Electronic controlled multi stage counter-current micro extractor in the flow range of 1 to 10 ml min-1

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Abstract title	ELECTRONIC CONTROLLED MULTI STAGE COUNTER-CURRENT MICRO EXTRACTOR IN THE FLOW RANGE OF 1 TO 10 ML/MIN
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Introduction

Micro reactors are more and more used in chemical industry laboratories, as the continuous production accelerates the development of novel products. However, the workup of the reactor effluent is still done mostly batch wise and is now the bottleneck of product development. Because liquid-liquid extraction is one of the main workup operations, developing a multi stage counter-current micro extractor at a typical laboratory flow range of 1 to 10 ml min^{-1} will enhance the use of micro reactors for product development.

Micro extractor with pressure controlled pumps

The development of the micro extractor started with the design of the contactor for efficient mass transfer between the extract (water) and raffinate (heptane) phases. For a Taylor flow of water (dispersed phase) and heptane (1 to 1 ratio) in a 30 cm long and 1 mm wide Teflon tube, we observed liquid-liquid mass transfer coefficients of 0.1 to 0.7 s^{-1} for total flow rates of 1 to 10 ml min^{-1}. In this single channel contactor, the extraction of benzoic acid from water to heptane resulted in concentrations at 90% of the equilibrium value between the two phases.

After the mass transfer operation, the organic and aqueous phases were separated through a Teflon and a glass capillary. The capillary pressure of 400 to 600 Pa in these 0.2 mm high, 5 mm long and 10 mm wide slit shaped channels prevented liquid flowing to the incorrect exit (1).

The complete extraction device consists of three stages, connected in a counter-current flow direction (Figure 1). Each stage consists of a contactor and a slit shaped capillary separator. At the outlets of each separator, two piezo electric micro pumps control the respective flow rates. The pressure difference between the two outlets of the separator must not exceed the capillary pressure to prevent breakthrough. This pressure difference was measured and used as the input for the PID controller of the micro pump. This control loop successfully prevented breakthrough and undesirable back mixing of the phases.

A three stage counter-current extraction simulation was done using the observed mass transfer coefficients of the contactor. The model simulation results corresponded to the exit concentrations of both the extract and raffinate phases for the benzoic acid extraction from water to heptane (Figure 2).

Conclusion

Combining micro flow technology with micro electronics resulted in an extraction device which is ready to use as a continuous work up line as part of continous flow process in a micro reactor. The separation of the extract and raffinate phases is the most delicate as pressure disturbances of a few hundred Pascal will influence the performance. Pressure controlled micro pumps eliminate these disturbances for flow rates up to 5 ml min^{-1} for each phase in a counter-current flow regime.

Acknowledgment

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References

(1) W.A. Gaakeer, M. H. J. M. de Croon, J. van der Schaaf and J. C. Schouten, Chem. Eng. J., 207-208 (2012) 440-444

Images

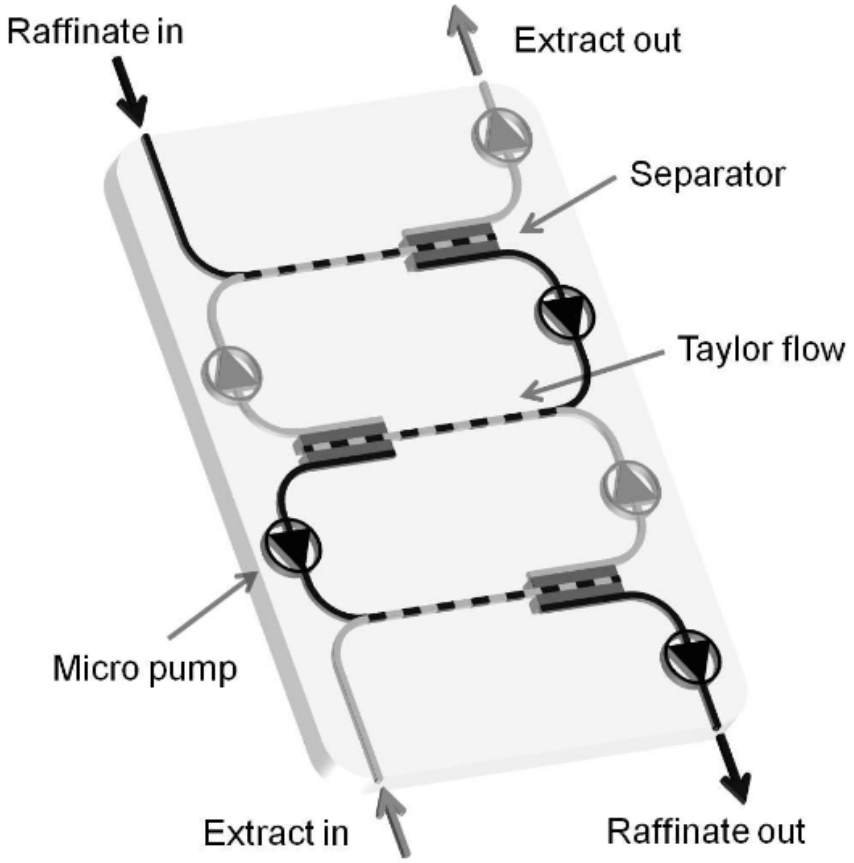

Figure 1: Flow scheme of three stage counter-current micro extractor

Flow scheme micro extractor

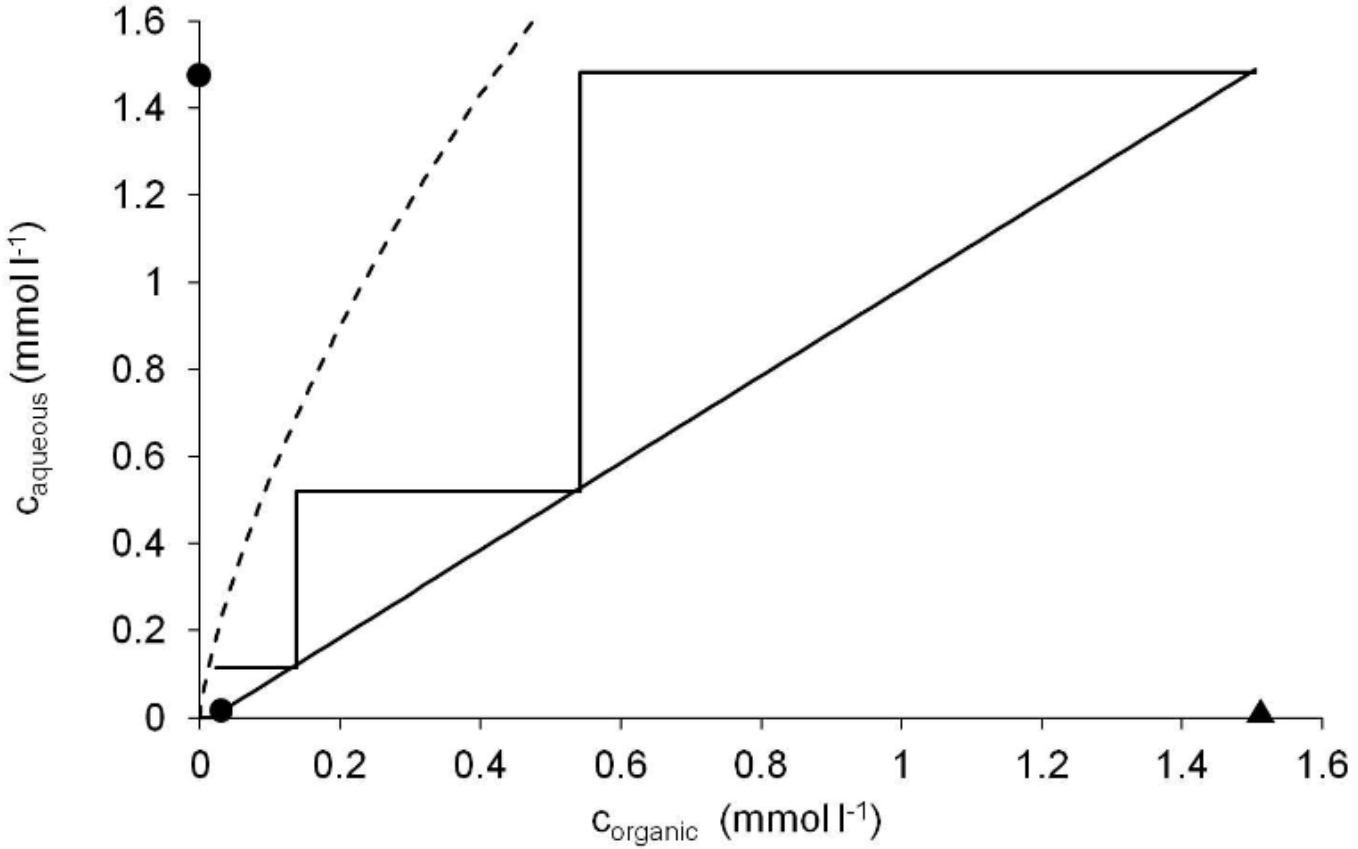

Figure 2: Simulated and experimental McCabe-Thiele diagram with extraction efficiency of 90% for 3 stage counter-current micro extractor. Measured start concentration in raffinate(▲) and end concentration in raffinate and extract(●)

Simulated and experimental McCabe-Thiele diagram

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