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The Importance of Particle Shape

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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

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

Powder Flow

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- 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

Powder Flow*

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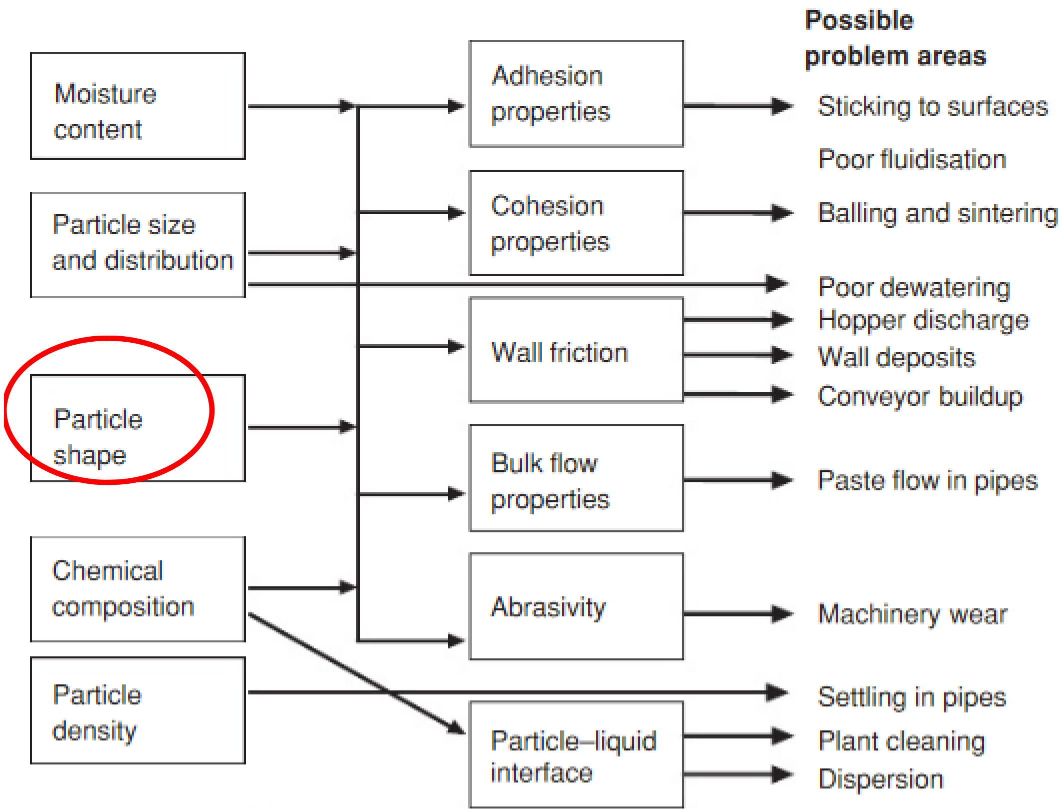
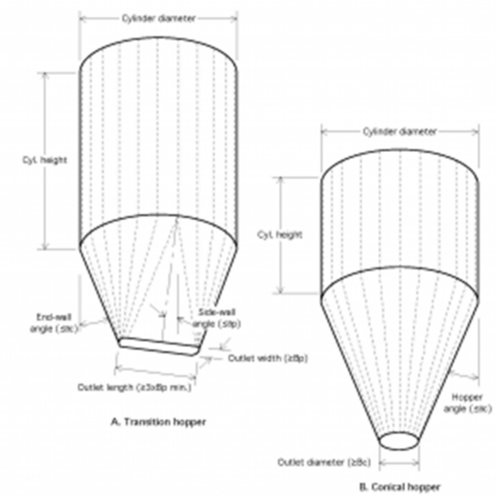
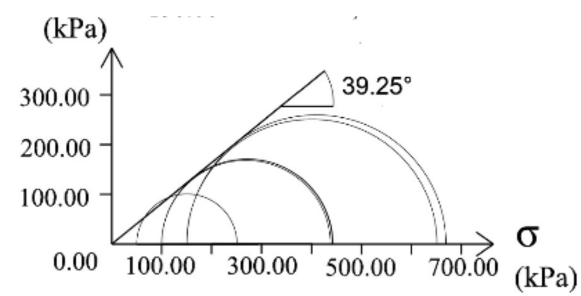




Θc =hopper angle

BC = outlet diameter



Angle of internal friction

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

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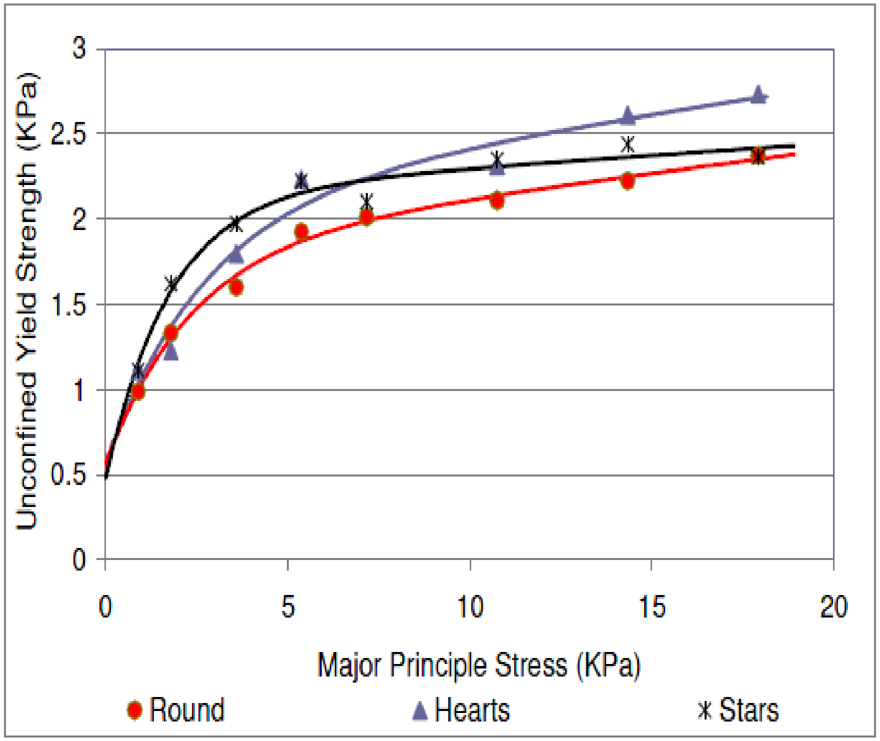
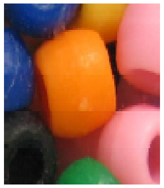
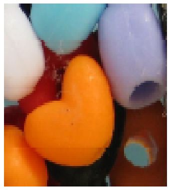
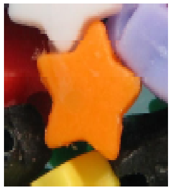
Powder Flow

- 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









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

Powder Flow*

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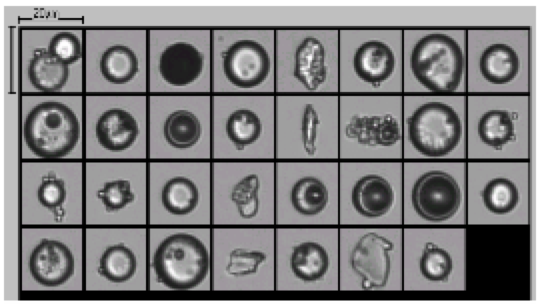
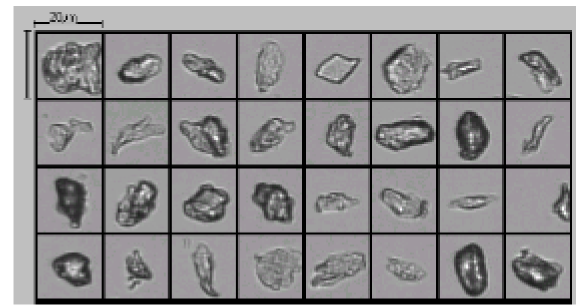


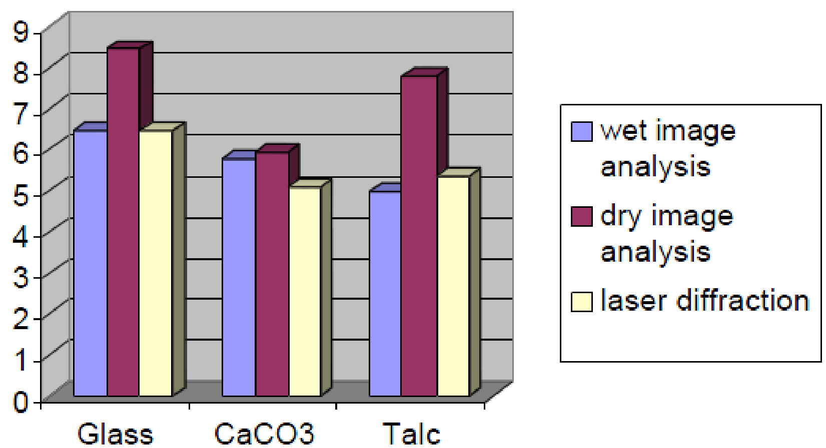
Glass spheres

$CaCo_3$

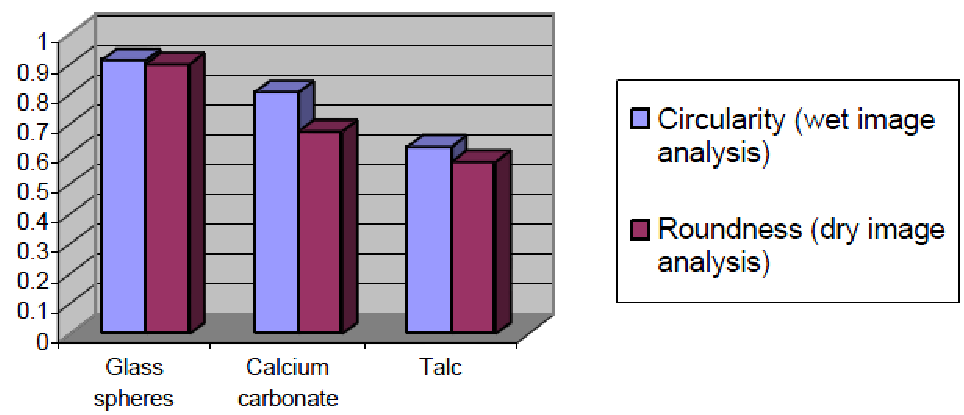


Talc

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

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

Powder Flow*

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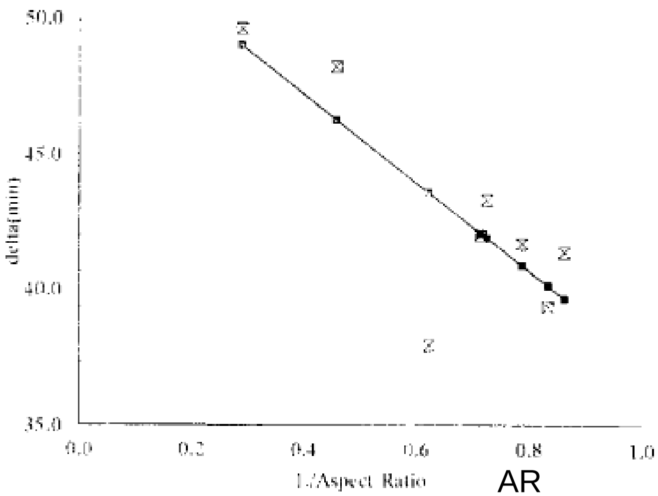
Powder	d_{vs} [μm]	Shape (BP)	AR	SF
Pregelatinized starch	103.2	Angular	1.38 ± 0.26	7.54 ± 0.35
Paracetamol	537.6	Angular	1.61 ± 0.65	7.38 ± 0.53
Calcium carbonate	4.6	Cubic	1.20 ± 0.20	7.66 ± 0.36
Potassium chloride	481.1	Cubic	1.27 ± 0.29	7.70 ± 0.56
Maize starch	49.2	Round	1.16 ± 0.12	3.86 ± 0.18
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

δ



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

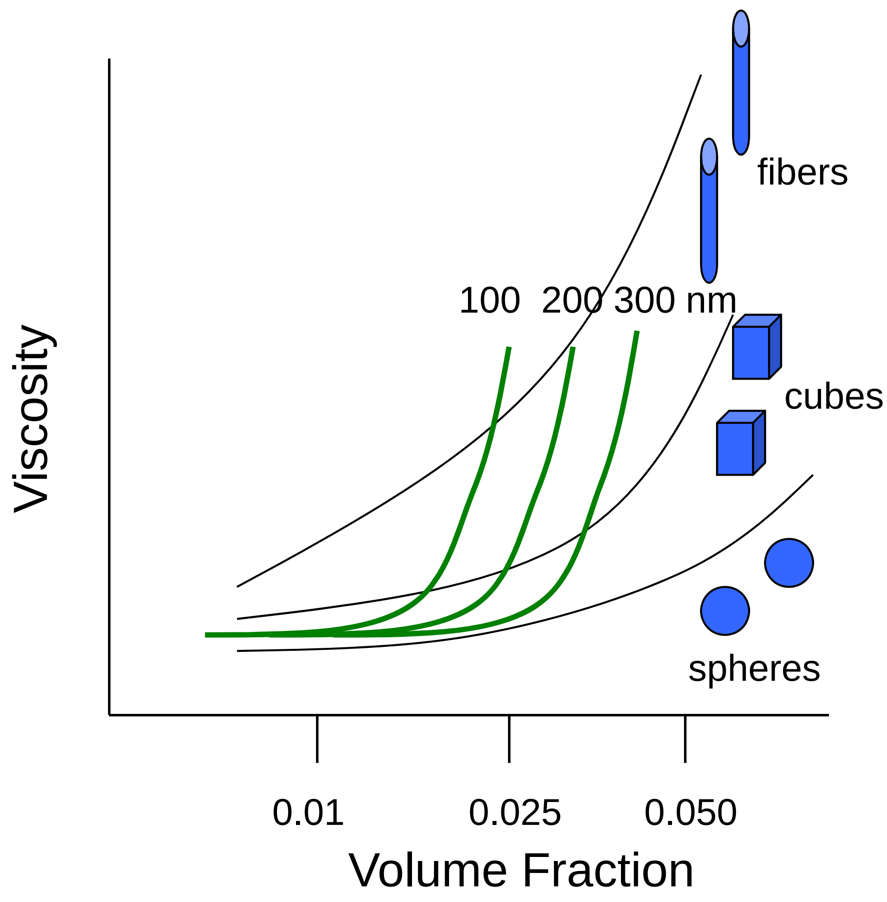
$$\delta = 0.347 \cdot e^{AR} - 2.434 \cdot \ln d_{vs} + 59.336$$

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

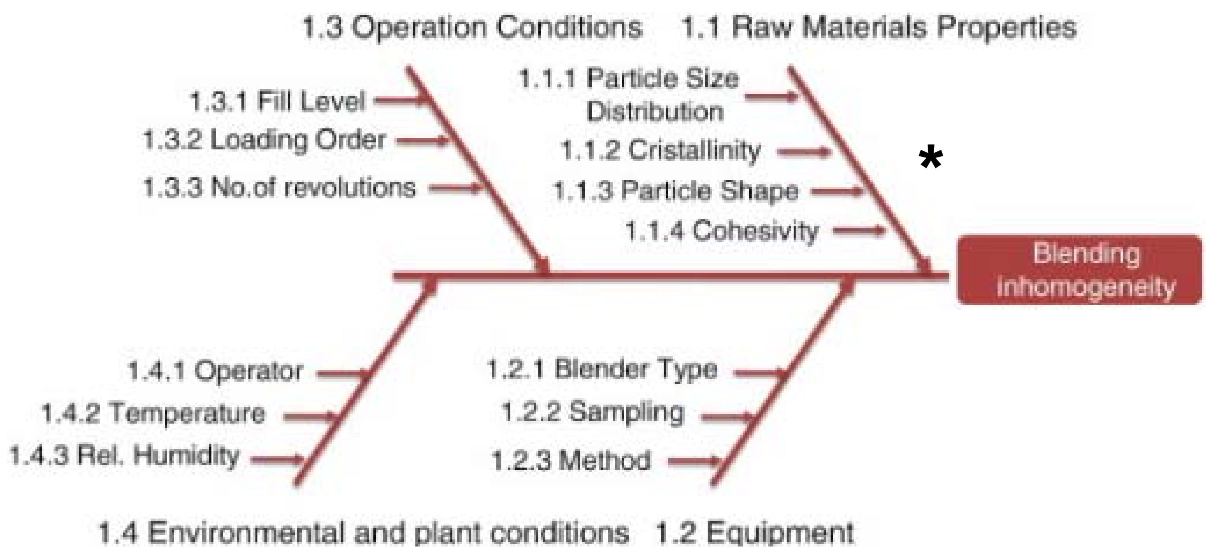
Rheology/Viscosity

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



Mixing

- Shape affects mobility and therefore causes segregation
- Smaller effect than size
- Shape differences but 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

Powder Mixing*

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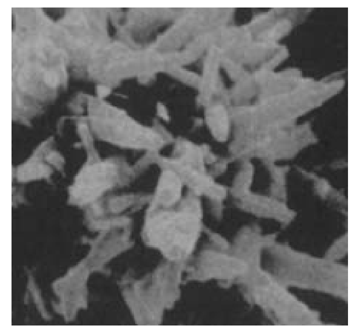
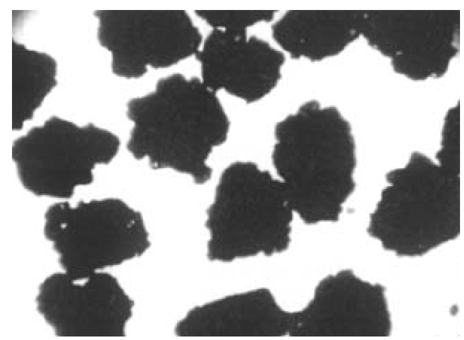
- Ordered mixtures of powders comprising a microfine active ingredient adsorbed onto coarser particles of an excipient offer significant advantages in the manufacture of pharmaceutical solid drug delivery systems



Lactose/calcium carbonate mixtures



$$\alpha = S_w \rho_s d_e + N \quad (1)$$

This is a modified form of Heywood's expression (Nikolakakis & Pilpel 1985) where N is the elongation ratio = (Length, L)/(Breadth, B) and d_e is the

•Wong & Pilpel, Effect of Particle Shape on the Mixing of Powders, J. Pham. Phamacol. 1990,42: 1-6

Powder Mixing

Standard deviation of concentration

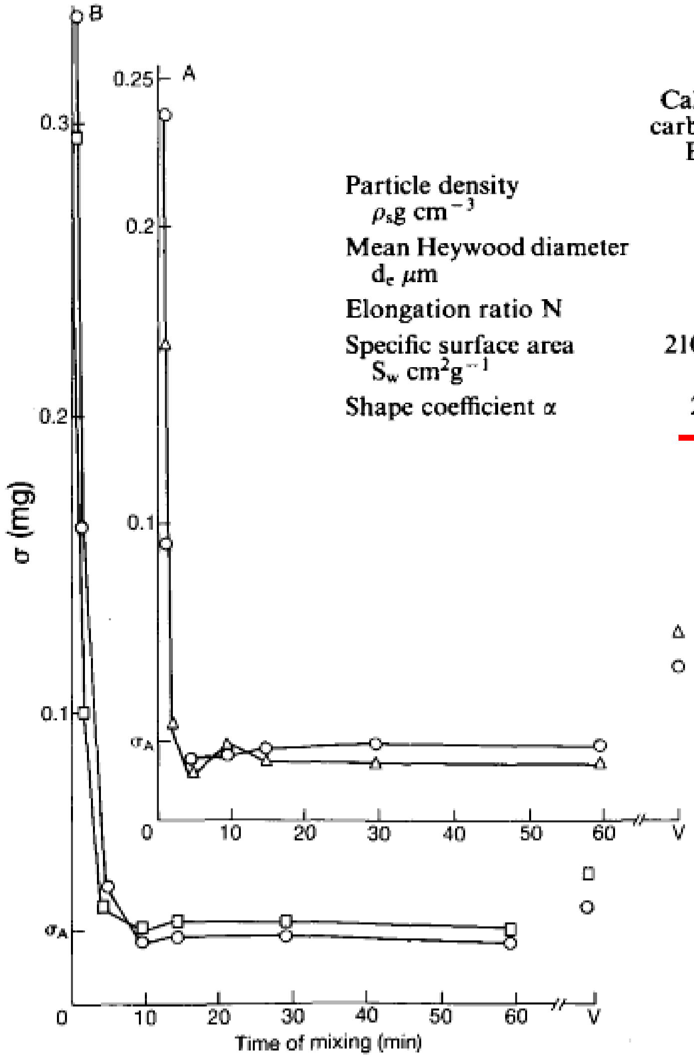


FIG. 1. Standard deviation versus mixing time for different shaped calcium carbonates (A: △ BM, ○ CH) (B: □ SL, ○ SH). V = 120.

	Calcium carbonate BM	Calcium carbonate CH	Calcium carbonate SL	Calcium carbonate SH	Lactose 1	Lactose 4	Lactose 8	Lactose 12
Particle density ρ_s g cm^{-3}	2·71	2·62	2·66	2·66	1·54	1·54	1·54	1·54
Mean Heywood diameter d_e μm	3·3	3·1	4·0	3·7	595	605	606	640
Elongation ratio N	1·50	1·50	1·30	1·40	1·01	1·04	1·06	1·08
Specific surface area S_w cm^{2}g^{-1}	2100	3800	3500	5000	325·4	342·4	368·7	410·8
Shape coefficient α	20·6	32·3	38·5	50·6	30·8	32·9	35·5	41·6

σa achieved more slowly when mixing with shape SL and SH (higher shape coefficient) and BM and CH (lower shape coefficient)

Product Performance

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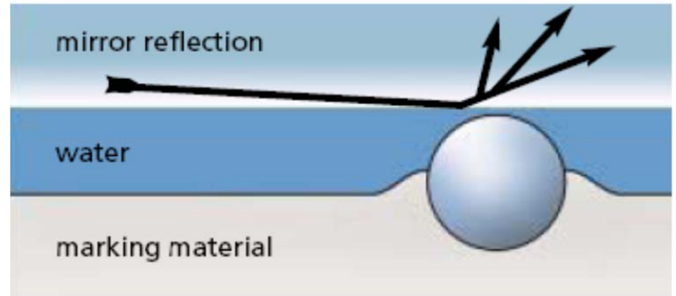
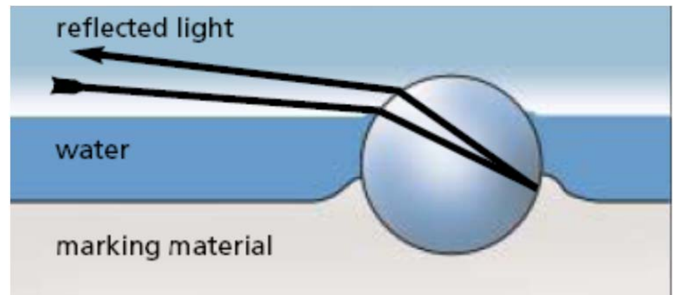
- Just as shape influences processing, also changes product performance
- Some products perform better when more spherical
  - Glass beads for highway paint
  - Proppants
- Some products perform better when less spherical
  - Abrasives

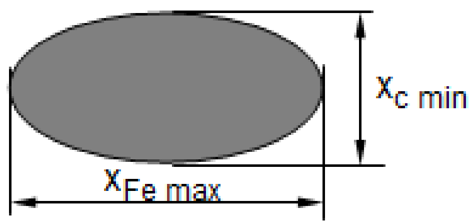
Glass Beads for Highway Paint

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



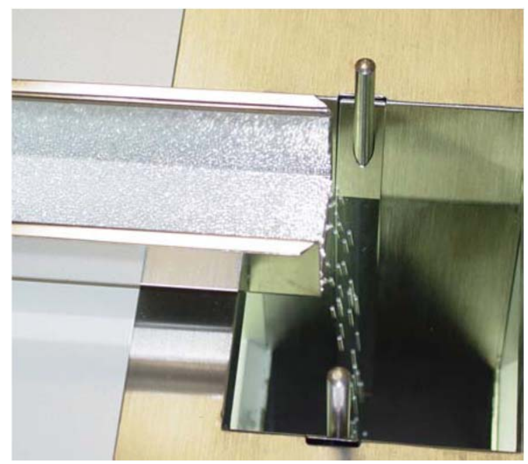


- **Breadth-/Length-ratio**



Dry

In paint

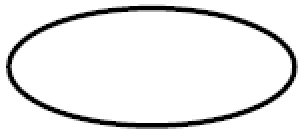


Glass Beads for Highway Paint

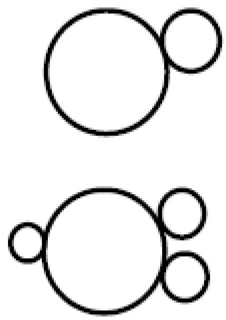
Defects from round particles

Quantified by Camsizer

Oval

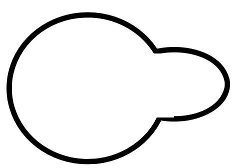


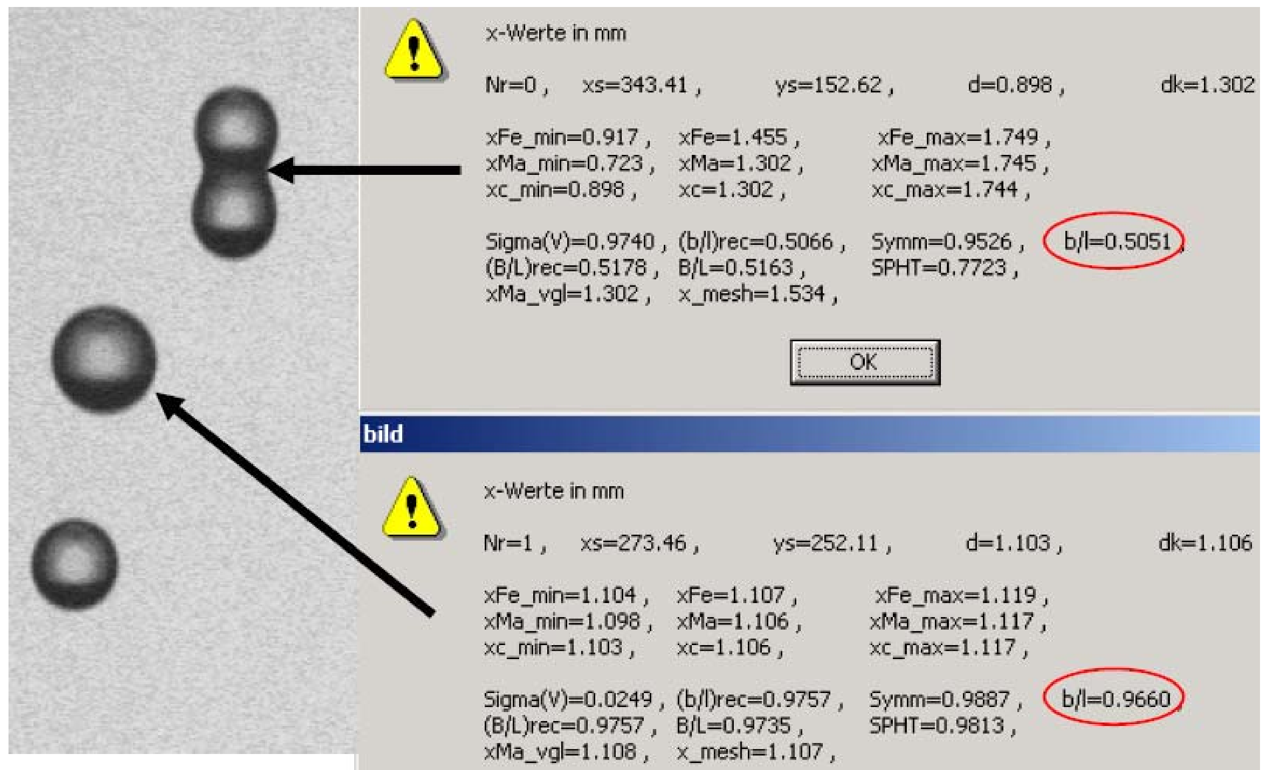
Satellites



Pointed

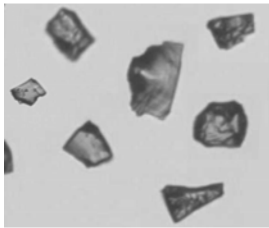
Aggregates





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



Dynamic Image Analysis

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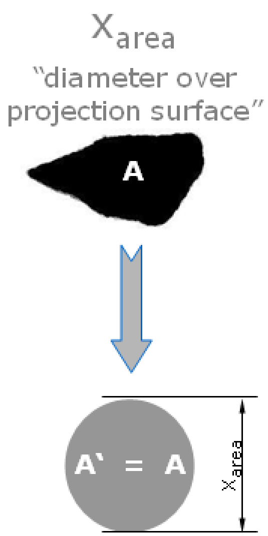
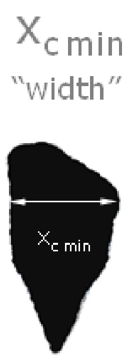
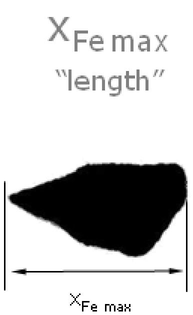
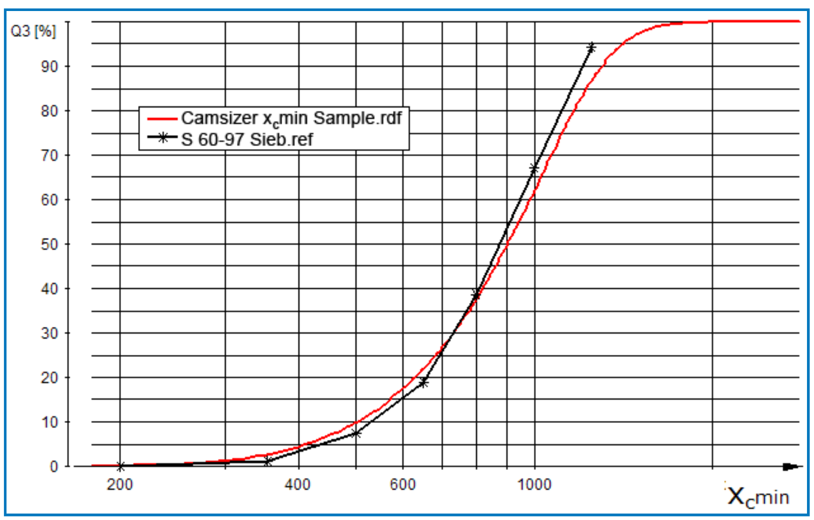




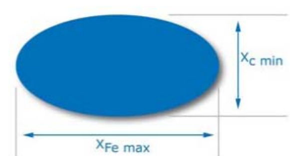
By choosing proper size parameter, Xc min, results can match historic sieve Data. Also generates shape data proven To correlate with abrasive performance.





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

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



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

Sand: Round vs. “Edgy”

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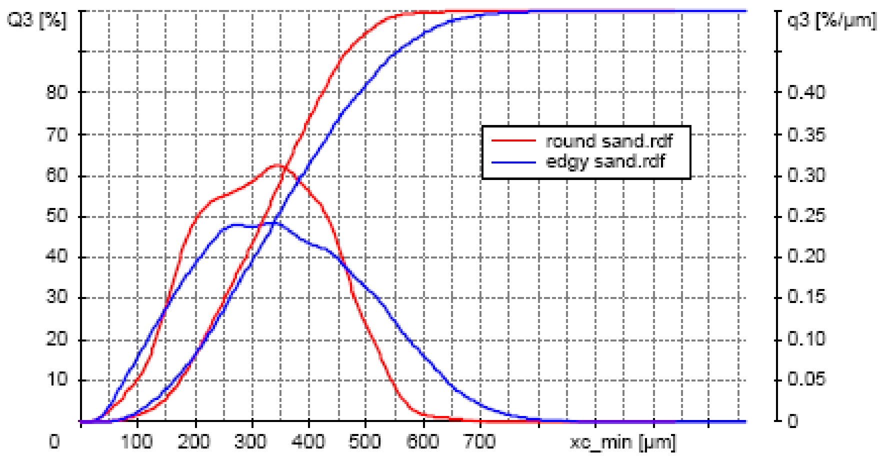
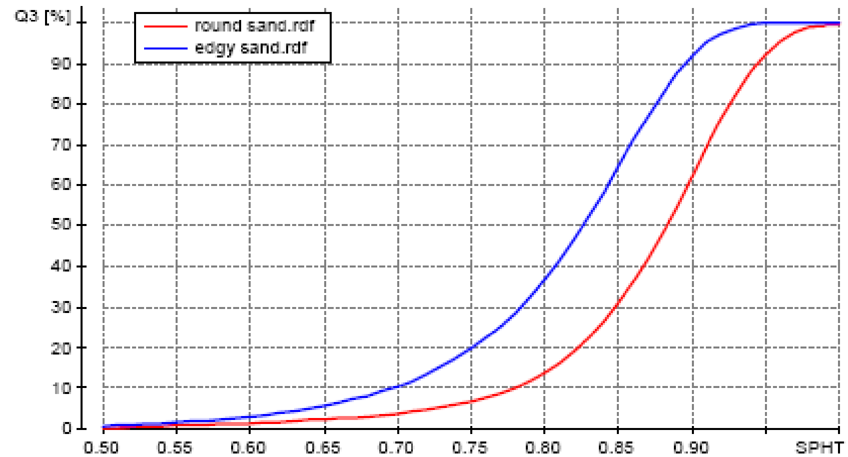
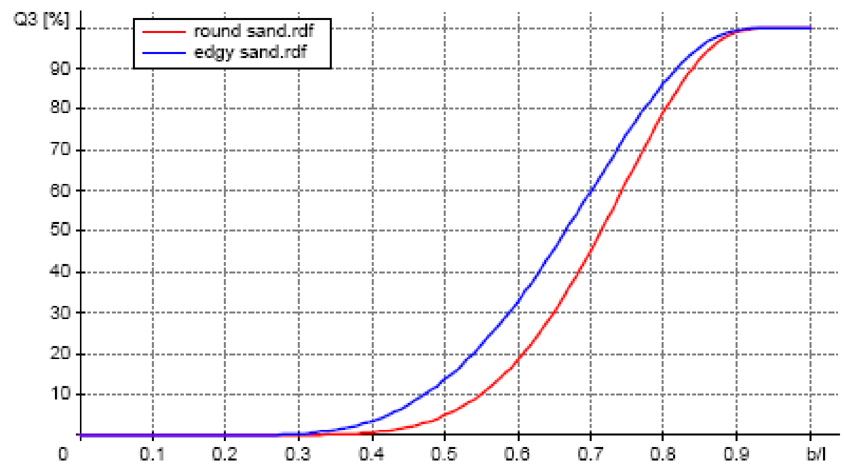




Sphericity

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



Breadth/length (b/l)

Other Shape Parameters

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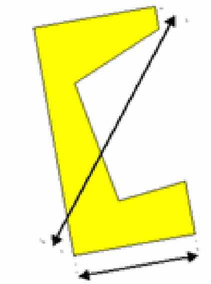
Spherical Volume — Median particle size based on the volume distribution assuming particles are spheres*

$$V_{sph} = \frac{\pi}{6} \text{Circular diameter}^3$$

Circle Diameter = $\text{Mean chord} \times 1.27324$

Roundness — 4 x Area/(π x L x L)
A sphere has a roundness value of 1.0. This value decreases (.9, .8, .7, etc.) as the particles become less spherical

Aspect Ratio — $\dfrac{\text{Longest Feret Length}}{\text{Shortest Feret Length}}$

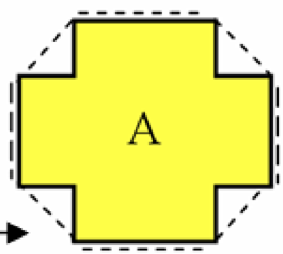


PSA 300
Static Image Analysis

Compactness — 4π Area/ Convex Perimeter^2

A = Area

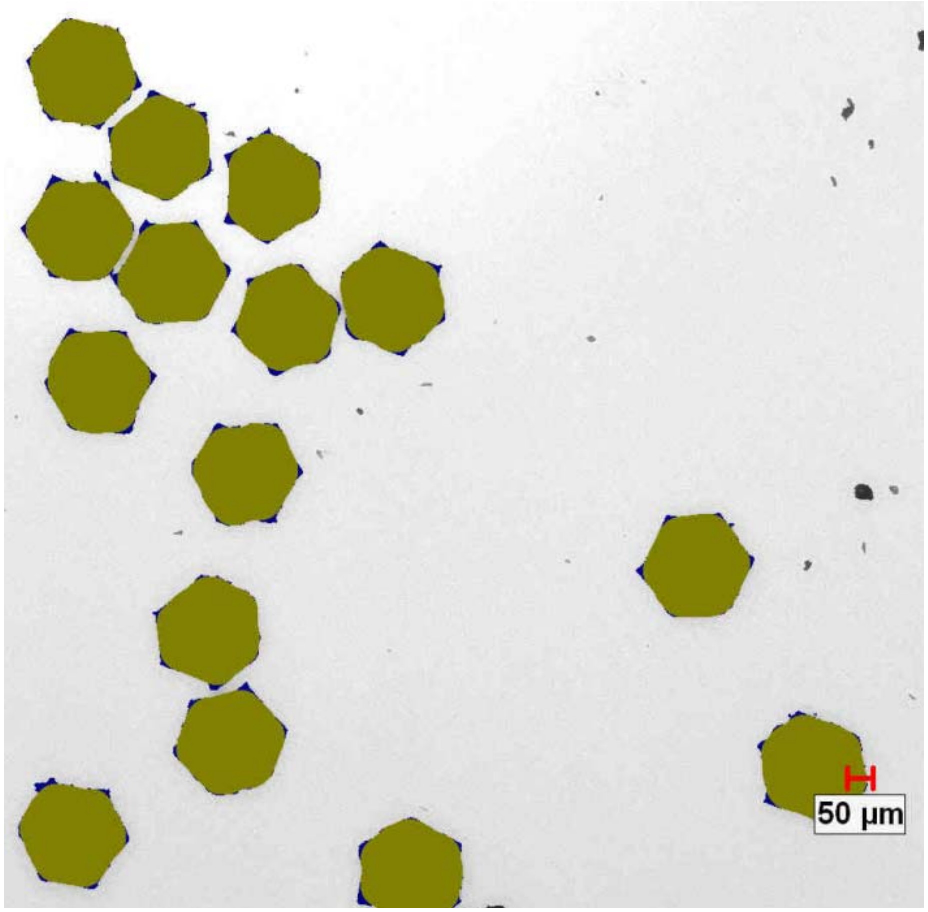
Convex Perimeter

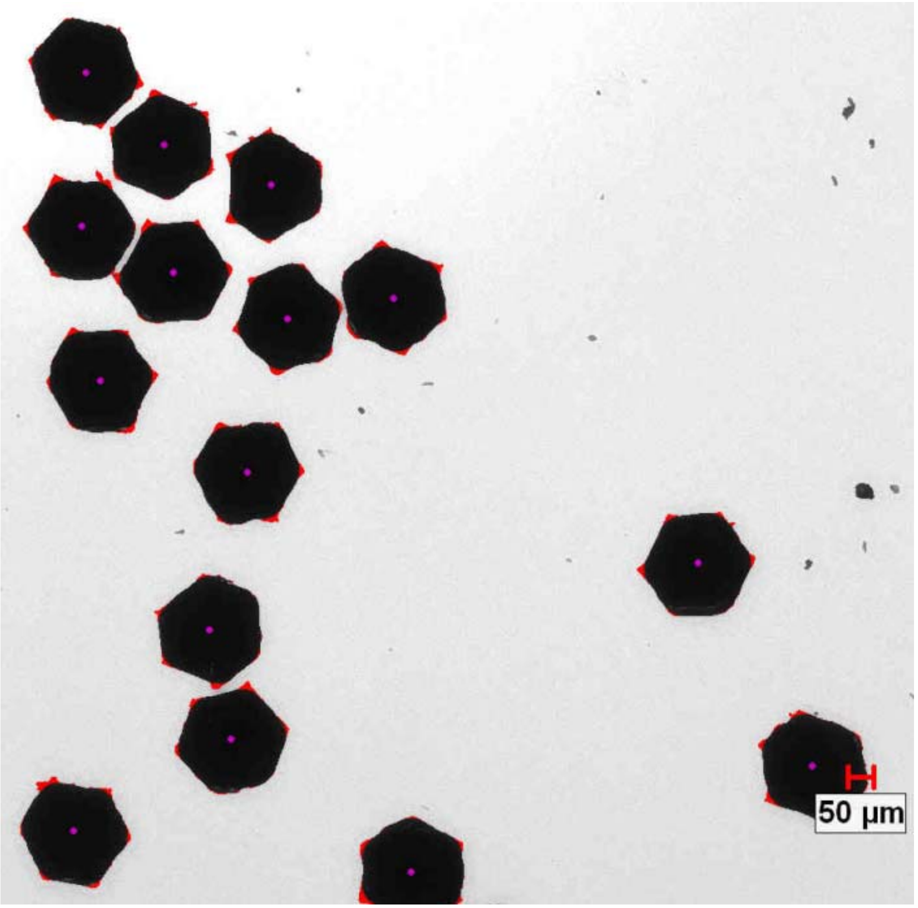


PSA300 Calculation

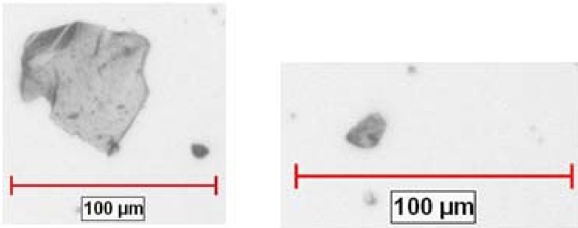
- Find sharp edges at the tips of the particles
- Count number of tips (child count)
- Number of tips alone not sufficient, long particles w/2 tips not as good as hexagon w/6 tips
- Define angularity roundness as child count x roundness
- Thus triangle w/3 points less angular than octagon of 4 sharp edges & 4 rounded edges

PSA300 Calculation

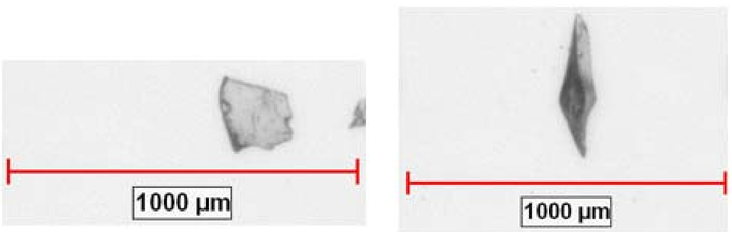




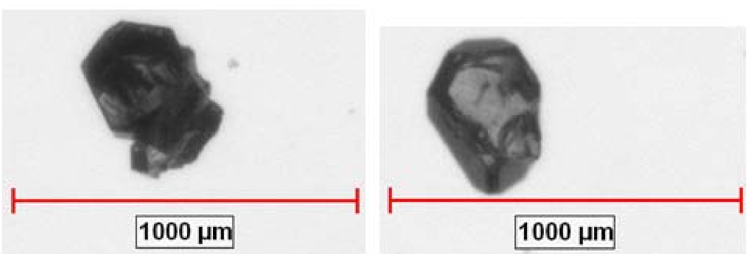
Abrasives



Small abrasive, high angularity (left), low angularity (right)



Medium abrasive, high angularity (left), low angularity (right)



Large abrasive, high angularity (left), low angularity (right)

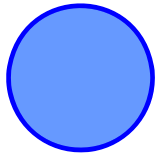
Sample	Vol	Round	Comp	AR	Ang
Small	(µm)				
d10	16	0.5	0.8	1.1	1.2
d50	38.8	0.7	0.9	1.3	2.3
d90	63.1	0.9	0.9	1.8	3.6
Medium					
d10	140	0.3	0.5	1.2	1
d50	211.8	0.5	0.7	1.6	1.9
d90	319.9	0.7	0.8	2.5	3.3
Large					
d10	332.9	0.6	0.7	1.1	2.1
d50	375.2	0.7	0.8	1.3	3.4
d90	421.4	0.8	0.9	1.5	5

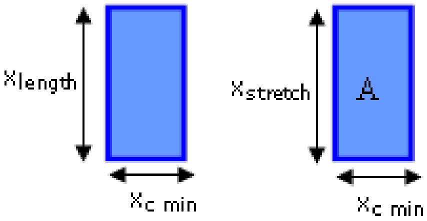
Angularity Roundness: count sharp tips, create child areas

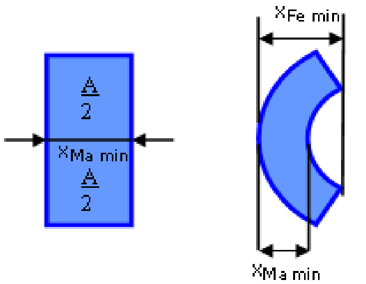
AR: roundness x child area

Catalysts Size/Shape by CAMSIZER

- Spherical catalysts
  - Easy, no special effort
- Cylindrical catalysts
  - Length, width
- Bended extrudates
  - Use other parameters





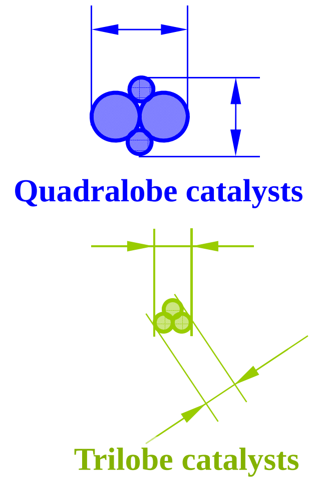


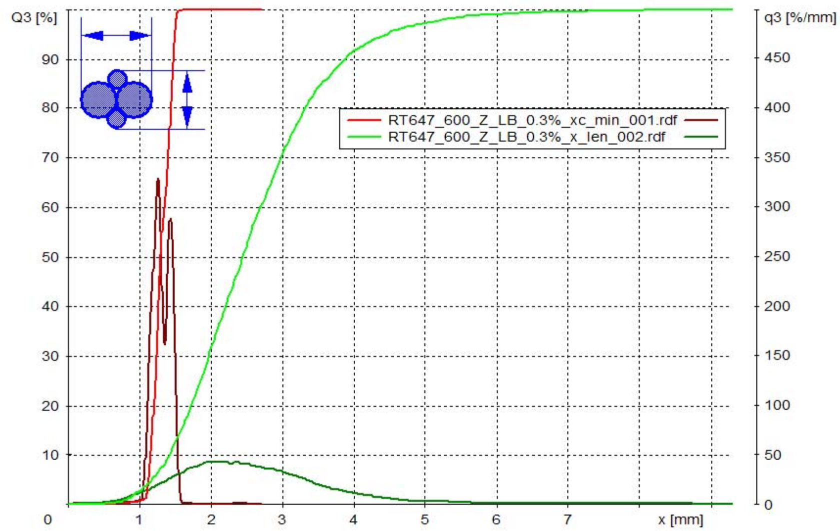
$$x_{length} = \sqrt{(x_{Fe\max})^2 - (x_{c\min})^2}$$

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

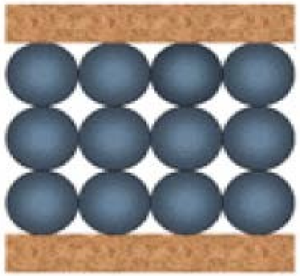
Catalysts Size/Shape by CAMSIZER

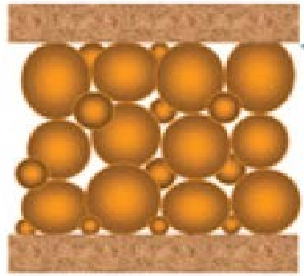
- Tri & quadralobe
  - Possible to distinguish between different diameters
  - Shorter green distribution = length
  - Taller maroon distribution= width
- Size helps define shape

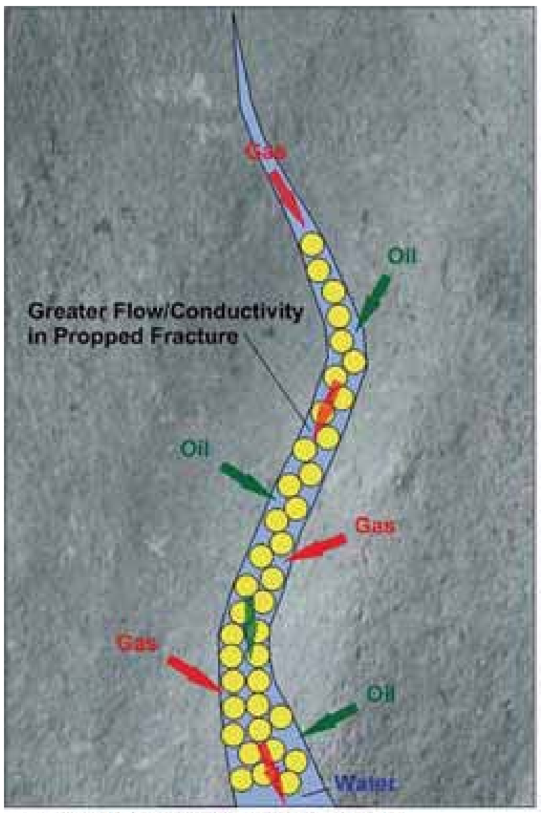




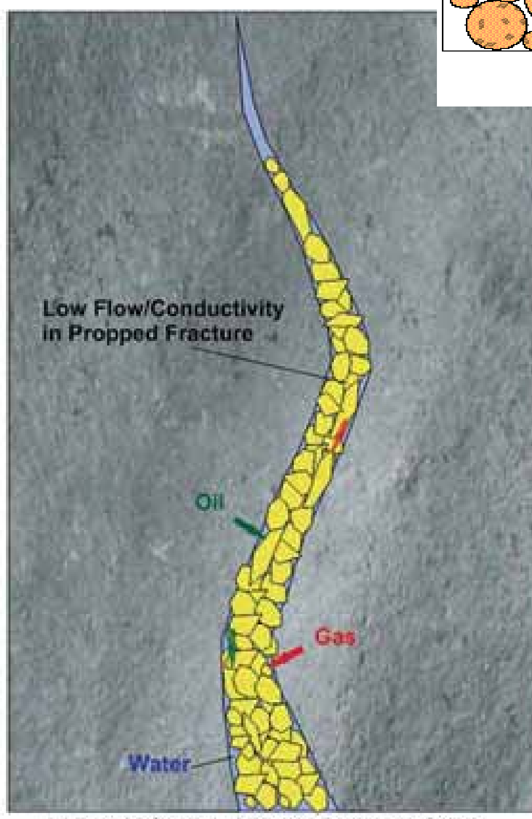
Proppant Packing



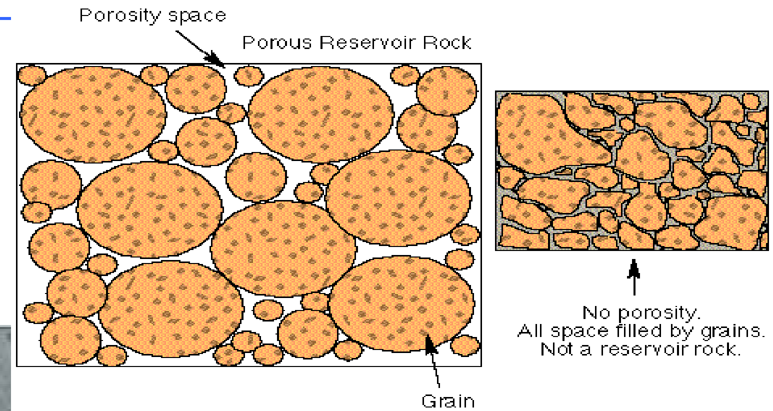


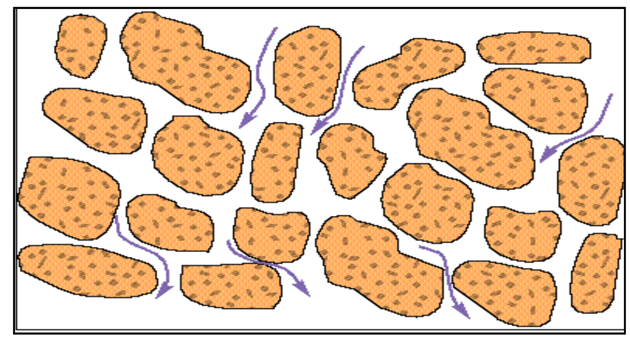


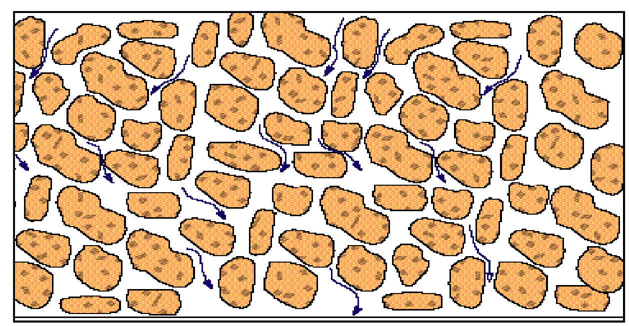
a. Well Rounded Ceramic Proppant



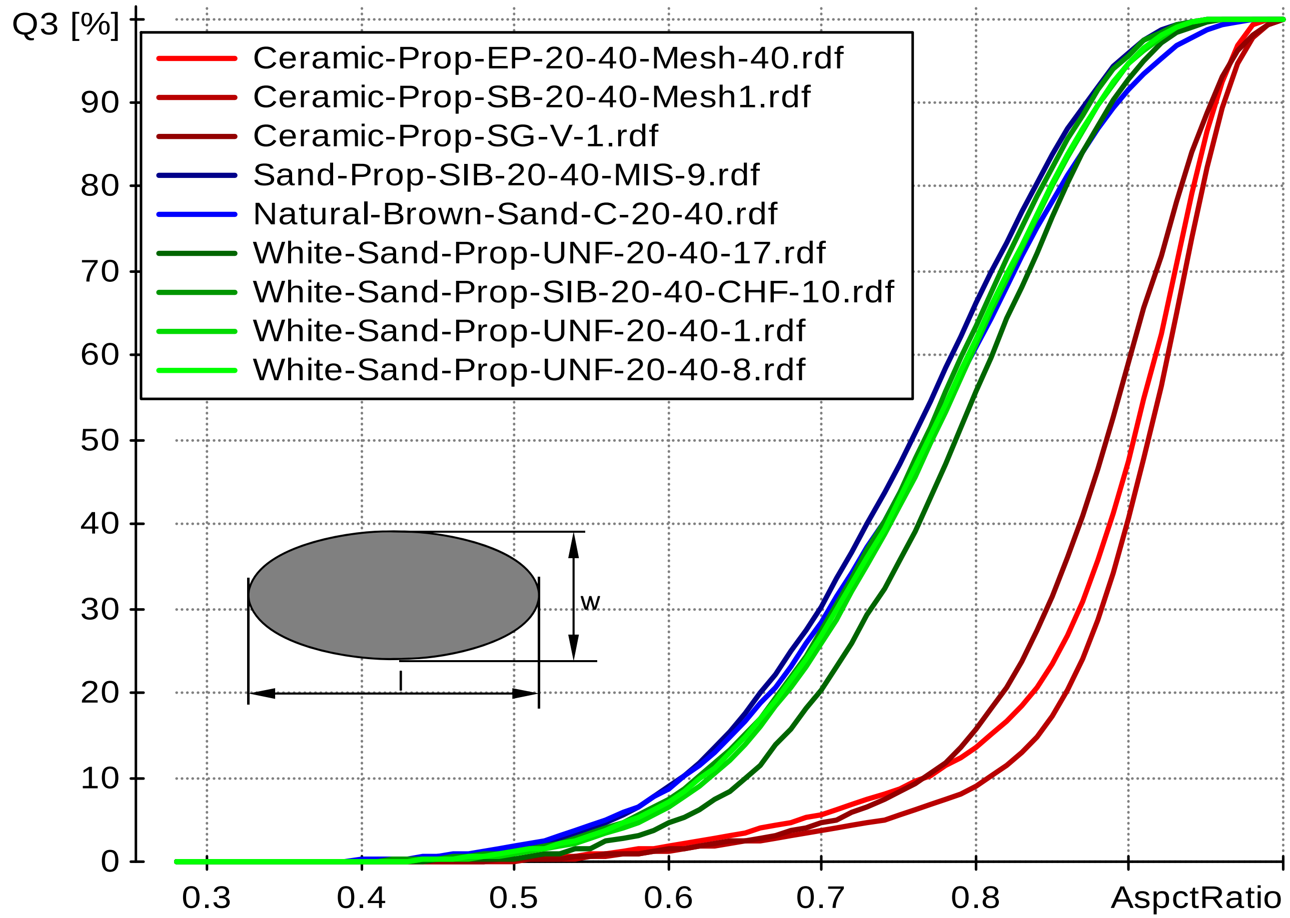
b. Poorly Sorted Angular Proppant Sand







Shape Comparison



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.)

Proppants

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Sand proppant

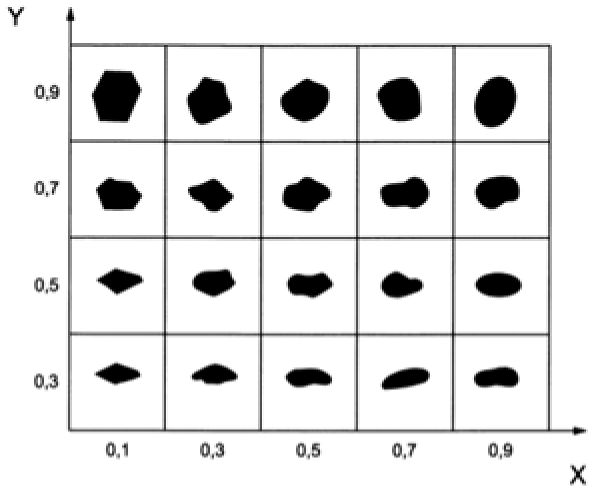
Resin coated sand

Ceramic proppant

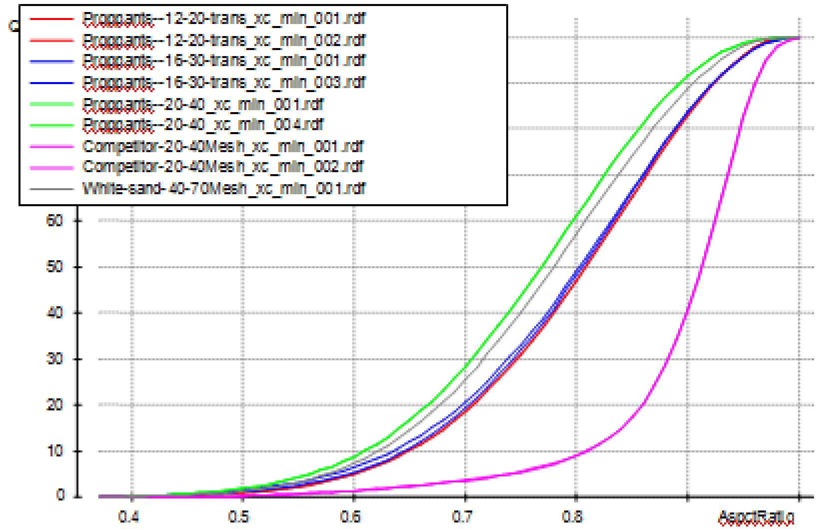
Resin coated ceramic proppant

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

Traditional method



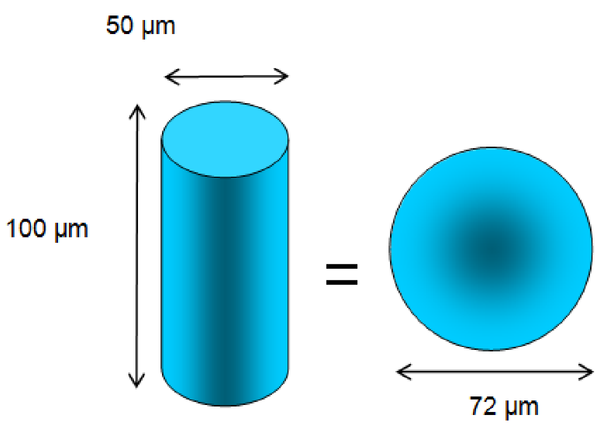
CAMSIZER



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

$V = \pi r^2 h$



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

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

Modeling/Predicting Differences

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

PDA Journal
of Pharmaceutical Science and Technology

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

Modeling/Predicting Differences

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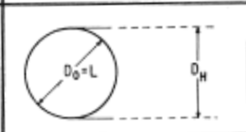
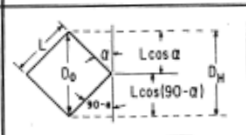
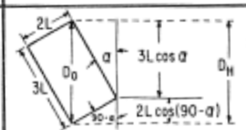
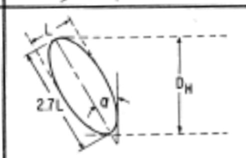
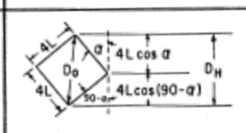
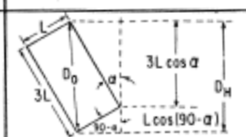
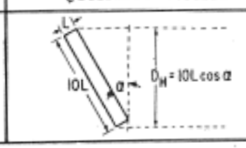
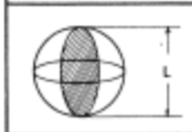
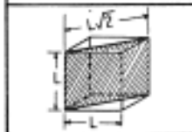
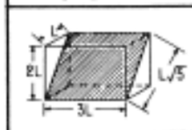
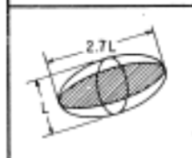
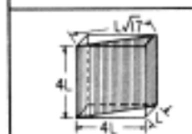
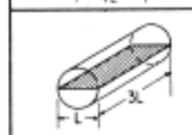
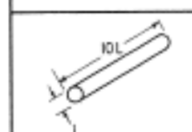
SHAPE	MICROSCOPE VIEW	LONGEST DIMENSION D_0	HORIZONTAL PROJECTION D_H
SPHERE L DIA.		$D_0 = L$	$D_H = D_0 = L$
CUBE LXLXL		$D_0 = L\sqrt{2}$	$D_H = \frac{\int_0^{\pi/2} L[\cos\alpha + \cos(90-\alpha)]\,d\alpha}{\pi/2}$ $= 1.27L \quad \therefore D_H = 0.90\,D_0$
EQUANT 3LX2LXL		$D_0 = L\sqrt{13}$	$D_H = \frac{\int_0^{\pi/2} [3L\cos\alpha + 2L\cos(90-\alpha)]\,d\alpha}{\pi/2}$ $= 3.18L \quad \therefore D_H = 0.88\,D_0$
PROLATE ELLIPSOID 2.7LXL		$D_0 = 2.7L$	CIRCULAR COORDINATES $\bar{Y} = x\sin\alpha + y\cos\alpha$ $D_H = \frac{\int_0^{\pi/2} \bar{Y}\,d\alpha}{\pi/2} = \frac{L(2.7+1)}{\pi/2} = 2.36L$ $\therefore D_H = 0.87\,D_0$
FLAKE 4LX4LXL		$D_0 = L\sqrt{32}$	$D_H = \frac{\int_0^{\pi/2} 4L[\cos\alpha + \cos(90-\alpha)]\,d\alpha}{\pi/2}$ $= 5.08L \quad \therefore D_H = 0.90\,D_0$
ROD 3LXL DIA.		$D_0 = L\sqrt{10}$	$D_H = \frac{\int_0^{\pi/2} [3L\cos\alpha + L\cos(90-\alpha)]\,d\alpha}{\pi/2}$ $= 2.55L \quad \therefore D_H = 0.81\,D_0$
RIGID FIBER 10LXL DIA.		$D_0 = 10L$	$D_H = \frac{\int_0^{\pi/2} 10L\cos\alpha\,d\alpha}{\pi/2} = 6.4L$ $\therefore D_H = 0.64\,D_0$

INSTRUMENT VIEW	LIGHT BLOCKAGE D_A	ELECTROLYTE DISPLACEMENT D_V
	$\frac{\pi D_A^2}{4} = \frac{\pi L^2}{4},\ D_A = L$ $\therefore D_A = D_0$	$\frac{\pi D_V^3}{6} = \frac{\pi L^3}{6},\ D_V = L$ $\therefore D_V = D_0$
	$\frac{\pi D_A^2}{4} = L \times L\sqrt{2},\ D_A = 1.34L$ $\therefore D_A = 0.95\,D_0$	$\frac{\pi D_V^3}{6} = L \times L \times L,\ D_V = 1.24L$ $\therefore D_V = 0.88\,D_0$
	$\frac{\pi D_A^2}{4} = 3L \times L\sqrt{5},\ D_A = 2.92L$ $\therefore D_A = 0.81\,D_0$	$\frac{\pi D_V^3}{6} = L \times 2L \times 3L,\ D_V = 2.25L$ $\therefore D_V = 0.62\,D_0$
	$\frac{\pi D_A^2}{4} = \frac{\pi}{4} \times 2.7L \times L,\ D_A = 1.64L$ $\therefore D_A = 0.61\,D_0$	$\frac{\pi D_V^3}{6} = \frac{\pi}{6} \times 2.7L \times L^2,\ D_V = 1.39L$ $\therefore D_V = 0.52\,D_0$
	$\frac{\pi D_A^2}{4} = 4L \times L\sqrt{17},\ D_A = 4.58L$ $\therefore D_A = 0.81\,D_0$	$\frac{\pi D_V^3}{6} = 4L \times 4L \times L,\ D_V = 3.13L$ $\therefore D_V = 0.55\,D_0$
	$\frac{\pi D_A^2}{4} = 3L \times L,\ D_A = 1.95L$ $\therefore D_A = 0.62\,D_0$	$\frac{\pi D_V^3}{6} = \frac{\pi L^2}{4} \times 3L,\ D_V = 1.65L$ $\therefore D_V = 0.52\,D_0$
	$\frac{\pi D_A^2}{4} = 10L \times L,\ D_A = 3.57L$ $\therefore D_A = 0.36\,D_0$	$\frac{\pi D_V^3}{6} = \frac{\pi L^2}{4} \times 10L,\ D_V = 2.47L$ $\therefore D_V = 0.25\,D_0$

Modeling/Predicting Differences

TABLE II. Summary of Sphericity Correction Factors Based on Longest Linear Dimension

Shape	D_0 Longest Dimension	D_H Horizontal Projection	D_A Light Blockage	D_V Electrolyte Displacement
Sphere	1.00	1.00	1.00	1.00
Cube (1:1:1)	1.00	0.90	0.95	0.88
Equant (3:2:1)	1.00	0.88	0.81	0.62
Prolate ellipsoid (2.7:1)	1.00	0.87	0.61	0.52
Flake (4:4:1)	1.00	0.90	0.81	0.55
Rod (3:1 dia.)	1.00	0.81	0.62	0.52
Fiber (rigid, 10:1)	1.00	0.64	0.36	0.25

My sample is a rod, what will my instrument report?
Coulter counter: 52 µm, laser diffraction: 81µm

Defining Size

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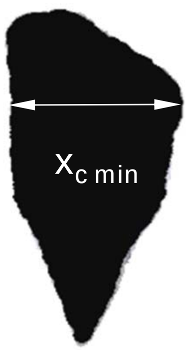
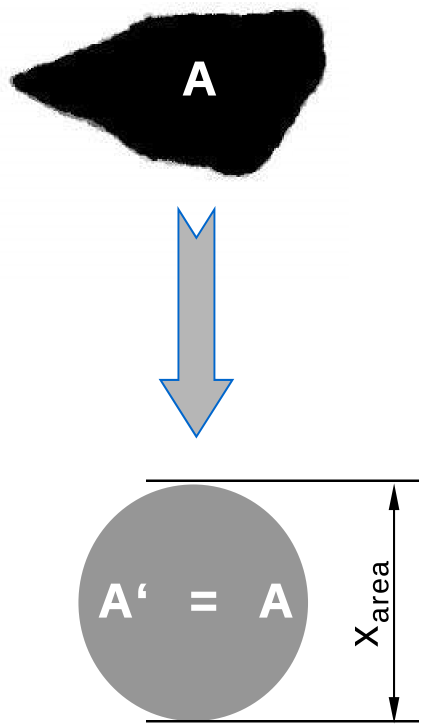
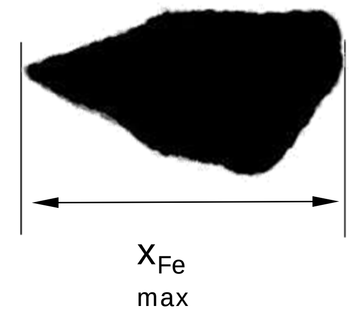
$X_{c\,min}$	X_{area}	$X_{Fe\,max}$
"width"	"diameter over projection surface"	"length"





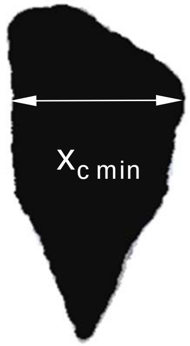


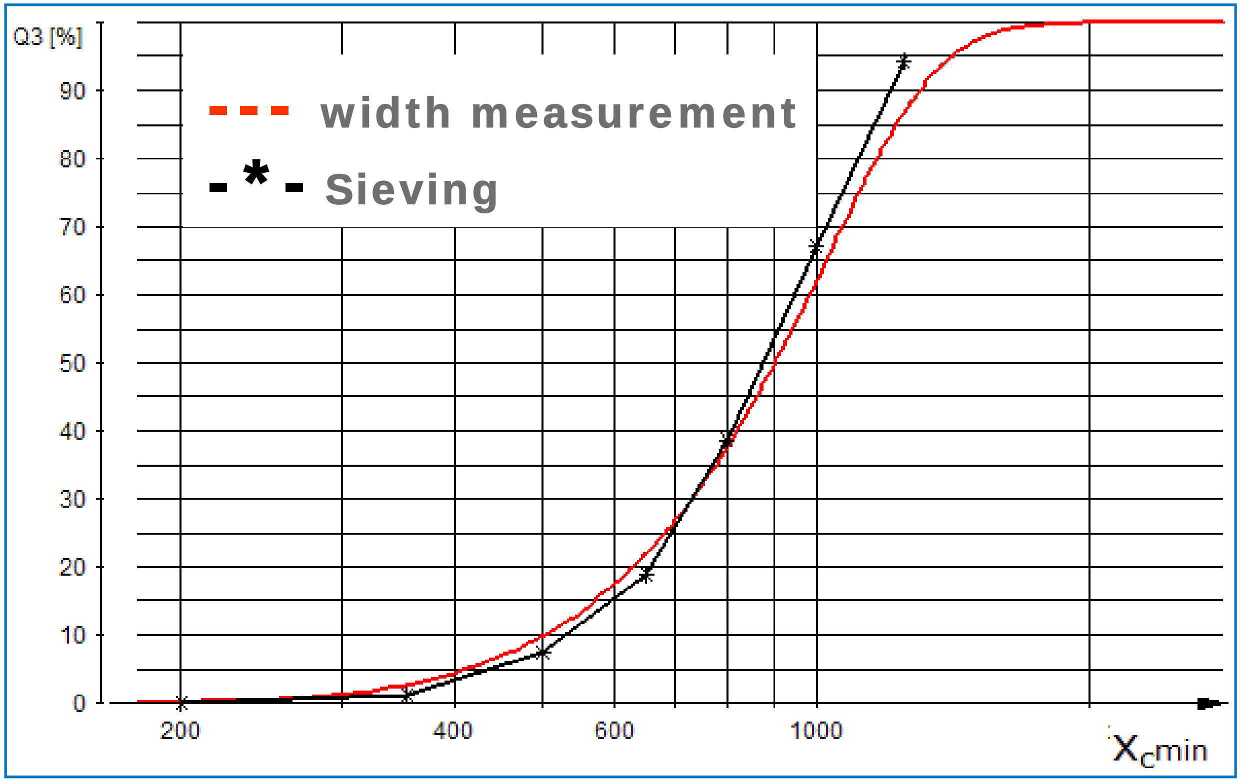
CAMSIZER results are compatible with sieve analysis

Width ⇔ Sieving

$X_{c\,min}$

"width"





comparison

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

Laser Diffraction vs. Sieves

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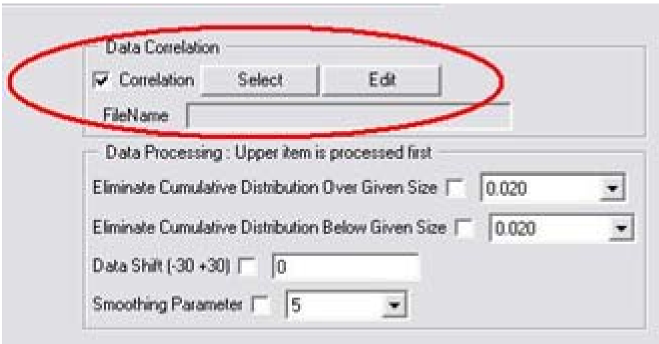
- Sieves report smaller size
  - Typically 10-30% smaller
- Depends on shape
- Options to correlate
  - Shift reported results
  - 50% passes 325 mesh (44 µm)
  - D50 by diffraction = 53 µm
  - Report value @ 53 µm ads pass 325 mesh result
- LA-950 correlation tools



Fiber: Laser Diffraction vs. Images

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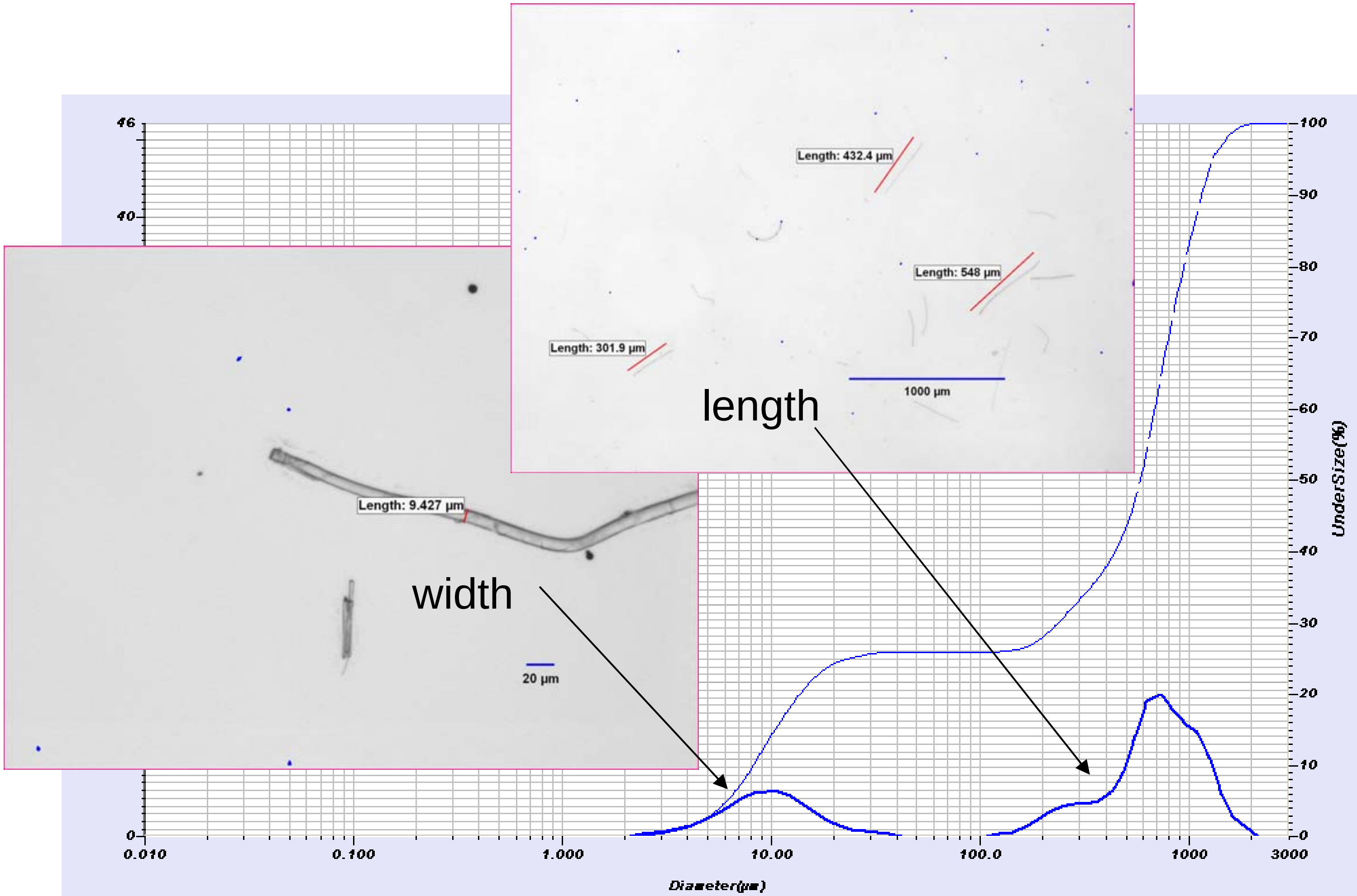
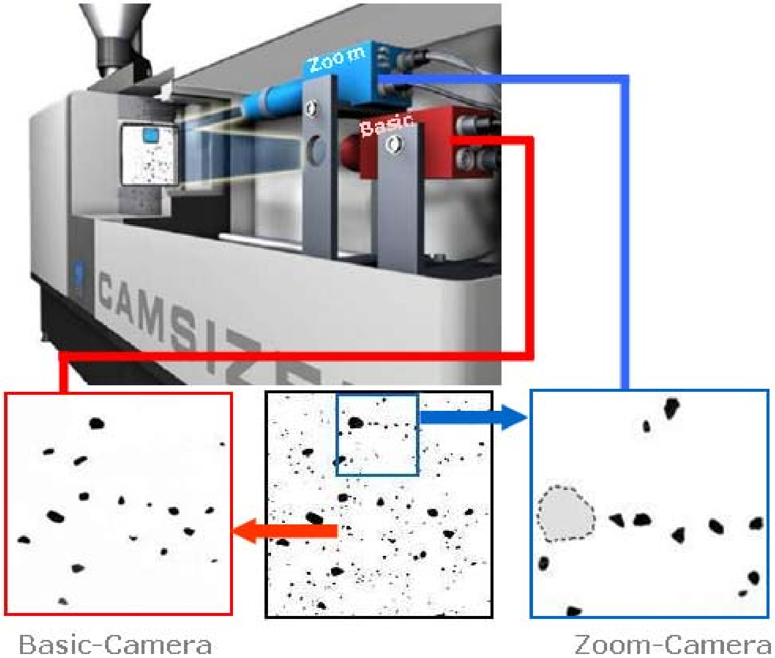


Note: both distributions widened by laser diffraction

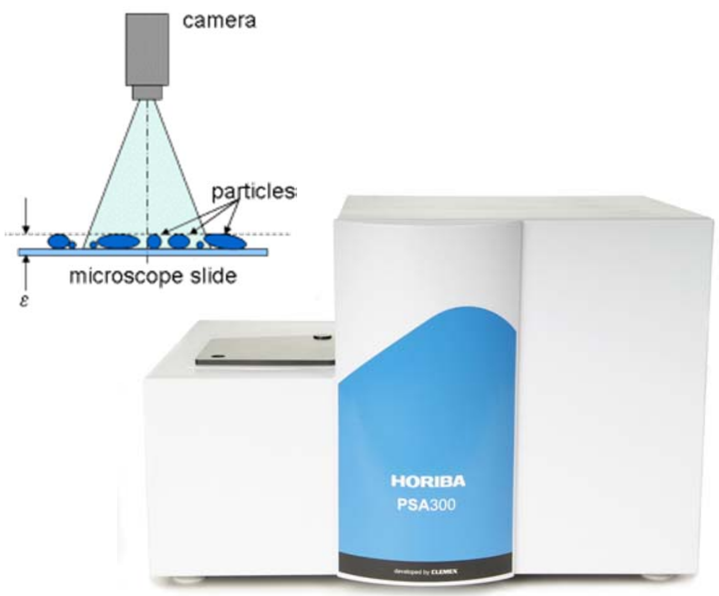
Want to Avoid Influence of Shape?

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

CAMSIZER



PSA300



Summary

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- 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

Resources: www.horiba.com/particle

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