



Polymerization monitoring

- Inline analytical technology for: monomer
 - concentration
 - polymer concentration
 - degree of polymerization
 - polymerization break off

Increasing (

With high

Robust, ac







quality, saving resources: LiquiSonic[®].

-value, innovative sensor technology.

curate, **user-friendly.**

LiquiSonic[®] is an inline analytical system, that detects the concentration in a liquid directly in the process and without any time lag. The device is based on the accurate measurement of sonic velocity and temperature and enables so the monitoring of processes and complex reactions.

Benefits for the user include:

- optimal plant control through online information about the state of the process
- maximum efficiency of processes
- increasing of the product quality
- reduction of costs for laboratory measurements
- saving of energy and material costs
- · optimal process utilization
- reproducible process management with proprietary "fingerprint" technology

Using the latest digital signal processing technology ensures a highly accurate and fail-safe measurement of the absolute sonic velocity and the concentration. In addition, integrated temperature sensors, a sophisticated sensor design and the know-how resulting from numerous series of measurements and many applications guarantee a high reliability of the system with a long lifetime.

Advantages of the measuring method are:

- absolute sonic velocity as a well-defined and retraceable physical value
- independent of color, conductivity and transparency of the process liquid
- installation directly into pipelines as well as tanks and vessels
- robust and completely metallic sensor design without gaskets or moving parts
- · maintenance-free
- · corrosion resistance by using special material
- · use at temperatures up to 200 °C
- high, drift-free measuring accuracy even with high concentrations of gas bubbles
- · connection of up to four sensors per controller
- forwarding of measuring results through fieldbus (Profibus DP, Modbus), analogue outputs, serially or Ethernet

Inline process analysis

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Fundamentals of polymerization



1.1 Introduction

In connection with the need to closely monitor and control processes, the capability to determine conversion in chemical reactions in general and in particular in polymerization reactions is of outstanding significance.

Just like concentration measurements, conversion measurements are becoming more and more important in all branches of industry on account of their impact on the efficiency of processes, their potential for material and energy savings, quality improvements as well as for environmental reasons.

For measuring concentrations and conversion, a number of measuring methods have been developed, including methods based on density measurement, refraction index measurement, conductivity measurement, the measurement of color, turbidity and viscosity. All of these methods are characterized by specific physical and technological limitations.

It has been known for quite some time that concentrations can be determined by measuring the sonic velocity, and this method has become the standard measuring technique in recent years.

1.2 Physical fundamentals

The propagation velocity v of ultrasonic waves in liquids is dependent on their density and adiabatic compressibility in accordance with the following relationship:

$$v^2 = \frac{1}{\rho \cdot \beta_{ad}}$$

 $\begin{array}{l} \mathsf{v} &= \text{sonic velocity} \\ \rho &= \text{density} \\ \beta_{\mathsf{ad}} &= \text{adiabatic compressibility} \end{array}$

The fact that the compressibility is the determining variable for the sonic velocity causes that, as the sonic velocity increases, density and compressibility may show a differing behavior. This, in turn, causes that even if there are only minor differences in density or none at all, large differences in the sonic velocity may occur. It very rarely happens that the reverse case takes place.

The sonic velocity is determined by the structure of the material concerned, i.e. by groups of atoms and molecules, isomerism or chain lengths. This correlation, thus, allows to characterize materials with the help of ultrasonic.

The table below shows the sonic velocity v of a few selected monomers and polymers at 20 °C.

As concerns monomer and polymer systems, it generally applies that the differences existing in the sonic velocity between monomers and polymers are primarily determined by the chain length and the extent of their branching and cross-linking. The table already shows significantly that the differences existing between monomers and polymers are extremely large and, thus in some extent, also between start and end of polymerization reaction.

Monomer	v [m/s]	Polymer	v [m/s]
styrene	1,354	polystyrene solid	2,330
vinyl chloride	897	polyvinyl chloride solid	2,260
vinyl acetate	1,150	polyvinyl acetate, dispersion 50 wt%	1,940
butyl acrylate	1,233	polybutyl acrylate, dispersion 50 wt%	1,375
butadiene	961	polybutadiene, solution 20 wt%	1,373



Processes

Depending on the reaction mechanism the polymerizations are divided into:

- · solvent polymerization
- emulsion polymerization
- suspension polymerization
- · polycondensation

Depending on the number of copolymers and the product changing additives, the change of sonic velocity shows a characteristic course. Typically, the sonic velocity of all components involved is determined depending on the temperature to be able to compensate it later on. Then it is possible to derive the course of reaction from the time course of the sonic velocity and to calculate the materials conversion.

The following description explains this as an example for the emulsion polymerization of styrene butadiene latex. The determination of parameters like concentration, degree of polymerization etc. occurs analog in other polymerization types.

Emulsion polymerization of styrene-butadiene-latex

The individual components and lattices were examined for a butadiene-styrene emulsion polymerization reaction system.

In the diagram below it can be seen, that the sonic velocity of the monomers clearly differs from that of the polymers.



Sonic velocity of components of butadiene-styrene polymerization

The sonic velocity and the concentration are linked to each other. Furthermore, the degree of polymerization reflecting the share of polymer in monomer correlates with the concentration. With ultrasonic measurement systems, it is possible to determine the concentration and the degree of polymerization. The following diagram describes this relationship during a polymerization of butadiene-styrene.

In case of emulsion polymerization of butadiene and styrene, the degree of polymerization can be determined with an accuracy of 0.1 %.

Variation of sonic velocity, concentration and degree of polymerization



Applications



Our knowledge in the field of polymerization acquired by numerous applications at the customer and by in-house technology is the result of varied experiences over 20 years. This knowledge is integrated into new projects by always handling customer data as confidential.

In polymerizations, not only polymers are the focus of monitoring, but also a number of monomers, additives and solvents as well as recovery processes.

SensoTech offers the following secondary literature to different production processes:

- · optimization of polyamide production
- · optimization of polyurethane production
- styrene-butadiene-latex (SBR) production safe and efficient

The following applications have been examined so far:

- styrene-butadiene-latex
- phenol-formaldehyde resin
- · polymethyl-meta-acrylate PMMA
- polyvinyl acetate PVA
- polyvinyl chloride PVC
- · polyamide PA
- polyvinylidenchloride PVdC
- epoxy resin
- · polystyrene PS
- · polycarbonate PC
- · polyester PE
- · polyethylene
- · formaldehyde urea resin
- · elastane
- · aldol in acetaldehyde
- polyurethane PU
- · polysiloxane
- · isoprene rubber IR
- · methyl silicone resin
- · slicone acrylate
- · potassium methyl siliconate
- · silicone resin
- · polysulfide polymer
- · paraphenylen-terephtalamide PPTA
- hindered Amine Light Stabilizers HALS
- · methacrylamide MAA
- · customer-specific compositions

4 LiquiSonic[®] 50



With LiquiSonic[®] 50 it is possible to determine the degree of polymerization and the respective concentration. The system consists of one sensor and one controller.

Additionally, the reaction can be monitored, because in reaction processes always following the same basic principle with almost identical initial values and process parameters, a typical path (fingerprint) for the sonic velocity or concentration is built up over time.

For monitoring purposes of such reaction processes a band can be defined for the desired magnitude of the measurement value. This band is described by two envelopes, which are stored in the controller.

Generally, the current concentrations or sonic velocities should be stored in the defined band. This will be visualized for the user in fingerprint menu of the controller. The current concentration or sonic velocity is displayed as a red cross blinking if activated. The values recorded by that time are displayed as a blue curve.

All relevant measuring values being detected during the process are stored as data and graphics in the controller. Furthermore, these information are provided the process control system via diverse analog outputs or field bus interface.

The LiquiSonic[®] 50 analyzer enables to optimize the process flow, to save raw materials and energy, to monitor the process and to ensure the product quality.



Fingerprint in LiquiSonic® 50 controller

The fingerprint view of the controller summarizes all necessary information for the user:

- · minimum / maximum values of y-axis,
- · localization of the current fluid concentration,
- · elapsed batch time,
- · deviation to the ideal curve in %,
- · current fluid temperature.

The yellow curve in the above figure is the ideal line showing the ideal reaction path; this curve is calculated from the mean values of the upper (+100 %) and lower (-100 %) envelopes (red lines).





Enthusiasm for technical progress is the driving force behind our company as we seek to shape the market of tomorrow. As our customer you are at the centre of all our efforts and we are committed to serving you with maximum efficiency.

We work closely with you to develop innovative solutions for your measurement challenges and individual system requirements. The growing complexity of application-specific requirements means it is essential to have an understanding of the relationships and interactions involved.



Creative research is another pillar of our company. The specialists in our research and development team provide valuable new ways to optimise product attributes, such as testing new types of sensor designs and materials or the sophisticated functionality of electronics, hardware and software components.

Our SensoTech quality management also only accepts the best production performance. We have been certified according to ISO 9001 since 1995. All device components pass various tests in different stages of production. The systems have all gone through an internal burn-in procedure. Our maxim: maximum functionality, resilience and safety.

This is only possible due to our employee's efforts and quality awareness. Their expert knowledge and motivation form the basis of our success. Together we strive to reach a level of excellence that is second to none, with a passion and conviction in our work. Customer care is very important to us and is based on partnerships and trust built up over time. As our systems are maintenance free, we can concentrate on providing a good service to you and support you with professional advice, in-house installation and customer training.

Within the concept stage we analyse the conditions of your situation on site and carry out test measurements where required. Our measuring systems are able to achieve high levels of precision and reliability even under the most difficult conditions. We remain at your service even after installation and can quickly respond to any queries thanks to remote access options adapted to your needs.



In the course of our international collaboration we have built up a globally networked team for our customers in order to provide advice and support in different countries. We value effective knowledge and qualification management. Our numerous international representatives in the important geographical markets of the world are able to refer to the expert knowledge within the company and constantly update their own knowledge by taking part in application and practice-oriented advanced training programmes.

Customer proximity around the globe: an important element of our success worldwide, along with our broad industry experience.



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osolute spirit of development.

SensoTech is a provider of systems for the analysis and optimization of process liquids. Since our establishment in 1990, we have developed into a leading supplier of process analyzers for the inline measurement of the concentration and density of liquids. Our analytical systems set benchmarks that are used globally.

Manufactured in Germany, the main principle of our innovative systems is to measure ultrasonic velocity and density in continuous processes. We have perfected this method into an extremely precise and remarkably user-friendly sensor technology. As well as the measurement of concentration and density, typical applications include phase interface detection or the monitoring of complex reactions such as polymerization and crystallization.

Our LiquiSonic[®] measurement and analysis systems ensure optimal product quality and maximum plant safety. Thanks to their efficient use of resources they also help to reduce costs and are deployed in a wide variety of industries such as chemical and pharmaceutical, steel, food technology, machinery and plant engineering, car manufacturing and more. It is our goal to ensure that you maximize the potential of your manufacturing facilities at all times. SensoTech systems provide highly accurate and reproducible measuring results even under difficult process conditions. Inline analysis eliminates safety-critical manual sampling and is immediately available for your automation system. All parameters can also be adjusted with high-performance configuration tools, so that you can react quickly and easily to process fluctuations.

We provide excellent and proven technology to help improve your production processes, and we take a sophisticated and often novel approach to finding solutions. In your industry, for your applications – no matter how specific the requirements are. When it comes to process analysis, we set the standards.





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In liquids, we set the measure.