PhD Thesis Defense

Process Experimentation Department

Intensification of experimentation Department

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«Use of multipoint Near Infra-Red Spectroscopy and chemometrics for in-line characterization of scattering media»

Résumé

The aim of this thesis is to demonstrate the potential of multi-point near-infrared spectroscopy to monitor scattering and absorbing media whose physical and chemical properties change over time.

The work first focused on the study of an environment where only scattering varies over time. The monitoring of the precipitation reaction of the silica has been chosen to illustrate such a

medium. In a first step, collimated transmission spectra were measured on samples taken during the process. Thanks to the application of the Beer-Lambert law, it was possible to obtain the diffusion coefficients of each sample and thus to find the different stages of the reaction. A principal component analysis of these coefficients showed the presence of two diffusion regimes in the reaction medium.

In a second step, a multi-point measurement probe was immersed in a manufacturing reactor. Spectra were measured at angles of 30 °, 90 °, 150 °, 170 ° and 180 ° to the source throughout the precipitation.

Univariate analysis, at different angles, showed differences and similarities between the positions, in relation to light-matter interactions.

Then, a multivariate multi-block analysis ACCPS (Analysis in Common Components and Specific Weights) was applied. This methods combines information from different angles and show common and specific information. This method validated the use of multipoint measurements for process control.

Through the analysis of global scores, different types of scattering phenomenon and the specificity of certain angles in the detection of physical changes, have been identified. The analysis of the individual loadings confirmed the different reaction steps identified and revealed phenomena related to light scattering such as optical pathlength widening.

The same approach, for laboratory and then online analyzes, has been applied to an environment in which scattering and absorption vary. The monitoring of the products resulting from the processes for the improvement of the oil extraction was chosen to illustrate such a medium.

At first, microemulsions (water, oil and surfactant) have been manufactured so that the chemical and physical properties are different. Then, each phase was analyzed separately.

The Principal Component Analysis allowed to characterize the systems through the phase identification. It also showed that the differences between them were related to both absorption and scattering.

To separate these two phenomena, a Multivariate Curve Resolution (MCR-ALS) by Alternating Least-Squares has been applied. This method explained in more detail the differences between the phases to obtain semi-quantitative monitoring.

Finally, in the last part, the same samples were analyzed in circulation with a multipoint probe.

ACCPS was again applied and showed that different scattering phenomena occurred between classes. In addition, it highlighted a heterogeneity in the composition of the phases, which could not be detected with static measurements.

Key words: near-infrared spectroscopy, chemometrics, scattering and absorption, multipoint measurements, physical and chemical properties

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