OPUS | Ultrasonic Extinction

Particle Measurement | Process | Wet

Size and Concentration | < 0.1 µm to 3,000 µm

Sympatec develops, manufactures, sells, services and supports a range of best instruments for particle size and shape analysis in laboratory and process applications for customers worldwide. With continuous innovations Sympatec makes a prominent contribution to laser diffraction, image analysis, ultrasonic extinction and photon cross-correlation spectroscopy.

The Sound of Your Particles

on-line Measurement of Particles in Highly Concentrated Liquids

No light in the dark

When making chocolate, cosmetic creams, paints, salts or crude oil products, highly concentrated ⊙ dispersed systems are produced. These slurries, pulps, pastes and emulsions contain high particle or droplet concentration impenetrable for light waves.

In order to be able to use optical methods for particle size analysis, a sample is taken from the production stream and usually diluted by several orders of magnitude. This may negatively affect the analysis quality and often leads to misinterpretations of measuring results. Dilution modifies the original sample state and can cause instability due to pH changes, emulsifier concentration or zeta potential differences.

A meaningful on-line analysis in the mass flow usually allows no dilution and thus excludes optical methods. Where the limits for electromagnetic light waves is reached, the acoustic waves of ultrasound unfold its full strength. Ultrasound propagates almost unhindered in liquids and solids and sheds "light into the darkness", i.e. to optically impenetrable media.

The sound of particles

Imagine a room filled with people. If the light conditions are good, we can count the number of people and assign most of them to an age group, according to their physical appearance. In the dark, this is not possible. However, if we ask those present to speak, we can deduce quantity and age by their voices.

Ultrasonic principle

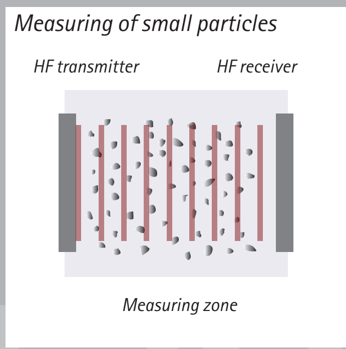
Usually, particles themselves are not a source of sound. That is why they are made "audible" by excitation from the outside. Particles and droplets embedded in a liquid matrix often form an optically impenetrable suspension or emulsion which is then penetrated with low energy ultrasound generated by a high frequency (HF) transmitter. On the receiver side, the incoming sound waves are detected. While the sound waves in pure liquids propagate nearly unresisted, they are scattered and absorbed in the presence of disperse phases.

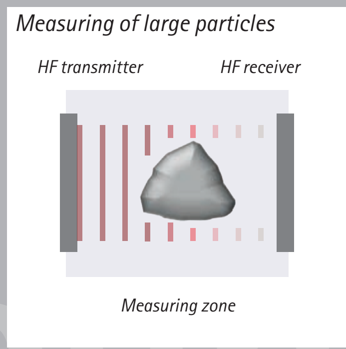
$$-ln\left(\frac{I}{I_0}\right)_{f_i} = \Delta l \cdot C_{vol} \cdot \frac{1,5}{x} \cdot K(\sigma)$$

Lambert-Beer-Law

Caused by the characteristic of the high frequency set-up, the deflection of the waves from their direction of origin leads to a loss of acoustic energy recorded on the receiver side. This effect is determined by the size ratio between wave length and particle and is also called attenuation. Comparatively small particles exert a low attenuation effect on sound waves. As the particle size increases, the attenuation increases. If large and small particles are present at the same time strong and weak attenuation effects superimpose.

The physical description of the acoustic effect applies analogous to optical waves based on the Lambert-Beer-Law. Assuming monodisperse particles, we have a simplified, reduced equation.

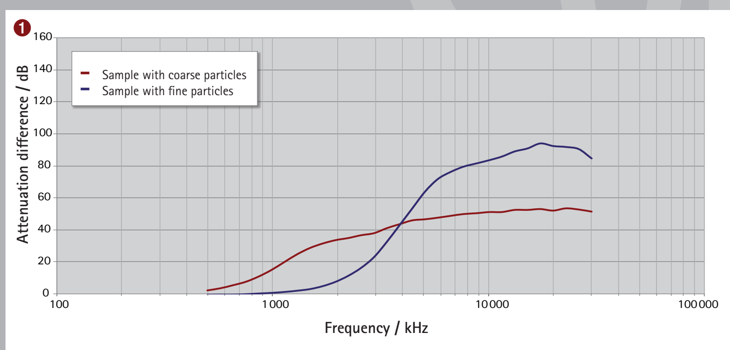




The equation shows a linear relationship between the measurement effect reflecting ⊙ the sound attenuation on the left side and the term written on the right side including the thickness of the suspension layer Δl, the solid volume concentration C_{vol} and a reciprocal relationship with the particle size x. ⊙ The factor K(σ) symbolizes the extinction function and describes the interaction of the particles in a suspension with the ultrasonic waves as a function of their size ratio.

In order to detect several particle size classes within a sample or a mass flow, attenuation measurements are required with different frequencies. The resulting frequency-dependent attenuation spectrum is reverse calculated to the underlying particle size and volume concentration using appropriate software.

❶ The diagram shows two attenuation spectra. The sound attenuation in dB is plotted over the frequency in kHz. While the sample with the coarser particle size distribution at low frequency (large wave lengths) produces significant sound attenuation, the fine sample achieves comparable attenuation only at higher frequencies (smaller wave lengths). This spectral shift from low to high frequencies forms the basis of particle size analysis using ultrasonic extinction.



⊙ *Dispersed systems are mixtures of substances consisting of two or more phases. In this case, one substance is distributed in a different substance in the finest form.*

⊙ **Sound attenuation** *refers to the obstruction of sound propagation through the absorption of airborne sound from particles in liquids. It is determined from the ratio of the received and emitted sound intensity I at a given frequency f_i.*

⊙ **K(σ)** *is a counterpart to the refractive index, known from light-optical physics.*

Product adapted. Durable. Robust.

Process-proven Method for Particle Size Analysis

OPUS with ultrasonic extinction

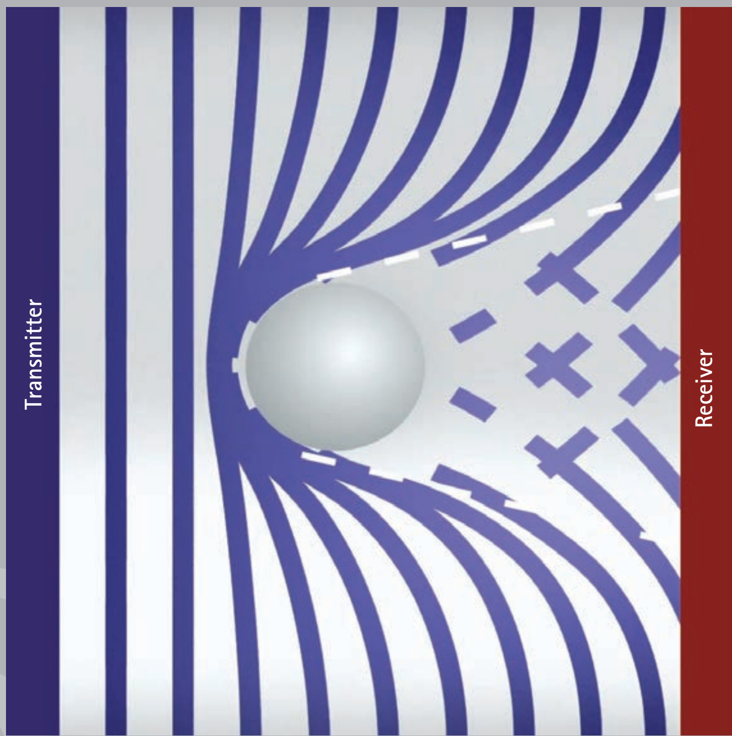
Since 1995, the robust, process sensor OPUS based on ultrasonic extinction, for simultaneous detection of particle or droplet size and volume concentration for process control and production monitoring in liquid process stages, has been applied to many applications.

The design is deliberately chosen in a way that the measuring zone – similar to a temperature sensor – is immersed directly into the suspension or emulsion to be analysed. Various process adapters make it easy to install the sensor in large process pipes and vessels, as well as in small hose lines of a few millimetres. Even compact desktop devices for laboratory use are available.

OPUS covers the entire pH range from 1 to 14. Employing a total of 31 measurement frequencies takes only a few seconds, so that analysis results are available by minute cycles in real time operation.

Powerful and gentle

Despite roughest operational conditions the precision of the ultrasonic technology remains constantly at a high level. Variations of typical process conditions, such as temperature, pressure, flow rate and direction, concentration, or the particle size distribution itself, do not affect the accuracy of the analysis. Thanks to a wide dynamic range of up to 160 dB, attenuation values can be scanned over a wide frequency range of 0.1 to 200 MHz.

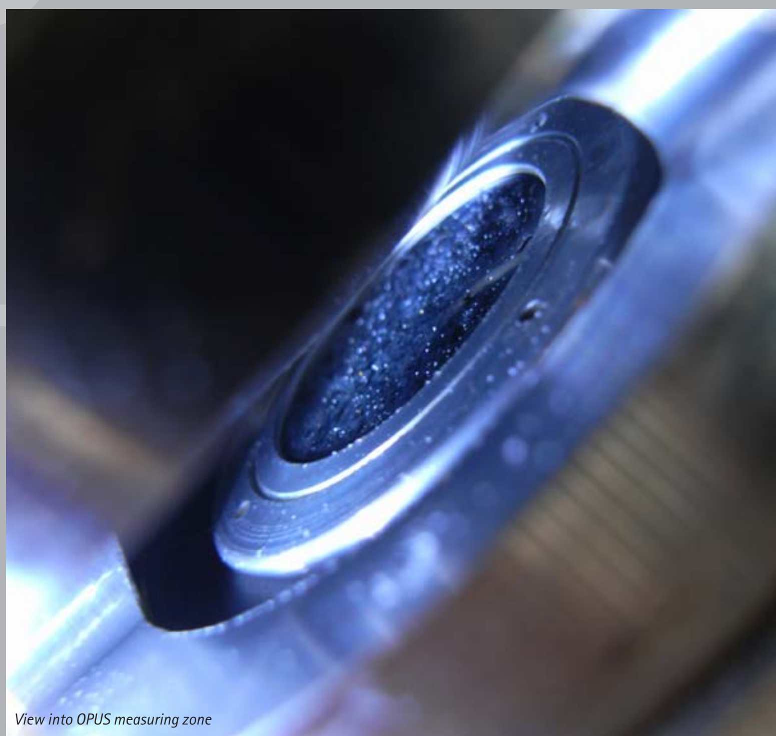


»Just as whales and dolphins scan their cloudy and dark surroundings with ultrasound, OPUS ensures that your particles are identified and described precisely.«

By the application of ultrasound technology, the use of mechanically, chemically and thermally resistant materials is possible. In areas where sensitive lenses and glass elements of photo optics are not allowed to come into contact with the products, ultrasonic extinction with impact and scratch-resistant sensor surfaces made of SIGRADUR®, a carbonised glass, stays in close contact with the process media. High-alloy stainless steel and sealing materials made of KALREZ® and TEFLON® allow unprotected immersion of the measuring finger in up to 120 °C, up to 40 bars of pressure, abrasive and fast flowing media.

The acquired measuring values yield a comprehensive characterisation of the disperse system. A precision of < 1 % standard deviation with high sensitivity, the detection of broad distributions over 3 to 4 decades as well as the particle size measurement of highly concentrated paste-like slurries up to 70 % by volume are realised.

The energy level of the sound waves introduced into the medium is below 2.5 mW, so that even for sound-sensitive applications such as crystallisation processes or fragile dispersions there is no need to worry about any manipulation by the analysis itself.



View into OPUS measuring zone

OPUS in-line | on-line | off-line

with OPUS-AF | OPUS-BP | OPUS-FT

on-line & in-line | Production Control of Highest Perfection

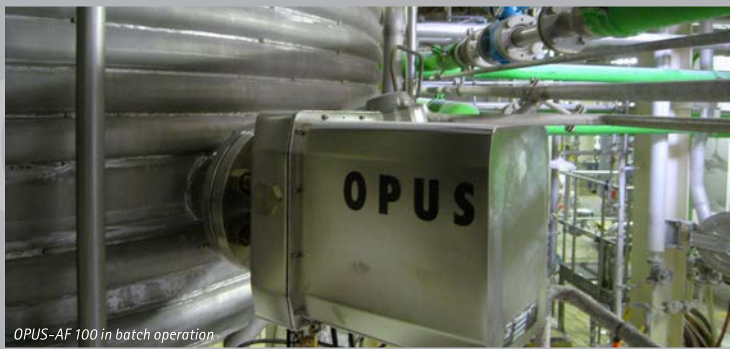
Real-time measurement in the product stream

OPUS was designed as a probe with the aim of submerging it directly in the product streams. This makes it ideal for ⊝ in-line and ⊝ on-line measurement of the particle size distribution in the process under harsh production conditions.

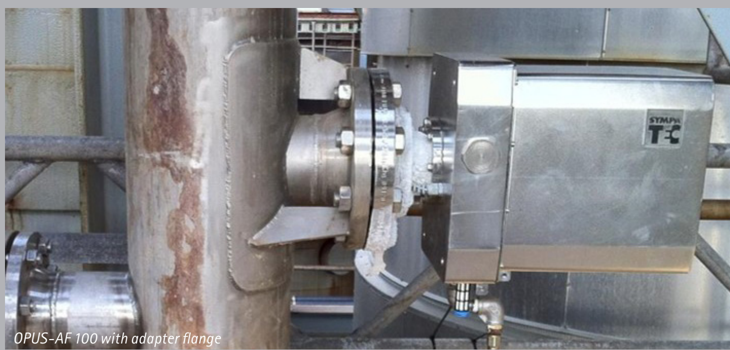
At the tip of the OPUS measuring finger the measuring zone with ultrasonic transmitter and opposing ultrasonic receiver is integrated. The distance between transmitter and receiver is controlled by the software and varies from 1 mm to 10 mm. This gap variation allows an ideal adaptation of the sensor to a wide variety of applications. For products with very high concentration, rather small measuring gap widths are used, while low concentrated samples are analysed with a larger measuring gap. To immerse the sensor in product streams which produce a strong wear, such as fast flowing ore slurries, the measuring finger is made of thick-walled stainless steel (DIN 1.4571 / SS316 / V4A).

The external housing of the sensor is home to the mechanics for the adaptation of the measuring gap and the electronics for the generation and reception of ultrasonic waves. This part of the sensor is protected by a sealed cover, which can be removed quickly in case of service, while the sensor finger remains in the product stream.

Trouble-free service and maintenance are thus possible even while the process is running.



OPUS-AF 100 in batch operation


OPUS-AF 100 with adapter flange


OPUS-BP with bypass adapter BP 50

Installation in pipe or vessel with OPUS-AF

The finger probe with a diameter of 89 mm and a custom-made length between 350 and 3,500 mm is integrated by means of a standard flange into pipes and reactors with diameters of DN 200 or larger. The measuring finger remains directly in the mass flow and transmits all measured data in real-time.

Installation in pipelines with OPUS-FT & OPUS-BP

For probe installation in small pipelines, flow-through (FT) and bypass (BP) adapters are available, which are mounted onto the measuring zone of the sensor finger. Product streams in 25 mm lines and smaller are pumped through the OPUS measuring zone and completely analysed without relevant flow mechanical disturbances occurring.

For larger pipeline diameter, pressure losses may arise. Therefore, so called bypass adapters are available for product lines above 25 mm, in which only a partial flow streams through the measuring gap, while the remaining partial flow circumscribes the measuring finger, without leaving the adapter.

The dust and splash water protected OPUS sensor meets the protection class IP65. For use of the probe in hazardous conditions and areas an OPUS-EX-version according to ⊝ ATEX (Ex II 2G EE p II T5) is available.

*⊝ During **in-line** measurement, the sample remains in the process line. The measurement occurs in real time under process conditions.*

*⊝ The **on-line** measurement is directly linked to the process. Sampling happens close to the process as quasi-continuous real-time measurement.*

*⊝ The **ATEX** directive 94/9/EG regulates the usage of devices and protection systems in explosion endangered areas.*

off-line | Comprehensive Support in Product Development

OPUS-FT in the laboratory

New products and processes are mainly developed in the laboratory. If the syntheses and process management succeed there, in a second step, a pilot plant will be built on a technical scale to ensure safe use in production. OPUS accompanies the product and process development in each of these phases. From the laboratory to the production, OPUS provides reliable and consistent principles for assessment.

In the development of OPUS, attention was paid to a wide range of applications in order to use this technology for both, a process measuring probe and a laboratory device for analysis of small quantities. Equipped with an FT adapter, a standby rack, a laboratory pump and a beaker, a sample circuit is created. With this set-up, volumes of typically about 300 ml to several litres are circulated and analysed in the laboratory or technical centre.

For non-segregating materials, a pumped circuit is not required. In this case sample volumes of less than 50 ml are completely sufficient for an OPUS analysis. Due to these flexible application options, the desired on-line measuring method can intensively be evaluated in advance in the laboratory before the sensor is integrated into the production process.

NIMBUS the compact lab device

With NIMBUS we take another step. It combines a compact, controllable sample circulation loop with the OPUS core components, making ultrasonic extinction suitable for use in the laboratory with incremental liquid samples of lower viscosity.

NIMBUS offers an easy and convenient way to analyse your manual samples from 300 to 1,000 ml. The system features a high chemical resistance to aggressive media. Like its "big brother" OPUS, NIMBUS covers a range from 0.1 to 3,000 microns.

Its compact, space-saving design, easy handling and fast filling and draining make NIMBUS an ideal laboratory device.

Whether you are considering OPUS-FT in the off-line version or NIMBUS – both instruments not only use the proven core technology but also apply the same intuitive and powerful PAQXOS software for operation, display and evaluation of results.

» Full sonication of optically impenetrable media in laboratory and process provides reliable knowledge about particle size and concentration. We rock your particles! «

OPUS - FT with standby rack

NIMBUS

Live is Life | Real-time Measurement

Process Monitoring and Control in Real-time

Crystallisation

The performance features of ultrasonic extinction open a wide range of different fields of application. In situations, where either sampling and dilution are not possible or dilution is not sufficient for optical analysis, the benefits of the acoustic measuring method come into effect. Crystallisation processes, in which crystalline products are deliberately dissolved and recrystallised in order to obtain an optimised particle shape or size, are a perfect showcase. Typical products of crystal suspensions are salts, such as ammonium sulphate, ammonium perchlorate or ammonium nitrate.

The production of crystalline materials usually starts from a saturated mother liquor by shifting the thermodynamic equilibrium of the solution while controlling it. The solubility of the crystals is either deflected by cooling the hot liquor down or by evaporation of the liquid phase in favour of crystal formation.

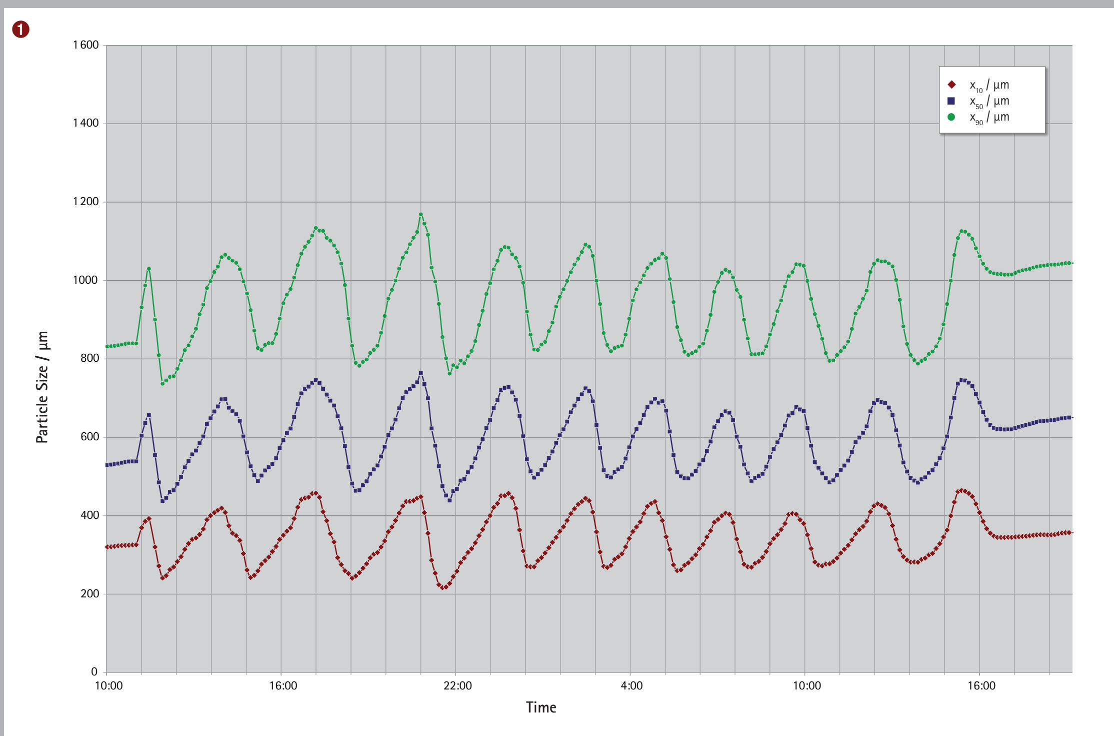
Manual sampling in crystallisation carries the risk of distorting the solution equilibrium and thus the sample condition. In addition, if a sample dilution is necessary, the thermodynamic system collapses completely.

OPUS succeeds in analysing the crystals in the original state without sampling and dilution. However, besides pure sample handling, the frequency of crystallisation analysis often plays an important role. Particularly, continuous crystallisation processes are subject to a strong process dynamic due to the change of supersaturation and subsaturation.

❶ The trend diagram clearly shows this process dynamic by the saw-tooth fluctuations in the particle size of sodium chloride. In subcritical conditions, the crystals mature to large particles until a constant saturation degree of the crystal solution is reached and the free surface becomes too small for further growth. A spontaneous crystallisation of the finest primary crystals occurs and the particle size values suddenly drop. The high analysis frequency of the OPUS probe of approx. 1,000 measurements per day guarantees a reliable monitoring of the current process situation at any time and allows targeted pinpoint interventions, e.g. with ⊙ seed crystals.

Salt crystals

❶



⊙ **Seed crystals** *are fine primary crystals and used as the starting crystal for the growth of larger crystals. In order to prevent excessive variations in the quality of crystalline products, in practice seed crystals are introduced into the crystal suspension at appropriate times. The prerequisite for recording suitable vaccination moments is a real-time observation of the present crystal size distribution.*

Extremely Resistant Even with Abrasive Media

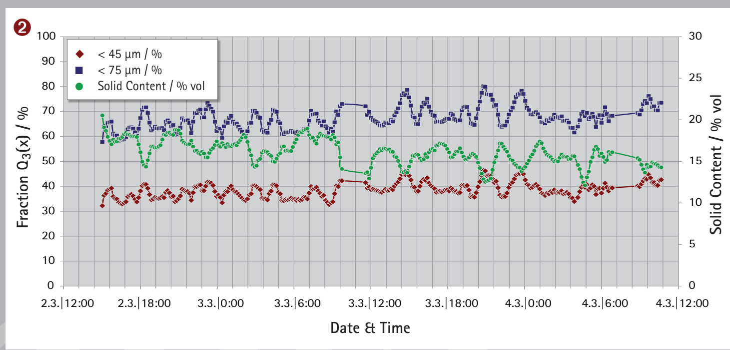
Grinding

The ultrasonic extinction is an effective and simple method of measurement for particle size determination during wet and micro-fine grinding. It finds a wide range of applications in the analysis of ore slurries, mineral suspensions, metal pigments and battery compounds.

Particularly for ore dressing applications, OPUS fully develops its strengths in continuous operation. The metal extraction begins with the comminution of the ore in crushers, autogenous mills, ball or rod mills. The ore slurry is transferred into ⊕ flotation cells where single particles are classified into mineral and burden material. The addition of chemical additives is just as crucial for the selection, as the compliance of the solids concentration and the particle size of about 1 to 200 microns. Despite the optically impenetrable ore slurry, the measurement of grain size with OPUS succeeds in full suspension flow without sample conditioning. The absence of moving or damageable components provides the required resilience to sharp-edged and abrasive particles.

Using MULTIPLEXER, up to 4 suspension streams are controlled and linked to the sensor in flow optimized conditions. Lower flow velocities through OPUS not only increase operational safety and availability but also minimise the maintenance requirements. The design of MULTIPLEXER guarantees long operation life and maximum sample throughput specifically for the mineral processing industry.

Ore slurry in flotation cell



OPUS with MULTIPLEXER

With statistically relevant sample quantities of approx. 50 to 150 litres per measurement, undiluted mineral slurry can be analysed in real-time and in 24/7 operation. This concept is rounded off with primary samplers, which can be individually adapted to specific process requirements.

❷ The trend diagram shows an on-line grinding monitoring of copper ore over approx. 2 days with the particle passing values Q_3 (x) for 45 µm and 75 µm as well as the solid material volume concentration. As a consequence of a mill overload, the typical inverse correlation of particle size values to the solid content is clearly displayed. If too much rock material is fed into the grinding chamber per unit time, just a little more material leaves the grinding process overall, but also with too coarse particles, since the mill's performance is usually kept constant. In order to grant a smooth process operation, a constant control of the mill feed by simultaneous monitoring of particle size and solid concentration is necessary.

OPUS always delivers precise and sensitive real-time analysis of the grinding material flow in the original condition, whereas a time-consuming laboratory analysis of discrete samples becomes unnecessary. No matter wether the OPUS sensor is integrated into the main process stream or in to a bypass line. In either case, a statistically relevant volume of suspension is captured in each analysis delivering meaningful and reliable measuring results.

⊕ **Flotation** *is a physico-chemical separation process for fine-grained solids based on the different surface wettability of the particles. If the ore is too coarse, the valuable material can no longer be separated from the deaf rock.*

Keep an Ear on Your Process

Analyses in Opaque Liquids in Original Conditions
No Dilution required

Classifications and mixtures

During the production of high-quality finished products, it is often not sufficient to pay attention only to the generation of targeted particle sizes. To achieve the desired product quality, a selected addition or separation of specific fractions or components can also be essential, e.g. in the production and processing of drilling muds, oil sands, foundry sand or coke.

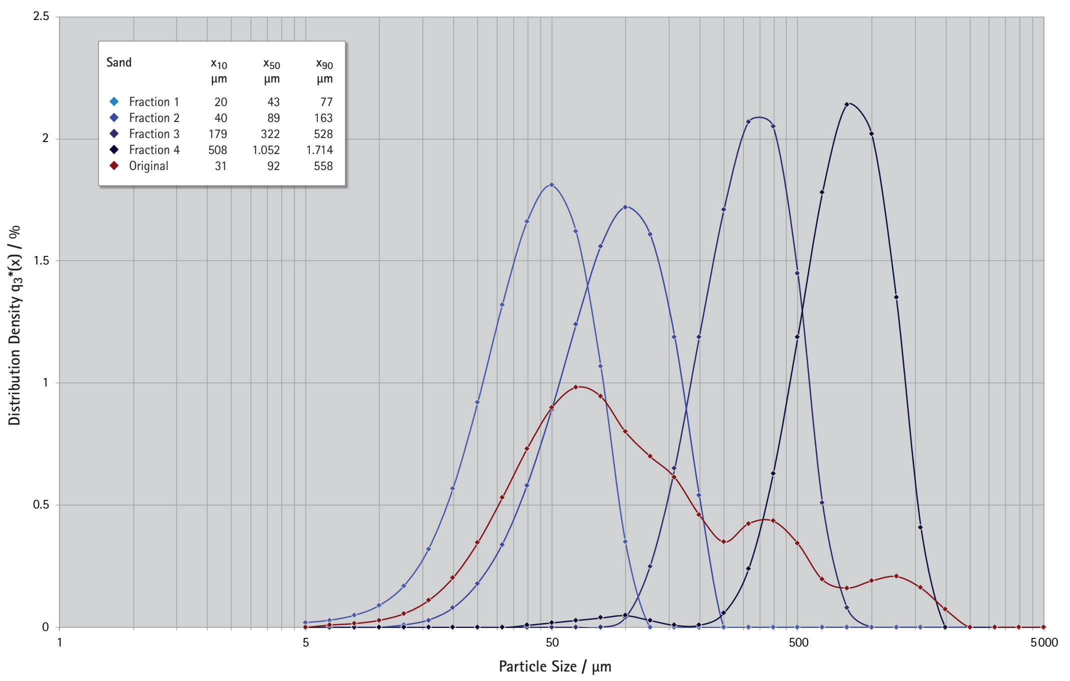
A typical example of a multi-phase separation is the oil sand processing. Oil sands are a very tough mixture and essentially consist of quartz sand, water, minerals and oil. When separating the mineral constituents from the brown-black crude oil, both the particle size and the solids concentration play an essential role. An optical analysis to monitor the separation success is not constructive due to the opaque properties of the crude oil. For ultrasonic technology, however, the light-impermeable crude oil matrix does not constitute a problem.

Many processes involve wet classification using hydrocyclones or wet screens to fractionate a widely distributed feed material into separate product streams in order to blend these fraction to a well defined particle size distribution. This holds true e.g. for sintered materials, such as refractory bricks, smelting anodes or foundry sands. For the production of solid casting moulds and cores, aggregate materials are needed which are characterised by a high packing density and low permeability. The demanding specifications regarding quality and precision of sintered materials require an accurate knowledge and control of sand grain size distributions even in their wet dispersed state.

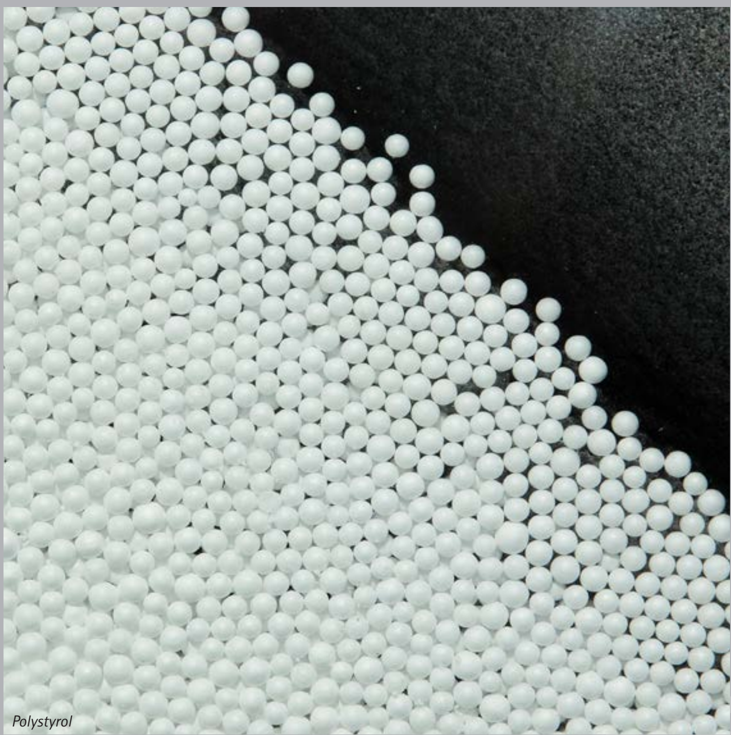
❶ The particle size distribution chart presents the classification of a foundry sand mixture. The presence of fine minerals such as silt and clay in the sand suspensions leads to an opacity which is impenetrable to light-optical methods. If coarse sand particles (fraction 4) are present at the same time, sufficient dilution to obtain optical transparency would lead to inadequate representation of the coarse particle fraction. OPUS analyses the sand mixture in its original state and considers all particle size classes (blue curves) according to their actual volume fractions.


Different fractions of sand

❶



Effective Early Warning System for Process Upsets



Polystyrol

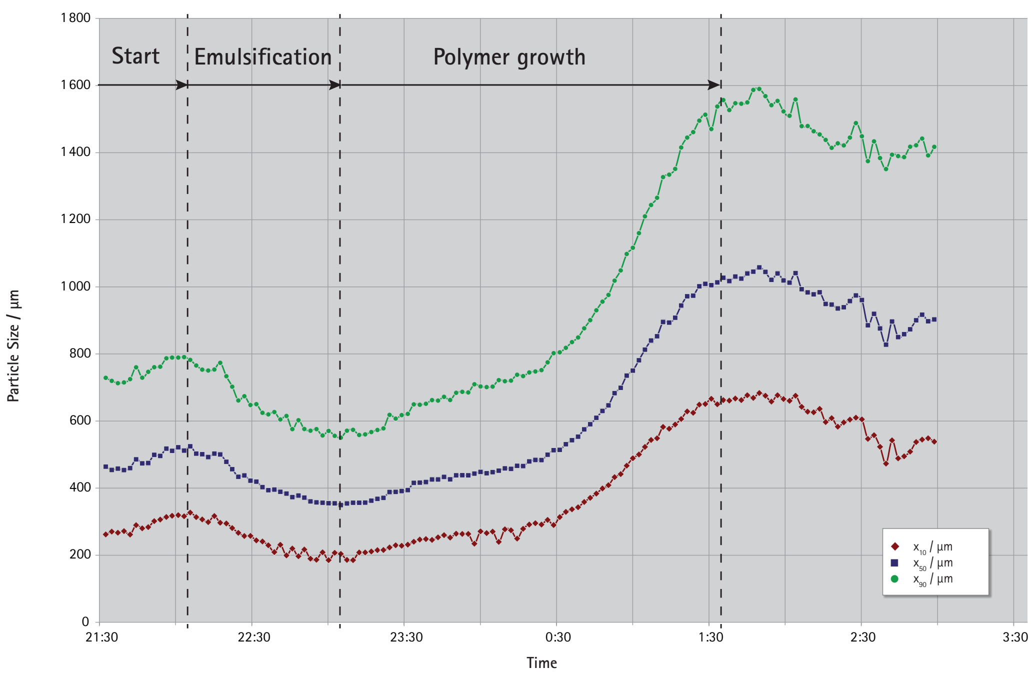
Emulsifications

The production of cosmetic and pharmaceutical lotions and creams, but also industrial lubricants and coolants, is usually achieved in a homogenisation process for the involved liquid phases. Long term stability of the emulsion in addition to the product performance is often dictated by the droplet size. It avoids segregation during storage ensuring a longer shelf life.

In the field of polymer chemistry, particle and droplet sizes are also analysed for unstable emulsions. ❷ The trend graph shows the course of ⊖ polymerisation from the start, through the emulsion phase to the polymer growth, right up to the end of the reaction when the target size of the EPS beads is reached.

During the production of polystyrene coarsely-dispersed emulsions of several 100 µm are kept in meta-stable conditions via permanent input of mechanical shear forces. At the same time, the polymerisation of the disperse phase from liquid droplets to solid particles takes place. In the transition zone, drop collisions due to increasing surface adhesion lead to ⊖ coalescence and thus to a drop growth of approx. 400 µm to more than 1,000 µm in x_{50} value, which is tracked by OPUS with a chronological resolution of 20 to 30 measurements per hour. If the desired particle size of about 1 mm is reached, the particles are coated by adding suitable release agents and prevented from further growth before they are thermally hardened and separated from their carrier liquid.

❷



Both types of emulsifying present difficult to overcome hurdles for optical measurement methods. In the first case of the stable micro-emulsions, an optical analysis means a sample dilution of several orders of magnitude. At improper process conditions the emulsions break up and the actual particle size to be detected is destroyed. In the second example, sampling is absolutely impossible because the mixture of phases immediately separates with the elimination of mechanical stress. OPUS requires neither sampling nor dilution to reliably detect and monitor the dispersity degree of the emulsion. Targeted use of the inherent strengths of ultrasound extinction manages to fill in the gaps in the process control safely and install an effective early warning system for process upsets.

⊖ **Polymerisation** *is a chain reaction in which simple starting materials (monomers) connect via reactive double bonds to long chains.*

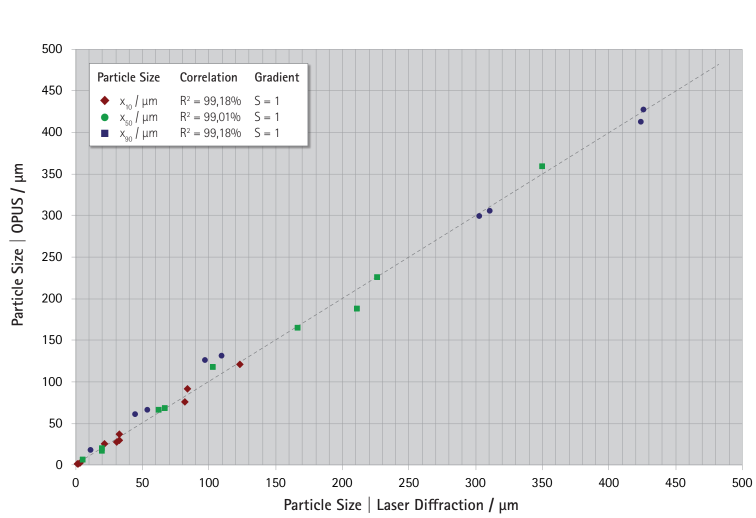
⊖ **Coalescence** *is the confluence and fusing of disperse liquid droplets into a unitary phase.*

PAQXOS Software

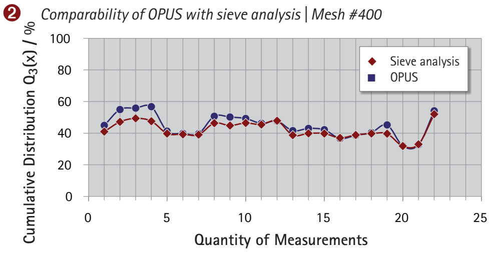
Process Interfaces

Control | Communicate | Automate

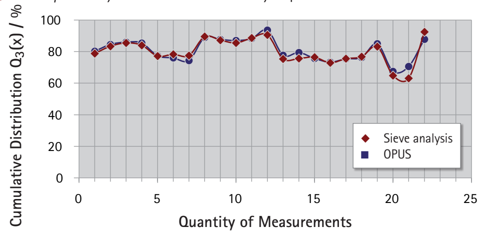
❶ *Comparability of Ultrasonic Extinction with OPUS and Laser Diffraction with HELOS*



❷ *Comparability of OPUS with sieve analysis | Mesh #400*



❸ *Comparability of OPUS with sieve analysis | Mesh #70*



PAQXOS combines our collected metrological expertise in a user-friendly application software implemented as network-ready 64-bit software for Windows® environments. A step-by-step assistant also guides users without expert knowledge quickly to first measurement success.

After each measurement the raw data are automatically stored in the database. As a result, a subsequent change of the evaluation parameters and the subsequent evaluation without renewed sample analysis is possible.

PAQXOS provides graphical presentations, tables, and reports whose formats are selected from a wide range of predefined templates or may be user-defined. Easy drag & drop results from the measurement database are simply displayed or measurement parameters taken over for the next measurement. Data browser and filter functions facilitate the presentation of already existing analysis data. Using the operator-friendly user-interface, all functions are conveniently activated and intuitive solutions for realisation of reliable measurements are created.

With its ➔ versatility, PAQXOS is ideal for flexible user applications in production and research. Instruments and databases are supported in a network environment, thus allowing remote access, control and data sharing.

The OPUS system can be flexibly integrated by means of common communication standards such as Modbus® RTU, Modbus® TCP, Profibus®, OPC, TCP/IP, FTP, MQTT or analogue PLC signals in a control system to get connected to remote control or autonomous measurements. The real-time information about the particle size distribution and the concentration are made available to the process control system for the process monitoring or control.

The software also allows FDA-compliant use in pharmaceutical environments and has all security mechanisms, access controls and authentications required by Directive 21 CFR Part 11.

The measurements achieved with OPUS show outstanding comparability with established analytical methods such as laser diffraction, sieve analysis or sedimentation devices. In chart ❶ the particle distribution values x_{10}, x_{50} and x_{90} of 10 sand samples are analysed with laser diffraction and ultrasonic extinction. The deviations from the ideal, represented as a diagonal, amount to less than 1 %. Comparing ultrasonic extinction with sieve analysis data, 22 individual samples ❷ with a 400 mesh (38 µm) and ❸ a 70 mesh (212 µm), the values of both methods are close together.

A conversion of the product or production specifications is usually not required. Where conventional measurement methods are not or only insufficiently applicable due to their limitations, OPUS sets new standards.

➔ *Through an integrated* scripting environment, *sophisticated routines can be programmed to make repeat measurement processes efficient and reproducible.*

Furthermore, measurement parameters can be specified as obligatory measurement instructions, so called Standard Operation Procedures | SOPs.

Brand Home of Sympatec's Premium Instruments

Development of Innovative Methods for Particulate Systems Characterization

Laser Diffraction | Image Analysis | Ultrasonic Extinction | PCCS

Perspective

"A classic is timeless and at the same time ahead of its time."

The variety of disperse products requires innovative and sustainable technologies to master the challenges in today's research, development, quality and production control.

With dry dispersion we have introduced product orientation and adaptation to laser diffraction.

The HELOS sensor family and a great range of dispersing units – spearheaded by RODOS – offer you premium performance. Our laser diffraction instruments allow for a significant extension of your particle knowledge concerning size and size distributions.

New questions and desires inevitably arise with unbowed progress. Power of innovation consequently remains key to future developments.

Today, if we encounter application limits of laser diffraction e.g., in suspensions of high optical concentration, we offer efficient solutions with ultrasonic extinction (OPUS).

If particle shape becomes of interest, we provide a great spectrum of powerful solutions with high-speed dynamic image analysis (QICPIC family). Now even sophisticated fibre analysis is amongst the range of multifaceted particle shape aspects.

And in case particles predominantly belong to the nanometre range, we have brought the unique Photon Cross-Correlation Spectroscopy (PCCS) to market with Sympatec's NANOPHOX.

By nature, we also keep an eye on the production of disperse systems when developing methods of particle characterisation. Hence, you may also trustfully address us in case process control becomes an issue. Laser diffraction with MYTOS, ultrasonic extinction with OPUS and dynamic image analysis with PICTOS are hundredfold approved process applications from Sympatec.

Designed with a consistent technological basis, our in-, on-, at-line systems reliably deliver results that are perfectly comparable to those of our laboratory instruments – most accurate, reproducible and at the shortest measuring times.

As "Particle People" we originate from the powder technology field. This is why we have a natural approach to process engineering and the production of disperse systems. The collective particle expertise of our physicists, mathematicians, computer scientists, engineers, electronic and mechanic technicians is built into our instruments.

Your particles in the best of hands with us.

Particle Measurement and Know-how from Pulverhaus

Several Thousand Installations
At Particle Professionals Worldwide

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