# SAFE SCALE UP CALORIMETRIC STRATEGIES AVOIDING DANGEROUS PRESSURIZATION AND EXPLOSIONS OF CHEMICAL REACTORS

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Runaway reactions are often characterized as highly exothermic and fast. If the heat flux generated by the reaction exceeds the heat flux removed by the cooling system, this can result in a loss of temperature control. This can lead to a further increase in the reaction rate and heat generation, where the reaction continues to accelerate uncontrollably, with possible triggering of secondary decomposition reactions. Such reactions can lead to an increase in pressure within the reactor and the formation of toxic or flammable products, ultimately causing the reactor to explode.

Such an unwanted phenomenon can take place into every type of reactor (continuous and discontinuous), operated in whatever temperature control mode (isothermal, isoperibolic, polytropic, etc.) and fed by the most disparate dosing strategies (linear, ramp, etc.).

The causes of runway phenomena can be different:

1) insufficient understanding of kinetics and thermochemistry
2) accumulation of reagents leading to an uncontrolled increase in reaction rate
3) failure or inadequacy of the cooling system resulting in an inability to dissipate heat
4) inadequate agitation
5) human error

Systematic search for hazards, risk assessment and identification of possible remedies are the basic steps of risk analysis.

In a batch production process involving a chemical reaction with a significant heat effect, the reaction is performed using a semibatch mode: one of the reactants (co-reactant) is slowly added to the second component, which has already been fully loaded to the reactor. This method allows for better control of the reaction rate and heat evolution. By adjusting the addition rate of the first reactant, the heat flux generated by the reaction can be controlled and set to a desired value. This approach ensures that the reactor cooling system is able to remove all the heat flux generated during the reaction.

Once a set of safe operating conditions has been selected at the laboratory scale and validated through reaction calorimetry experiments, it must be scaled-up to the industrial reactor scale. However, such a scale up process should be performed not only under safe conditions but also maximizing industrial reactor productivity; so scale up criteria must be easy to use and not expensive.

To ensure the safe and productive operation of a batch reactor, the following conditions should be met:

1. The dosing speed of the coreactant should be equal to the reaction speed. This ensures that there is no accumulation of coreactant within the reactor. By maintaining a balanced dosing and reaction rate, the cooling system can effectively dissipate the heat generated during the reaction.
2. The maximum temperature reached inside the reactor must be below the maximum allowable temperature (MAT). Staying within the maximum allowable temperature ensures that the reactor operates within safe limits and avoids any potential risks or adverse effects associated with overheating.

To determine the optimal dosing time that guarantees isothermal conditions on an industrial scale, the following steps can be followed:

1) Choose a laboratory-scale dosing time and determine the overall heat transfer coefficient (U) and heat capacity (Cp) of the reactor system.
2) Calculate the dosing time at the laboratory scale that ensures isothermal conditions.
3) Determine the overall heat transfer coefficient (U) and heat capacity (Cp) of the reactant mass at the industrial scale.
4) Using the determined values of U and Cp at the industrial scale, calculate the dosing time required to achieve isothermal conditions.

The neutralization reactions of the following acids were chosen as a case study:

1) $H_3PO_3 + 2KOH \rightarrow K_2HPO_3 + 2H_2O$
2) $HCl + NaOH \rightarrow NaCl + H_2O$
3) $CH_3COOH \rightarrow CH_3COONa + H_2O$

These reactions exhibit a simple kinetics without complex reaction mechanisms or intermediate steps and the reactions proceed at a relatively fast rate.

To evaluate the parameters on the laboratory scale, tests were carried out using Mettler Toledo's Easymax 102 reactor, which has a volume of 100 ml. For the tests on the industrial scale, the Optimax 1001 reactor from Mettler Toledo was chosen. This reactor has a larger volume of 1000 ml.

The experimental tests conducted validated the developed procedure, indicating that the dosing time calculated on the industrial scale ensures isothermal conditions. This means that the chosen dosing time allows for maintaining a constant temperature throughout the reactions. Furthermore, all the reactions are carried out under dosing time control.