Possibilities of Dynamic Image Analysis in the Laboratory and Process Environment

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ABSTRACT

Since high-speed dynamic image analysis (HDIA) with powerful dry dispersion was introduced at ACHEMA in 2003. The technique has been extended into various fields of applications: (1) the addition of wet dispersion allows for characterisation of suspensions and size ranges down to 1 µm; (2) the introduction of a family of instruments with representative sampling, dry and wet dispersion for the process environment, including GMP versions and versions for hazardous areas; and (3) the development of software extensions for special shape parameter determination, sophisticated characterisation of fibres, and large samples quantities comprising more than 100,000,000 particles. These enhancements are presented and discussed.

Keywords: Dynamic image analysis, wet dispersion, process environment, characterisation of fibres

1 INTRODUCTION

The unique combination of a pulsed light source with light pulses shorter than 1 ns, telecentric illumination and imaging, a high speed mega-pixel camera with adjustable acquisition rates up to 500 frames per second, and a powerful database for storage of the image data, measuring conditions, and evaluated results enabled the characterisation of particle size and shape with formerly unimaginable statistical relevance, as shown by Witt, Köhler and List in 2004 and 2005. This technology has been continuously improved since its introduction.

2 WET DISPERSION

Wet dispersion has been introduced, allowing for the characterisation of suspensions. Today, two devices are available:

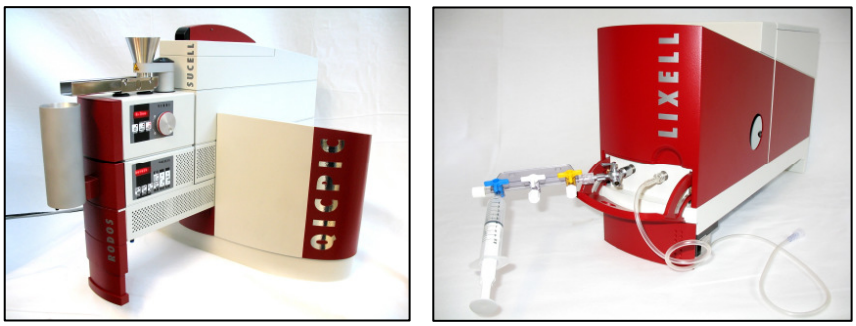

Figure 1: (left) OASIS, comprising the wet (SUCELL, top) and dry disperser (RODOS, bottom) inserted into the measuring zone of the HDIA system QICPIC; (right) wet disperser LIXELL with syringe and valves connected.

The **SUCELL** is designed for fully automatic operation and closed loop applications as a piggy back add-on for the dry disperser RODOS/L. Samples can be homogenised by stirrers in a basin with up to

500 ml of liquid, optionally dispersed by ultrasound with adjustable power from 0 to 60W, and circulated by a peristaltic pump. The change between dry and wet dispersion is possible manually by a single key stroke or under software control e.g. by a laboratory automation system selecting the appropriate treatment for the sample.

For the **LIXELL** a flow-cell set-up is used, obtaining a maximum flexible solution for complex user applications. The Luer™ connectors to the cuvette help to simplify experimental set-ups. As they are widely in use in medical applications, a large variety of hoses, valves, syringes etc. is commercially available for the adaption of user specific sampling and/or dispersion units.

Both systems are equipped with an auto-focus unit for the optimum clearness of the particle images. The gap-width of the flow cell is detected via RFID. A sophisticated focussing procedure automatically aligns the position of the cuvette under software control.

2.1 Extension of the Measuring Range

Wet dispersion enables size ranges down to 1 µm due to the well-defined position of the particles in relation to the optical depth-of-field. So for wet applications additional measuring ranges have been introduced: M5 (3.3 to 1,135 µm / 3,413 µm) and M4 (2 to 682 µm / 2,048 µm). M3 (1 to 341 µm / 1,024 µm) will be released shortly. The two values of the upper size limit represent ISO 13322-2 and the maximum detectable image dimension.

A new telecentric zoom-illumination unit varies the beam diameter from 1 to 38 mm and adapts it to the magnification. So the brightness of the images is constant for all measuring ranges simplifying the acquisition and optimising the signal to noise ratio of the images. The resolution of the optical system was checked (e.g. by a standard USAF target) showing that M4 can resolve structures below 2.7 µm, as shown in Figure 2.

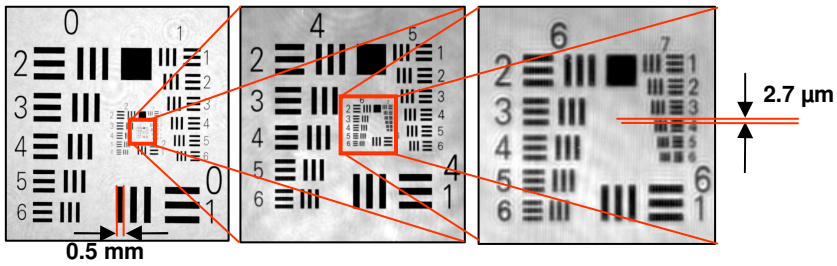

Figure 2: Check of the resolution via a standard USAF target. M4 clearly resolves bars of <2.7µm (right image, near 6).

2.2 Application with Highly Transparent Particles

The combination of telecentric illumination and telecentric imaging create images with high contrast at the particle edges even for nearly transparent gel particles in water as displayed in Figure 3.

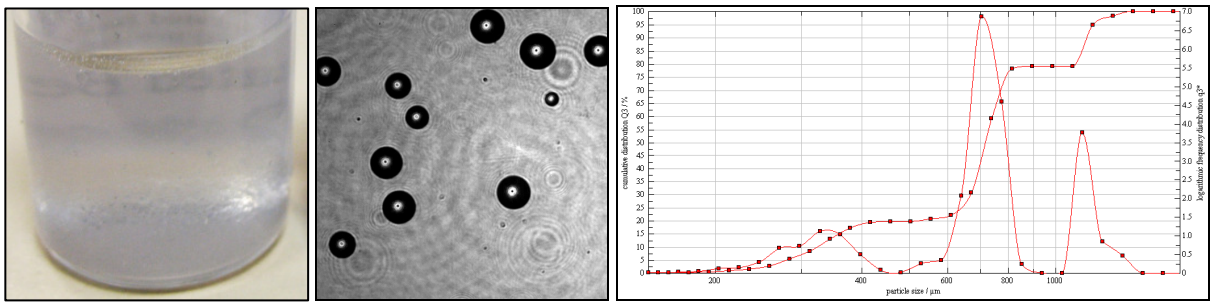

Figure 3: Highly transparent gel particles (left), their grey image (centre), and particle size distribution (right).

This optical set-up filters the refracted light and only the rays nearly parallel to the optical axis can

reach the camera (bright spot in the centre). Feret diameters calculating the edges of the surrounding rectangle should be used for precise evaluation of the particle size, avoiding errors from the bright spot when calculating the particle diameters for equivalent projected circles (EQPC) instead. Multimodal distributions are resolved, as seen in Figure 3 on the right hand side.

3 PROCESS ENVIRONMENT

The principle of the presented HDIA is not limited to laboratory use and can be adapted to the requirements of the particle characterisation in rough process environments. As with particle size analysis by laser diffraction representative sampling and proper dispersion is of decisive importance for results of high significance. A family of instruments combining sampling, dispersion and HDIA has been released.

5.1 Systems with Dry Dispersion

For dry applications two different types of instruments are available. PICTOS uses the well-established dry disperser RODOS and addresses applications with wide size distribution or sticky materials. It can be directly connected to samplers, e.g. to the representative samplers of the TWISTER family. These samplers have been originally introduced for particle size analysis by laser diffraction and currently cover process pipe diameters from 50 to 660 mm. PICTIS combines a gentle gravity disperser based on a fall shaft with impact plates and HDIA. For both units special embodiments are available for the use in pharmaceutical (GMP) and hazardous environments. Figure 4 shows one of the installations in our test house before delivery. Five representative TWISTER samplers with 150 mm and 100 mm process pipe diameters are connected to a single PICTOS sensor (left). The process lines are measured in cycles. Sampling, measurement, data acquisition and storage are performed by a Pentium M™ processor. The evaluation of the image data is performed concurrently by a quad-core processor. The results of all five process lines are displayed on a 32” high resolution flat panel, each with parameters, size distributions, trend graphs, and selected characteristic values (Figure 4, centre). Once every minute a line is scanned and the result of the former scan displayed. Due to the introduction of a new loss-less compression algorithm 1 TByte of disk space is sufficient for storing the image data and results of about 20,000 images per measurement for more than 30 weeks.

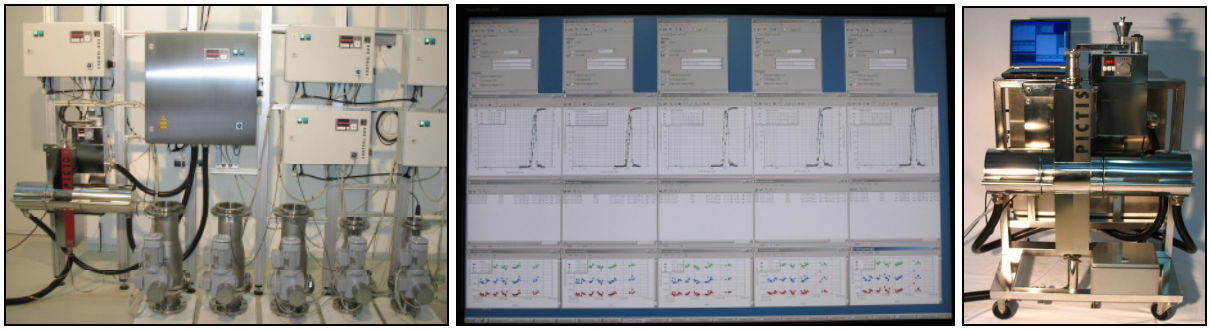

Figure 4: (left) PICTOS combined with five TWISTER, (centre) process control of five pipes in the polystyrene production, (right) PICTIS with built-in gravity disperser, mounted on a trolley for GMP applications.

Figure 4 (right) shows a PICTIS in a GMP application. The sample is supplied from an external sampler to the gravity disperser via hopper of the vibratory feeder on the top. The box on the rear contains the pulsed light source, the control, the pneumatics and an embedded dual-core processor for data acquisition, storage, evaluation and transmission of the results by WLAN to the laptop on top.

5.2 Systems with Wet Dispersion

The technology of the LIXELL, i.e. combining a flow cell of different width indentified by RFID and an auto-focus mechanism, has been recently transferred from the laboratory to the process environment.

Figure 5 shows drawings of the PICCELL. The system is chemically resistant against most liquids, e.g. acids, oils etc. Process pressures up to 10 bar are possible without further adaptations. The unit can by directly connected to a preceding sampling and dilution stage.

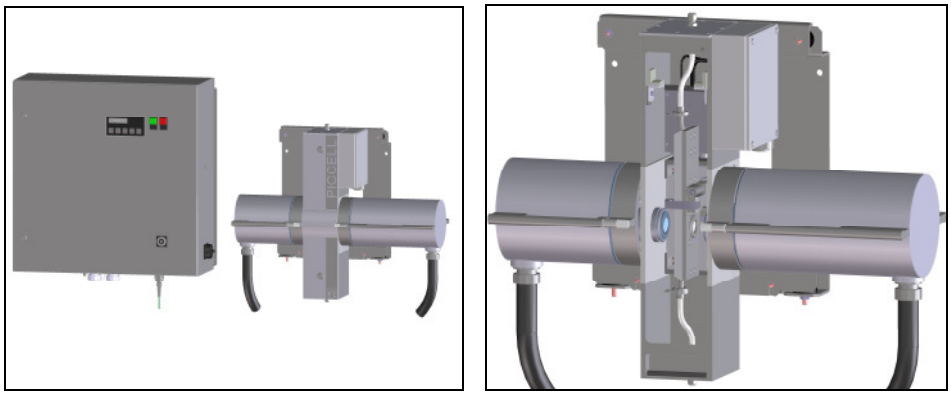

Figure 5: (left) PICCELL control box with pulsed light source, control unit and embedded PC, and HDIA sensor with integrated flow cell, (right) inside view of the measuring zone with flow cell and the connecting hoses.

4 OTHER IMPROVEMENTS

Also the software has been continuously improved. While most of the improvements dealt with the evaluation of specific size and shape parameters, such e.g. Feret diameters, sphericity, aspect ratio, convexity, elongation and straightness, and the graphical representation of these results, also the fundamental structure of the acquisition and evaluation process was optimized. Today particle numbers of more than 100.000.000 particles per measurement can be acquired, stored and evaluated. The acquisition time for a single measurement has been extended to more than 1.5 hours at a frame rate of about 450 images per second. As all particle images are stored, this allows e.g. for searching the proverbial “pin in a haystack”. The evaluation software fully supports multi-core processors with up to 16 cores, the calculation speed is currently increased to about 110.000 particles per second for calculating the EQPC on a 2.4 GHz quad-core Q6600 Intel processor.

5.1 Characterisation of Fibres

Fibres occur in many different processes like the production of cellulose, wood wool, minerals as well as textile fibres. The dry disperser RODOS can be used in combination with HDIA to disperse even very sticky assemblies of fibres, when they are prepared as displayed in Figure 6 (Witt 2005).

Figure 6: Original wood fibres (left), manually pre-dispersed fibres in the chute of a vibratory feeder VIBRI (right) feeding to the funnel of the dry disperser RODOS/L.

Most of the fibres are well dispersed as shown in Figure 7. The strong dilution by the driving gas of the disperser widely avoids overlapping particles. Skeletonisation and topological analysis have been successfully applied even to fibres of very complex topologies (Witt 2006).

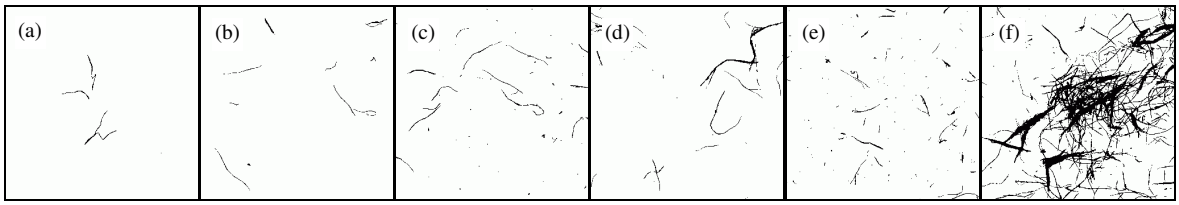

Figure 7: subsequent binary images of the dispersed fibres (a-c), rare examples of overlapping fibres (d-f)

For each individual particle/fibre evaluation steps (1) to (6) are performed, as displayed in Figure 8. By using graph theory the length and the diameter, the straightness, the elongation and the volume-based fibre diameter are calculated.

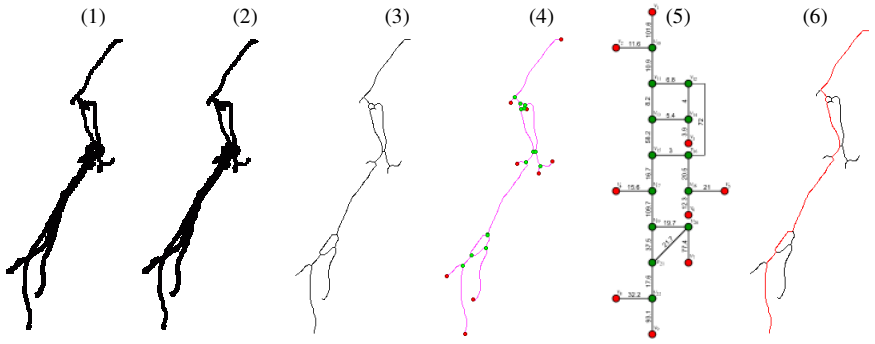

Figure 8: evaluation steps: primary image (1), closing holes (2), skeleton (3), vertices (4), graph (5), result (6)

5 CONCLUSION

Within five years after its introduction, HDIA has become a powerful tool of size and shape analysis for dry and wet applications, as well in laboratory as in process environments. Today HDIA covers a size range from 1 µm to 20 mm at high resolution. It allows for sample sizes of more than 10^8 particles per measurement, resulting in a statistical relevance of the results comparable to laser diffraction but with the possibility to back-trace to the image and properties of the individual particle.

The method is traceable to the standard metre.

6 REFERENCES

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