In-line Particle Sizing for Process Control in New Dimensions

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ABSTRACT

The combination of laser diffraction with upstream sampling built the break through for the in- and online particles size analysis in industrial applications. Today, especially the combination of representative sampling, dry dispersion, particle size analysis by laser diffraction and integrated feedback of the sample is well approved in many industrial applications. No more interactions of the user are required, and for standard applications the on-line monitoring of particle sizes became nearly as simple as the monitoring of any other parameter of the process. The increase of inspection intervals for a 24-hour operation into the region of months has increased the confidence users have in this technology, and industries with more demanding measuring conditions want to benefit from this performance. This challenge could not be solved with simple scale ups or downs. New solutions had to be found for the sampling system, the measuring sensor, the adaptation to the environmental conditions and the processing of the fast growing amount of data.

Keywords: In-line Laser Diffraction, Representative Sampling, on-line Monitoring

1 INTRODUCTION

An on-line system for particle size analysis (PSA) with the subsequent stages of representative sampling, effective dry dispersion (Leschonski 1984) and PSA by means of laser diffraction (LD) was already displayed about two decades ago (e.g. Heuer 1984). As many subsequent process stages are connected by pipes an innovative solution was offered by (Witt 1995 and 1998) which integrated the complete sampling and measuring system inside a pipe – representing a real in-line system. This solution consists out of two devices, the representative sampler "TWISTER" and the subsequent dry disperser and PSA "MYTOS".

1.1 TWISTER: The Representative Sampling Stage

As modern LD PSAs usually have a reproducibility in terms of standard deviations of less than 0.1% for well splitted samples, the errors result from sampling strongly dominate. So for reliable in-line particle size analysis representative sampling is of decisive importance. In a pipe the whole cross-section has to be scanned and all areas must contribute equally weighted. The mechanical solution offered by the TWISTER mechanics is shown in . The opening of the sampling tip is driven on a spiral line with varying velocity v. The velocity is controlled in a way that equal areas are scanned in equal times. The change of the aspect ratio of the opening due to the inclination is also taken into account. In the patented embodiment e.g. a system of connected levers is used, as displayed in Figure 1b. Combined with the rotation a nut is driven on a vertical thread varying the value of z and hence the value r via Equation (1).

$$r = \frac{L}{2H} \left[\left(\sqrt{l^2 - H^2} + z \right) + \frac{(H^2 - l^2)}{\left(\sqrt{l^2 - H^2} + z \right)} \right]$$
(1)

This system offers many advantages:

- The partial flow is kept inside the process pipe, so any pressure in the pipe does not affect the sampling.
- > Space saving 90° elbows are avoided. This reduces wear.
- > No moving gaskets and the use of bellows instead allows for the operation in hazardous areas.
- Small moving masses enables a small drive unit.
- All parts accessed by the product flow have wide mechanical tolerances and can be manufactured out of hardened material (e.g. ceramics).

- > The adaptation to different throughputs Φ can be made by variation of the diameter d of the inlet cap. If d is smaller than all subsequent stages, the cap acts as a 'security screening'.
- Simple adaptation to different diameters D of the process pipe by variation of the length of the sampling pipe.
- A parking position at the intersection point of the trajectories allows for a break without stopping in the product stream, which would result in an overestimation of that position. This also allows to interrupt the partial flow for a reference measurement or idle condition. A scraper at the parking position cleans the inlet.



Figure 1: a: trajectories of sampling orifice

b: lever action

c: sampling finger mechanics

Finally the particles have to be sucked into the sampling pipe. This is done quasi-isokinetic (Röthele, 1982), i.e. the sampling is performed at nearly constant velocity of the particles. The controllable flow pump of the subsequent dry disperser is used for this purpose.

1.1 1.2MYTOS: The Dry Dispersion and PSA Stage

The complete system is displayed in Figure 2. The well established dry disperser of RODOS is used for the dry dispersion stage of the MYTOS. Particle-to-particle, particle-to-wall collisions and centrifugal forces due to strong velocity gradients smoothly disperse particles down to 0.1 μ m. Although the lifetime of the dispersing line covers more than 100.000 measurements of 5g Cement PZ35F the benefit of no effect of wear during idle periods while sampling pipe is in parking position, lead to lifetimes of approximately 2 years with 6 measurements/hour. Additionally cleaning by an integrated flush-back stage safely avoids blocking.

The subsequent LD system is identical in all major parts to the standard off-line LD PSA (HELOS). A separate control box comprises power supply, laser, the sampler and disperser control unit and a fibre-optical communication link to PC. All components are encapsulated as IP65, hazardous areas are supported as option.

The measuring zone is equipped with a flow focussing injector with concentrates the aerosol beam along the centre line and keeps the windows free of particles. The mechanics of the measuring zone depends on the measuring range. For the three measuring ranges from 0.25 μ m to 875 μ m and the two ranges from 9 μ m to 3,500 μ m different optimised set-ups are used.

In the final merging stage the partial flow is re-combined with the process flow in direction of the centre line of the process pipe eliminating any possible wear at the process walls. The built-in valve allows for complete separation of the PSA from the process.



Figure 2: Cross section of dry disperser and LD PSA, Sympatec "MYTOS", with 200mm inner diameter

In combination with a cap in the parking position this creates a bypass inside the pipe and enables cleaning while the process is running. Bayonet fastenings of the light source and detector unit simplify the serviceability.

Basing of this concept a complete family of TWISTER and MYTOS units have been developed, currently covering process pipe diameters from 50 to 200 mm and particles size ranges from 0.25 μ m to 1,750 μ m. Their performance has been proven in various applications.

2 SCALE-UPS AND SCALE-DOWNS

For process pipe diameters larger than 200 mm the sampling tube cannot be elongated offhand. The enhanced forces had to be taken into account. Completely new concepts for mechanics, sensor technologies, control, electronics and operating sequence had to be developed and were accomplished. Currently pipe diameters up to 860 mm are supported.

Scale-downs to pipe diameters below 100mm require that the drive unit of the TWISTER and the MYTOS has to be moved outside the pipe. For 50 mm diameter a new sampler has been developed, that still performs the scan on a spiral line and incorporates a parking position. A quick release mechanism for all components follows the cleaning request in pharmaceutical applications. Te device is best suited for the control of small classifier-mill combinations.

Even more demanding is the extension to lager particles. The output of a laser has usually a Gaussian intensity distribution I(r) with $2w_0$ specifying the full width at $I/I_0 = 1/e^2$. As the diffraction pattern on the detector is the Fourier transform of the spatial distribution of the particles and the illuminating beam, the centre of the detector is also illuminated by a Gaussian intensity distribution. The width w is

transformed by Equation (2) which defines diameter of the focal point. This means that the beam itself reacts as a large particle which is transformed.

$$w = \frac{\lambda \cdot f}{\pi \cdot w_0} \tag{2}$$

For the analysis of large particles it is important, that $w_0 >> x_{max}$ because large focal lengths *f* have to be used for the measurement and significant intensity has to be detected very near to the centre of the detector, i.e. to the bright focal point.



Fig. 3: The spot diameter w as function of the required beam diameter 2 w_0

Ideally the beam diameters has to be adapted to the measuring range. Up to $f = 500 \text{ mm} (x_{max} < 875 \mu\text{m})$ it is possible to use a relatively small beam diameters of 3 mm to 10 mm and a multi-chamber system is sufficient to avoid contaminations of the windows. From $f = 1000 \text{ mm} (x_{max} = 1750 \mu\text{m})$ on the beam must have a minimum diameter of 30 mm to keep the focal point sufficiently small. Here a multi-chamber system would be in the size of the tube diameter. So a new concept had to be developed: a special flow chamber with the advantage of less gas consumption and small design. In addition a new Fourier Optics was developed, in order to improve the mechanical stability. Now an optical system with $f = 2000 \text{ mm} (x_{max} = 3,500 \mu\text{m})$ fits in a cylinder with a length of only 300 mm. Figure 3 shows the design. The unit has to be operated vertically.



Figure 3: Dry disperser and PSA with new flow concept for focal length of 2000 mm (x_{max} = 3500 μm), Sympatec "MYTOS".

Finally the software has to be scaled-up as well. Cycle times down to about 1 minute and the combination of several PSA in a single process create an enormous amount of data – up to about 500.000 measurements per sensor and year. So the concept of the controlling software had to be expanded. A new powerful database (INTERBASE[™]), the implementation of a well defined client/

server structure and an integrated security systems compliant with CFR 21 Rule 11, allows for the unlimited parallel operation of several PSA units and proven to handle more than 1,000,000 data sets.

3 APPLICATIONS

Out of a variety of already established applications, the following two examples are specified in detail: metal spraying and coal milling.

Metal spraying of aluminium		Milling of raw coal	
particles up to	400 µm	particles up to	x ₈₀ = 3000 μm
product temperature	100 °C	product temperature	40 °C
mass flow rate	1000 kg/h	mass flow rate	400 t/h
air flow rate	60,000 m³/h	distance MYTOS – PC	200 m
air velocity	40 m/s		
tube diameter	DN 660 mm		TR
pressure	0.1 bar		W AW
set up	upside down		all the war
TWISTER sampling time	20 s		
distance MYTOS – PC	200 m		
(Ex)-class zone 10 inside	zone 11 outside		
antistatic design			

Table 1: Specifications of process parameters for two selected applications, installation of MYTOS (x_{max} = 3500 µm)

While the challenge of the first example is the very low concentration, the very high air velocity and the high temperature with temperature change, the challenge of the second is the large maximum particle size, the sticky material and the high throughput, which required a pre-sampling unit.

4 CONCLUSION

The combination of representative sampling, dry dispersion, particle size analysis by laser diffraction has created a powerful family of devices, suitable for the in-line and on-line particles size analysis of dry powders and suspensions. Several scale-ups and scale-downs have been performed and were possible within this technology, widening the field of applications. Currently size ranges from 0.25 μ m to 3,500 μ m and pipe diameters from 50 mm to 860 mm are supported. Applications with high temperature, high particle velocities, low concentrations, abrasive or sticky particles in standard or hazardous areas (also following the new ATEX 20/200 standard) have been successfully implemented. The new software allows for the parallel operation of many PSAs on the same database. It can handle very large amount of data, as acquired over years, and is fully compliant with CFR 21 Rule 11.

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