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process under control

In-line tracking of reaction progress through the speed of sound

Many material parameters that are crucial for optimal process control are still measured using indirect variables such as temperature, pressure or flow. This often involves decisive changes information lost or are not available in a timely manner. Determining and processing the absolute speed of sound represents an interesting alternative to being able to determine material parameters such as concentration, density or the course of the reaction directly in the process.

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Almost all substances are subject to change
technical processes converting
changes in the density and concentration of the substances involved. However, the inline determination of the concentration usually encounters difficulties, since almost all available measurement methods require direct contact of the sensor with the usually harsh and aggressive process conditions. For this reason, known measuring methods such as determining the pH value or conductivity, optical principles such as determining the refractive index, turbidity or color, and density measuring devices based on oscillating tube systems are usually only suitable to a limited extent for monitoring the course of reactions.

Conventional measurement methods are inadequate

Figure 1 shows typical application specifications for these measurement methods. For process control le in demanding process engineering Processes such as polymerisation and crystallisation are covered by most measurement methods mainly due to limited temperature and pressure ranges or can due to other boundary conditions such as sensitivity to contamination only with a significantly reduced service life or a unreasonably high maintenance costs are used. Classic principles such as pressure or temperature measurement are therefore often used as an alternative solution. However, these measured variables only provide direct, delayed or falsified process information.

The sound velocity measurement, on the other hand, is almost the only method predestined for use under harsh process conditions due to its wide operating temperature and pressure range and its unlimited service life. Figure 2 shows a sensor for determining the speed of sound and an associated evaluation unit. The fork-shaped sensor is installed directly in the container or in the pipeline and digitally transmits the measurement data for the sound velocity and the process temperature to the evaluation unit.

The evaluation unit takes over the Be Calculation of customer-specific variables such as concentration, material conversion, degree of polymerisation or supersaturation. The special advantages of this measuring method are obvious:

- purely inline solution, installation directly in pipelines or tanks;
- regardless of color, conductivity or transparency of the medium;
- maintenance
- free; robust against mechanical vibrations and pressure surges; full metal
- finish, none gaskets or glued joints;
- chemically resistant due to special materials.

The installation of the sensors at the measuring point does not require any specific adapters or retractable fittings.

2: *ultrasonic analyzer Liquisonic, consisting of sensor and evaluation unit*

1: Typical application specifications for the various measurement methods

measurement method	Operating temperature	Operating pressure	Service life	remark
refractive index	150ÿ	25 bars	restricted	sensitive to pollution
conductivity	160°C	40 bars	Can only be used	without restrictions for inorganic material systems
PH value	130ºC	15 bars	restricted	ongoing maintenance through calibration
density	125ºC	50 bars	restricted	Bypass required, sensitive to dirt
speed of sound	200ºC	250 bars	unrestricted	resistance to corrosion special material

Macromolecules, polymers and plastics are ubiquitous products and must meet the highest standards. The processes developed for production often take place under high process pressures and temperatures. Due to these boundary conditions, the monitoring and control of these processes must meet the highest standards.

Avoid bad batches

In addition to the safety-related relevance targeted process control is required to maintain the desired properties of the polymer. For these tasks, sound velocity measurement systems are successfully used in various polymerisation processes: solution polymerisation of

- butadiene; emulsion polymerization of
- vinyl acetate,

butadiene styrene, vinyl chloride or acrylates;

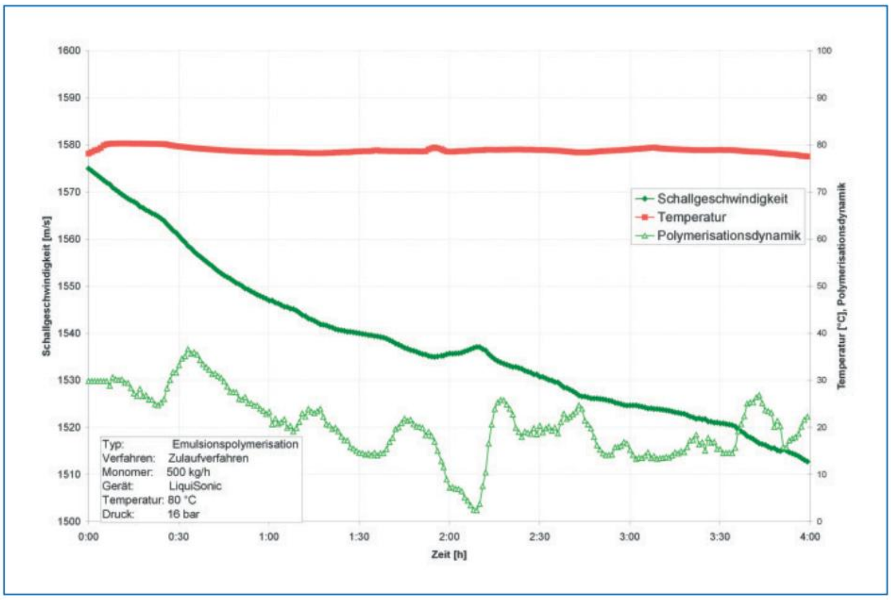
- suspension polymerization of styrene or vinyl chloride;
- Polycondensation of urea and formaldehyde as well as phenol and formaldehyde.

Here, the property is used that the speed of sound between the monomer and the polymer changes significantly as a result of the chain length and the degree of branching and crosslinking. The evaluation unit (controller) uses the measured speed of sound to calculate the variables required for process control, such as the degree of

- polymerisation (conversion),
- polymerisation dynamics (speed) and the
- concentration of the monomer or Po lymers.

A process that is frequently encountered is emulsion polymerization in the feed process. This batch process is generally used in agitator tanks carried out and is used, for example, for the production of paints, adhesives and lei men. In such a process, a sound velocity meter is used directly in the batch container. Figure 3 shows the trends in temperature, sound velocity and polymerisation dynamics during a complete batch. In the process shown, it came about two hours after the reaction beginning of a polymerisation disturbance, which could be clearly detected by the reduction in the calculated polymerisation dynamics. The temperature remained inconspicuous during this time and did not allow any conclusions to be drawn about this process disturbance. The fault itself resulted from an error in the monomer dosage. If this fault had not been recognized and rectified, the entire end product would have had to be discarded due to insufficient quality.

Similarly, a Ver delay in the start of polymerisation, which leads to an accumulation of monomers and thus poses a safety risk. The use of sound velocity measurement technology enables both the reaction monitoring of the process and thus the constant control of the product quality as well as the detection of safety-critical conditions.

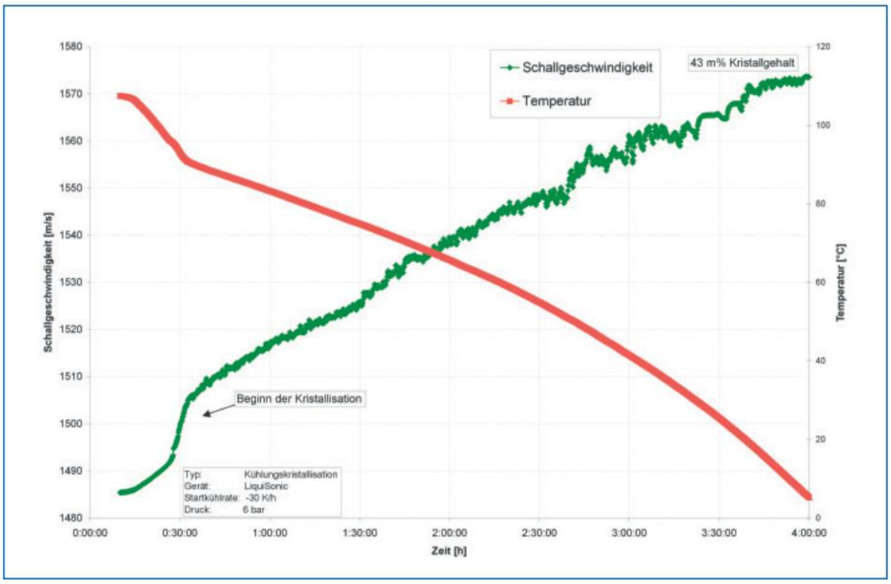


3: Trend of temperature, sound velocity and polymerisation dynamics of a feed polymerisation

defined crystallization result

Crystallization is one of the basic operations in thermal process engineering with the aim of creating a defined solid phase. As a thermal process for separating, cleaning and concentrating substances, it is of great importance in the food (sugar, fruit juice), pharmaceutical (tablet active ingredients, vitamins) and chemical industry (fiber raw materials, fertilizers, salts). Depending on the specific requirements, the solid phase is formed from a solution or a melt by evaporation or cooling crystallization.

Figure 4 shows the course of the process during the discontinuous cooling crystallization of an inorganic substance in water. The aim of the inline measurement is to monitor the



4: Trend of temperature and sound velocity of a cooling crystallization

Crystallization process with particle sizes of around 50 to 100 µm. Setting defined product qualities requires inoculation with crystals at defined degrees of supersaturation. No vaccination was carried out before the use of the sound velocity measurement technique. Crystallization started spontaneously. The onset of crystallization could le only be observed visually.

In the case of cooling crystallization, however, the average crystal size distribution is largely determined by supersaturation and the cooling rate at the start of crystallization. Inoculation enables a uniform crystal size distribution and thus better product quality.

The controller calculates from the sound speed directly determines the degree of supersaturation and thus enables inoculation at reproducible and defined process states. In addition, the system can determine the following parameters of a crystallization process:

- Supersaturation or temperature difference to the saturation temperature,
- crystal
- content,
- temperature, cooling
- rate and concentration of the mother liquor.

With the sound velocity measurement, a measurement option is available to ensure a reproducible course of the reaction in demanding technical processes. This enables a high degree of process reliability and thus the defined setting of reproducible end product qualities.

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COMPACT

speed of sound possible Inline process tracking

The speed of sound of ultrasound in liquids depends on the density and the adiabatic compressibility. Since both the density and the compressibility are temperature-dependent, material-specific constants, the sound propagates in every material at a characteristic speed that is temperature-dependent. The speed of sound is less than 700 m/s for gases, in the range from 700 to 2,500 m/s for liquids and up to 7,000 m/s for solids. The

The speed of sound is determined by the structure of the substance, ie by groups of atoms and molecules, isomerism or chain lengths. This connection thus offers the possibility of characterizing substances by means of ultrasound.