Choosing the right particle characterization tool: Laser Diffraction or Imaging?
Customer perspectives

● “I need to measure my particles. What technique should I choose?”

● “I think I know what I need, I'm but curious to see if there are more suitable alternatives”

● “I know exactly what I want to measure and the best instrument for my application, can I have a quote please?”
Vendor perspectives

- “Where will the instrument be used?”
- “What particle parameters do you want to measure?”
- “What particle size range do you need to cover for your applications?”
- “Do you need to measure particle shape?”
- “Do you need to be able to compare data with other depts/sites?”
- “How much budget do you have?”
What you will learn today

- Basic principles of laser diffraction and imaging techniques
- Key information these techniques can provide
- Typical applications where they are used
- Guidelines for choosing between the two techniques
- Application examples
- Overview of instrumentation offered by HORIBA
Techniques - Particle size range

Laser Diffraction

Imaging Dynamic/Static
Laser Diffraction – How It works
Laser Diffraction – What it measures

Intensity vs. Angle => Particle size distribution

- Sphere equivalent diameter
- Volume weighted distribution
- Cumulative response of all particles in beam
- Does not measure individual particles
LD – Key Strengths/Limitations

**Strengths**
- Wide dynamic range - sub micron to millimetre
- Fast measurement - good statistical sampling
- Very repeatable - great for bulk characterization QC
- Widely used in regulated environments

**Limitations**
- Only gives sphere equivalent size
- Great for milled particles but less relevant to irregular shapes e.g needles
- No individual particle data
Laser Diffraction – Typical Applications

Incoming raw material QC
  • Pharmaceutical ingredients

Product/process development labs
  • Rapid characterisation/prototyping

Troubleshooting in central lab facility
  • Comparison of production sites

Outbound product QC and monitoring
  • Customer satisfaction, regulation etc.
Imaging – How It works

IMAGE ACQUISITION
Captured with a digital (CCD) camera

THRESHOLDING
Separates particles from the background

CALCULATIONS
Generation of results
Imaging – Dynamic vs Static

Static

Dynamic
Imaging – Size range per lens

Stream of particles

- The basic camera detects the larger particles.
- The complete particle flow is recorded by two cameras.
- The zoom camera analyzes the smaller particles.
Imaging – What it measures
Digital 2D image of individual particles

Particle size
- Length, Width, Circle equivalent diameter

Particle shape
- Sphericity, elongation, roughness etc.

Number/frequency weighted distributions
Imaging – Key strengths/limitations

Strengths
• More complete characterisation of size and shape for irregular particles
• Individual images - information rich
• Orthogonal technique for validation of LD

Limitations
• More care with sample dispersion required
• Data interpretation can be more complex
• Limited resolution sub-micron
Imaging – Typical applications

Quality control

Applications where particle shape is important
Additional visual proof required - qualitative

Research and development

Quick “look see” characterization type measurements
Validation of other methods such as LD

Troubleshooting

Forensic investigations, contaminants etc.
Sample dispersion
Wet vs Dry Dispersion

Dry/Air Dispersion
• Passive (freefall) or active (gas flow)
• Convenient for non-cohesive dry powders
• Not suitable for fragile cohesive materials

Wet/Liquid Dispersion
• Greater control over energy input - stirring, ultrasound etc.
• Convenient for particles already in suspension
• Particle solubility may be an issue
• Image contrast issues
Number vs volume

Number

Volume

Size

Size
## Diffraction/Imaging Comparison

<table>
<thead>
<tr>
<th></th>
<th>Laser Diffraction</th>
<th>Imaging</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle size</td>
<td>***</td>
<td>***</td>
</tr>
<tr>
<td>Dynamic range</td>
<td>***</td>
<td>**</td>
</tr>
<tr>
<td>Particle shape</td>
<td></td>
<td>***</td>
</tr>
<tr>
<td>Dry dispersion</td>
<td>***</td>
<td>***</td>
</tr>
<tr>
<td>Wet dispersion</td>
<td>***</td>
<td>**</td>
</tr>
<tr>
<td>Sampling/repeatability</td>
<td>***</td>
<td>**</td>
</tr>
<tr>
<td>Individual particle data/images</td>
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<td>***</td>
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</tbody>
</table>
Guidelines for choosing

- Particle size range
- Degree of polydispersity
- Dispersion method
- Non-spherical particles?
- Is particle shape important?
- Routine QC or research/forensic tool?
- Comparing/collaborating with data?
- Regulated environment?
Application example 1

Stainless Steel Powder for 3D printing
  Formed by gas atomisation => Spherical particles
  PSD determines packing density and uniformity
  Mechanical strength and surface properties

Suitable for dry dispersion
  Laser diffraction
  Dynamic Imaging
Application example 1

Stainless Steel Powder for 3D Printing

<table>
<thead>
<tr>
<th></th>
<th>LD</th>
<th>DIA</th>
</tr>
</thead>
<tbody>
<tr>
<td>D50</td>
<td>43.1</td>
<td>45.0</td>
</tr>
<tr>
<td>D10</td>
<td>23.4</td>
<td>22.8</td>
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<tr>
<td>D90</td>
<td>73.6</td>
<td>78.9</td>
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<tr>
<td>Std Dev</td>
<td>1.3</td>
<td>0.4</td>
</tr>
<tr>
<td>CV (%)</td>
<td>1.8%</td>
<td>4.9%</td>
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</tbody>
</table>
Stainless Steel Powder B

**Laser Diffraction (LD)**

**Dynamic Image Analysis (DIA)**

<table>
<thead>
<tr>
<th></th>
<th>LD</th>
<th>DIA</th>
</tr>
</thead>
<tbody>
<tr>
<td>D50</td>
<td>49.4</td>
<td>50.2</td>
</tr>
<tr>
<td>D10</td>
<td>36.0</td>
<td>33.7</td>
</tr>
<tr>
<td>D90</td>
<td>71.4</td>
<td>76.2</td>
</tr>
<tr>
<td>Std Dev</td>
<td>0.6</td>
<td>1.6</td>
</tr>
</tbody>
</table>
| CoV   | 1.1%   | 4.7%   | 4.6%
Application example 2

Alumina powder for abrasives
Hard, irregular shaped particles
Size and shape => abrasive properties
Application example 2

<table>
<thead>
<tr>
<th></th>
<th>D50</th>
<th>D10</th>
<th>D90</th>
</tr>
</thead>
<tbody>
<tr>
<td>LD (Dry)</td>
<td>97.2</td>
<td>11.2</td>
<td>135.2</td>
</tr>
<tr>
<td>LD (H₂O)</td>
<td>102.4</td>
<td>72.4</td>
<td>153.2</td>
</tr>
<tr>
<td>DIA</td>
<td>105.7</td>
<td>73.5</td>
<td>159.3</td>
</tr>
<tr>
<td>CoV</td>
<td>4.2%</td>
<td>1.6%</td>
<td>8.4%</td>
</tr>
</tbody>
</table>
Alumina Shape

Q3 (SPHT=0.9) = 52.5 %
Q3 (Symm=0.9) = 30.0 %
Q3 (b/l=0.9) = 93.9 %

Mean value SPHT3 = 0.874
Mean value Symm3 = 0.914
Mean value b/l3 = 0.754

Sphericity => 1 – less abrasive
Application example 3

Solubility of pharmaceutical active ingredient

Efficacy of drug measured with dissolution profile
Particle size distribution an important factor
Smaller particles dissolve more quickly

Particle shape can also be important
Rough particles – higher surface area – faster dissolution
Application example 3

- Trial A (Rough Core), Sphericity = 0.87
- Trial B (Smooth Core), Sphericity = 0.97

Graph showing the percentage of drug dissolved over time for two trials.
Complementary Tools

Certain circumstances may be advantageous to use both

Laser Diffraction – daily workhorse

Imaging (microscope) – primary reference method

Validation of Laser Diffraction methods in regulated environments e.g. Pharma
Disclaimer

There is no one size fits all!

If in doubt ask your vendor to measure samples with both techniques!
Dynamic Range of the HORIBA Particle Characterization Systems

https://static.horiba.com/fileadmin/Horiba/Products/Scientific/Particle_Characterization/Particle_Guidebook.pdf
Contact Info

Dr Carl Levoguer - Consultant
Carl.levoguer@googlemail.com

Julie Chen Nguyen
julie.nguyen@horiba.com

Product Enquiries
info@horiba.com