Process optimization based on reaction calorimetry

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Safety is often perceived as a hindering factor for productivity. In a practical example stemming from the industry it is shown that the safety study may also give essential hints for the process optimization.

A process safety study of a Diels Adler reaction (A + B → P) was investigated by calorimetric measurements. At first the thermal process safety parameters, e.g. reaction heat, accumulation, adiabatic temperature increase (ΔT_{ad}) and Maximum Temperature of the Synthesis Reaction (MTSR) were determined by reaction calorimetry (CPA). Then the thermal stability of the reaction mass was studied by Differential Scanning Calorimetry (DSC) in order to elaborate a failure scenario and define the criticality class.

In this work the reaction was performed under pressure in a semi-batch mode with the reactant be fed at a constant flow rate. This process showed a strong exothermic reaction with an energy of 773 kJ/kg, which corresponds to an adiabatic temperature increase of 320°C. Thus, in case of cooling failure at the time of maximum accumulation, the temperature will reach 223°C.. In addition the thermal stability of the initial reaction mass is problematic: reactant (A) is unstable. Two exotherms were observed: the first one with an energy of 52 kJ/kg and 562 kJ/kg for the second one. However the thermal stability of the final reaction mass poses no problem: the product P is stable, since no exotherm was detected. Thus the process belongs to the criticality class 3. This class requires a perfect control at the maximum temperature for technical reasons (MTT), here at 143°C, which corresponds to the opening of the safety valve. These results clearly mean a “no go” for scale up.

Nevertheless, based on the considerations above, essentially on the fact that the final reaction mixture is stable, an alternative process with a simultaneous dosing of reactant A and B on a precharged base of P was developed. Its safety parameters are studied and discussed in this presentation.

This example shows how a well conducted safety study opens possibilities for improving the process in such a way that it can be scaled up in a safe way.