



A robust multistage mesoflow reactor for liquid–liquid extraction for the separation of Co/Ni with cyanex 272



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ABSTRACT

Solvent extraction is a key principle for separations of components often requiring relatively large separation times due to large droplet size distributions. A continuous mesoflow reactor setup was evaluated for solvent extraction and phase separation in terms of efficiency of Co/Ni separation with cyanex 272 as extractant in a kerosene mixture. Several mixer types have been evaluated for the solvent extraction capability combined with a thin film coalescer/separator, expediting separation times, resulting in a settling volume that was only 1–2 times the volume of the mixer effectively reducing total residence time compared to classic mixer-settlers, where the ratio is 4–8 times higher. This was mainly attributed to the droplet homogeneity generated in the mixer, resulting in a short separation time. The thin film coalescer partitions in the settler expedited the phase separation further reducing the required settler volume. This paper shows that continuous mesoflow reactor setups can be utilized for small-scale liquid extractions with narrowly defined droplet sizes, enhancing the phase separation time, making it a viable screening utility. It also shows that the useful volume is much higher than those for conventional mixer-settler with a phase-separation-volume-to-equilibrium-volume ratio of 1–2 compared to 4–8 for the conventional mixer-settler expediting parameter screening.

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1. Introduction

Purification of a mixture of metals in solution is the second step in the refinement of metals occurring in the hydro-metallurgical industry. This is referred to as solvent extraction, wherein two immiscible liquid phases are contacted, between which the metals partition themselves over both phases. A discussion and experimental evaluation regarding the energy dissipation in microflow reactors and the resulting droplet size and mass transfer rate influence was recently presented by our group [1]. From these mixers, several mixers have been selected for a case study of the separation of Co/Ni (Cl^- in aqueous phase) with cyanex 272 in kerosene (organic phase). The results are compared with batch processes and with several other mixer types.

Solvent extraction is an alternative to component separation when distillation or crystallization are considered to be impractical, implemented in both medical and industrial applications. Conventional solvent extraction is performed in continuous stirred tanks in series (CSTR), batch vessels, centrifugal extractors or membrane extractors [2–6]. All except the latter types are relying on the dispersion of one liquid into another liquid to facilitate mass

transfer across the interface. When the extraction is performed under the form of a reactive extraction the extraction rate is generally accepted to be limited by mass transfer rather than reaction rate in case of reactive extraction [7,8]. To expedite the extraction rate, the interface between the two phases should be maximized. The only way this can be done in the case of dispersions is by decreasing the dispersed phase droplet size. To achieve smaller droplets, the liquids have to be mixed vigorously to generate a small droplet size. By doing so, due to difference in shear rate (close and far from the mixer axle) the droplet size distribution is large, resulting in a major fraction of micron sized droplets whilst other droplets are still in the mm-scale, resulting in less optimal conditions for extraction [9]. A significant problem arising with decreasing droplet size is the increase in phase separation time. The phase separation is in most cases defined by the difference in density in these reactor types. To achieve a full separation the smallest droplets have to phase-separated as well. The rate at which the droplets migrate due to density differences is expressed as the terminal velocity, which scales with $\sqrt{4gd(\Delta\rho)(3\rho_{cont}C_{drag})^{-1}}$ in case of low Reynolds numbers (less than 2). The drag coefficient is linear to d , resulting in a relation of terminal velocity with d^2 while the surface-to-volume ratio is proportional to d^{-1} [10,11]. Since in these reactor types a large droplet size distribution is common, the terminal velocity of the smallest droplets is the rate

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Nomenclature

k_l	mass transfer rate constant (m/s)	P	pressure (bar)
a	specific area ($\text{m}^2 \text{m}^{-3}$)	ε	energy dissipation rate (W/kg)
E	extraction efficiency (%)	\dot{m}	mass flux (kg/s)
C	species concentration (mol/m^3)	μ	dynamic viscosity (Pa s)
Q	volumetric flow rate (ml/min)	ρ	density (kg/m^3)
V	volume (m^3)		

determining step of phase separation, requiring larger settler/mixer volumes [12,13]. The cyclone extractor compensates the need for large settler volumes by maximizing the angular velocity, but the velocity is still proportional to d^2 [14]. It is a more efficient method of phase separation for large droplets but the smallest fraction typically still has to be separated in a secondary separator (cutoff size).

To omit the need for phase separation a lot of research has been conducted towards membrane extractors in the form of flat sheet membrane extraction and hollow fiber extraction units [15–17]. By utilizing these reactor types, the membrane acts as a phase barrier allowing for intense liquid–liquid contact without dispersion, thus making a subsequent phase separation obsolete. The main drawback lies within the fact that the membrane performance is restricted by the limited stabilizing capability of the achievable Laplace pressure. This has been thoroughly investigated in flat membrane extractors and in hollow fiber membranes [15,16].

Coming back to dispersive extraction, the most efficient way to perform the extraction and separate the phases is by increasing the density difference or by confining the droplet size distribution to a narrow range. Such a narrow control of the droplet size distribution cannot be achieved in classical setups. Intensification of a chemical process aiming at more effective chemistry and energy transfer goes hand in hand with the miniaturization of chemical reactors. These mesoflow reactors are carefully defined reactors containing a number of specific features such as pillars, obstructions and orifices, using the high surface-to-volume ratio and the short length scales of the mesoflow reactors. Especially in the industry these reactors are currently used for rapid experimentation, leading to shortened product development cycles [18].

These reactor types rely on geometrical features for dispersion, often resulting in a narrower droplet size distribution [19,20]. At low flow rates the CFR can be operated in a segmented flow regime, resulting in instant phase separation but limited throughput [19–28]. When increasing the flow rate the transition from segmented to dispersive flow has been observed and studied extensively [29–31].

Several CFR mixer types have been evaluated in combination with a settler that utilizes wettability of a solid interface in the settler as coalescer, which results in a significant reduction in total residence time in single and a multistage setup. This setup has been evaluated in a single stage and multistage mode for Co/Ni (Cl^-) separation with saponified cyanex 272 in kerosene.

The results obtained in this paper demonstrate that exploiting the beneficial properties of process intensification by size reduction yields a faster solvent extraction rate and enhanced the phase separation rate, thanks to a more precise droplet size distribution control, which is often limiting when using conventional setups.

2. Experimental methods

2.1. Reagents

The organic phase is comprised of C12–C14 alkanes (escaid 110 – Shell) with cyanex 272 from Cytex Inc (50 wt%), as extractant.

The organic phase was saponified for a molar fraction of 60% by addition of 10 M NaOH addition. The aqueous phase contained 35 g/l Co and 9 g/l Ni in chloride medium (industrial feed) which was replenished on daily basis to prevent precipitation.

2.2. Mesoflow reactor designs

The customized vortex reactor Fig. 1a is inspired by a concept suggested by Ansari et al. [32], known as centrifugal mixers and features cylinders with off center top/bottom outlets and has an internal volume of 1 ml [2]. The fluids were contacted in the reactor by a 2 mm wide T-junction to reduce inlet mixing effects. The connecting channels between the cylinders were 1 mm wide and 1 mm in height. The cylinders had a radius of 1.5 mm and a height of 3 mm. The device was constructed from 4 layers of 1 mm tick poly-methyl methacrylate sheets (PMMA, Eriks-Baudion, Hoboken, Belgium) that were milled with a computer numerical control (CNC) milling robot (Datron, NH, USA), using carbide tools (Datron, NH, USA). This reactor will be referred to as the vortex reactor throughout this article.

The LTF-MX reactor, from Little Things Factory (LTF, Elsoff, Germany), features discrete crossing slits which facilitate liquid contact by the split-and-recombination, as illustrated in Fig. 1b. In this reactor, the two fluids make contact as they enter the extraction unit. The internal volume of the reactor is 0.2 ml and the height in the mixing compartment is (2×0.125) mm, whilst the width of this compartment is 10 mm (see Table 1).

The separation of the organic and aqueous phase was facilitated by an in-house developed settler with a serpentine design enforcing droplet coalescence on the surface and with an angular acceleration in bends (Fig. 2). The settler has a thin film contacting zone (0.3 mm depth) alternated by a settling zone (3 mm width). The entire volume of the settler is 25 ml. The settler was built from

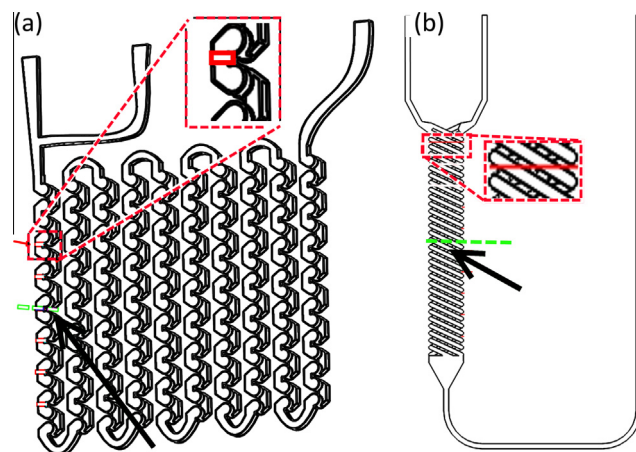


Fig. 1. Reactor designs: with several areas zoomed in and the 35 μl point marked with an arrow (a) vortex reactor – in-house developed reactor (total volume 1 ml – volume of 1 repetition unit: 5.3 μl). (b) LTF-MX reactor (total volume 0.1 ml ml – volume of 1 repetition unit: 7.0 μl).

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