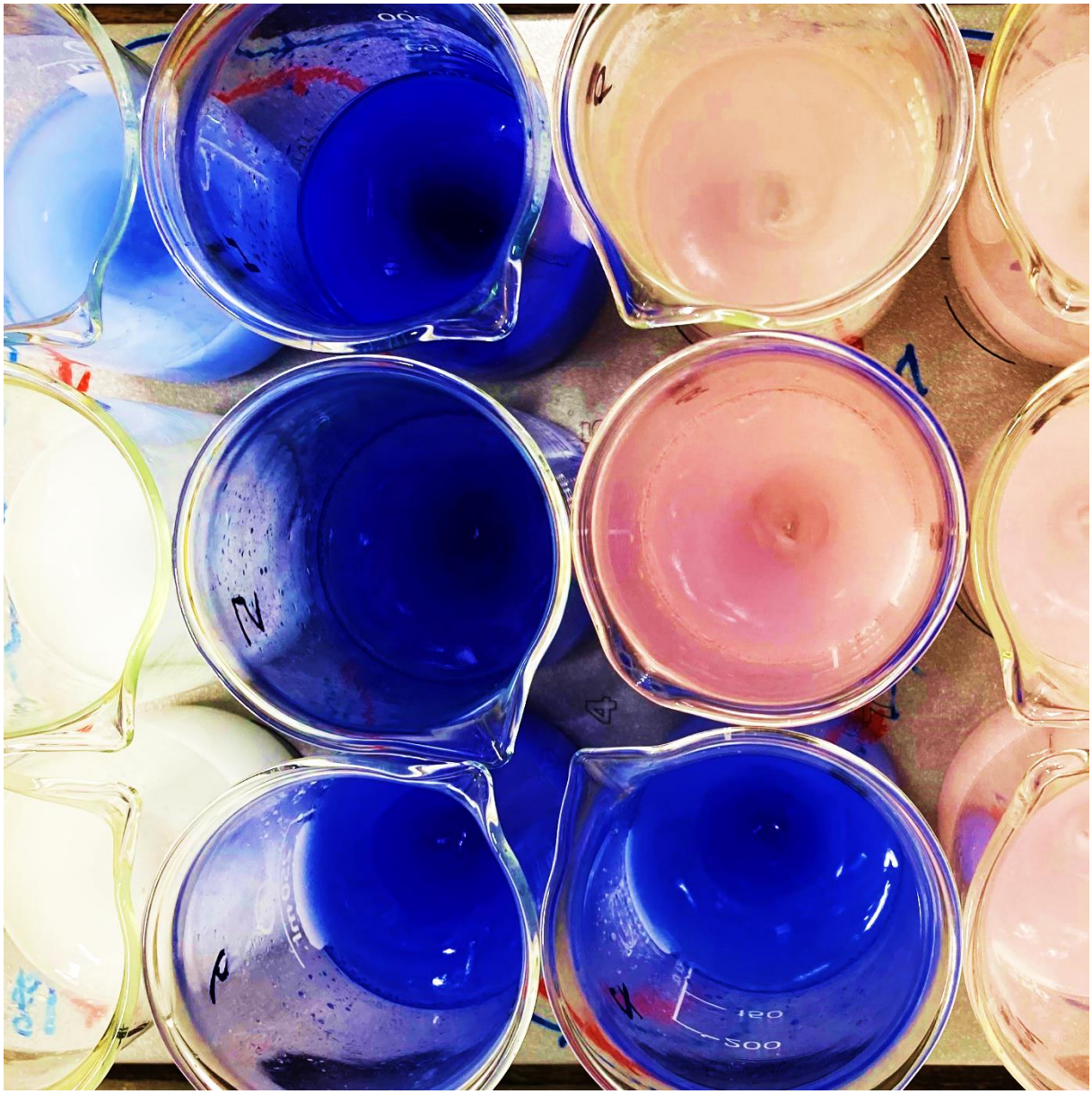
LIÈGE université

PEPs CHEMICAL ENGINEERING

Separation and Purification



2024

Content

title photo: Marc Philippart de Foy - experimental validation of the SisClever process

Dear Reader

We have been waiting for quite some time now to be able to talk about our new proposed process for efficient recovery of individual components from mixed streams. The application examples we are looking at are hydrometallurgical processes for the recycling of used Li-ion batteries, Nd magnets, and electronic scrap in general. Together with the university, we have applied for a patent for this process, which we call the SisClever process (SImultaneous Separation of multi-Component systems to high LEVEl of Recovery). While in principle the process can be viewed as a tightly coupled sequence of binary separations, the strength of the process only unfolds, if the process is viewed as a single process with free exchange of components along the entire process. In this way, it is possible to quantitatively separate many components in a single continuous process and to define the purity and the product concentration of all individual product streams. We are actually happy to receive funding from the university to validate the SisClever process in a sufficiently large mixer-settler battery. Accordingly, this annual report will begin with a description of this process, simulations, and experimental validation.

Also this year, the collaborations with the Sirindhorn International Thai-German Graduate School of Engineering (TGGS) at the King Mongkut's University of Technology North Bangkok (KMUTNB) in Thailand and with the Industrial University of Ho Chi Minh City in Vietnam have continued to develop. For the cooperation with TGGS we progress to link the experimental findings in Bangkok with the drop-based simulations of liquid-liquid phase separation that had been developed by David Leleu. The work on the process proposed for the recycling of phosphorous-containing wastewater sludges in Vietnam is approaching finalization, since Vu Dinh Khang plans to submit his Ph.D. thesis that summarizes the results beginning of next year.

Activities with Scientists for Future continued this year, again on Zukunftsbilder.net and with presentations and workshops on sustainability at various events.

2024 was once again a year full of diversity, of which we try to give an impression in this annual report.

So: Enjoy reading!

Andreas Pfennig

SisClever Process, a New Separation Process for Multi-Component Mixtures: Concept and Modeling

Marc Philippart de Foy, Andreas Pfennig

Introduction

The recycling of critical metals is essential to reach sustainability. The Circular Economy Action Plan established by the European Union aims to support metals recycling in order to reduce dependence on primary raw materials (European Commission, 2024).

Hydrometallurgy is a typical route for metal recycling, where metals are first dissolved in an aqueous acid, then separated using organic solutions in which they are complexed with an extractant. Conventional processes consist of several steps, in which conditions such as *p*H and extractant used differ. Each step recovers one metal, so that several steps are required to handle multi-component mixtures. This route is limited in terms of the flexibility and scalability of the process with regard to the composition of the waste processed, which generally varies over time in e.g. electronic scrap recycling (Lakshamanan, 2016).

In conventional processes, the concentration of metals recovered is defined by the flowrate ratio of the phases used. Using a low flowrate in the receiving phase enriches the component relative to its initial concentration in the feed. At the same time, the operation of liquid-liquid extraction equipment becomes more complicated as the phase ratio increases. Components present in small quantities are therefore difficult to recover at high concentrations.

A new process has been developed to meet these challenges.

SisClever Process

The SisClever process for Simultaneous Separation of multi-Component systems to high Level of Recovery is a single process in which each transfer component can be recovered at high concentration and purity. The idea is to control the partition coefficient of the various transfer components throughout the process, so as to guide them to specific points in the process, different for each component, where they accumulate and from which they can be recovered.

A schematic representation of this counter-current process and the behavior of a transfer component *i* is shown in Fig. 1. In case of metal recycling by reactive extraction, an aqueous and an organic phases are the two main streams, referred to as $\dot{L}$ and $\dot{G}$ respectively.

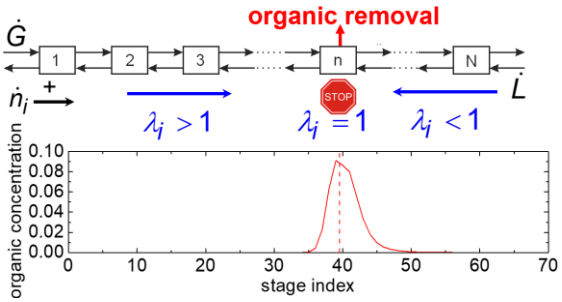


Fig. 1: Schematic representation of the SisClever process for one component

In a counter-current process, the net flowrate of a component *i* at any point in the process is

$$\dot{n}_i = \dot{G} Y_i - \dot{L} X_i \tag{1}$$

using the notations in Fig. 1, where X_i and Y_i are the loadings of the component in the two phases. The net flowrate is defined as positive when the component moves on average with the *G*-phase. The extraction factor for component *i* is then defined as

$$\lambda_i = \frac{\dot{G} Y_i}{\dot{L} X_i} = \frac{\dot{G}}{\dot{L}} K_i \,, \tag{2}$$

with the partition coefficient K_i. This gives for the net flowrate of component *i*

$$\dot{n}_i = \dot{L} X_i \left(\lambda_i - 1 \right). \tag{3}$$

Thus, the net flowrate is positive when the extraction factor $\lambda_i > 1$, meaning that the com-

ponent moves on average with the *G*-phase. When $\lambda_i < 1$, the component moves on average with the *L*-phase. The idea is thus to control the partition coefficient of the component so that the component is guided to the point in the process where its $(\lambda_i - 1)$ changes sign. The component will accumulate at this point as it is pushed towards it from both sides of the process. A small side stream will allow to retrieve it at this point at high concentration.

In the reactive extraction of metal cations, the partition coefficient depends on *p*H. Thus, in view of Eq. (2), controlling the extraction factor involves controlling the partition coefficient by varying the *p*H. At low *p*H, metals remain in the aqueous phase, so the partition coefficient is low, while it increases with *p*H as the metals are extracted into the organic phase. To control the partition coefficient throughout the process so that the behavior shown in Fig. 1 occurs, a *p*H-profile is generated. On the right side of the process, where the acidic leach liquor is fed to the process, the *p*H is low, so the component moves with the aqueous phase, while the *p*H increases as the aqueous flow moves to the left. At a certain point along the *p*H-profile, the accumulation point for each component is then reached, beyond which the component moves back to the accumulation point with the organic phase.

The new process therefore enables a metal to be accumulated and recovered from a specific point in the process. The idea can be extended to a multi-component mixture. Since the *p*H-dependence of the partition coefficient differs between metals, the $(\lambda_i - 1)$ term for each metal changes sign at different *p*H-values. Thus, each metal accumulates at a different *p*H, and thus a different point in the process, from where it can be recovered. The separation of several components can therefore be achieved in a single process.

Modeling and application

For process optimization, a Fortran program was implemented simulating the process behavior. The user enters the *p*H-profile throughout the process, and the tool predicts the steady-state concentration profiles of all components. Acid and base consumption can also be assessed, including the acid or base injection required to generate the desired *p*H-profile, as well as the acid and base requirements for the leaching, stripping and precipitation steps of the process. An optimization of the *p*H-profile then allows to improve purities at removal points or to minimize acid and base consumption.

The simulation requires data on the extraction reaction. For cation-exchange extraction with D2EHPA, the general form of the reaction is

$$\mathrm{M}^{m_i+} + n_i\,\overline{(\mathrm{HR})_2} \rightleftarrows \overline{\mathrm{MR}_{m_i}\,\mathrm{HR}_{(2n_i-m_i)}} + m_i\,\mathrm{H}^+,$$

where overbars represent components of the organic phase. Here, M^{m_i+} is the metal cation, $(\mathrm{HR})_2$ is the free D2EHPA considered here as a dimer, $\mathrm{MR}_{m_i}\mathrm{HR}_{(2n_i-m_i)}$ is the metal complex formed in the organic phase and H^+ are the protons released when the metal is extracted from the aqueous to the organic phase by cation-exchange. The stoichiometry of the reaction and its equilibrium constant must be known to model the metal behavior throughout the process. These data are generally determined by slope analysis in the literature.

The Fortran tool has been applied to several metal recycling cases, such as the recycling of cobalt and lithium from Li-ion batteries, or the recovery of lithium from brines. The results obtained for the recycling of neodymium magnets after aqueous leaching are presented here, where only the main metal cations present in this type of waste are taken into account. Reaction stoichiometries and equilibria were assessed from the literature for each metal (Sahu, 1997, Mohammadi, 2015, Sarangi, 1999, Padhan, 2017).

The simulation tool was applied to minimize acid and base consumption, while using a limited number of stages to achieve the desired purities for all metals at their respective removal points. The optimum *p*H-profile for these aspects is shown in Fig. 2.

The steady-state organic concentration profiles achieved when the process is operated with this *p*H-profile are shown in Fig. 3. These profiles have been normalized to show the enrichment achieved in the process over the respective metal concentration in the aqueous feed. This shows that very high enrichment can be achieved in the process, since an enrichment factor of 20 is reached for four metals at their corresponding removal point, represented by a vertical dashed lines of the same color as the metals. The fifth metal present in the feed

is cobalt, which is the metal extracted at the highest *p*H among the metals present in the input stream. With the *p*H-profile shown in Fig. 2, Co passes through the process with the aqueous stream and is never extracted into the organic phase, as the *p*H of the aqueous outlet is chosen to be too low to extract Co. Co is thus recovered as a pure component in the main aqueous outlet. Of course, it would be possible to introduce a Co accumulation point within the process as well.

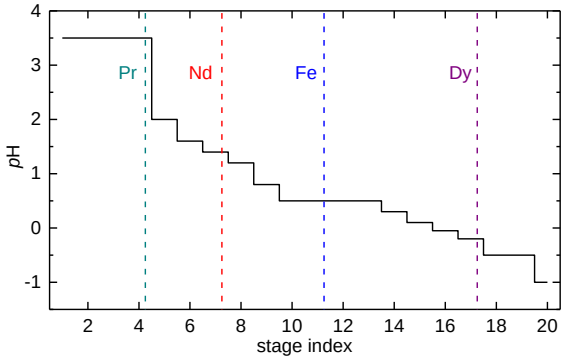


Fig. 2: Optimal *p*H-profile for Nd magnets recycling

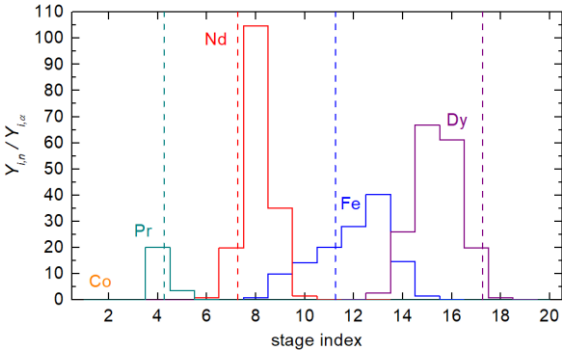


Fig. 3: Normalized organic concentrations profiles at steady state

It was thus found that with just 20 stages the 5 main metals present in neodymium magnet scrap could be separated in a single process. High metal enrichment was achieved, as the removal concentration is 20 times higher than the input concentration. The purity of each metal is high at the different removal points, as shown in Fig. 3. Dy is removed from the process with a purity of 97.3%. All other metals are recovered with purities above 99.8%.

Advantages of the SisClever process

Simulations have shown that the separation of several components is possible within the process. Experimental validation has also been carried out and is described in the contribution on page 6 of this annual report. The main advantages of the SisClever process can now be summarized.

Firstly, with the SisClever process, the separation of several components can be achieved in a single process, whereas a unit operation per component to be separated is generally required in conventional processes.

The components can be recovered with high purity and, in principle, arbitrary enrichment. In steady-state operation, the concentration reached at the removal point of each component is defined by the removal flowrate. Decreasing the removal flowrate allows to increase enrichment, which is limited only by practical considerations such as extractant availability or solubility. This also allows valuable components present in trace amounts in the feed to be recovered at a high concentration, thus increasing process income without additional operating costs. At the same time, the main flowrates passing through the process remain essentially unchanged, so that the operation of the equipment is not impaired by extreme phase ratios which would be required for high enrichment in conventional processes.

It is expected that the CAPEX for the SisClever process will be lower than for a conventional process and that higher purities can be achieved. In conventional processes at the process step to remove metal *i* some fraction of *i* may be leaving with the raffinate. This amount typically ends up in the extract of the next separation step to remove the subsequent metal. In the SisClever process, the removals for all metals are linked so that each metal is returned to the appropriate accumulation zone. In conventional processes, at each separation step, the metal is first extracted and then individually stripped/scrubbed. In the SisClever process, the zone for extracting one metal corresponds directly to the zone for stripping/scrubbing the next metal. Of course, these two aspects are linked, where in the SisClever process the purity and number of stages required can be freely optimized to meet the specific requirements.

Comparison of the OPEX shows that the OPEX of the SisClever process will be at most that of a conventional process, since the flowrate of base needed in the example is exactly that to shift the feed stream from its acidic

inlet state to the higher *p*H at the end of the process. This is exactly what is required in the conventional process as well. Due to the potentially higher concentration, the dilution effect in a conventional process can be avoided.

Another major advantage of the SisClever process is its flexibility with regard to feed composition. If feed concentrations vary over time, it is always possible to achieve constant removal concentrations by varying the removal flowrates. Additional components initially absent from the feed but appearing over time can be separated and recovered without a significant change in process operation by adding new removals between those already present.

Perspectives

To separate metal cations in the SisClever process, a *p*H-profile is generated. In the above example, the *p*H is increased along the aqueous flow. This induces the consumption of bases such as NaOH, which, in addition to representing a significant operating cost, generates salt that has to be treated afterwards. An alternative to this base consumption could be the use of an electrochemical *p*H-shift. This idea was e.g. applied to membrane-assisted reactive extraction by (Gausmann, 2020). A coupling of this idea with the SisClever process could replace NaOH consumption by the electrochemical generation of OH^-. Fig. 4 shows a potential configuration for this coupling, where the *p*H of the aqueous phase could be increased by an electrolysis cell passing from one stage to the next. This would allow base injections to be replaced by green electricity consumption, reduce the environmental impact of process operation, and support electrification of the recycling process. The H^+ generated in the second half cell can be used in the leaching of the metals from the input scrap.

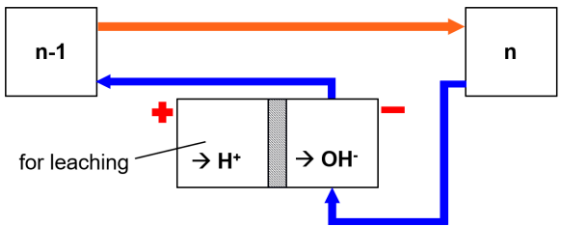


Fig. 4: Use of electrochemical *p*H-shift to adapt the aqueous *p*H

The SisClever process has been presented here for metal-cation recycling in hydrometallurgy, where the extraction equilibrium depends on *p*H. This new process idea is, however, general and can be applied to any counter-current process in which a parameter can be controlled to sufficiently influence the λ_i of the different transfer components and induce their separation, as presented in this work.

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Experimental Validation of the SisClever Process

Ezgi Uslu

Efficient recycling of valuable metals is critical to the sustainable use of resources, given their role in advanced technologies such as batteries and high-performance magnets. The SisClever process, which is described in detail on page 2 of this annual report, offers an efficient alternative to traditional multi-step extraction methods. To validate the SisClever process experimentally, it was run pseudo-continuously for the separation of zinc and cobalt.

Starting conditions have been chosen according to the steady-state concentration profiles for Co^{2+} and Zn^{2+} as obtained from Fortran-based simulation tool, also described in the contribution on page 2 in this annual report. The obtained *p*H-profile is shown in Fig. 1. This profile was optimized to reduce acid and base consumption and minimize the number of stages required. The optimization determined that 13 stages were required to achieve effective separation. The organic removal stages were strategically chosen based on the *p*H values at stages 4 and 9, which were identified as optimal for maximum extraction of cobalt and zinc, respectively. First, at lower *p*H, Zn accumulated around stage 9, while Co kept moving with aqueous phase. Around stage 4, at higher *p*H, Co accumulated. At these points, where the extraction factor equals one, efficient separation is achieved, ensuring the recovery of high-purity components.

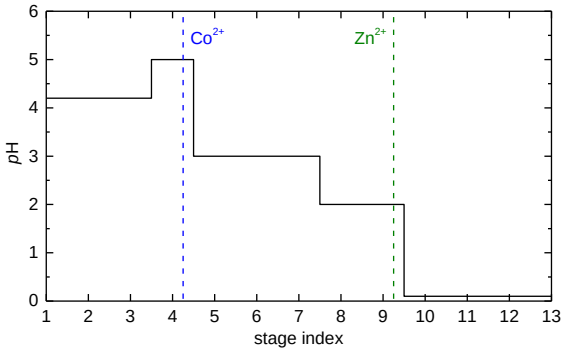

Fig. 1: Optimal *p*H-profile for Co-Zn separation obtained from simulation

All chemicals used in the validation experiments were analytical grade. Zinc sulfate heptahydrate ($ZnSO_4 \cdot 7H_2O$) and cobalt chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) were used to prepare aqueous solutions. The extractant di-(2-ethylhexyl) phosphoric acid (D2EHPA) was dissolved in Ketrul D80 to form the organic phase. The organic phase was composed of 90%-vol Ketrul D80 and 10%-vol D2EHPA. The aqueous feed contained metal concentrations of 0.53 g/L Co^{2+} and 0.8 g/L Zn^{2+}. The organic and aqueous phases were mutually saturated prior to the experiment and distilled water saturated with Ketrul D80 was used to prepare all the solutions. All experiments were performed at approximately 16 °C. The equilibrium *p*H for each stage was maintained throughout the experiment via adding of 5 M HCl or 5 M NaOH.

A quasi-continuous separation of zinc and cobalt in a counter-current setup was then carried out to effectively reproduce the behavior of a continuous process, analogous to operations conducted in a continuous mixer-settler battery. The experimental procedure involved mixing the phases until equilibrium was achieved, followed by settling, and subsequently partially transferring the phases to their next respective stage.

Each stage of the process was represented by a 250 mL beaker, as shown in Fig. 2. At each stage, 50 mL of aqueous phase and 50 mL of organic phase were mixed at 650 rpm for a minimum of 5 minutes, followed by a minimum settling period of 5 minutes. After the mixing and settling step, 10 mL from each phase which is 20% of the total volume per phase was collected using syringes from all the beakers. Viton tubes connected to the syringes were used to collect organic phase from the bottom part of the beakers. Then, both phases were transferred into the next respective stage. The flowrate ratio of both phases was thus 1. The aqueous phase flows from stage 13 to stage 1 while the organic phase flows countercurrently from stage 1 to stage 13.

Organic removal streams at stage 4 for Co^{2+} and stage 9 for Zn^{2+} were established by collecting 1 mL of organic phase after each mixing step, which was 10% of the main organic

stream. Due to the flowrate ratio, this was also equivalent to 10% of the main aqueous inlet stream. Based on the steady-state mass balance, a 10-fold enrichment in metal concentrations was thus anticipated at these removal points compared to the inlet. To ensure constant organic main flux, 1 mL of fresh organic phase was added to the stage following the partial removal.

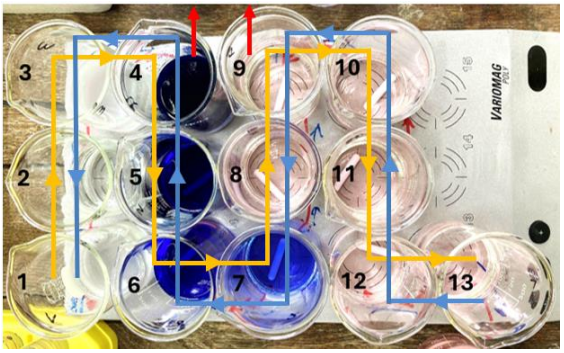

Fig. 2: Experimental set-up: aqueous stream flows shown by blue arrows, organic stream flows shown by orange arrows, and two partial organic removal streams are shown by red arrows

The process was operated for a total of 5 residence times, corresponding to 25 cycles, during which 250 mL of each phase were transferred. The final organic concentration profiles are compared with the predicted simulation results in Fig. 3.

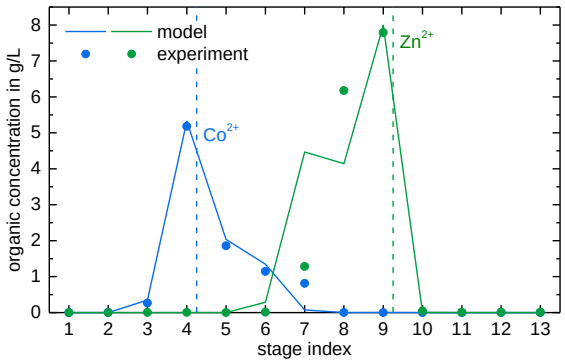

Fig. 3: Co and Zn concentration profiles in organic phase at the end of the experiment

Two distinct concentration peaks were observed, corresponding to the removal points of the metals. The simulated steady-state profiles, used as initial concentration profile, closely matched the experimental results. This proves that steady state was achieved at the end of experiments. The only minor deviation in zinc concentration was observed at stages 7 and 8, which may be due to the mutual interaction of the metals with D2EHPA.

Cobalt and zinc were recovered with purities of 99.88% and 100%, respectively. These results demonstrate the ability of the SisClever process to achieve high purity. Also, metal concentrations were obtained that achieved an enrichment factor of 10 relative to their input concentrations. This shows that high enrichment can be achieved even with lower inlet concentrations.

The findings confirm that the proposed process effectively separates zinc and cobalt from a mixed aqueous input, achieving high recovery rates by enriching the concentration and reaching high purity. The experimental results demonstrate the successful separation and recovery of cobalt and zinc from an aqueous feed phase.

In the future, the goal is to validate the SisClever process experimentally in continuous operation and to test the process control towards technical application of the process. A pilot-scale mixer-settler battery, which allows efficient mass transfer, will be put into operation. The setup will consist of 18 mixer-settlers MSU 0.5 supplied by MEAB Metallextraktion AB and will be equipped with *p*H and flowrate control to help maintain the system in a steady state, guided by simulation results. Process parameters will be controlled and monitored using LabVIEW, ensuring stability and adaptability during operation. Initial experiments will focus on two-metal systems such as Co-Zn and Co-Cu.

In a second step, the experiments will be extended to systems with up to four metals, as the modular design of the equipment provides operational flexibility to adapt the system to different metal-separation requirements. The selection of additional material systems for application of the SisClever process will be guided by industrial partners in the areas of Li-ion battery recycling and other precious metal mixtures e.g. for rare-earth metal separation.

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Force Model for Electrical-Driven Separation (EDS) Using High Voltage and Alternating Current of Biodiesel-Glycerol Mixture System

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Starting Point

Our research group has investigated the effects of a high-voltage alternating current (AC) on the separation of a biodiesel-glycerol mixture using a sharp conductive needle electrode employed in a point-to-point configuration to create a non-uniform electric field, shown in Fig. 1, as outlined in the 2023 annual report. This electric field facilitates the coalescence of glycerol droplets within the biodiesel phase and leads to a faster settling time, which promotes more efficient separation of the mixture.

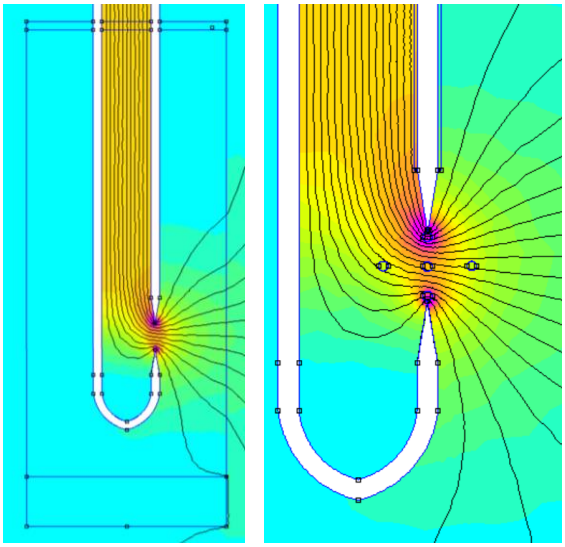


Fig. 1: FEMM Simulation of electric and magnetic field for point-to-point Fe electrode configuration at 3 kV for glycerol droplets at 5 different locations representing different levels of electric field

Currently, the dynamics of glycerol-droplet movement under the influence of a non-uniform electric field are examined in detail. The trajectories of glycerol droplets are systematically analyzed by tracking their motion and evaluating their behavior in relation to the electric potential and electric field distributions, see Fig. 2.

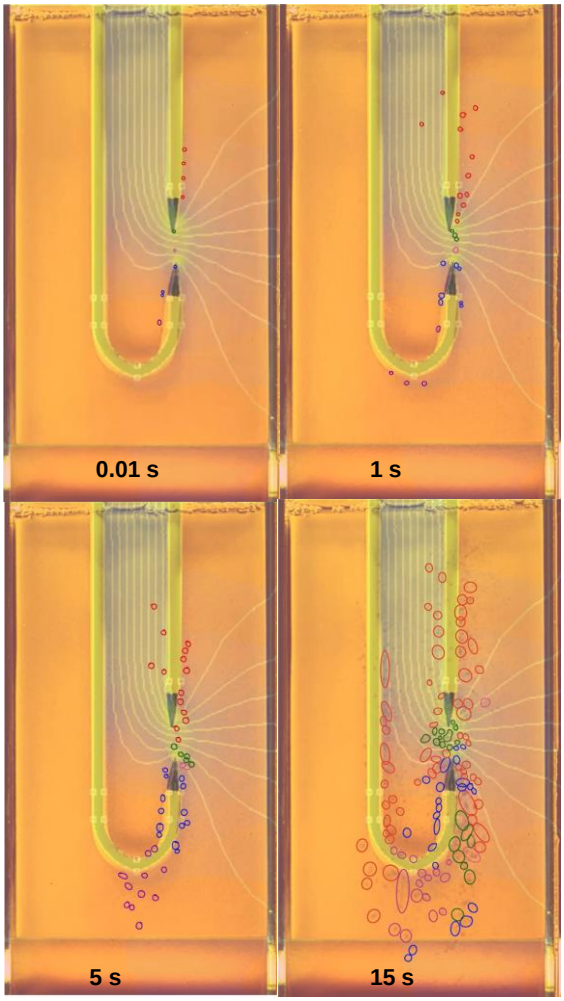


Fig. 2: Image analysis of glycerol droplets trajectories in electrical field distribution during EDS at 0.01, 1, 5 and 15 s

The first droplets are generated at the upper tip (green), the lower tip (blue), in between tips (pink) and along the side of electrodes (red) at 0.01 s. The first coalescence occurred at the upper electrode tip where the maximum electric-field stress was generated. The droplets continuously moved downward along the non-uniform electric field lines. The drops were also

arranged in a form of chained or elongated structure (purple). As the droplets got larger (purple), the electric field was not strong enough to dominate the movement, then the droplets were falling under the gravitational field.

To explore the dynamics of this system, both qualitative and quantitative analyses are employed. The qualitative analysis involves visual examination of coalescence patterns, captured through slow-motion video recordings, providing insight into the behavior of glycerol droplets under non-uniform electric fields with high electric stress. In the quantitative approach force calculations based on the Helmholtz force density model are used to evaluate the interactions driving the coalescence process.

The observed coalescence phenomena are influenced by several key forces, including electrostatic force, dipole–dipole interaction, migratory coalescence (electrophoresis), and dielectrophoresis. These forces play important roles in facilitating the aggregation of glycerol droplets. Their interaction with the electric field contributes to the overall separation efficiency of the biodiesel-glycerol mixture. In addition, the motion of glycerol droplets can be influenced by Coulombic forces acting on the charged droplets, which enhances the probability of collisions between glycerol droplets, thereby promoting migratory coalescence. Migratory coalescence is primarily driven by electrophoresis when AC-HV under high electric stress is applied. The efficiency and extent of coalescence are affected by a variety of factors, including the electrical and physical properties of the fluids, the magnitude of the external electric field, the presence of impurities in the mixture, fluid flow and turbulence, as well as the size and dispersion of the glycerol droplets within the emulsion.

The Method

Helmholtz electric force density model consists of the contribution of force acting on free charge density, force acting on an inhomogeneous dielectric and electrostrictive force $\overrightarrow{F_e}$ (Fitzpatrick 2016). The electric force density $\overrightarrow{F_e}$ can be expressed as:

$$\overrightarrow{F_e} = \rho_f \vec{E} - \frac{\varepsilon_0}{2} E^2 \nabla\varepsilon + \frac{\varepsilon_0}{2}\left(\frac{d\varepsilon}{d\rho_m}\rho_m\right)\nabla E^2 \quad (1)$$

The first term in Eq. 1 represents the standard electrostatic force density due to the presence of ρ_f, a free charge density in the medium, describing how the electric field interacts with free charges to generate a force. The second term represents the electrostriction force, which arises due to the non-uniformity of dielectric permittivity ε or inhomogeneous dielectric within the material. This term refers to the mechanical deformation or force generated when a material with spatially varying permittivity is exposed to an electric field which describes how the permittivity gradient $\nabla\varepsilon$ interacts with the square of the electric field E^2 which is related to the energy stored in the electric field and the influence on the medium's structure. The last term involves the rate of change of the dielectric permittivity with respect to the material density ρ_m. This accounts for the dielectrophoresis effect, where the force on the material is influenced by the spatial variation of the electric field and how the permittivity changes with respect to the material's properties. In other words, it describes how the field exerts a force based on both the electric field intensity and the material's spatially varying response to the electric field. Note that the electrostriction term gives zero as $\nabla\varepsilon = 0$ because when considering large finite regions of dielectric material, it usually does not contribute to the calculation of the total forces acting on the dielectric bodies.

The forces acting on the droplets under the influence of electric field are gravitational, $\vec{F}_g$, buoyancy, $\vec{F}_b$, drag, $\vec{F}_d$, and electrical, $\vec{F}_e$ forces, as illustrated in Fig. 3. The sum of all forces acting on the droplet then results as.

$$\sum \overrightarrow{F} = m\frac{dv}{dt} = \vec{F}_g - \vec{F}_b - \vec{F}_d - \vec{F}_e \quad (2)$$

where

$$F_g = \frac{4}{3}\pi r^3 \rho_d g \quad (3)$$

$$F_b = \frac{4}{3}\pi r^3 \rho_c g \quad (4)$$

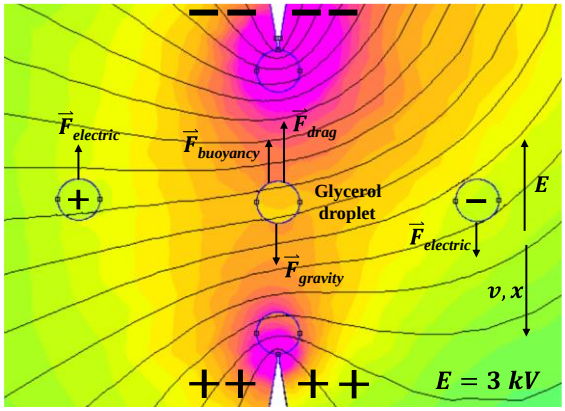
$$F_d = \frac{1}{2} c_d \rho_c v^2 \pi r^2 \quad (5)$$

ρ_d is the density of the glycerol droplet, ρ_c that of biodiesel, η is the dynamic viscosity of glycerol, r is the radius of glycerol droplet, c_d is the drag coefficient, v is the velocity of glycerol droplet, and g is the gravity.

Also of interest is the evaluation of the interfacial tension force, which is expressed as

$$F_\sigma = 2\pi r \sigma h \quad (6)$$

where σ is the interfacial tension of glycerol in biodiesel and h is Harkins-Brown correction factor.



Electric field (AC mode)

Fig. 3: Schematic representation of all forces acting on glycerol droplets in biodiesel under the influence of the electric field

The two-dimensional system has been considered in the simulations with an assumption that the droplet has a spherical shape at a uniform diameter of 10^{-4} m. Since the electrical field is non-uniform, the forces acting on the droplets are evaluated at 5 different locations representing in different levels of electric field, see Fig. 3. The first and third locations are at the tips of upper and lower electrodes respectively, where the maximum electric field stresses are generated (pink) with the value of which is in the order of 10^{6} V m^{-1}. The second location is at the center between two electrode tips where the electric field stress is lower (yellow) with the value of which is in the order of 10^{5} V m^{-1}. The fourth and fifth locations are to the left and the right of the second location at the same distance, where the electric field stress is slightly lower (green) than the one at the second location and the value is in the order of 10^{5} V m^{-1}.

In this preliminary analysis, the values of the gravitational and buoyancy forces are in the order of 10^{-7} N whereas the drag force is in the order of 10^{-8} N and the interfacial tension force is in the order of 10^{-5} N.

The electric force density can be determined by using Eq. 1 but the free charge density in the medium, ρ_f, as expressed in Eq. 1 is not available. However, the force can be determined from the zeta potential, ζ, and the electric field. Then, the electric force can be expressed as

$$\vec{F}_{e,1} = 6\pi r \varepsilon_r \varepsilon_o \zeta \vec{E} \quad (7)$$

and the value is in the order of 10^{-7} to 10^{-9} N. With the EDS, it is assumed that the temperature and density are constant. Then there is no change in the dielectric constant in the biodiesel phase and the other two contributions to the electric force become zero. As a result, the total force acting on the glycerol droplet under the influence of electric field has the same magnitude as the electric force in the opposite direction.

For the next phase of this study, the numerical and simulation results based on this mathematical model for EDS will be validated with the experiments.

Acknowledgements

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Drop-Based Simulation of Electrical-Driven Separation

Andreas Pfennig

Starting Point

Based on the experimental findings presented in the previous contribution of this annual report, it appears to be possible to link the theoretical insights and the drop-based simulations developed by David Leleu (Leleu, Pfennig, 2020). The main question is, if the effects of electrically driven interactions between the droplets can be integrated into the simulations. This should be possible, since one of the main aspects of progress of David Leleu was to explicitly introduce the evaluation of interaction forces between the drops into the simulations. That allowed already in the past to account for the different forces acting, e.g., between freely sedimenting drops and those in continuous contact in the close-packed zone.

Challenges

As first step gravity separation of biodiesel and glycerol phases should be described. In the experiments, which were performed at TGGS in Bangkok, it was found that even after one day the remaining glycerol phase continues to separate. This generated two challenges.

The first challenge was that the simulations with several 100 000 drops were to be performed depicting up to 170 000 seconds, resembling 48 hours, as shown in Fig. 1. Considering that the usual simulations covered few minutes at most, a new strategy needed to be introduced. Since the dependence of the coalescence probability on contact time between the drops had been formulated in a physically meaningful way (Pfennig, Leleu, 2020), a logarithmic time scale can be used. For a smooth onset of the logarithmic scale, at a switching time t_{switch} chosen by the user, the program switches from adding a time step Δt to using a factor f for determination of the next time,

$$f = \frac{t_{switch} + \Delta t}{t_{switch}} \quad . \tag{1}$$

That factor is then used to determine the successive times simulated according to

$$t^{(n+1)} = f t^{(n)} \quad . \tag{2}$$

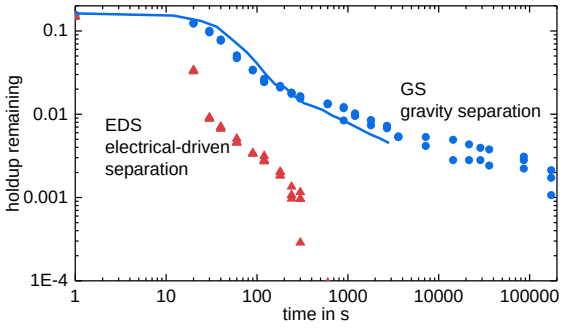

Fig. 1: Experiments performed at TGGS in Bangkok compared to simulations

The second challenge is that the initial drop-size distribution has to contain a large number of very small drops, since only then a significant volume of remaining drops is still settling after a day. It is not possible to describe this behavior with typical distribution functions. Thus, an initial distribution is implemented with small drops having a significantly higher probability. First results of corresponding simulations are presented in Fig. 1, which show that the two challenges have been solved.

As a next step the electrical forces acting have already been implemented, which will now be tested and refined so as to depict the very fast separation found experimentally with electrical field as shown in Fig. 1.

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Climate Change and Your Choices

Khang Vu Dinh

In 2024, where have you been when the climate change disasters happened? Were you safe? If you were safe, you were lucky. But, with the same question in 20, 30, or 50 years from now, will the answer still be positive? I am not sure. I believe that humanity is standing just one step away from the brink of global climate-change disasters. Wrong choices can plunge humanity into disaster and extreme suffering.

The climate disasters that happened in 2024 are a very serious reminder of the escalating and irreversible climate-change crisis across the globe. Typhoon Yagi in Vietnam, catastrophic floods in Valencia, and Superstorm Milton in the United States illustrate the terrible devastation caused by climate change. Typhoon Yagi, the strongest storm to hit Vietnam in 70 years, devastated northern provinces with winds exceeding 220 km/h and heavy rainfall exceeding 400 mm. More than 318 people were killed, and millions were affected by flooding, landslides, and infrastructure damage. The agricultural sector was devastated with thousands of hectares of crops destroyed. Although the storm's strength was predicted and storm preparedness plans were strictly implemented, the damage was still extensive. Unusually prolonged heavy rains caused by the storm's circulation caused severe landslides in many northern provinces of Vietnam. In particular, a peaceful village of 169 residents was almost completely wiped out after a terrible accident, 68 people were killed and went missing.

The scale of damage caused by climate-change disasters shows the vulnerability of countries, whether developing or developed countries. Climate disasters are becoming more severe than predicted with ever breaking records. The catastrophic floods in Valencia, Spain, and Hurricane Milton in the United States show that the climate crisis is not a problem of one individual country, or one continent, but a global one.

For many years, scientists from above 150 countries, including a group of more than 15,000 scientists from the Intergovernmental Panel on Climate Change, overwhelmingly agree that these extreme weather patterns are caused by climate change, fueled by the accumulation of greenhouse gases (GHG) (William et al., 2024). The concept of Carbon footprint focuses on the negative impact of human activities on climate change, such as burning fossil fuels for energy and transportation. Larger carbon footprints contribute more to global warming and climate change due to GHG accumulating in the atmosphere, causing rising sea levels, warmer ocean temperatures, and affecting atmospheric systems, contributing to the intensity and frequency of such disasters.

The warnings from scientists are a wake-up call about the imminent risks of climate change due to increasing greenhouse gas emissions (Ripple et al., 2020). As nations grapple with the aftermath of extreme climate, the need for both preventative and adaptive measures becomes evident. However, are we in the right direction when fossil fuel emissions have increased to an all-time high, and the temperature increase possibly reaching 2.7 °C by 2100 is very high? (UNDP, 2023). The three globally hottest days ever occurred in July of 2024 (Guterres, 2024).

The trajectory of climate change is not inevitable. It hinges on our choices as a global society and as individuals. The handprint concept refers to the vision, direction, and opportunity to address global climate change as the positive effects of actions or solutions that reduce others' carbon footprint. It measures the extent to which an individual, organization, or product contributes positively to mitigating climate change. Carbon handprints contribute to slowing or reducing the impacts of climate change in various ways, such as developing low-carbon technology, carbon neutrality, improving energy efficiency, using renewable energy, etc. Increasing carbon handprint will create a net positive impact and promote changes that will help reduce emissions. Governments are key in determining whether to increase or reduce climate change. Therefore, policies enacted by governments should prioritize climate change first. At the same time, the government must lead by changing friendly policies, transi-

tioning to renewable energy, enforcing stricter emissions regulations, investing heavily in research and development of new clean energy, and investing in climate-resilient infrastructure to limit climate change.

Individuals play a crucial role in shaping demand and supporting sustainability. Every individual decision, such as choosing a country's leader who cares about climate change, is also important. Individual lifestyle consumption, such as reducing energy use, adopting plant-based diets, and advocating for climate-friendly policies, can collectively drive significant change. Ruminant livestock is predicted to produce 250 to 500 L of methane per day (Johnson et al., 1995). Therefore, changing habits toward choosing to reduce meat consumption contributes significantly to limiting greenhouse gas emissions.

While the climate change events of 2024 paint a grim picture, they also offer an opportunity to raise global actions, in which each individual plays a key role. Recovery efforts in Vietnam, Spain, and the United States demonstrate the resilience and solidarity of affected communities. However, all of that is not enough. The fight against climate change really needs more unity and resilience from all individuals in the world to act together to reduce greenhouse gas emissions. Communicating climate change awareness to each individual is also a solution to promote carbon handprint. Like-minded individuals will create a strong group in the fight against global climate change no matter what country or regime you live in. Each individual should be a resilient pioneer who not only sets an example but also conveys a message that helps other individuals take action against climate change. If each pioneer acts and helps other individuals follow suit, the total number of climate action takers can quickly increase exponentially, which can be calculated by the formula

$$T_n = \frac{r^{n+1} - 1}{r - 1}$$

where, T_n (people) is total reach after n rounds, r is transmission rate (people), which is The number of people that each individual communicates or transmits the information to in one round, n is the number of rounds, which is the number of times the transmission process occurs. If $r = 5$, then with just about 15 rounds, the entire world population of about 8.1 billion people can access the climate action message.

At the government level, climate action can bring about rapid change to reduce climate change. However, that is up to the leaders. Do they really care and act to reduce climate change, or is it something else? It is clear that action on climate change may require a long-term compromise at the government level in many countries. Individual action worldwide may not completely reduce the problems of global climate change, but it is the fastest way to reduce the risks and disasters from climate change. The living environment of current and future generations depends on our choices today.

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Department Activities of This Year

Ezgi Uslu, Marc Philippart de Foy

Introduction

As in previous years, several events were organized in 2024 within the department, bringing together the various groups that make it up. Here is a summary of some activities over the year.

Forestia

This year, as part of our annual team-building activities, the entire Chemical Engineering Department at ULiège had the opportunity to visit Forestia Park in Belgium. It was a day filled with adventure, bonding, and relaxation in nature.

The day started with a thrilling climbing session among the trees, where we challenged ourselves on various ropes and zipline courses. Fig. 1 shows our colleagues on parkours from Forestia. It was not only a physical activity but also a great chance to practice teamwork, as we encouraged and supported one another through the challenges.

Fig. 1: Our colleagues at Forestia

After the climbing adventure, we enjoyed a delicious meal together, which allowed us to relax and connect on a more personal level. Sharing laughs and stories over good food made the experience even more special.

To wrap up the day, we explored the park's natural wildlife area, where we observed animals in their habitats as shown in Fig. 2. It was a peaceful and educational experience, reminding us of the importance of balance between human activities and nature.

Fig. 2: Deers in their natural habitat

Overall, the trip to Forestia Park was a memorable experience that strengthened our bonds as a team and gave us a refreshing break from our academic routines.

Annual barbecue

Every July, our department gathers for a long-standing tradition: the Annual Summer BBQ. This event is a highlight of the year, giving everyone a chance to take a break from their busy schedules and enjoy a relaxing day in the summer - the "Belgian summer" as shown in Fig. 3.

The BBQ is held in a “picturesque” outdoor setting, providing the perfect backdrop for good food and great company. Professors, researchers, PhD students, technicians and secretaries come together to share delicious grilled dishes, refreshing drinks and homemade desserts But it's not just about the food; the BBQ is also a chance to strengthen our sense of community.

It's always a wonderful opportunity to welcome new members to our department and celebrate our collective successes in a relaxed and friendly atmosphere.

The annual summer BBQ is more than just a meal - it is a cherished tradition that reflects the warmth and camaraderie of our Chemical

Engineering Department. We always look forward to it as a moment to connect and make lasting memories.

Fig. 3: Preparation of the BBQ under not so sunny conditions

Christmas events

The Christmas spirit also touches our team at the end of each year. In December 2023, Andreas resumed the tradition of baking Christmas cookies together, after a few years in which this had not been possible due to the Covid-19 pandemic.

Andreas invited our entire team to his home to spend the day together and complete the preparation of hundreds of little Christmas cookies. Four recipes were followed in parallel to prepare vegan treats based on coconut, almonds, fruit and chocolate. The team was made up of Andreas, Khang, Hong, a student who did her master's thesis with us, Martine, the department secretary, and us. Teamwork enabled us to prepare all the cookies in one day. While some prepared the dough, others mixed and baked the paste and the last ones gave shape to the cookies. A real team effort! And to give us even more energy, Andreas had prepared a delicious meal for lunch!

The cookies were then distributed to the people we work with in the department, such as the laboratory technicians or the executive secretaries. Numerous treats were also offered as a buffet to students at the annual Christmas lecture, during which we presented our work to the master students in chemical engineering. A photo of us in front of our cookie production is shown in Fig. 4, where you can see the smiles on the faces, proud of all the baking accomplished, and where you can appreciate the dedication we put into baking these delicious cookies!

Fig. 4: The baking team in front of the delicious result of our work

Another Christmas-related event in the department is the annual Christmas lunch, which took place last year at the “Wok Bon Sel” restaurant. It was a great opportunity to meet our colleagues from different teams who we do not always see at work. The Christmas cookie production, Christmas lecture and Christmas lunch will of course also take place this year, and we are looking forward to it!

In December, PhD students, postdocs, and researchers also usually visit Liège’s Christmas Market as shown in Fig. 5. Enjoying warm drinks, local treats, and the festive atmosphere, we created great memories and recharged for the new year.

Fig. 5: Happy faces from Christmas market in Liège

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2024, a Year of Conferences and Trips

Marc Philippart de Foy, Ezgi Uslu

Introduction

2024 was an excellent year as regards our participation in international conferences. Both of us were able to take part in various events bringing together colleagues from different locations working in similar fields. Here is a summary of the events we attended.

DECHEMA extraction meeting in Dresden

On February 14 and 15 this year, DECHEMA organized an annual meeting on Extraction in Dresden, Germany. Over the course of two days, oral and poster presentations were held in sessions covering a variety of subtopics related to extraction. Contributions encompassing the full thematic range of the field were delivered, “from experimental results to theoretical approaches, numerical methods, instrumental implementations, and the development of new extractant and overall processes”. The aim of this annual, closed event is to review the projects of numerous researchers working in the field of extraction and to provide a platform for networking and collaboration among those pursuing similar goals.

Ezgi participated in the conference with her presentation, “Mass Transfer and Reaction Kinetics for Reactive Extraction” as shown in the Fig. 1.

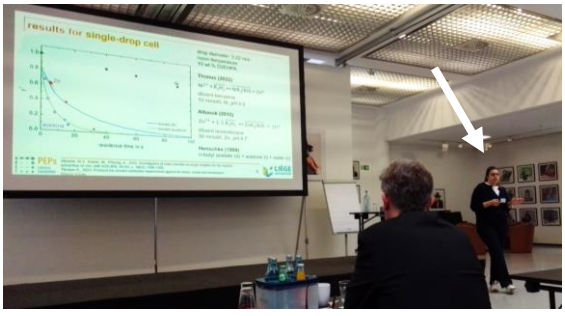

Fig. 1: Ezgi while presenting

Following the presentations on the first day, the entire conference team gathered for dinner. Meeting new people from various cultures who are working in the same field is invaluable for networking. The second day of the conference presentations continued. During breaks, there was great interest in the poster sessions. At the closing of the conference, the best poster received a unique gift: Beer produced by the students at the Technical University of Dresden! Produced in an experimental brewery run jointly by Professor Jan J. Weigand and Professor Thomas Henlen, this project combines education and hands-on experience for chemistry students. The brewery also works in cooperation with the local “Watzke Brewery”.

The next DECHEMA Extraction Assembly will be held in Aachen in February 2025, where we plan to participate with both an oral and a poster presentation.

During our free time, we also had the chance to explore Dresden. The Old Town (Altstadt) is especially worth visiting, with landmarks such as the Zwinger Palace, Frauenkirche, Fürstenzug, and Brühlsche Terrasse. Fig. 2 shows an exterior view of the Zwinger Palace. If you are passing through Dresden, be sure to enjoy this historic city, the capital of Saxony.

Fig. 2: Zwinger Palace, Dresden

DECHEMA forum 2024 in Friedrichshafen

In July 2024, the one-year confidentiality period of the patent for the SisClever process (see

other contribution in this annual report for process details) came to an end, meaning that we could now talk freely about our new process idea. Marc therefore took part in several events at which he was able to present the process and seek industrial collaboration. These events included two conferences, the first being the DECHEMA forum 2024.

This conference brought together engineers in Friedrichshafen from September 11 to 13. The theme of the conference was “How can the chemical, pharmaceutical and life sciences industries make their production more sustainable?” Scientists and industrial partners were able to meet during the conference and discuss future challenges in this field. The event was divided into presentations, poster sessions and workshops, which was a good balance to allow discussion between all participants and reflection on hot topics in the chemical industry. Andreas and Marc together presented the SisClever process to the public for the first time.

The DECHEMA forum also offered networking events, such as a carrier forum, where brief interviews with companies were possible, or a dinner. This was a good opportunity to meet colleagues from elsewhere, such as the PhD students we met at the doctoral seminar held in Aachen the previous year. The conference was also an opportunity to visit the beautiful city of Friedrichshafen. The city is located on the shores of Lake Constance, which offers stunning views, as shown in Fig. 2. This photo was taken from the top of the Mole Tower, which rises to a height of 22 meters, accessible by 117 steps. The Zeppelin Museum is the town's main attraction, Friedrichshafen being an important place in Zeppelin's history, but simply strolling around the town and in particular the main street by the lake was Marc's favorite activity.

Fig. 3: Sunset in Friedrichshafen

SFGP 2024 in Deauville

Marc also attended a second conference this year, the SFGP 2024 in Deauville, France, from October 15 to 17. Every two years, this conference brings together around 500 French-speaking process engineers. It was another opportunity to present the SisClever process to potential future partners, as well as to see new advances in the field of chemical engineering.

The conference strongly promoted networking, especially among doctoral students. Several events were organized to bring them together, such as a “break-the-ice” mission through Deauville or a chemical-engineering quiz. Other events included a gala dinner, as shown in Fig. 3. The gala dinner took place in the magnificent Salle des Franciscaines, a former convent converted into a public library.

Fig. 3: Gala dinner in the Franciscaines, photo by Gael Kazaz

Others

Other presentations were also given during the year too, such as departmental seminars. The annual Christmas lecture was also presented to master students in December 2023 and will be again this year. This event is an excellent opportunity to show them more clearly what the dry matter from their studies can be used for in real engineering practice. Christmas cookies prepared by us are also shared on this occasion, for which more details are given in the contribution on page 14 in this annual report.

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Shariff, Z.A., Leleu, D., Pfennig, A. (November 2022). Phosphorous Recovery From Sewage Sludge Utilizing Reactive Extraction. Presented at The International Symposium for Green Solutions, ISGS 2022, Ho Chi Minh City, Vietnam.

Pfennig, A. (November 2022). Sustainable Bioeconomy or CO2-Economy: What Does the Future Hold? Paper presented at The International Symposium for Green Solutions, ISGS 2022, Ho Chi Minh City, Vietnam.

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Pfennig, A., Trettler, L., & Müller-Hermes, A. (September 2022). Morgen-Land: Generationswechsel und Kulturpädagogik als Bildung für Nachhaltige Entwicklung. Paper presented at 17. Jugendkunstschultage NRW, Kleve, Germany.

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Shariff, Z.A., Leleu, D., Pfennig, A. (September 2022). Phosphorous Recovery From Sewage Sludge Utilizing Reactive Extraction. Paper presented at International Solvent Extraction Conference (ISEC 2022).

Leleu, D., Pfennig, A. (September 2022). Drop-Based Settler-Design Tool Developed Based on Iso-Optical Systems. Paper presented at ProcessNet & DECHEMA-BioTechNet Jahrestagungen 2022 & 13th ESBES Symposium.

Shariff, Z.A., Leleu, D., Pfennig, A. (September 2022). Recovery of Phosphorus From Dried Sewage Sludge for Fertilizer Formulations. Paper presented at ProcessNet & DECHEMA-BioTechNet Jahrestagungen 2022 & 13th ESBES Symposium.

Bohn, F., Lenze, A., Pfennig, A., & Schleicher, C. (June 2022). Workshop Lebensmittelverschwendung. Paper presented at Nachhaltigkeitstage 2022 der Asten der RWTH und der FH Aachen, Aachen, Germany.

Pfennig, A. (June 2022). Klima- & Krisen-Wende-Zeit: Worauf kommt es wirklich an? Paper presented at Nachhaltigkeitstage 2022 der Asten der RWTH und der FH Aachen, Aachen, Germany.

Leleu, D., Pfennig, A. (May 2022). Drop-Based Modeling of Batch Settlers: The Final Model. Paper presented at Jahrestreffen der ProcessNet Fachgruppen Extraktion, Phytoextrakte und Membrantechnik.

Exam problem:

Please provide two goals to be achieved with energy input and two methods for energy input to extraction columns.

Student response:

goals:

- heat the liquid to bring it to boiling temperature

- if extraction is endothermic, as we want to stay in steady state

ways:

- add a hotter feed in the column

- use a reboiler

[Comment AP: I should assure you that our students are taught thermal separation processes according to international standards, i.e. this answer is not compatible with our teaching material and lecture content...]

Student presentation in the Integrated Project to estimate the cost for a proposed process:

EAOC = equivalent annual operating cost

EAOC = 29 441 888 € $\Rightarrow$ needs to be refined