Mastering a Reaction beyond Decomposition Temperature by Moving from Batch to Continuous Processing

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The conversion of chlorosulfonic acid with isocyanates to imidosulfuryl halides has to be considered as a very challenging reaction due to severe corrosion, CO_2 evolution during the reaction and a very limited thermal stability. A semibatch process has been the initial approach to perform the reaction within a stirred tank reactor in an industrial framework. For the thermal safety management, three main heat sources must be considered. The isocyanate as well as the product show significant decomposition enthalpies (>-500J/g each). Additionally, the reaction enthalpy contributes with >-500J/g to the potential. Since no solvent is used, the adiabatic temperature rise of the reaction is >500K. Moreover there's only a small window between the reaction and decomposition temperature which makes a sound process safety strategy indispensable. An early, never realized, production concept consisted of a $4m^3$ semibatch reactor connected to a large flooding volume of chlorosulfonic acid to decelerate the reaction in case of a runaway.

Considering all these difficulties, a continuous process promised to reduce the challenges in various areas. A short feasibility study in the laboratory confirmed that the targeted product yield is obtained in a plug flow reactor at higher pressure, higher temperature and extremely reduced reaction time. Due to the precise heat control the reaction temperature can be raised even above the decomposition point. The total reactive volume is reduced to 15L (vs. $4m^3$ for same productivity in batch). The safety concept is substantially simplified, reducing both CAPEX and OPEX for the system. For the scale up from lab to pilot to production, the maximum dimensions of the reactor were determined with AKTS-Thermokinetics software, so the hot-spot formation could be controlled. The approach of AKTS using the differential scanning calorimetry signal to determine kinetics was the only possible method to characterize this highly reactive system. There is no analytical way to detect all species involved that could have been used to achieve this goal.

The combination of appropriate process design, predictive software and exploration of formerly uncommon reaction conditions made it possible to intensify the synthesis of imidosulfuryl halides. In the resulting setup both safety and investment costs benefit from the extremely reduced reaction volume. The chosen approach will be illustrated during the presentation. Furthermore the experience of large scale production will be shared.