Control and Optimisation of Cement Quality with Laser Diffraction Particle Size Analysis and Dry Dispersion

Dr.-Ing. Ulrich Kesten

Sympatec GmbH, D-38678 Clausthal-Zellerfeld, Germany

1 Introduction

Improvement and guarantee of product quality on highest level have become one of the important issues for industrial production in general and on a global scale.

In the production of cement, the particle size distribution of the final products is one of the important parameters for quality, as its guarantees for example the final strength of the concrete. However, the particle size distribution is not only important for the product quality, it can also be used to characterise the efficiency of the production devices such as the cement mills and the classifiers. Optimisation of operation cannot only help to improve product quality it also has an important impact on the economical aspect of cement production. If due to immediate control of the milling and classifying circuits the amount of the re-circulated coarse fraction can be decreased by only few percents, the yield increases and the cost for energy per ton cement decreases remarkably.

Fortunately today the particle size distribution of cement can easily and reliably be determined.

With the introduction of the first laser diffraction instruments some 25 years ago, the basis for industrial particle size analysis and production control was laid.

The first generation of laser diffraction particle size analysers was presented in the early 1970's. It had been developed in close co-operation with the French cement industry and it was applied exclusively with suspension dispersion, i.e. as wet liquid systems.

In 1984 a new generation of laser diffraction analysers was introduced. It is based on the simple idea that the instruments used for the characterisation of quality parameters have to adapt to the products and processes and not vice versa, i.e. that the products have to adapt to the instruments. This was the established way of how analytical equipment was designed.

Dry powders should be dispersed dry, wet products and suspensions should be dispersed in the wet state. This is the practical and consequent conclusion, and the new idea consequently led to the development of a range of laser diffraction analysers and dispersing systems that are of modular design thus being easily adaptable to a wide variety of different products and processes.

Sympatec GmbH from Germany have developed a to date unique instrument for the dry dispersion of powders in the particle size range from 0.1 µm to nearly 10 mm. This patented instrument, the RODOS, allows for a smooth, complete and product adaptable dispersion of all kinds of dry powders.

In combination with the laser diffraction instrument, HELOS, a powerful system for industrial particle size analysis of the powders is available.

2 Laser Diffraction Principle

The history of Laser Diffraction as an advanced optical analysis principle for fast determination of particle size distributions in a wide size range, i.e. from 0.1 to x.000 µm, begins in the early 19^{th} century with the experimental work of Mr. Fraunhofer in Munich (table 1) [1].

Elements	Definition	Year
Theory	**Fraunhofer** diffraction physics	1840
Method	He-Ne-laser as high energy, coherent light for the generation of **particle-light interaction**	1960
Sensor	**multi-element semiconductor** first used as aiming device for military application	1965
Processing	high performance **microcomputers**	1970
Result	**particle size analysis with laser diffraction**	since 1972

table 1: History of Laser Diffraction

Those experiments are the basis of what is today known as Fraunhofer diffraction physics.

From these first experiments it took about 130 years for the technical realisation of the first laser diffraction particle size analyser in 1972 [2].

The optical set-up for the generation of diffraction patterns (fig. 1) consists of a laser as light source, an optical beam expander that widens the very fine laser beam to more than 20 mm in diameter, and a measuring zone where the sample is introduced and taken out.

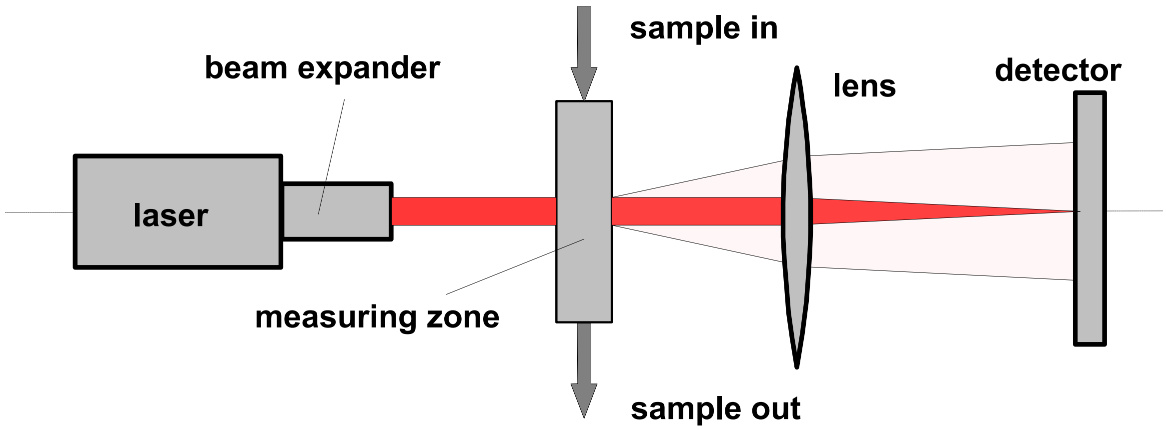


fig. 1: optical set up for the generation of diffraction patterns

The measuring zone is the position where the interactions between the monochromatic, coherent light and the particles take place and the diffraction patterns are generated.

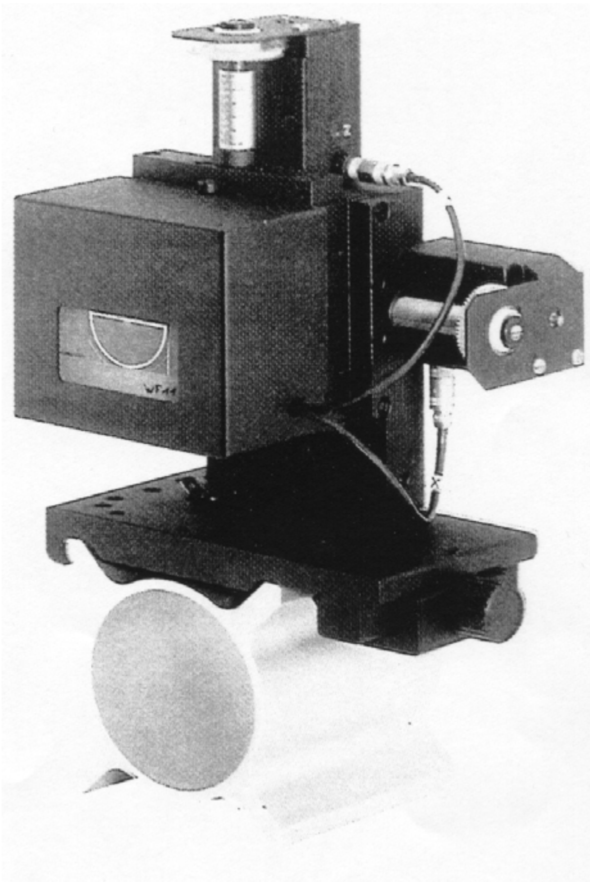


fig. 2: multi-element detector and AUTO-FOCUS unit

These phenomena of intensity distribution of diffracted light are focused on the surface of an optical detector with the help of a Fourier transformation lens of a distinctive focal distance. The detector usually consists of up to 31 semicircular elements. They are transforming the optical light intensity into the corresponding electric current (fig. 2).

A mathematical relationship was established between the diameter of the particles and the intensity patterns [3]. For a particle size distribution, consisting of fine, intermediate and coarse particles, the diffraction pattern monitored with the detector incorporates a two-parameter information; it is the distribution of particle sizes and the quantities in which the different particle classes contribute to the distribution.

Amongst other elements of the laser diffraction sensor, such as the mechanical stability, the optical components and the electronics of highest quality, an extremely important feature that determines the quality of the measurement to a very large extent is the accurate alignment of the optical set-up. The precise focusing of the central laser beam to the centre of the detector determines the quality and long-term stability of the measurements. Sympatec have realised a focusing system, which automatically and continuously checks this alignment and, if necessary, corrects it before a measurement can be released (fig. 2).

3 Laser Diffraction Systems and Dispersing Units

As a result of the idea of adaptation of the instruments to the products and processes a completely modular series of instruments is now available and it comprises instruments for off-, at-, on-line and in-line particle size analysis with laser diffraction.

The Laser Diffraction Sensor HELOS is the central unit of the analytical system. It comprises the laser light source, the different Fourier lenses, the multi-element detector and the system computer for control of the central unit and the evaluation of the particle size distribution data. Today it covers a size range between 0.1 µm and 8750 µm.

Dispersers are the instruments that are preparing the sample for presentation to the laser beam of the central unit. The sensor will see the particles as they are presented, i.e. agglomerates of fine particles will be measured as big particles. Hence the disperser must guarantee a complete and proper dispersion of the agglomerates down to the primary particle size. If a dry powder has to be analysed, a dry powder disperser is the best choice, if a wet product, a suspension, or an emulsion has to be analysed, a suspension or wet dispersing system is necessary. Approximately 40000 different disperse matters are being produced in worldwide industry. As a consequence appropriate dispersers for dry and wet products have to be provided and must be combined with the laser sensor.

As for cement the dry dispersion is the ultimate objective, a closer look to the dry powder disperser RODOS [4], will be useful.

Due to its two-stage design RODOS can be used with powders in a very wide size range from below 0.1 µm to more than 8 mm (fig. 3).

Depending on the dispersion characteristics of the powders, different adapters are applied to feed the sample to the gas-solids injector, being the centrepiece of the disperser.

For very fine and adhesive powders the sample is fed in surplus into a rotary groove with the help of a vibratory feeder. Additional scrapers and rollers are compacting the powder in the groove. Thus a homogeneous mass flow of powder is presented to the rear end of the disperser. The gas-solids injector is driven by compressed air. In a nozzle it is accelerated to a velocity of about 50 m/s.

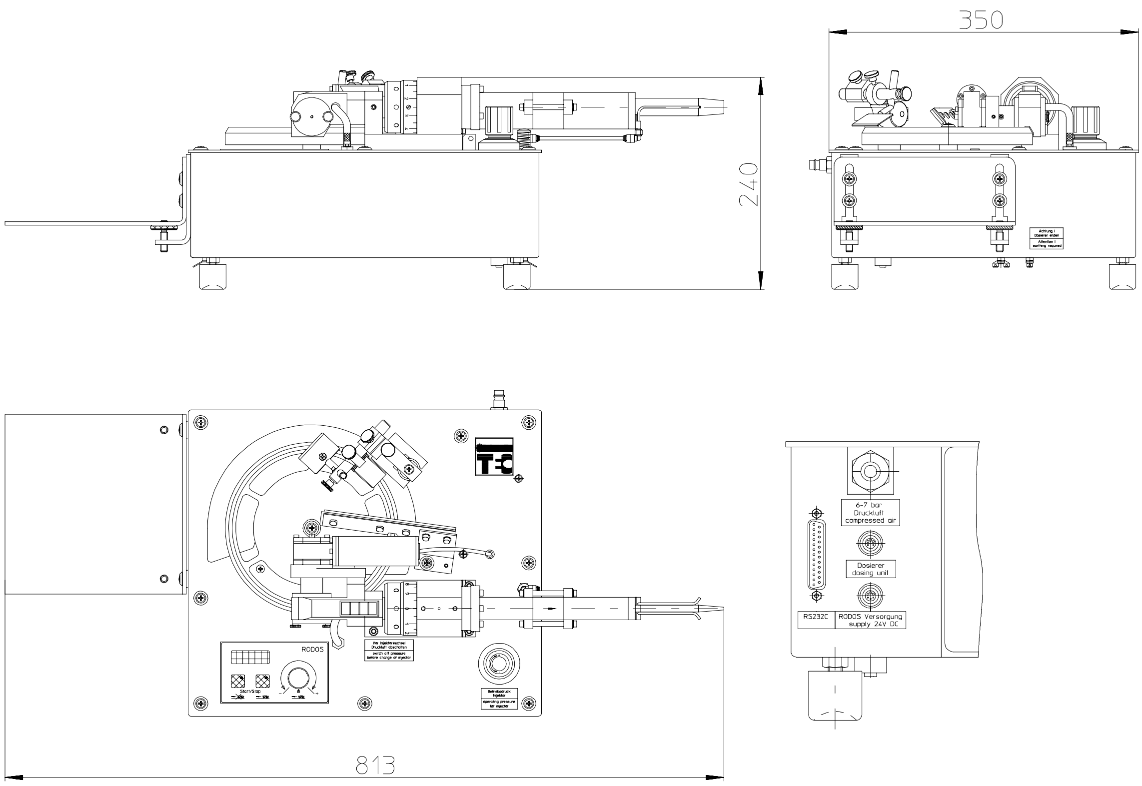


fig. 3: dry dispersing system RODOS

Due to the acceleration of the compressed air, a vacuum is produced at the rear end of the disperser and the previously homogenised powder is taken into the disperser. In a zone of steep velocity gradient between the air and the agglomerated particles, shear forces are applied to destroy these particle aggregates. Additional particle-to-wall and particle-particle collisions and centrifugal forces generated by the rotation of such aggregates completely and smoothly take the agglomerates apart, down to the primary particle sizes.

The powder leaves the disperser as an aerosol in a free jet and crosses the laser beam of the HELOS instrument at a very short distance from the exit of the disperser. After passage of the laser beam the aerosol is completely taken out of the air with the help of an extraction nozzle and a vacuum cleaner.

The advantages of the dry dispersion with RODOS over suspension (wet) dispersion are manifold and include complete and proper product specific dispersion, adaptable dispersing forces and variable sample size, highest measurement frequency available to date, as well as a guaranteed long life time of the disperser with minimum operating costs (table 2).

H **complete and proper dispersion of all dry powders from 0.1 µm**

Ä sensitive to changes also in the fine particle range (< 10 µm)

H **adjustable dispersing forces**

Ä best adaptation to dispensability of different products

H **variable sample size**

Ä minimising the statistical sampling error

H **high measurement frequency due to short cycle time**

Ä no cleaning, no rinsing, of sample container

H **no chemical interaction between particles and dispersing fluid**

Ä no dissolution, no surface reaction, ...

H **no reprocessing of contaminated liquid**

Ä (e. g. alcohol filtration)

H **guaranteed long lifetime of disperser**

Ä low operating cost
Í maximum up time

table 2: ADVANTAGES of dry dispersion with RODOS

With regard to application in the cement industry RODOS is the natural way for the dispersion of

- cement
- raw meal
- coal dust
- fly ash
- ....

4 Automation of Industrial Particle Size Analysis

In addition to the off-line instruments designed for application in labs of all branches with manually operated dispersing systems, Sympatec have created a new line of instruments, called at-line, for application in automatic laboratories for continuous quality control. These instruments automatically perform a particle size analysis once a sample has been filled into the disperser. They can be operated as stand alone solutions and, due to their ability to co-operation with robots and central process control systems, they can be integrated into the laboratory automation systems supplied by well-known plant manufacturers.

A typical cycle time for a measurement with an integrated auto-line system with the automatic dry dispersing unit auto-RODOS module totals to less than two minutes (table 3).

step	action	auto-RODOS	auto-SUCELL
1	information input	5“	5“
2	initialisation	10“	10“
3	fill-in sample	15“	60“
4	dispersing & sampling	10“ - 30“	10“ - 30“
5	sample take-out	15“ - 35“	60“ - 120“
6	data evaluation	5“	5“
7	information out	5“ - 60“	5“ - 60“
Σ	**1. - 7.: [minutes]**	**< 2**	**5**
8	stand-by mode	1“ - ∞	1“ - ∞

table 3: operation modes and cycle times for automatic dispersers

Included in the cycle is the information exchange between the central control computer of the automatic lab and the HELOS system computer, the fill in of the sample, the dispersion and measurement of the size distribution, the evaluation of data and transfer to the host computer [5].

For all those applications where not only the size distribution of cement has to be controlled but also those of the raw meals, the coal dust for the heating of the furnaces and the fly ashes as additive to the cement, a fully automatic version of the HELOS central unit allows for automatic change of the measuring range (auto-ranging).

It means that the size distribution of an extremely fine cement quality can be analysed on the same instrument as a relatively coarse and broadly distributed raw meal without any operator touching the instrument. In case the at-line instrument is integrated into an automated central laboratory, the host computer of the automatic lab announces for example the different types of products to the HELOS system computer and this will initialise the central unit to change the measuring range by selection of the appropriate lens and relocation of the detector unit to the related position on the optical bench; after auto-focusing the system will run the sample and determine the particle size distribution with the measuring range that fits the products in the optimum way.

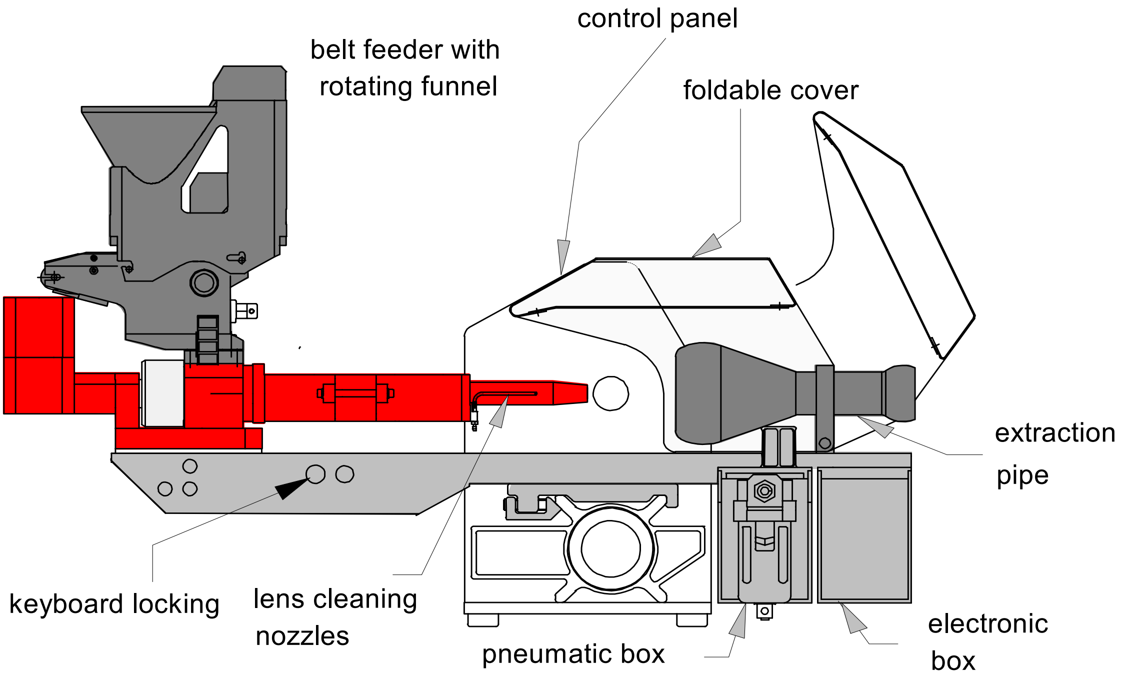


fig. 4: auto-RODOS module for automatic particle size analysis

Two typical realisations of automatic cement laboratory systems are shown in the photos (fig. 5 and 6). One solution is based on a single robot, being centrally installed in the lab and serving the different analytical equipment sequentially, the other disposes of different cabinets with one small robot being dedicated to the service of one or several analytical instruments at a time.

5 Cement Applications

Since 1986 a remarkable number of HELOS systems have been installed in the cement industry world wide. All of them are equipped with a RODOS dry dispersing unit. 65 % are operated off-line in the manual mode and 45 %, with quickly increasing trend, are operated in the automatic version as part of automatic cement laboratories.

As typical results for particle size distributions of products in the cement industry the differential distribution curves q_{3lg} of Portland cement in the PZ 35, 45, 55 qualities analysed

fig. 5: ***HELOS and auto-RODOS installations in an automatic cement Lab: ROBOLAB of F.L. Smidth, Copenhagen***

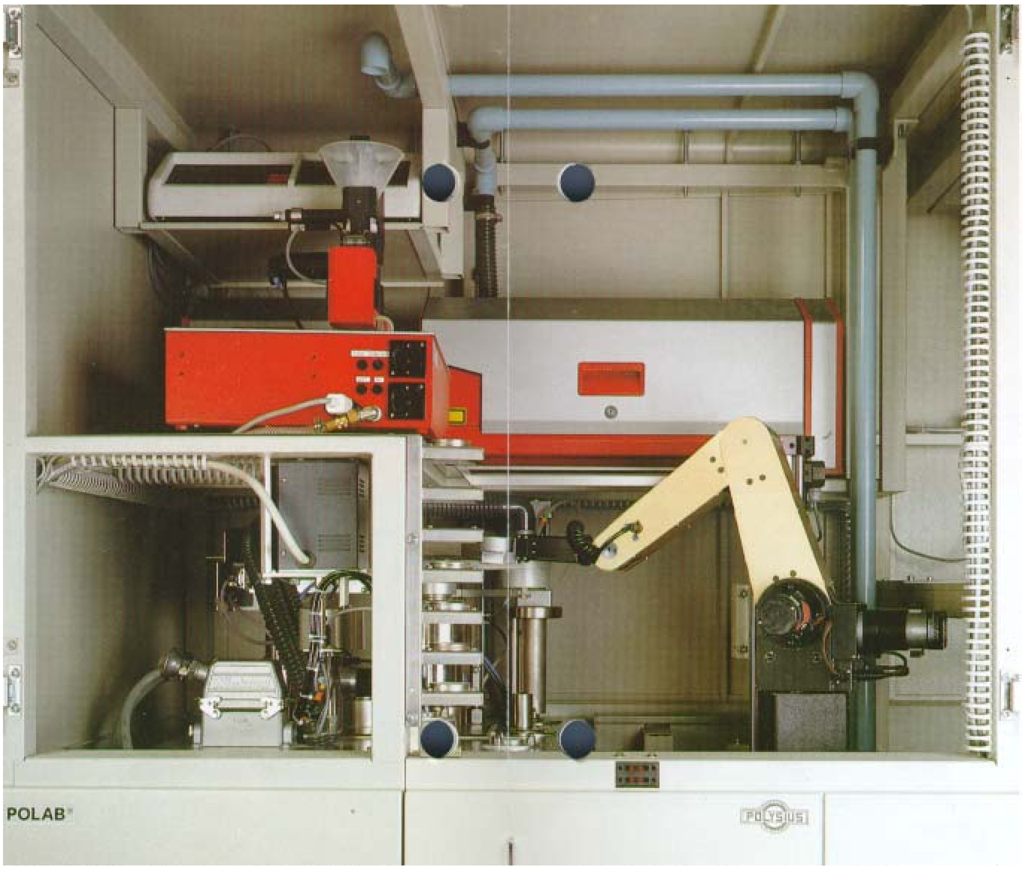


fig. 6: HELOS and auto-RODOS installations in an automatic cement Lab: POLAB, Krupp-Polysius, Neubeckum)

with HELOS and auto-RODOS module, are plotted on a logarithmically subdivided abscissa (fig. 7). The presentation is in accordance with the ISO 9276-1 standard. The distribution curves show the typical multi-modal shape of cement with expressed fine tails.

The ability of the RODOS dry disperser to even properly dispersing the extremely fine powders of the new generation of Micro-Cements is proven by the next graph (fig. 8). The cumulative distribution curves by volume Q_3 and the corresponding differential distributions curves q_{3lg} of three ultra-fine cements show that these qualities contain more than 20 to 30 % of particles below one micrometer and a maximum particle size of only 20-30 microns.

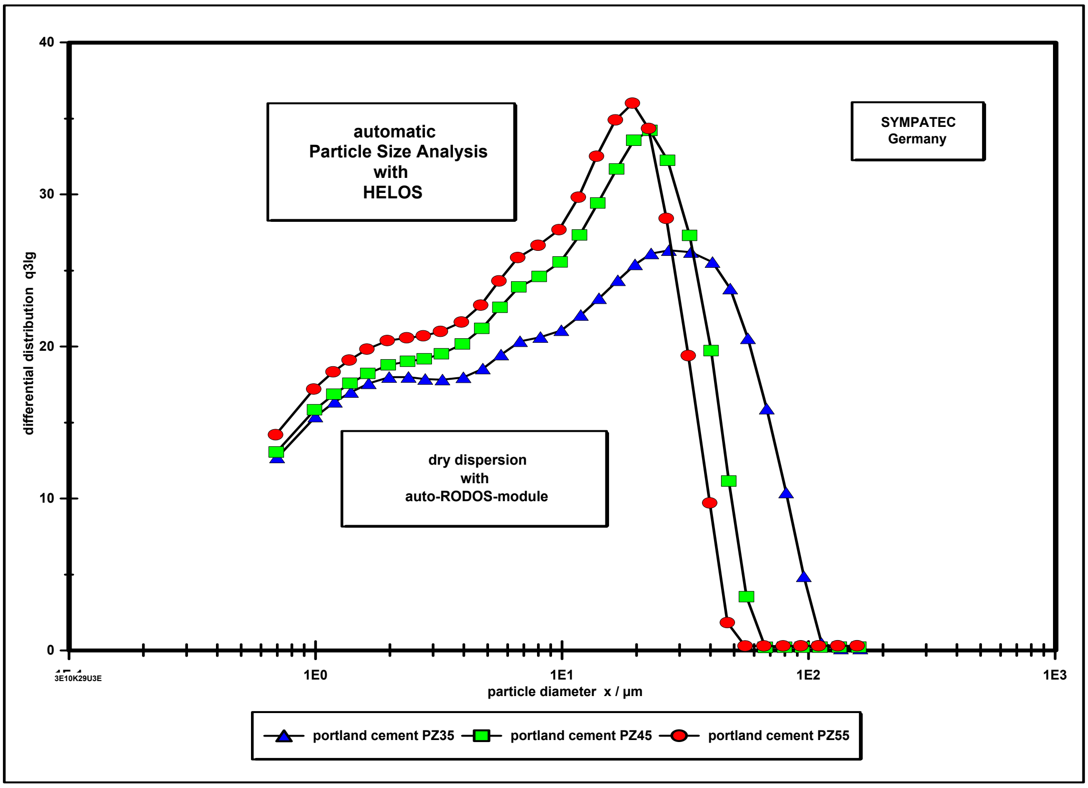


fig. 7: typical particle size distributions of Portland cements

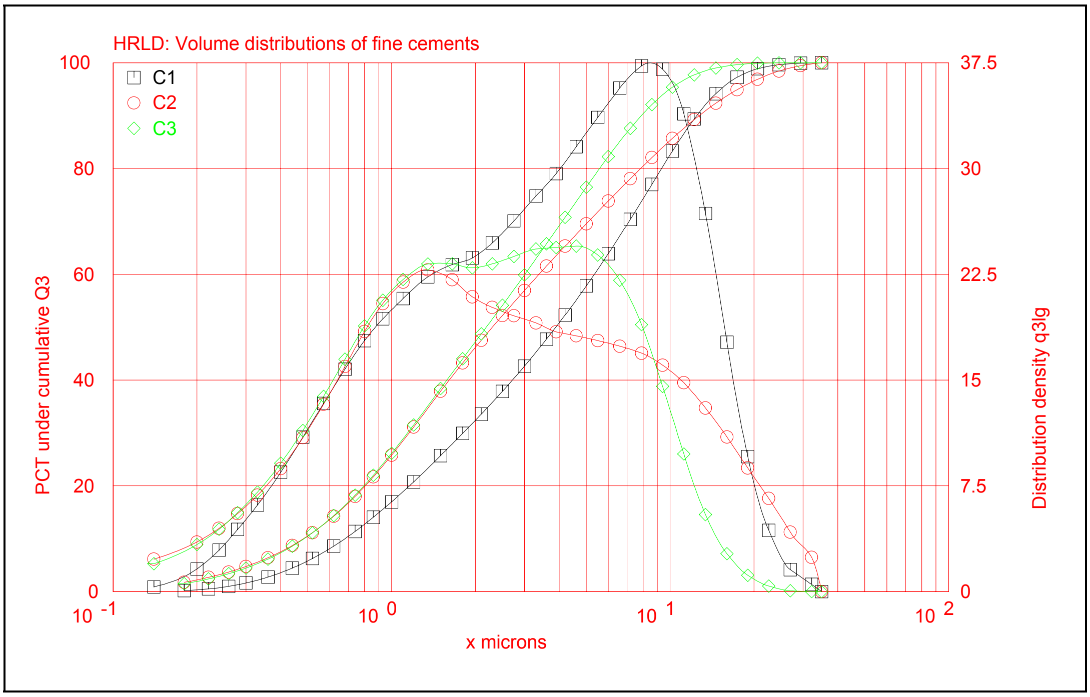


fig. 8: RODOS: dry dispersion of ULTRA-FINE (SUBMICRON) micro-cements

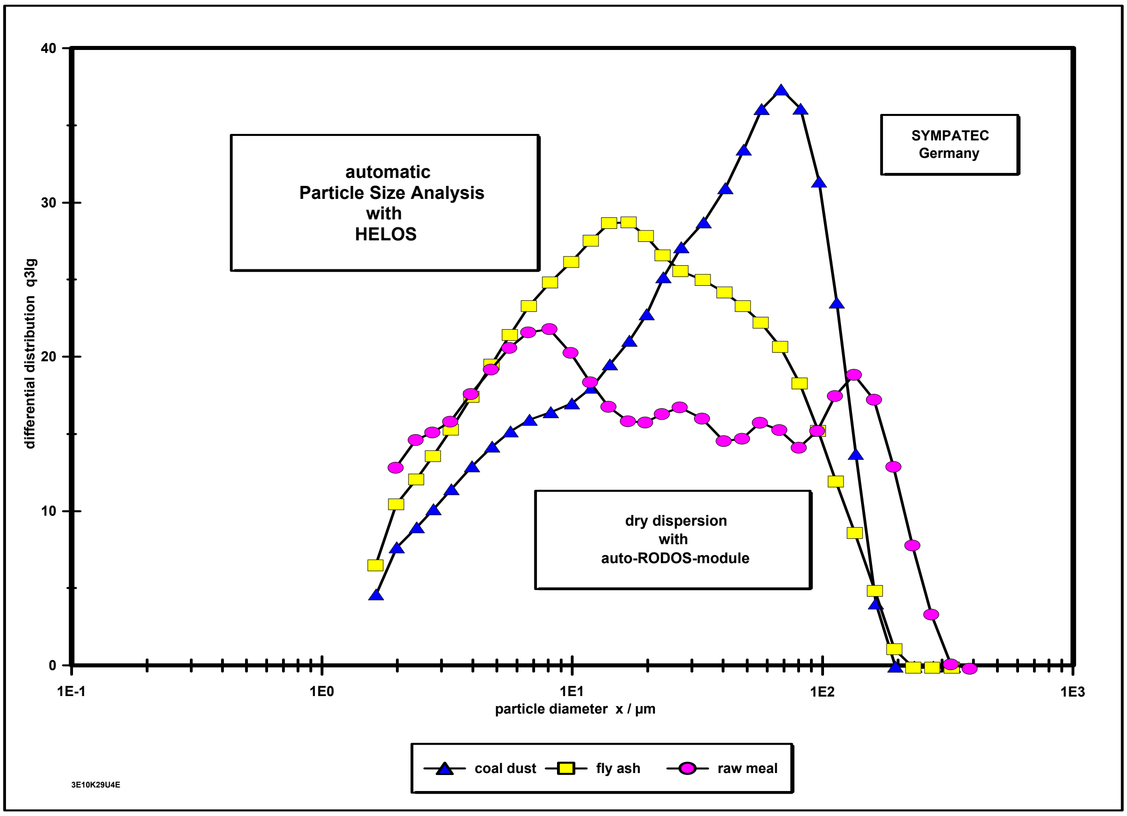


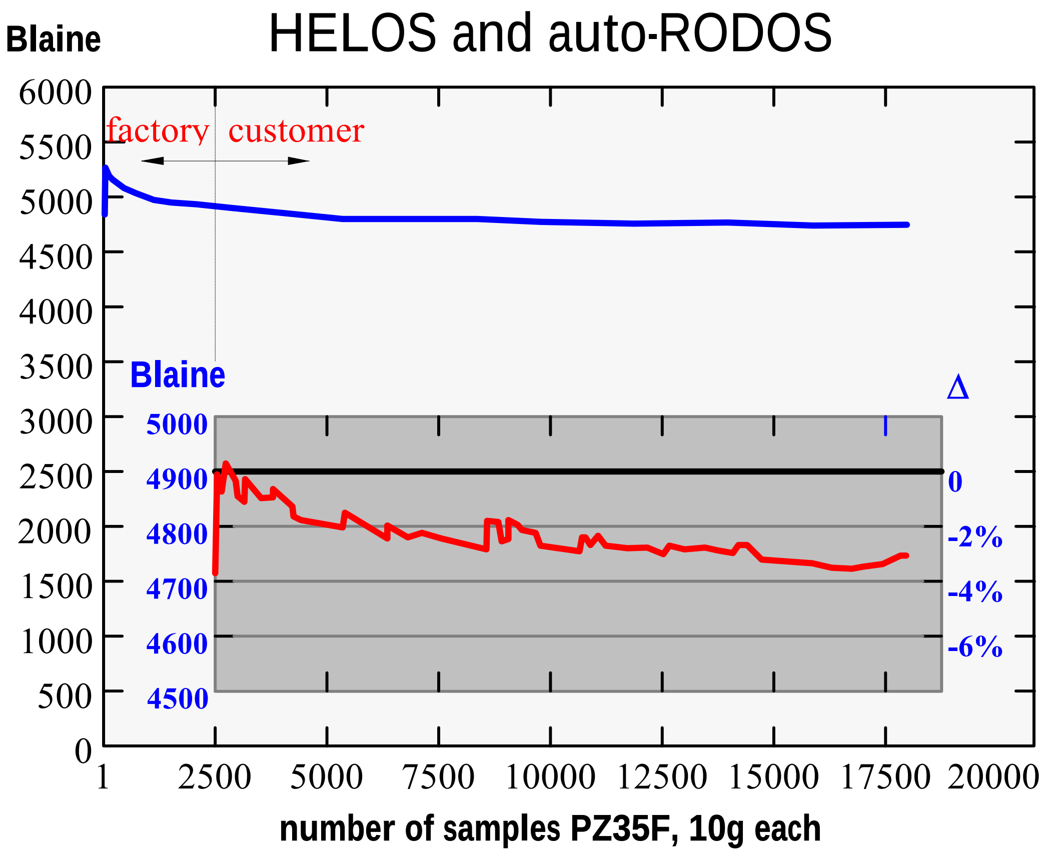
fig. 9: particle size distributions of raw meal, fly ash, coal dust

In an additional graph the results of the analysis of raw meal, coal dust and fly ash are shown. Of special interest is the extremely broad distribution of raw meal with a manifold of modes in the differential curve that might be related to the numerous components of raw meal (fig. 9).

The extreme long time stability of the auto-RODOS module as operated with the HELOS laser diffraction instrument is illustrated in fig. 10.

The standard deviation over the lifetime is less than 1 % or expressed in terms of the BLAINE surface, inferior to +/- 40 cm²/g for 4000 cm²/g (fig. 10). With regard to the long time stability of the dispersing line of the auto-RODOS instrument, Sympatec guarantees a life time of minimum 25.000 measurements of PZ 35 F, i.e. a life time of typically far more than one year in an automatic production control laboratory.

Compared to the operating cost of systems that are using suspension dispersion in alcohol, the dry dispersing system RODOS has an advantage of probably more than 50%.



Blaine-values

$$\boxed{S_M = \frac{6\,f}{\rho}\sum_i \frac{q_3}{x_i}\Delta x_i}$$

fine fraction <10 µm is significant for the specific surface !!

standard deviation:	**< 1%, typical 0,4%**
Blaine values:	**± 40 cm²/g** for 4000 cm²/g
lifetime:	ca. **250 kg** PZ 35 F, i.e.
	>25.000 samples, 10 g each,
	or typical **1 year in 24h**
	continuous operation

fig.10: guaranteed long-time stability of RODOS and HELOS measurements

6 Process Optimisation

Optimisation of operation of unit operations with particles can not only help to improve product quality it also has an important impact on the economical aspect of cement production. If due to immediate control of the milling and classifying circuits the amount of the re-circulated coarse fraction can be decreased by only few per cent, the yield increases and the cost for energy per ton cement decreases remarkably.

For the characterisation of the efficiency of mills and classifiers the ***Grade Efficiency*** or ***Tromp curve*** has been used successfully.

From the complete mass balance of feed material into the unit operation, e.g. the classifier, the fines (product) and the coarse (re-circulated material) and the particle size distributions of at least two of the components, the Tromp curve T(x) can be determined according to the following equation:

$$T(x) = \frac{C \cdot q_c(x)}{q_f(x)}$$

T(x) represents the ratio of the relative amount of material of a certain size, present in the coarse material (oversize, reject) ($cq_c(x)dx$), to the relative amount of the same size initially present in the feed material ($q_f(x)dx$).

C = relative amount of coarse material

$$C = \frac{M_c}{M_f} = \frac{M'_c}{M'_f}$$

M_f, M'_f = amount, mass flow rate of feed material

M_c , M'_c = amount, mass flow rate of coarse material

Fig. 11 represents the Tromp Curve T(x) as determined from the particle size distributions of the Feed, the Coarse and the Fines of a separation or classification process.

And with fig. 12 an explanation and discussion of a typical course of a Tromp curve is given.

An ideal separation would be such that for a desired and set separation diameter x_t the fines would not contain any amount of course particles and the coarse would not contain any amount of fine particles. Hence the Tromp curve T(x) would be a straight line parallel to the ordinate of the Tromp curve graph. In reality Tromp curves have an S-shaped course with a gradient becoming steeper with a better operation of the classifier. Very often we find the Tromp curve not even ending at the bottom line of the abscissa but also moving upwards again in the fines.

The distance from the minimum of T(x) to the bottom line of the abscissa is referred to as τ. It represents the amount of feed material that is passing through the classifier without separation. This is often due to an overload of the separator.

The increase of the Tromp curve in the fine particle range refers to the amount of fine particles in the coarse fraction. This is usually due to the fact that the classifiers are fed with incompletely dispersed powder. The agglomerates hence are taken as coarse particles and during the particle size analysis with properly operating dispersers they are detected as the fine particles they have been in reality.

Potentials for the optimisation of the classification process are to be found in the improvement of the dispersion of the feed material before it enters the classifier zone. The resulting reduction of the amount of fines in the coarse fraction decreases the re-circulated mass flow into the mills. This decreases the energy consumption per ton of product remarkably. In addition the amount of the fine (product) fraction increases, and hence the yield of the process is much better. Already the improvement of this detail in the large number of parameters affecting the process of cement manufacturing has a dual positive effect. It can be achieved with the control of the particle size distribution using the laser diffraction principle and the dry dispersion.

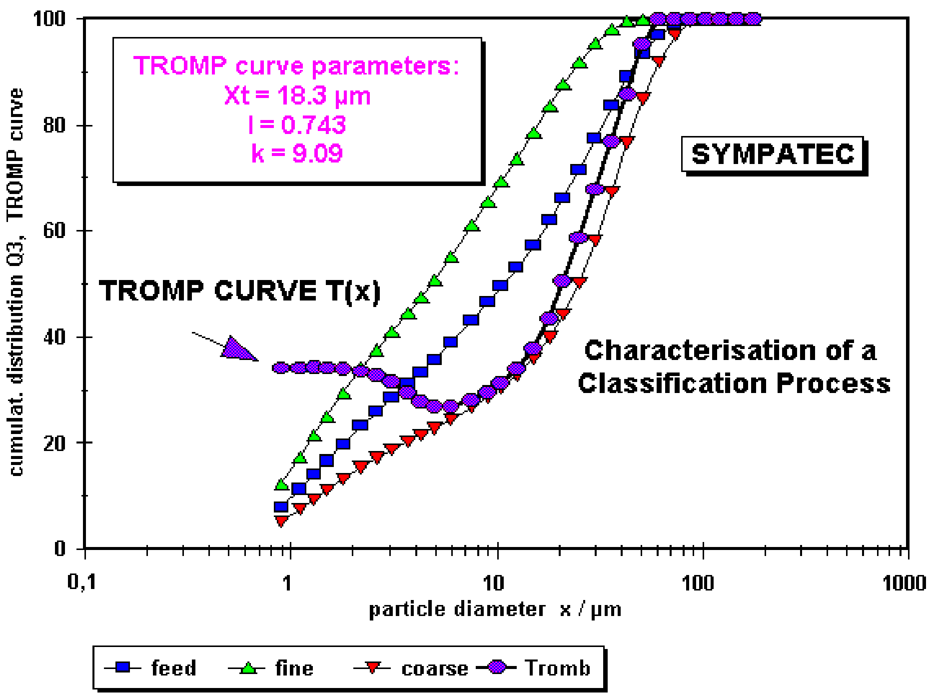


fig. 11: characterisation of classification process with TROMP curve

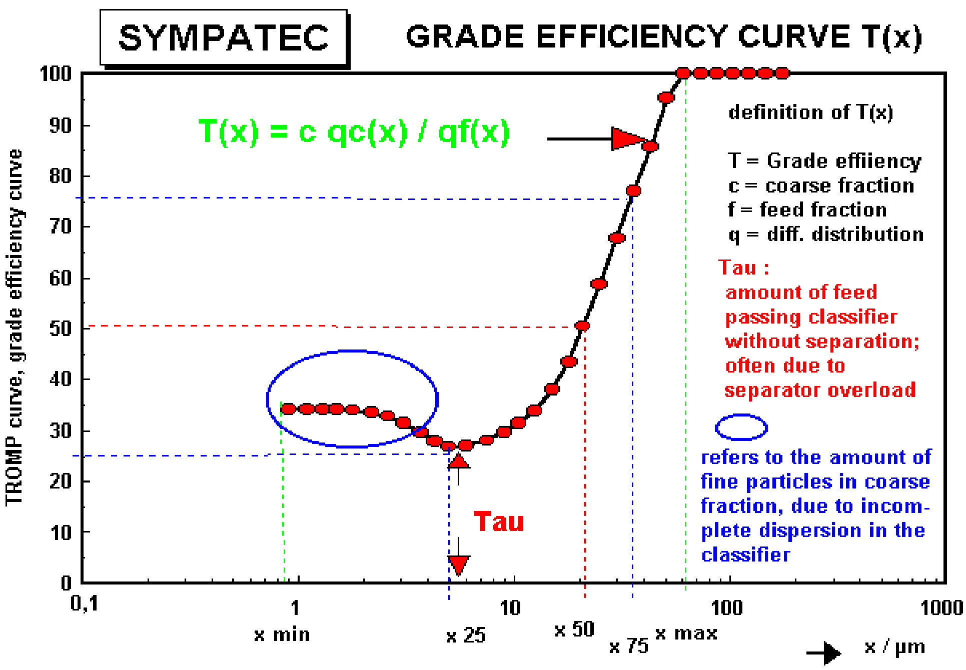


fig. 12: typical course of a TROMP curve of a real classification process

7 References

[1] *J. Fraunhofer*
Bestimmung des Brechungs- und Farbzerstreuungsvermögens verschiedener Glasarten
Gilberts Annalen der Physik, 56 (1817) 193-226

[2] *Stephan Röthele, Ulrich Kesten*:
The Application of Laser Diffraction Technology,
2nd World Congress of Particle Technology 1990,
19. - 22. September 1990, Kyoto, Japan

[3] *Michael Heuer, Kurt Leschonski*:
Results Obtained with a New Instrument for the Measurement of Particle Size Distributions from Diffraction Patterns,
Part. Charact. 2 (1985) 7 - 13

[4] *Kurt Leschonski, Stephan Röthele, Ulrich Menzel*:
A Special Feeder for Diffraction Pattern Analysis of Dry Powders,
Part. Charact. 1 (1984) 161 - 166

[5] *Stephan Röthele, Heinz Naumann, Udo Brandis*:
on-line Sampling and Sample Splitting -
Principle and Instrumentation
6th Conference Granulometry, 21. - 22. Nov. 1989, Dresden

[6] *Kurt Leschonski*
Representation and Evaluation of Particle Size Analysis Data
Particle Characterisation 1 (1984), 89 - 95