SAM-Spec

CHAUVIN ARNOUX INDATECH

Chemicals



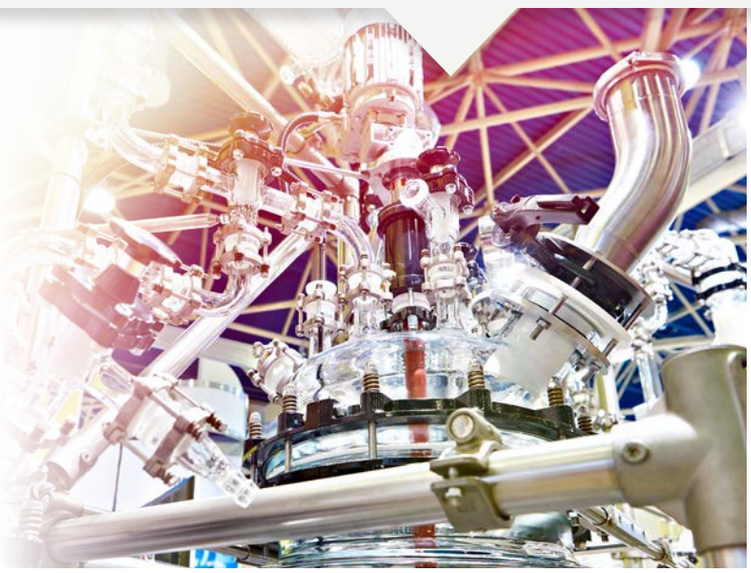
In-line monitoring of silica precipitation reaction using multi-angle spectroscopy

The production of silica is a high-stakes industrial application. The physical properties of silica, such as the size, specific surface and porosity, make it a high added-value product which can be used for a large number of applications. The precipitation reaction phase is crucial in the silica production line because the main characteristics of the end-product depend on it. In-line monitoring of this reaction and understanding of its mechanisms represent a critical step in the process which we will seek to analyze.

Objective: qualitative monitoring of the size of precipitated silica particles

Use of the SAM-SPEC technology helps to improve understanding of the scattering phenomena occurring during the silica precipitation reaction.

This makes it possible to monitor the evolution of the particle size in the reaction medium and to determine the freezing point.



SFX-IM: the multi-angle solution for monitoring your critical parameters in-line

Indatech offers a unique inspection solution capable of checking both the physical and chemical characteristics of liquids.

The SFX-IM probe can be used for quick, non-destructive, in-line analyses.

It is capable of:

- measuring with up to 7 different angles in transmission and/or reflection mode
- providing information on the density and mean particle size
- performing very fast simultaneous acquisition of the measurement points (< 10 ms)

How can you measure the precipitation reaction in-line?

The silica precipitation reaction always involves neutralization of an aqueous sodium silicate solution using sulfuric acid.

All the syntheses were performed in a pilot laboratory in an agitated 25-liter reactor. An aqueous sodium sulfate solution was prepared and placed in the bottom of the reactor vessel before adding the reagents.

The reagents were then added alternately according to the protocol defined by the industrial company.

The SFX-IM probe was installed vertically on the fitting of a fast loop made-to-measure for the tests (cf. figure 01). The probe is connected to a Hy-Ternity spectrometer which is used to acquire the near-infrared spectra from all the angles simultaneously in the spectral range from 900 to 1650 nm.

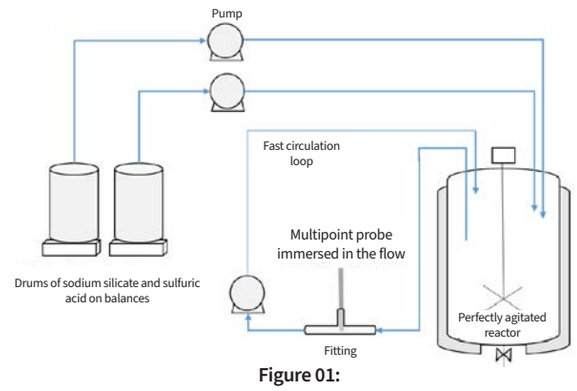


Figure 01:
Diagram of the experimental setup for in-line monitoring of the syntheses

Results and discussion

To refine our study of precipitation reaction monitoring with a multipoint probe, a Common Components and Specific Weights Analysis was performed. This multivariate analysis can be used to find dimensions common to the angles. The result obtained is a compromise based on the individual scores and loadings.

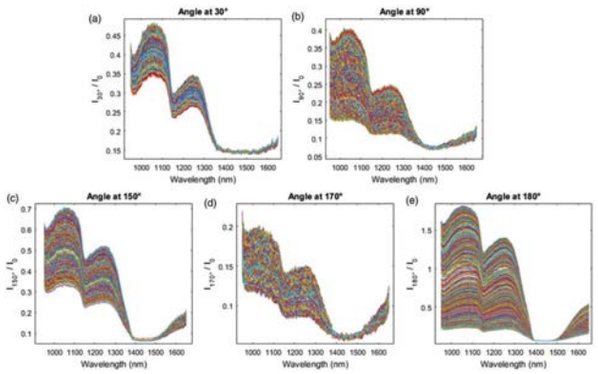


Figure 02:
Near-infrared spectra of the batch, obtained with five angles

Monitoring of the global scores enabled us to identify various scattering regimes according to the dimension. (Cf. Figure 03)

The first dimension reveals multiple scattering of the photons, while the second dimension represents simple scattering. This second dimension is very precise, allowing you to detect the freezing point unambiguously.

Finally, the third dimension is directly linked to the anisotropy coefficient and therefor the size of the particles during the precipitation reaction.

All the information obtained at the three dimensions demonstrates the complementarity of the angles for monitoring this reaction and the advantages of multipoint probes.

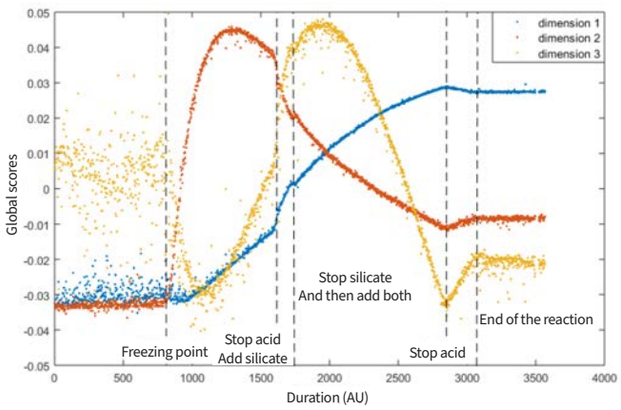


Figure 03:
Scores common to the different dimensions according to the duration of the batch with parallel presentation of the actions linked to the progress of the reaction

Advantages of SFX-IM probes

SAM-SPEC multi-angle technology offers a flexible solution for in-line inspection of precipitation/crystallization processes:

- Multi-angle measurement enabling you to monitor the evolution of the particle sizes by means of the different light scattering phenomena
- NIR or UV-Vis spectroscopy associated with chemometrics allowing physical and chemical characterization of the product of interest

For further information, please see the research paper: Rey-Bayle, M., Bendoula, R., Caillol, N. & Roger, J. M. (2019). Multi-angle near-infrared spectroscopy associated with common components and specific weights analysis for in line monitoring. Journal of Near Infrared Spectroscopy, 27(2), 134-146. https://doi.org/10.1177/0967033519830062

INDATECH's teams will be delighted to answer your questions by telephone: +33 4 80 78 01 40



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CA INDATECH
4, rue Georges BESSE - 34 830 Clapiers - France
Tél. : +33 4 80 78 01 40
sales@indatech.eu | **www.indatech.eu**