



Separation and Purification



2023





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Dear Reader

The year 2023 was politically very turbulent and challenging. This, as well as the late effects of COVID-19, is still affecting life at the university. For example, my impression is that student performance has deteriorated somewhat compared to before the crisis. This may also be due to the more turbulent times and the fact that all lectures are now available on video. These videos are offered to students to support their learning, but some rely completely on the videos and only rarely attend lectures. A regular timetable and the personal presence of the lecturer, who can act much more freely on the lecture stage than when recording a video, seem to have a positive effect on students' learning outcomes.

On the one hand, 2023 was a very quiet year for us. Everyone in the team worked on his or her work packages in the projects. 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. On the other hand, two things should be emphasized in particular. We filed a patent application this year for the new process structure, which I reported on for the first time in last year's annual report, and are now hoping that it will be granted. Secondly, this year I can report for the first time on a collaboration with Evonik on the Rheticus project - albeit only in a very generic way. Rheticus is about artificial photosynthesis, i.e. a power-to-X process that produces e-chemicals from CO₂ and green hydrogen.

For me personally, the activities with Scientists for Future continued. On the one hand, this relates to the project on visions for the future (Zukunftsbilder), in which positive, sustainable visions for Germany in 2040 are being developed in significant detail. Meanwhile a first version of the Website <u>Zukunftsbilder.net</u> is online and is currently filled continually with more and more content. On the other hand, there were of course various lectures and workshops on the topic of sustainability and how we can personally adopt more sustainable behavior.

All in all, it was another very eventful and multifaceted year, of which we try to give an impression in this annual report.

So: Enjoy reading!

Andreas Pfennig





P-Recovery Process: Stages Validation

Vu Dinh Khang

According to the results of assessing the potential for phosphorus (P) recovery in waste sources in southern Vietnam by Khang et al. (2023), biological (BIOS) and chemical (CHES) sludge from rubber-latex processing wastewater-treatment plant (RWWTP) was determined to be the most suitable raw material source for P recovery under current conditions.

In this study, the procedure for P recovery from sludge of RWWTP is step-by-step developed. Through a combination of results from lab experiments and simulations, process parameters are optimized and validated on the pilot. A schematic representation of the sequential P recovery process is provided in Fig. 1. This operation comprises principal stages: leaching acid solution, first precipitation, dissolution of the precipitated material, and precipitation product. These are interspersed with secondary stages involving the separation of liquid and solid phases. The first precipitation stage has the main role of precipitating compounds of P for recovery as Al-P at appropriate equilibrium pH using NaOH solution. The final precipitation stage involves producing the Ca-P mineral by using a Ca(OH)₂ solution.



Fig. 1: A schematic representation of P-recovery process

Findings obtained through simulation utilizing the Matlab tool (Shariff et al. 2023) and the cascaded option tree methodology reveal that, at a phase ratio (ml: g) of 5, P leaching efficiency from CHES can approach about 100% by using sulfuric acid. Nevertheless, the empirical results unveiled a notable drawback. That is the formation of gypsum mineral from the reaction of Ca²⁺ in sludge and sulfate ions SO₄²⁻. Mixed leaching solution is shown as Fig. 2. The formation of gypsum mineral in the leaching mixture at phase ratio 5 greatly hinders the ability to phase separation. Similarly, at phase ratio of 6 to 9 shows that the liquid-solid phase separation is impossible. At phase ratio 10, more volume of acid solution is used, so the formation of gypsum does not greatly affect the ability to phase separate and recover the liquid after leaching. In addition, the P leaching efficiency at this phase ratio reached over 90%. Therefore, phase ratio 10 is considered suitable for P leaching.



Fig. 2: Mixed solution after leaching at phase ratio 5 with CHES

BIOS generated from the biological treatment stage of RWWTP was also used for simulation and experimentation. The results of laboratory experiments and simulations at phase ratio 5 were used to optimize P leaching parameters and validated on pilot for BIOS. Fig. 3 shows the pilot P leaching stage.







Fig. 3: Pilot leaching stage

Adjusting the *p*H of the leached liquor by adding NaOH solution not only facilitates the formation of Al-P minerals but can also cause unwanted precipitates such as Al(OH)₃, Fe(OH)₃ and Fe-P salt to appear. The appearance of these unwanted precipitates hinders the separation and removal of unwanted substances in the P recovery process. Therefore, it is necessary to perform the precipitation process at a selected *p*H to achieve the maximum Al-P precipitation efficiency as well as limit other precipitation products of P. Research results show that at equilibrium *p*H 5.2 for BIOS and 5.1 for CHES, the degree of Al-P mineral precipitation is most effective.

Results obtained from laboratory experiments and pilot-scale operations and also simulations for BIOS and CHES have revealed a notable congruity in the extent of phosphorus precipitation, specifically in the form of aluminum-phosphorus. The degree of P precipitation as AI-P is in the range of 0.9 to 1.0 for both, BIOS and CHES.

The dissolution stage aims to dissolve precipitated minerals such as AI-P and AI(OH)₃ obtained from previous precipitation stage in an alkaline liquid, adjusted with NaOH. Other solid compounds such as $Fe(OH)_3$ are not soluble in highly alkaline environments and will be removed through liquid-solid phase separation. Fig. 4 shows the solubility result of the main precipitated minerals under different equilibrium *p*H conditions. Results from the lab experiment and simulation of the precipitated mineral dissolution stage were used to optimization and validation on the pilot for both BIOS and CHES. The suitable equilibrium *p*H for dissolution of precipitated minerals for BIOS and CHES is 9.2 and 9.1, respectively. The degree of dissolution at equilibrium pH of precipitant from BIOS and CHES are in the range of 0.85 to 0.95.



Fig. 4: Simulation of dissolution with CHES

The dissolved solution obtained after the phase separation is added with Ca(OH)₂ solution to continue the last precipitation stage to create the final product of the process, Ca-P.

The P recovery process from RWWTP sludge is developed step by step through combining results from lab experiment, simulation and finally validation on pilot scale. Through the stages of the process P in the sludge is gradually dissolved, separated and recovered as Ca-P product; and unwanted metal impurities such as AI, Ca and Fe are removed through precipitation at selected equilibrium pHs.

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Electrical-Driven Separation Using High Voltage and Alternating Current

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Starting Point

Electrical-driven separation (EDS) using high voltage and alternating current has been proven to be an alternative liquid-liquid separation technology with high separation efficiency as mentioned in the 2021 and 2022 annual reports. The focus application in our research is the separation of biodiesel and glycerol using EDS at the optimal conditions (the maximum separation efficiency of 99.8% determined at the shortest separation time and low energy consumption) of 3 kV with the point-to-point electrode configuration and the vertical distance between electrodes of 3 cm for separation time of 240 s. As observed in the experiments, the alycerol droplets moved toward the electrode tips where the electrical field was generated, while the charge on the surface of the glycerol droplet changed. The more droplets were in the electrical field, these droplets would align along the streamlines and the coalescence of droplets occurs. This phenomenon took place in a shorter time than in gravitational settling.

However, the behavior of glycerol droplets movement under the influence of non-uniform electric field has not been investigated in detail. Therefore, in the current phase of this study, the trajectories of glycerol droplets are first investigated by tracking the movement of glycerol droplets and then analyzing their motions along the potential and electric field distributions, which was obtained from the Finite Element Method Magnetics (FEMM 4.2) computer simulation.

The Method

The experiment of the separation of biodiesel and glycerol using EDS at the optimal conditions was recorded using a 4K camera and evaluated frame by frame every 0.1 s. The movement of individual glycerol droplet was tracked in the entire chamber. In general, the glycerol droplets move freely without any external force applied to the system. However, when applying electric field, the glycerol droplets intensively moved near the electrodes as illustrated in Fig. 1. By applying an AC high voltage, a strong electric field strength was generated between two sharp-edged electrode tips, as shown in pink color in Fig. 2. The charge on the surface of droplets was induced by the applied electric field causing these charged droplets to move toward the electrodes. Any nearby charged droplets moved closer and then coalesced. Typically, this first coalescence was observed at the active electrode tip, the top one. Note that the ground electrode tip is the bottom one. As this coalesced droplet moved downward along the electrode, it attracted other small droplets nearby and further coalesced to larger drops, which settled towards the bottom of chamber under the influence of gravity. On some occasions, the small, charged droplets could move upward near the electrode causing small fluid currents, which promotes more separation.



Fig. 1: Schematic diagram of glycerol droplets trajectories at 5 s, 10 s and 20 s

FEMM simulation results in Fig. 2 show the electric field distribution and electric field strength or electric stress (E). Lines show the equipotential lines, and the color shades represent the magnitude of E. The dark pink color corresponds to the highest E while the light blue color corresponds to the lowest E. Obviously, the highest electric stress is observed in the non-uniform field of point-to-point electrode configuration as shown in curved lines. At constant voltage, the maximum field E is generated under non-uniform conditions when having the small radius of curvature as corresponding to



$$E_{\text{oil}} = \frac{\varepsilon_{s}(\varepsilon_{s}+1)V}{2(d+2R(1-\cos\theta))} \cong \frac{V}{d+2R(1-\cos\theta)}$$

In general, the electric stress or electric field strength of oil (E_{oil}) depends on the voltage supplied (V), the gap distance between electrodes (d), the electrode radius (R), the polar angle on electrode (θ), and the relative permittivity of glycerol as solute (ε_s) as given in the equation above (Takuma, 1991).



Fig. 2: FEMM Simulation of electric field and magnetic field for point-to-point Fe electrode configuration

The trajectories of glycerol droplets were further analyzed together with electric field lines obtained from FEMM simulation results. The image analysis process shown in Fig. 3 started by capturing the image at each frame from the recorded video. Then, these images for the entire experiment time were converted to video with FEMM electric field lines as a background. This process was accomplished by superimposing images of many sequences of glycerol motion images over a base image of the electric field background from FEMM with transparent fitting using the MATLAB tool. Finally, the image sequences were converted back into a video using the MATLAB tool. This full video is available at https://myqrcode.com/qr/559fe7b4/view.

The video of glycerol droplets motion along with the electric field lines confirmed that the droplets moved toward the electrodes and the first coalescence occurred at the top electrode tip where the maximum electric field stress was generated. The droplets continuously moved downward along the non-uniform electric field lines. As the droplets got larger, the electric field was not strong enough to dominate the movement, then the droplets were falling under the gravitational field.



Fig. 3: Process flow diagram for image analysis of glycerol droplets trajectories in electrical field distribution

For the next phase of this study, all important forces acting on this glycerol droplet under the influence of electrical field will be evaluated. Later, the mathematical model for EDS will be developed along with the Computer Fluid Dynamics (CFD) simulation. The numerical and simulation results will be validated with the experiments.

Acknowledgements

This work including the preparation of the apparatus and the experimental works was mainly performed by Rossarin Ampairojanawonga, Ajalaya Boripuna and Sayan Ruankon.

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Nitsch Cell – First results

Ezgi Uslu

Introduction

For the design of reactive-extraction columns and calculation of the extraction efficiency, it is important to understand and precisely determine the reaction and mass-transfer kinetics. These parameters are commonly determined using specific laboratory cells, such as the modified Lewis cell known as the Nitsch cell shown in Fig. 1. This type of cell is used to determine the kinetic parameters of interfacial reactions and their rate-determining step. The Nitsch cell is characterized by a particularly careful design concerning the fluid dynamics in the two phases and thus appears to be a good starting point for our investigations (Bertakis et al., 2008).



Fig. 1: Schematic representation of the Nitsch cell (Kalem, 2015)

Nitsch Cell

The working principle of the Nitsch cell is a mixing cell with a fixed interfacial area, in which liquid-liquid extraction mass transfer can occur while the contact area between the two phases is kept constant and stable. The Nitsch cell is surrounded by a double-walled glass cylinder, which is used for temperature control by connection to a thermostatic bath. The light phase in blue and the heavy phase in green are shown in Fig. 1. Both phases can be stirred independently of each other, which is especially useful for stirring both phases at identical Reynolds numbers, which will generally have different densities and viscosities. The stirrers drive the liquids through sections with vertical cylinders, which ensure that the essentially axial flow of the phases towards the interface is realized. Flow breakers are used to avoid tangential flow and to reach laminar flow between the tubular flow guide and the outer wall of the cell. Thus, in both phases, the flow is ideally transferred towards the interface at the center of the cell and passes the interface in a radial direction. In the ideal case, the geometric area of the interface is well-defined. Via measurement of the concentrations in both phases, the transferred amount of substance can be determined with different characterization methods. Thus, it is possible to determine the area-specific masstransfer rate.

The Experimental Method

To measure mass transfer, a transfer component is added to one of the phases, and samples of either phase are taken at specified times and analyzed for composition. For each condition, especially for a specified stirrer speed, the mass transfer rate is then evaluated from the time-dependent concentrations via appropriate balances as a function of time.

First experiments with the Nitsch cell were performed with a physical test system to validate the equipment: n-butyl acetate, water, and acetone. The solvents were saturated before the experiments. The transition component, acetone, was added to the organic solvent. First, the aqueous phase was filled into the measuring cell, then the organic phase was added slowly from the top. The addition of the organic phase had to be carried out with special care to avoid that the organic phase would be partially dispersed into the aqueous phase during the introduction and thus a non-negligible proportion of the acetone would already be extracted into the aqueous phase during the filling process.



Samples are then taken from both phases at certain time intervals and the amount of acetone in each phase is determined using a UV-Vis spectrophotometer. The concentration profiles of both phases are shown in Fig. 2.

Results

The mass-transfer coefficient for the organic phase k_{org} is defined as

$$-\frac{m_{\rm org}}{A}\frac{dw_{\rm org,Ac}}{dt} = k_{\rm org}\rho_{\rm org}\left(w_{\rm org,Ac} - w_{\rm org,Ac}^*\right), (1)$$

where *A* is the mass transfer area, m_{org} is the mass of the organic phase, ρ_{org} is the density of the organic phase, $w_{\text{org,Ac}}$ is the acetone mass fraction in the organic phase, where the star indicates equilibrium with the aqueous phase which can be evaluated by using the distribution coefficient K_{Ac} (Kalem, 2015):

$$w_{\rm org,Ac}^{*} = K_{\rm Ac} w_{\rm aq,Ac} \,. \tag{2}$$

Eq. 1 can then be integrated as:

 $\int_{w_{\text{org},\text{Ac},0}}^{w_{\text{org},\text{Ac}}} \frac{dw'_{\text{org},\text{Ac}}}{w'_{\text{org},\text{Ac}} - w'_{\text{org},\text{Ac}}} = -\frac{k_{\text{org}}A}{V_{\text{org}}}t.$ (3)

As shown in Fig. 3, the experimental data for the argument in the integral can be plotted and fitted by a second-order polynomial. This polynomial can the be integrated easily to quantify the left-hand side of Eq. 3 and thus back out the value of the mass-transfer coefficient.

The mass-transfer coefficient for acetone transferred from n-butyl-acetate phase to water is calculated as around 5×10^{-5} m/s. Kalem (2015) worked with another physical test system: toluene, water, and acetone. The mass transfer coefficients that he obtained were between 2×10^{-5} m/s and 4×10^{-5} m/s depending on stirrer speed, thus in a comparable range.

Meanwhile it has been shown that the direct measurement of the concentrations in both phases yield identical results to analyzing only one phase and determining the concentration of the other phase by solving the appropriate balances. To obtain reliable results, in solving these balances, it must be taken care that also the volume of samples removed from the cell are properly accounted for.



Fig. 2: Concentrations in both, the aqueous and the organic phase



Fig. 3: Fitting the term using a polynomial function

After the experimental optimizations are completed with the physical test system, studies will continue with the reactive systems. First the EFCE standard test system for reactive extraction with zinc and D2EHPA will be utilized. After that systems relevant for recycling of valuable metals from scrap will be investigated.

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Reactive Extraction Equilibrium and Stoichiometry Study of Cobalt-D2EHPA System

Marc Philippart de Foy

Introduction

An important aspect of liquid-liquid reactive extraction process design is knowledge of the chemical reactions taking place. One approach to modeling extraction processes is to use theoretical stages, where the process is separated into different stages and chemical equilibrium of the reactions is assumed to be reached between the output streams of each stage. In this modeling approach, it is therefore essential to correctly describe the chemical equilibrium of the reactions taking place. The general form of extraction reactions for the extraction of a metal M with an extractant RH is

$$\mathsf{M}^{\beta+} + \alpha \overline{\mathsf{RH}} \underbrace{\longrightarrow} \overline{\mathsf{MR}}_{\beta} \left(\mathsf{RH}\right)_{\alpha-\beta} + \beta \mathsf{H}^{+}, \qquad (1)$$

where the stoichiometry α of the extractant is unknown. The aim of the experimental work presented in this report was to determine this stoichiometric coefficient and the extraction equilibrium constant K_c defined as

$$K_{\rm c} = \frac{C_{\rm MR_{\beta}(RH)_{\alpha-\beta}}C_{\rm H^{+}}^{\beta}}{C_{\rm M^{\beta+}}C_{\rm RH}^{\alpha}}.$$
 (2)

This study focuses on the reactive extraction of cobalt with D2EHPA with the aim of finding a test system for further general reactive extraction studies. The experimental work was carried out as part of a master's thesis (Singh, 2023).

Experiments

Extraction experiments were carried out in glass tubes of 25 mL, where approximately 10 mL of aqueous phase was mixed with a similar volume of organic phase. Cobalt chloride was initially dissolved in the aqueous phase, where it was mixed with distilled water, and a so-called '*p*H-changer' solution, used to vary the *p*H of the aqueous phase. This *p*H-changer solution contained either NaOH or HCI. Kerosene was used as solvent in the organic phase and D2EHPA as extractant. Once both, aqueous phase and organic phase, were placed in the tubes, the tubes were mixed in a water bath at the desired temperature for 10 minutes allowing to reach the extraction equilibrium (Singh, 2023). They were then left in the water bath for 1 hour to allow phase separation at the desired temperature. The phases were finally separated using a syringe. The organic phase was first recovered and placed in a new tube, then the interface was removed using the same syringe. The aqueous phase was left alone in the tube and could be analyzed by UV-vis spectroscopy to calculate its cobalt mass fraction. The cobalt mass fraction remaining in the aqueous phase could finally be compared with its initial mass fraction to assess the degree of extraction achieved. The degree of extraction is defined as follows, based on the initial 'i' and final 'f' cobalt masses in the aqueous phase:

$$E = \frac{m_{M_{i}^{\beta+}} - m_{M_{f}^{\beta+}}}{m_{M_{i}^{\beta+}}}.$$
 (3)

As the degree of extraction depends on the *p*H reached at equilibrium, extraction experiments were carried out to assess the degree of extraction at different *p*H values, as shown in Fig. 1. It can be seen from the image that the higher the *p*H, the higher the degree of extraction, as the pink color of the cobalt in the aqueous phase fades with increasing *p*H, while the blue color of the cobalt complex in the organic phase intensifies, meaning that more and more cobalt is extracted as the *p*H increases.



Fig. 1: Equilibrium of Co extraction experiments with D2EHPA at an increasing *p*H from left to right





A second method was also used to assess the degree of extraction. A schematic representation of this method is shown in Fig. 2.



Fig. 2: Representation of back-extraction method

This method involved collecting the organic phase obtained after the extraction and backextracting the cobalt present in it into a new acidic aqueous solution containing HCI. A photo of the samples obtained at equilibrium of backextraction is shown in Fig. 3.



Fig. 3: Equilibrium of back-extraction of the samples presented in Fig. 1

The aqueous solution obtained after back-extraction was then again analyzed by UV-vis spectroscopy to evaluate its cobalt mass fraction. The mass fraction of cobalt in the organic phase obtained after extraction was calculated from this result using mass balances and assuming that all cobalt initially present in the organic phase had been recovered via back-extraction. This allowed to deduce the degree of extraction from the extraction experiment.

Results

Both characterization methods were used for all extraction experiments. This gave greater confidence in the results, and meant that problems were sometimes noticed during experiments or evaluation. This is illustrated in Fig. 4 for an example, where three extraction experiments were carried out for similar cobalt and D2EHPA concentrations, but with varying equilibrium pH. The graph shows good consistency between the two methods, validating the characterization

methods. The mean value between the degrees of extraction obtained with the two methods was used in the further numerical evaluations.



Fig. 4: Degree of extraction obtained via extraction and back-extraction

Once the analytical methods had been validated, experiments were carried out under different conditions. The initial cobalt concentration was kept constant at 2 g/L, and the extractant concentration was varied. The preparation of samples with different *p*H values enabled several data points to be obtained, which were then used to evaluate K_c and α . Fig. 5 shows the experimental data points obtained for different D2EHPA concentrations and *p*H values.



Fig. 5: Experimental degrees of extraction for varying D2EHPA concentrations

Based on these experimental results, the values of K_c and α could be computed by data fitting. Eq. 2 can be expressed with the partition coefficient *K* defined as

$$\mathcal{K} = \frac{C_{\mathsf{MR}_{\beta}}(\mathsf{RH})_{\alpha-\beta}}{C_{\mathsf{M}^{\beta+}}}, \qquad (4)$$



leading to

$$\mathcal{K}C^{\beta}_{\mathsf{H}^{+}} = \mathcal{K}_{\mathsf{c}}C^{\alpha}_{\mathsf{R}\mathsf{H}}.$$
 (5)

Computing the equilibrium value of the partition coefficient and free extractant concentration, it was possible to plot the graph of the left side of Eq. 5 as a function of the free extractant concentration and fit the values of K_c and α . The obtained values are 4.86×10^{-5} and around 3, respectively, which is close to the values found in the literature (Keller, 2022). Another approach would be to fit the logarithmic form of Eq. 5:

$$\log(\kappa) - \beta \rho H = \alpha \log(C_{\rm RH}) + \log(\kappa_{\rm c}), \qquad (6)$$

where α is here the slope and log(K_c) its y-intercept. This linear fitting is presented in Fig. 6 as it is most commonly used in the literature. However, the power fit allows to minimize the absolute error between the curve and the data, which is not the case of the linear fit in Fig. 6.



Fig. 6: Linear fit of experimental results for K_c and α evaluation

Since D2EHPA reacts as a dimer with cobalt (Keller, 2022), the extraction reaction becomes

$$\operatorname{Co}^{2+} + 1.5 \overline{\left(\operatorname{RH}\right)_2} \Longrightarrow \overline{\operatorname{CoR}_2\left(\operatorname{RH}\right)} + 2\operatorname{H}^+.$$
 (7)

Knowing the equilibrium constant and the stoichiometry of the reaction, cobalt extraction curves with D2EHPA can be modeled for different conditions. The model is implemented using Eq. 2 and mass balances on metal and extractant. Using fixed-point iteration, it is possible to predict the equilibrium concentrations of all components for a defined *p*H, for any initial concentration of cobalt and D2EHPA. This model was used to predict the cobalt extraction curve



for the different initial D2EHPA concentrations used in the experiments, which are compared with the experimental points in Fig. 7. Quite excellent agreement can be observed.



Fig. 7: Experimental and correlated extraction curves for varying *p*H and D2EHPA concentrations

Next Steps

The experimental protocol and evaluation methods have been validated for cobalt extraction with D2EHPA. The next steps will be to further investigate this material system by varying extraction conditions such as metal and extractant concentrations, and the acid and base used. A new master student is currently working on this topic to provide a better understanding of the system for other uses. All results will also be compiled in the experimental database for the evaluation of K_c and α . The experimental procedure will then be applied to other material systems that are technically relevant in metal recycling.

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Technical Process for Artificial Photosynthesis

Andreas Pfennig

Lessons Learned from Previous Projects

Over the past decades, several collaborations with Evonik Industries AG and in particular with Creavis, "Evonik's innovation incubator", have been implemented. Our team has regularly been responsible for developing and supporting solvent extraction downstream of biotechnological production steps. The fermentation broth typically has properties that pose a significant challenge to solvent extraction. Microorganisms and solids can lead to stabilization of an emulsion when the organic extractant is added. Electrolytes in the broth strongly influence coalescence of the resulting dispersion and modulate the influence of solids. Unfortunately, the biology of the process is usually worked out and optimized before the process is handed over to those responsible for the downstream. Such a sequential process development unfortunately ignores the positive feedback that could result from directly considering the influence of, for example, the nutrient salts or buffer system used in fermentation on the downstream process.

Because of these challenges, much work resulted for extraction design in the projects like Diamine, CarboxyFun, or Bioxamine, some of which are reflected in the corresponding Ph.D. theses like that of Andreas Bednarz (2019). Our studies included measurement and modelling of the equilibrium of the reactive extraction commonly used. In addition, mass-transfer studies were carried out, which form the basis for the design of technical equipment. The challenges then typically arose when studying the phase-separation behavior together with the influencing factors mentioned above.

The Rheticus Project

This collaboration has continued until today, but – of course – regularly comes along with corresponding secrecy agreements. As there has been some news about some general details of the Rheticus project that started in 2018, it can finally be briefly presented here. The basics of this project have been published by Haas et al. (2018) and are nicely presented in a video by Dr. Haas (2023). The basic idea is to use artificial photosynthesis for the production of specialty chemicals. In a first step, green hydrogen is produced by electrolysis with electricity from PV by the project partner Siemens. The hydrogen together with CO₂ is then converted into products such as hexanoic acid by fermentation with bacteria such as Clostridium. The product is then separated from the fermentation broth by solvent extraction and purified by distillation. The pilot plant shown in Fig. 1 and especially the extraction recently received the ultimate honor when the Dutch King Willem-Alexander and Hendrik Wüst, Minister President of the State of North Rhine-Westphalia, laid their hands on it!



Fig. 1: Rheticus pilot in Marl. Photo: Evonik

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Liège Autumn

Vu Dinh Khang

During my stay in Liège for learning and doing research at Liège University, I had the opportunity to experience 4 different seasons in Liège City. Liège autumn really gives me the most unforgettable impressions and memories.

The most striking feature of Liège in autumn was undoubtedly the breathtaking foliage. As the leaves of the trees lining the streets and parks gradually turned into vibrant shades of red, orange, and gold, the cityscape became a picturesque canvas. Walking through the Boverie park, or along the Meuse River, I was surrounded by a symphony of colors that created a serene and inviting atmosphere. The crisp air, tinged with the scent of fallen leaves, added to the enchantment of the season.



Fig. 1: Meuse river in Liège autumn afternoon

Liège's cultural richness also shone during autumn. The city's numerous cafes offered warmth where I could savor delicious Belgian chocolates, waffles, and aromatic coffees while watching the world go by. The October Fair along the Boulevard d'Avroy and local markets were brimming with seasonal delights, from freshly harvested apples and pears to regional specialties that were a delight for the taste buds.

Autumn in Liège is a time when the city's cultural events came to life. From art exhibitions to music festivals and theater performances, there was always something happening to stimulate the mind and soul. Furthermore, as a student, the learning atmosphere during this season is especially exciting. The University of Liège's libraries and grounds provide the perfect backdrop for focused study and research, complemented by the charm of the changing leaves outside the windows.



Fig. 2: Late autumn afternoon in Liège, view from the dormitory of ULiège

Liège's autumn has a charming beauty when the weather begins to get cold, containing within it the sadness of a person living far away from family. I am fortunate to have the warm care of teachers, colleagues and friends here.



Fig. 3: Autumn outside at the ULg campus

Liège autumn is so beautiful to me.

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Chemical Engineering Department and Food, a Never-Ending Story

Marc Philippart de Foy, Ezgi Uslu

Introduction

Our Department of Chemical Engineering is made up of around 70 people. So, it is hard to get to know each other well, and we are sometimes surprised to meet someone we have never seen before in the corridor. One way of extending relations between us is to organize departmental lunches. And it is fair to say that in this respect... we cannot complain!

Christmas Lunch 2022

The first event on this list took place at the very end of 2022: the traditional Christmas lunch! The whole department got together that day to share a delicious lunch provided by a caterer. As is tradition in our department, the new arrivals had also prepared the aperitifs. In the end, we had a fairly copious buffet, from which everyone could choose their favorite dishes. And for dessert, a colleague had even prepared some delicious Yule logs, of course!

Exotic Lunch

A few months later, the department got together for an exotic lunch. It was an opportunity to try dishes that we're not really used to. For example, cooked bananas were on the menu, as well as noodles or meat prepared according to an African recipe. Fig. 1 shows the group photo taken on this occasion. As always, everyone got together, first to set the tables, then to eat and finally to wash up together. At this kind of event, it's not just the tasting that brings everyone together, but also the washing up!



Fig. 2: Exotic lunch, photo by E. Plougonven

Annual Barbecue

In July, the annual barbecue was held, but this time outside, to take advantage of the fine weather on the day. As you can see from Fig. 2, everyone in the department enjoyed the good food and the good atmosphere. Once again, it was an ideal opportunity to discuss the latest departmental news with colleagues outside of work. The main topic that day was a colleague's PhD defense which had taken place the week before.



Fig. 2: Barbecue of the department, photo by E. Plougonven

Perspectives

These were the three main lunches organized last year, as well as the international lunch to which an individual chapter is devoted in this annual report. But as far as food is concerned, even though the year is almost over, we still have some great prospects ahead of us. Christmas lunch will be arriving soon, as will the Christmas lecture our team will be giving to master students. During this talk, we will be presenting our work to them, and then enjoying Christmas cookies we have baked together especially for the occasion! So, it is safe to say that in our department, the pleasures of the taste buds are rarely forgotten...

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International Lunch

Andreas Pfennig

Another food event organized at the department was an international brunch on October 30. All members of the department were invited to prepare dishes from their home country and contribute them to the brunch. In addition, all those who wished to do so were asked to make the recipes available on the department server, from which the recipes were later compiled together with photos into an International Cookbook 2023 to help digest the impressions of this brunch. Practically all contributors also made their recipes available. On the next page you will find one of the recipes that obviously represents Germany, sauerkraut rolls, which are also suitable as finger food. The diversity of nations at the department was reflected in the range of dishes prepared. From Liège waffles and French quiche Lorraine to Turkish potato börek and Vietnamese salad rolls. The photos on this page, all of which were photographed by Erwan Plougonven, give a first impression of the delights offered to the eyes and taste buds.



Fig. 1: Liège waffles by Antoine Rouxhet



Fig. 2: Vietnamese salad rolls by Ms. Xuan Ngoc Anh Tranm, Mr. Tan Nhat Luong, and Ms. Hong Do Thi Thu





After the success of this brunch, which also showed the breadth of the nations present at the department, it can be assumed that this event will be repeated.

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Sauerkraut Rolls (vegan)

for about 20 pieces

	Drain
400 g sauerkraut,	squeeze out carefully, and collect the sauerkraut juice. In a bowl mix
400 wheat flour,	
1 sachet dry yeast,	
2 tsp sugar,	
1 tsp ground caraway seeds, 1 tsp salt.	
	Make a depression in the center. There pour
1 tbsp olive oil,	
50 ml sauerkraut juice	and
150 ml lukewarm water	and knead everything into a smooth dough. If the dough is too dry, add a little more water. Cover the bowl and let the dough rise in a warm place for an hour, until the dough volume has about doubled.
	Meanwhile, finely dice
1 red onion,	
1 yellow or red bell pepper	and
100 g smoked tofu	and mix in a bowl with
50 g of grated vegan cheese.	
4=0	In another bowl mix
150 g vegan sour cream	with
1 tsp sait	and
2 tsp lemon juice	to a soft cream.
	surface as rectangularly as possible. Spread half of the sour cream, the diced ingredients, and the sauerkraut thinly and evenly on each half.
	dough with the topping. Press the uncovered 2 cm firmly so that the
	place them on baking trays lined with baking paper so that they are
	spaced apart enough, and flatten them a bit.
	Let the sauerkraut rolls rise for 15 minutes.
	Meanwhile, preheat the oven to 200 °C.
	Brush the sauerkraut rolls with water. Sprinkle
1 tbsp of caraway seeds	and
50 g grated vegan cheese	on top.
	Bake the sauerkraut rolls for 15 to 20 minutes until lightly brown.

Comment

Sauerkraut is typically prepared adding salt. Thus, the amount of added salt in this recipe should be adjusted depending on the saltiness of the sauerkraut used.







Ph.D. Extraction Seminar – DECHEMA in Aachen

Ezgi Uslu, Marc Philippart de Foy

DECHEMA is the expert network for chemical engineers and biotechnologists in Germany. This year on September 28th and 29th they organized a Ph.D. seminar on Extraction in Aachen, Germany, where we had the opportunity to present our work to other Ph.D. students from all over Europe who are working in the field of extraction. We would like to thank Prof. Andreas Jupke, Professor of Fluid Process Engineering at RWTH Aachen University, for hosting the seminar and inviting us to participate in this workshop. The goal of this seminar was basically to network with Ph.D. students within the same field and to discuss overarching questions and problems in the field of extraction to identify further collaborations.



Fig.1: Seminar group photo

In this seminar, which was attended by about 16 students as shown in Fig. 1, each of us gave a 15-minute presentation about his or her work, followed by a 15-minute discussion period. Ezgi Uslu presented 'Reactive Extraction Mass Transfer Kinetics and Results' while Marc Philippart de Foy presented 'Stoichiometry-Equilibrium Experiments and Results'. Photographs taken during our presentations are shown in Figs. 2 and 3.

On the second day of the seminar, in addition to the presentations, we had the opportunity to visit the chemical engineering laboratories of RWTH Aachen University. RWTH Aachen students showed and explained the equipment they were working on. All the experimental equipment and laboratories used for extraction were also shown by the technician. It was a very nice and informative technical trip.



Fig. 2: Ezgi Uslu presenting the single-drop mass-transfer cell



Fig. 3: Marc Philippart de Foy presenting reactive-extraction results

It was not only a highly informative Ph.D. seminar, but it also exposed our work to different viewpoints and perspectives within our field, thus broadening our understanding of the topic and helping us to think critically about our research from various angles. The next Assembly on Extraction by DECHEMA will be held in Dresden in February 2024, where we will attend to present our work to a broader audience.

We also had the opportunity to spend some time in Aachen during our free time. We visited the Aachen Cathedral, the Charlemagne Square, and the City Hall. And we tried some of Aachen's specialties, especially the Aachener Printen, which was our favorite. If you happen to be in Aachen, give it a try!

For further questions and comments, please contact: Marc Philippart de Foy, Ezgi Uslu phone: +32 (0) 4 366-3559 marc.philippartdefoy@uliege.be ezgi.uslu@uliege.be





An up to date list of publications is available at: <u>https://orbi.uliege.be/ph-search?uid=U222548</u>

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