

Separation and Purification



2021

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Dear Reader

The year 2021 was again very much dominated by the COVID 19 pandemic, where this year we knew what to expect and how to deal with it. All options for videoconferencing were already installed and all errors with sound or video we had already experienced, so virtual communication worked smoothly most of the time. With the new academic year starting in Belgium in mid-September, the rector decided that the pandemic was essentially over for us and that wearing masks would be enough to keep the virus away. And if it is possible to keep a distance of more than 3 meters, there is no need to even wear a mask as a lecturer. It should be noted that in Belgium any mask will do, including community masks. Now, as a cautious person, I always wear a mask, in lectures FFP2 masks, which have a relatively good effect (Bagheri et al., 2021). Since teaching in a foreign language requires good articulation to be understood with the mask, I use a voice amplifier to help me speak more quietly and clearly while saving my voice.

A turning point this year was July 15, when climate change also hit Belgium with severe flooding, especially in Pepinster, the small town where I live, which even made it into the German evening news. Luckily, we live a bit uphill so we weren't directly affected, but to this day I have to take detours to get to work because the bridges I used to cross no longer exist. I used to drive along a small river, the Vesdre, where today it is quite depressing if you try to take that route. For about 20 km, destroyed houses, ruins for sale, collapsed walls, piles of rubble every umpteen meters and closed stores line the road. And lots of construction traffic lights, especially for some bridges or their remains. The human destinies associated with this destruction, along with the Corona pandemic, leave one worried and a little humbled about our own fate, which so far has allowed us to get through these crises quite well.

As for our research, the Phos4You project was completed, with Zaheer and David successfully running the demonstrator at three different sites. In addition, a new project has just been approved that will look at a new process structure that will allow separation of essentially any number of components - here we are looking for industrial partners to potentially help us filing a patent. The main application of this new process structure is foreseen in the context of urban mining for the separation of metallic components such as rare earth metals from electronic scrap or other similar sources.

Finally, towards the end of the year, the long-standing collaboration with TGGS (Thai-German Graduate School of Engineering) in Bangkok was revived. We have remained in contact during the last years, but currently a new project is being launched, which hopefully will soon be supported by a jointly supervised PhD student. Our Memorandum of Understanding is currently being renewed. Therefore, this annual report includes a guest article by Assoc. Prof. Dr. Tawiwan Kangsadan with her co-authors, Assoc. Prof. Dr. Kraipat Cheenkachorn having been with our team in Aachen for a few months many years ago.

So: Enjoy reading!

Andreas Pfennig

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Lab-Scale Five-Stage Mixer-Settler Battery

Marc Philippart de Foy

Mixer-Settlers

Every three years since 2011, the European Union updated the EU list of critical raw materials. This list includes all raw materials that are both economically important and present a high supply risk. In 2011, the list contained 14 materials. It has been extended to 30 in 2020 (European Commission, 2020). Recycling plays a role in reducing dependence on the supply of these materials. Thus, recycling processes e.g. for metals in urban mining should be developed. Streams from urban mining usually contain several metallic components that need to be separated, for which different methods are available. Reactive extraction is studied here as one option. Given the equipment available in the laboratory, experiments will be carried out with mixer-settlers, which are the subject of this report.

Mixer-settlers consist of a combination of a mixing chamber and a settling tank. They can be used in extraction processes that require first mass transfer and then phase separation. A drawing of a mixer-settler is presented in Fig. 1. The heavy and light phases first enter the mixer, which induces dispersion of one phase into the other. The mass transfer of the component to be extracted occurs in the mixer. The mixture then flows through the settler where the phases separate. The heavy phase is removed at the bottom and the light phase at the top.

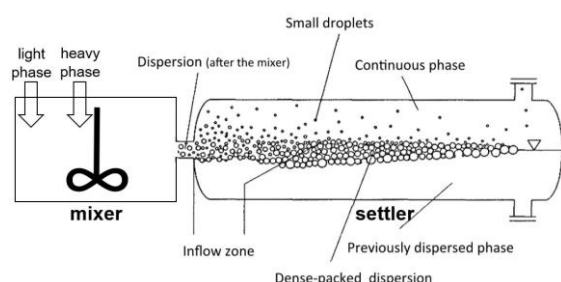


Fig. 1: Mixer-settler (after Henschke, 1995)

The main advantages of mixer-settlers are their flexibility and the possibility of achieving good separation even at extreme phase ratios. The main drawbacks are the large amount of energy required to operate them, the high ground-area demand and the high process costs.

Another characteristic of such equipment is that a mixer-settler is a good approximation of a theoretical stage, provided that the mixing intensity and residence time are sufficient to achieve extraction equilibrium. Several mixer-settlers can also be connected to form a mixer-settler battery. Such equipment can be operated in a continuous fashion, in cross-current or in counter-current. Mixer-settlers are, therefore, a good tool to experimentally validate theoretical models of extraction processes.

Lab-Scale Five-Stage Mixer-Settler Battery

A small-scale five-stage mixer-settler battery made of borosilicate glass 3.3 is available in the laboratory, as shown in Fig. 2. It was built by De Dietrich Process Systems - QVF in the early 2000's and has been used for two master theses by Coruh (2003) and Börcek (2005).



Fig. 2: Five-stage mixer-settler battery available in the laboratory

The equipment consists of five successive mixer-settlers that can be operated in counter-current. The two phases are mixed in the mixers before entering the settling tanks. The mixing is generated by rotating magnetic parts at the bottom of the mixer. The rotation is transferred by belts that rotate magnetic parts under each mixer. In the settlers, a tube is used to manually adjust the overflow level to control the position of the interface. Fig. 3 shows the connection between a mixer and the settler into which the two phases enter after the mixing stage. The mixer can be seen at the bottom left while the settler is the horizontal tank at the top.

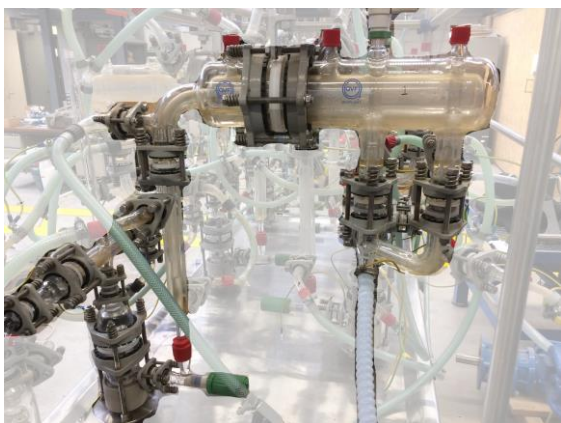


Fig. 3: Mixer and settler in the equipment

The temperature of the fluids can be controlled during operation of the equipment. The glass walls of the unit are double-walled to allow water flow for thermostating. The water circulates in a closed loop and a chiller is used to adapt both its temperature and flowrate. The special feature of this equipment is that all the glass parts in contact with the phases are temperature controlled. This also prevents temperature changes between the mixers and settlers.

Fig. 4 shows the flows of both, the light and the heavy phase in the mixer-settler battery. The two phases enter at either side of the battery and each phase follows a pre-defined path. The phases meet in counter-current, which means that the entering stream of the first phase is mixed to the leaving stream of the second phase, etc.

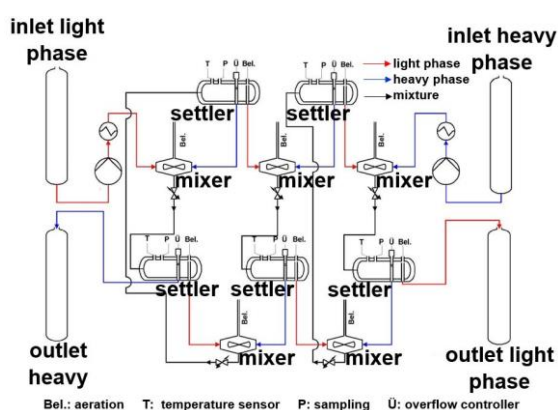


Fig. 4: Scheme of the five-stage mixer-settler battery (after Coruh, 2003)

This five-stage mixer-settler battery had not been used for nearly two decades, so some repairs were needed before it could be operated. After cleaning the dust off the walls, the belts

were replaced, the few missing or broken pieces were ordered and the two-phase and temperature-control circuits were tested with water to ensure that the equipment does not show any leakage. As volatile diluents will be used, all metal parts need to be grounded to comply with ATEX regulations. This will be the last step in the preparation of the equipment before conducting experiments.

Use in the Future

The lab-scale mixer-settler battery will be used in the coming years to conduct reactive extraction experiments. Different metals will be studied. The metals will be selected among those coming from urban mining to obtain practically useful results for their recycling. A student will also conduct beaker experiments to write her master thesis on the validation of a prototype workflow based on lab experiments. Ni(II) and Co(II) will serve as test systems as these metals have already been studied thoroughly in the literature. The results obtained will be used to design experiments with the mixer-settler battery.

The results of the experiments conducted in the mixer-settler battery should contribute to the development of a new process that could improve metal recovery yields in urban mining.

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Settling Experiments Using Iso-Optical Systems

David Leleu

Starting Point

A coalescence model has been developed during the last years (Leleu and Pfennig, 2019a). The model is very general and can be adapted to different scenarios, e.g. sedimenting drops or drops in a close-packed environment. In parallel, a simulation tool, based on the ReDrop approach, has been built in order to simulate the separation of two-phase systems. The coalescence model is integrated into this tool as well.

The ReDrop tool allows detailed representation of the settling by evaluating the hold-up profile versus time and height of the dispersion. However, usual settling experiments result in limited data, e.g. the characteristic curves of the separation. To validate the ReDrop program and thus the models on the available level of detail, ideally, experimental hold-up profiles should be available. One especially simple solution to this challenge is the use of iso-optical systems. In an Iso-optical system, two liquid phases have an identical refractive index. This leads to the interfaces and thus the drops not being visible. A dye is then added to one of the phases so that during the settling experiment the color intensity is directly proportional to the drop concentration and thus to the hold-up. This can be quantitatively evaluated with the Beer-Lambert law.

Settling experiments using iso-optical system have been performed to measure experimentally the hold-up evolution, as shown for one system in Fig. 1. The results are then to be reproduced with the ReDrop simulation.

Chemicals and Experimental Set Up

The settling cell initially developed by Henschke (Henschke 1995, Leleu 2017) is used to perform the settling experiments. The dispersion is created by stirring at e.g. 800 min^{-1} during 30 s. The cell is illuminated from behind with an LED panel. A thermostatic bath is connected to the equipment to control temperature and a SOPAT probe is used to measure the drop size. Experiments are recorded with a video camera. The movie is then treated with MATLAB to evaluate the evolution of the hold-up profile.

The iso-optical system is generated with three major chemicals: hexane, ethylene glycol, and water. Sodium chloride is added to the aqueous phase at a concentration of 50 mmol/L. Methylene blue is added to the system at very small concentration to obtain the hold-up profile.

Density and viscosity of each phase are measured with the DSA 5000M combined with Lovis 2000ME (Anton Paar). The interfacial tension is measured using the Du Noüy ring method. Those data are necessary for reliable ReDrop simulations.

As the interface is not visible at the iso-optical point, measurement of the drop size with the SOPAT probe is not possible. To determine the drop-size distribution, slight changes in concentration are introduced so that the interfaces become visible and can be detected with the help of this optical probe.

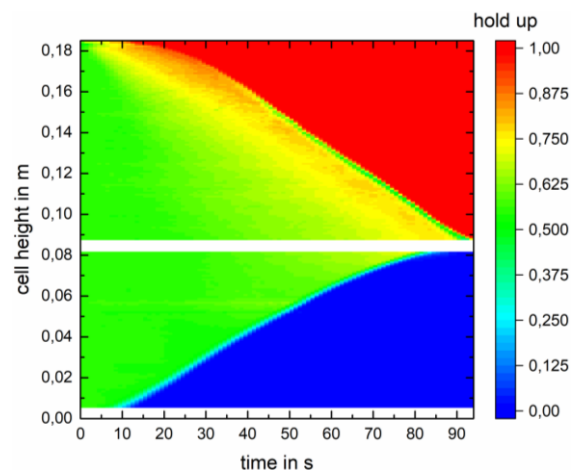


Fig. 1: Iso-optical settling experiment, organic-phase fraction of 53 %

Results

Different experiments have been performed at different hold-up and different stirring speed to generate different initial drop-size distributions. Fig. 2 shows the influence of the stirring speed on settling behavior. After the first seconds, the sedimentation curves have the same slope, i.e. identical average drop size. Nevertheless, the

two sedimentation curves show a shift, which is induced by a different curvature of the sedimentation curve during the first seconds. An increase of the stirrer speed reduces the initial drop size. As the slope increases over time, the drop size increases. Within few seconds, apparently an average drop size is reached, which is rather independent of stirrer speed.

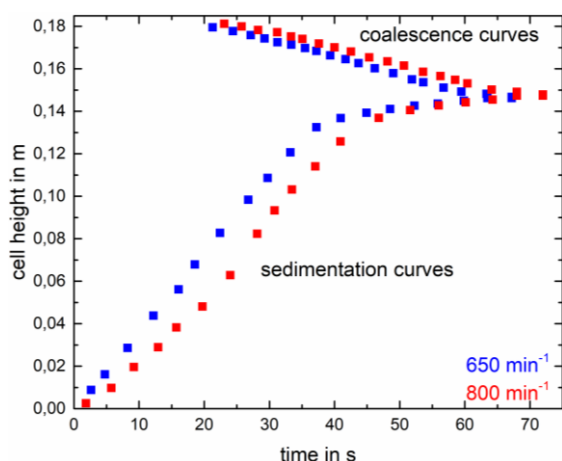


Fig. 2: Characteristic curves for two different stirring speeds, 20 % organic-phase

Measuring the evolution of the drop-size at the beginning of the settling with the SOPAT probe confirms this interpretation, as shown in Fig. 3. For the case shown, the initial mean diameter differs by 100 μm between the two stirrer speeds. After few seconds, the drop sizes become quite similar. This was observed for all the settling experiments. It shows that coalescence occurs during the first seconds of the settling until a given drop diameter characteristic for the material system is reached.

Different phase ratios were investigated. In each case, the hold-up increases smoothly from the initial hold-up to a maximum of 85 % near the major interface. Squeezing of the drops sets in beyond a hold-up of spherical close packing, i.e. 0.74. This value thus corresponds to the hold-up where the drops are just entering into the close-packed zone (Henschke, 1995).

In Fig. 1, the close-packed zone, shown in orange, has an almost constant height over time. However, the hold-up in the close-packed zone reaches its highest value at the beginning of the experiment, i.e. when the coalescence with the interface is still slow compared to few seconds later. These two observations are related. Coalescence between drops with the interface induces an excess of continuous phase that has

to flow through the channels present between the drops. This flow induces a pressure drop acting in the opposite direction as buoyancy. By this pressure drop, the drops tend to separate and thus the hold-up decreases. The influence of the flow of continuous phase through the close-packed zone is quite significant. It needs to be accounted for in the ReDrop simulations.

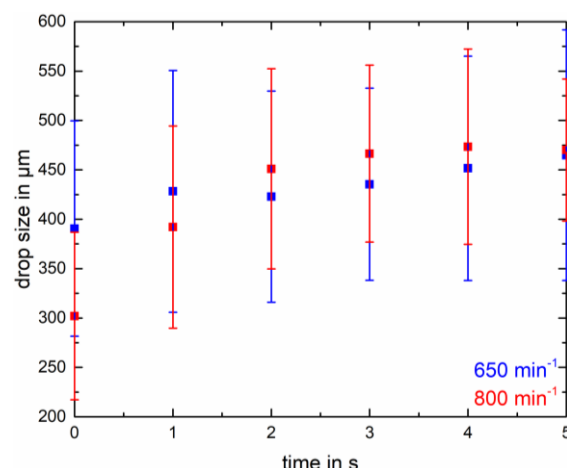


Fig. 3: Drop-size evolution during the first seconds for different stirring speeds.

Simulation and fitting with the ReDrop tool are still continued at this moment to model exactly the coalescence behavior of the droplets during batch-settling experiments

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Electrocoagulation Separation Process

Tawiwan Kangsadan, Thanapong Suwanasri, Kraipat Cheenkachorn

Starting Point

Biodiesel has been promoted as alternative diesel fuel by blending with the fossil fuel at 7% with increasing consumption for B10 in 2020 for the transportation in Thailand. As of December 2020, Thailand is ranked 5th in the global biodiesel production with the production capacity of more than 8.5 million liter per year. Commercially, biodiesel is produced via a transesterification of triglycerides (e.g., refined palm oil or waste cooking oil) and short-chain alcohol (e.g., methanol or ethanol) in the presence of homogeneous alkali-catalysts at low temperature and pressure to yield up to 98% conversion.

After the reaction is completed, the main product of biodiesel must be separated from other by-products using a conventional separation process such as gravitational settling or centrifuge. However, one of the major concerns of the desired biodiesel product from the gravitational settling (GS) as the separation process is the contamination of impurities such as excess amounts of unreacted reactants (e.g., alcohol, catalyst, saturated or unsaturated of glycerides) and glycerol. Since this process takes a long time to separate biodiesel from other products, the long settling time leads to a reverse reaction, resulting in a decrease in a product yield. Another method to purify biodiesel in order to meet the standard requirements is a warm-water washing or acidic wash. Nevertheless, water washing has drawbacks because it is a time-consuming process for settling biodiesel from water in order to achieve complete separation. Large amounts of water are consumed for the process leading to the waste water treatment. Moreover, water washing must eventually be followed by an evaporation process at high temperature in order to remove the remaining water during the biodiesel phase, resulting in high capital and operation costs.

Additionally, for the biodiesel purification process, biodiesel is neutralized using acidified water in order to eliminate soap, free glycerol, and catalyst residue. Common acids used in this method include hydrochloric acid (HCl), phosphoric acid (H₃PO₄), and citric acid (C₆H₈O₇). This neutralization method can lead to

remaining acid, where it is possible to obtain a total acid number higher than the maximum limit of EN 14214 (the maximum value of 0.5 mg KOH/g). It also leads to environmental issues in terms of wastewater generation.

The electrocoagulation separation (EC) process has been widely used for a variety of separation multi-components, emulsion solvents, and treatment wastewater. This is an efficient technique because it can remove various small colloidal particles from aqueous media, especially those with charges. By applying an electric current, the ion of small particles is neutralized and destabilized which leads to the aggregation of tiny particles. Then, these massive particles form into larger groupings of enough density to be settled out under the force of gravity. The EC operation modes include low voltage, high direct current (DC), high voltage, low alternating current (AC), and pulse mode of DC or AC. Previous studies done by other researchers have shown that the overall separation efficiency of AC and DC was comparable. However, AC was preferred due to shorter separation time, high removal efficiency as well as impressive color and turbidity, effective energy consumption, and low corrosion rate of electrodes.

Though EC finds an application in wastewater treatment, the application of EC in biodiesel-glycerol separation has been rarely investigated. Therefore, the full investigation of the EC technique for biodiesel separation should be studied in detail starting with this preliminary research by varying factors, including the type of electrode configurations, the distance between the electrodes, and applying high voltage (kV). Moreover, the effects of remaining catalyst and soap before purification were investigated. The final product after purification of biodiesel (B100) was also analyzed and compared to ASTM D6751 and EN 14214 standards.

The Method

Refined palm oil (RPO) and sodium hydroxide derivative, which were used as raw material and a catalyst, respectively for a transesterification reaction, were obtained from a non-disclosure local biodiesel producer in Thailand. The fatty

acid composition of RPO consists of both saturated and unsaturated ones at relatively the same ratio. Saturated fatty acids are Palmitic acid (C16:0) of 43.45% as the major composition while Stearic acid (C18:0) of 4.23% and Myristic acid (C14:0) of 1.10% and Lauric acid (C12:0) of 0.80% are small constituents. Oleic acid (C18:1) of 40.90% and Linoleic acid (C18:2) of 9.10% are unsaturated fatty acids. An analytical-grade methanol was purchased from a local chemical supplier.

Fatty acid compositions and total glycerol in biodiesel were analyzed according to EN 14105 while the methyl ester content according to EN 14103. The remaining soap and catalyst contents were determined by the titration method.

The transesterification reaction of excess methanol and oil at the ratio of 6 to 1 in a presence of sodium hydroxide derivative (2.16%wt) at the optimum temperature of 60°C and ambient pressure for 1 hr was carried out in a batch reactor (Fig. 1) with a constant stirring speed of 500 rpm to produce biodiesel and by-products. At the end of the reaction, the product mixture containing biodiesel, glycerol, methanol, remaining catalyst, and unreacted reactants was then separated EC and compared with GS.

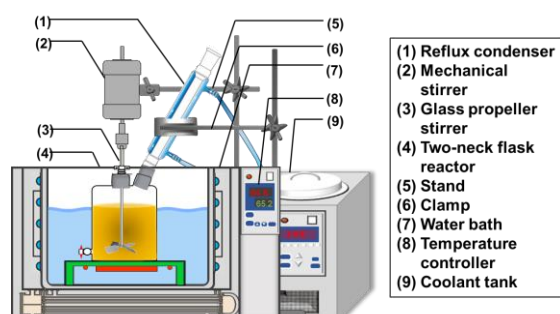


Fig. 1: Experimental set up for transesterification of biodiesel production from refined palm oil in a batch reactor

The EC setup consists of a testing chamber, a transformer unit, and a control unit. As illustrated in Fig. 2, an AC source of 50 Hz and 220 V from the electrical system line was transmitted to an auto-transformer to increase the voltage from the primary to the secondary side of 0-100 kV. Finally, the desired high voltage in range of kV_{ac} was applied to the test chamber. The test chamber comprised of a pair of Fe electrodes that were filled with the product mixture for the separation test. The desired voltage was adjusted by a control box. The experiments were conducted in a Faraday cage for safety.

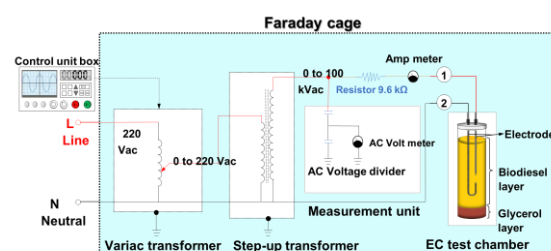


Fig. 2: Schematic diagram of EC set up

Previous experimental results demonstrated that the point-to-point electrode configuration as displayed in Fig. 3 generated the strongest electric stress resulting in the highest separation efficiency of 99.8%. The experimental results had been confirmed with the simulation results of electric and magnetic fields using Finite Element Method Magnetics (FEMM 4.2) as demonstrated in Fig. 4. For the safety operation, the smallest vertical-distance between electrodes (d) was recommended to 3 cm and the applied AC voltage of 3 kV was sufficient to reach the same separation efficiency.

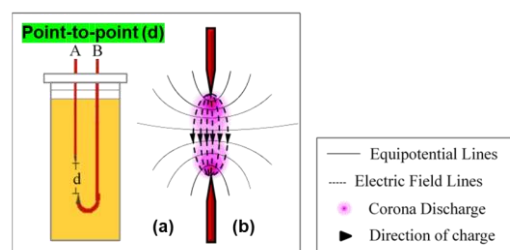


Fig. 3: Schematic of point-to-point Fe electrodes configuration (a) and generated electric and magnetic fields (b)

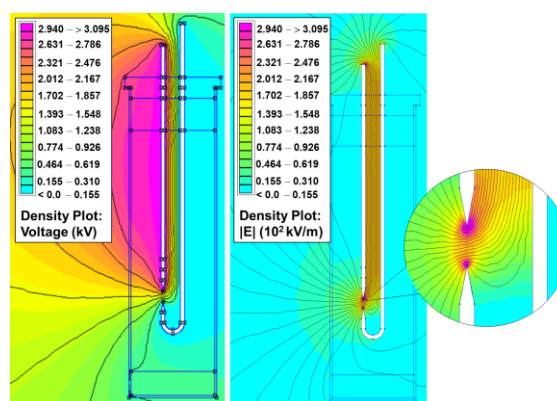


Fig. 4: FEMM Simulation of electric field and magnetic field for point-to-point Fe electrode configuration

As seen in Fig. 5, higher operation time results in higher separation efficiency compared to shorter operation time. The difference is more pronounced at a higher distance between electrodes. The vertical distance between electrodes of 2 cm and 3 cm shows the highest separation efficiency of 99.8% at the operation time of 240 s. Since electrical stress is inversely proportional to the distance between electrodes, higher distance shows less electric stress, resulting in decreased induction of free particles and released charges between electrodes. Hence, the conductive zone between electrodes was reduced and the EC process did not show a significant effect on the sedimentation of the glycerol particles.

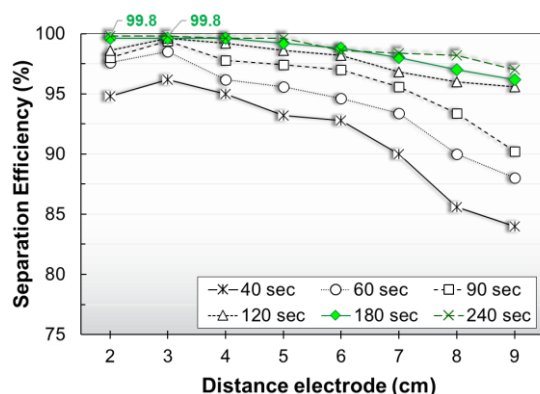


Fig. 5: Effect of vertical-distance between electrodes on separation efficiency of EC process by applying AC at 3 kV

A clear interface between the biodiesel and glycerol layers was first observed at a short separation time within 40-60 s. However, the purity of the final biodiesel product did not meet the standards and the remaining catalyst and soap contents were still high. In order to meet the standards and to reduce the remaining catalyst, the separation time was extended as illustrated in Fig. 6. The removal of glycerol was faster for the applied AC voltages higher than 3 kV. In order to achieve more than 99% separation efficiency, the separation time had to be longer than 120 s for an applied AC voltage greater than 3 kV. The optimal conditions for the EC separation process with the maximum separation efficiency of 99.8% determined at the shortest separation time and low energy consumption (low current) were at 3 kV for separation time of 240 s. Biodiesel can be contaminated with glycerol, the homogenous catalyst, and the remaining excess methanol. It is essential to remove these chemicals in order to meet the acceptable standards. Generally, the catalyst is

simultaneously removed from biodiesel via the precipitation of glycerol using the gravitational method because of the polarity of both species. However, the trace amount of catalyst still remains in the biodiesel phase. Fig. 7 and 8 show the normalized concentration of remaining catalyst value versus separation time for GS and EC, respectively. GS takes more than 24 hr to totally eliminate the remaining catalyst from the biodiesel. In contrast, EC significantly improved the catalyst removal process with totally removed from biodiesel in a shorter time of 240 s.

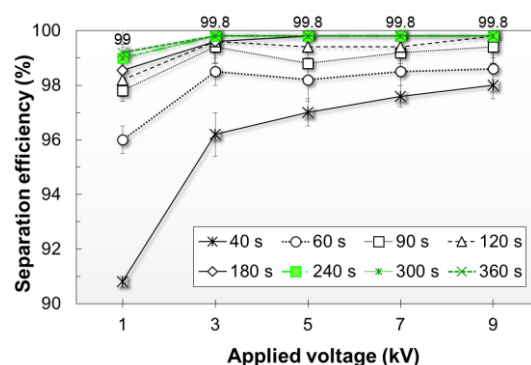


Fig. 6: Effect of applied AC high voltage on separation efficiency of EC process at vertical-distance between electrodes of 3 cm

Since soap can be formed from the reaction of the remaining catalyst and glycerol in the presence of water as saponification reaction, it must be removed from the biodiesel by washing with water because the soap can lead to an emulsion formation in the biodiesel, requiring longer washing time, and resulting in loss of biodiesel yield during purification process with water-wash. The soap value content in the biodiesel final product prior the purification process was determined using the titration method and results presenting in the normalized concentration of remaining soap value versus the separation time for GS and EC processes are depicted in Fig. 7 and 8, respectively. GS required a separation time of more than 24 hr in order to obtain the lowest normalized concentration of remaining soap value of 0.037. For EC separation process, the normalized concentration of remaining soap value in the biodiesel layer gradually decreased from 0.083 at a separation time of 30 s to 0.050 at a separation time of 180 s. After that, it obviously decreased to 0.035, still higher than the standard of 0.0084, at the separation time of 240 s. However, the separation time showed no significant decrease of normalized concentration of the remaining soap value after 240 s.

This is due to the fact that the catalyst and glycerol were efficiently eliminated from the biodiesel during the EC process, resulting in less soap formation.

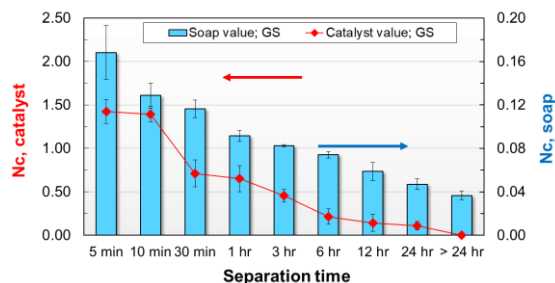


Fig. 7: Normalized concentration (Nc) of remaining catalyst and soap value in biodiesel obtained from GS prior purification process

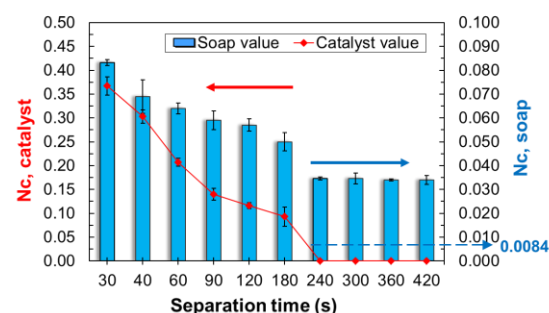


Fig. 8: Normalized concentration (Nc) of remaining catalyst and soap value in biodiesel obtained from EC prior purification process

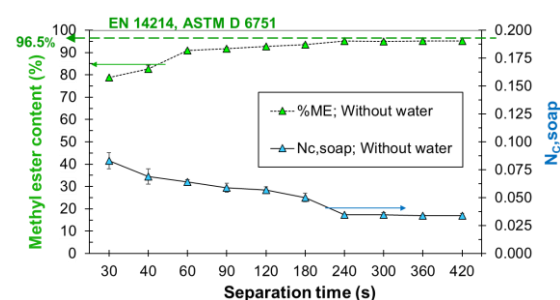


Fig. 9: Methyl ester content and normalized concentration of soap content (Nc, soap) in biodiesel obtained from EC prior purification process

EC takes only 60 s to meet the methyl ester content of $90.9\% \pm 0.5\%$ as shown in Fig. 9 whereas for GS, it takes more than 12 hr to achieve the ester content of $93.3\% \pm 0.2\%$. This is because most of the contaminants were removed for the

EC process, resulting in a more purified product. According to Thai commercial standards and international standards, the required methyl ester content should be $\geq 96.5\%$ wt. Though the ester content from the EC process did not meet the requirements, further purification process is necessary for a higher percentage of methyl ester content, such as water washing, etc.

All of the main properties of the biodiesel obtained from the EC separation process meet the standards using either the ASTM or EN methods. Interestingly, after washing only three times with warm water, the ester content was $98.5\% \pm 0.25\%$ wt., which is considerably high compared to the conventional process after washing five times with warm water. The acid value of such a product was also low. Moreover, the contents of the mono-, di-, tri-glycerides, the total glycerol and free glycerol met the standard requirements. Present research has demonstrated that the EC separation process shows high separation efficiency is a time-saving process. This method can also be applied to other multi-components OH and emulsion mixtures.

Acknowledgements

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Phosphorus Recovery Process: Applied for Waste From Vietnam

Khang Vu Dinh

Goals and Approaching Methods

Chemical (CheS) and biological sludge (BioS) from rubber wastewater-treatment plant (WWTP) in Southern Vietnam are two studied materials for phosphorus (P) recovery. A P-recovery process is considered effective when it achieves at least three objectives:

- 1 high P-recovery efficiency,
- 2 low operating costs, and
- 3 is environmentally friendly.

The cascaded option-tree (COT) method (Bednarz et al. 2014) was used to analyze the potentials, advantages, and disadvantages of currently published processes. This is the basis for proposing the most appropriate solution for a P-recovery process. PASCH, SEPHOS and Aqua Reci® processes showed the greatest attraction for P recovery due to their individual advantages. The advantages of each process were considered as the basis for improvement and included in a P-recovery process suitable for raw-material characteristics of Vietnam. Rubber WWTP mainly uses poly aluminum chloride to remove P in wastewater. Approaching P-recovery technologies by wet chemistry such as these processes provides dual goals, which is to recover P and also Al in the leach liquor. Figure 1 shows the approach and the steps to perform the impurity separation and P recovery in sludges from the rubber WWTP.

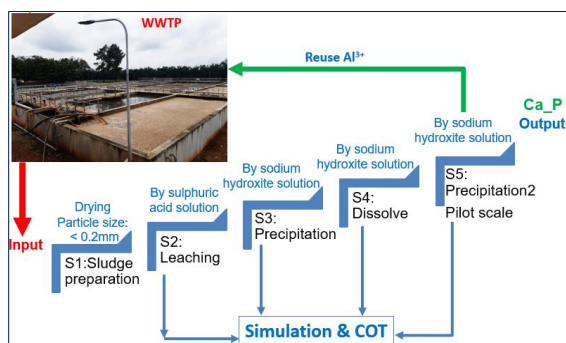


Fig. 1: Phosphorus-recovery process steps

The P-recovery process is carried out in five main steps. This includes the preparation of raw

materials, followed by the steps of metal separation, liquid and solid phase separation. The final product was recovered as a Ca-P precipitate. The research steps of metal separation and precipitation are simulated based on input parameters. Establishing COT is the most reliable basis for finding the most suitable options.

Experimental Results

The leach liquor contains not only high P content but also Ca, Al, and Fe. These metals need to be separated before producing clean recovered products. P and metal are separated by precipitation with a sodium solution in a pH range from 3 to 12.5. The Aqion-Pro hydration program was used to simulate the products with the corresponding saturation indices (SI) (Fig. 2). SI represents the ability to form mineral precipitates in the reaction of leach liquor with sodium solution. If $SI > 0$, the solution is super-saturated. It indicates the possibility of mineral precipitation.

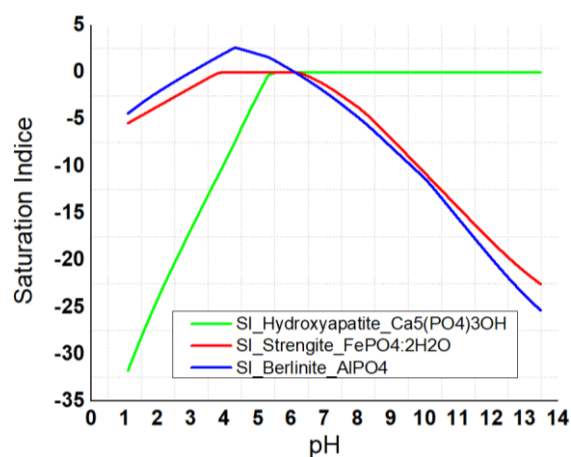


Fig. 2: SI from simulation by Aqion-Pro 7.9

The results for precipitation of P and other metals from CheS leach liquor are shown in the Fig. 3. This shows that the color of the precipitate is different at different pH values. The milky color precipitate appears at low pH values. The color changes to dark orange at higher pH. Iron precipitation products and iron oxide are the main cause of the reddish-brown color.

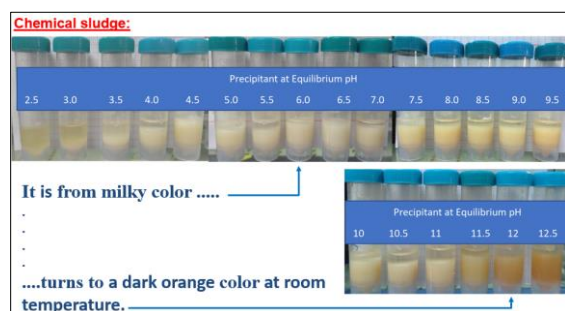


Fig. 3: Precipitation of CheS leach liquor at different pH values

The recovered precipitate was dried at 105 °C to constant weight, as shown in Fig. 4. The precipitate was then analyzed to determine the components, including P content, metals such as Al, Fe, Ca and heavy metals by ICP-OES equipment. The precipitates after drying showed a difference in color. The leach liquor from the chemical sludge had a higher Fe content than that from the biological sludge. The reddish-brown color produced by Fe^{3+} in the precipitated product from the leach liquor of CheS indicates that difference.

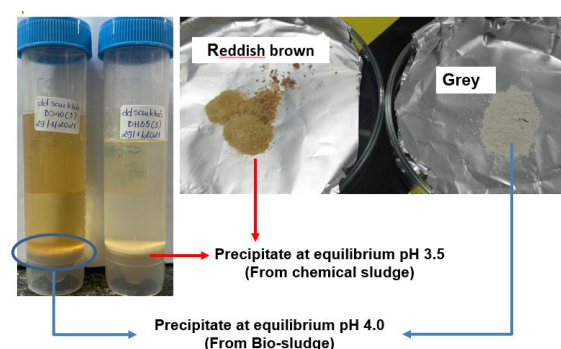


Fig. 4: Precipitate from BioS and CheS leach liquor

The experiments of dissolving the precipitated product with sodium-hydroxide solution at high pH were carried out in the laboratory to evaluate the solubility of the precipitated product, called Pre-Product. The composition of the substance in the dissolved solution is shown in Figs. 5 and 6.

Experimental results show that the solubility of Al and P compounds increases sharply with the increase of alkali content. Fe and especially Ca showed negligible solubility as alkalinity increased. The difference in the solubility of Fe, Al and P compounds in NaOH solution is the basis for separation of impurity metals. Al and P

compounds have the highest solubility in the range of pH 12 to 13 for both pre-products.

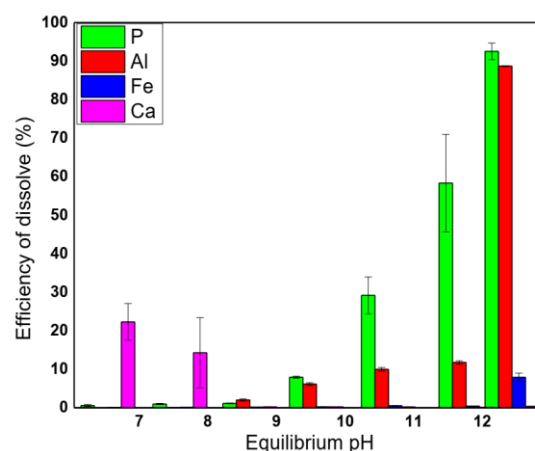


Fig. 5: Ability to dissolve substances in the precipitate from CheS at different pH

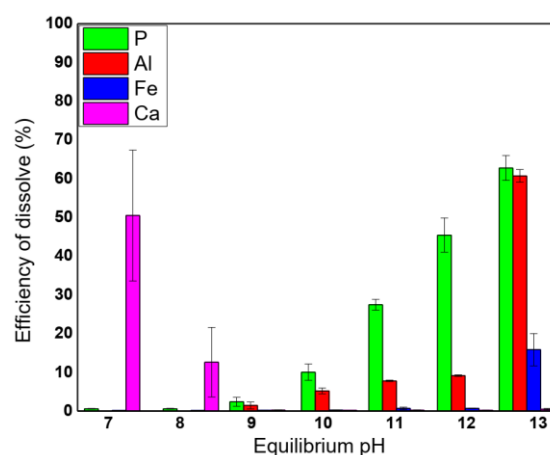


Fig. 6: Ability to dissolve substances in the precipitate from BioS at different pH

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Visit of the Dong Nai Rubber Corporation

Khang Vu Dinh

During the assessment of the potential for P recovery from waste sources in South Vietnam, I was very impressed by the development of the rubber latex processing industry in the Dong Nai province. I had the opportunity to visit and work with one of the largest rubber corporations in Vietnam, the Dong Nai Rubber Corporation (DONARUCO) in Dong Nai province, shown in Fig. 1.



Fig. 1: DONARUCO's headquarters

DONARUCO is a unit of the Vietnam Rubber Industry Group. The main business fields are planting, tending, exploiting, processing and exporting natural rubber. DONARUCO has 10 farms and the total managed area is 33,182 hectares. Vast rubber farm in Dong Nai province is shown in Fig. 2.



Fig. 2: Rubber tree farm in Long Khanh, Dong Nai province

The total annual production was about 30,000 tons. The Xuan Loc-An Loc rubber processing enterprise belongs to DONARUCO. To get to Xuan Lap-An Loc wastewater treatment plant (WWTP) see Fig. 3, I had to go on a road through the green farm with the shade of rubber trees. The scene was so fresh and peaceful.



Fig. 3: Xuan Lap-An Loc WWTP

I had been to Xuan Lap-An Loc WWTP many times before, and this was the first time it did not rain. The flowers blooming in the bright sunshine in the middle of the WWTP create a strange feeling (Fig. 4). It is an emotional interweaving between pollution and vitality. It's wonderful, in the midst of seemingly simple things I find many extraordinary things. Research on P recovery from potential waste sources in South Vietnam is also the solution for me to find miracles.



Fig. 4: Flowers blooming at WWTP

DONARUCO needs a solution to connect a perfect green circle, that is the solution to recover P from waste sources for reuse as fertilizer at rubber farms.

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PULSE Process Demonstrator Trials

Zaheer Shariff

The Phosphorus ULiège Sludge-Extraction (PULSE) process developed at ULiège under the Phos4You project was demonstrated at pilot scale with sludge from Belgium, Germany, and Scotland. The trials were conducted at the Sart-Tilman ULiège campus, on-site at Oupeye wastewater treatment-plant near Liège and at Scottish Water Development Center in Bo'ness (Scotland). The PULSE process is depicted in Fig. 1, in which phosphorous (P) is obtained from dried sludge by acidic leaching followed by extraction of heavy metals to obtain a high-quality P-product that can be used for fertilizer production.

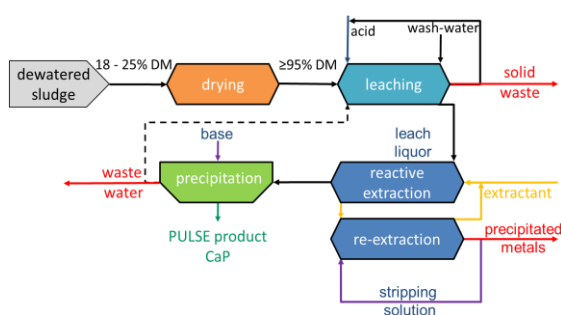


Fig. 1: Process flowsheet of PULSE process

The PULSE pilot plant was designed and constructed so that it can be transported to different locations for demonstration. About 80 to 100 kg of dewatered sludge can be treated per batch in the pilot plant. The pilot plant consists of a batch forced circulation drier, a jaw-crusher, leaching and precipitation tanks, a five-stage horizontal mixer-settler unit, and cartridge filters. The PULSE pilot plant during the trials in Scotland is shown in Fig 2.



Fig. 2: PULSE pilot at Scottish Water Development center in Bo'ness

During the pilot trials it was found that the P content of the 3 sludges was quite different as shown in Fig. 3 and also the content of the other metals and heavy metals.

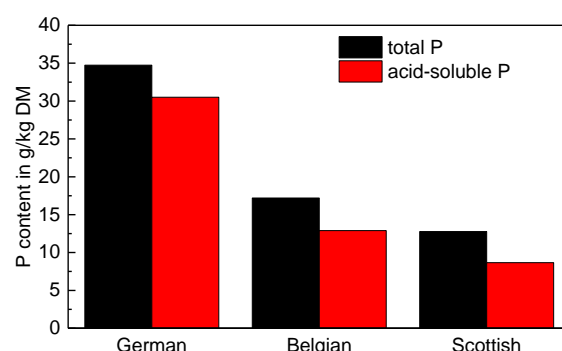


Fig. 3: P content in the different sludges

The process operating parameters for the different input sludges such as leaching pH, solvent composition, organic-to-aqueous phase ratio, and base requirement for P precipitation were optimized using lab-scale experiments and solid-liquid-liquid chemical equilibrium (SLLE) simulations (Shariff, et al. 2020). An example of P-leaching efficiency from German sludge at lab and pilot scale is compared to that obtained with the SLLE tool in Fig 4. In principle, acidic leaching only dissolves the inorganic P contained in sludge, which defines the maximum P recovery efficiency that can be reached. As can be seen in Fig. 4, the results from the pilot trials agree well to those obtained in lab and with the SLLE tool.

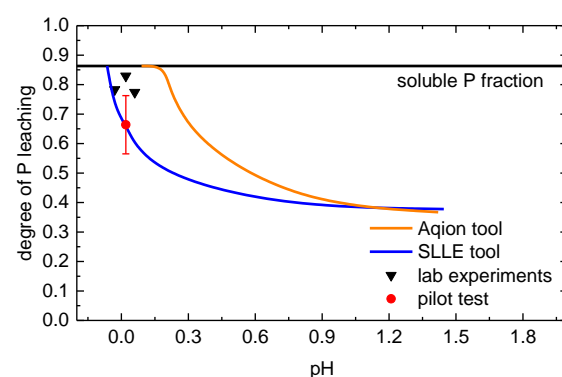


Fig. 4: Leaching efficiency of P from dried German sludge, solid-to-liquid ratio 1:4

After leaching, the sludge is washed with water to recover residual P and acid that is present in the wet solids before disposal. The leach liquor containing the dissolved P and metals is purified by reactive extraction to remove the metals before precipitation of the P product. Metals are extracted in a two-stage counter-current operation using a solvent system containing Alamine 336 as the extractant. The degree of extraction of metals from the German sludge liquor evaluated using the SLL tool and that obtained during the pilot trials is shown in Fig. 5. The used solvent is regenerated by stripping the metals with an alkaline solution of ammonia and ammonium hydrogen carbonate. During stripping, a golden-brown precipitate is obtained which mostly contains Fe, while the other metals such as Cu, Cd, Pb, and Zn remain dissolved in the stripping solution. It is possible to further separate and valorize some of these recovered metals.

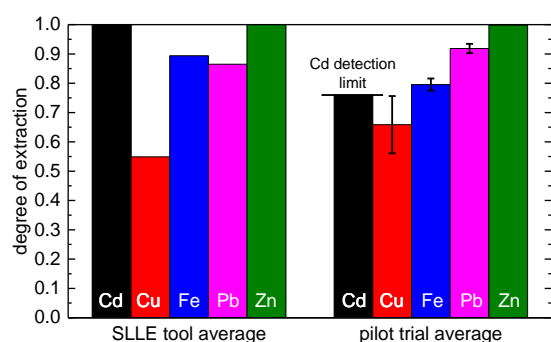


Fig. 5: Metal extraction efficiency during pilot trials with German sludge

After the extraction of metals, the pH of the liquor is increased by addition of NaOH, or $\text{Ca}(\text{OH})_2$, or both to precipitate P as calcium phosphate salts. After precipitation, the product is separated by filtration and dried. The composition of the product obtained from the different sludges is shown in Fig. 6.

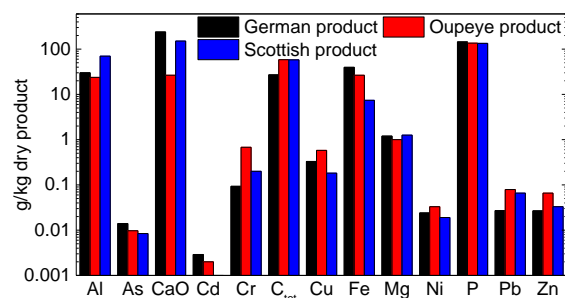


Fig. 6: Composition of the product obtained from PULSE process

The PULSE product has a P content sufficiently high to be used as fertilizer ranging between 10 to 14 % P/kg dry matter. The molar ratios of Al and Fe to P in the product are quite low ensuring a good solubility of P in soil. The content of other toxic metals was well within the limits of the EU fertilizer regulation 2019/1009. The product also responded well to granulation tests performed by Prayon S.A. The summary of the PULSE pilot trials is given in the Table 1.

sludge		German	Belgian	Scottish
source		Dortmund-Deusen	Oupeye	Sterling
site		at ULiège	at wwtp	Bo'ness
amount	kg	~340	~280	~250
P recovery	%	60 to 65	60 to 65	48 to 52
P ₂ O ₅	%	~33	~31	~29

Tab. 1: Summary of PULSE pilot trials

Based on the pilot-plant results, it can be concluded that the PULSE process can be used to recover P from different sludges even with high concentrations of metals such Cd, Cu, Fe, Pb, and Zn without compromising overall P recovery efficiency while guaranteeing a sufficient removal of the detrimental metal impurities. An estimation of the cost of the resource consumption for P recovery by the PULSE process indicates that it would only increment the cost of wastewater treatment by about 9 €-cent/m³.

Acknowledgements

The research work carried out at ULiège in the context of the Phos4You project, was jointly funded by Interreg North-West Europe and the Walloon Region. Complementary product analysis and granulation tests were performed by Prayon S.A. Samples of Exxal 10 were supplied free of charge by ExxonMobil Petroleum & Chemical (Belgium).

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Visit of the Scottish Highlands Between Pilot Tests

David Leleu & Zaheer Shariff

The Phos4You project brings us, the authors, to a small town located at the periphery of Edinburgh, called Bo'ness. The plan in Bo'ness was to demonstrate the PULSE process for recovery of phosphorus from sewage sludge at Scottish Water test facility for a duration of five weeks.

Our trip began with a quarantine of 10 days in the city of Edinburgh prior to the actual pilot trials. During that time we planned our work for the next weeks. Of course, being that close to the Scottish Highlands, working without any sight-seeing was not an option, so we also planned visiting the region during our free time.

The city center of Edinburgh was the first place that we visited. The history behind King Arthur is really well presented with, for example, the Castle and the Arthur seat in the Holyrood Park, from which we have a beautiful view of the city, as shown in Fig. 1.



Fig. 1: View of Edinburgh city from the Holyrood Park.

During one of our weekends, after a full week of leaching tests, we visited the region of Calander, a city at the entrance of the Highlands. We visited the town before climbing Ben Ledi, which reaches an altitude of 879 m. The walk to the summit is 4 km long and rather physical. Unfortunately, the weather conditions were not ideal and the view from the top was a bit foggy.

Our next trip brought us close to Fort William in the Glencoe National Nature Reserve. We walked for 13 km and reached the summit Stob Dubh. This time the weather was better and offered us a stunning view at the top (Fig. 2).



Fig. 2: Glencoe National Nature Reserve.

We finished our stay by a road trip to the Western part of the Scottish Highlands: the Isle of Skye. It takes about 5 to 6 hours to reach the Isle of Skye by car from Edinburgh. This part of the country is typical of what people know usually about Scotland: herds of sheep, cliffs, fog and also castles, as shown in Fig. 3.



Fig. 3: Eilean Donan Castle publicly known from the Highlander movie.

On our way back, we passed by the Loch Ness, but unfortunately, we did not see any prehistoric monster. Then, we stopped in Fort William to climb the Ben Nevis, the highest point of UK, that reaches 1345 m. This was only for the sporting challenge because due to the fog and the rain we were not able to enjoy fully the experience.

In conclusion, the Scottish Highlands offers stunning views that require a bit of effort due to the elevation and a bit of patience and luck to have for the right weather.

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Press Conference of the Phos4You Project: Behind the Scenes

David Leleu & Zaheer Shariff

One goal of the Phos4You project is to promote the technologies that can be used to recover phosphorous (P) from sludges. For that purpose, the PULSE process pilot plant was designed so that the pilot plant can be moved easily from site to site to demonstrate the feasibility and functioning of such process to the different stakeholders such as the wastewater management and P-fertilizer producers.

During our trials in Oupeye near Liège, a press conference was organized with the local and national media. Different stakeholders were also invited to the event such as representatives from P industry, water boards and the Walloon Region. The event began with a presentation in the auditorium of the wastewater treatment plant, see Fig. 1. Angélique Léonard, co-manager of the project at ULiège, presented Phos4You and the PULSE pilot particularly. Angélique is also responsible of the LCA analysis of the various P recovery technologies demonstrated in the project.

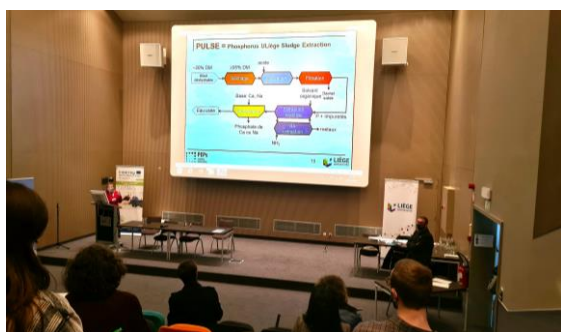


Fig. 1: Presentation of the Phos4You project to journalists and stakeholders

Then, the visit of the plant followed, as shown in Fig. 2. The different process steps were explained to the participants. During the visit, the leaching of P from sludge was running. The intermediate products obtained from other unit operations were also shown to the participants. The press conference lasted for half a day and a total of 15 journalists were present. The pulse process was the subject of a television

report on 3 different channels and appeared 9 times in the written press.

Prior to the press conference, a photo session and a filming of the process were coordinated with the photographer and the media-team of the University. The press release about the event along with a short film is available on the ULiège website (University of Liège 2021).



Fig. 2: Visit of the pilot

The press conference was a very successful event, as different companies expressed their interest in testing or developing the PULSE process at the industrial level.

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Acknowledgements

The press conference was organized by the press and communication service of the University of Liège. The AIDE provided also the auditorium for the press conference.

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Food Waste

Andreas Pfennig

Background

In recent years, the media have repeatedly reported that a large fraction of food is wasted. A study commissioned by the UN, for example, finds that about one third of all food is lost globally (Gustavsson, 2011). This discussion was also one of the triggers for food-sharing initiatives and food banks. Also, in talks on sustainability, where I promote vegan nutrition, I am often told that we should first avoid these losses before demanding such fundamental changes in behavior. The high values for food losses are also frequently repeated by various organizations and initiatives on their websites as a motivation (e.g. WWF, 2021).

In the context of the 'Visions of the Future' (Zukunftsbilder), developed by the Scientists for Future, I took a closer look at the underlying data. The Visions of the Future are about developing a feasible, scientifically sound, and complete picture of how humanity may live in 2040, covering a wide variety of areas of life (S4F, 2021). To this end, descriptive texts are first developed, which will then be brought to life on a website with the support of journalists, artists and web developers. Of the four Visions of the Future that are currently being developed, I am coordinating the development of the 'Focused' vision with the support of a small team. The trigger for the closer examination of the topic of food waste was that we wanted to present the visions of the future at the Zero Waste Berlin Festival with a workshop and that changes in nutritional habits are key in the 'Fokused' vision.

When starting to look into the numbers, I was struck by the fact that one of the studies on food waste cited indicated that 25% of cereals are wasted at the household level in Europe (FAO, 2011). This figure seemed significantly too high to me from personal experience. Others I asked also reported that their losses of cereal products are certainly not that high. It also seems implausible that a quarter of bread, cake, pasta, and breakfast cereals are thrown away in households. It is true that a loaf of bread may go bad once in a while, or children may not finish their pasta, but that this should account for a quarter in total seemed strange to me.

Causes for High Values

The quite high values for food losses found in partly older studies, which are often quoted on the net and in the media, are achieved with some 'tricks' in data collection and numerical evaluation. Some of these are:

- Losses are not distinguished between avoidable and unavoidable fractions. Unavoidable losses are, for example, coffee grounds, leached tea leaves, outer leaves of vegetables, lettuce trimmings, egg shells, bones, and similar portions of food that end up in the trash during normal consumption. Since in evaluating the waste all the proportions are considered that can be attributed to food as a whole, the unavoidable proportion is thus included. According to recent studies, however, the avoidable portion, the actual food waste, accounts for only about half of all losses.
- The losses are not taken in relation to the initial quantity, but to the quantity of food consumed. If one-third of an original 100 kg of food is lost, that's not 33 kg, but only 25 kg - one-third of the remaining 75 kg.
- The losses are only roughly estimated from the percentages of the garbage disposal. In one study, for example, the losses for individual food categories are chosen as 15% or 30%. It is then logical that the study finds something in between, averaged for all foods, namely 26 % losses. The inaccuracy of at least 7.5 % is not indicated.
- Water is not excluded from the losses. The dry quantities of flour and pulses, for example, are assumed for the quantity produced. In the case of losses, it is the mass of prepared meals to which water has of course been added. This water content is not factored out even in more recent studies.

It should also be kept in mind that losses are usually expressed as mass, with naturally water-rich foods being more likely to spoil. The fraction of losses in mass are higher than the loss expressed as nutritional value or calories. The motivation for such high values is obvious. The larger the numbers, the more media attention they attract and also the higher the chance

that follow-up projects to investigate food waste more closely will be funded.

The Correct Values

In more recent studies, food losses have been assessed more systematically and accurately. In addition, attempts have been made to consolidate different data sources. The results still show quite significant scatter, but overall present a fairly homogeneous picture. In Fig. 1, first the values for Germany are shown. Across the entire value chain, losses amount to 8%, of which about half is unavoidable and the other about 4% is wasted. Of this share, about half, i.e. around 2%, occurs in the household sector.

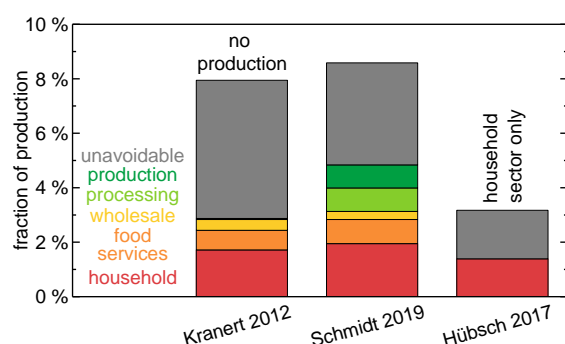


Fig. 1: Food waste and loss in Germany

Fig. 2 shows that in Europe the percentages are somewhat higher. In Belgium and the Netherlands in particular the shares in the processing sector are strikingly high. These countries have the largest ports in Europe, Antwerp and Rotterdam, where products are imported from overseas. The resulting losses are then attributed to these countries, which, however, have significantly lower production than other European countries due to their relatively small area. As a result, the shares of these losses relative to production in these countries are particularly high.

These results show that by far not one third of food is wasted, as it often appears in the media. Waste and losses account for about 12 to 14 % in the EU. Waste is expected to account for about half of this, 7%, and about 3.5% is wasted in households. Food waste is therefore far from being as dramatic as often portrayed. However, about one in ten people worldwide is undernourished. About 40% of all people experience some form of malnutrition, either in terms of quantity or nutrients.

Against this background, it is of course unethical to waste any food. Avoiding food waste may

significantly help to overcome malnutrition globally. However, it cannot contribute significantly to making it easier for us to meet the other challenges of the future, for example by using more biomass to produce materials and provide energy instead of using fossil sources. This will only be possible by avoiding waste of land area in animal production, where only 20% of calories are provided animal-based on 80% of the land area used for food production.

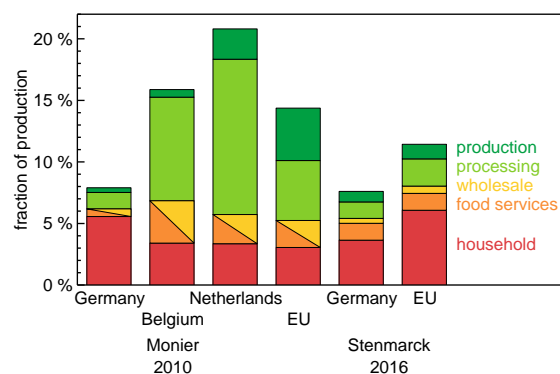


Fig. 2: Food losses and waste in different European countries and the total EU

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The Return of Face-to-Face Events at the University

Marc Philippart de Foy

Life at the University During the Pandemic

The academic year 2020/2021 has been an unusual year at university. Teachers, researchers and students have really come to understand what homeworking is all about and everyone has had to adapt their lifestyle to comply with the governmental rules curbing the COVID-19 pandemic.

New communication habits were taken such as podcasts for the courses and online meetings between colleagues. Events of all kinds were held online. Not only were working seminars and international conferences held remotely, but even other festive events were organised via video-conferencing platforms. The final presentation of the 2020 Bachelor Project is a good example: all participants received samples to taste at home while attending the party online. Such events were a way to get some distraction from the pandemic that prevented everyone from meeting in person and enjoying each other's company.

New Year, New Dynamics

Since the beginning of this academic year, new regulations have been introduced. Classes can be given entirely face-to-face and full-time homeworking is no longer compulsory. Through vaccination, life on the campus has resumed and communication among colleagues has intensified, if only because the cafeteria has reopened. The university is also playing a role in the fight against the spread of the virus by offering vaccination opportunities open to all. The SARSSURV study was also launched almost a year ago. Its objective is to evaluate the spread of the virus, the efficiency of antibodies and the level of seroprevalence in a large population (SARSSURV, 2021).

The introduction of the Covid-Safe-Ticket (CST) in Belgium has also made it possible to organize large-scale events at the university. As communicated by the rector in October 2021, events can now be organized in the university premises

on condition that each participant presents a valid CST at the entrance.

Unfortunately, the health situation has worsened since November and stricter measures have had to be taken, so the organization of some festive events is jeopardized.

Events of the Department

For the Chemical Engineering Department, the first annual barbecue since 2019 was held in September, as shown in Fig. 1. This event was the first time that most members of the department had been able to see each other in a year and a half. Some also realized that it was the first time they had met other co-workers in person, and the introduction of the list of 'newbies' was very long this year! This barbecue was a great opportunity to put a face to every name that was previously just an e-mail address or a pixelated image in an online meeting. The atmosphere was very pleasant, the sunny weather helped, and everyone seemed happy to finally share a meal with colleagues and chat without having to open and close the computer microphone every time.



Fig. 1: Return of the annual barbecue of the Chemical Engineering department

A second team-building event organized by the department was a blind-test in mid-November. This was a competition in which each team had to recognize as many songs as possible from those played. The songs were classified in different musical categories such as French or English oldies, covers, duets, country, and even

mysterious themes. The blind-test was also the opportunity to increase the bonds between different people in the department as NCE and PEPs workers were mixed in the teams, as well as foreigners and Belgian participants.

Unfortunately, the department's traditional Christmas dinner had to be cancelled this year due to the health situation. There will therefore be no meal shared by the whole department, and the fancy Christmas sweaters will remain in the cupboards for another year. But that will only increase the anticipation for the 2022 Christmas dinner!

Events of the University

Events were also organized by the University, always restricted to people with a valid CST. For the students, the 15th edition of the 'Unifestival' took place in October on the Sart-Tilman campus. Bands and DJs played music on three stages, while food and drinks were available for purchase in the vicinity. The Unifestival was the first student event organized by the university for over a year and gave first- and second-year students the opportunity to finally see the campus from a different perspective.

A second event was the 'Afterwork of the university staff'. As the name suggests, this event was open to all university workers and was attended by around one thousand people at the Colonster castle in Liège, as can be seen in Fig. 2. Food and first drinks were offered by the university and a concert of the Belgian band Suarez closed the evening. This was an opportunity to meet colleagues from other faculties and discuss the working conditions of everyone during the lockdown, for instance. The Chemical Engineering department was well represented, with about fifteen people attending the afterwork and sharing the delicious free food.

In the Future

Other events are planned in the next semester, such as the 'Souper chimie' organized by the Master students in Chemical Engineering or the theatre play 'Revue des ingénieurs' featuring students from the faculty of Applied Sciences. These events will only be organized if the pandemic situation improves compared to the end of 2021. It is therefore everyone's responsibility to be cautious and to follow the protective measures to inhibit infections. Even vaccinated people should keep in mind that they can still be

contagious and develop serious health problems due to COVID-19, although the risks are lower than for unvaccinated people.



Fig. 2: Afterwork of the university staff – photo by @Goldo

The continuation of face-to-face events depends thus on everybody's behaviour. Only time will tell whether such festive events can be organized again under safe conditions in the future, especially if the omicron variant of the COVID-19 virus should be even more infectious than the delta variant, as initial investigations suggest.

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Presenting Sustainability

Andreas Pfennig

A large part of my private time in recent years has been devoted to sustainability. For example, I am involved in various initiatives at Scientists for Future, Germany, where I am also a member of the expert panel that plans and implements strategic projects. The Future Images is one of these projects in which I am actively involved, as described in the article on Food Waste. The aim is to bring to life positive, liveable images of a successful future, instead of just pointing the finger at everything that is going wrong today.

Together with Jürgen Blümer, we have, for example, brought the Scientists for Future standard slides on climate change up to a consistent level and updated them. These slides are especially intended for education in schools. There are slides of varying complexity for each of the topics, allowing their use in age-appropriate education. These slides on climate in general and many other sustainability topics are freely available on the Scientists for Future website: <https://info-de.scientists4future.org/materialien/>

In the second half of 2021 in particular, presentations were scheduled at almost two-week intervals, mostly online, but also a few on site. Remarkable were for example two climate camps in Aachen, one in the Frankenberger Park, one on the Katschhof (see Fig. 1). It is a special experience to talk about sustainability in the shadow of Aachen Cathedral, which has already survived so many changes and developments.



Fig. 1: Talking at the Climate Camp at the Katschhof in Aachen

As in previous years, one of the challenges was to adapt the way of communicating to the respective age of the audience. At the 'Morgen-Land' workshop at 'Aber Hallo' in Alsdorf, younger children were guided by experts to think about what our world might look like in the future. Using various media, houses and landscapes were designed in a model, which is shown in Fig. 2. Everything was included, from a flying house that can escape all the rigors of the weather to a floating house that can withstand rising sea levels.



Fig. 2: Morgen-Land (Tomorrow Land) at Aber Hallo in Alsdorf
(Photo: Andreas Basler)

On the other hand, older students about to graduate from high school or adults at the Adult Education Center (VHS) were trained for more sustainability.

A special and very memorable event I would like to highlight was a virtual presentation at the Adult Education Center (VHS) in April to which I invited my former Ph.D. students. I hope I didn't forget anyone of whom I have the contact data... Even if we only saw each other partially, it was nice to once again get into contact and partly into live conversation and discussion even during Corona.

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