Bettersize
BETTER PARTICLE SIZE SOLUTIONS

APPLICATION NOTE

Oversize particle analysis combining laser diffraction and dynamic image analysis with the Bettersizer S3 Plus

The limitations of laser diffraction in particle size analysis

Dr. Alex Spieß , Düsseldorf
Dr. Christian Oetzel , Odelzhausen
3P Instruments GmbH & Co. KG, Odelzhausen

Keywords: Oversize analysis, Particle size measurement, Optical combination measuring device, Laser diffraction, Dynamic image analysis

Introduction

In various applications such as the manufacturing of inkjet printing powders or abrasives, the presence of a small number of coarse particles within a predominantly fine sample presents a significant challenge. Conventional methods like laser diffraction [1] face difficulties in accurately and quantitatively detecting these oversize particles due to their limited representation in the measured scattering light spectrum and subsequent mathematical fitting process. Consequently, the resulting particle size distribution curve either fails to capture their presence entirely or includes them in inadequate proportions.

The Bettersizer S3 Plus [2] offers a sophisticated solution to overcome this challenge by employing a synchronous measurement approach that combines laser diffraction with dynamic image analysis [3] . By integrating a high-speed CCD camera within the device, it captures images of particles larger than approximately 40 µm simultaneously to the determination of the scattered light spectrum. These images are intelligently processed and combined with the laser diffraction data by an advanced algorithm, enabling the precise determination of the particle size distribution of the sample. In order to demonstrate the capabilities of this measuring device, various mixtures of two quartz powders will be utilized as examples.

2.Experimental Section and Results

The samples of the quartz sands "P3A" and "FH31" and their mixtures were measured in water using the Bettersizer S3 Plus[3] with BT-803 circulation and dispersion system. All samples were measured at least three times using a standard operating procedure (SOP) outlined in Table 1. The obtained results were averaged to ensure accuracy and consistency.

Table 1. *SOP measurement and evaluation parameters for all measurements*

Sonication	50W, 60s
Stirring Speed	1200 U/min
Obscuration	8-12 %
Mathematical Model	Mie theory, Refractive index:1,54-0,01i
Modes	Laser diffraction + CCD (X0.5), maximum Feret diameter Fmax > 500 µm

The mode used in this study combines laser diffraction and CCD camera measurements with a 0.5x objective lens (15x magnification). The selected equivalent diameter for image analysis is the maximum Feret diameter, Fmax, and the threshold value for particle inclusion is 500 µm.

Dry mixtures of both powders were prepared in a roll mixer, with varying weight percentages of FH31 (0.5, 1.0, 1.5, and 2.0 wt.%) in P3A. The quantity ratio of the two quartz samples at a FH31 content of 1wt.% is depicted in Figure 1, showing that the amount of FH31 corresponds to only a few grains.

Figure 1: Representation of the amounts of 99 wt.% P3A (left) and 1 wt.% FH31 (right) to illustrate the 1 wt.% FH31 content in a mixture with P3A.

The Bettersizer software offers a valuable functionality to extract individual results from the combined measurement of laser diffraction and image analysis. The data presented in Table 3 correspond to the laser and combined measurements derived from the same experimental setup. Figure 2 illustrates the histograms of the pure quartz samples P3A and FH31, with detailed size values provided in Table 3.

Figure 3 demonstrates that even at a low weight percentage of only 0.5 wt.% FH31, the combined measurement reveals a distinct peak around 500 µm. This peak represents the presence of oversized particles that cannot be effectively characterized through laser diffraction alone. While the characteristic values such as D10, D50, and D90 remain consistent between the two experiments, a significant increase in D100 is observed, from 277 µm in laser diffraction to 539 µm in the combined test result. Similarly, an increase in FH31 content to 1 wt.% yields comparable results, with D100 reaching 538 µm (as indicated in Table 3).

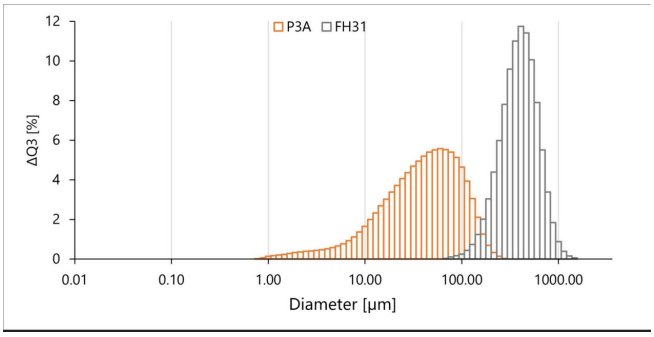

Figure 2: Histograms of the particle size distribution of pure quartz samples P3A and FH31 (combined method).

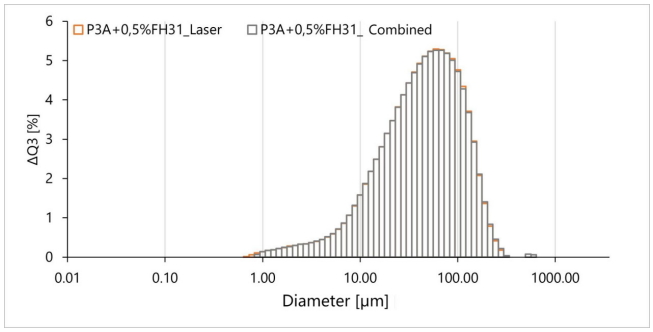

Figure 3: Particle size distribution of 0.5 wt.% FH31 in P3A, determined by a combined measurement (gray) and the extracted laser diffraction experiment (orange).

Table 2 provides an overview of the five largest particles captured by the CCD camera for the P3A+0.5%FH31 sample, along with selected characteristic particle shape parameters calculated by the software. Notably, only one particle precisely aligns with the mode specified in Table 1 (Fmax >500 µm) and contributes to the overall particle distribution as Particle No. 1. Through the integration of both optical measurement techniques, the results of the image analysis are weighted based on the number of particles detected. Furthermore, the particle size distribution is presented as a volumetric distribution (Q3), where the presence of a few large particles exerts a significant influence on the overall distribution due to their larger volumes.

Table 2. *Images of the five largest particles captured by the camera for P3A+0.5%FH31,including some characteristic particle shape parameters [4], sorted by CE diameter.*

No.	1	2	3	4	5
CE diameter [µm]	486,7	394	331,2	306	280,6
Length [µm]	504,3	405	345	394,4	345
Width [µm]	485,4	390	330	235,4	240
L/D	1,039	1,038	1,045	1,675	1,437
Circularity	0,903	0,903	0,894	0,876	0,847
Fmax [µm]	556,5	439,3	358,8	394,9	376,3
Fmin [µm]	479,1	387,7	330	235,4	235,9
Image					

The difference between the D100 value of the combined measurement in Table 3 (539 µm) and the Fmax value of the image analysis in Table 2 (556.5 µm) is attributed to the fact that D100 is determined from a cumulative smoothed overall curve, whereas Fmax represents an equivalent diameter genuinely measured on an individual particle.

With further increase in the oversized FH31 particles in the mixture to 1.5 wt.%, it can be observed that the coarse peak can be better resolved by laser diffraction (Figure 4). However, the results of pure laser diffraction and the combined measurement are not entirely identical in the coarse range. The coarsest particle detected by the CCD camera has an Fmax value of 587.4 µm, while the D100 value of the combined curve (due to smoothing and the representation of classes in the distribution) is 614.2 µm. However, laser diffraction shows a D100 value of 698.5 µm, significantly coarser. Therefore, the combination with image analysis leads to a more accurate result, in this case, corrected to smaller diameters. The use of a sample with 2 wt.% FH31 in P3A yielded the same results.

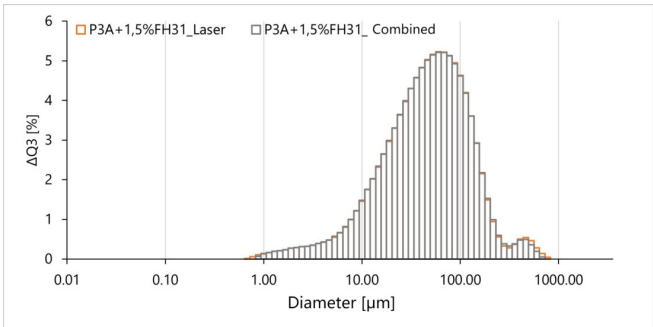

Figure 4: Particle size distribution of 1.5 wt.% FH31 in P3A, determined by a combined measurement (gray) and the extracted laser diffraction experiment (orange).

Table 3. *Summary of the characteristic particle size values for all discussed samples.*

Sample	D10 [µm]	D50 [µm]	D90 [µm]	D100 [µm]
P3A	9,1	38,8	103,0	242,3
FH31	197,5	364,0	609,5	1354
P3A+0.5%FH31, Laser	9,7	42,0	117,5	277,3
P3A+0.5%FH31, combined	9,7	42,1	118,8	539,0
P3A+1%FH31, Laser	9,9	43,7	120,9	315,5
P3A+1%FH31, combined	9,9	43,7	122,1	537,7
P3A+1.5%FH31, Laser	10,2	44,6	132,5	698,5
P3A+1.5%FH31, combined	10,2	44,4	131,7	614,2

Conclusion

Laser diffraction is a powerful method for rapidly measuring particle size across a wide size range. However, it faces challenges in accurately determining the size of low volume fractions of oversized particles. To overcome this limitation, the combination of laser diffraction with dynamic image analysis proves to be a valuable solution. The integration of these techniques in the Bettersizer S3 Plus enables efficient and reliable particle size analysis, as exemplified by the case study involving two quartz sands of varying coarseness. This combined approach offers a comprehensive and precise characterization of particle size distribution, ensuring accurate results for a wide range of applications.

Reference

[1] ISO 13320: 2020 Particle size analysis - Laser diffraction methods

[2] ISO 13322-2: 2006 Particle size analysis - Image analysis methods - Part 2: Dynamic image analysis methods

[3] Particle World Edition 19, p. 4- 8, 3P Instruments

[4] ISO 9276-6:2008 Presentation of particle size analysis results: Descriptive and quantitative presentation of particle shape and morphology

Bettersize

BETTER PARTICLE SIZE SOLUTIONS

www.bettersizeinstruments.com
info@bettersize.com

Bettersize Instruments Ltd.

Address: No. 9, Ganquan Road, Jinquan Industrial Park, Dandong, Liaoning, China

Postcode: 118009

Tel: +86-415-6163800

Fax: +86-415-6170645

Bettersize Inc.

Address: Suite K-2, 3188 Airway Ave, Costa Mesa, CA 92626, United States

Tel: +1 833-699-7493 (SIZE)

Download Our Application Notes:

Visit Our Bettersizer S3 Plus Site: