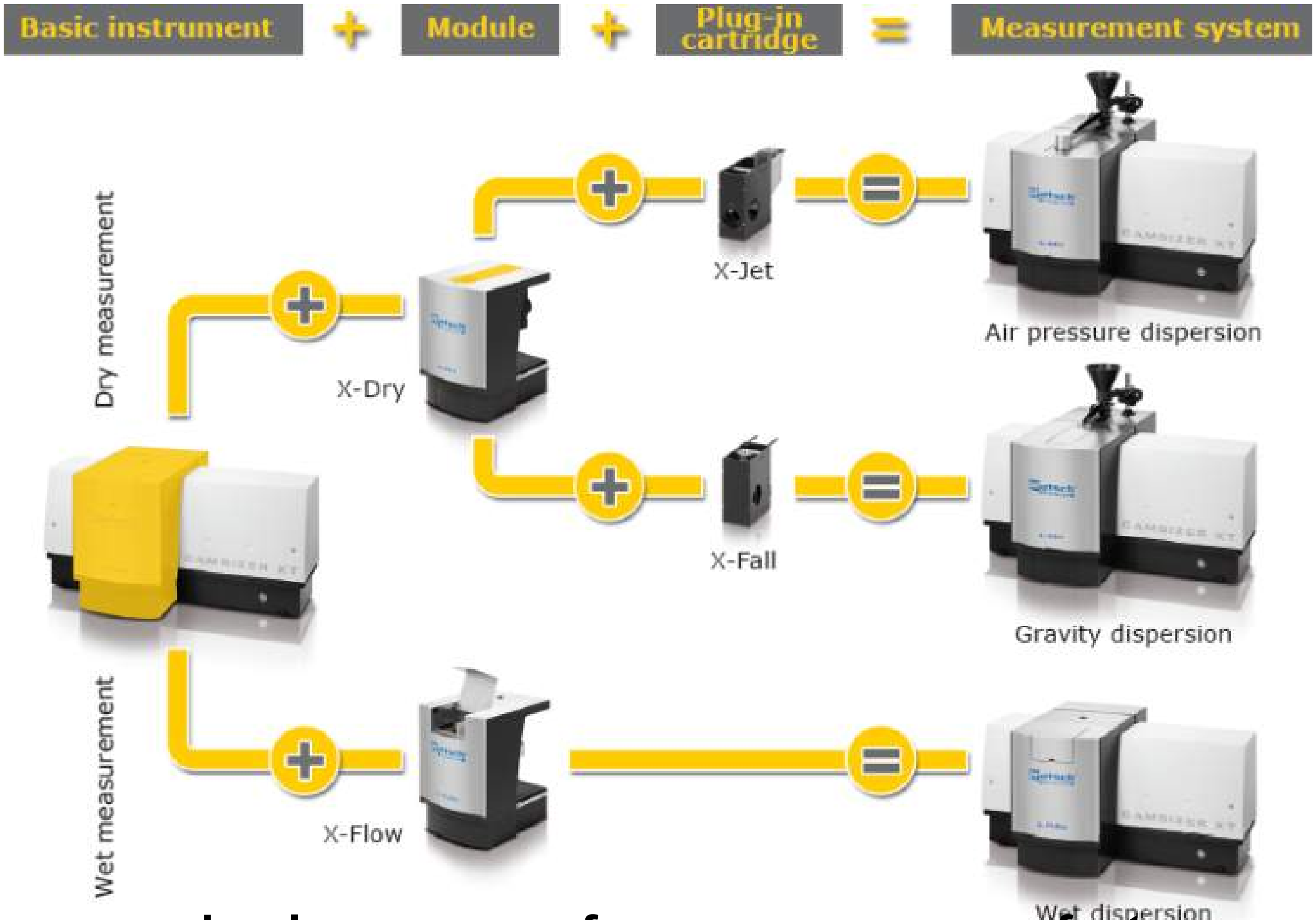
Modular "X-Change" Concept

Flexible configuration for a wide application range



simple • safe • fast

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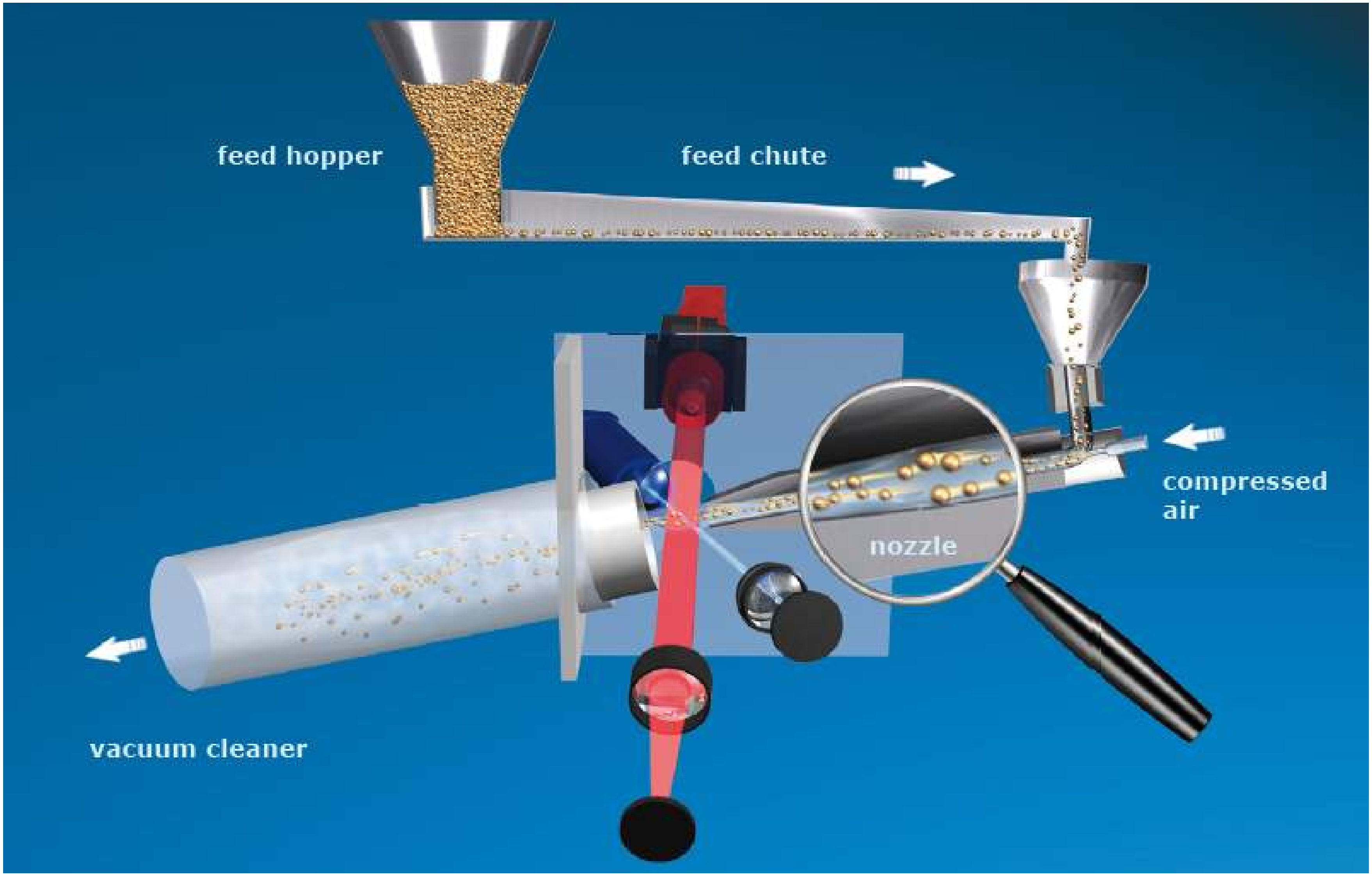
Measurement principle – X-Jet



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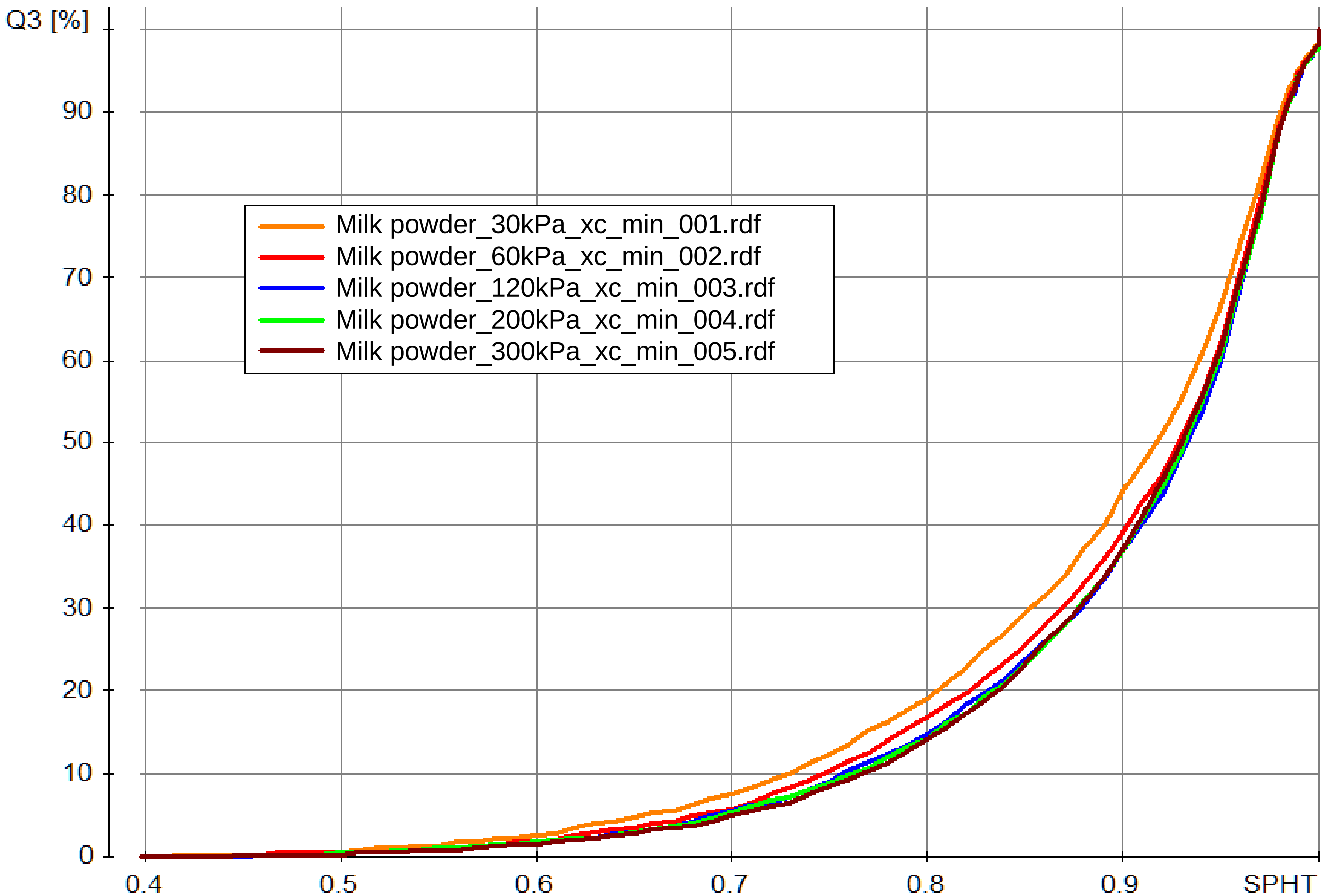
X-Jet Measurement Results

Influence of dispersion on shape parameters



Milk powder with *X-Jet* at 30, 60, 120, 200, 300 kPa

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Resolution Difference

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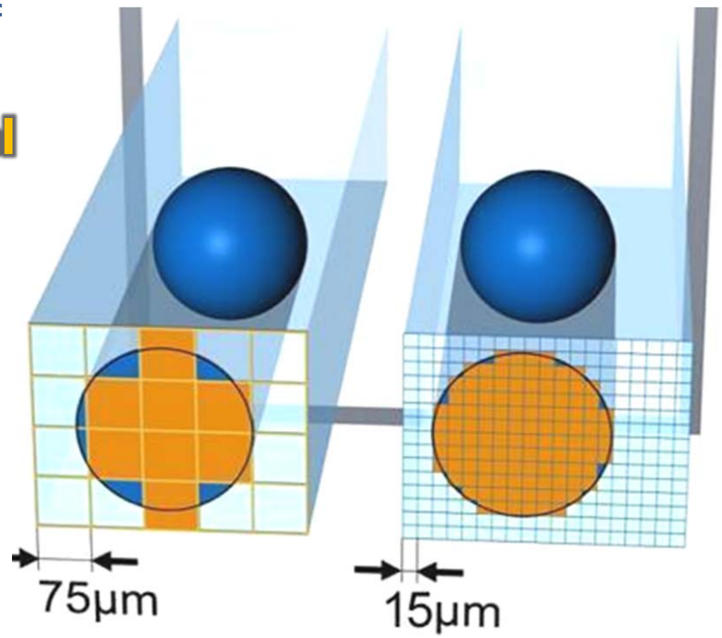
Detection of particles

One pixel is element of a projection when **at least half of the pixel** is covered.

CCD - Basic

CCD - Zoom



	CCD - Basic	CCD - Zoom	Size Range
CAMSIZER	75µm	15µm	30 µm -> 30 mm
CAMSIZER XT	14µm	1µm	1 µm-> 3 mm

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Measurement Range

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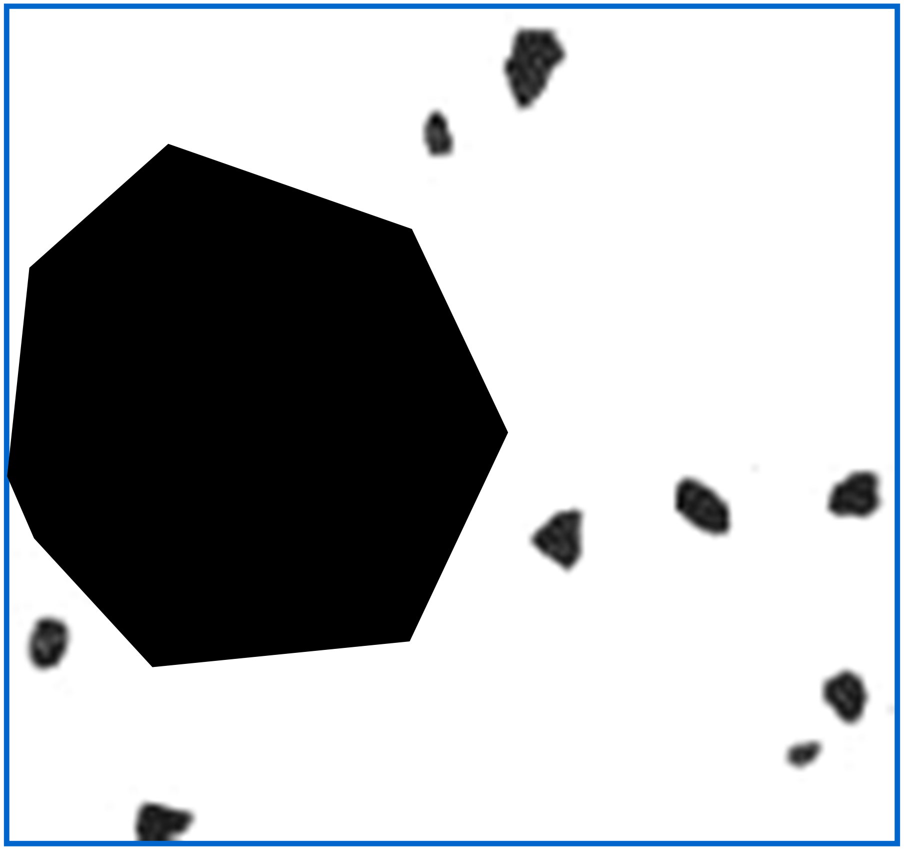
Large particles cannot been measured properly even they fit in the frame.



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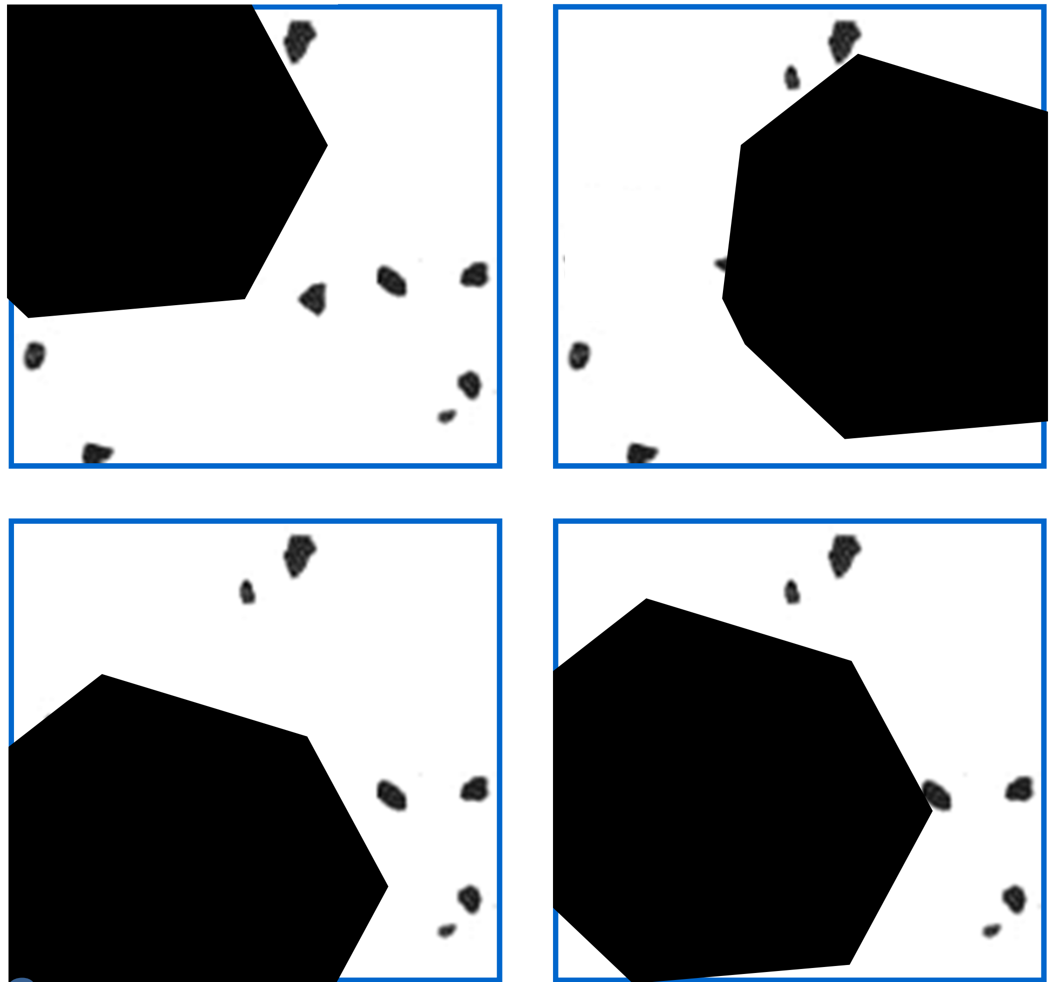
Measurement Range

The probability of large particles touching the edge of the frame is higher than for smaller particles.

=> Large particles cannot been measured sufficiently

→ Upper limit of measurement range



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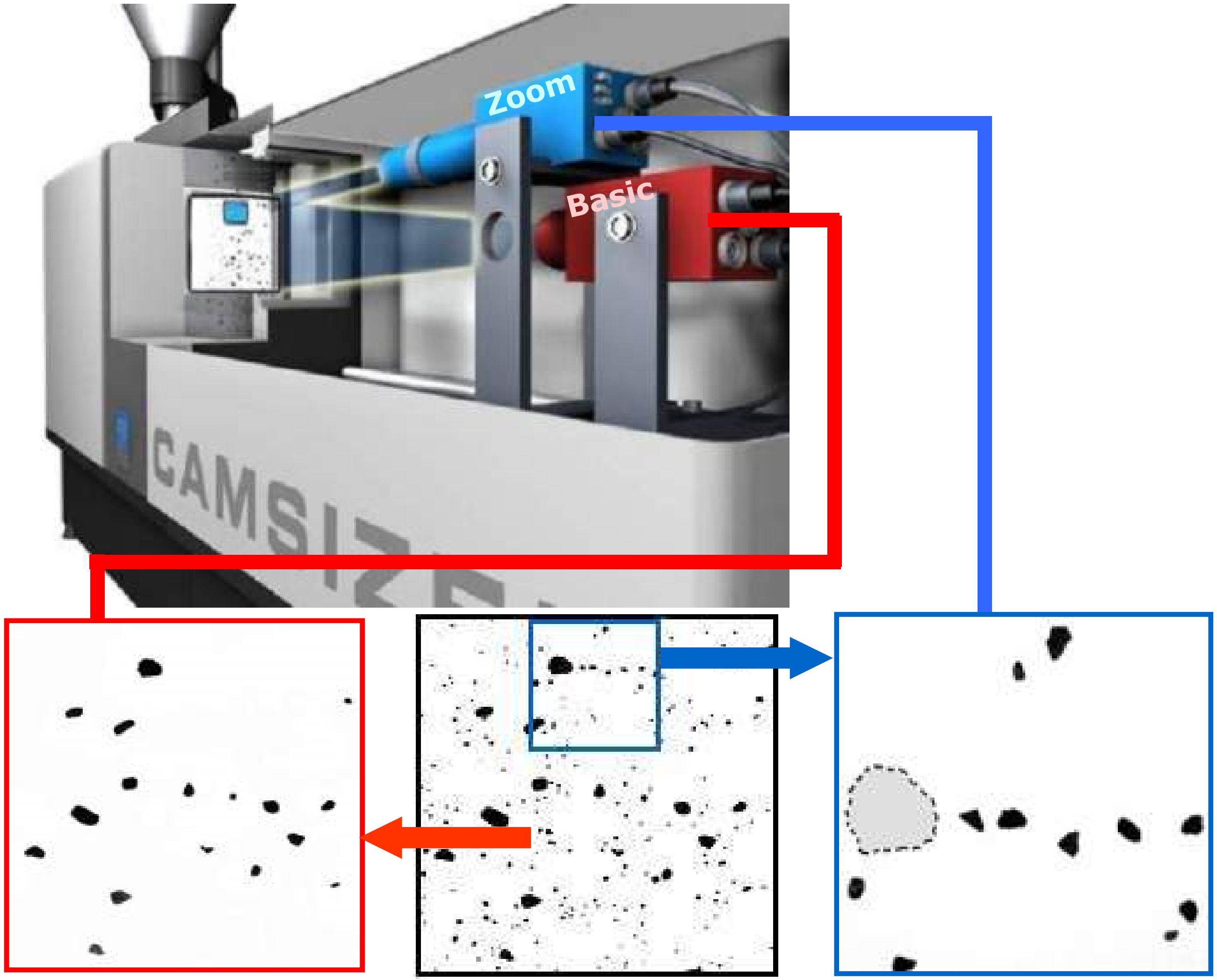
Two-Camera-System



Basic-Camera

Zoom-Camera

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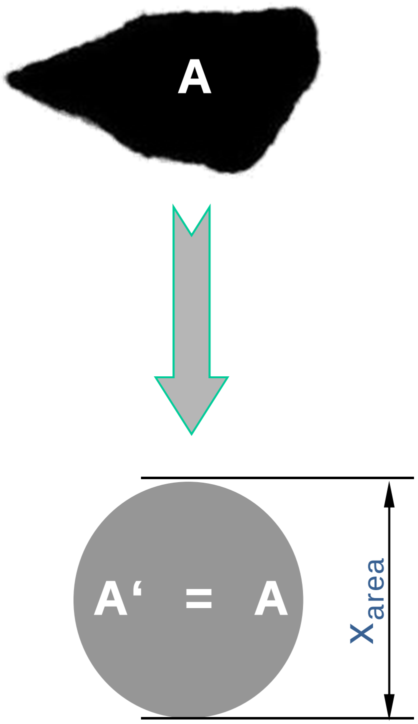
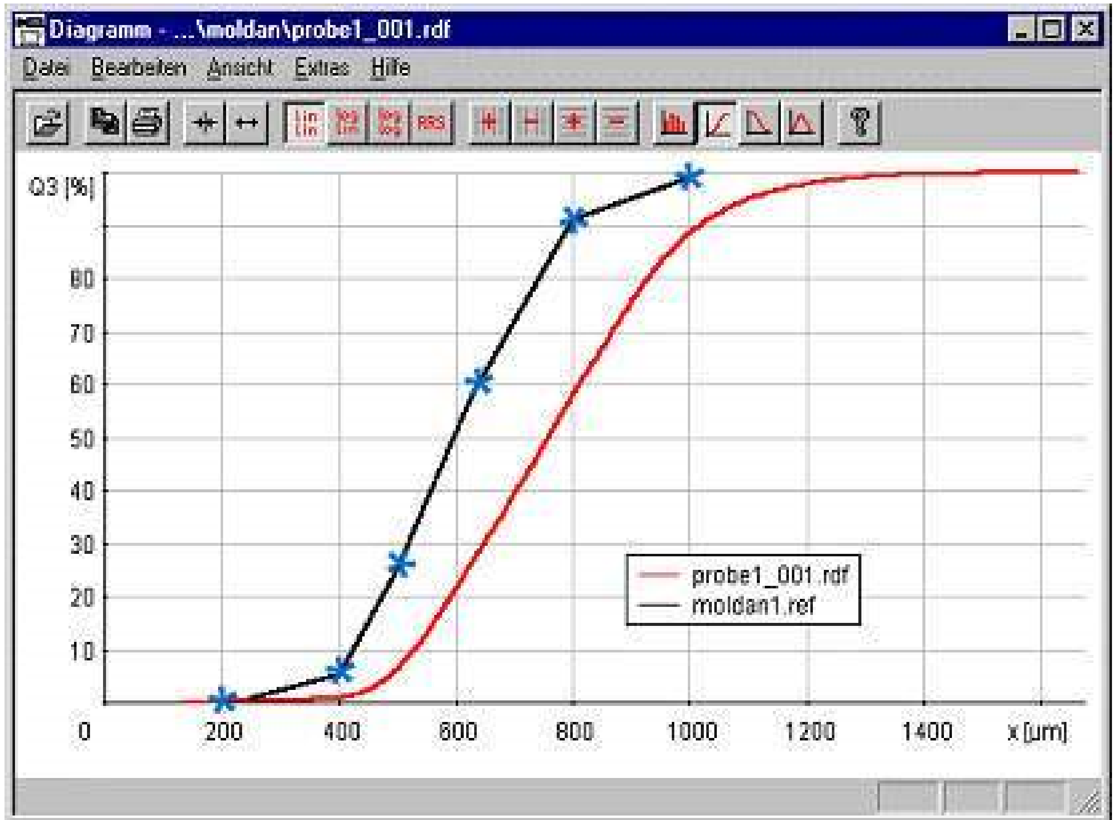
Digital Image Processing

Area Measurement ⬄ Sieving



x_{area}

"diameter
via projection surface"



comparison

CAMSIZER-measurement x_{area} (**red**)
and sieving * (**blue**)

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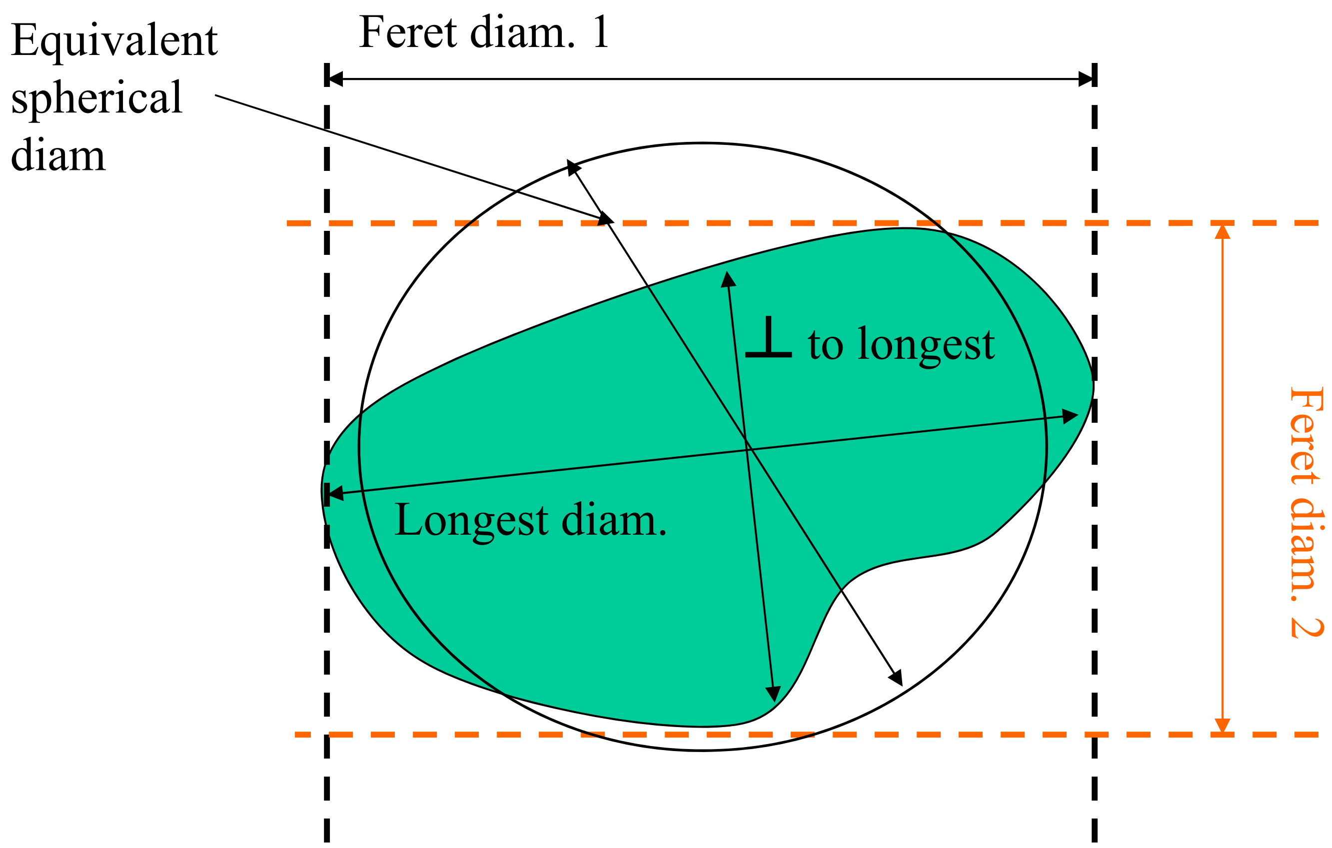
Size Descriptors



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Shape: Aspect Ratio

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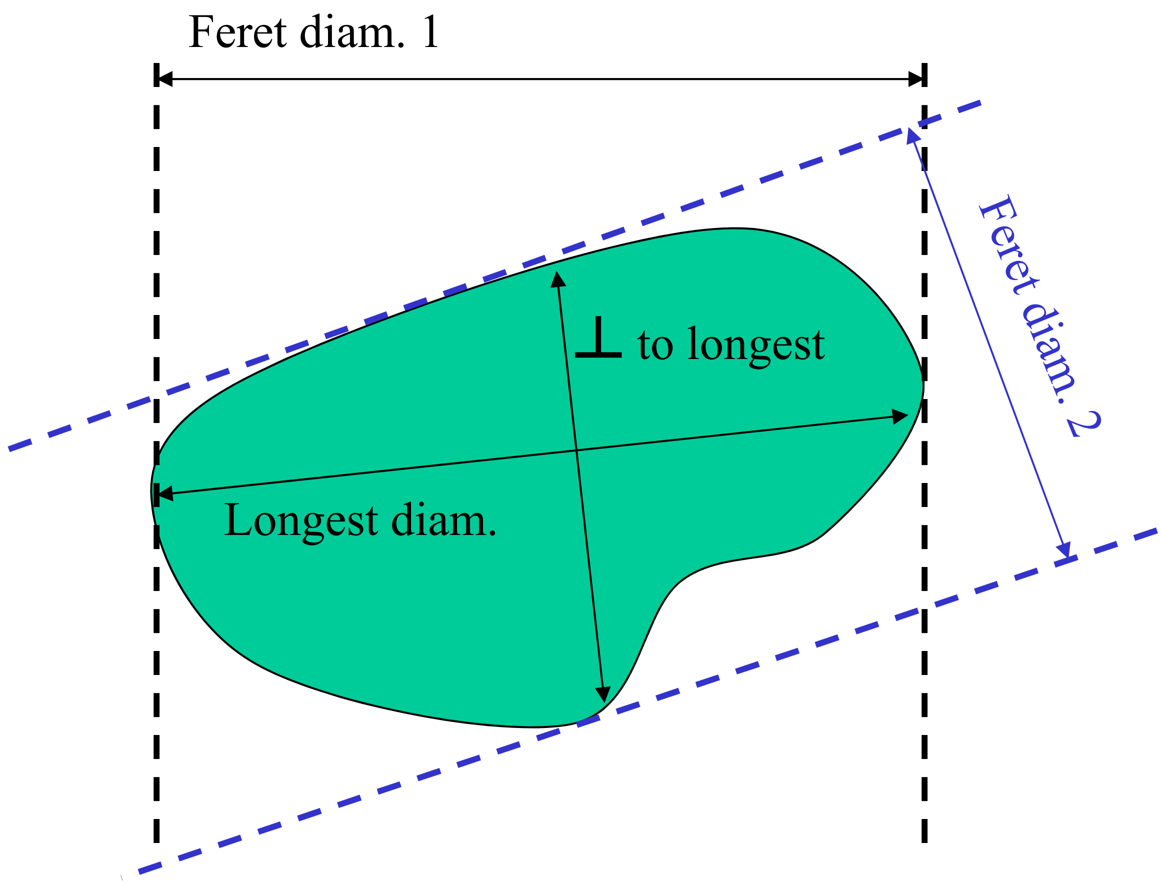
Aspect ratio

= shortest diam / longest diam

= ⊥ to longest diam / longest diam

= shortest Feret diam / longest Feret diam

= three different numbers!



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More Shape Descriptors

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Roundness

A shape measure that quantifies the "roundness" of an object's edges:

$$\frac{4 \times \text{Area}}{(\pi \times \text{L} \times \text{L})}$$

Compactness

Ratio of area over convex perimeter:

$$\frac{4\pi \text{ A}}{\text{Convex perimeter}^2}$$

Roughness

A shape measure that quantifies the jaggedness of an object's edges:

$$\frac{\text{Convex perimeter}}{\text{Perimeter}}$$

Fractal Dimension

Numerical characterization of irregular contours through fractal geometry.

$$P = P_{e}\delta^{1-D}$$

D is the Fractal Dimension, d is the unit length of the scale used for the measurement and P is the perimeter of the object (1<D<2).

Aspect Ratio

Ratio of length over width.

$$\frac{\text{Length of longest feret}}{\text{Length of shortest feret}} = \frac{\text{Length}}{\text{Width}}$$

Sphericity

Estimate of the sphericity of an object:

$$\frac{4\pi \text{ A}}{\text{p}^2}$$

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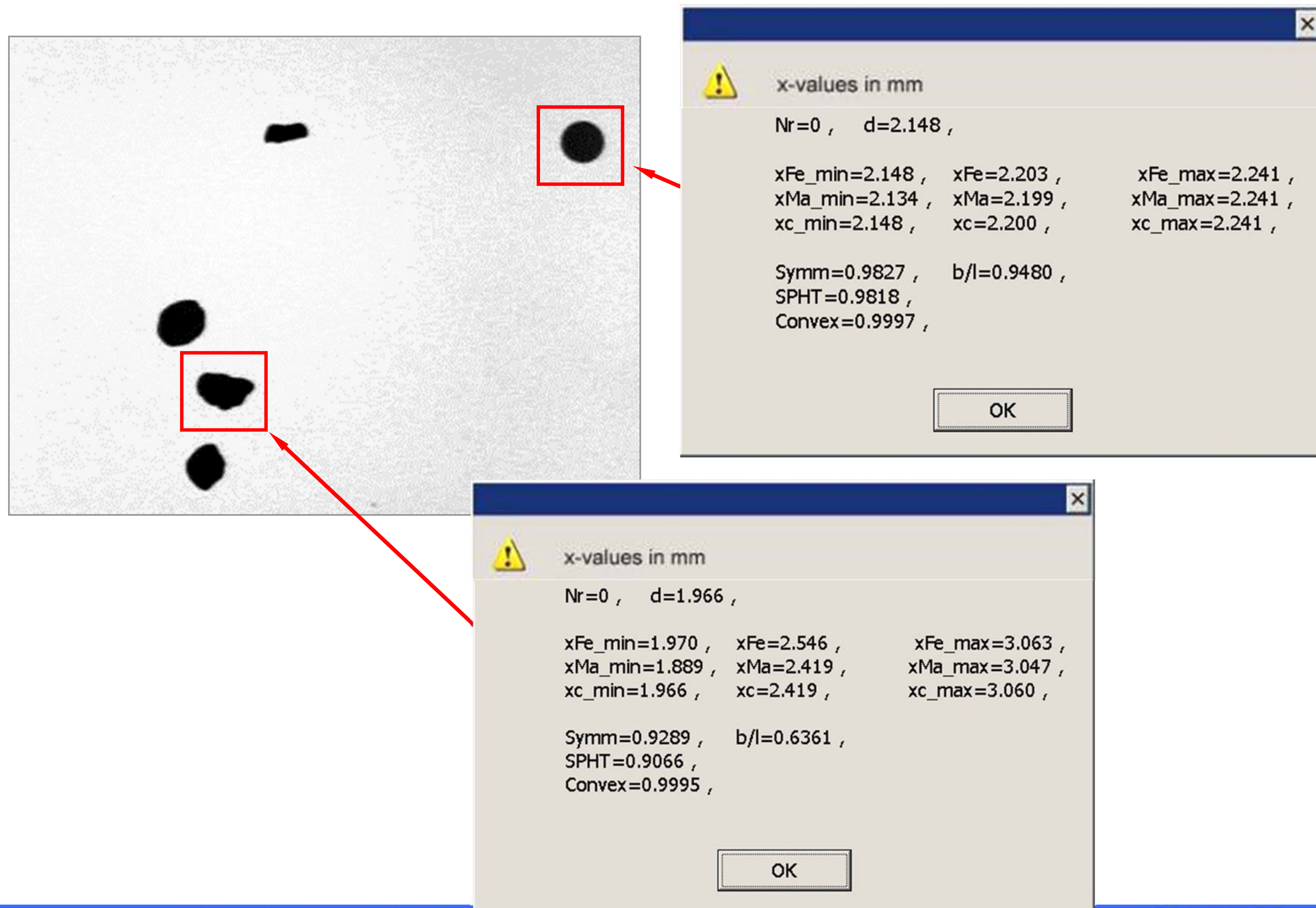
Optical Process Control

analysis for size and shape



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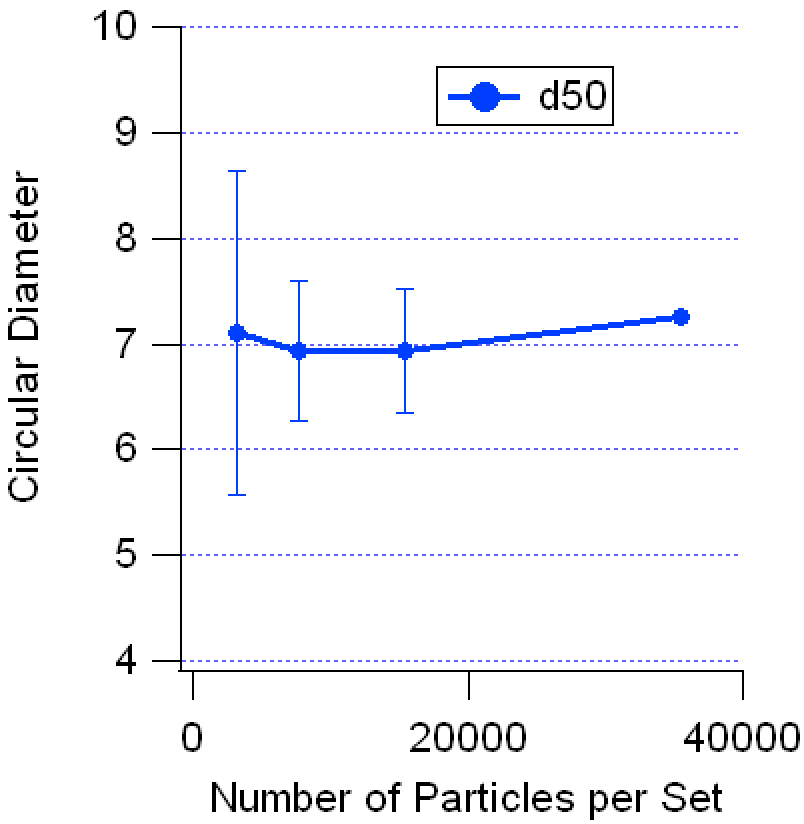
Number of Particles



- Error bars are one standard deviation from repeated measurements of the same number of particles from different parts of the sample.
- The error bars get smaller as you evaluate more particles.

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CAMSIZER Advantages

- Measurement of very broad particle distributions (due to speed and two cameras)
- Direct particle definition
  - by width (analogue to sieving)
  - by length
  - or projection surface
- Two camera system for more accuracy and reproducibility
- Easy operation
- Fail-safe, robust
- Ideal for particle shape analyses
- Measurement of density, counting of particles

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Watch out for:

- Sample preparation
- Image quality
- Measure enough particles

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What we’ll talk about

- Importance of particle shape
- Dynamic image analysis principles
- Examples
- Q&A

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Product Performance

- Some products perform better when more spherical
  - Glass beads for highway paint
  - Proppants
- Some products perform better when less spherical
  - Abrasives
  - Catalysts
- Finding particles by shape

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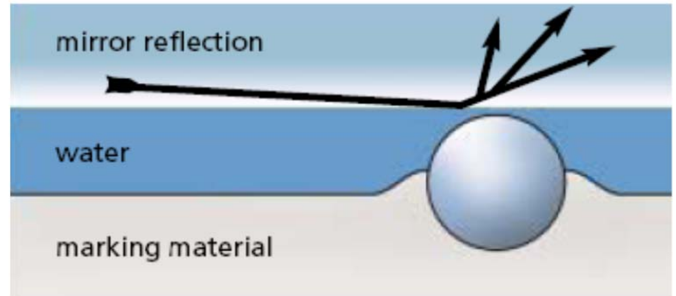
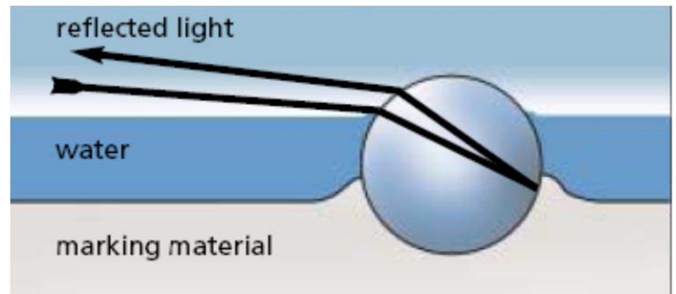
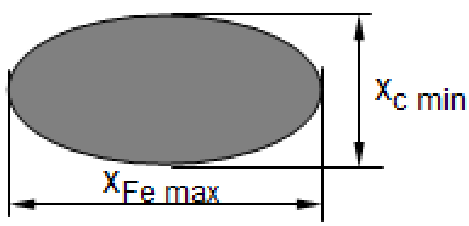
Glass Beads for Highway Paint

- Size and shape critical to reflective properties
- More round = more reflectivity back to source
- CAMSIZER uses b/l ratio to quantify roundness



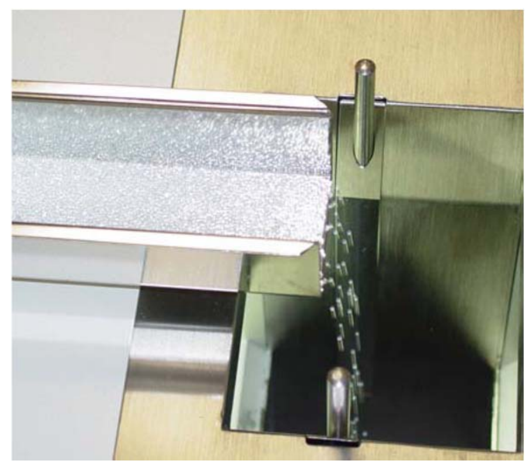


- **Breadth-/ Length-ratio**



Dry

In paint



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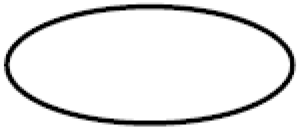
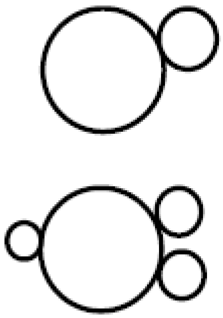
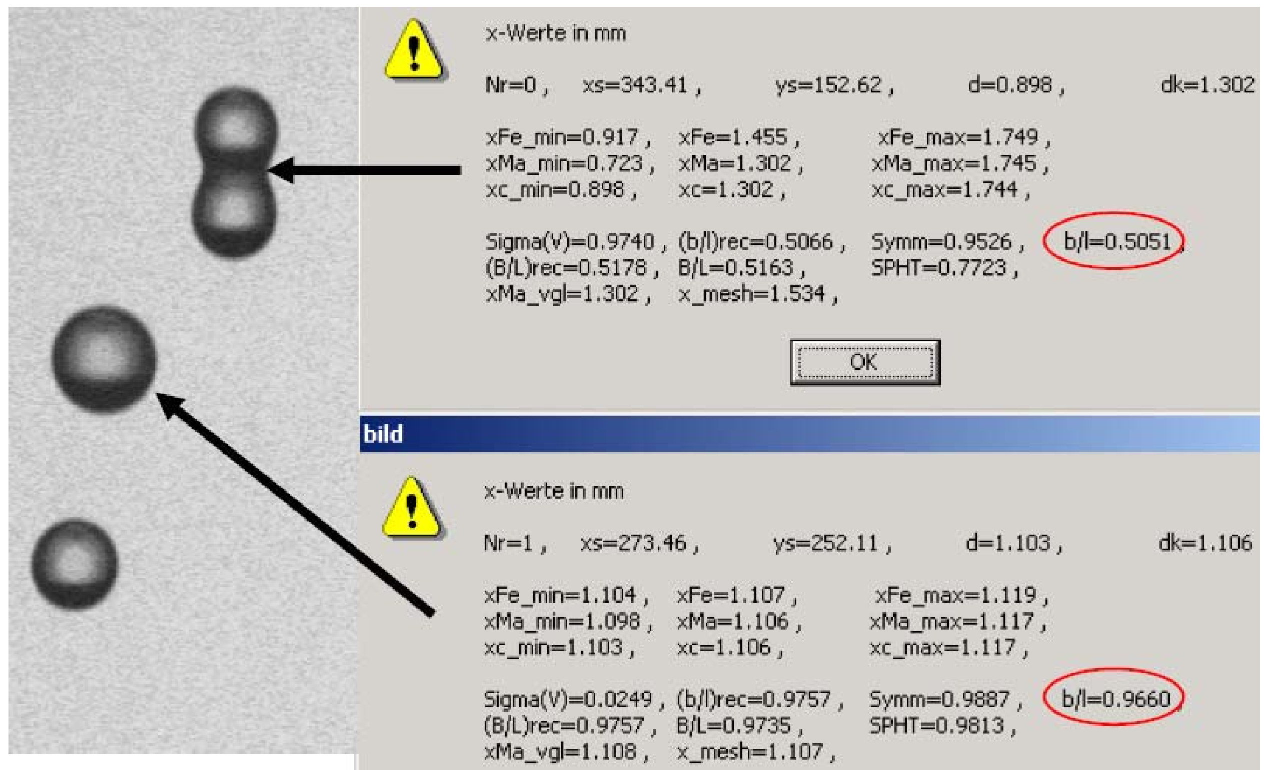
Glass Beads for Highway Paint

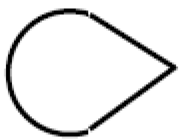
Defects from round particles

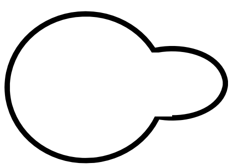
Quantified by Camsizer

Oval


Satellites




Pointed


Aggregates


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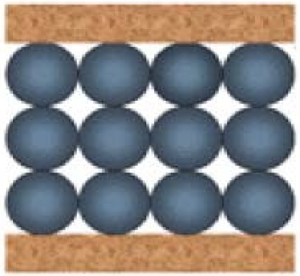
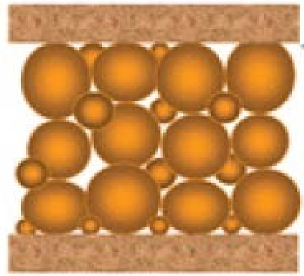
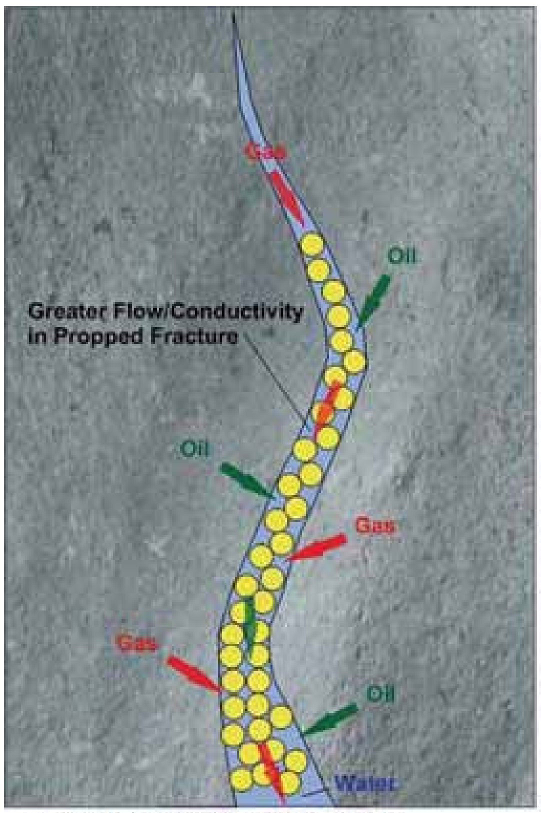
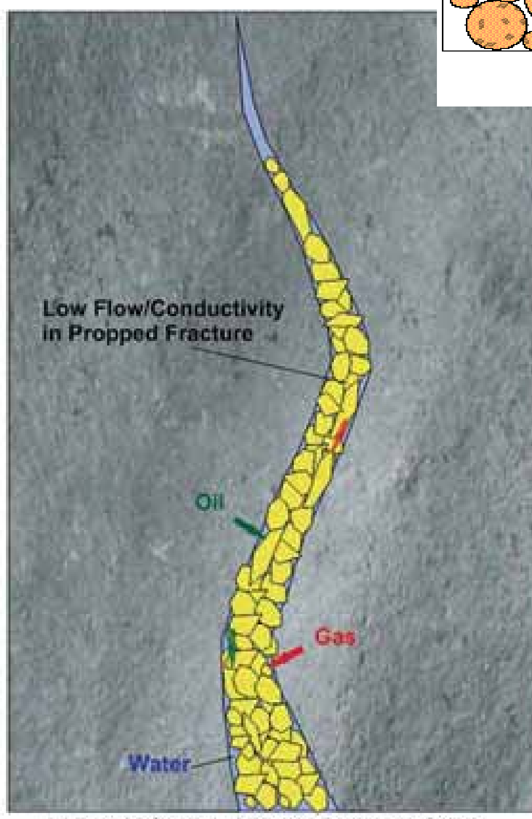
Proppant Packing



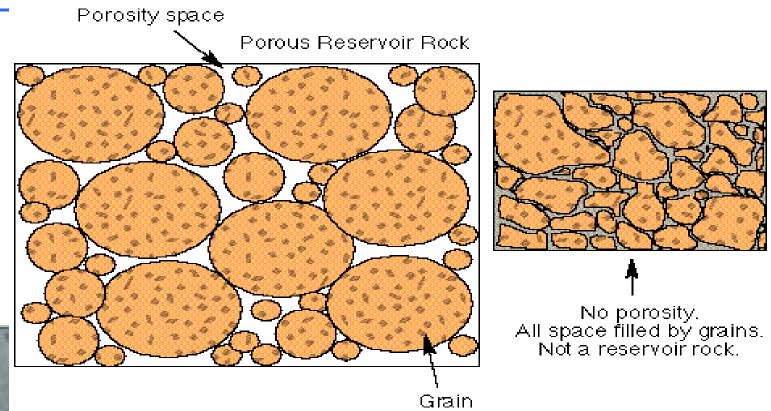


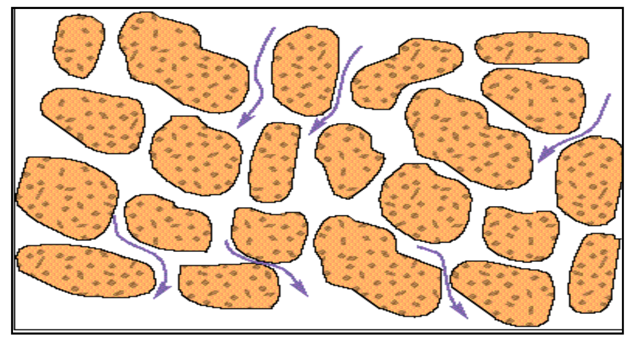


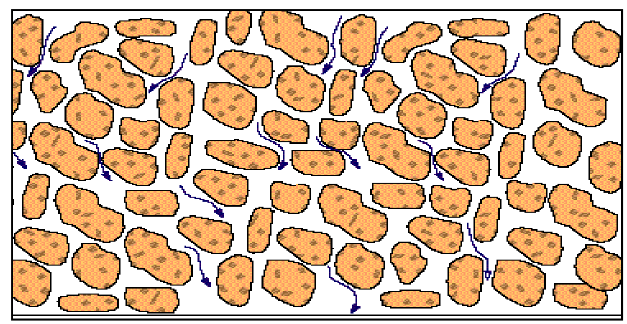
a. Well Rounded Ceramic Proppant



b. Poorly Sorted Angular Proppant Sand







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Shape Comparison

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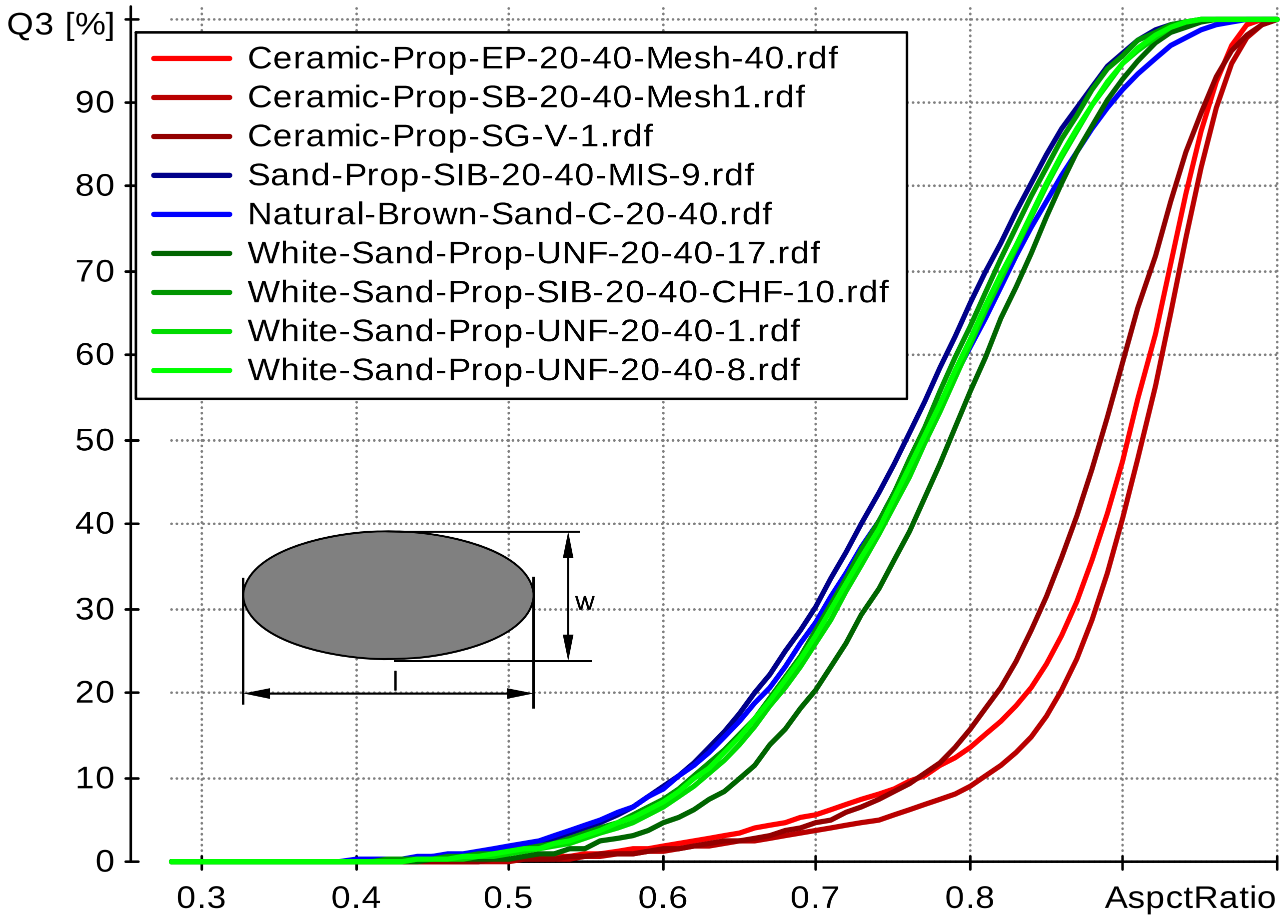

Shape comparison between natural **sand proppants** and **ceramic proppants**. There are two clearly different ranges of Aspect Ratio (**Krumbein's Sphericity**). Analysis of other shape parameters are possible as well (Convexity for ceramic bead twins, Symmetry for good and broken ceramic beads, **Krumbein's Roundness** etc.)

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Proppants


Sand proppant

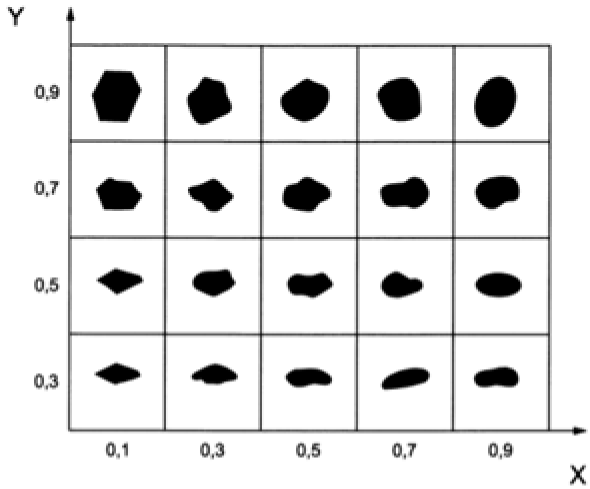
Proppant	Shape	Strength	Conductivity
Ceramic	Uniform	High	High
Resin coated sand	Irregular	Medium	Medium
Sand	Irregular	Low	Low


Resin coated sand

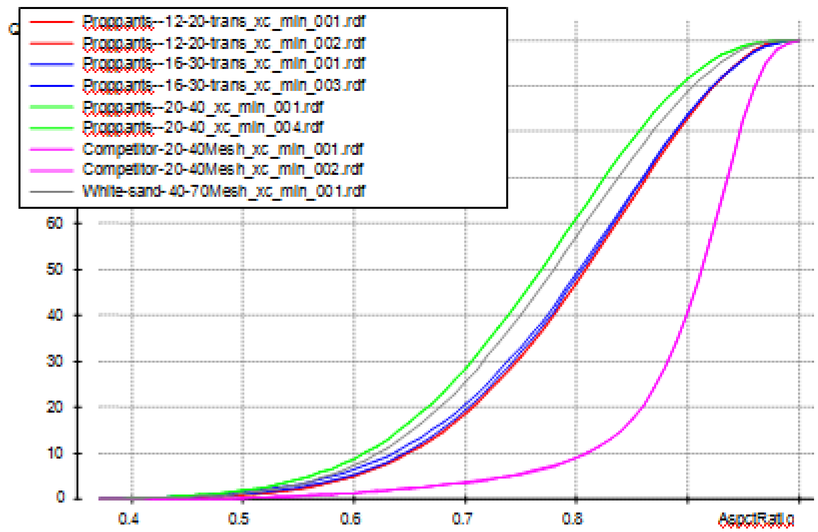

Ceramic proppant


Resin coated ceramic proppant

Traditional method



CAMSIZER



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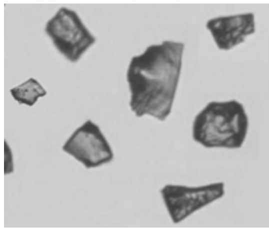
Abrasion Mechanics

- Difference in hardness between the two substances: a much harder abrasive will cut faster and deeper
- Grain size (grit size): larger grains will cut faster as they also cut deeper
- Grain shape: sharp corners help some mechanisms
- Compactness helps in others



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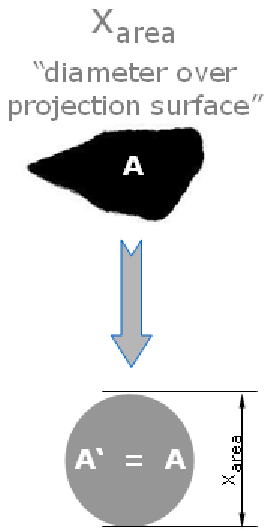
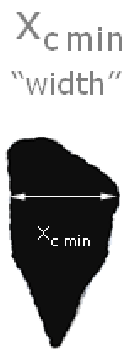
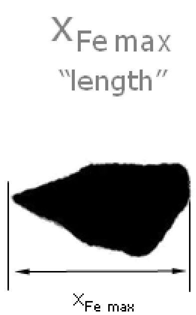
Dynamic Image Analysis

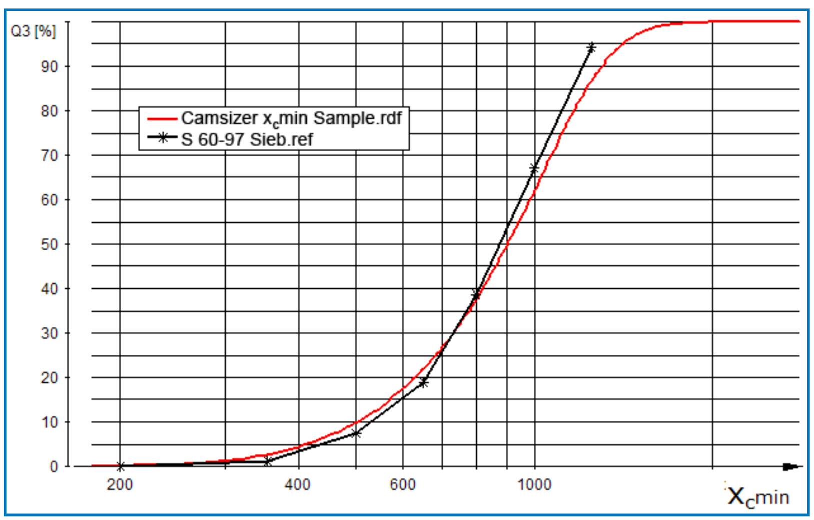




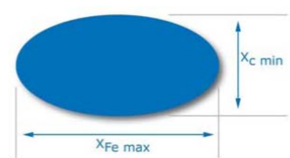
By choosing proper size parameter, Xc min, results can match historic sieve Data. Also generates shape data proven To correlate with abrasive performance.

Roundness; Sphericity $\frac{4\pi A}{P^2}$





Elongation; Width-/Length-Ratio $\frac{X_{c\ min}}{X_{Fe\ max}}$



Convexity $\sqrt{\frac{A_{real}}{A_{konvex}}}$

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Sand: Round vs. "Edgy"

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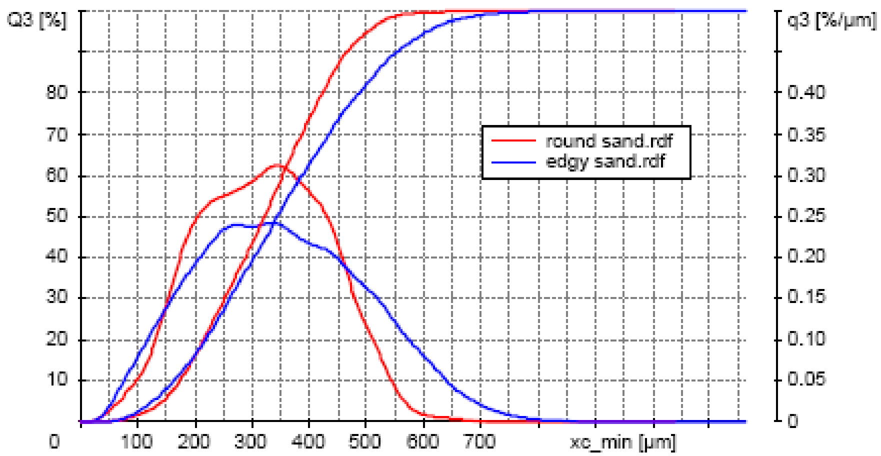
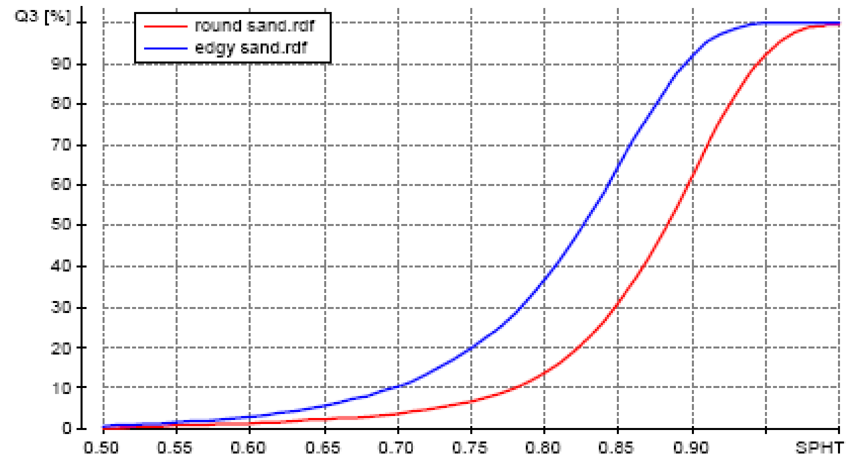
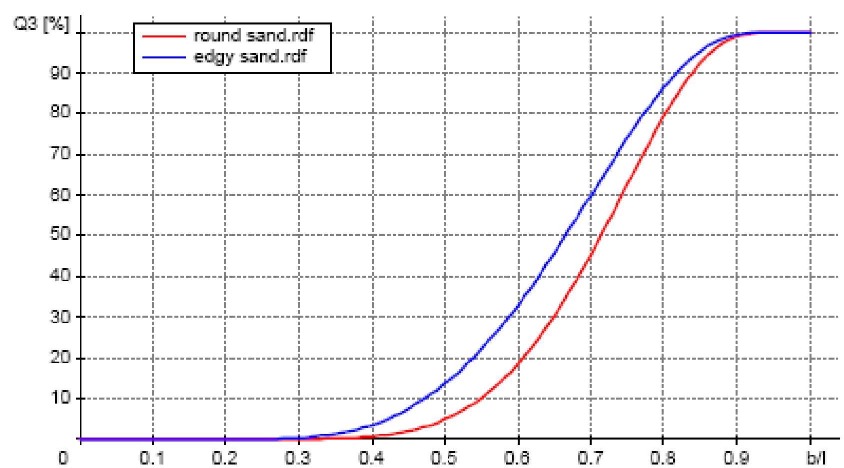


Similar in size.
Shape difference seen in b/l and sphericity.
Edgy would make better abrasive.



Sphericity



Breadth/length (b/l)

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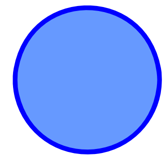
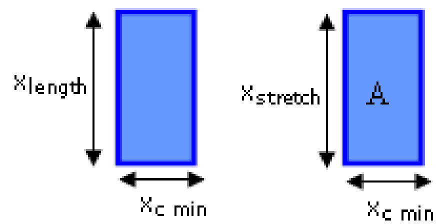
Catalysts Size/Shape by CAMSIZER

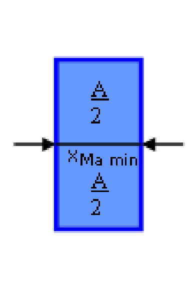
- Spherical catalysts
  - Easy, no special effort



- Cylindrical catalysts
  - Length, width



- Bended extrudates
  - Use other parameters



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

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

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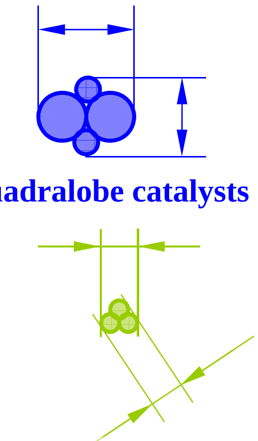
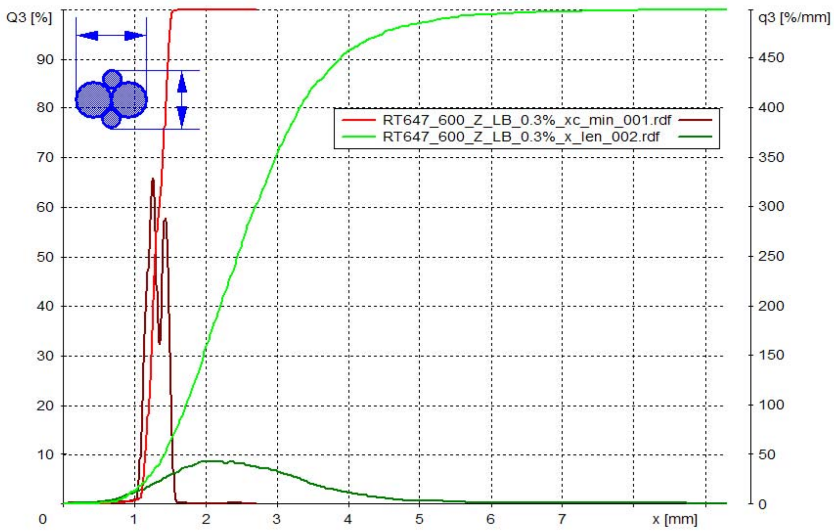
Catalysts Size/Shape by CAMSIZER

- Tri & quadralobe
  - Possible to distinguish between different diameters
  - Shorter green distribution = length
  - Taller maroon distribution= width
- Size helps define shape





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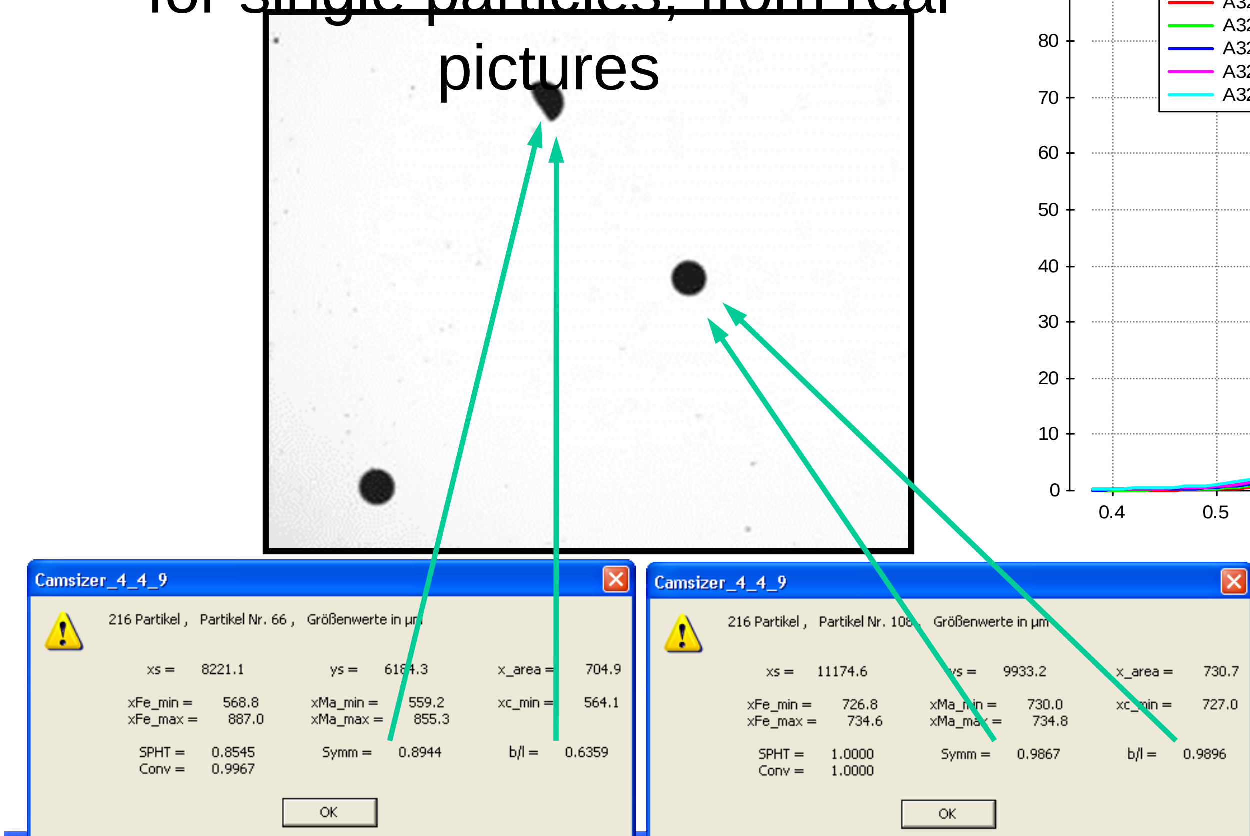
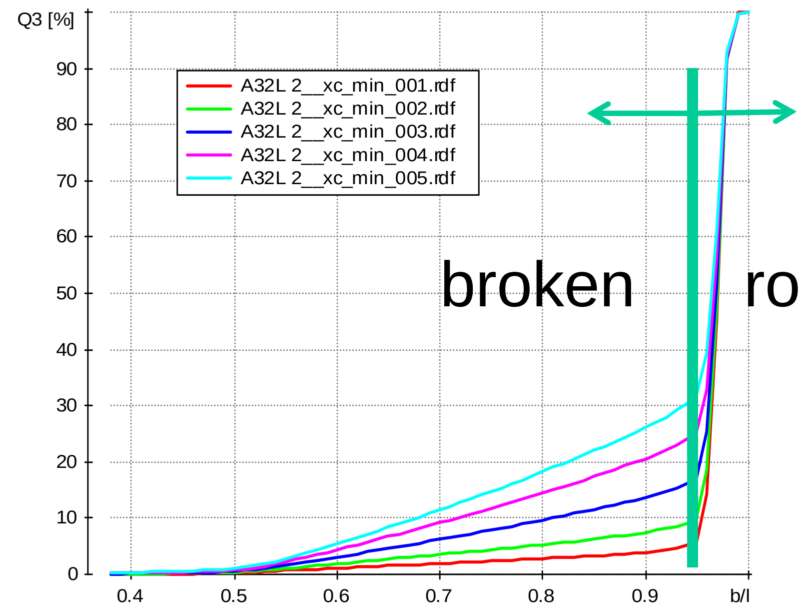
Results X-Flow

Shape analysis for detection of broken particles:
„What you see is what you get“

Length and shape numbers for single particles, from real pictures





Precise analysis of the amount of broken particles by shape detection

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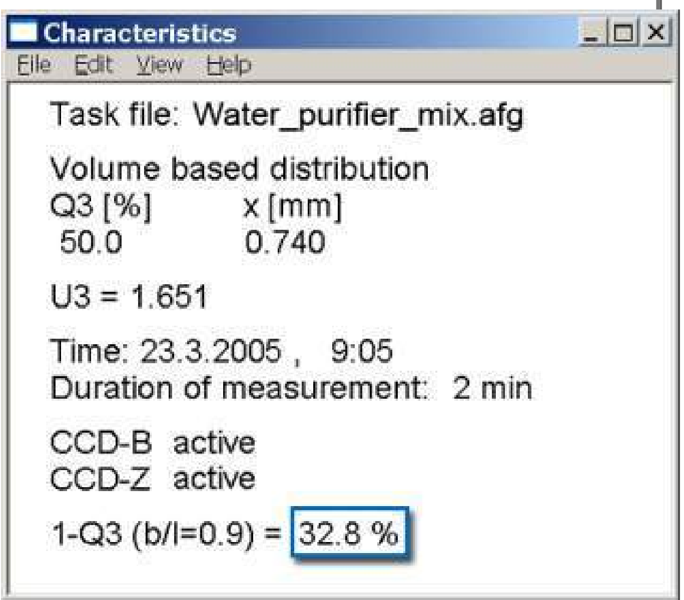
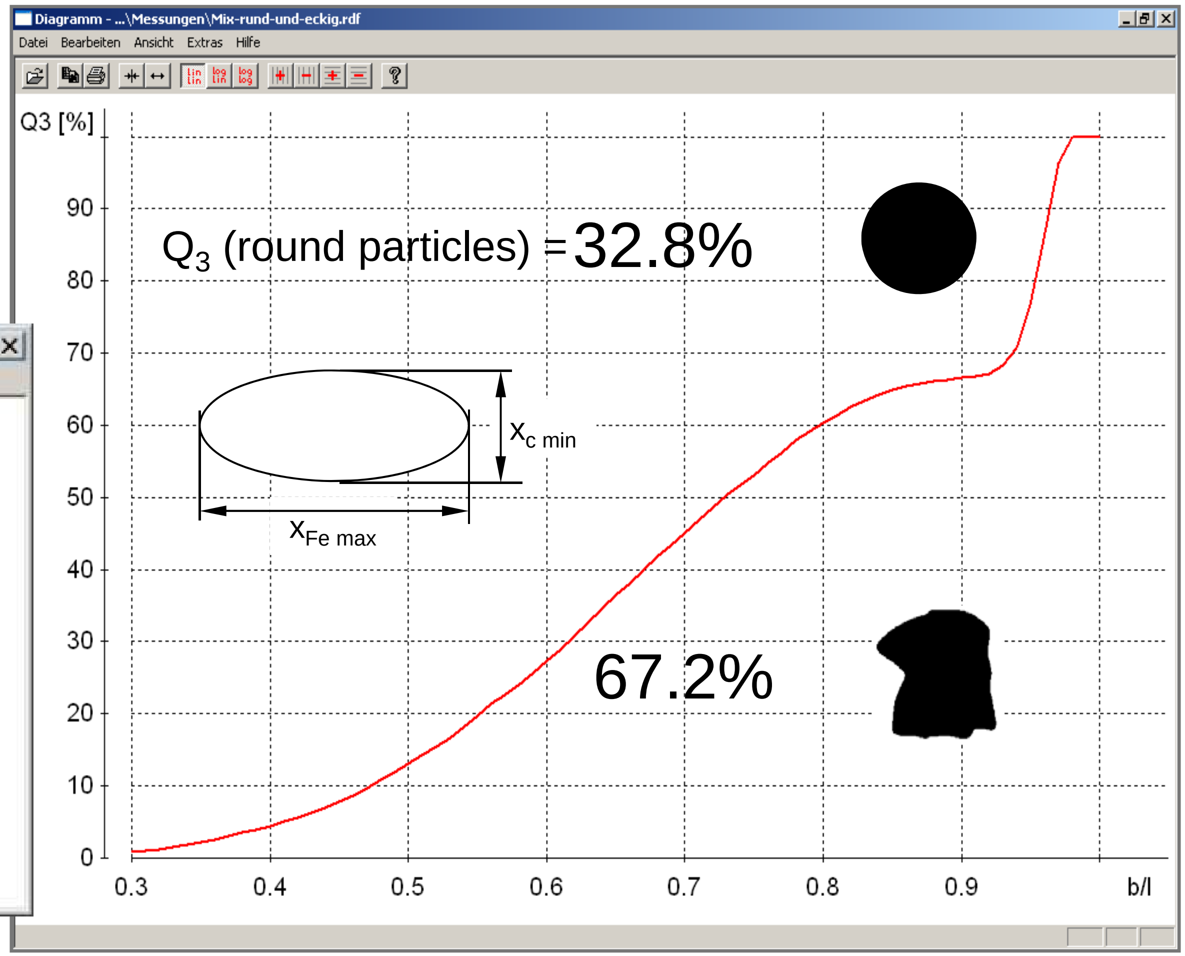
Find the componentsof Ion Exchanger and Charcoal in a mixture





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What we’ll talk about

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- Importance of particle shape
- Dynamic image analysis principles
- Examples
- Q&A

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Thank you

ありがとうございました

ขอบคุณครับ

谢谢

اشْكُر

Gracias

Grazie

Σας ευχαριστούμε

धन्यवाद

நன்றி

Tacka dig

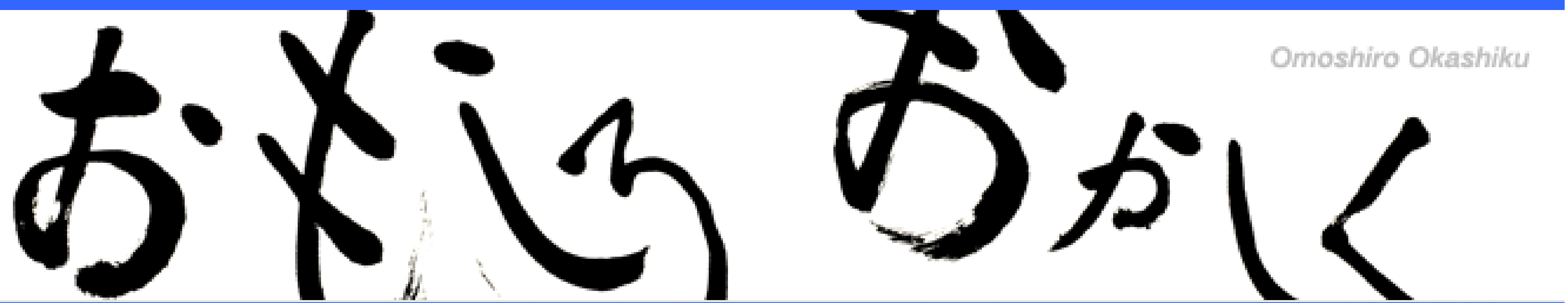
Danke

Merci

Obrigado

감사합니다

Большое спасибо



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