SisClever: Pure and Concentrated Products From Multicomponent Mixtures – Applied to Urban Mining

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The SisClever Process facilitates the simultaneous separation of multicomponent systems, achieving high concentrations and purities of individual products. A notable application is the hydrometallurgical separation of metals using cation-exchange extractants, where the pH is systematically varied along the counter-current process. The process is modeled based on phase equilibria and validated through experiments separating Zn^{2+} and Co^{2+}. The efficiency of the process is demonstrated for the separation of a complex mixture of rare-earth metals, where only roughly a third of equilibrium stages is required compared to an optimized proposal from literature. Possible applications in recycling, generalization to other unit operations, as well as limitations are discussed.

Keywords: High concentration, High purity, Hydrometallurgy, Multicomponent separation, Recycling

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1 Introduction

Efficient processes for the separation of components from multicomponent waste streams are essential for achieving a circular economy and minimizing the extraction of primary raw materials. In hydrometallurgical processes, the objective is to obtain pure metals from ores or to recover them from waste. Typically, each valuable component is separated with an individual separation step, leading to complex separation schemes [1, 2]. Simplifying such a sequence is desirable.

In a general counter-current process in steady state, which can be subdivided conceptually into a succession of theoretical stages, where in each equilibrium is reached for the exiting streams, the direction in which any component i is moving on average depends on λ_i, which, depending on the process considered, is called, e.g., extraction or stripping factor:

$$\lambda_i = \frac{\dot{G}}{\dot{L}} K_i \tag{1}$$

where $\dot{G}$ and $\dot{L}$ are the streams in the counter-current process and K_i denotes the equilibrium partition coefficient:

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

where Y_i and X_i are the equilibrium concentrations of the $\dot{G}$ and $\dot{L}$ streams, respectively. Here, the streams are either the overall molar or mass flow rates and the compositions correspond to mole or mass fractions. Alternatively, the flow rates are those of the carrier component(s) of each stream and the compositions refer to the loading, defined as the amount of substance or mass of a transfer component relative to the amount of substance or mass of the respective carrier component(s). In the context of solvent extraction, the carrier components are typically water for the aqueous phase and the extractant for the organic phase. This results in $\dot{G}Y_i$ and $\dot{L}X_i$ being the partial flow rates of component i with the respective stream. The average net flow rate of component i is then:

$$\dot{f}_i = \dot{G}Y_i - \dot{L}X_i = \dot{L}X_i\left(\frac{\dot{G}Y_i}{\dot{L}X_i} - 1\right) = \dot{L}X_i\,(\lambda_i - 1) \tag{3}$$

which can be expressed for amount of substance or mass. It is apparent that with $\lambda_i > 1$ component i will on average move with $\dot{G}$ while for $\lambda_i < 1$ it will move on average with $\dot{L}$. Typical separation processes are thus designed such that for some component(s) $\lambda_i > 1$ while for some other(s) $\lambda_i < 1$, so that the component(s) can be separately recovered at either end of the process. For a feed with two components to be separated both can be recovered with an arbitrary

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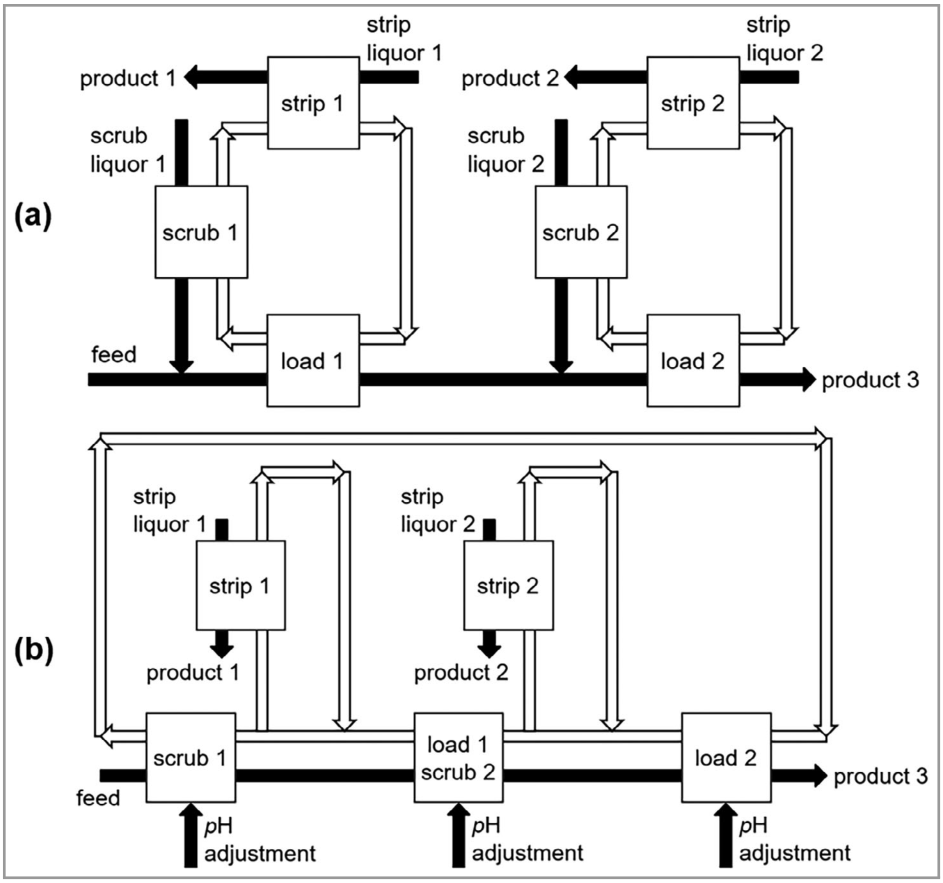

Figure 1. Schematic representation of (a) conventional processes in hydrometallurgy, taken from [3], and of (b) the SisClever process. The aqueous streams are in black and the organic streams are in white.

purity relative to the respective other component by adjusting the number of theoretical stages of the process. For more complex mixtures only one component can be recovered essentially pure, while the other stream exiting the process is recovered as mixture of the remaining components. Alternatively, both exiting streams can be mixtures of several components, where the cut is defined by $\lambda_i > 1$ for one group of components exiting the process at one end while $\lambda_i < 1$ for the other leaving at the opposite end.

An interesting case results, if $\lambda_i = 1$ or $\lambda_i - 1 = 0$ at a certain point in the process. In that case, component i will have zero average flow rate at that specific point, implying that it will accumulate there. To maintain steady state, it is necessary to withdraw the component at that particular point with a side-stream. It should be noted that no assumption has been made about the nature of the streams up to this point. Consequently, the considerations apply to any steady-state counter-current process.

2 Application

The considerations above can be efficiently applied, if λ_i is varied along the process, either by varying the flow rates, e.g., by introducing or removing side streams, and/or by varying the partition coefficient. The latter can, e.g., be realized by varying the pH in reactive extraction with a liquid cation-exchange extractant in hydrometallurgy. After a first acid leaching step, the pregnant leach liquor typically contains a range of dissolved metals. Since the dependence of extraction equilibrium on pH generally varies for different metals, a pH-profile along a counter-current reactive-extraction process will lead to $\lambda_i - 1$ switching sign at different positions along the process for the different metals present in the multicomponent feed. By systematically adjusting the pH along the process, each metal can be strategically guided towards an individual position where $\lambda_i - 1$ switches sign. This approach enables the accumulation of the metals at these specific points, facilitating their withdrawal at high concentrations and purities.

To illustrate the distinctions between a conventional hydrometallurgical metal-separation process and the SisClever process based on this concept, a schematic comparison is presented in Fig. 1. Each of the boxes in Fig. 1 corresponds to a particular process step, which is accomplished through a series of equilibrium stages, which can be realized as mixer-settlers. In the conventional process, each metal is separated using an individual cycle of extractant. Consequently, each metal undergoes a distinct extraction and scrubbing step. In contrast, the SisClever process employs a more streamlined approach, where the extraction of metal 2 occurs concurrently with the scrubbing of metal 1. This process offers a significant advantage, allowing up to 50 % reduction in the number of stages typically required in conventional extraction processes.

Furthermore, when the objective is to attain a high metal product concentration, conventional processes necessitate a low strip flow rate due to the overall balance. Consequently, achieving a high concentration requires a high organic-to-aqueous flow-rate ratio in the stripping section, which is challenging to operate and control. In contrast, the SisClever process only requires the flow rate of the product-removal side stream to be small to achieve high concentration since each metal is removed essentially exclusively at its accumulation point, while the overall flow-rate ratio along the main separation process can be chosen in a convenient range. In principle, the concentration can be arbitrarily high, although this is limited by other effects, such as solubility limits or extractant loading capacity.

In conventional processes, each metal is obtained using a dedicated extractant. In contrast, the SisClever process utilizes a single extractant phase. Consequently, the objective of developing new extractants for the SisClever process differs from that of the conventional approach. While conventionally, an extractant is optimized to remove a specific component from a mixture, the equilibria for all desired products should exhibit a substantial difference in the SisClever process, favoring not just the extraction of a single component.

The product purities are predominantly influenced by the number of theoretical stages between two adjacent accumulation points for the respective metals, which can be adjusted to reach a desired purity. It is therefore possible to achieve essentially arbitrary purity and concentration individually for an arbitrary number of components in a single separation process. This approach differs significantly from the conventional design of such processes and may offer substantial advantages, some of which will be detailed below. This paper first presents the experimental validation of the simulation tool predicting process behavior, then the application of the process model to the example of a complex case of rare earth element separation.

Because of its advantages, this is called the SisClever Process: SImultaneous Separation of multi-Component systems to high LEVEl of Recovery, namely, concentration and purity.

3 Validation

To validate the performance of the SisClever process, a simulation tool to describe and optimize this process has been developed in Fortran. This tool takes into account the underlying reactive extraction equilibria and is based on an equilibrium-stage approach. A dynamic approach is used to model the path of metals along the process over time until steady state is reached. Equilibrium is calculated at each time step between the exiting streams of each theoretical stage. A metal ion $M_i^{m_i+}$ with a charge m_i+ is extracted with an extractant like D2EHPA (di-(2-ethylhexyl)phosphoric acid, $(HR)_2$) according to:

$$M_i^{m_i+} + n_i\overline{(HR)_2} \rightleftharpoons \overline{M_iR_{m_i}(HR)_{2n_i-m_i}} + m_iH^+ \qquad (4)$$

where the overbars indicate components in the organic phase. D2EHPA is known to form dimers in organic solution, which is accounted for in Eq. (4). The result of optimizing the pH-profile so as to minimize chemical consumption was compared to experiments for a simple example of separating Co^{2+} and Zn^{2+}. The chemicals as specified in Tab. 1 were used without further purification. The parameters of Eq. (4) were obtained from slope analysis of equilibrium experiments reported in previous studies [4, 5].

Table 1. Chemicals used and suppliers.

Component		Supplier
Zinc sulfate heptahydrate	$ZnSO_4{\cdot}7H_2O$	VWR
Cobalt(II) chloride hexahydrate	$CoCl_2{\cdot}6H_2O$	Roth
Di-(2-ethylhexyl)phosphoric acid	D2EHPA	Sigma Aldrich
Hydrochloric acid	HCl, 5 mol L^{-1}	Fisher Chemicals
Sulfuric acid	H_2SO_4, 95–97 %	Sigma Aldrich
Sodium hydroxide	NaOH	Sigma Aldrich
ELIXORE 205		Total Energies

The experimental validation of the SisClever process was realized in 250-mL beakers, between which quantities of both phases were exchanged in a quasi-continuous counter-current mode. Each beaker contained 50 mL of each phase, which were initially prepared with concentrations and pH according to the optimized steady-state simulations. In each transfer step, 10 mL of each phase were first withdrawn from all beakers using plastic syringes. The syringes used for collecting the aqueous bottom phase were equipped with short Viton tubes that allowed passing through the upper phase. The bottom phase was collected by first sucking air into the syringe and then ejecting it after positioning the tip of the Viton tube in the bottom phase to avoid undesired collection of the upper phase. In a subsequent step the 10 mL of each phase were transferred to the neighboring beakers in counter-current fashion. At the inlets 10 mL of the aqueous feed solution and pure extractant phase were introduced, while at the outlets 10 mL of the corresponding phases were collected in separate beakers. The product removals at the accumulation points were achieved by extracting 1 mL of organic phase per transfer step from the corresponding beakers using plastic syringes. To compensate for the withdrawal to mimic a constant flow rate of organic phase, 1 mL

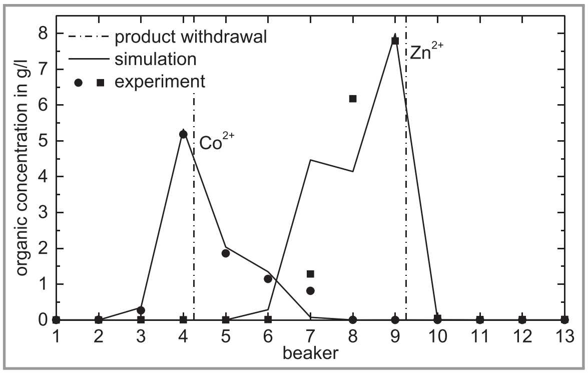

Figure 2. Experimental results of purification of Co^{2+} and Zn^{2+} with the SisClever process utilizing an extractant phase of 10 vol. % D2EHPA dissolved in ELIXORE 205 at 16 °C compared to results of an optimized simulation.

of pure extractant phase was added to the corresponding beaker.

For each transfer step, first equilibrium was achieved by stirring the phases in the beakers at 650 rpm on a Variomag Multipoint 15 multi-stirrer from Fisher Scientific for 5 min. Subsequently, the pH was adjusted to the optimized value obtained from the simulation by adding small quantities of 5 M HCl or 5 M NaOH solution. The pH was measured with a R3620 pH-meter (Consort) equipped with an InLab Semi-Micro-L pH-sensor (Mettler-Toledo).

A total of 25 transfer steps were performed resulting in the transfer of 250 mL of each phase, which corresponds to five residence times per phase in a beaker. The results obtained via ICP-MS (ICAPS 6500, Thermo Fisher) for the organic phase are shown in Fig. 2, where back-extraction with 2 M H_2SO_4 at a 5:3 aqueous-to-organic volume ratio was applied. The vertical dot-dash lines indicate the stages between which the product streams are withdrawn. These data are compared to the initial concentration profiles, which correspond to the steady-state result of the simulation. The simulated steady-state concentration profiles are essentially identical to those obtained after five residence times. This suggests that the simulations accurately represent the process. The observation that there was hardly any change in the concentration profiles during five residence times indicates that steady state has been achieved.

Comparison of the final concentrations in the product streams to the initial feed concentrations of 0.53 g L^{-1} for Co^{2+} and 0.80 g L^{-1} for Zn^{2+} indicate an enrichment factor of 10. This finding corresponds well with the withdrawal flowrate being only 1/10 of the feed flow rate. These results demonstrate the effectiveness of controlling the concentrations of product side streams by adjusting their flow rate. The results also illustrate that high purities can be obtained with 99.88 % for Co^{2+} and 100.00 % for Zn^{2+} with merely 13 equilibrium stages. The purity of Zn^{2+} at its removal point is extremely high but properly reported, as the concentration of Co^{2+} measured by ICP-MS in the aqueous phase obtained after re-extraction of the organic side stream is only 0.07 mg L^{-1}, which is above the detection limit of 0.002 mg L^{-1} for Co^{2+}, from which a purity of 99.9981 % results in the organic phase.

Table 2. Ion charges, stoichiometric coefficients, and reaction-equilibrium constants.

Component	n_i	m_i	$\log_{10}(K_{c,i})$	Source for n_i
Y	2.0	2.9	0.809	[6, 7, 8, 9]
Gd	3.0	2.0	1.058	[10, 11]
Sm	3.0	2.1	0.432	[12, 13]
Nd	1.0	1.9	−2.413	[6, 14]
Pr	2.0	1.5	−0.543	[9, 15]
Ce	2.5	1.9	−0.744	[16, 17]
La	2.0	1.2	−0.769	[9, 15, 16]

4 Application to a Challenging Example

After experimental validation of the process concept and of the simulation tool, the SisClever process model is now applied to a practical example of a recycling case. The SisClever process's capabilities are illustrated here through the challenging separation of rare-earth metals from a coal-based source. A 104-stage separation process was developed by Srivastava [2] for this purpose. The present study follows a similar approach, as the optimization is also based on modeling. The equilibria are described in this study via reaction-equilibrium constants and stoichiometry:

$$K_{\mathrm{c,i}} = \frac{c_{\mathrm{M}_i\mathrm{R}_{m_i}(\mathrm{HR})_{2n_i-m_i}}\, c_{\mathrm{H}^+}^{m_i}}{c_{\mathrm{M}_i^{m_i+}}\, c_{(\mathrm{HR})_2}^{n_i}} \tag{5}$$

where c_i are the molar concentrations of species i. As in [2], the extractant was D2EHPA at 0.152 mol L^{-1} along with the modifier tributyl phosphate (TBP) at 0.364 mol L^{-1} dissolved in Orfom (C13 to C16 isoalkanes). The values employed for the ion charge, the reaction-equilibrium constant, and the stoichiometric coefficients are collected in Tab. 2. The values of the stoichiometric coefficients have been obtained from the literature as indicated in Tab. 2. The other values have been determined by minimizing the deviation in $\log(K_{c,i})$ for those experimental values given in [2], where the degree of extraction lies between 0.05 and 0.95 and for Y between 0.01 and 0.99 because of the limited number of data for that species.

It should be noted that only a single charge is fitted per metal ion and that no speciation is currently considered in the equilibria modeling. For this reason, the metal charges are not +3 as is usually the case for these rare earth elements, but represent an average charge at which the metals are extracted, enabling proper fitting of the pH-dependence of the experimental extraction results. Since speciation is

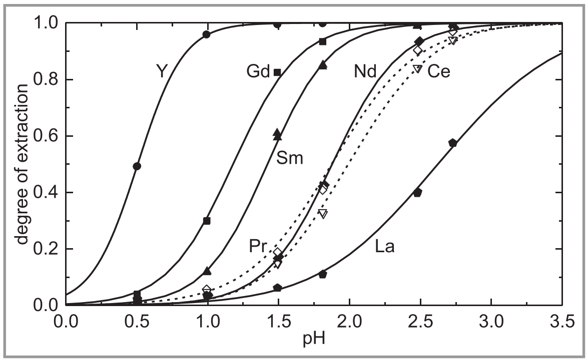

Figure 3. Comparison of experimental and modeled equilibrium data for the example application; data taken from [2].

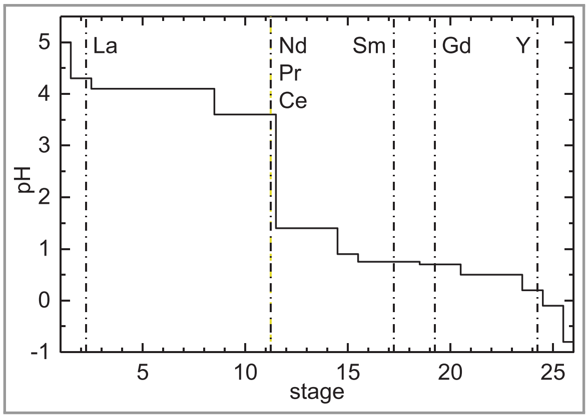

Figure 4. pH-profile along the SisClever process for example 1. Volume flow-rate ratio O/A = 4:1; fraction of organic stream withdrawn at respective removal point: La = 5 %, Ce/Pr/Nd = 16 %, Sm = 5 %, Gd = 5 %, Y = 3 %.

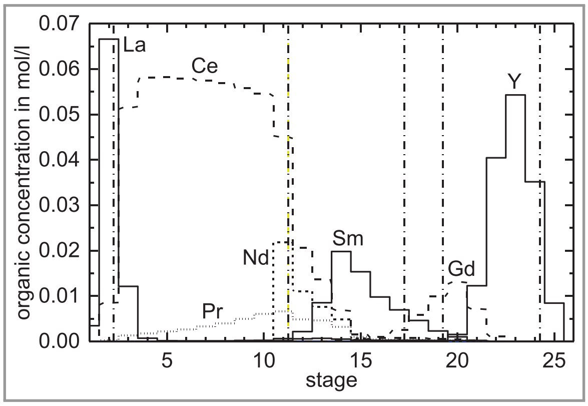

Figure 5. Concentration profiles for example 1 along the SisClever process. Conditions identical to Fig. 4.

also only effectively accounted for in [2], this is a fair comparison. The results shown in Fig. 3 demonstrate that with this approach the equilibria can properly be described.

In a first step, the goal was to minimize the number of theoretical stages for identical feed composition and extractant specification while meeting the same purities and concentrations as optimized by Srivastava [2]. To reach this goal, the pH-profile along the process was adjusted together with the flow rates of the organic phase in the main process and of the product withdrawals. The stripping of the metals from their organic removal streams has been realized at a flow-rate ratio of 10, which is also the limiting flow-rate ratio in [2]. In each stripping step, the organic stream is mixed with an acidic solution in a single step, and it is assumed that 100 % re-extraction is achieved for all metals present in the organic side stream. Metal concentrations in the final aqueous product streams are therefore 10 times higher than those in the corresponding organic withdrawal stream.

The regenerated extractant is re-introduced into the process to maintain the major flow-rate ratio constant along the process. Also, since Srivastava based his optimization on simulations of equilibrium stages, this is a fair comparison. The result is presented in Tab. 3 as example 1 and in Figs. 4 and 5. While Srivastava's approach requires 104 theoretical stages, the SisClever process requires only 26 stages to separate the components into five withdrawal streams. These streams can then be stripped individually with one stripping stage each. Consequently, a total of 31 stages – less than a third of the stages proposed in [2] – is sufficient to achieve products that are identical or even superior. Notably, the concentrations of metals in several product streams are considerably higher than those reported by Srivastava [2].

Table 3. Comparison of SisClever products with those of Srivastava [2].

Metal	Feed	Srivastava [2]		Example 1		Example 2	
	Conc. [mmol L^{-1}]	Purity [%]	Conc. [mmol L^{-1}]	Purity [%]	Conc. [mmol L^{-1}]	Purity [%]	Conc. [mmol L^{-1}]
Y	4.3	99.5	9.7	99.6	351.8	99.6	352.6
Gd	2.5	72.6	6.8	73.7	98.7	92.6	308.2
Sm	2.3	39.7	1.5	62.8	69.6	87.8	293.3
Nd	14.1	28.4	216.6	29.4	218.7	24.8	174.7
Pr	4.4	8.4	65.9	8.9	65.9	7.7	54.2
Ce	30.8	56.7	445.5	60.6	450.9	54.5	384.2
La	13.6	85.4	6.1	88.4	666.2	99.9	343.9

As was the case in [2], it was not possible to completely separate the components Nd, Pr, and Ce, since under the conditions that were selected their equilibria with the extractant phase are too close. These three metals are therefore withdrawn in a single removal stream. To achieve their separation, it would be necessary to use a different extraction agent, or to modify the concentration of D2EHPA in the extraction phase so that extractions of these three metals take place at different pH. In both cases, this could be achieved in a single process by

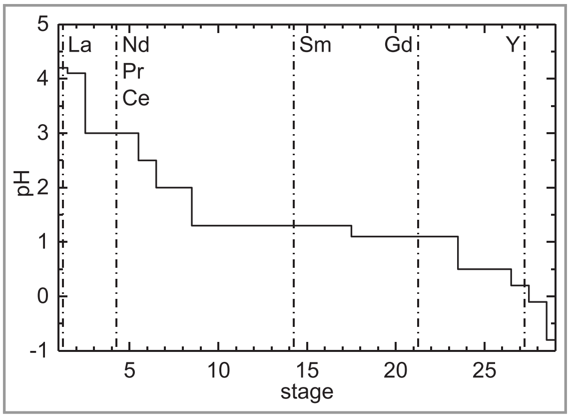

Figure 6. pH-profile along the SisClever process for example 2. Volume flow-rate ratio O/A = 4:1; fraction of organic stream withdrawn at respective removal point: La = 5 %, Ce/Pr/Nd = 20 %, Sm = 1 %, Gd = 2 %, Y = 3 %.

applying the SisClever concept, where the metals would accumulate around different pH values.

In a second test, the goal was to increase the purities of the products to as much as possible while keeping a high enrichment. The other boundary conditions are identical to example 1. The results of a corresponding process are given in Tab. 3 as example 2 as well as in Figs. 6 and 7. For example 2 in the SisClever process, 29 theoretical stages are required and 5 re-extraction stages. Thus, a total of 34 equilibrium stages are sufficient with the SisClever process, i.e., with only a slight increase in number of theoretical stages, which is still significantly smaller as by Srivastata [2], much higher enrichments and purities can be achieved.

5 Conclusions

It has been demonstrated that by controlling λ_i along the SisClever process it is possible to separate several components simultaneously with high concentrations and high purities. The process concept and modeling tool were validated by a pseudo-continuous experiment, and the model was applied to the case of rare earth separation to compare the advantages of the SisClever process with a conventional separation route taken from the literature. The next step in the development of the SisClever process is experimental validation with a continuous mixer-settler battery.

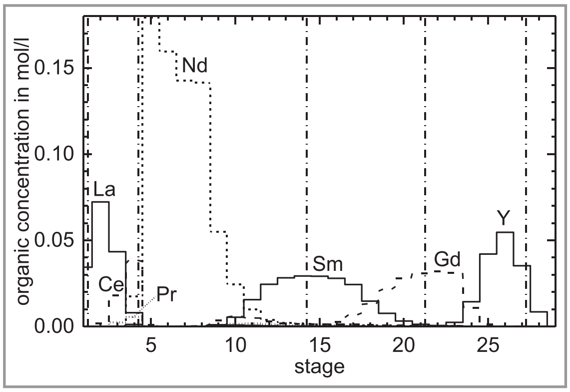

Figure 7. Concentration profiles for example 2 along the SisClever process. Conditions identical to Fig. 6.

For the challenging example applications, it has been demonstrated that the number of theoretical stages can be significantly reduced and, concomitantly, higher purities and enrichments can be attained when compared to the conventional approach. One reason for the reduced number of theoretical stages is that in the conventional arrangement, where typically one component is removed in each of the separation steps, if some of that component is transferred to the next step, it will constitute an impurity in the product(s) of the successive step. The SisClever process, by contrast, circumvents the subdivision into individual separation steps, thereby enabling each component to progress through the entire process to its designated accumulation point. In addition, each step of the conventional hydrometallurgical process necessitates the extraction, scrubbing, and stripping of each metal individually. Conversely, the SisClever process utilizes the same equilibrium stages for the extraction of one component and the scrubbing of another.

In conventional metal extraction, achieving a high product concentration requires a large flow-rate ratio, which, beyond certain limits, poses significant challenges to process operation and control. In contrast, the SisClever process utilizes a reduction in the product-withdrawal flow rate without altering the flow rates in the primary process, thereby decoupling the flow rates in the main process from the product concentration. This renders the process particularly well-suited for components with such low concentrations in the feed that conventional processes requiring extreme flow-rate ratios would be uneconomical. The SisClever process offers the potential to enhance the purity and concentration of these components while demanding minimal additional effort and operating within reasonable flow-rate ratios, making it a promising solution for metal extraction processes.

Finally, it should be reiterated that in the fundamental presentation of the SisClever process concept reference has only been made to λ_i and especially not to the nature of the phases. Thus, application also in contexts other than hydrometallurgy may be possible, if the flow-rate ratio and/or the partition coefficient can be sufficiently varied. Examples can be the addition of antisolvents in extraction or entrainers in absorption processes. This will be explored in the future.

While it is obvious that the SisClever process, for which a patent application has been filed, will not be able to solve every complex separation task, even in hydrometallurgy, it is believed that this process structure has significant opportunities in cases where, e.g., extractants are available that provide favorable equilibrium behavior supporting the realization of multiple accumulation points along the process.

The SisClever process is thus not an incremental optimization of conventional processes but a new process concept that allows to obtain many product streams in a single process, where in principle each purity is defined by the theoretical stages between adjacent accumulation points and the concentrations by the flow rate of the product side withdrawals.

Potential applications are not only rare-earth separation but also separation from complex mixtures, e.g., in recycling of Li-ion batteries of different types, even if the different types are mixed, and the recycling of valuable metals from electronic scrap. Similarly, anions can be separated using anion-exchange extractants. Thus, the SisClever process may help closing the recycle loops in a circular economy by facilitating efficient urban mining.

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Symbols used

c	[mol L^{-1}]	concentration
$\dot{f}$	[kg s^{-1}] or [mol s^{-1}]	net flow rate of a species, positive direction is in the direction of flow of the G-phase
$\dot{G}$	[kg s^{-1}] or [mol s^{-1}]	flow rate of the G-phase stream
K	[–]	partition coefficient
K_c	[$(\text{mol}l^{-1})^{m_i-n_i}$]	chemical reaction equilibrium constant
$\dot{L}$	[kg s^{-1}] or [mol s^{-1}]	flow rate of the L-phase stream
m	[–]	net charge of the extracted metal ion
n	[–]	stoichiometric coefficient of the reactive extraction with D2EHPA
X	[–]	concentration in the $\dot{L}$-stream
Y	[–]	concentration in the $\dot{G}$-stream

Greek letters

λ	[–]	extraction or stripping factor

Sub- and superscripts

c	chemical reaction
i	species index

Abbreviations

SisClever	SImultaneous Separation of Multi-Component systems to high LEVEl of Recovery

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