DESIGN AND CONTROL TECHNIQUES FOR THE NUMBERING-UP OF CAPILLARY MICROREACTORS WITH UNIFORM MULTIPHASE FLOW DISTRIBUTION

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Summary

The results of experimental studies on eight parallel capillary microreactors for both single phase and biphasic liquid-liquid slug flow applications are presented. Four different flow distributor designs were investigated with respect to the uniformity of the liquid-liquid flow in the individual capillaries. A novel multichannel phase splitting unit was installed at the downstream end of the capillaries to ensure well-defined phase separation. The uniformity of flow rate and flow structure could be further enhanced by using a non-invasive capacitative measurement to characterise the flow in conjunction with specially developed microvalve and actuator techniques for the on-line control of flow rates and the manipulation of slug flow structure in individual capillaries.

Keywords

Capillary Reactor, Numbering-up, Liquid-Liquid slug flow.

Over the past two decades the principal benefits of microscale operation, such as rapid and thorough mixing, negligible temperature gradients, uniform processing, histories and intensified interphase mass transport, have been extensively documented in a large number of publications (e.g. [1, 2]).

In spite of this promise, instances of the industrial application of this technology remain few and far between. Closer inspection of the literature in this field reveals that the task of numbering-up is far more challenging than the pioneers of microtechnology originally anticipated. Even with precise fabrication techniques and advanced simulation tools, it remains extremely difficult to achieve a reliable uniform flow distribution over multiple parallel microchannels. The presence of a second phase in the capillary dramatically increases the complexity of the flow distribution task, since not only the flow rate but also the biphasic flow structure must be uniform.

The aim of this work is to develop a reliable, robust and affordable numbering-up strategy for liquid-liquid slug flow microreactor systems. In the first section the principles and phenomena which have to be taken into consideration for the numbering-up of liquid-liquid capillary microreactors are reviewed.

In addition to the performance of the four different flow distribution units studied, a novel multichannel separator for biphasic liquid-liquid systems located at the end of the capillaries is presented.

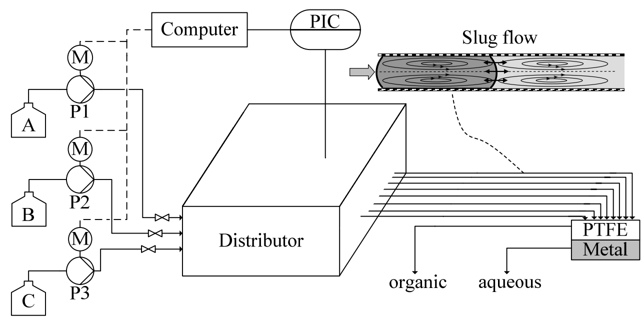

Figure 1 Schematic of experimental set- up

The distributors used are proprietary in-house developments using a variety of flow splitting concepts. The impact of manufacturing tolerances on flow maldistribution and mixing variances has been examined. It was observed that even minor modifications can result an immense improvement in the uniformity of distribution.

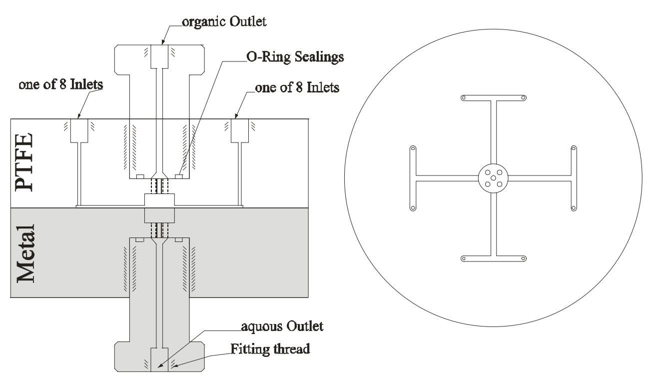

Figure 2 Splitter

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The key question is: what are the most important factors influencing the distribution (e.g. surface effects, fabrication tolerances, etc.) for each unit? In all the devices studied, a strong interaction between the flow rate and the slug length was observed. Smaller slug lengths give rise to increased pressure drops and thus diminished flow rates. It can be concluded that an external numbering-up of biphasic liquid-liquid systems is feasible down to tolerances of within 4% (standard deviation, as opposed to <1% for single phase flow).

For each specific application it must be estimated how much the expected deviations will decrease the selectivity of the desired reaction. For example, in simple mass transport limited extraction tasks, numbering-up will probably only have marginal influence on the capillary reactor efficiency.

In some cases, such as polymerisation reactions, the conversion as a function of the residence time and flow rate in each capillary strongly affects the pressure drop. In the presence of such interactions, dynamic fluctuations or even complete maldistribution can occur.

To overcome such problems, a non-invasive capacitative measurement technique was developed, as a basis for fine-tuning the flows in each capillary. By measuring the displacement current between two adjacent ring electrodes, it is possible to measure the rate of change of the liquid capacitance in the intervening section of the capillary. By reprocessing the measurement signal, using a so-called log-in amplifier, it is possible to characterise both the biphasic flow rates and slug structure.

The second prerequisite for manipulating the flow in individual capillaries was to develop appropriate new microvalves, since commercially available valves are expensive and exhibit unsuitable pressure drop characteristics. As the objective is only to tweak a flow distribution, which is already uniform to within a deviation of less than 10%, many of the demands made on conventional valves become superfluous.

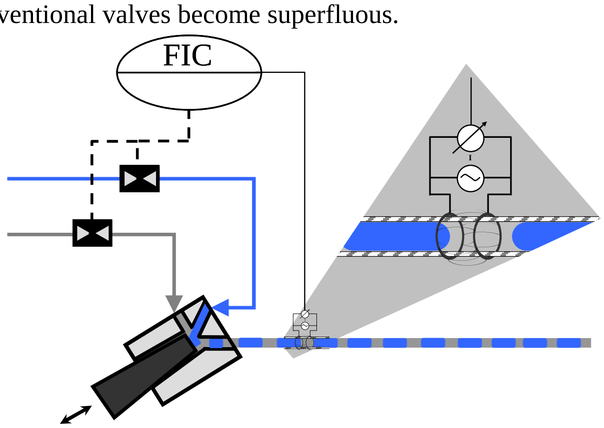

Figure 3 Capacitative Measurement and control of slug flow

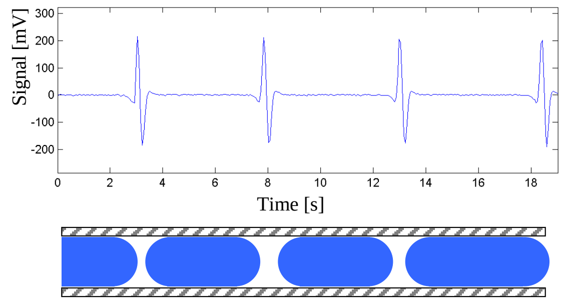

Figure 4 Signal of capacitative measurement

Besides the control of overall flow rates and flow ratios, the new measurement also provides the opportunity to regulate slug length. For this purpose, the geometry of the location at which the slug is formed and the initial contacting of the two phases must be adjustable. Since the slugs are formed by a reciprocating process, with each phase displacing the other until it encounters the opposite wall of the mixing element, this can be achieved by installing a set-screw to influence the exact point of slug disengagement.

Although issues concerning the integration of the measurement and control techniques described into a single robust unit and the precise control algorithms remain to be resolved, the underlying feasibility of the concept has been demonstrated. Furthermore, the regulation of slug flow in this manner should make it possible to employ simpler liquid delivery systems in place of the costly high precision, pumps used previously in such work.

References

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[2] Hessel, V. and H. Löwe, Microchemical Engineering: Components, Plant Concepts, User Acceptance - Part III. *Chemical Engineering & Technology*, **2003**, 26(5): p. 531-544.