

Process Optimization

Cuts High Chemical Production Costs

- Solvent consumption cut by 50%
- Production costs lowered by 10%
- Batch time reduced from 10 to 4 hours

The challenge

A major speciality chemicals manufacturer sought to reduce the high costs of solvent consumption in a current production process. The problem involves both high recycling costs and evaporative losses to the environment. The potentially hazardous nature of the process made in-plant optimization difficult. The plant in present use includes extensive safety measures to prevent a thermal runaway in case of equipment failure. With the METTLER TOLEDO RC1_e Reaction Calorimeter ways shall be explored to reduce solvent costs while maintaining process safety as well as product quality and yield.

The procedure

The existing procedure was first simulated in the RC1_e (Fig.1): While heating the reactor content of raw material A to 40°C, a stoichiometric portion of reaction partner B is added. The reaction mass is held isothermally until the heat production has decreased.

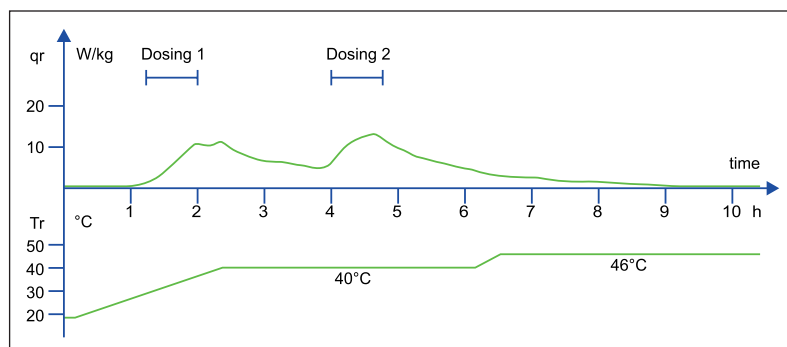
In order to accelerate the reaction, an excess of reactant B is then added and the reaction mass is again kept at 40°C. Finally, the temperature is raised to 46°C for the reaction to run to completion. The heat production curve shows that only about 10% of the feed was converted at the end of the



first addition. If a malfunction were to occur at this stage (e.g. cooling failure, stirrer breakdown) the continued reaction of the accumulated amount of B would cause a runaway without the use of special emergency measures.

Figure 1

Substitution reaction according to procedure in current use (RC1_e experiment)



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The reaction mass would heat up leading to possible secondary reactions that can be estimated from the microcalorimetric study (DSC curve, Fig.2). In the temperature range 100 to 200°C, an exothermic reaction of -290 kJ/kg takes place. At 300°C a violent decomposition then sets in with a heat production of another -220 kJ/kg. This is the very reason why the safety of this process was considered critical. Any changes in the process must thus first be checked thoroughly by measurements to prevent a trade of lower production costs for an increased safety risk.

Modification of the procedure

The analysis of the existing procedure suggests a semi-batch process with dispensing rate, reaction temperature and concentration of reactants was investigated. Thanks to the reproducible operation of the RC1_e a wealth of data was acquired from each individual experiment. These data were then used to design an optimum procedure (Fig.3). The reaction is now performed at 62°C (boiling point of the

solvent). The higher temperature and the selected addition time result in a considerably enhanced reaction rate. The increased heat production of the reaction can be dissipated without problem through efficient use of evaporation cooling. In addition, the faster reaction keeps the accumulation of non-reacted material now below 0.4 = 40%. The resulting adiabatic temperature rise was found to be at a non-critical level.

Realized benefits

The **solvent consumption is cut by half**. Total **production cost savings** amount to **10%**.

Since the reaction runs considerably faster, the **cycle time** has been **reduced from 10 to 4 hours**.

Increased process safety – although not a primary aim of the work, an appreciably safer design of the process was achieved owing to the exact knowledge of the process data. As a consequence, certain expensive emergency measures were rendered unnecessary.

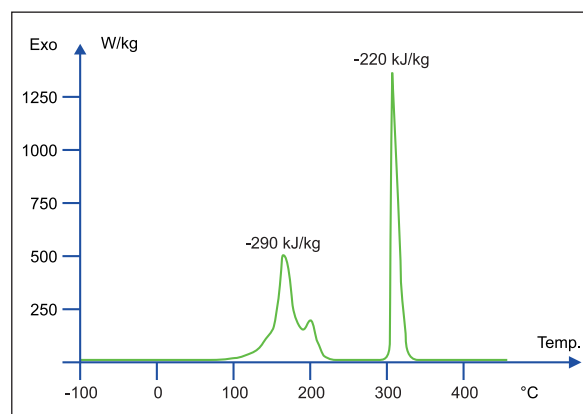


Figure 2
DSC curve of the final reaction mass

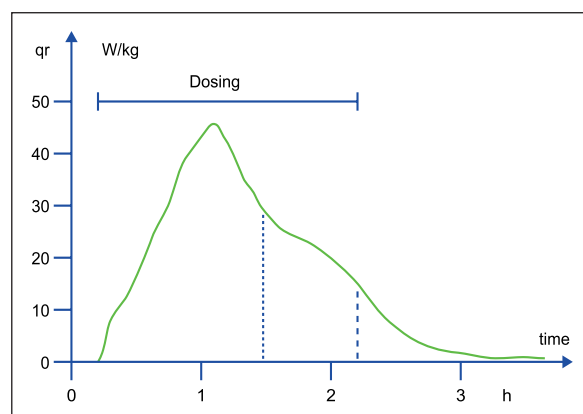


Figure 3
New procedure run at 62°C isothermally and at higher concentrations (RC1_e experiment)

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