Experiences with Dry Dispersion and High-Speed Image Analysis for Size and Shape Characterisation

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ABSTRACT

An innovative imaging sensor has been developed, which allows for the direct use of well established dry dispersers. It combines a new light source which provides an exposure time of less than 1 ns and an adaptable optical system for perfect illumination and imaging of the fast particles on a high speed camera, an integrated image pre-processor and a Gigabit digital transmission line to the computer. A first device was exhibited on the ACHEMA 2003 and presented on the Innovative Forum of the PSA 2003, showing that particles can be clearly imaged and analysed at the output of a well established and proven dry dispersing injection system. Meanwhile, the device has proven its projected performance in a multitude of applications. It allows for the acquisition and analysis of more than 10^7 randomly oriented particles in short times, resulting in very low statistical errors. Examples of possible applications are displayed and discussed. Results of resolution, detection limits, reproducibility and system-to-system-reproducibility are reported, showing that image analysis now even reaches the reproducibility of particle sizing of non counting based techniques with traceability to the individual particles and shape information in addition.

Keywords: High Speed Image Analysis, Dry Dispersion, Particles Size, Particle Shape

1 INTRODUCTION

Image analysis (IA) is widely in use for the characterisation of shape and size of particles. As with all counting methods, low particle counts usually lead to large statistical errors. It might be acceptable to investigate only a few particles when the sample is nearly monodisperse. However, for polydisperse or even wide or multimodal distributions acceptable measurement repeatability is only obtained, if a very large number of particles are acquired.

1.1 Statistics

If a single particle from a sample of size *n* is considered, its probability of being finer than x_i is $Q_0(x_i)$. Since there are only two possible results (finer than x_i or not), the fundamental error (expressed as variance σ) of the fraction $Q_0(x_i)$ is calculated as the variance of Binomial distributions (Leschonski 1974, Sommer 1986, Allen 1990, Rhodes 1998):

$$\sigma_{0,i}^2 = \frac{Q_0(x_i)\cdot(1-Q_0(x_i))}{n} \quad \textbf{(1)}$$

The distribution of a sample is an estimation for the distribution of the whole product. Under the assumption, that the estimations for the Q_0-values are asymptotically normal distributed, the estimation theory (e.g. Bronstein 1989) provides the tools to calculate the maximum error E_{max}:

$$E_{\max} = \sigma_{0,i} \cdot z_c \quad \textbf{(2)}$$

where z_c is the critical *z*-value related to the defined confidence level *c* according to the standard normal distribution. Inserting eq. 2 into eq. 1 leads to the minimum particle number n_{min} required for a defined error E_{max} at confidence level c because the maximum of $Q_0(1-Q_0)$ is ¼:

$$n_{\min} = \frac{z_c^2}{4 \cdot E_{\max}^2} \geq \frac{Q_0(x_i) \cdot (1 - Q_0(x_i)) \cdot z_c^2}{E_{\max}^2} \qquad \textbf{(3)}$$

Example: E_{max} = 1%, c = 95% (z_c=1.96) $\Rightarrow$ $n_{min} \approx 9600$. So as a rule of thumb $n_{\min} \approx 1/E_{\max}^2$ can be used as a first approximation.

Unfortunately, the general calculation for a volume- or mass-based distribution is not as simple. The coming ISO standard 14488 (2003) recommends to convert the measured mass-distribution into the corresponding number distribution by a spreadsheet program. Then the equations mentioned above can be used. As the mass *m* of the particles is proportional to n^3, the errors at the coarse end of the number distribution will be decisive for the maximum error of the mass distribution.

1.2 New approach

For IA the number of particles can be increased by two independent methods: the number of particles per image (npi) and the number of images (frames) per second (fps). The npi is limited by the risk of overlapping particles, which usually require sophisticated and time consuming image processing algorithms for their separation. The maximum number of fps is not only limited by the camera, but strongly by the time for the analysis of the image and the bottleneck of the subsequent data path to the storage media.

A new device has been exhibited on the ACHEMA 2003 and presented on the Innovative Forum of the PSA 2003 by Witt et. al. (2002), combining well established powerful dispersers with a high speed image analysis sensor. The dispersers are used to create a particle flow with well separated particles, so that high npi's can be obtained. The combination of a highly stable 1 ns pulsed light source illuminating the particles by a parallel beam with adjustable width, and the imaging of the particles by a lens system with aperture stop eliminates any motion blur and creates images of high contrast for even transparent particles. This is a prerequisite for fast analysis and enables high fps. Meanwhile the device has reached its projected performance and operates at up to 500 fps. It allows for the acquisition and analysis of more than 10^7 randomly oriented particles in short times, resulting in very low statistical errors.

2 REALISATION

Figure 1 shows the current realisation of the system. Up to 500 fps with 1024x1024 square pixels of 10 µm x 10 µm and 256 grey levels resulting in 500Mbytes/s are acquired, processed, compressed, transferred and stored in a high-performance database.

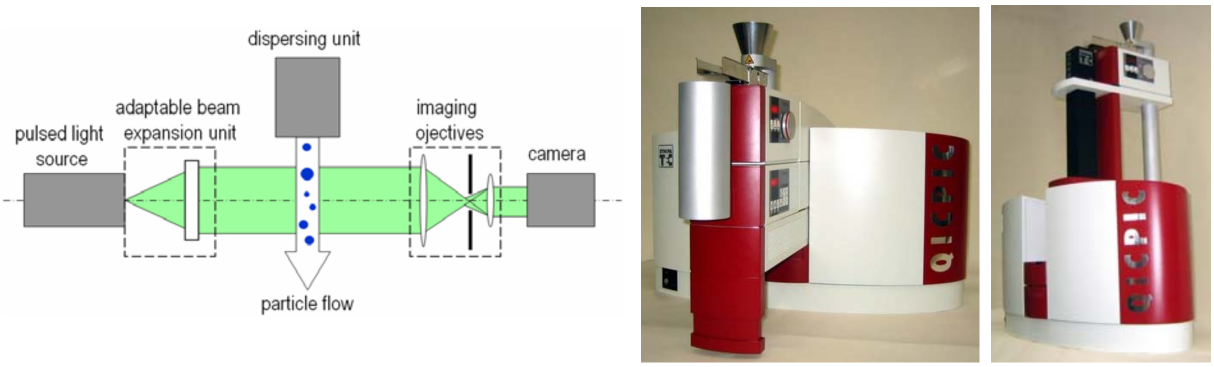

Figure 1: Optical set-up and view of the image analysing sensor QICPIC (left), QICPIC with dry disperser RODOS (centre, please refer to Leschonski, Röthele, Menzel (1984)), QICPIC with gravity disperser GRADIS (right), both using with vibratory feeder VIBRI.

Different measuring ranges can be selected by software: 3.3 to 1,140 µm, 10 to 3,410 µm and 20 – 6,830 µm (according to the coming ISO 13322-2) are currently implemented. Different dispersion units can be applied to an open measuring zone, e.g. a dry disperser with injection system for fine particles (RODOS) or a gravity disperser (GRADIS) for coarser particles. A powerful selection and display facility of particles has been implemented in the software, which is fully compliant with CFR 21 Rule 11 and concurrently supports other particle sizing instruments using laser diffraction (HELOS), ultrasonic extinction (OPUS) or photon-cross-correlation (NANOPHOX) in an extended network.

2.1 Camera System

The camera system and especially the image pre-processing plays the most important role to reach high fps. As current PC interfaces are not able to handle continuous data transfer streams of up to 500 Mbytes per second, specialised signal processing hardware has to be embedded in the camera system to reduce the data transmission requirements. The used embodiment is able to reduce the data transfer rate without any trade-offs concerning the flexibility and quality of the grey value image analysis. The three major steps of the image evaluation are performed within the camera, as displayed in Figure 2. Most of the time is spend on grey value analysis of the particle images and the background to obtain accurate threshold values. The threshold values are applied at the binarisation step which reduces the data to 1/8th of the original image. The binary data are further compressed, before the results are sent to the computer and stored in the database in real-time. Every particle imaged by the system is stored for subsequent evaluation and re-evaluation. The final image evaluation of particle shapes and sizes is currently performed by the external computer system.

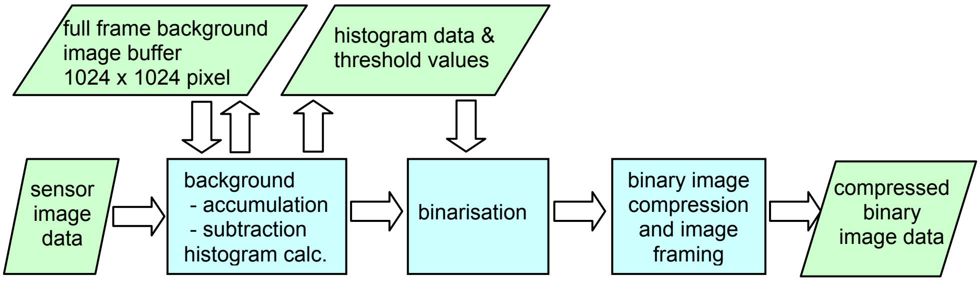

Figure 2: Internal image processing steps of the camera system

Using a high performance signal processor of latest technology the image capture, data transfer, image analysis, binarization and compression are performed concurrently. An optimized assembler code, features like single instruction/multiple data and an efficient buffer management with several direct memory accesses (DMA) working concurrently, shrink the overall processing time for a complete image to less than 2 ms as shown in Figure 3.

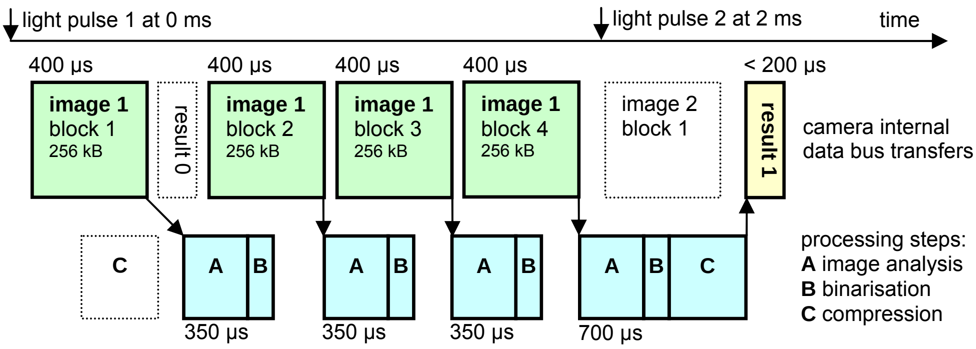

Figure 3: Timeline of image block data transfers and processing steps within the camera system. Image block processing and block transfers must be done in parallel to achieve maximum frame rate.

3 RESULTS

3.1 Dispersion of Particles

As efficient dispersers can be used, which are able to create de-agglomerated, diluted fields of particles, the overlap of particles is widely avoided. So high npi can be obtained, as demonstrated in Figure 4.

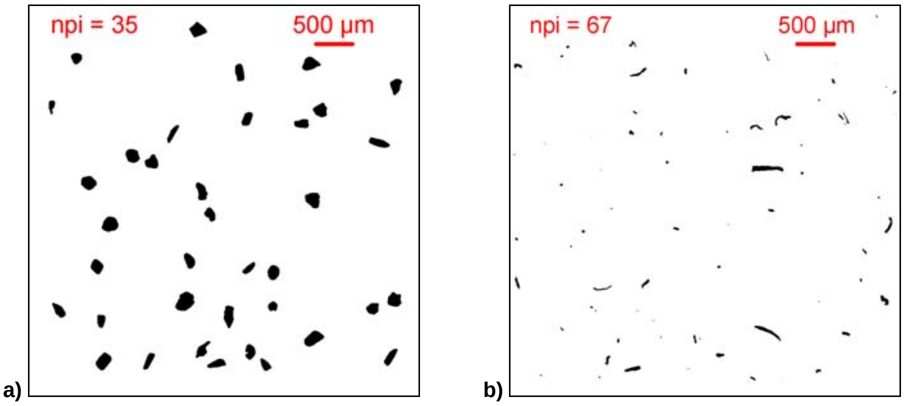

Figure 4: Two example of binary images showing that the dispersion units create aerosol beams with no overlapping particles even at very high npi's. Left: SiC particles, right: methyl cellulose fibres, both dispersed with RODOS.

3.2 The Influence of the Minimum Particle Number on the Measurement Repeatability

Figure 5 shows results of coffee measurements and the influence of the coarse particles on the mass distributions in detail. m is the sample mass used, n_i is the number of particles acquired.

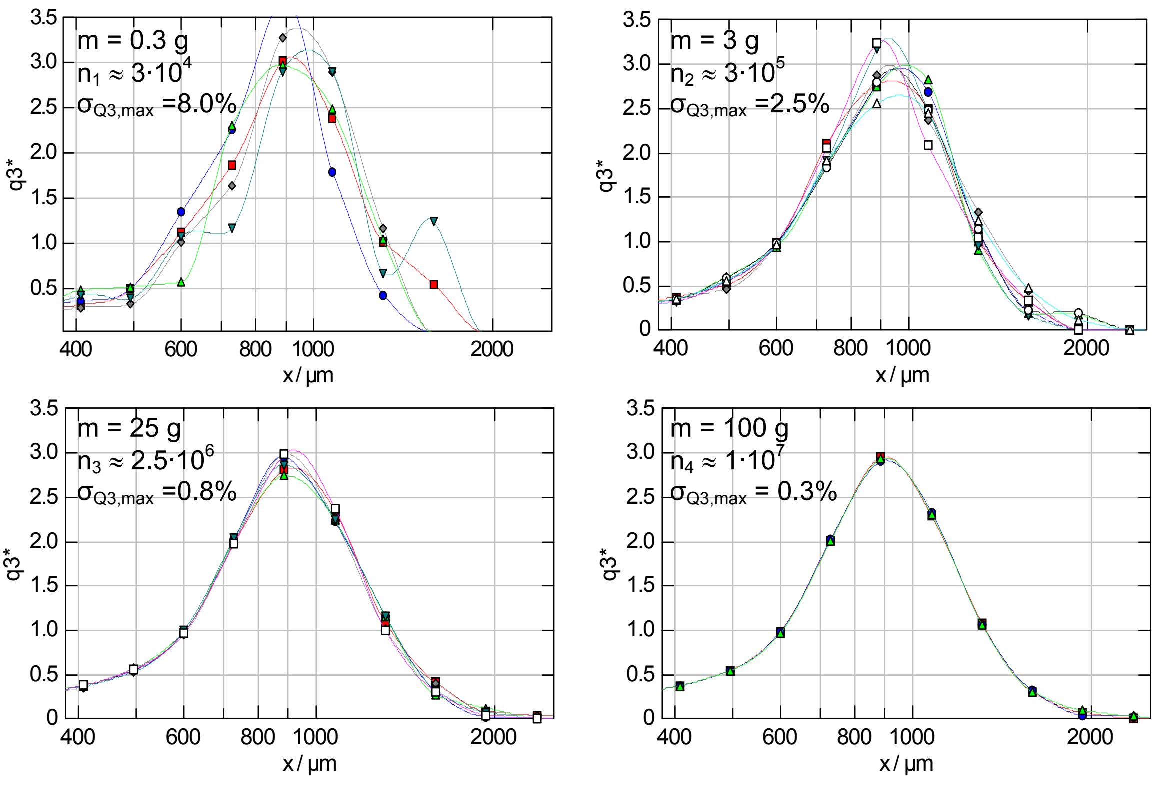

Figure 5: Coffee, excerpt the coarse part of the logarithmic density distributions for different number of particles (n_i), all measured with QICPIC with measuring range M7 (10 µm to 3,410 µm) and dry disperser RODOS

As expected the standard deviation $\sigma_{Q3,max}$ decreases significantly with an increasing number of particles. For the last measurement series the maximum standard deviation $\sigma_{Q3,max}$ is located at x = 1750 µm. At the same size the corresponding number distribution gives a standard deviation σ_{Q0} = $1.5\cdot10^{-4}$.

As mentioned by the ISO guideline 14488, the mass distributions have been converted to number distributions. The resulting $\sigma_{Q0,max}$ values are plotted in Figure 6(left). The comparison with the theoretical values for E_{max} shows a good compliance. Because real samples were used, the remaining errors by sampling and segregation become more and more dominant for large particle numbers. Hence the $\sigma_{Q0,max}$ values for large particle numbers differ from those predicted by statistics only.

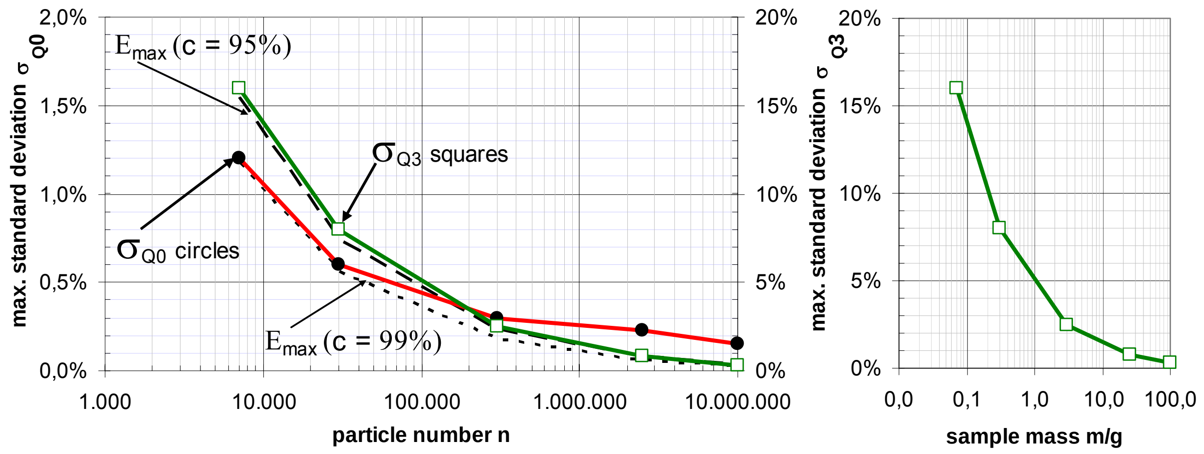

Figure 6: left: E_{max} (dashed) for c = 95%, c = 99% (dotted) and $\sigma_{Q0,max}$ (circles) on left axis; $\sigma_{Q3,max}$ (squares) on right axis, all versus particle number n; right: $\sigma_{Q3,max}$ as function of sample mass m

On the second axis the maximum standard deviation for the mass distributions Q_3 is plotted for comparison. Naturally the relation of both depends on the product and its kind of PSD. Nevertheless, if such a graph is generated for a specific product, it provides an estimation of how many particles are necessary for a defined maximum deviation as demonstrated in the diagram on the right hand side. For this product and PSD m ≥ 20 g or n ≥ 2,000,000 particles are necessary to reach $\sigma_{Q3,max} \leq 1\%$.

4 SUMMARY

The presented combination of powerful dispersers with high speed image analysis fundamentally overcomes the weakness of typical image analysis systems – low particle numbers resulting in large statistical errors. Investigations of particle size at the outlet of the dry disperser RODOS at 500 fps acquire particle numbers of more than 10^7 resulting in statistical errors comparably small as achieved with renowned laser diffraction instruments. In addition various size and shape parameters are available. All results are traceable to the individual particles. This opens new fields of applications with outstanding precision.

5 REFERENCES

ALLEN, R., (1990), Particle Size Measurement, Chapman and Hall

BRONSTEIN, I.N., (1989), Taschenbuch der Mathematik, Harri Deutsch, p.685

ISO/DIS 14488, (2003), Sample Preparation – Sampling and Ssample Splitting of Particulate Materials for the Characterization of Particulate Properties (not published yet)

KOGLIN, B., LESCHONSKI, K., ALEX, W., (1974), Teilchengrößenanalyse (Probennahme, Dispergieren), Chem.-Ing. Tech. 46(7), p.289-292

LESCHONSKI, K., RÖTHELE, S., MENZEL, U., (1984). Entwicklung und Einsatz einer trockenen Dosier-Dispergiereinheit zur Messung von Partikelgrößenverteilungen in Gas-Feststoff-Freistrahlen aus Laserbeugungsspektren; Part. Charact., 1, 161-166

RHODES, M., (1998), Introduction to Particle Technology, John Wiley & Sons

SOMMER, K., (1986), Sampling of Powders and Bulk Materials, Springer, p.64ff

WITT, W., KÖHLER, U., LIST, J. (2004), Direct Imaging of Very Fast Particles opens the Application of the Powerful (dry) Dispersion for Size and Shape Characterization, PARTEC 2004, Nuremberg, Germany