

CURRENT LIMITS OF PARTICLE SIZE AND SHAPE ANALYSIS WITH HIGH SPEED IMAGE ANALYSIS

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ABSTRACT

Since the introduction of high speed image analysis with powerful dry dispersion at the PARTEC 2004 [1], this technique has been extended into various fields of application. The unique combination of a pulsed light source with light pulses shorter than 1ns, telecentric illumination and imaging, a high speed mega-pixel camera with adjustable acquisition rates up to 500 frames per second, and a powerful database made the characterisation of particle size and shape possible at extreme particle numbers (e.g. 10^7) of even sticky materials in a well dispersed aerosol beam [2] – creating results of formerly unimaginable statistical relevance. It has been demonstrated that dry dispersion can also be effectively used for the dispersion of fibres. Subsequent skeletonisation, and topological analysis have been applied even to fibres of very complex topologies [3], evaluating the length, width and curl-index etc. of the fibres.

Now this technology has been expanded into the wet regime with particles dispersed in liquid, allowing for size ranges down to $1\mu\text{m}$, and into the process environment, with a family of instruments combining representative sampling, dry dispersion and image analysis for on-line characterization of particles at highest statistical relevance. The current limits of this technology are presented.

1 INTRODUCTION

For the characterisation of particles dynamic image analysis (DIA) is becoming the dominating technique whenever particle shape is an issue.

1.1 The New Concept

At the PARTEC 2004 a new concept of DIA has been presented [1], combining for the first time high speed image analysis with powerful dry dispersion in a table-top instrument.

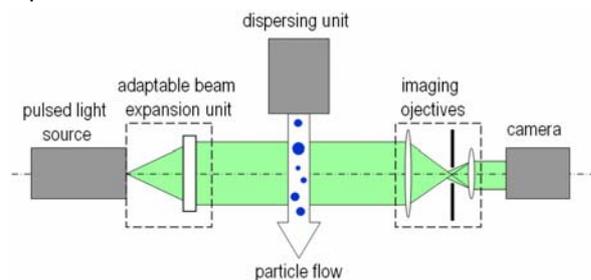


Figure 1: Principle set-up used for high speed dynamic image analysis in combination with dispersion.

As shown in figure 1 the device works in transmission with a parallel light beam. An innovative light source is generating stable visible light pulses with a pulse duration $\tau \approx 1 \text{ ns}$ and lowers any motion blur even at particle speeds of 100m/s far below one pixel size. The output power is sufficient for the correct exposure of images captures by the high speed CMOS camera. A fibre outlet of the light source acts as a point source in the adaptable beam expansion unit. The beam diameter is adapted to the magnification of the imaging objectives, so that always the same amount of light exposes the camera sensor. High optical contrast is

achieved as the imaging objective is equipped with an aperture stop, allowing only light rays propagating nearly parallel to the optical axis to reach the camera. The light source and the camera act synchronously. Frame rates adjustable up to 500 fps are possible at full resolution of 1024×1024 $10 \times 10 \mu\text{m}^2$ pixels per frame. The frames are background corrected, binarised and compressed in real-time ($< 2\text{ms}$ per frame) by a built-in signal processor unit of latest technology and transferred by a serial 1.25 Gbit link to a PC for subsequent evaluation and characterisation of the particles [2] by a variety of size and shape parameters and distribution types.



Figure 2: (a) DIA sensor QICPIC with dry disperser RODOS/L inserted in the measuring zone, (b) view of the outlet of the dispersing line and vacuum inlet in the measuring zone.

1.2 Improvements

Since its first presentation at ACHEMA 2003, the preceding technology has been continuously improved in many aspects, e.g.: High speed imaging, fast handling of large particle numbers per measurement ($> 10^7$), evaluation of very complex topologies such as fibres, introduction of wet dispersion, lowering of the

smallest particle size to about 1 μm , and expansion of the field of applications into the production, and/or pharmaceutical environment.

2 RESULTS

2.1 High Speed Imaging

The high frequency of up to 500 fps can be used to image slow particles more than once from different aspects, giving a more realistic 2D representation of the 3D shape of the particles.

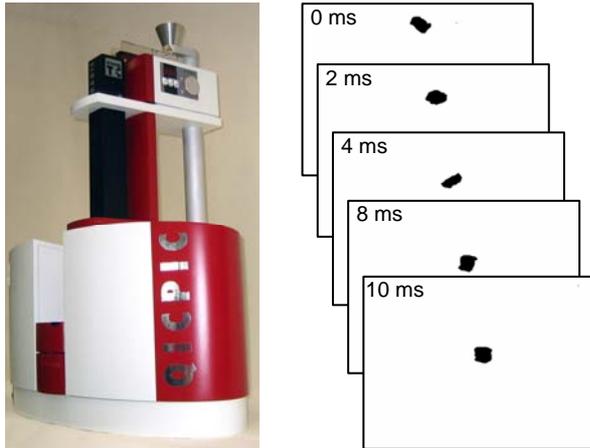


Figure 3: A gravity disperser is used to capture images of the same particle from different aspects. The 5 binary images are acquired in direct sequence at about 500 fps.

2.2 Large Particle Numbers

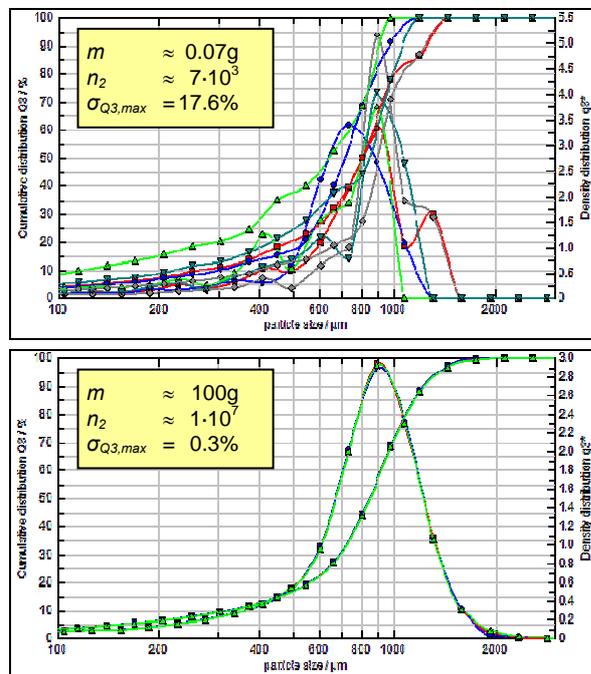


Figure 4: Size distributions of 6 subsequently measured coffee samples of different sample sizes m resulting in different particle numbers n and rel. maximum standard deviations σ .

It has been observed [2] that the dry dispersion even of sticky powders usually creates well dispersed and diluted images at high numbers of particles per image.

In combination with high frame rates extreme numbers of particles can be acquired in short measuring times.

For coffee powder sample sizes up to 100 g could be measured in less than 10 minutes. About 10^7 particles per measurement have been acquired resulting in a maximum rel. standard deviation of about 0.3 %. Using an Intel Core 2 Duo processor clocked at 2.8 GHz, the complete evaluation of an equivalent projected circle diameter distribution was performed in less than 5 minutes.

2.3 Dispersion and Evaluation of Fibres

One of the most challenging tasks is the characterisation of complex fibrous particles. Various trials with the dry disperser RODOS have demonstrated, that even very sticky fibre assemblies are well dispersed and captured by DIA. As an example figure 5 shows the primary wood fibres and the manual preparation of the fibres on to a feeder chute. The V-shape of the chute smoothes the flow.



Figure 5: Wood fibres are manually pre-dispersed in the V-chute of a vibratory feeder dosing the fibres into the dry disperser RODOS/L for DIA.

Most of the fibres are well dispersed as shown in figure 6. The strong dilution by the driving gas of the disperser widely avoids overlapping particles.

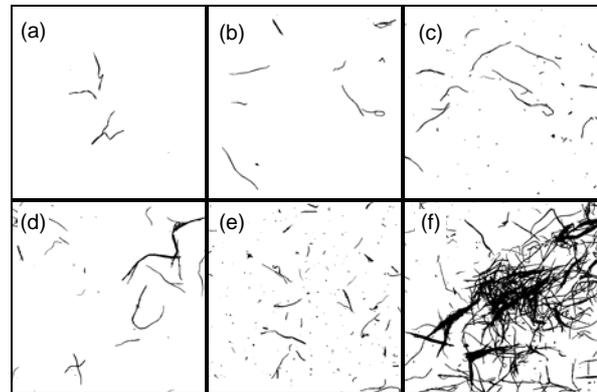


Figure 6: (a) to (c) show subsequent binary images of the dispersed fibres, (d) to (e) displays rare examples of too high concentration with overlapping fibres.

For each individual particle/fibre a complex evaluation algorithm with steps (1) to (4) was applied, as displayed in figure 7.

Subsequently, all edges were detected, transferred into a frequency polygon, their length was calculated and the skeleton was finally converted into its graph representation as (5) displayed in figure 8.

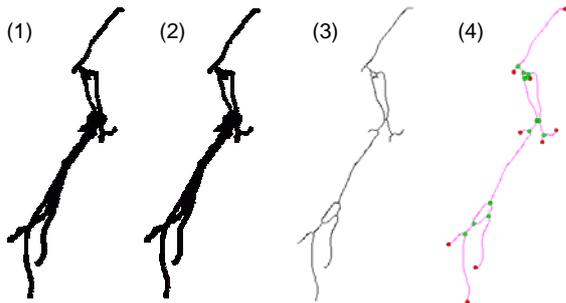


Figure 7: Evaluation steps of the complex fibre evaluation algorithm: (1) primary binary image, (2) closing tiny holes by dilation & erosion, (3) skeletonisation, (4) finding all vertexes

Using graph theory now the length of the fibre (shortest path between the most distance endpoints (6)), the width of the fibre (projected area divided by the sum of all segments), the curl index, straightness, complexity etc. can be calculated [3].

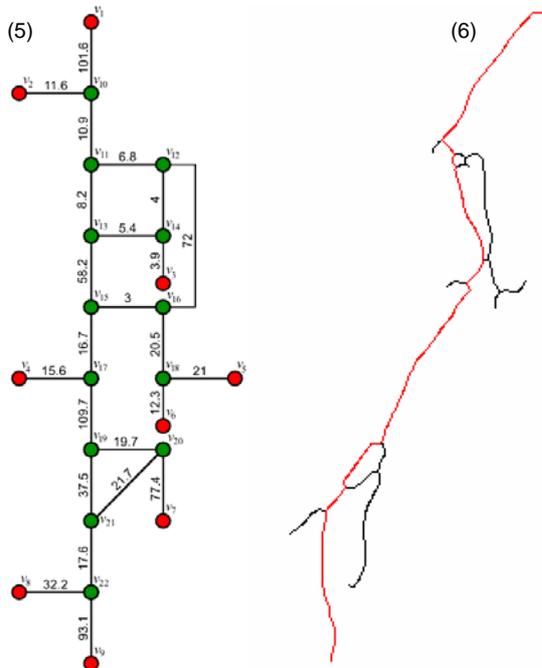


Figure 8: (5) graph representation of the skeleton of the fibre. The numbers represent distances in micrometers between the different vertexes. (6) the grey/red coloured path used for calculation of the length of the fibre.

2.4 Wet Dispersion

The special optical set-up with parallel illumination and an imaging objective with aperture stop creates high contrast images even for hardly visible highly transparent gel particles suspended in water. Figure 9 shows how the gel particles are pumped by a syringe through a flow cell applied to the measuring zone of the DIA sensor.

Examples of the resulting grey value images are shown in figure 10. Due to the refraction of the light by the gel spheres the particles are imaged nearly black, each with a light centre spot as light beams passing the centre of the spherical particles remain parallel to

the optical axis and thus pass the aperture stop of the imaging objective.



Figure 9: (a) highly transparent gel particles of about 1 mm diameter suspended in water, (b) pumped by a syringe through a flow cell with 2 mm optical path length.

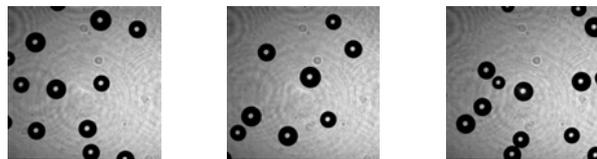


Figure 10: Resulting grey value images of the gel particles, showing that they are nearly spherical. A high contrast is obtained at all particle borders and a bright spot is visible in the centre. Feret diameters calculating the edges of the surrounding rectangle should be used for precise evaluation of the particle size.

Commercial versions of the flow cell set-up are shown in figures 11 and 12. As the depth of field becomes smaller with increasing magnification of the imaging objective, flow cells with different optical path length of 0.2, 0.5, 1, 2 and 4 mm are provided. A built-in autofocus algorithm is used to optimize the cuvette position prior the measurement. The path length of the cuvette used is automatically monitored by a radio frequency identification technology (RFID).

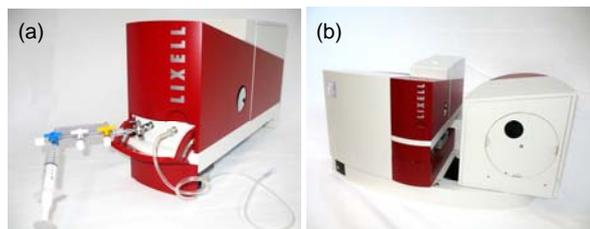


Figure 11: (a) commercial version of the flow cell LIXELL. (b) the DIA sensor QICPIC is opened for insertion of the LIXELL.



Figure 12: Commercial version of a closed loop cell (SUCCELL/L) on top of the dry dispersing unit RODOS/L, allowing the selection of dry or wet dispersion for DIA by software.

While the LIXELL allows for single shot set-ups and flexible adaptation to user requirements, the SUCCELL/L is designed for closed loop applications with basin,

pump, stirrers, ultrasonication, valves included.

2.5 Range Extension

While the upper size range is mainly limited by the maximum available beam diameter, the lower size range is strongly dependant on the depth of field, which becomes very small with increasing magnification. As with standard microscopy the position of the particles has to be precisely controlled, e.g. by the use of flow cuvettes with small path lengths.

Currently a size range of 2 μm to 20,480 μm (6,820 μm according to ISO 13322-2) is covered by 5 imaging objectives mounted on a carousel. An improved beam expansion unit acting as a zoom-objective adapts the diameter of the illuminating light beam to the selected imaging objective.

2.6 Process Environment

Finally, special embodiments of DIA in combination with dry dispersion have been designed for use in process and pharmaceutical (GMP) environments.

Basing on the comprehensive experiences with in-/on-line particle sizing by means of laser diffraction the instrument in figure 13 (a) combines the dry disperser and the DIA sensor components of QICPIC in a robust instrument. Samples can be fed by a vibratory feeder, either manually (off-line, as displayed in figure 13)), semi-automatic (at-line) or directly connected (on-line) to a preceding representative sampler (e.g. the TWISTER, which is currently available from 50 to 660 mm diameter of the process pipe).

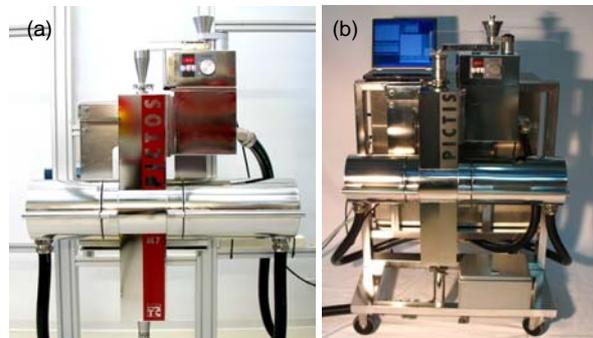


Figure 13: (a) on-/at-line DIA sensor PICTOS with built in dry disperser. (b) on/at-line DIA sensor PICTIS with built-in gravity disperser, mounted on a trolley for GMP applications. Both sensors are shown with a vibratory feeder.

For well flowing or fragile particles a version with the gentle gravity disperser of figure 13 (b) is provided.

The supporting housing on the rear contains the pulsed light source, all necessary controls and the image processing unit. As an option the embedded PC for the acquisition and control (Pentium M 2 GHz) is combined with a second embedded PC (Pentium Dual Core 2.8 GHz) for the concurrent evaluation of the previous result. This enables the stand-alone operation with measurement cycle times of less than 2 minutes for about to 10^6 particles per measurement. The particle size data are transferred by TCP/IP or WLAN and are presented on a PC in the control room.

2.7 Software

A software (WINDOX 5) was developed to specifically support the needs of real time storage and processing of high speed DIA. Basing on the powerful open source database firebird™ and encapsulated by a sophisticated application server, several clients can operate concurrently on different PCs in an extended network environment. The software supports all preceding Sympatec DIA instruments and includes features like fibre analysis in addition to particle size analysis by means of laser diffraction, photon-crosscorrelation, ultrasonic extinction, and sensors for laboratory and process applications. It is fully compliant with requirements of 21 CFR rule 11.

3 CONCLUSION

The presented high speed image analysis working in transmission with nanosecond exposure in combination with powerful dispersion has turned out to be a fruitful basis for the precise characterisation of the size and the shape of particles.

Since the first presentation at PARTEC 2004 a family of instruments have been developed, covering today laboratory and process applications in the dry and wet regime over a size range from about 1 to 20,000 μm .

The possibility to acquire, store and evaluate large particle numbers of $> 10^7$ in short times results in a formerly unknown quality and statistical relevance of the measured data. Today the maximum standard deviations $\sigma_{Q3\text{max}}$ for a series of measurements approach the precision formerly only known from non-counting methods, as e.g. laser diffraction.

This is combined with the advantage, that all information is traceable down to the individual particle. Galleries of particle images and distributions of various types can be created and displayed on various combinations of size and shape parameters.

Currently the main limitations are: (1) the lower particle size in combination with the restriction of the depth of field and (2) the limited width of the single size range. It will be possible to improve both by future sensor chips, light sources and the ongoing progress in the computing hard- and software.

REFERENCES

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