COMPARISON OF LASER DIFFRACTION AND IMAGE ANALYSIS UNDER IDENTICAL DISPERSING CONDITIONS

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ABSTRACT

Laser diffraction has developed into the leading principle for particle size analysis. This principle assumes a spherical particle shape in its optical model to obtain a particle size distribution. The size distribution will be different from those obtained by methods based on other physical principles (e. g. image analysis). Not all particles are spherical but all diffraction patterns are point symmetric. Sympatec's particle size analyser HELOS uses a multi-element photo detector with semi-circular rings. The results are independent of a systematic orientation of the particles. Laser diffraction instruments with asymmetric detectors are also used to measure average shape and size characteristics of the particles, but the results strongly depend on the orientation of the particles in the measuring zone and change with the flow condition.

For shape and size characterisation Sympatec has introduced the QICPIC dynamic image processing system [1]. The dispersers for dry powders and suspensions are modular and exchangeable between QICPIC and HELOS. This combination of powerful dispersion and high frame rate allows for the acquisition and analysis of extreme numbers of even > 10^7 particles in short times. With statistical errors far below 1%, image analysis now even reaches the reproducibility of laser diffraction with traceability to the individual particles and shape information in addition.

The comparison of image analysis and laser diffraction measurements under the same particle dispersing conditions is now possible.

1 INTRODUCTION

How to handle the influence of different particle shapes on Laser Diffraction (LD) results is an issue in powder handling with a long history. In all LD Systems an angular variation of the intensity pattern of the diffracted light is recorded. The intensity pattern, however, does not contain the phase information of the diffracted light anymore and is averaged on the measuring time. Exact reconstruction of the shape is impossible from the reduced data. It is even difficult to interpret the relationship between the diffraction pattern obtained and the equivalent spherical size distribution in the case of non-spherical particles.

Nevertheless, some extensions of LD systems are available to measure a size distribution and some shape characteristics of the particles [1].

Variations in the diffraction signal with the azimuthal angle can be detected with the help of wedge shaped photo elements. Here the signal not only depends on particle shape but also on the orientation of the particles in the measuring zone. Thus the signal depends strongly on the flow condition of the dispersing system.

It is even theoretically possible to include ellipsoidal or rectangular particles with a pre-known aspect ratio in the optical model of the LD evaluation software. This method can correct the influence of the spherical particle assumption on the particle size distribution. But the diffraction matrix for non-spherical particles is less well conditioned for the inversion procedure.

In our opinion the best strategy is to stay with the classical concept of LD and to accept the influence of the particle shape on the result. The orientation dependence within the projection plane at least should be eliminated with a semi-circular detector.

If shape information should be detected, an IA system shall be used. But until now the comparison of IA with LD has been limited because of differences in dispersion and average particle orientation. In many IA systems the particles are statically observed on glass slides, where we have free flowing particles in an LD system. Because particle detection had been time consuming in the past and because of low frame rates of standard imaging devices only a few thousand particles had been measured.

2 LINK OF TWO METHODS

2.1 Realisation

At the PARTEC 2004 a new concept of digital IA has been presented [2], combining for the first time high speed image analysis with powerful dry dispersion in a table-top instrument. The short exposure time of 1 ns eliminates any motion blur even at particle speeds of 100m/s. This makes it possible to apply the same dispersing devices which have been developed for the standard laser diffraction line of instruments (figure 1 and 2).

The high frame rate of up to 500 fps at full resolution of 1024x1024 pixel and the fast handling of large particle numbers per measurement (> 10^7) fundamentally overcomes the weakness of typical image analysis systems – low particle numbers resulting in large statistical errors [3].

Now for the first time image analysis data is available with the same statistical significance as of LD results. The results of both instruments can now be obtained under identical dispersion conditions. Therefore a direct comparison of both methods is possible.

Figure 1: OASIS/L wet and dry dispersing system set-up in the QICPIC image analysis sensor.

Figure 2: OASIS/L wet and dry dispersing system set-up in the HELOS laser diffraction sensor.

2.2 Comparison of detection principle

The optical set-up of the QICPIC image analysis system (figure 3) and the HELOS laser diffraction system (figure 4) is in its principle very similar. In both systems a parallel beam of light is created by an adaptable beam expansion unit. This beam of light is directed to the measuring zone of the dispersing system. In the LD system a lens transforms the diffracted light to a diffraction pattern. This pattern is in fact an amplitude and phase distribution of the electromagnetic wave. In the LD system a multi-element photo detector records the radial intensity distribution, which contains only half of the information in that pattern. In this simplified comparison this lens may be regarded as a first part of a telecentric imaging lens. With the help of a second lens the full amplitude and phase distribution of the diffraction pattern is back-transformed to a real image, which is recorded by the image sensor.

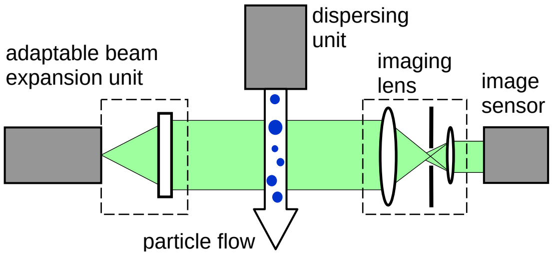

Figure 3: Optical set-up of the QICPIC image analysis sensor.

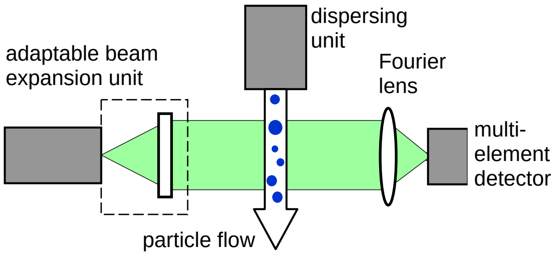

Figure 4: Optical set-up of the HELOS laser diffraction sensor.

To measure the particle size in SI length units, the IA system is calibrated with a certified standard scale. The effective magnification of the imaging lens and the size of the sensor are measured and are thus traceable back to the standard metre. The scale may be determined using pixel or even sub-pixel accuracy if the image quality allows for it. In practice the calibration is a small correction to the theoretical value of the optical design.

A laser diffraction system like an IA System is based on first principles. In a strict sense a calibration is not required, but also a small correction of the theoretical value is necessary. To measure the particle size in SI length units, the wavelength of the light, the scale of the detector and the exact focal length of the system must be known. This length is not just the focal length of the lens but depends also on the collimation of the beam. The HELOS LD system uses an auto-focus procedure which can detect the focus location accurately. The focal length then can be measured by a ruler and is in principle also traceable back to the standard metre.

With IA and LD Systems it is still necessary to qualify the whole system (sensor and disperser) with the help of reference materials. This will confirm the correct scale of the instrument and reveal misalignment, optical defects or malfunction of the dispersing or feeding system. We will show later that this material does not need to be spherical.

A primary method is defined as one where the dimensions of length and weight are directly traceable to International Standards. With respect to the presented comparison it is difficult to understand why IA is considered a primary method but LD is not.

2.3 Differences in experimental conditions and sensitivity.

Even when the same dispersing systems are used for both IA and LD some experimental conditions still remain different.

In IA the depth of focus must be considered carefully. For wet dispersion the design of the standard cuvettes is slightly different. It is not always possible for an IA system to share the same optical path length with an LD system.

In LD instruments, contamination of liquid is subtracted as a background signal with the help of a reference measurement. In IA a particle filter on shape and size may be applied to recognize and eliminate air bubbles and overlapping particles.

An IA system does not detect particles below the measuring range but is very sensitive to coarse and overlapping particles. The overlapping of particles is a two dimensional effect compared to the three dimensional effect of multiple scattering. It can be minimized only by using a very low optical concentration. In LD a higher concentration should be used to obtain a good signal to noise ratio. In IA the optical concentration is defined as a geometrical obscuration. In LD systems it is defined as the extinction of the laser beam in focus. According to the Fraunhofer diffraction theory this value is twice the geometrical obscuration of an IA system.

A LD system in contrast may detect particles below the measuring range and include them into the smallest size class, but is less sensitive for large particles because of the reduced signal to noise ratio near the focus.

Even in the case of a monodisperse sample the LD result will be a distribution of sizes because smoothing constraints are commonly required in the inversion procedure. Thus it is advisable to compare polydisperse particle samples with a size distribution within at least three size classes of the LD system.

These differences can be minimized with a careful selection of the particle system under test with a narrow but still sufficient broad size distribution.

2.4 Comparison of the evaluation modes

The LD technique assumes a spherical particle shape in its optical model. For non-spherical particles a size distribution is reported, where the predicted diffraction pattern for the volumetric sum of spherical particles matches the measured diffraction pattern.

Many different diameters and evaluation modes may be selected as an evaluation mode within the software of IA systems. The equivalent projection area of a circle (EQPC) is assumed to give the best agreement with LD.

In theory both evaluation modes will give the same results if the particles are spherical.

It is a common statement that IA can only be compared to LD with spherical particles. It is even claimed that a spherical standard reference material is absolutely necessary to certify a LD system.

As far as diffraction by opaque particles is concerned, the diffraction pattern of that particle corresponds to the two dimensional Fourier transform of the particle shape projection. For arbitrarily shaped particles it is possible to calculate an equivalent LD pattern from the images of the IA measurement.

The scale of both systems may now be compared and aligned by direct signal comparison without even relying on evaluation modes.

3 RESULTS

3.1 Comparison with spherical particles

Opaque spherical glassy carbon particles with a narrow size distribution have been selected for the first comparison between QICPIC and HELOS sensors. The result of the laser diffraction analysis is obtained with the advanced HRLD iterative Method. A standard Phillips-Twomey inversion method will show a broader distribution. The IA evaluation is based on the equivalent projection area of a circle (EQPC).

The particle size distributions are presented in figure 5. They show a perfect alignment between the image scale and the focal length of the LD lens. The inversion process only leads to a slightly broader size distribution. The alignment of both techniques can be cross-checked by direct comparison of the measured diffraction pattern with the simulated pattern from IA data. A difference in scale of the focal length of the HELOS compared to the magnification of the QICPIC is then observed as a shift between both patterns.

For spherical particles the results of both methods are nearly identical. These results may be used to qualify both the LD evaluation method and the HELOS LD instruments. For the evaluation software it is only necessary to do this test once with a constant dataset.

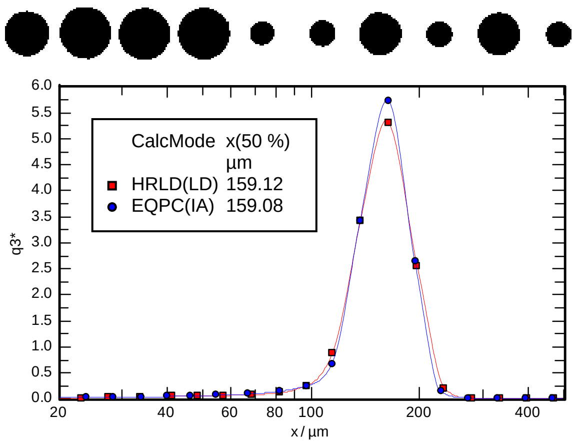

Figure 5: Size distribution of opaque spherical glassy carbon particles. Sample images are shown above. The HELOS (LD) result is obtained with the HRLD evaluation mode. The QICPIC (IA) result is obtained with the EQPC evaluation mode.

3.2 Comparison with irregular particles

Irregular silicon carbide particles are used by Sympatec to certify and recertify IA and LD sensors according to Sympatec's own specifications. Advantages compared to spherical material are the much simpler wet and dry particle feeding resulting in a better instrument repeatability. A huge database of results is available at Sympatec and the long-time availability is guaranteed.

The particle size distribution results are presented in figure 6. This is an experimental demonstration that for non-spherical particles the EQPC and the equivalent spherical diameter measured by LD are not the same.

To overcome this fundamental problem all 6886 images of the same QICPIC measurement are converted by a Fast Fourier Transform and accumulated to an equivalent laser diffraction signal. These values are passed through the LD inversion algorithm. This procedure leads to a very good agreement of both results (figure 7).

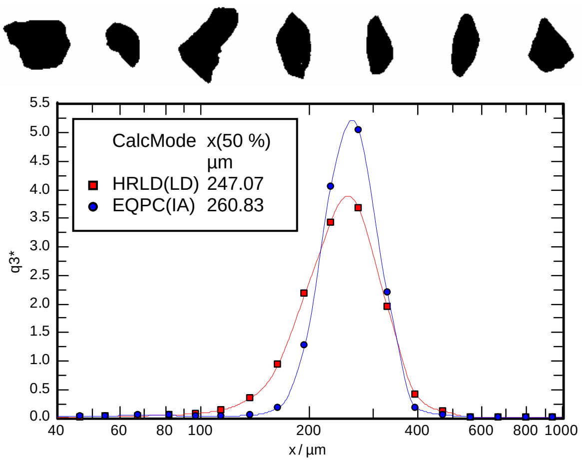

Figure 6: Size distribution of irregular SiC-P80 particles. Sample images are shown above. The HELOS (LD) result is obtained with the HRLD evaluation mode. The QICPIC (IA) result is obtained with the EQPC evaluation mode.

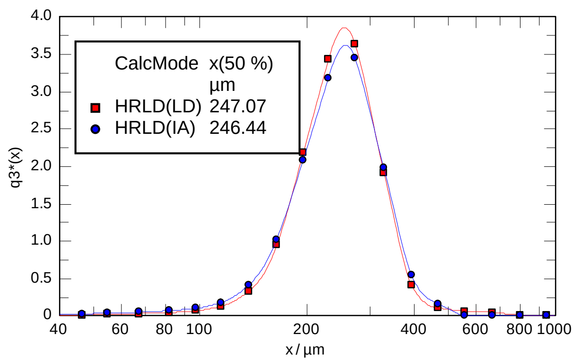

Figure 7: Size distribution of irregular SiC particles calculated from the same measurement data as in figure 6. The HELOS (LD) result is obtained with the HRLD evaluation mode. The QICPIC (IA) result is obtained after calculating an equivalent diffraction pattern by Fast Fourier Transform and using the HRLD laser diffraction evaluation mode.

This first result shows that the observed main differences between LD and IA only arise from the effect of the particle shape on the evaluation procedure.

4 CONCLUSION

A non spherical material has been characterised to an equivalent laser diffraction result only by using image analysis data. This approach has finally solved the fundamental problem of how to handle the influence of the infinite form of appearances of particle shapes on the LD results.

This data has been obtained by the QICPIC image analysis sensor, where the image scale is calibrated by a certified standard. It is a common opinion that a laser diffraction instrument can only be traced back correctly with spherical reference particles only. This is true for most instruments where corresponding high quality IA data is not available. It is possible even with irregular shaped particles, if shape and alignment of the particles in the dispersing system are characterised and certified completely by IA, as demonstrated.

It is thus completely valid to prove the performance of individual HELOS LD instruments with irregular shaped particles. The validation of the LD evaluation software with the help of spherical particle data is only needed once in a software development lifecycle.

This approach is currently in a conceptional phase. It is planned to be used in future by Sympatec to improve the comparability of both techniques and to tighten the internal specifications used for production control.

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