Milli-scale continuous extraction with coiled flow inverter connected to phase separator
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MILLI-SCALE CONTINUOUS EXTRACTION WITH COILED FLOW INVERTER CONNECTED TO PHASE SEPARATOR

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Abstract:
Process design intensification situated under the umbrella of Novel Process Windows heads for process integration with the coupling of flow devices and here most development is needed for flow separators [1]. We first investigated continuous liquid-liquid extraction in flow with a setup including a T-mixer, straight tube where partitioning takes place in slug flow and a membrane phase separator (FLEEX module from Syrris Ltd., UK). Maximum flow rate achievable was 1 ml/min. With the confirmation of good performance using model extraction systems, the setup was tested for metal catalyst separation. The use of different types of copper catalysts and metal scavengers were investigated together with variation of metal scavenger concentration and pH. Such analysis allowed to achieve excellent extraction efficiency up to 99%. The unit was then coupled to the copper-catalyzed azide-alkyne cycloaddition (click chemistry) reaction as shown in Fig. 1. Triazole product was obtained at a yield of 92% at 30 minutes residence time using 5% mol catalyst loading. Copper catalyst was efficiently removed and the level of contamination limit for APIs of 15 ppm was met in a single stage of separation [2].

The vision now is to achieve such multi-step synthesis in flow on pilot scale. Scale-up of separation is rare and thus conducted here. The coiled flow inverter (CFI) was considered as the right tool to work at higher flow rates. It was tested already for pilot plant scale with tube internal diameter of 10 mm allowing for a liquid flow rate up to 2000 l/h [3]. The high performance of CFI is documented for single phase and gas-liquid mixing [4]. Thus, we report hereby for the first, to best of our knowledge, the use of the CFI for immiscible liquid mixing. In the scale-up setup, a CFI with tube internal diameter of 3 mm and tube length of 210 cm is used (Fig 2.). Phase separation is achieved with membrane flow separator (PTFE 0.5 μm pore size) or slit-type flow separator (separation based on wettability with glass and Teflon slit shaped capillaries). The setup is tested for EFCE recommended model extraction systems and extraction efficiency above 90% is achieved which gives performance close to thermodynamic equilibrium. Compared to straight tube CFI performs about 15% higher. Currently, good operation (maximum 20% breakthrough in separator) with flow rate of up to 120 ml/min (7.2 l/h) is attained. This equivalent to 50 m3/y i.e. with 10 parallel units container-type capacity is achieved. We like to point out that the microfluidic pattern of the slug flow does not change, even at such pilot scale. This setup will be further tested for continuous homogeneous metal catalyst separation in flow with the aim to couple to fine chemical synthesis.


Highlight 1: Continuous extraction unit is developed for up to 1 ml/min and tested for metal scavenging
Highlight 2: Scale-up setup developed with coiled flow inverter (3mm ID) connected to phase separator
Highlight 3: Extraction efficiency for two model systems were > 90% and up to 7.2 l/h was achieved