

Fluitec Documentation No. 11.136 Rev. 1

From Batch to Conti: Process Intensification by Fluitec

Transforming batch processes of chemical reactions into continuous operated systems serves numerous distinctive advantages, such as improved safety, constant product quality and maximised space-time yields. If designing such a continuous operating plant, some important factors must be considered already at the planning stage: kinetics, thermodynamics, mass transport rates, selectivity and the flow of energies are among the most important influencing parameters. Taking these factors into account, the change from batch to continuous production becomes a reliable and very economic opportunity.

Advantages of Continuous Reaction Processes

Once started-up, the continuous operation of chemical reactions serves many advantages such as:

- improved reaction control
- reduced reaction volume
- lack of dead spots
- improved local energy dissipation
- stable operating parameters
- no time needed for filling, draining and cleaning
- reduced space required, etc.

In addition, static mixers possess no moving or rotating parts, thus there is no wear and no maintenance is required.

Characterising the Reaction

Before transforming a batch reaction into continuous operation, the most important physical and chemical parameters must be thoroughly characterised. Depending on the complexity of the process, the preliminary experiments can be performed simply in a stirred beaker or more sophisticated in a very precise reaction calorimeter. The quality of the experiments, however, must be high enough to get reliable informations about the reaction.

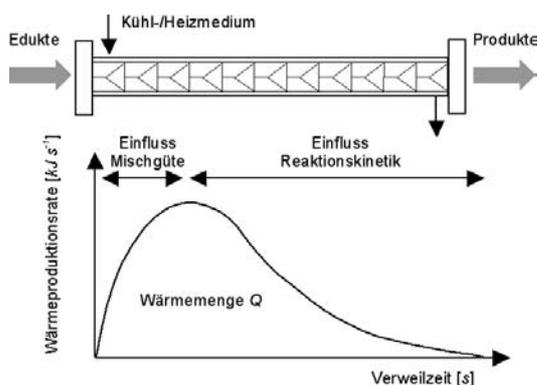


Fig. 1 The profile of the local heat production rate in continuous tube reactors can be calculated by the appropriate batch reactions.

Transforming Batch Reactions into Continuous Reactions

Data from batch processes can be transferred to continuous operating systems: the reaction time of batch reactions becomes to the residence time in tube reactors, if considering the specific mixing energy and the heat transport (Fig. 1), thus allowing the precise location of the hot spot.

Fluitec is capable of simulating the temperature profile in tubular mixer/heat-exchangers with high accuracy. Based on the quality of the preliminary experiments, the deviation of the calculated local temperatures to the measured values can be +/- 1°C. A very precise simulation is essential if performing exothermic reactions using temperature sensitive or even explosive compounds.

The equation of the heat transfer balance for continuous ideal tubular reactors is a good fundament for the calculation of the heat transfer balance of the CSE-XR reactor (mixer/heat-exchanger):

$$\frac{\partial Q}{\partial t} = -\bar{c}_p \cdot m \cdot \frac{\partial [w_z \cdot T]}{\partial z} + (-\Delta H_R) \cdot V \cdot r_v + \alpha \cdot A \cdot (T_{gr} - T)$$

Eq. 1 Equation of the heat transfer balance for continuous ideal tubular reactors

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Fluitec's product line mini production plant (mpp) is based on its modular and extremely versatile design, thus allowing the fast, economic and autonomous characterisation of continuous reactions. By this means, the user can test the possibility of transforming any batch reaction into continuous processes by himself. Of course, the competent and well-experienced Fluitec staff is permanently assisting and analysing the results gained. Design, components and choice of material of the final mpp plants are always custom based.

Fig. 3 shows an mpp plant with PTFE tubes of 8 mm inner diameter, allowing a throughput rate of 1'200 kg per day. The ex-proofed plant is equipped

with several temperature-, pressure- and pH-sensors to perform the 5-step reaction. It is connected to the companies internal process leading system (PLS) and all material in contact with the product is out of PTFE, Hastelloy C22 or Tantalum.

In many cases, however, it is sufficient to characterize a single reaction step only. For this reason, Fluitec provides standardised tubular reactors for mixing and heat exchange. They can be equipped with double shell, diverse probes, injection nozzles, dosing pumps, etc on the customers demand. Dimensions, materials, fittings and pipings are defined in close cooperation with the customer. The reactors can be used for low and highly viscous components.

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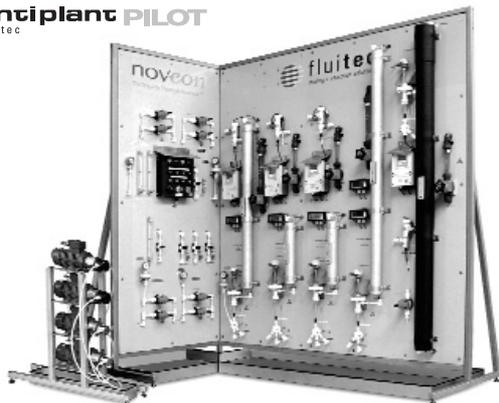


Fig 3: mini production plant (mpp) by Fluitec for the flexible characterisation of continuous, exothermic reactions of up to 5-step by using explosive and corrosive media

Fast Reactions

A reaction can be classified as fast, if it is completed within (the fraction) of a second. The limiting factor in these cases is the mass transport of the components only, respectively the mixing intensity of the system. Due to the stationary interfacial films, multiphase systems such as liquid/liquid, gas/liquid, or e.g. the use of dispersed solid catalysts are often limited by mass transport rates.

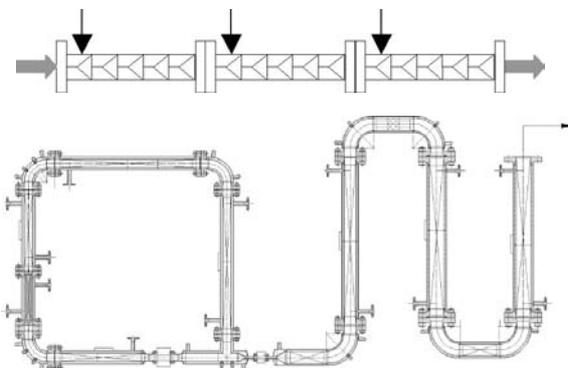


Fig. 4 Performing fast reactions, the additive must often be dosed repetitive or in a loop in order to control the heat generated

Fast exothermic batch-reactions can only be controlled by the very slow dosing of the additives, under intense cooling and stirring. Scale-up ability, however, is rapidly limited by the decreasing specific heat transfer rate, thus creating a safety

hazard. In continuous tubular reactors, on the other hand, it is possible to localize and to predict the hot-spot very precisely. In combination with the small reaction volume, the large cooling area and the controlled and intense mixing performance the complete control over the reaction can be guaranteed.



Fig. 5 The patented CSE-XR mixer/heat-exchanger guarantees controlled and intense mixing and high heat transfer rates.

Slow Reactions: Residence Time Reactors

The continuous performance of slow reactions often requires a very narrow residence time distribution, expressed as Bodenstein-numbers, at long residence times. The tendency to back-mixing can be characterised by a Dirac-pulse and detecting the particles at the outlet. Static mixers induce continuous and intense radial mixing, thus generating a plug-flow regime and high Bodenstein-numbers. The plug-flow regime is influenced by several factors such as (changing) viscosities, thermal induced convection, L/D-ratio of the reactor, etc. Static mixers in combination with especially designed inlet and outflow sections generate plug flow regime even at residence times of several hours. Using for example a reactor of 70 m total length, 4 hours of residence time and a diameter of DN 450 Bodenstein-Numbers > 300 are achieved by using Fluitec's technology and know-how.

The flow pattern is characterised by many specific experiments, industrial experience and numerous CFD-calculations.

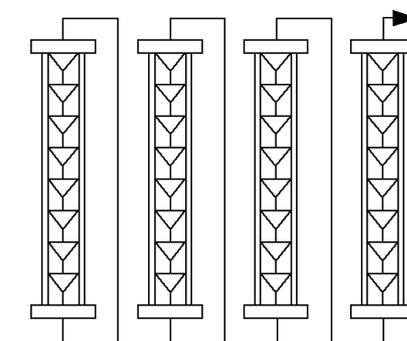


Fig. 6 Large continuous reactors for slow reactions often have to be built in segments

Examples of Application

Continuous tubular reactors of Fluitec are used e.g. for the following applications: polymerisations, polymer modifications, esterifications, nitrations, diazotations, alcylation, halogenations, hydrations, oxidations, neutralisations, etc.