

LIQUID-LIQUID SLUG FLOW CAPILLARY MICROREACTORS

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Abstract

Liquid/liquid biphasic reactions and extractions play an important role in the chemical and pharmaceutical industries. The concept of liquid-liquid slug flow capillary microreactor enables a new strategy in process intensification, providing considerable advantages over conventional liquid/liquid contactors, both in terms of the extremely high mass transfer coefficients and the low energy requirements. Fast biphasic reactions with mass transfer limitations can be enhanced in this type of reactor, in which alternating uniform slugs of the biphasic reaction mixture provide well-defined mass transfer areas and flow patterns.

The design basis for exploiting this concept in multiphase chemical microreactors entails the precise analysis of interfacial area between the two liquids and ascertaining the mass transport characteristics. Experiments were carried out to determine the mass transfer rates and effective interfacial area in a liquid-liquid slug-flow capillary microreactor. The effective interfacial areas measured by physical and chemical methods were compared and confirm the presence of the previously conjectured organic wall film, which provides a much higher interfacial area for mass transfer. Flow visualisation measurements have revealed that this wall film often exhibits a non-uniform thickness along the length of the non-wetting slug, a feature associated with the presence of multiple circulation vortices and stagnant zones within the slug, little appreciated in the past, which have been confirmed by CFD calculations.

A segmented flow splitter for continuously separating aqueous and organic slugs is also presented, which provides well-defined contacting times and permits the direct cascading of individual capillaries in various overall flow configurations. This simple 'Y' shaped device separates the two phases by exploiting the preferential wettability of a particular phase on different channel surfaces. Experimental measurements on and CFD simulations of the new phase splitter performance will be described.

Extraction is a separation process particularly well-suited to microscale operation, since it involves no temperature gradients and the low hold-ups are conducive to the application of highly selective advanced solvent systems, such as ionic liquids. The microcapillary reactor-extractor together with the phase splitter thus offers considerable potential for microscale downstream processing. The issues involved in microscale counter-current flow arrangements will be discussed in this context.

The preferential wettability of ultrafine suspended catalyst particles can also be exploited to retain them within one of the immiscible liquid phases and thus facilitate catalyst recovery without the need for subsequent filtration or sedimentation of the reaction medium. The excellent mass transfer characteristics within and between the catalyst carrier phase and reaction medium, together with the minimal catalytic pore diffusion resistances at the micrometer scale make such a biphasic heterogeneous catalysis an attractive alternative to homogeneous catalysis for microscale operation. The principle feasibility of this concept has been demonstrated for the catalytic transfer hydrogenation of m-nitrotoluene with aqueous formate using palladium catalyst supported on hydrophobic active carbon suspended in toluene.

Finally, numbering-up strategies to enable the benefits of the liquid-liquid slug flow capillary microreactor operation to be extended to higher throughputs have been developed. Due to fabrication tolerances, CFD modelling is of limited utility in designing appropriate distributors. To achieve both flow rates and slug lengths that are uniform to within less than 5%, it is necessary to regulate the behaviour of individual capillaries. Simple non-invasive capacitance measurements have proved able to provide a detailed characterisation of the slug flow and actuators to manipulate flow rate and slug size are the subject of ongoing investigations.