CHARACTERIZATION OF HOT SPOTS IN MICROSTRUCTURED REACTORS FOR FAST AND EXOTHERMIC REACTIONS IN MIXING REGIME

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Summary

The intensification of fast exothermic reactions can be achieved by using microstructured reactors (MSR) which provide improved mass & heat transfer rates leading to higher overall reaction kinetics. But for highly exothermic reactions the heat evacuation becomes not efficient enough and unwanted hot spots are formed. In this study, first the mixing in MSR is quantified for different geometries and then temperature profiles are measured using a novel quantitative IR-thermometry method. The results are fitted to a lumped parameter model of overall reaction kinetics and the influence of the operating conditions is established.

Keywords

Exothermic reaction, Hot spot, Infrared thermometry, Microstructured reactor, Mixing

Introduction

Nowadays, more and more efforts are made to make processes continuous due to their advantages over batch and semi-batch processes such as better production quality and lower operating cost. This tendency of substantially improving process efficiency (in terms of safety/energy/space/time) through innovative ideas is often referred to as "process intensification". The intensification of very fast and strongly exothermic reaction requires an improved control of heat release which can eventually be achieved by increasing transport rates by virtue of changing reactor geometry with higher surface to volume ratio.

Microstructured reactors (MSR), defined as miniaturized systems with at least one dimension in submillimeter range, are considered as one of the tools of process intensification. The small sizes (in the range of 10^2µm) result in very high specific interfacial area which in turn increases heat- and mass transfer significantly. Besides, the low hold-up in the MSR allows its operation under harsh conditions.

In the case of fast reactions, the productivity of the process can significantly be improved using MSR due to high mixing (or mass transfer) rates. The apparent reaction time in MSR is strongly influenced by mixing time which varies significantly from millisecond to seconds depending on the operating conditions used[1]. The heat production increases with decrease in apparent reaction time for a given set of operating conditions as shown in Figure 1.

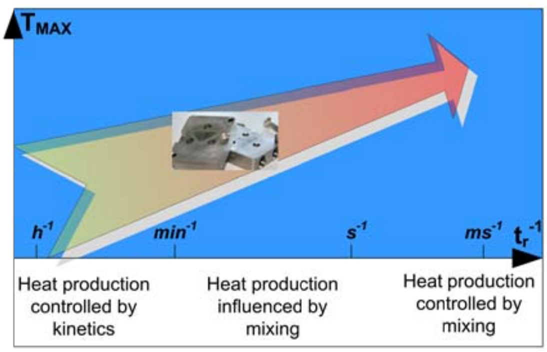

Figure 1: Hot spot temperature as function of reaction time (t_r).

Though the small size of MSR offers sufficiently low characteristic cooling time, the excessive heat production in the case of fast exothermic reactions results in unwanted hot spots. This has a detrimental impact on the product quality (coloration, broad molar mass distribution, etc.) and/or selectivity due to undesired side reactions[2].

Methodology

In the present work, experiments were carried out using the classic methods of mixing to investigate the mixing quality in different types of MSR under isothermal conditions. Two different parallel competitive reaction systems were used: the acetal cleavage reaction and the Villermaux-Dushman reaction [3].

Further a reacting system with fast intrinsic kinetics and high exothermicity was used and the mixing influence and controlled regimes were established. A newly developed method based on infrared thermometry was applied to investigate the temperature profile in the MSR. This is a non-intrusive method and gives quantitative measurements of temperature along the length of the reactor. The temperature was monitored on the surface of MSR wall and the reaction mixture temperature was investigated using a mathematical model considering the physical properties of wall material. If the setup is operated under ambient conditions, the reactor loses significant heat which is difficult to quantify. Therefore, the modifications were done in the experimental setup where the reactor is operated under vacuum resulting in adiabatic boundary conditions. To take infrared snapshots inside the vacuum chamber infrared mechanically stable transmitting windows made up of ZnSe were installed on top of the vacuum box in which the MSR is enclosed.

A mathematical model has been established considering the main scaling effects in MSR and the experimental data were fitted by lumping the appropriate parameters in the model.

Results

Three specially designed MSR were used. The influence of the MSR geometry on mixing efficiency was quantified over a wide range of flow rates under isothermal conditions. As expected, strong influence of two-fluid contacting geometry angle and flow rates on mixing quality was observed. Subsequently, the region of mixing influenced and controlled has been established. A fast exothermic reacting system was introduced and the temperature profiles were measured using infrared thermometry as depicted in Figure 2. The experimental data were fitted to the mathematical model using software Mathworks Mathlab 7.0 to investigate the lumped parameters. Finally, a correlation of lumped parameter as a function of operating conditions was established.

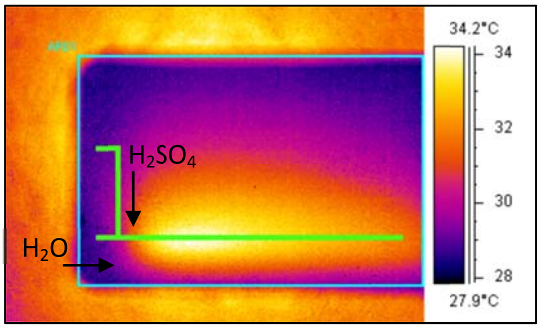

Figure 2: Temperature profile obtained by mixing sulfuric acid with water at low flow rates (0.5 ml/min).

The data obtained give a clear guideline for rational design of MSR for fast highly exothermic reactions. An *a priori* estimation of the hot spot is possible from the quality of mixing in the MSR.

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