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Fluitec continuous reaction calorimeter

Reaction calorimetry is a key requirement for economical and safe process design. As this has been practiced for years in batch-mode, it is now possible to be done directly in a continuous manner. Novartis could recently show that the heat of reaction could depend on the reaction mode (flow vs. batch) [2]. We therefore present a commercially available continuous reaction calorimeter similar to the one used by Novartis. In the following report, the equipment setup, determination of the heat of reaction and the range of operation will be described. This new generation of reaction calorimetry has a high potential for safe scale-up of continuous processes.

There is a trend of developing continuous processes in tubular reactors for industrial applications. In order to make these continuous processes safe and sustainable, a precise knowledge of the heat of reaction will be required. Traditionally, reliable heat measurements have been carried out in well-developed batch-like reaction calorimeters [1]. Although they are able to mimic batch-like processes, they are not always suitable to represent continuous processes in tubular reactors [2]. Fluitec presents therefore a commercially available continuous reaction calorimeter similar to the one used by F. Mortzfeld et al. [2]. In contrast to micro-reactor-like continuous calorimeters, the surface-to-volume ratio of this tubular calorimeter is so as to be easily scaled with Fluitec mixer-heat exchangers. It thus opens the way for an economical and safe scale-up.

Equipment setup

The Fluitec continuous reaction calorimeter comprises two flow-controlled dosing systems, one jacketed tubular reactor with static mixers inside, one axial temperature sensor, temperature sensors for the heat transfer medium side as well as a precise thermostat (Fig. 1). The equipment is available in stainless steel and in Hastelloy.

Fig. 1: Fluitec reaction calorimeter setup.

Determination of the heat of reaction

The heat of reaction will be determined by evaluation of the temperature profile along the tubular reactor. Fluid properties and well described heat transfer characteristics are required for a precise calculation of the actual heat of reaction. We further recommend applying a screening of different flow rates to find the highest possible heat production for safety reasons.

Range of operation

The operation range of the Fluitec reaction calorimeter is described in Tab. 1. We recommended a temperature range from room temperature to 250 °C, which however could be extended to -40 and 300 °C by using appropriate sealing materials. It was experienced that a minimal back pressure of about 1 bar should be applied for a constant dosing performance. Using gear pumps, it is possible to apply a maximum back pressure of 10 bar. By using other pumps, such as HPLC pumps, even the maximum permissible pressure of the tubular reactor (60 bar) could be applied. We recommend a flow rate range from 20 to 100 ml/min for a good performance in the heat exchanger since the heat transfer characteristics are well defined within a range of 0 < Re < 100. We further recommend a viscosity range from 1 to 120 mPas, which could be extended up to 1000 mPas by using appropriate pumps.

Tab. 1: Operation range of the Fluitec reaction calorimeter.

	min	max	min (extended)	max (extended)
Temperature [°C]	RT	250	-40	300
Pressure [barg]	1	10	-1	60
Flow rate [ml/min]	20	100	2	400
Re number [-]	0	100	0	100
Viscosity [mPas]	< 1	120	< 1	1000
Reaction time [min]	< 1	2	< 1	unlimited

The Fluitec reaction calorimeter is suitable for exothermic reactions leading to a polytropic temperature profile in the tubular reactor. Its full heat can only be determined if the reaction is 100 % converted within the reactor. This applies for chemical reactions with a reaction time of maximum 2 min when working with the minimum flow rate of 20 ml/min. The range of reaction time could even be extended by using appropriate analytics at the outlet of the reactor to determine the chemical conversion.

This kind of reaction calorimetry allows to design continuous flow reactors in an economic and safe manner, since the actual heat of reaction in continuous mode will be determined. The flow rate screening further serves as a safety tool to find the maximum possible heat generation to describe the worst-case scenario.

References

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[2] F. Mortzfeld, J. Polenk, B. Guélat, F. Venturoni, B. Schenkel, P. Filipponi, Org. Process Res. Dev. **2020**, 24 (10), 2004–2016. DOI: 10.1021/acs.oprd.0c00117.

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