Contribution to topical session „Mi²Pro“ at Achema 2018

Measurement and online quality control of continuous chemical processes

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In this contribution we present first results of a cluster of Universities and companies to develop a solution starting from batch processing of chemical products to a future of continuous processing. A core enabling technology for this is the in-line quality monitoring in chemical as well as pharmaceutical production and various other life sciences. A huge variety of basic principles are tested and frequently found to be not suitable. Concerning aspects like cost of ownership, reliability, process adaptability and specificity a focus for in line optical measurement technique can be recognized. The processes monitored in the collaborative research project Mi²Pro typically employ mixtures of liquids and solids – so called dispersed systems.

To solve this challenge a combined technology of UV-, VIS-, NIR-Spectroscopy while fluorescence and Raman-technology is used for the liquid phase. Particle measurement systems including turbidity, Mie scattering, image analysis or scanning are used for the dispersed part of the emulsions, dispersions, suspensions or aerosols. Amongst these technologies in particular Raman-spectroscopy and related methods show the most significant progress. The presentation will elaborate on this specifically in comparison to the classical solutions. The targets to be controlled are defined by extremely low temperatures in one case, highly concentrated pigment systems, organic and heterogenic phased targets in other cases.

Within this project two partners are responsible for the measuring technique: The HORIBA Group, with its worldwide subsidiaries, offers a wide range of measuring instruments and systems for the automotive inspection systems, process & environmental technology, medical technology, semiconductor and scientific sectors as well as OEM solutions for the optical market providing gratings and spectrometers. The University of Applied Science (UAS) Mannheim is represented by the Institute for Process Measuring Technique and Innovative Energy Systems (PI). This institute with 43 coworkers and currently leading the ranking of powerful institutes at UAS in Baden-Württemberg is embedded in the M2AIND consortium, a FH-Impuls project which is for the next 4-8 years a central German center in measuring technique at UAS.

In the first section the principles of Raman-scattering and -spectroscopy are covered. The advantages of Raman techniques like selectivity, in-line capabilities, resisting rough production circumstances, availability of many and special probe heads are explained.

Subsequently the very low excitation cross sections will be discussed and solutions by using new generations of sensitive detectors leading to smaller laser power, longer lifetime and service intervals are shown. Standard measurement models are presented followed by specialities, necessities and challenges within our project. For the project-related measurement task a customized Raman spectrometer has been designed with outstanding sensitivity and signal-to-noise ratio. Core of the Raman system is a spectrometer optimized for an excitation wavelength of 785 nm. The usable temperature range of the probe extends from (-100 ... +300) °C and the applicable pressure is well above 400 bar.

Examples for different measurement settings are demonstrated. With an outlook to the expected improvements and inventions for the next near future the lecture will end.