Advanced Particle Size and Shape Analysis for the Process Environment

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ABSTRACT

Size and shape of particles are sensible parameters for the quality of most dispersed matters. It is thus advantageous for the production process of these materials to monitor and control these parameters in real time. With the availability of fast measuring techniques such as e.g. laser diffraction (LD), a few instrument manufacturers have migrated particle sizing equipment originally designed for laboratory use only to the process environment. Since the early 80s a fast growing family of commercially available in-line and on-line instruments has been developed, covering a wide field of applications.

In this paper our recent developments of in-line and on-line sensors, dispersers, samplers and structural improvements are displayed.

Our LD sensors MYTOS, dynamic image analysis sensors (DIA) PICTOS and representative samplers TWISTER have been extended to cover extreme environmental conditions such as ATEX 95 Zone 20 or 1 inside and 21 or 1 outside, i.e. to operate under dust or even under gas explosion proof conditions. Hybrid systems for the combination of gas & dust explosion proof conditions with Zone 1 and 20 inside and 1 and 21 outside have been introduced recently. GMP versions for pharmaceutical applications are available as an option. The recent PICCELL sensor comprises a high pressure flow cell and extends DIA process applications to suspensions and emulsions in a wide size range.

A cost effective handling of multiple sampling points is supported by multiple samplers connected to one LD, DIA or ultrasonic extinction (UE) sensor. Typical cycle times are about one minute per sampling point. For faster response or wider spatially distributed sampling points the concurrent operation of multiple sensors is provided and simply setup: All sensors are using a standard TCP/IP LAN/WLAN connection to the computer. So only one computer is required and can be located anywhere in the network, preferably outside the hazardous area. The data of all concurrently operating sensors are stored in a common local or remote database. Multiple users can access the results in real time network-wide. Tables, graphs and/or trends can be adapted by the user to his specific requirement. New data are signalled and displayed instantaneously without any user interaction. Various methods are provided for co-operation with the central process control. As typical on-line DIA applications can easily generate about one TByte of data per week, a configurable data back-up is provided concurrently to the normal operation of the system.

Special applications of these advanced particle size and shape analysers of the process environment are shown and discussed.

Keywords: Particle Size Analysis, Particle Shape Analysis, Process Environment, GMP, ATEX

1 INTRODUCTION

Since the introduction of representative sampling in combination with dry dispersion for particle size analysis (PSA) basing on laser diffraction (LD) in the 80s, the technology of PSA in the process environment has been continuously improved. Meanwhile, robust sensors basing on various methods such as dynamic image analysis, (DIA) ultrasonic extinction (UE) etc. in addition to laser diffraction (LD) has been introduced. This strongly widens the possible fields of application.

2 EVOLUTION OF PSA FOR THE PROCESS ENVIRONMENT

2.1 Representative Sampling

Sampling and sample splitting contributes the largest errors to the results of the PSA. Proper sampling is thus of decisive importance. The proven concept of the ROPRON continuous representative sample splitter has been further developed by Witt (1998) to the TWISTER family of sample splitters. Common to all is representative sampling by scanning the cross section in a process pipe on a spiral line.

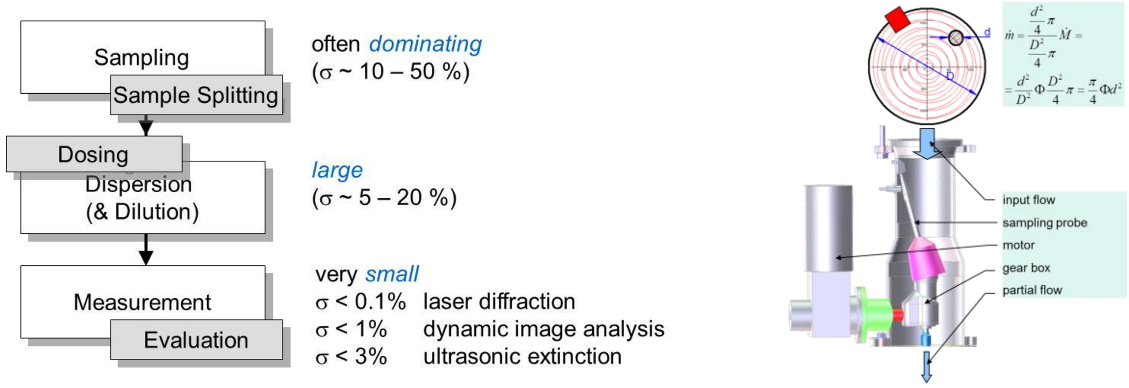

Fig. 1: Experienced contributions of errors to the measured particle size (left), principle set-up of the continuous representative sampler TWISTER (right), a finger probe scans the cross-section of the process pipe on a spiral line.

2.2 Laser Diffraction (LD)

The family of LD devices for the process environment has meanwhile developed to a large family, comprising currently true in-line devices for process pipe diameters from 100 to 250 mm and 50 to 660 mm diameter for on-line applications (Witt et. al. 2007). At-line installations for manual or automated feed of the samples are also available. The control is performed via a standard TCP-IP network.

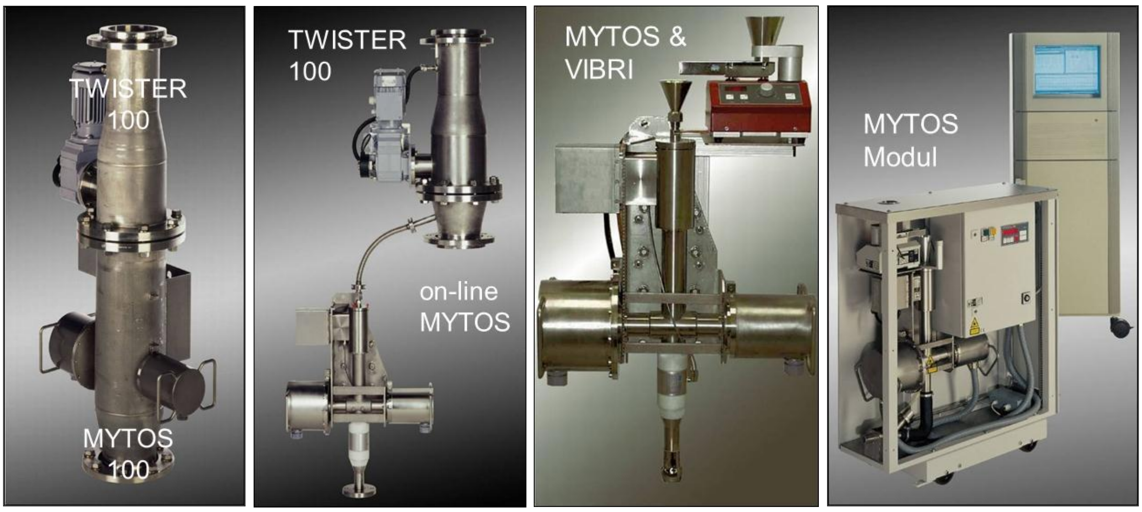

Fig. 2: Examples of the LD devices for the process environment: In-line (sampling and measurement inside the product flow), installation height saving on-line (sampling inside, measurement outside), at-line device for manual or automated feed of the dry disperser e.g. by a robot, LD module with built-in dry disperser and vibratory feeder, as used e.g. in cement plants (from left to right). All devices are using the renowned dry dispersing line of RODOS.

2.3 Dynamic Image Analysis (DIA)

The family of instruments basing on DIA has been extended recently (Witt et. al. 2008). PICTIS widens it to the dispersion of coarser, gravel-like or fragile dry applications using the built-in fall-shaft gravity disperser with baffles. PICCELL is equipped with a rigid 10 bar flow cuvette with selectable width of the optical path, and auto-focus, suitable for applications in suspensions and emulsions.

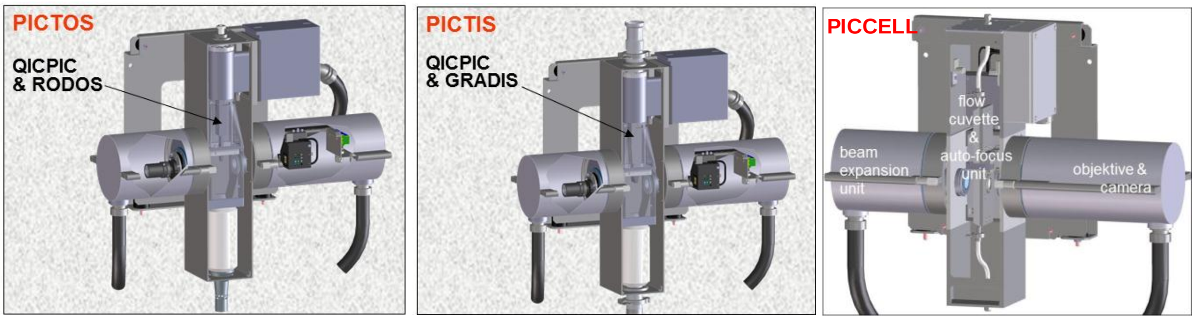

Fig. 3: Examples of DIA devices for the process environment: PICTOS is using the technology of the DIA sensor QICPIC and the dry dispersing line of RODOS, PICTIS is for coarser material or fragile particles with fall-shaft gravity-disperser GRADIS and PICCELL for applications with suspensions or emulsions with flow cuvette and auto-focus unit.

2.4 Ultrasonic Extinction (UE)

UE has shown its suitability in various in-line and on-line installations for PSA in concentrated suspensions under very harsh environmental conditions (Geers et. al. 2003). Today, a dynamic range of > 160 dB and a full frequency range of 100 kHz to 200 MHz widens the field of OPUS applications. The control is performed via the standard TCP-IP network protocol allowing for error-free, low-cost transmission even over expanded installations.

Fig. 4: UE probe OPUS in an in-line (left), in-line with by-pass adapter (centre) and off-line application. The devices cover applications with suspensions and emulsions in the range of 0.01 to 3000 mm, 0 to 70 %vol. without dilution, 0 to 40 bars, 0 to 120°C, pH 1 to 14, with GMP and ATEX versions as an option

3 GOOD MANUFACTURING PRACTICE (GMP)

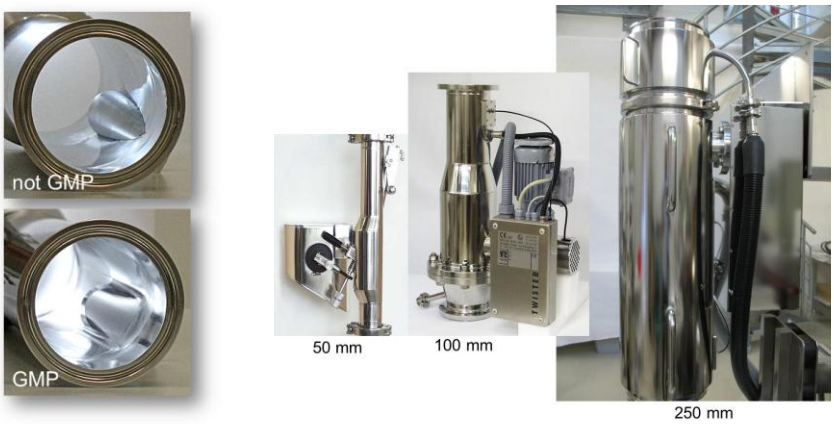

Fig. 5: Non- GMP and GMP inner surface structure of the process pipe of the representative sampler TWISTER 50 (left) with strongly reduced surface roughness and no dead stores (on the picture on the lower left), and examples of representative GMP samplers TWISTER for process pipe diameters with 50, 100 and 250 mm

GMP has been introduced by the US FDA in 1962 as part of the quality control process. The goal was that products are consistently produced and controlled to their requirements and their intended use, as defined by their product specifications. Since that date numerous rules, guidelines and literature has been released teaching what but not how it has to be done. So all guidelines have to be selected, interpreted and implemented by experts case-by-case, resulting in different requirements for every instrument and application, and challenging the manufacturer to find intelligent solutions serving as many applications as possible. For in- and on-line PSAs e.g. the steel quality has to be traceable, all non-metal parts require FDA certificates, cleanability (cleaning in place CIP, SIP) is important, the construction has to avoid dead storages and all relevant parts and all procedures have to be documented comprehensively.

Meanwhile nearly all members of the TWISTER sampler family, various probes, all our LD (MYTOS, MYTIS), and DIA (PICTOS, PICTIS, PICCELL) sensors are optionally available as GMP version.

4 ATEX

The use of bellows instead of moving gaskets makes the representative sampler TWISTER suitable even for hazardous areas by design. In the European Community the operation of devices in these environments is covered by the EU directives on explosion protection: ATEX 95 (94/9/EC) has to be observed by the manufacturer and ATEX 137 (1999/92/EC) by the operator. The operator is responsible for the definition of the appropriate zone, the manufacturer has to prove that the device satisfies the corresponding category, as shown in Fig. 6. The operation is only permitted, if the device is correctly approved and labelled with the specific ATEX label, as shown in this figure on the right.

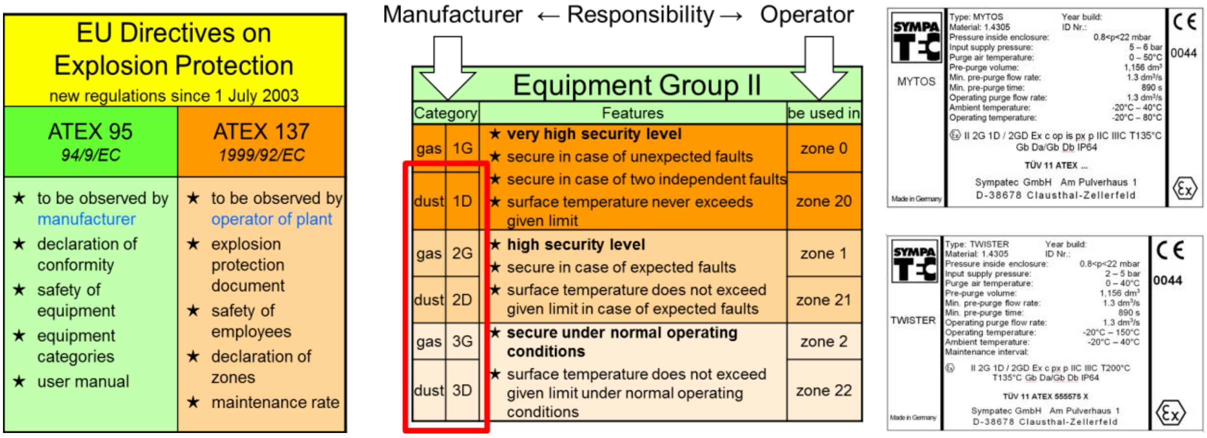

Fig. 6: EU directives for explosion direction for manufacturer and operator and the required documentation (left), zones and corresponding categories for the different security levels (centre), labels showing that MYTOS and TWISTER are accepted for category 2G1D/2GD and can be operated even in the very high security level zone 20 (dust) / zone 1 (gas)

We have successfully applied for approval for the complete MYTOS & TWISTER design for category 3D to 1D, so even the risks of gas (zone 1), dust (zone 20) and combinations of gas and dust are covered. For this purpose e.g. a risk of ignition analysis had to be performed and documented. The final documents comprise for the MYTOS and for the TWISTER design all necessary information. For the DIA design of PICTOS the approval has been recently initiated.

5 MULTIPLE SENSOR SUPPORT

In real applications it is meaningful to combine several samplers or a manual sample input stage with one PSA in order to reduce the costs per sampling point. Larger applications may comprise several of such combinations, sometimes even using different analysis methods for the different stages in the production line, e.g., LD or UE at the beginning of the production process and DIA for the final product. In the control room the possibility to combine results from various sources on a single screen is advantageous. For this purpose an adequate software and hardware structure is important. In our implementation, a powerful database is the core of the software of all our PSA systems. It is shielded and managed by an application server providing and storing all data for the clients.

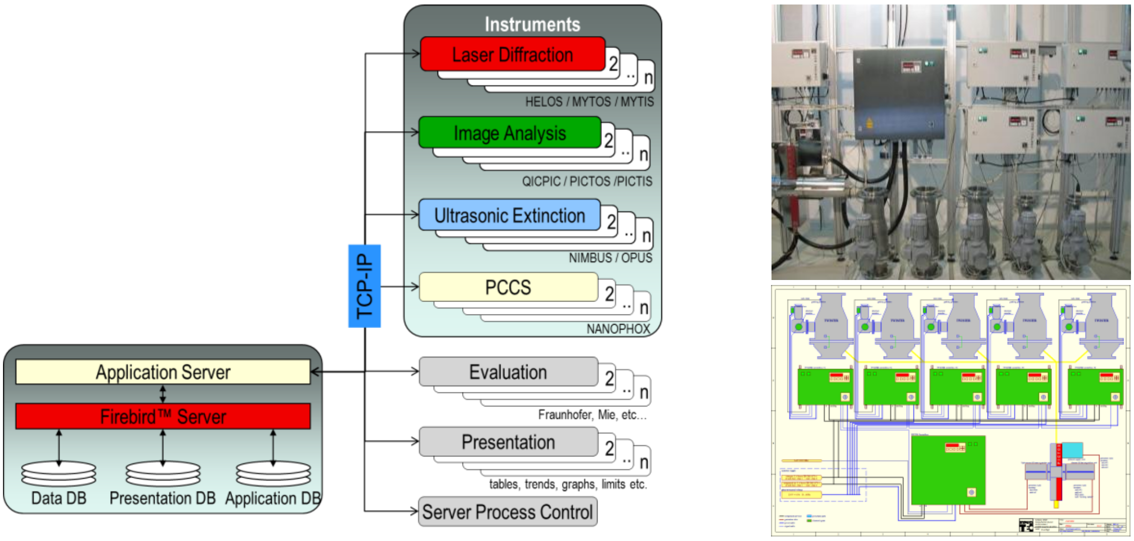

Fig. 7: Strict client-server structure of the WINDOX 5 software used for the operation and control of all Sympatec particle size and shape analysers (left), different types of multiple sensors each connected to (different types) of samplers can operate concurrently, using the same data base / application server on a single or multiple computers in a network. Example with 5 TWISTERs with their control boxes connected to one PICTIS sensor (right).

A strict client-server structure enables the combination of sensors, each having access to multiple samplers/dispersers. Evaluation and presentation modules are connected in the same way, all using the standardised TCP-IP protocol in a standalone (i.e. using a single computer) or distributed (on several computers) in a network configuration. Fig. 7 shows the example of 5 TWISTERs connected to a single PICTIS DIA sensor, with one PC performing the measurement, one PC (with Intel Core™2 quad processor) performing the evaluation of the images and running the database and separate PCs for presenting the results in various ways at different locations. Terabyte back-ups of excerpts of the database can be performed concurrently.

6 CONCLUSIONS

Since its introduction in the 80s in- and on-line particle size and shape analysis has been successfully extended into various fields of applications. Representative samplers covering a wide span of pipe diameters have been developed in addition to static or moving probes. Robust sensors basing on laser diffraction (LD), dynamic image analysis (DIA), ultrasonic extinction (UE) and other methods have become available. LD and DIA have been extended to the dry and wet regimes. GMP and hazardous environments with very demanding security requirements such as for gas (zone 1), dust (zone 20) or even for the combination of gas with dust explosion protection classes compliant with the latest ATEX regulations have been approved recently. A special software and hardware design with server-client structure and multiple-sensor/method and/or multiple-sampler support offer well-structured, highly modular, and thus simply extendable systems at reduced costs per sampling point.

7 REFERENCES

WITT, W., RÖTHELE, S., (1998), In-line Laser Diffraction with Innovative Sampling, PARTEC 1998, Nuremberg, Germany

WITT, W., PANKEWITZ, A., (2007), Particle Size and Shape Analysis in the (wet) Process Environment, Colloquium "Grinding and Dispersing with Stirred Media Mills", 2007, Braunschweig

WITT, W., LIST, J., KÖHLER, U., (2008), Possibilities of Dynamic Image Analysis in the Laboratory and Process Environment, PSA 2008, Stratford-upon-Avon, 2008

GEERS, H., WITT, W., (2003), Ultrasonic Extinction for in-line Measurement of Particle Size and Concentration of Suspensions and Emulsions, Particulate Systems Analysis 2003, Harrogate, UK