DEVELOPMENT AND VALIDATION OF A SAFE PROCEDURE TO SCALE-UP HAZARDOUS CHEMICAL PROCESSES

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Abstract

Loss of reactor temperature control, due to activation of side reactions or decomposition of the reacting mixture, has been thoroughly analyzed in the process safety literature from the well known accident occurred in Seveso (Italy, 1976). This phenomenon, known as "thermal runaway", represents one of the major problems to scale-up a process from laboratory to full plant scale. In fact, because of the increase of the reactor volume consequent to scale-up, the ratio between thermal power removed by refrigeration system and that one developed by the reaction is penalised.

Operating conditions of semi-batch reactors can be considered safe if they correspond to a sufficiently low dosed reactant accumulation, so that the cooling system can control the heat evolution. Once a set of operating conditions has been selected at the laboratory scale, it must be then scaled-up maximizing the industrial reactor productivity. In order to obtain a practical solution of the problem, every scale-up procedure of such processes must be simple and general at the same time. Such goals are not easily achieved in practice since, especially in fine chemical industries, a wide range of products in relative small amounts are produced. As a consequence, detailed mathematical model of the single process is often not available because of money and time constraints.

In this work $ClC_6H_3(NO_2)CF_3$ synthesis by $ClC_6H_4CF_3$ nitration in mixed acids has been studied to develop and validate a reliable scale-up procedure.

The most important parameter to be optimized is dosing time since initial reactor temperature arises from laboratory tests in order to obtain the desired product selectivity. Once found optimum dosing time at laboratory scale, it is necessary to scale up it to industrial scale. The procedure developed in this work permits to find full plant scale dosing time through the usage of two different typologies of diagrams: temperature (TD) and boundary diagrams (BD). TD allows to verify that the process does not operate close to Maximum Allowable Temperatures (MAT) beyond which undesirable exothermic phenomena may occur. BD allows to optimise process parameters, identifying optimal operative conditions, both from the productivity or safety point of view, through the minimization of the reactants accumulation into the system.

In order to validate the developed scale-up procedure, several experiments has been carried out to: 1) characterise the reactants, products and reacting mixture in a given temperature range; 2) define the MAT; 3) determine the reaction kinetic parameters; 4) simulate the isoperibolic reactor temperature profile. The results obtained have shown that BD and TD are suitable tools to characterize thermal behaviour of semi-batch processes, allowing the optimisation of their fundamental parameter, namely dosing time.