CAMSIZER XT
- the latest in Dynamic Image Analysis

Webinar
Wednesday, 14th of September 2011

Speaker: Gert Beckmann
Retsch Technology GmbH

Host: Ian Treviranus
HORIBA Instruments International
History of Retsch Technology GmbH

- **2000**
  - Transfer of RETSCH Technology into an independent company owned by VERDER

- **2001**
  - Appointment as exclusive distributor of HORIBA Particle Sizers within Central Europe

- **2003**
  - Launch of the Autosampler System

- **2006**
  - Development of an Improved CAMSIZER Version
  - New possibilities

- **2011**
  - Development of CAMSIZER XT (for agglomerated powders: 1 μm – 3000 μm)
Content

**Instrument**

1. Measurement principle

2. Results

**Applications**

3. Markets and applications

4. Alternative analysis methods
Comparison between Static ↔ Dynamic Image Analysis

ISO 13322-1

ISO 13322-2
Measurement principle

Resolution

Detection of particles

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

Particle Size Range from 1µm to 3mm

Three modes in 2 modules (dry and wet):

**X-Fall**: for dry and free flowing particles

**X-Jet**: air pressure dispersion for fine and agglomerated powders

**X-Flow**: wet module for emulsions and suspensions, with ultrasonic probe, optional for organic solvents
Modular "X-Change" Concept

Flexible configuration for a wide application range

- Basic instrument
- Module
- Plug-in cartridge

= Measurement system

- Dry measurement
  - X-Dry
  - X-Jet
  - Air pressure dispersion

- Wet measurement
  - X-Flow
  - X-Fall
  - Gravity dispersion

- Wet measurement

simple • safe • fast
Dispersion Modules

Dry Dispersion Inserts (2 Plug-In Options)

X- Fall
(Gravity dispersion)

X-Jet
(Air pressure dispersion)
Dispersion Modules

Dry Dispersion with X-Jet

Measurement range from 1 µm to 1.5 mm

For fine powders and agglomerating materials

Dry Dispersion by pressurized air: pressure range from 0.2 bar to 4.5 bar, 3psi – 66psi
Measurement principle – X-Jet
Measurement results with – X-Jet

Lactose Powder

100, 150, 200, 300, 460 kPa => particles break

20, 30 and 50 kPa air pressure => stable results / particles don’t break
Customer had sent 30 different samples to Retsch Technology but some of these samples were the same (red, blue and green). We found out the groups and showed to the customer the good reproducibility of CAMSIZER XT (and proofed his sample splitting as well)
Measurement principle – X-Fall

Dry Dispersion with X-Fall

Measurement range from 10 µm to 3 mm
For free flowing materials
similar to standard CAMSIZER

Sample recovery after analysis
• Complete sample recovery
• No contamination
Measurement principle – X-Fall
Measurement principle – X-Flow

Wet Dispersion with X-Flow

Measurement range from 1 µm to 600 µm for emulsions and suspensions

stronger dispersion with ultrasonic module

Optional for organic solvents
Measurement principle – X-Flow

- Flow cell
- Level sensor
- Dispersion bath
- Ultrasonic probe
- Centrifugal pump
- Outlet
Pharmaceuticals using Wet Cell

Cancer Pharmaceuticals
Measurement principle

Advanced, patented optics design

Light source 1

Light source 2

Sample flow

Basic Camera

Zoom Camera
Measurement principle

Advanced, patented optics design

Light source 1

Light source 2

Sample flow

Basic Camera

Zoom Camera
Measurement principle

Advanced, patented optics design

Light source 1

Light source 2

Sample flow

Basic Camera

Zoom Camera
Measurement principle

Advanced, patented optics design
Features of the CAMSIZER XT

Calibration Reticule
Features of the CAMSIZER XT

Calibration Reticule
Physical Dynamic Partical Standards

Whitehouse Glass Bead Standard XX030 for X-Dry and X-Fall
Results X-Flow

Particle Size Distribution
2.5µm + 5µm, Wet Dispersion

Certified Particle Size Standards
- 9000 Series Borosilicate Glass (2–10 microns)
- 9000 Series Soda Lime Glass (30–2000 microns)

Dry Glass NIST Traceable Size Standards
Features of the CAMSIZER XT

X-Change of wet and dry modules in < 1minute
Highlights of the optics setup design:

• More than 275 images per second
• Full frame cameras with > 1.3 Megapixel resolution
• Separate light sources for optimised brightness, homogeneity, and contrast
• 2 Cameras: High resolution combined with excellent statistic for a wide dynamic range
• Image processing in real-time: Each particle in each image is analysed
• Hundreds of particles in each image: Excellent statistics in short time

CAMSIZER XT can measure in a wider dynamic range with better statistics and reproducibility than any other image processing system.
Advantages

- fast
- repeatable and reproducible
- precise
- maintenance free and robust
Content

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Results

For agglomerating powders

- Metal powder
- Coal dust
- Wheat flour

Graph: Relative Sample Volume vs. Particle size
Velocity Adaption

When you illuminate the backlight two times within one photo you will see every particle two times on the image.
Particles of different size have different speeds.
Velocity Adaption

Particles of different size move different distances
Velocity Adaption

\[ v = \frac{s}{t} \]
Velocity Adaption

Results with and without velocity adaption

![Graph showing Q3 (%) versus xc_min [µm] with multiple lines representing different conditions.](Pharma-Product-30kPa_xc_min_1.rdf)

- Pharma-Product-30kPa_xc_min_1.rdf
- Pharma-Product--30kPa_xc_min_2.rdf
- Pharma-Product-30kPa_2_xc_min_4.rdf
- PharmaProduct-30kPa-VitesseAdaption_xc_min_8.rdf
- PharmaProduct-30kPa-VitesseAdaption_xc_min_9.rdf
- Pharma-Product-lot002-tamis-Sieve-Analysis.ref
Crushed glass in DM 200 (Retsch Disk Mill)

Different gap settings of the disc mill
Results X-Jet

Detection of oversized particles

Sample: PMMA Beads

Δ ~ approx 0.5% Vol.

4 Samples:
Red
Green
Blue
Violet

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Identical results to sieve analysis
Particle Size Distribution
1µm - 10µm, Dry Dispersion

Sample: SiC
At the lower resolution limit .......
Particle Size Distribution
10µm + 12µm, Wet Dispersion

Results X-Flow (Calibration)

q3 [%/µm]

Duke10um12um_gl0_xc_min_009.rdf
Duke10um12um_gl0_xc_min_010.rdf
Duke10um12um_gl0_xc_min_011.rdf
Results X-Flow (Calibration)

Particle Size Distribution
10µm + 12µm, Wet Dispersion
Better Size Analysis due to Understanding of Particle Shape: Length, Width, Average Diameter
Shape Analysis with CAMSIZER XT

- Area of the particle
- Circle with same area as particle
- Perimeter of the particle
- Diameter of circle of same area

\[ P_{ASPHT} = \frac{\pi}{4} \times \text{max} \times \text{min} \]

\[ Fe = \frac{x \times x}{l} \]

Q3 [%] vs. \( \frac{b}{l} \)

Q3 [%] vs. Aspect Ratio

- Pharma-Product-1-30kPa-bonne-forme-_xc_min_009.rdf
- Pharma-Product-1-30kPa_Vitesse-Adaption_xc_min_008.rdf
- lactose-30kPa_xFemax_003.rdf
- Pharma-Product-2-460_xc_min_008.rdf
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
Excellent repeatability and detection efficiency for „large particles“ and small sample volumes

Sample: Glass beads, multimodal
Advantages

- Digital image processing with patented 2-camera system (ISO 13322-2)
- Wide dynamic range from 1 µm to > 3 mm
- Newly developed optical system with ultra bright LEDs for sharp contrasts and large depth of focus
- Short analysis time 1 – 3 minutes for few million particles
- Safe detection of oversized and undersized
- Modules for dry and wet dispersion
- Analysis results optionally compatible to sieve analysis
Content

Instrument

1. Measurement principle
2. Results

Applications

3. Markets and applications
4. Alternative analysis methods
Application areas

- Industrial labs
- Research institutes
- Production control
- Quality control for final products
- Quality control of incoming raw materials
- Immediate control and optimisation of production processes
Typical sample materials

- Pharmaceutical powders, granules or small pellets
- Pulverized and granulated food, spices
- Detergents, enzymes, fillers for washing powders
- Metal or ore powders
- Abrasives (medium and small grit)
- Sand and cement, building materials, limestone
- Fibres
Content

Instrument

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## Alternative Methods

<table>
<thead>
<tr>
<th>Performance Features</th>
<th>CAMSIZER XT</th>
<th>Sieving</th>
<th>Laser Diffraction</th>
<th>opt. Microscope</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Wide dynamic measurement range</td>
<td>++</td>
<td>+</td>
<td>++</td>
<td>-</td>
</tr>
<tr>
<td>2. Reproducibility and repeatability</td>
<td>++</td>
<td>+</td>
<td>++</td>
<td>-</td>
</tr>
<tr>
<td>3. High resolution for narrow distributions</td>
<td>++</td>
<td>-</td>
<td>-</td>
<td>++</td>
</tr>
<tr>
<td>4. Particle shape analysis</td>
<td>++</td>
<td>-</td>
<td>-</td>
<td>++</td>
</tr>
<tr>
<td>5. Direct measurement technique</td>
<td>++</td>
<td>++</td>
<td>-</td>
<td>++</td>
</tr>
<tr>
<td>6. Compatibility of results with other techniques</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>7. Reliable detection of oversized grains</td>
<td>+</td>
<td>++</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>8. Robust hardware, easy operation for routine analysis</td>
<td>++</td>
<td>++</td>
<td>+</td>
<td>-</td>
</tr>
<tr>
<td>9. Analysis of individual particles</td>
<td>+</td>
<td>-</td>
<td>-</td>
<td>++</td>
</tr>
<tr>
<td>10. High measurement speed, short measurement times</td>
<td>++</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Feature</td>
<td>Sieving</td>
<td>CAMSIZER XT</td>
<td></td>
<td></td>
</tr>
<tr>
<td>----------------------------------------</td>
<td>--------------------------</td>
<td>---------------------------</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Size range</td>
<td>10 µm - 63 mm</td>
<td>1 µm – 3 mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shape analysis</td>
<td>no</td>
<td>yes</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Detection of oversized particles</td>
<td>each particle</td>
<td>few big particles from &lt; 0.1% Vol.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resolution</td>
<td>poor</td>
<td>high resolution</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Multi-modal distributions</td>
<td>poor size resolution</td>
<td>better resolution</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Repeatability and lab-to-lab comparison</td>
<td>„difficult“</td>
<td>superior</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Comparison with sieving</td>
<td>identical results possible</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Handling</td>
<td>simple, but time consuming</td>
<td>easy and fast</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Results X-Jet

Identical results to sieve analysis
Spheroidal Particles

$$\varnothing = d = X_{c\ min}$$

$$X_{c\ min} = \text{particle-width}$$
Influence of Mesh Width

**Theory:**
- Nominal Sieve Mesh = 1400µm
- only beads < 1400µm will pass the sieve mesh
- beads > 1400µm will not pass the sieve mesh

**Reality:**
- Real Sieve Mesh >1400=1455
- Upper mesh size range ~1455µm
- sieve No. 03033531 (nominal 1400µm)
Real Mesh Width

Passing [%]

Sample-1__xc_min_002.rdf
Sieving-upper-range-S1.ref
Sample Reproducibility of CAMSIZER XT measurements of $x_{c\text{ min}}$ (red, and blue) with Basic + Zoom or Zoom only, Retsch sieve result (real mesh sizes from optical inspection) AS 200 TAB (*black), Customer nominal sieve results (*blue)
Glass Recycling

Crushed Glass
Similar results to sieve analysis

![Graph showing Q3 (%) vs. xc_min [µm]](image)
Applications: Metal powders
Material: Cu

Identical results to the sieve analysis

Automatic reports, many languages available
Digitale Imaging ↔ Sieving

![Diagram showing the relationship between Digitale Imaging and Sieving](image.png)
Glass Recycling

Very Acicular and Angular Particles without and with Elementary Fitting
### CAMSZIER XT ↔ Laser sizer

<table>
<thead>
<tr>
<th>Feature</th>
<th>Laser sizer</th>
<th>CAMSZIER XT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Size range</strong></td>
<td>down to 20nm</td>
<td>&gt; 1µm</td>
</tr>
<tr>
<td><strong>Shape analysis</strong></td>
<td>no</td>
<td>yes</td>
</tr>
<tr>
<td><strong>Detection of oversized particles</strong></td>
<td>percent range</td>
<td>few big particles &lt; 0.1% Vol.</td>
</tr>
<tr>
<td><strong>Resolution</strong></td>
<td>good for fines</td>
<td>better resolution for large particles</td>
</tr>
<tr>
<td><strong>Multi-modal distributions</strong></td>
<td>more difficult</td>
<td>better volume model, better size resolution</td>
</tr>
<tr>
<td><strong>Comparison with sieving</strong></td>
<td>not possible</td>
<td>identical results</td>
</tr>
<tr>
<td><strong>Information content</strong></td>
<td>black box + mathematics</td>
<td>pictures</td>
</tr>
</tbody>
</table>
Reliable results, closer to sieving

Sample: Petrolcoker for Electrodes
### CAMSIZER XT ↔ Optical Microscope

<table>
<thead>
<tr>
<th></th>
<th>Microscope</th>
<th>CAMSIZER XT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Size range</strong></td>
<td>0.5 – 500 µm</td>
<td>1 µm -3 mm</td>
</tr>
<tr>
<td><strong>Shape analysis</strong></td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td></td>
<td>superior image quality</td>
<td></td>
</tr>
<tr>
<td><strong>Detection of oversized particles</strong></td>
<td>no</td>
<td>few big particles</td>
</tr>
<tr>
<td></td>
<td></td>
<td>&lt; 0.1% Vol.</td>
</tr>
<tr>
<td><strong>Resolution</strong></td>
<td>better</td>
<td>good</td>
</tr>
<tr>
<td><strong>Statistics</strong></td>
<td>Low, few 1,000 particles</td>
<td>million particles/minute</td>
</tr>
<tr>
<td><strong>Comparison with sieving</strong></td>
<td>not possible</td>
<td>identical results possible</td>
</tr>
<tr>
<td><strong>Handling</strong></td>
<td>time consuming</td>
<td>fast</td>
</tr>
<tr>
<td><strong>Representative Sample Amounts</strong></td>
<td>difficult, only narrow distributions</td>
<td>yes, small and large amounts</td>
</tr>
</tbody>
</table>
Effect of Removing 6 particles

Sample PPO 646

- **Before Removal**
  - 29752 particles
  - Mean: 136 microns
  - D50: 162 microns

- **After Removal**
  - 29746 particles
  - Mean: 28 microns
  - D50: 19 microns
<table>
<thead>
<tr>
<th></th>
<th>CAMSIZER</th>
<th>CAMSIZER XT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size range</td>
<td>30 µm – 30mm</td>
<td>1 µm -3 mm</td>
</tr>
<tr>
<td>Shape analysis</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Detection of oversized</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>particles</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Images / second</td>
<td>60</td>
<td>277</td>
</tr>
<tr>
<td>Resolution CCD-Cameras</td>
<td>790,000</td>
<td>1,300,000</td>
</tr>
<tr>
<td>Comparison with sieving</td>
<td>identical results possible</td>
<td>identical results possible</td>
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<tr>
<td>Handling</td>
<td>fast</td>
<td>fast</td>
</tr>
<tr>
<td>Representative Sample</td>
<td>yes, small and large</td>
<td>yes, small and large</td>
</tr>
<tr>
<td>Amounts</td>
<td>amounts</td>
<td>amounts</td>
</tr>
</tbody>
</table>
Results of CAMSIZER (black) and CAMSIZER-XT (red) of sample #30

CAMSIZER distribution is wider, the results are not that accurate and repeatable as results from CAMSIZER XT.
Thank you for your attention!