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The influence of operating conditions on the mass transfer performance of a micro capillary contactor with liquid–liquid slug flow

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ABSTRACT

The application of two-phase slug flows for extraction processes in micro capillaries was investigated and the mass transfer performance under different conditions was analysed. As contacting capillaries PTFE or FEP tubes were used with internal diameters ranging from 0.5 mm to 1 mm. Two extraction systems were experimentally tested for the evaluation and the extraction efficiencies were measured for various flow rates and slug lengths. In the experiments the slug length was manipulated at a constant flow rate by means of a specially developed slug valve and a complete separation was ensured by a newly designed robust phase separator. Besides single-step experiments, a counter-current microfluidic flow was realised by connecting co-current flow modules in a counter-current arrangement and performance results for a two-stage counter-current operation were also obtained.

1. Introduction

For process intensification the utilisation of micro-scaled devices in combination with a counter-current operation is a promising strategy to amalgamate the advantages of both techniques as powerful tools for achieving excellent mass transfer rates and for overcoming equilibrium constraints in mass transfer limited reactions or separations. The achievable mass transfer rates, described by the product of the mass transfer coefficient k_L and specific surface area a, in slug flow microreactors are at least ten times higher than in other contactors [1–4]. This makes the use of microreactors particularly attractive for reactions or separations limited by mass transfer. Presently, the major bottleneck impeding the widespread industrial application of micro- processing technology is achieving the higher throughputs sought. Nevertheless, it still has an important role as a laboratory technique permitting shorter design times for the experimental equipment for specific small-scale syntheses or separations and identifying asymptotic performance limits.

Several techniques and various flow regimes are available for contacting two-phase-systems in both co- and counter-current micro-scaled flow modes [5,6]. The most common arrangements are depicted in Fig. 1.

Within this portfolio of possible devices and techniques, the use of slug flow, also referred to as segmented flow, satisfies the needs for an efficient and well-defined contacting of the phases and also for a facile separation of the two phases after each contacting zone [7]. Additionally, for the operation with slug flow an overall counter-current flow can be achieved by connecting individual co-current flow modules in an overall counter-current arrangement [8].

In earlier studies Burns and Ramshaw investigated the use of slug flow for the extraction of acetic acid from dispersed kerosene slugs into a continuous (wall-wetting) aqueous phase. They found that with increasing flow velocity the mass transfer rates also rose. They concluded that there are at least three pertinent operating parameters – residence time, flow rate and slug length – responsible for the extraction efficiency [9]. Okubo et al. also observed the relationship between the flow rate and the extraction efficiency. Their results too indicated that, for efficient mass transfer, both slug lengths have an influence and thus should be optimised [3]. Furthermore, Kashid et al. characterised four different test systems in terms of their mass transfer and compared the mass transfer coefficients achieved with those in macro-scaled benchmark contactors [2].

Although the experimental results show an improvement in mass transfer at higher flow rates, the underlying mechanism for this is not fully understood. The mixing in slug flow is a combination of convective and diffusive contributions and the characteristic mixing times in such segmented flows are therefore

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Nomenclature

a	Specific surface area [$m^2\,m^{-3}$]
c_i	Concentration at position i [$mol\,L^{-1}$]
E	Extract
ID	Internal diameter [mm]
k_L	Mass transfer coefficient [$m\,s^{-1}$]
n	Number
p_i	Pressure at position i [Pa]
PTFE	Polytetrafluoroethylene
R	Raffinate
u	Velocity [$mm\,s^{-1}$]
ϕ	Phase ratio
FEP	Fluorinated ethylene propylene

lower than those in stratified flows [10]. One reason for the increased mass transfer rates at higher flow rates is that the internal circulations inside the droplets are intensified at higher flow rates, because of higher shear stresses. These so-called Taylor vortices are not only dependent on the flow rate but also on the slug length and the various regimes for the internal vortices, which depend on the flow rate, slug length and the system properties [11].

From the three operating parameters influencing the extraction performance, the residence time can be changed simply by adjusting the length of the contacting section, i.e. the length of the micro-capillary-contactor (MCC), but the remaining two parameters – slug length and flow rate – are often closely correlated with one another. Thus a change in the flow rate generally leads to different slug lengths. Many authors have attempted to characterise this relationship between slug length and flow rate [3,8,11–15] but the results have largely been unsatisfactory or system specific.

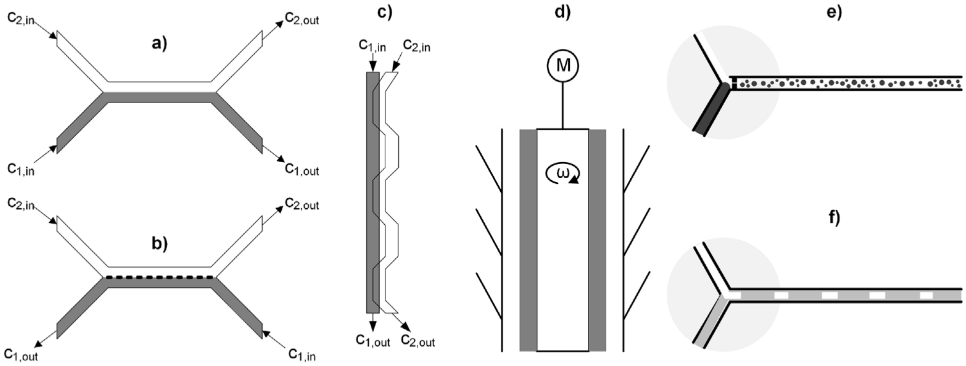

Fig. 1. Arrangements for co- and counter-current contacting of two-phase systems in micro-channels: a) Stratified flow regime, b) stabilisation of the interfacial area in stratified flows by membranes or sieve walls, c) partially overlapped channels, d) stabilisation of the interface with external forces, e.g. centrifugal force field in a Taylor-Couette reactor, e) emulsion flow with high specific surface area, f) liquid–liquid slug flow with medium-to-high specific surface area but well-defined conditions.

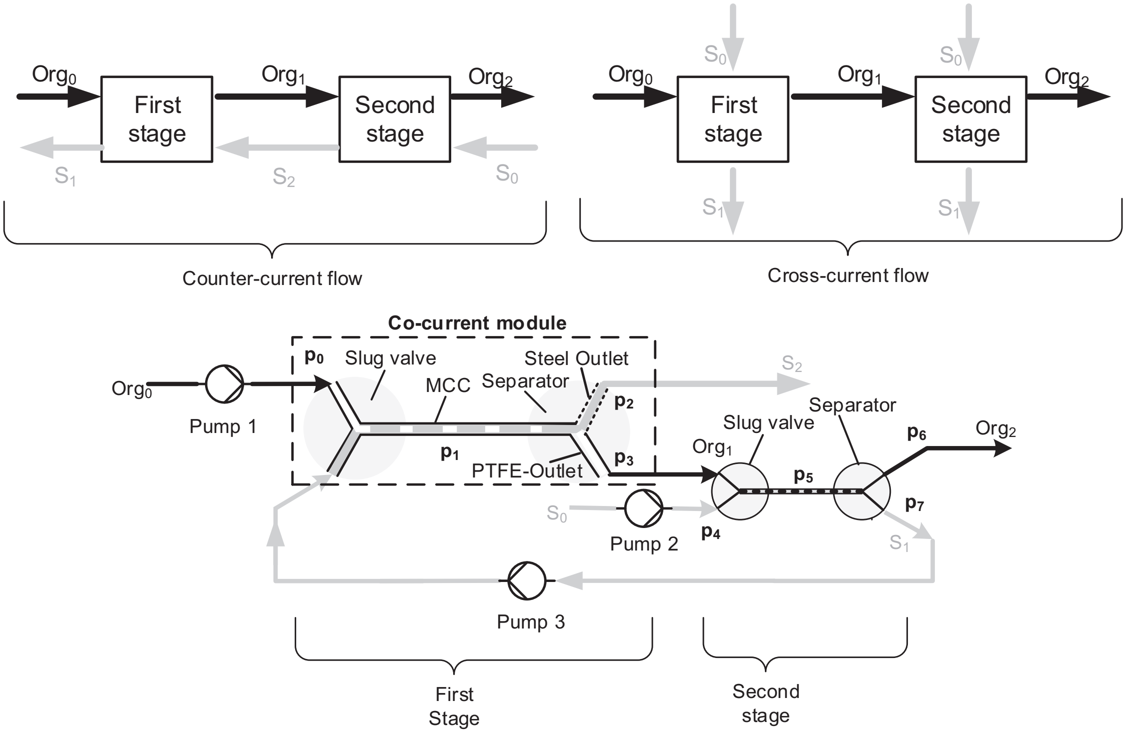

Fig. 2. Schematic setup for a multi stage operation of the co-current μ-mixer-settler modules.