Detection of unstable process conditions during continuous nitrate ester production

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It has been known for years that micro structured flow reactors are highly suitable to handle fast exothermic reactions due to the excellent mixing and heat transfer [1]. Moreover, for technical scale production remote-controlled and automated processing can ensure the safety of technical staff and infrastructure. Therefore the integration of inline sensors (eg. temperature, pressure, conductivity) is essential. Commonly, these sensors are mostly integrated in the peripheral pipes or tubes, allowing only a delayed identification of unstable process conditions inside reactors, which can be too slow relating to fast reactions. Nitrate esters are synthesized by esterification of different mono- or polyvalent alcohols with mixed acid (HNO3 / H2SO4). As for many other reaction of this type, the stoichiometry has to be maintained very carefully. An excess of nitrating agent is strictly necessary. Maladjustment of stoichiometry usually leads to runaway reactions and can cause serious disturbance and even deflagration in the reaction zone.

Here we present the integration of multiplexed online spectroscopy in order to detect unstable process conditions during the reaction in explosible nitrate ester production at kg-scale [2]. The flow reactor is equipped with a hyperspectral imaging system which allows spatially resolved spectroscopic monitoring [3] along the reaction zone (Fig. 1).

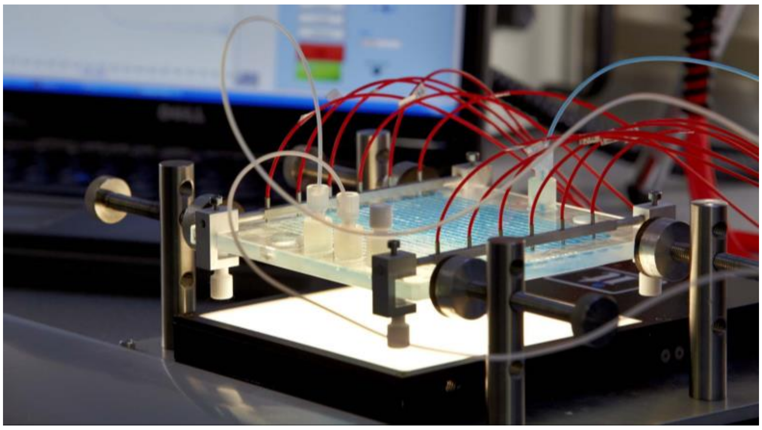

Fig. 1: Customized hyperspectral imaging for multipoint in-line measurements in continuous reactors

Thus, unstable process conditions and the resulting color change in the reaction mixture, caused by the decomposition of the product, can be identified in an early stage. The analytical data were implemented in a process control system and closed loop controls were realized (Fig. 2). This enables immediate automated adjustment of process parameters, i.e. flow rates of reagents.

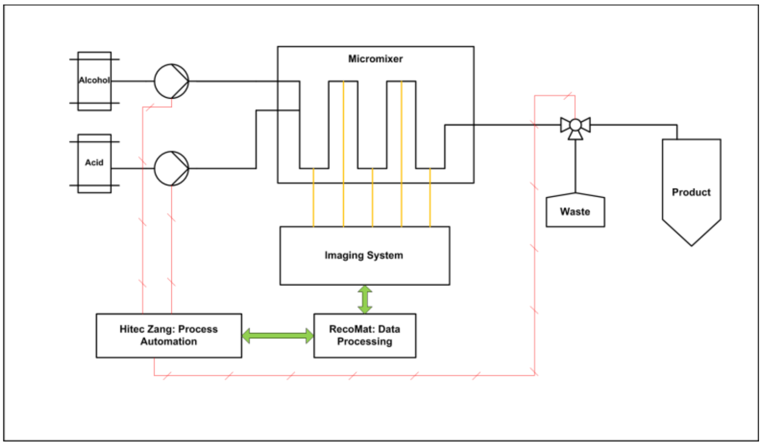

Fig. 2: Scheme of the control loop: Spectral acquisition of the reaction mixture through light guides (yellow); Data acquisition and processing (green); Process control with the lab automation system (red).

The setup is used in a pilot plant for the continuous production of various nitrate esters (methyl nitrate, ethylene glycol dinitrate, nitroglycerin, butanetriol trinitrate) with a capacity of 10 kg/day (Fig. 3).

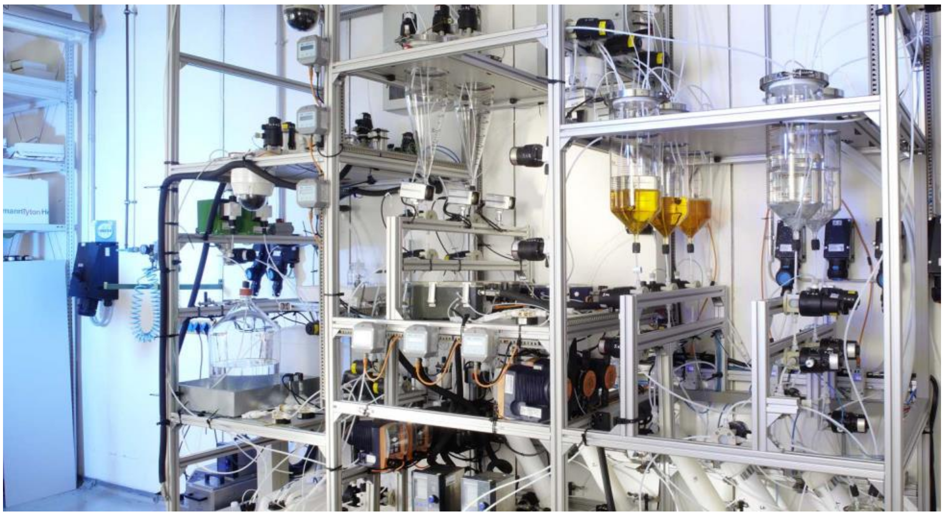

Fig. 3: Plant for continuous kg-scale synthesis of nitrate esters

The presented analysis and control strategy contributes significantly to the overall process safety. Moreover, it allows for more efficient reagent consumption and reduction of (acid) waste. Such closed loop system can also be realized with other spectroscopic system like Raman or Infrared spectroscopy.

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