In-line Laser Diffraction

Proven Particle Size Analysis in Industrial Environment

M. Puckhaber, S. Röthele and W. Witt

Reprint from

powder handling & processing

Volume 10, Number 4
October/December 1998

Vol. 10 · No. 4 · October/December 1998 powder handling & processing

In-line Laser Diffraction

Proven Particle Size Analysis in Industrial Environment

1. Introduction

The importance of information on particle size has grown extraordinarily during the last decades. In parallel the instrumentation was developed, and therefore formerly existing limitations are not valid anymore. This not only applies for the availability of measurement data but also for the handling of measuring equipment and for controlling and monitoring of processes.

Laser Diffraction (LD) has become the standard technique for particle size analysis (PSA) within the last thirty years because of its speed, reliability, outstanding reproducibility and applicable size range.

It is vision from long ago that an automatic measuring system should be available, which can be integrated into the process-line and allows direct reaction on the measured data. A decade ago, this dream became true for some special applications in the field of particle technology. A sampling device in a process pipeline takes a sample and somehow transports it out of the process area to the measuring facility. A subsequent automatic treatment (e.g. a robot) is used and then a standard instrument measures it. Finally the sample is either returned to the process or it is disposed off. No standard has been established for further processing of the measured data, but individual solutions have been found to automatically control the process.

For large quantities of particulate matters limitations arise because the capacity of the automated sample splitting is limited. Processes with pneumatic transport of the product request adapted sampling procedures. A particle free reference measurement, which is required for precise measurements, must be provided. Restrictions come from toxic and hazardous materials as well as from specific requests in pharmaceutical and food industries.

This situation has now remarkably improved. First, of course, even the off-line instrumentation has been automated. New sample preparation, dosing and dispersing systems have been developed and the handling of the system has become much more comfortable. Especially in particle size technology, either new ways have been found to solve some of the mentioned problems or developments even prohibited the problems to occur.

This article explains a new approach of real in-line analysis in general and examines a special application at a chemical plant. The production of a polymer additive has been monitored for several months and still is. Some results are presented and the performance of the measurement assembly is shown.

This presentation was contributed by M. Puckhaber, S. Röthele and W. Witt of Sympatec GmbH, Burgstädter Str. 6, D-38678 Clausthal-Zellerfeld, Germany.
Tel.: ++49 53 23 717 0; Fax: +49 53 23 717 229
e-mail: info@sympatec.com

Challenges of PSA in a Process Environment

Particle size information is a measure for the quality of processes. Vice versa, particle size information can be used to control the process; i.e. conclusions are drawn fast and aim at immediate reaction to keep the process in certain pre-defined limits. If there is a tendency into sensitive values, appropriate measures against leaving the acceptable tolerances can be taken. In fully automated systems these steps are performed with a programmable logical control (PLC), no operator action is necessary.

Several severe challenges have to be faced in common production environments:

1. A sample has to be taken from a pipeline with a throughput that is much higher than the analytical device can deal with.
2. The sample has to be taken representatively to gain information about the process and not just catching a number. A continuous sampling is preferred against a "snapshot".
3. The sample has to be treated in a proper way. It has to be dispersed, but a modification of the sample, however, must be avoided.
4. The analysis must lead to results that can be checked and approved.
5. The material to be analysed can be extremely valuable. Hence keeping it in the process line should be the most favourable solution.
6. If the tube is not pressureless and/or higher temperatures occur the short fall of dew-point must be avoided.
7. The analysis should be fast and the results should be presented in a way that consequences can be taken immediately. An automated closed loop reaction might be possible, as well.

It is obvious that a sampling device is advantageous. Hence this detail is not surprising at the presented solution. But all the other requests are not fulfilled naturally.

In our example we look at a chemical plant producing polymer additives. At the end of the production line the polymer additives are milled. To monitor the final state of the product the PSA system is installed behind the mill. Nevertheless, conclusions can also be drawn about the production process and the product. Hence the gained information can be used to control the milling as well as the production process.

TWISTER's Sampling Principle

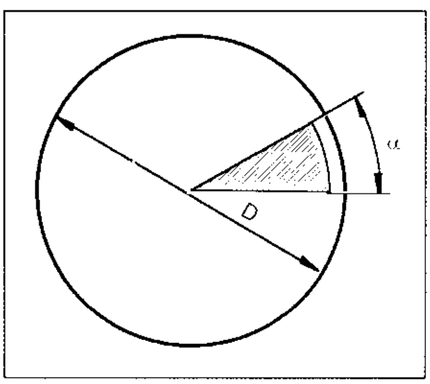
A simple probe or even just an outlet is often used to take a sample out of a larger amount of material. This is only representative if the medium carrying the particles is of high viscosity and the material is moving turbulently. Otherwise a segregation of different fractions of particles is perceived and the results of the analysis are quite arbitrarily.

So the challenge is to find a representative sampling device for in-line application under process conditions.

At the plant mentioned a dry production facility with an air jet mill is used. The connected pipeline is pressurised and the product is transported with the airflow. Particle measurement takes place in a position where the particles move vertically downwards.

First idea could be to take out the sample from the pipe with a constantly rotating sector. Thus a representative amount of sample out of the main stream, which has fallen into the sector element, is continuously available for analysis or further treatment.

Fig. 1: Sampling with a rotating circle segment



Experiences with this principle have been gained with the ROPRON sample coupler, a former development of Sympatec. The angel of the sector element α determines the share of the throughput of the pipeline that is separated. In many cases, however, the amount of sample is too big, so additional sample splitting steps are necessary.

Only scanning of the whole cross section of the pipe secures representative sampling. Hence the rotating sector element can be replaced by a small moving hole.

The new idea is to drive a small opening over the complete cross section of the pipe. If the sampling is isokinetic i.e. the speed of volume entering the opening is identical to the transportation speed inside the main pipeline, after completion of the whole cross section a representative sample has been taken.

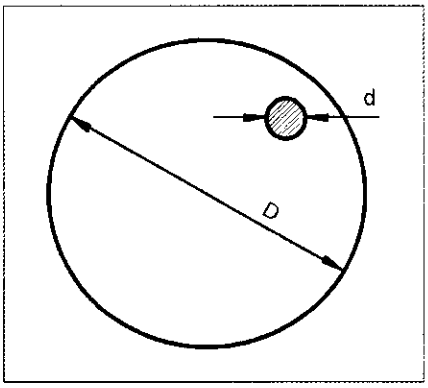


Fig. 2: Sampling with an in-line tube

As a side effect a necessary adaptation to varying amounts of sample in the partial stream can simply be realised by changing the diameter of the inlet of the sampling pipe.

As consequence of the sampling pipe scanning the cross section other questions arise:

1. Which is the best way to move the sampling pipe?
2. Along which way should this be done?
3. How to mount the sampling pipe in the process pipeline?

With the design of TWISTER a spiral movement of the sampling pipe was realised starting at the wall of the main pipeline.

The solution that was finally realised is passsing the centre of the process pipeline and ends at the wall of the pipe. The final point is a shield that covers the inlet and prevents it from continuing collecting particles when in the parking position and so offers the possibility of a particle free reference measurement.

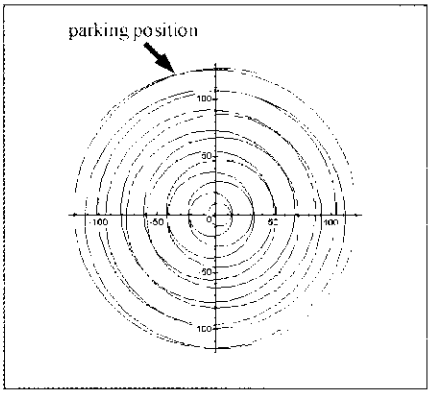


Fig. 3: Proper scanning with spiral way

A natural consequence of the spiral movement is of course that the sampling pipe is installed in the centre of the process pipe. This also reduces wear of the sampling device.

As the origin of the sampling pipe is fixed, it is directed from this origin and the projection area changes. The larger the angle is the smaller is the effective inlet area.

A compensation of this effect is achieved by adaptation of the speed of movement. The larger the angle is, the smaller is the sample inlet area and the slower the movement has to be.

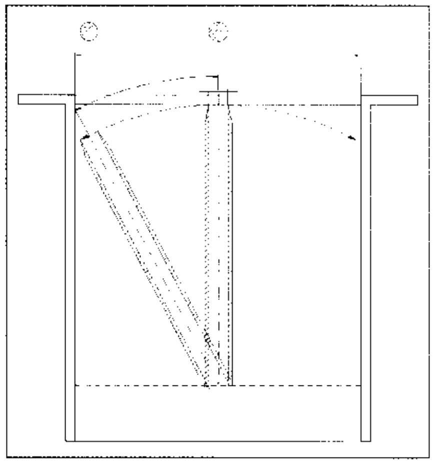


Fig. 4: Sample inlet projection

These measures together with isokinetic sample inlet ensure real representative sampling with the TWISTER.

TWISTER Design

For installation of TWISTER in the centre of a process pipeline only a single flange is necessary. The moving masses are very small. Therefore the necessary power is small, too. The drive unit is located outside the process pipeline at the mounting flange.

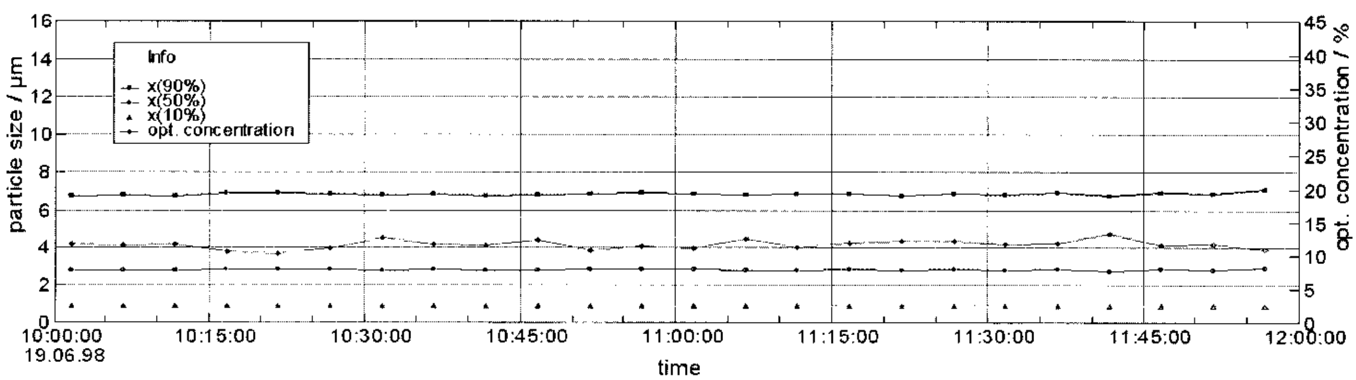
The only moving part, the sampling pipe, is protected without gaskets but with bellows, this allowing for operation in hazardous areas. A shield that reduces wear to a minimum covers the outer bellow.

A shielded parking position allows particle free reference measurements and further reduces the wear within the measuring system, as there are only particles in it while sampling.

Furthermore this simple and straightforward construction allows some modifications according to different requirements:

1. It is possible to adapt TWISTER to different diameters of pipes by simply shortening or elongating the length of the sampling pipe.

Fig. 5: Detection of a stable process shown in selected results of particle size measurement



2. Adaptation to different throughput is achieved by modifying the size of the sample inlet.
3. For abrasive or reactive materials the few and simple parts in contact with the product can be manufactured of virtually any material, e.g. hardened steel or ceramics.

The positioning of the sampling pipe in the middle of the process pipeline brought up the idea of having the analyser at the same location. Although an outlet can also be realised, the installation of the complete system in-line has advantages.

First of all, the pressure in the process pipeline does not have to be compensated. The dispersion medium is the only flow that enters and leaves the process - besides the demanded measuring results.

A second advantage is the avoidance of detours in the sample flow. In fact, the sample is moving almost straight forward inside the measuring system. Hence sedimentation is impossible and wear usually occurring at large angle elbows is excluded.

Assembly of MYTOS

MYTOS is a combination of a dry dispersing device with a laser diffraction sensor following on the TWISTER in the in-line system.

It is mainly build from parts and elements, which are identical to those used in off-line particle size analysers. The dispersing unit is well-established in the RODOS dry dispersing system and the optics is of the same type as in the standard HELOS laser diffraction spectrometer. These parts have proven to be very performing and of unrivaled long time stability and, with small adaptations, fit perfectly for the new task.

The RODOS injector is long and thin and can be installed parallel to the centreline of the pipe. Fortunately, the dispersing injector supplies the vacuum needed for the isokinetic sampling anyway. The control of the vacuum is coupled to the pressure inside the process pipeline. The quality of the dry dispersing system RODOS with excellent dispersion and reproducibility down to submicron range and a high lifetime is proven.

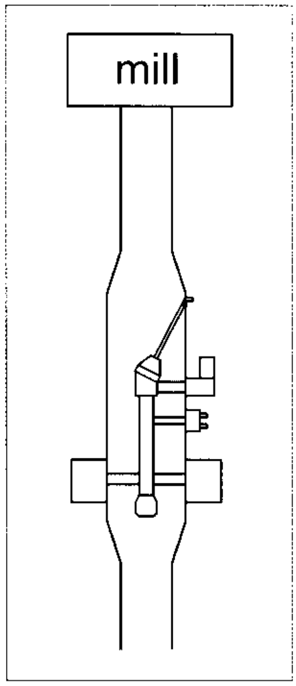


Fig. 6: MYTOS & TWISTER set up downstream the mill

Of course, in a suitable installation there is no room for the HELOS cabinet. As the sample does not go to the optics, the necessary beam expansion unit and the Fourier optics got new housings and go to the sample into the process pipeline.

The dispersed aerosol enters the measuring zone with a sheath flow used for focussing the aerosol and avoiding the optical system to get in contact with particles.

As light source a Helium-Neon laser is used. The beam expansion unit connected to it via fibre optic light transmission and the detector are mounted to the process pipe with bayonet fastenings to simplify serviceability. Identical elements to off-line HELOS systems are applied.

A demonstration installation similar to the one in the chemical plant is shown on the photo (Fig. 8). The transparent pipe is chosen to enable the view on the assembly. Only one detail is different in comparison to the installed TWISTER & MYTOS: Behind the measuring zone a pneumatic valve is installed, that allows the complete separation of the inside of the measuring system from the product flow.

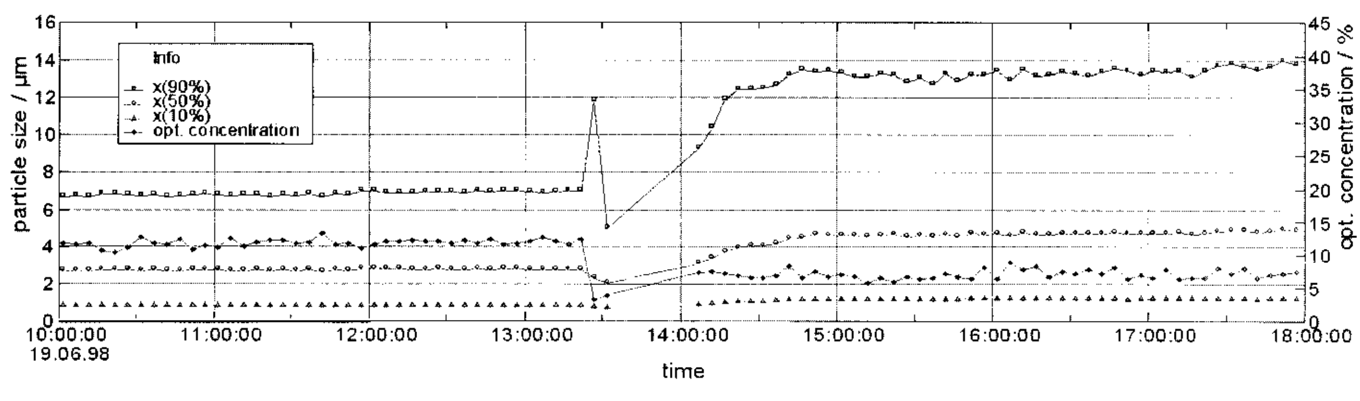
As the beam expansion unit and the detector are mounted inside of pipes within outer pipes, with the sampling pipe in parking position and after closing the valve it is a simple operation to take them out and clean them, or the protection windows, respectively. In the same way the complete service of the unit is possible without any side effect on the production process. In fact, the installation can be operated as a bypass - inside the process pipe.

The shield that covers the sample inlet in the parking position can include an inlet for air or whatever transport medium is used in the process. Therefore, also the possibility of cleaning, rinsing and particle free reference measurements is provided.

Experiments have shown that results obtained with the new in-line system are almost identical to off-line measurements. Considering the origin of the main parts this is no surprise. On the other hand, it shows that the system achieves the same high and unrivaled quality of measurement results as the Sympatec HELOS off-line system. And, most important of all, this fact shows that the sampling is really representative.

The apparatus is not sensitive to optical concentration. The range is from about 0.1% to about 50%, and, depending on the material, even higher. Experiments on this show that the results are very stable regardless to the sample amount and the optical concentrations exceeding a certain minimum.

Fig. 7: Detection of product change



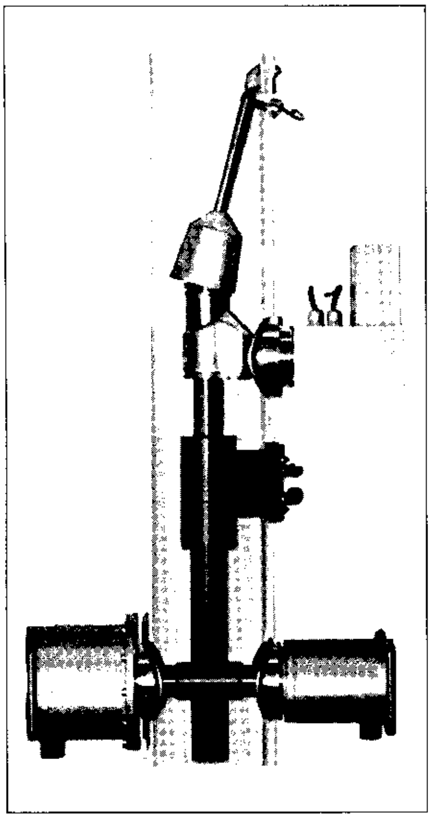


Fig. 8: MYTOS & TWISTER installed in a model pipe

The operational software WINDOX is well approved and extremely easy to use. It includes a variety of analysing tools like statistics, a trend analysis feature and graphical representation possibilities. Although the analysis of the data is remarkably powerful, the handling for the operator is very simple.

WINDOX is running on a standard personal computer under Windows™ 95/98/NT 4.0 user interface. The superior database features allow remote control via network or serial interface as well as measuring and exploring results at different locations with instant update of graphics and reports network-wide.

The user can define content and design of the desktop individually according to the requirements of the application. This includes graphs, trends, tables, and limit values.

If there is only a set of products, the operator just selects the actual product out of a list. Otherwise the name of the product is entered. All the other parameters like pressure needed for isokinetic sampling or measuring time settings are either fixed once or selected automatically.

In the presented example only the production process is changing. For the PSA this means, only the product name and the process conditions (e.g. pressure) are differing.

Trend analysis, for instance, shows user-selected values over the time. This means, every value of interest can be monitored, the share of product exceeding a certain size as well as the pressure inside the pipe.

Industrial Installation

The here reported industrial installation took place at a chemical plant producing polymer additives. The batches are changed every few days, so - besides the standard quality monitoring - any time the product changes, the process has to be adjusted.

One decisive criterion is the particle size. Therefore the in-line PSA is a perfect match for controlling the quality of the process. Different approaches had been checked. The solution presented here was chosen because it matched the necessity of sampling and the process conditions perfectly.

After the actual production step the product is milled in an air jet mill. The pressure of the milling process is used to transport the product within a DN150 pipeline (150 mm ID). The in-line system has been installed directly downstream the mill.

The flow is rather turbulent, but it is very typical for dry particle size measurement in-line that results strongly depend on the position of the sampling inlet inside the process pipeline anyway. Therefore, scanning of the whole cross section of the pipe is absolutely necessary to get information about the real particle size distribution.

Although particles up to 875 µm can be measured, the installed measuring range is R3, i.e. up to 175 µm. Generally, changing of the measuring range is possible as only an optical module has to be replaced.

As the amount of each product batch is relatively large and the products are quite special and therefore also quite expensive, the in-line control with the very short response time is of high economic value. Although it is very difficult to give detailed figures about the money saved, the installation is amortised within a few months.

Experience has shown that cleaning of the protection windows in the MYTOS was not necessary within three months. The flow focussing device obviously does a great job to prevent the optics to get in contact with particles.

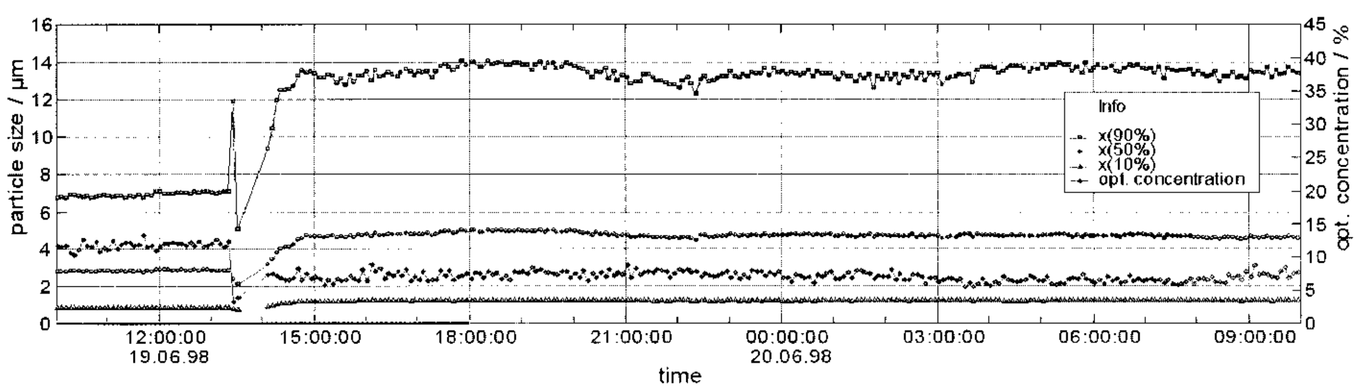
As an option the system is made of stainless steel and the whole equipment is manufactured according to GMP standards.

The measuring results are collected by a control-box at the production site. The process control is located in a room not to close to the mill.

The control personnel evaluate the data on a computer connected to the control box by a fibre optical link. If more than 500 m, fibre optical repeaters can extend the distance to several kilometres.

An operator reacts on changes in the results. The information, however, necessary for automatic control in terms of digital and analogue output is provided with the help of PLC connected to the PC. Thus fully automatic control is prepared and can be established with little effort. The idea of closing the gap is now becoming reality.

Fig. 9: A full day process monitoring



Measurement Results

The system measures the particle size distribution every five minutes. Exceptions within the first weeks were some periods when the process was interrupted. The results show the following:

1. The measurement is very reliable. Due to experience when the process runs stable the results reflect this perfectly. E.g. over a period of two hours there is almost no change in the values (Fig. 5).
2. The measurement is sensitive and the results perfectly correspond to the real situation: When changing the product the optical concentration decreases rapidly. The mill runs empty and the particle size oscillates because of remains in the process. Some random results appear and then the next production process is quickly adjusted using the results of the PSA. The oscillation at the beginning indicates the ringing of the pressure regulator of the mill (proved by manual variation of the pressure). After starting with the new product the data stabilises again (Fig. 7).
3. Although the optical concentration changes between different products, the results are very reasonable. A lower optical concentration is due to a coarser product. The measuring principle is performing well over a wide range, shown in extended off-line experiments.
4. Operation of the system is as easy as can be. The usual operation is without any user interference. Only when changing the product the MYTOS & TWISTER is stopped by pushing a button and the new product name is entered.
5. As the operational software is Windows™ based even inexperienced computer users handle the measurement control and evaluation easily and intuitively. Additionally the system can be controlled remotely also via serial interface or network which enables remote control over long distances.
6. Particle size information in formerly unknown quantity and quality is provided. A resolution of one measurement per five minutes gives a good idea of the process, especially if only sensitive values are under supervision. Higher resolution of one measurement per minute is chosen during the adjustment phase of a new product. Even small changes in the production process are realised immediately. The operator can react without delay.
7. Data of long term trial, i.e. more than 5000 measurements can be handled easily. They require less than thirty megabytes on a computer harddisk. Today's harddisks of several Gigabytes allow for the storage of data of several years.
8. The database allows detailed supervision of the monitored process. A more detailed analysis concerning other values is always possible thanks to the database support of the operating software. Especially the possibilities of investigating or following trends in arbitrary values are powerful features of the employed WINDOX software. Diagrams of any two parameters e.g. x_{50} vs. optical concentration allow for sophisticated analyses of the process behaviour.
9. Quality management is improved remarkably not only as far as particle size is concerned. Because the particle size distribution is a decisive criterion for the quality of the process

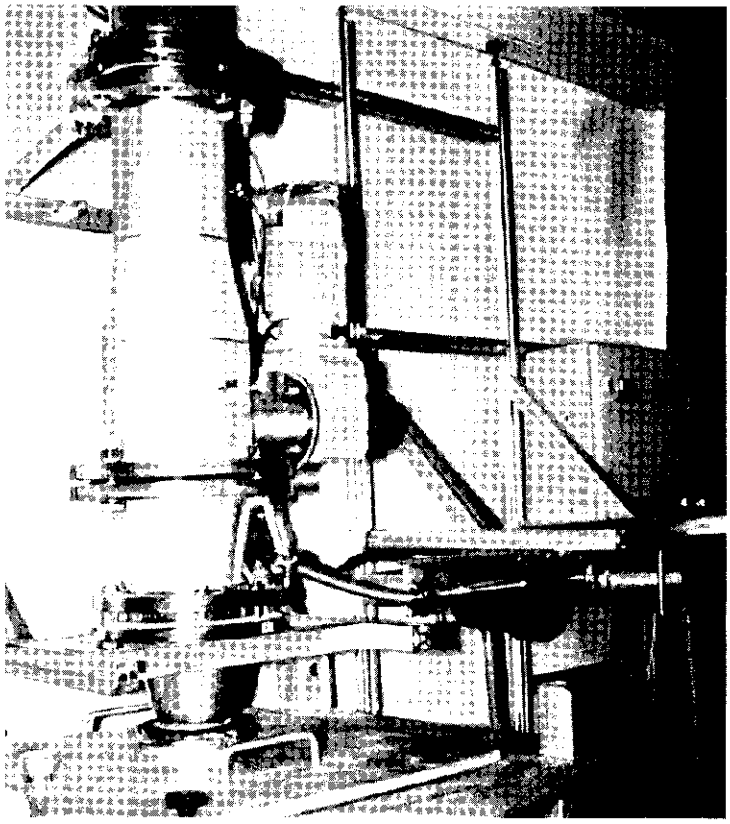
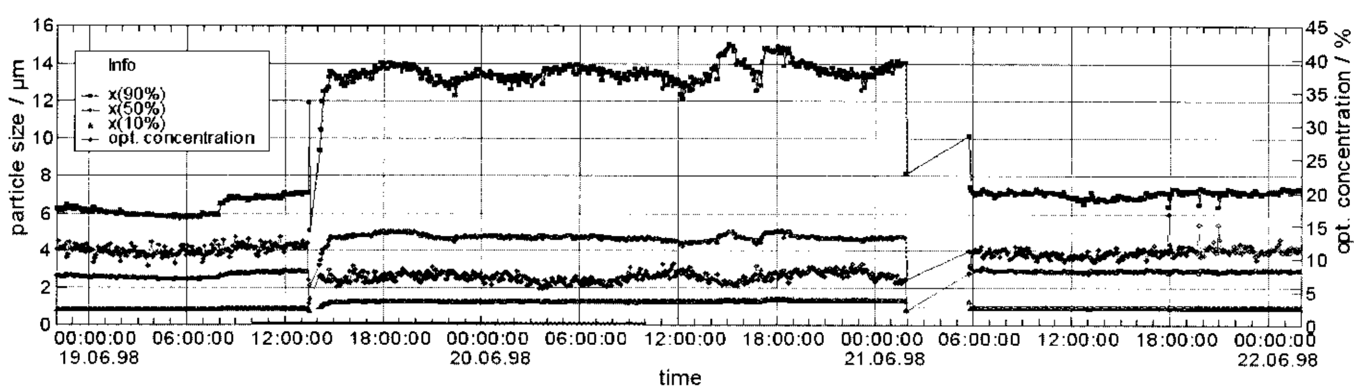


Fig. 10: TWISTER in-line installation with MYTOS on-line

Fig. 11: Batch-monitoring over three days



the information gained is used to improve at the same time the quality of the process and of the product.

10. The system runs with virtually no service. During the period presented in this trial several different polymeric additives were produced and the results stayed reproducible from the first to the 5000th measurement. Cleaning was not necessary even after this long time. Remote servicing by Sympatec via an arbitrary network connection is possible and avoids travelling costs. Today the system has run for more than six months continuously without service and with only one cleaning of the optical components.

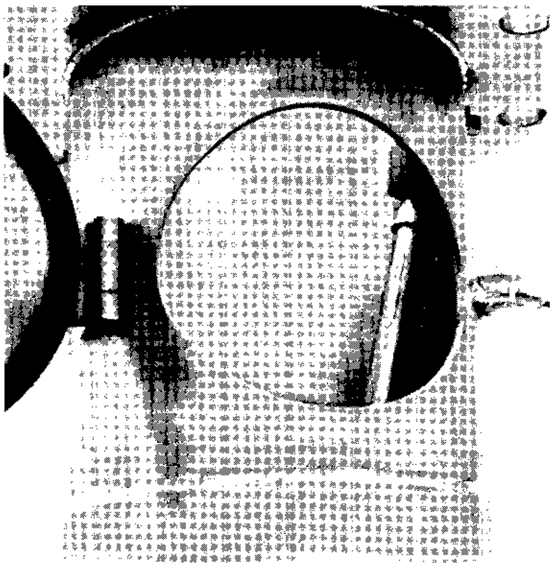


Fig. 12: TWISTER in parking position

Conclusion

The in-line measurement system fulfills all expectations. It is reliable, easy to handle and the features provided for analysis are powerful. As no service was needed and the measurements are performed automatically, interaction with the system only happens when the system is requested to stop or something has to be changed according to the measurement conditions. In general this is only the case when the product changes - and even this can be automated easily.

Of course, the importance of the particle size as a property of the product has been clear before. But especially with the representative sampling the knowledge about the process and the possibility of real time analysis and of immediate intervention are very much appreciated.

Due to the changing products the time of adjustment is crucial for the performance of the production. So the TWISTER & MYTOS system not only increases the product quality by improving the overall performance of the production process but also reduces waste and saves money and production time.

Whether the application of such a high-performance system is really economical can be calculated considering aspects of saved time, saved cost and increased throughput. Generally the more sensitive the particle size distribution reflects the quality of the product and the higher the mass or the value of the ingredients or of the final product the more money can be saved.

The most outstanding feature of the TWISTER & MYTOS particle size analysis is the excellent system-to-system comparability to other TWISTER & MYTOS as well as to off-line HELOS & RODOS. Reasons are the representative sampling in combination with reproducible in-line dispersion and the superior technological properties of the complete system.

In other words: The measuring result is really the particle size distribution and not just a number. Hence, in case of changing the process, confirmation of results in the laboratory and comparability to other production facilities increase the mentioned advantages of TWISTER & MYTOS for the process itself and point out that it is really unrivaled as a dry particle size analysis in-line system.

Summary

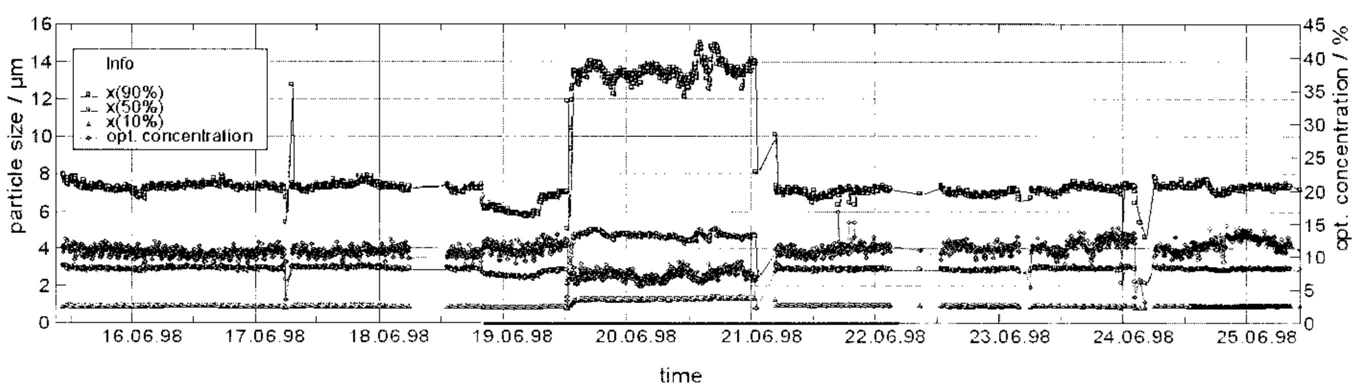
Sympatec GmbH as a supplier of particle size analysis equipment has been strongly involved in the process of developing new solutions. Laser diffraction spectrometer HELOS and dry dispersing instrument RODOS are accepted as most reliable dry particle size analysis system. Although the measuring principle with *Fraunhofer Laser Diffraction* has remained unchanged for the company's fifteen years history, Sympatec managed to establish not only dry dispersion for dry products but also supported the idea of on- and in-line PSA with sampling and measuring equipment.

During the last years several on-line and in-line attempts have been made of which the Sympatec MYTOS & TWISTER is the latest. This new in-line system combines for the first time representative sampling, effective dry dispersion and particle size analysis by means of LD in a single device integrated in a pipe of the production process. Additionally as a solution for undiluted measurement of high concentrated suspensions and emulsions the ultrasonic extinction system OPUS can be applied.

Operation of the measuring system is very easy. The WINDOX operating software offers unique analysis possibilities, remote system control and intuitive handling.

The results obtained with the new in-line system are identical to the off-line HELOS & RODOS analysers. In-line particle size analysis reduces remarkably the effort of sample handling especially with toxic materials or in hazardous areas (*Ex-(p)* optional). Direct control of the process is realised in combination with a standard programmable logical control-system (PLC).

Fig. 13: Production-scan over ten days



SYMPATEC

Sympatec GmbH
System-Partikel-Technik
Burgstätter Straße 6
D - 38678 Clausthal-Zellerfeld
Germany

Phone +49 / 5323 / 717 0
Fax +49 / 5323 / 717 229
e-mail marketing@sympatec.com