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SisClever: High Purity and Enrichment in a Single Process for Multicomponent Mixtures

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ABSTRACT

The SisClever process is presented. This is an acronym for "SImultaneous Separation of multi-Component systems to high LEVEl of Recovery", which refers to the concentration and purity of the products. The process concept is based on the control of the pH along a counter-current extraction process in hydrometallurgy. This control influences the net flowrate of the components to be separated. The net flowrates are controlled such that the components are guided to individual accumulation points along the process. There, they are recovered at high purity and concentration with organic withdrawal side-streams. This process concept has been validated by simulations based on reactive equilibrium stages and experiments with quasi-continuous conditions. The findings indicate that the concentrations in the product-withdrawal streams are determined by the flowrate of the side-streams, while the flowrates along the primary process can be maintained at a constant level. The findings of the study demonstrate that it is possible to attain high purities. A patent application has been filed for the SisClever process. First results with an 18-stage mixer-settler battery, which is currently under construction, are expected by November 2025.

INTRODUCTION

In hydrometallurgical processes for the processing of ores and in the context of urban mining, achieving high purities of individual metals and high concentrations simultaneously with minimal effort is challenging, particularly when individual metals are present in comparatively low concentrations. The SisClever process is designed to address these challenges.

In a counter-current process with an aqueous feed stream $\dot{L}$, e.g., an acid pregnant leach solution, and an extractant stream $\dot{G}$ the net flowrate $\dot{m}_i$ of any component i depends on its extraction factor λ_i:

$$\dot{m}_i = \dot{G}Y_i - \dot{L}X_i = \dot{L}X_i\left(\frac{\dot{G}Y_i}{\dot{L}X_i} - 1\right) = \dot{L}X_i(\lambda_i - 1), \quad (1)$$

where the extraction factor is the ratio between the capacity flowrates of both phases, i.e. the flowrate ratio multiplied with the ratio of equilibrium mass concentrations of the phases, Y_i and X_i. If λ_i exceeds unity, the net flowrate of the component considered is oriented in the direction of $\dot{G}$, if it is less than unity, in that of $\dot{L}$. Should the value of λ_i equal unity somewhere along the process, the component will accumulate at that specific point and can be withdrawn with one of the phases at high concentration.

The fundamental concept underlying the SisClever process is the variation the partition coefficient K_i with

$$K_i = \frac{Y_i}{X_i} \tag{2}$$

along the process by adjusting the pH such that for each component $\lambda_i - 1$ changes sign at a specific position, at which point an accumulation zone for that component occurs. At that point a small side-stream of extractant phase is withdrawn from which the respective metal enriched in that accumulation zone is stripped ideally with a single equilibrium stage. In the ideal case, at each accumulation zone only one metal partitions into the extractant phase, so that high purities can be reached. The stripped extractant phase can then be returned to the SisClever process directly behind the withdrawal point so that the overall flowrate ratio along the process does not change. In principle, the flowrate ratio along the process can be varied, which has a corresponding effect on the extraction factor. This also allows, for instance, changing the pH by means of dilution by adding water to the aqueous phase along the process.

The concentration of a metal in the product stream then depends on its feed composition and the flowrate ratio between feed stream and side-stream withdrawal. A decrease in the withdrawal stream results in an increase in product concentration. The enrichment is, naturally, constrained by the extractant capacity and solubility limits. Consequently, a high enrichment does not necessitate extreme flowrate ratios in the primary process, as is the case for a conventional hydrometallurgical process configuration. The SisClever process is particularly well-suited for minor components in feed solutions with highly disparate metal concentrations.

The SisClever process thus allows SImultaneous Separation of multi-Component systems to high LEVEl of Recovery, namely concentration and purity.

A schematic comparison between a conventional hydrometallurgical process and the SisClever process is shown in Figure 1. In a conventional hydrometallurgical process (see Figure 1a), each metal is extracted, scrubbed and stripped with an individual extractant cycle, where the extractants can differ between the different metals present in the feed. In the SisClever process (see Figure 1b) a single extractant is used that passes through the entire process. This allows combining the loading of the extractant with one metal with the scrubbing of the next along the process. Along the SisClever process one metal after another is thus accumulated, enriched in the organic phase, and withdrawn at its accumulation point. In the ideal case the organic product side-streams, in which each metal is rather pure and highly concentrated, are then stripped in a single equilibrium stage. Thus, the number of required equilibrium stages can be almost halved as compared to the conventional approach.

METHODOLOGY

Simulations

In order to validate the SisClever concept, a simulation tool was developed in Fortran. This tool allows solving the different equilibria, taking the chemical equilibria for the reactive extraction of all components into account. The competition of the different metals for the extractant is thus properly described. It is assumed that the pH-profile is specified, and the corresponding equilibria are evaluated in the counter-current process. The balances connecting the equilibrium stages are solved

by considering the transient transfer of fluxes between the stages. Consequently, a steady state is eventually achieved.

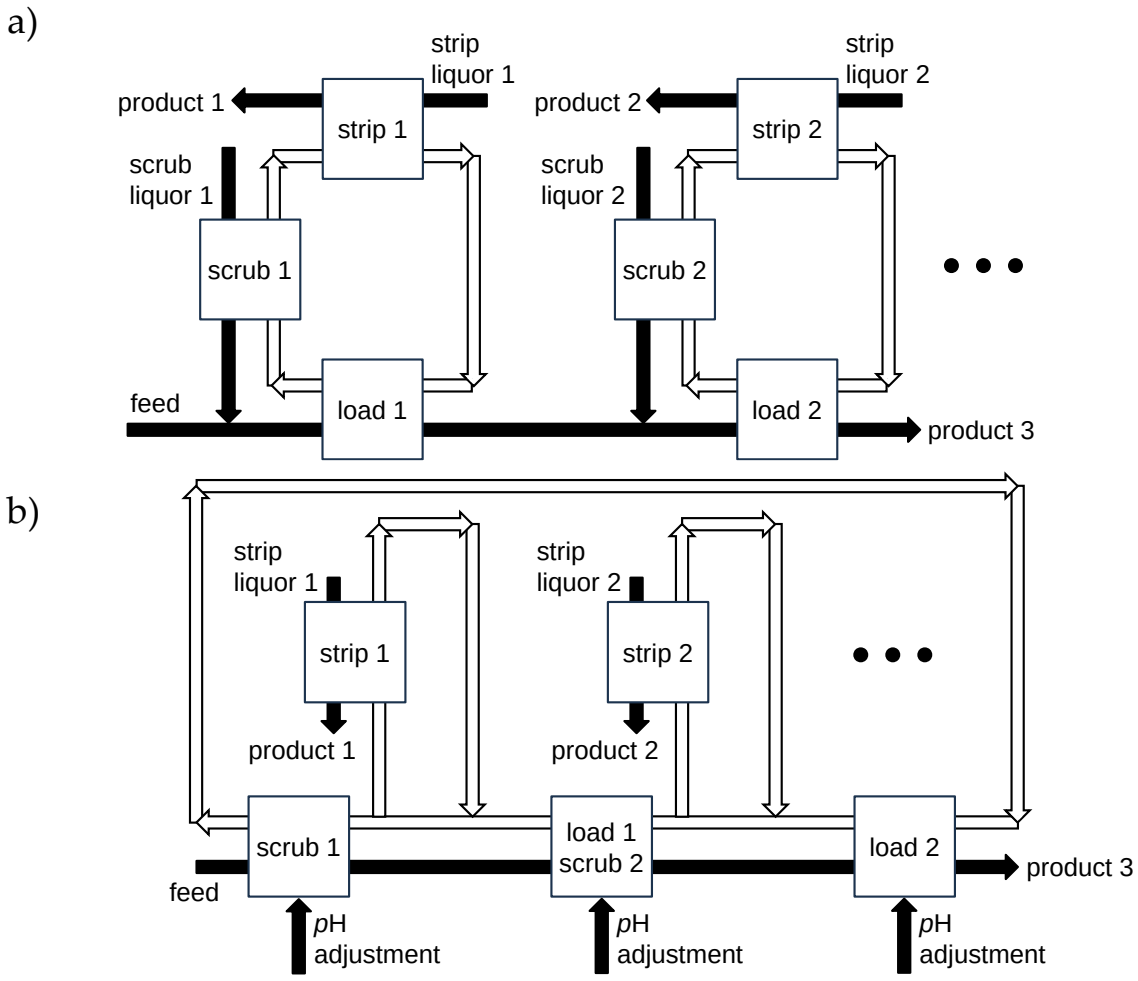

Figure 1 Schematic comparison of a conventional hydrometallurgical process (a) (Sole, 2008) to the SisClever process (b) for the extraction of 2 metal species, where the black arrows indicate aqueous and the white arrows organic phases

Based on the simulation for a given pH-profile, an optimization of the pH-profile is realized with a simplex approach. The objective functions can be defined in a flexible manner, such as reducing the number of base injections or minimizing the cost accounting for user-defined prices for acid, base, and products. Given that an arbitrary pH profile can only be maintained by the addition of acid and base throughout the process, where, in the ideal case, only base would be required, the minimization of acid addition can also be incorporated into the objective function. This typically results in the avoidance of any acid addition in the final optimized case.

The simulations demonstrate the effectiveness of the SisClever process, as outlined in the subsequent section.

Experimental Validation

To validate the SisClever process concept experimentally, a quasi-continuous process was realized with the help of 250-ml beakers. Each of the 13 beakers was filled with 50 ml of each phase for the separation of Zn^{2+} and Co^{2+} with D2EHPA dissolved in ELIXORE 205. The initial concentrations were selected to represent the steady-state results of the simulations, as illustrated in Figure 2. The equilibrium constants and stoichiometric coefficients for reactive extraction of both metals have been taken from previous studies, where slope analysis was applied (Singh, 2023, Bart & Slater, 2001).

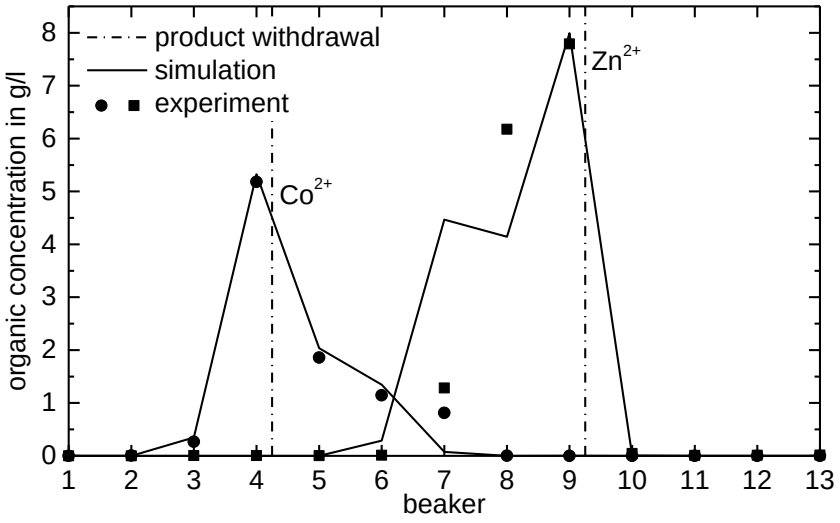

Figure 2 Initial concentration profile of Co^{2+} and Zn^{2+} along the SisClever process obtained from simulation (full lines) compared to final experimental results obtained by ICP (points)

In the following steps, 10 ml was withdrawn from each phase with the aid of syringes and subsequently transferred to the neighboring beaker according to the counter-current flow of both phases. Then, the two-phase systems were stirred at 650 rpm for five minutes, after which the pH was adjusted to the profile shown in Figure 3, which is also the result of the optimization, by adding small quantities of 5 M HCl or 5 M NaOH solutions. The aqueous feed was realized by the addition of 10 ml of an aqueous solution containing 0.53 g/l of Co^{2+} and 0.80 g/l of Zn^{2+}. The organic feed was realized by the addition of 10 ml of 10 vol.-% D2EHPA dissolved in ELIXORE 205. At the product-removal points at stages 4 and 9, 1 ml of the organic phase was withdrawn and collected, and 1 ml of fresh extractant phase was added. The procedure was repeated 25 times so that for each phase and each beaker, 250 ml have been exchanged, corresponding to five residence times of each phase. The final concentrations were determined by ICP after re-extraction of the organic phase.

ANALYSIS AND DISCUSSION OF RESULTS

As demonstrated in Figure 2, the final results closely mirror the initial concentration profile, thereby indicating that a steady state has been achieved in the experiment. The agreement between the simulation and the experiment also validates the simulation tool. It is noteworthy that the

concentrations of the product streams are ten times higher than the feed concentrations for both components. This finding corresponds well with the ratio between the feed and product-withdrawal volumes, thereby demonstrating that the enrichment achieved through the SisClever process is indeed defined by that ratio. The product streams exhibited purities of 99.88% for Co^{2+} and 100.00% (exactly 99.9981%) for Zn^{2+}, with a mere 13 equilibrium stages.

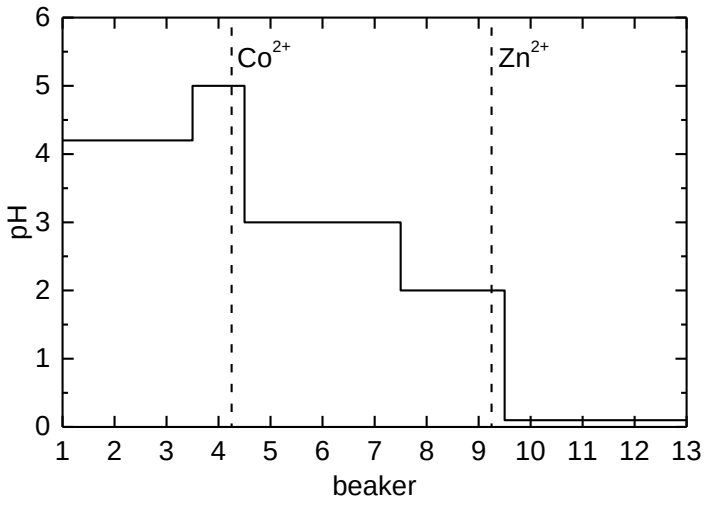

Figure 3 pH profile obtained from optimization, realized in the quasi-continuous experiment

CONCLUSIONS AND OUTLOOK

The results of the simulations and quasi-continuous experiments demonstrate the feasibility of the SisClever process, as outlined in the introduction. The SisClever approach facilitates achieving elevated purities and concentrations from multicomponent feed solutions. The utilization of a single extractant in the SisClever process demands that the extraction equilibria be uniformly distributed across the pH range in an ideal scenario. This establishes an optimization objective for novel extractants that diverges from those for conventional extractants. When the equilibria of several components are too similar, the components are withdrawn as a combined product at the respective accumulation point. Subsequent separation of these mixed products, potentially employing a different extractant, would be necessary. While the SisClever process may not be a universal solution to all separation problems, it is nevertheless believed that it may be beneficially applied in many cases.

The SisClever process has been patented (Pfennig, 2025), and an 18-stage mixer-settler battery is currently under construction for the purpose of validating the SisClever process concept in continuous operation. The first results are expected by November 2025.

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NOMENCLATURE

$\dot{G}$	organic flowrate
K_i	partition coefficient
$\dot{L}$	aqueous flowrate
λ_i	extraction factor of component i
$\dot{m}_i$	net flowrate of a component
X_i	concentration of component i in the aqueous phase
Y_i	concentration of component i in the organic phase

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