Multiphase mixing performance characterization of oscillatory baffled reactors applied to the hydrometallurgical separation of cobalt and nickel

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Highlights

- Separation of cobalt and nickel using 3D-printed continuous oscillatory baffled reactors
- Near equilibrium extraction and stripping in continuous process in less than one minute.
- High-speed imaging and droplet velocimetry confirming flow reversal and adequate radial mixing

1. Introduction

The European Union's Green deal has set the ambitious goal of 55% reduction in greenhouse gas emissions by 2030. A decisive step to meet this goal, is a transition to electric vehicles (EVs). This requires the production of an enormous amount of batteries, for which the availability of a high purity grade of metals such lithium, cobalt, manganese, and nickel is indispensable. This purity is often obtained through solvent extraction, a process where metal complexes are separated based on their difference in solubility in an aqueous feed and immiscible organic solvent. The transfer of metals complexes from the aqueous to the organic phase requires an intense mixing to create droplets that increase the interfacial area available for mass transfer. In this work, a 3D-printed oscillatory baffled reactor is investigated to create these dispersions and perform the hydrometallurgical separation of cobalt and nickel.

2. Methods

Cobalt(II) sulphate heptahydrate and nickel(II) sulphate hexahydrate were dissolved in ultrapure water. Cyanex 272 diluted in kerosene was used as the organic phase. The extractant was partially pre-neutralized by dissolving sodium hydroxide in the kerosene. The concentration of Cyanex 272 in kerosene was optimized in batch experiments to maximize the separation factor. Several concentrations of sulfuric acid were tested in batch and all yielded almost complete stripping of cobalt and limited stripping of nickel. A relatively low acid concentration was selected to reduce the consumption of chemicals. Analytical characterization was performed using a PerkinElmer Optima 8300 ICP-OES. The oscillatory baffled reactor was produced using a Formlabs Form3+ stereolithography printer in combination with the FLGPCL04 Clear resin. The 3D printer allowed to rapidly and cost-efficiently produce transparent custom-made designs. A Photron FASTCAM Mini UX100 was used for high-speed imaging. Images were pre-processed using GIMP and further analyzed with the PIVlab toolbox in MATLAB [1].

3. Results and discussion

The continuous set-up comprised of a 3D-printed oscillatory baffled reactor (OBR), an oscillator, and two pumps to supply each of the two phases. Five different flow rates were employed (always A/O 1:1) which resulted in residence times between 9 and 73 seconds for the extraction experiments. Since batch experiments indicated that the stripping was slower, two identical OBRs were placed in series to double the residence time at equal flow rate. Three different oscillation amplitudes and frequencies were selected and investigated in a full factorial experimental design. Without any oscillations, a parallel flow profile was present and the blue color of the cobalt complex in the organic phase was only observed at the interface. Even at low oscillation intensities where the flow remained parallel, the organic phase turned into a homogeneous blue color. This indicated the presence of mixing within the phases. At the lowest amplitude, the frequency had only a limited effect on the percentage extraction, which increased linearly with residence time as shown in Figure 1. This was due to the low oscillatory Reynolds number

(< 500), too low to create any dispersion (illustrated in inset of Figure 3). For the highest amplitude on the other hand, droplets were being created for both 1.0 and 2.0 Hz. Their respective size varied between 400-800 µm and 350-400 µm. The significant increase in interfacial area due to the creation of the droplets clearly had an effect on the extraction of cobalt. A percentage extraction of 80% was obtained within 9 seconds for the highest amplitude in combination with 2.0 Hz (fully dispersed). Within a residence time of 18 and 36 seconds, 92 and 97% extraction was obtained, respectively, as compared to the equilibrium of 99% in batch. The aqueous and organic phases from the extraction experiments were collected and additional shaking was performed to ensure that equilibrium was attained. The phases were separated and the organic liquid was subsequently used in the stripping experiments. Results from the stripping experiments were comparable to those from extraction with the exception that a higher stripping yield was obtained when the oscillatory Reynolds number was the result of a higher amplitude. This difference between stripping and extraction is somewhat surprising, but can be caused by the difference in interfacial tension and the need of two reactors connected in series because of the inherently slower kinetics of the stripping. For several oscillation conditions droplets were present that enabled to track the motion of the liquid. The resulting velocimetry data corresponded closely to the theoretical values and confirmed the presence of flow reversal as shown in Figure 2. A large standard deviation was obtained which was to be expected because of the turbulent character of the flow. Lastly, while never the aim of the current work, calculations of the space time yield and productivity indicated a performance among the highest in its field.

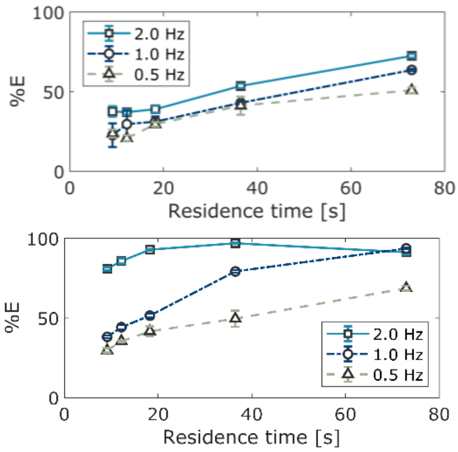

Figure 1. Percentage extraction as a function of residence time for (a) a low and (b) a high oscillation amplitude.

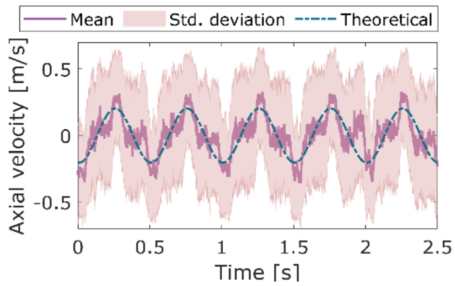

Figure 2. Axial velocity profile.

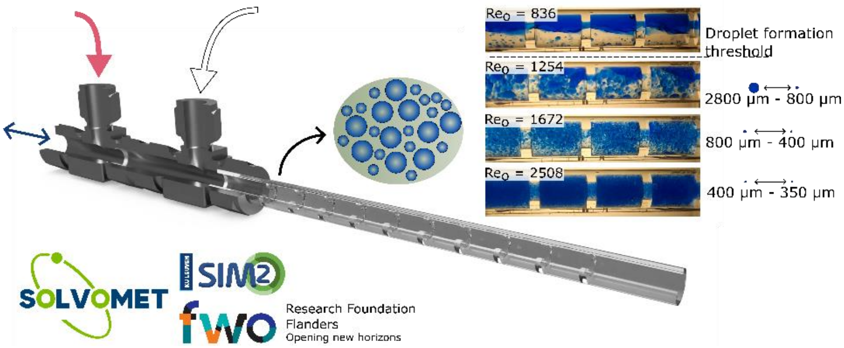

Figure 3. Digital render of the OBR along with color images at different oscillation intensities.

4. Conclusions

A 3D-printed oscillatory baffled reactor was successfully applied to perform the hydrometallurgical separation of cobalt and nickel. Near equilibrium extraction and stripping were demonstrated in a continuous process with a residence time of one minute. Multiphase mixing was further investigated with high-speed imaging and droplet velocimetry. This allowed to determine the droplet size and confirm the presence of full flow reversal and satisfactory radial mixing.

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Keywords

Continuous oscillatory baffled reactor; multiphase flow; process intensification; hydrometallurgy.