

# 19th NORDIC FILTRATION SYMPOSIUM

Techno-sustainability and Circular Economy

together with the

**1st Scientific seminar of the  
SCI-MAT-research platform**

» June 13-15, 2023 | Lappeenranta, Finland

- 9:00** **VENUE:** LUT University, Lappeenranta campus, Auditorium 1316  
Registration and morning coffee
- 10:00-10:20** NoFS19 Opening
- *Professor Mika Mänttari, NoFS Symposium Chair, LUT University*
  - *Morten Lykkegaard Christensen, Nordic Filtration Society, Aalborg University*
- 10:20-11:00** **KEYNOTE:** The role of circular economy in fulfilling the mineral needs of energy transition,  
*Kimmo Tiilikainen, Director General, Finnish Geological Survey GTK*

## Session: 1 Dewatering

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- CHAIR** *Dr Mohammad Golmaei, AFRY, Assoc. Prof. Teemu Kinnarinen, LUT*
- 11:00-11:20** How tailings dewatering with decanter centrifuges is outperforming filtration systems,  
*Amol Chinchankar, Alfa Laval Mining, Sweden*
- 11:20-11:40** Dewatering of lake sediment – Phosphorus and ecotoxicology,  
*Gustav Simoni, Aalborg University, Denmark*
- 11:40-12:00** Ultrasonic treatment and electrically assisted dewatering of P&P mill wastewater sludges,  
*Teemu Kinnarinen, LUT University, Finland*
- 12:00-13:00** Lunch
- 13:00-13:20** Methods for the measurement of water permeability for filter media,  
*Marina Ängeslevä, Porometer, Belgium*
- 13:20-13:40** Mechanical compression of lime mud, Tatu Kärki, LUT University, Finland
- 13:40-14:00** Acidic wastewater treatment plants in mining and metals industry,  
*Mohammad Golmaei, AFRY, Finland*
- 14:00-14:20** Future trends in material handling systems for minerals processing,  
*Timo Kupsanen, Laitex Oy, Finland*
- 14:20-14:50** Afternoon break

## Session 2: Separation of critical metals

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- CHAIR** *Docent Sami Virolainen, LUT*
- 14:50-15:10** Battery recycle filtration, case Roxia, Matti Luoma, Roxia Filtration, Finland
- 15:10-15:30** Process development of a continuous ion exchange process in battery metals recycling: From single column experiments towards the simulated moving bed configuration,  
*Tobias Wesselborg, LUT University, Finland*
- 15:30-15:50** Membrane distillation for lithium recovery from brine,  
*Aamer Ali, Aalborg University, Denmark*
- 15:50-16:10** Solvent extraction in metal recycling: Focus on phase separation,  
*Niklas Jantunen, LUT University, Finland*
- 16:15-16:45** Nordic Filtration Society board meeting
- 16:15-17:30** Lab tours (optional)
- 18:30-19:30** **VISIT:** Metso Filtration Competence Center, Address: Tukkipäät 1,  
*Bus transportation from LUT or hotels to Metso*
- 19:30-22:00** **GET TOGETHER EVENT:** Restaurant Lappeenranta Kasino,  
*Address: Ainaankatu 10, Bus transportation from Metso to Restaurant Kasino*

### Session 3: Membrane fouling

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- CHAIR** *Dr Hanna Kyllönen, VTT*
- 9:00-9:20** Inorganic scale prevention for the bipolar membrane electrodialysis, Frederick M.S. Christensen, Aarhus University, Denmark
- 9:20-9:40** Perspectives of membrane fouling monitoring by  $3\omega$  sensing, Mads Koustrup Jørgensen, Aalborg University, Denmark
- 9:40-10:00** Effect of fouling on recovery of recominant protein by membrane perfusion filtration, Malene Heilskov Veje, Aalborg University, Denmark
- 10:00-10:20** Morning break

### Session 4: Membrane prospects in protein shift

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- CHAIR** *Assoc. Prof. Morten Lykkegaard Christensen, Aalborg University*
- 10:20-11:00** **KEYNOTE:** Membrane processes in the times of the protein-shift, Frank Lipnizki, Lund University, Sweden
- 11:00-11:20** Recovery of food- and feed-grade proteins from fresh grass juice using membrane separation, Mads Koustrup Jørgensen, Aalborg University, Denmark
- 11:20-11:40** The application of ultrafiltration for the extraction of food grade proteins from grass, A. Kjær Jørgensen, Aalborg University, Denmark
- 11:40-12:40** Lunch

### Session 5: Novel applications of membranes

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- CHAIR** *Prof. Frank Lipnizki, Lund University, Assoc. Prof. Mads Koustrup Jørgensen, Aalborg University*
- 12:40-13:00** Development of nanofiltration membranes for offshore recovery of  $H_2S$  scavenger chemicals, Jens Muff, Aalborg University, Denmark
- 13:00-13:20** Cellulose membranes in treatment of spent deep eutectic solvent used to extract lignin from hardwood, Vadim Ippolitov, LUT University, Finland
- 13:20-13:40** Exploring novel universal 2D carbon-based materials for  $H_2$  gas separation from mixtures and further potential applications, Dániel Gardenö, University of Chemistry and Technology in Prague, Czechia
- 13:40-14:00**  $NO_3$  recovered from an industrial side stream, Hanna Kyllönen, VTT Technical Research Centre of Finland, Finland
- 14:00-14:20** Hydrophilic cellulose membranes in the treatment of wool scouring effluents, Tiina Rissanen, LUT University, Finland
- 14:20-14:40** Membrane modification strategies for high performance enzymatic membrane reactors, Ziran Su, Technical University of Denmark, Denmark
- 14:40-15:00** Sustainable power generation from salinity gradients via pressure retarded osmosis (PRO): Membrane modifications for improved mechanical stability, Magdalena Malankowska, Technical University of Denmark, Denmark
- 15:00-15:20** Afternoon break

### Session 6: Techno-sustainability in mining

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- CHAIR** *Industry Prof. Jutta Nuortila-Jokinen, LUT*
- 15:20-16:00** **KEYNOTE:** Sustainable solutions for future mining, Jani Kiuru, SVP Raw Materials, Finnish Mineral Group
- 16:15-18:00** **EXCURSION:** Visit to Nordkalk lime stone mine in Lappeenranta
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- 19.30-23:00** Conference Dinner & Closing of the 19th Nordic Filtration Symposium.  
*Restaurant Wanha Makasiini, Address: Satamatie 4*



Towards sustainable circularity of industrial inorganic side streams:  
collaboration across disciplines and industries

9:00 Registration and morning coffee

9:45 Opening speech, LUT SCI-MAT research platform, *Jarkko Levänen, Assistant Professor of Sustainability Science, Team Leader of SCI-MAT platform, LUT University*

**Session 1: Recovery of valuable metals from industrial side streams**

- 10:00-12:00
- Rare-earth elements (REEs) as business opportunity?  
*Erika Rova, Head of P-Competence Center, Yara Europe*
  - The recovery and purification of REEs from secondary source,  
*Santeri Kurkinen, PhD Candidate, LUT University*
  - Supply risks and development of the critical metals market,  
*Jarkko Vesa, Senior Specialist, Ministry of Economic Affairs and Employment*
  - Discussion
  - Chair, *Katja Lahikainen, Director of CST, LUT University*

12:00-12:30 Networking and refreshments

**Session 2: Treatment and valorization of industrial side streams**

- 12:30-14:00
- Introduction, *Sami Virolainen, Director of SCI-MAT platform, Head of Department of Separation Science, LUT University*
  - Panel discussion:
    - *Annica Lindfors, Business Development Director, Circular Economy, Nordkalk*
    - *Matti Lampinen, Development Manager, LUT University*
    - *Mikko Räisänen, Senior Researcher, Innovations, Betolar*
    - *Saija Luukkanen, Professor of minerals processing, Head of Oulu Mining School, University of Oulu*
    - *TBD, Metso Group*
  - Q & A
- 14:00 Lunch and networking

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The 19th NOFS is organized by LUT Centre for Separation Technology CST,  
SCI-MAT Research Platform and Xplorer network

# How tailings dewatering with decanter centrifuges is outperforming filtration systems

Ravi Kappel<sup>\*1</sup> & Amol Chinchankar<sup>2</sup>

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## Keywords:

Tailings; Dewatering; Decanter; Centrifuge

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Alfa Laval has provided decanter centrifuges for dewatering the tailings from a wide range of mineral processing plants all over the world. The applications are tailings from sulphide ores, iron ores, borate, oil sands and coal. Most of the customers has gradually extended their decanter array after starting out in a small scale and then added more machines after seeing the results.

The decanter centrifuge can be used for dewatering slurries with a wide range of solids content. It can be used for dewatering thickened tailings, but also for dewatering the flotation underflow, thus eliminating the need of thickeners.

There are several factors that influence the suitability of decanters in a processing plant: wear properties of the tailings, particle size distribution, mineral composition, expected dryness, available water resources, and environmental restrictions.

The results are compared to the expected performance of a filter press. Major savings can be achieved with the decanter centrifuge. The required space for the processing equipment is greatly reduced, with subsequent savings in building capital expenditures and operational expenses. The process equipment itself also has 15-20% lower capital expenditures and more than 30% lower operational expenses. The eliminated need of thickeners is the main reason for the savings, but the replacement of the filter presses has a very significant impact too, since the decanter centrifuges have a higher availability and less downtime needed for maintenance operations.

It has been proven that the decanter centrifuge technology provides the most cost-efficient way to dewater tailings for either dry stacking or paste backfilling. It is also a technology that is very well suited for the needs in the Nordic mining industry where cold climate demands leads to high building costs. Therefore, decanter centrifuges should be considered by everyone looking for an efficient tailings dewatering solution.

# Dewatering of lake sediment – Phosphorus and ecotoxicology

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## Keywords:

Belt filter; Biopolymer; Consolidation; Filtration; Flocculation

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Most of Danish lakes are eutrophic due to high concentration of phosphorus (P). Phosphorus are non-substitutable element required for all living organism and it is energy-intensive for mining. Hence, phosphorus should be removed from lakes and reused. High amounts of phosphorus are bound in lake sediment, which could be dredged and collected. The large water content in the dredging lake sediment causes the costly transportation and difficulties in directly utilizing the dredging sediment. Thus, sediment have be be dewatered. The current used dewatering technologies are treating the dredged sediment using synthetic polymer which can be harmful for the environment.

In this study, pilot-scale belt filter and filter bags were tested for separating water from solid sediment. The dredged sediment was flocculated using either the conventional synthetic polymer (polyacrylamide) or biopolymers (modified starch, low-MW chitosan and high-MW chitosan). The results showed that dry matter content (%DM) of dewatered sediment was increased from 3-7%DM of dredged sediment to 6-14%DM of dewatered sediment after belt filter and subsequently 12-16%DM of dewatered sediment in big bag. The reduction of P, N and Fe was 84-99%, 27-93%, and 89-94% of filtrate water compared to lake sediment. Toxicity tests with three aquatic model organisms representing bacteria, phytoplankton and zooplankton indicated low ecotoxicity of most polymer filtrates with median effective concentrations (EC50). Lake water with the synthetic polymer (polyacrylamide) showed the greatest ecotoxicity of the tested polymers whereas the biopolymers (modified starch, and cationized chitosan) displayed lower overall ecotoxicity.

The pilot test in this study was an important result obtaining good results with biopolymer (less ecotoxicity but increasing the filtration time). This expects to enhance the lake-sediment dewatering technology using biopolymers and supporting P recovery project with respect to technical feasibility and environmental criteria according to circular economy.

# Ultrasonic treatment and electrically assisted dewatering of P&P mill wastewater sludges

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**Keywords:**

Ultrasound; Dewatering; Electrofiltration; Sludge; Cake

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The objective of this experimental study is to introduce previous advances on ultrasonic pretreatment and electrically assisted filtration techniques to improve the treatment of pulp and paper (P&P) mill wastewater sludges. There is a lack of nutrients in biological wastewater treatment processes in most P&P mills. The intracellular nutrients of sludge particles might provide an undervalued source of nutrients for the process. Ultrasonication is a potential method to be used before mechanical dewatering of sludge to release the nutrient content trapped within the cell walls. On the other hand, one of the biggest problems in mechanical sludge dewatering is that the intracellular liquid cannot be removed techno-economically by mechanical compression. Therefore, technologies accelerating flow of water through cell walls are key to improving the dewatering result.

The experiments were carried out by using a tubular ultrasonication reactor, a vertical filter press, and a piston press for treatment of industrial wastewater sludges. Both sludge press units were equipped with electrodes, between which a pulsed electric field was applied. The influence of ultrasonication was evaluated by particle size measurements and analyzing the liquid phase for, e.g., TOC, TN, nitrate, and P. The solids contents of the dewatered sludge cakes were determined gravimetrically.

The results of this study show that the application of ultrasound showed promising effects on the release of nutrients. Ultrasound also made the particle size distribution of the sludge significantly finer. However, the release of water from the cells by ultrasound had a positive effect on the dewatering result: the solids content of the filter cake increased by max. 5 p.p. in tests with a vertical filter press. Electrically aided dewatering with the vertical filter press increased the cake solids content even more, in spite of some operational challenges with the equipment.

# Methods for the measurement of water permeability for filter media

Marina Ängeslevä<sup>\*1,2</sup>, co-authors (Riina Salmimies<sup>3</sup>, Graham Rideal<sup>4†</sup>, Janine Hilden<sup>5</sup>, Christian Wagner<sup>5</sup>, Andreas Wiegmann<sup>5</sup>)

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## Keywords:

Liquid permeability; Porous materials; Simulation; Woven meshes; Porous media flow

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Permeability is one of the most critical characteristics of filter media used for any kind of filtration. Currently, air permeability is one of the most commonly used parameters to characterise the performance of a filter medium in wet particle filtration and it is commonly approximated that air permeability is directly correlated with liquid permeability. Although air and water are both fluids, they obviously have different physical properties. Therefore, using air permeability to approximate the performance of a filter medium in wet particle filtration might be erroneous and misleading. This problem is further exacerbated by the lack of standards, standardised techniques or even widely accepted units for water permeability measurements. This work thus aims to demonstrate, discuss and compare methods developed for measuring liquid permeability for a particular type of filter media, namely woven meshes.

Seven woven metal wire mesh samples were studied in this work. Two experimental setups were specially designed to measure the water permeability of the wire meshes. One of the units worked under declining pressure, while another operated under constant pressure. The results obtained from both of these setups were compared with simulations of the flow through virtually constructed woven meshes analogous to the real samples.

As the results obtained in this work showed, all three techniques used to measure the water permeability of wire meshes produced comparable results when all measurements were done under turbulent flow conditions. These conditions were observed for the pore size above 30 µm. The samples whose pores were below 30 µm required laminar flow. The equipment working under constant pressure could provide such conditions. Moreover, we recommend that a standard method for measuring the water permeability of woven filter media be constructed based on constant pressure conditions.



# Mechanical compression of lime mud

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## Keywords:

Lime mud; Properties of lime mud; Compression; Density change; Solid content; Rheology

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Aim of this master's thesis was to study changes in properties of lime mud during mechanical compression. Changes in properties of lime mud have effect on kraft pulp mills white liquor plant and chemical recovery cycle. The density of lime mud had effect on transportation and solid content had effect on energy consumption of lime kiln.

The literature part of this master thesis includes a brief review of kraft pulp mill and chemical recovery cycle, introductions to rheology and compression and also chapter of properties of lime mud. Rheology and compression chapters are focused to powder form materials in addition to the general review. Chapter of properties of lime mud focuses on impurities, particle size and previous rheological studies.

In experimental part rheological properties of lime mud were measured. Viscosity- and flow curves were measured at different solid contents of lime mud. Loss factor experiments were done also for lime mud at different solid contents. Compression tests were performed with hydraulic press and screw press. Saturation, porosity, density, solid content and particle size changes were measured during compression experiments.

The rheological properties changed based on solid content of lime mud in all rheological experiments. Compression influenced particle size and density during experiments. In the screw press experiments the changes in particle size of lime mud were remarkable which effected density changes significantly with solid content.

# Acidic wastewater treatment plants in mining and metals industry

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## Keywords:

Acidic Wastewater Treatment; Mining and metals industry;  
Neutralization and Sulfate Removal, Hazardous Elements Removal; Circular Economy

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Acidic wastewater (effluent) treatment in the mining and metals industry is getting more attention due to the increasing disposal limitations in environmental regulations. Two of the main sources of acidic wastewater are smelter off-gas scrubbers and sulfuric acid plants. The acidic wastewater may contain a considerable amount of sulfuric acid, arsenic, and traces of heavy metals. Different hydrometallurgical processes are commonly applied to precipitate those impurities and separate them as filter cakes. Gypsum precipitation ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) is widely applied to neutralize sulfuric acid by using chemical reagents such as quick lime ( $\text{CaO}$ ), hydrated lime ( $\text{Ca(OH)}_2$ ), and limestone ( $\text{CaCO}_3$ ).

Usually, quick lime and hydrated lime are used to prepare milk of lime which will be dosed into the gypsum precipitation reactors. By adding milk of lime, the gypsum precipitation can be carried out in an alkaline condition not only to neutralize sulfuric acid but also to precipitate heavy metals as metal hydroxides. After precipitation stage, solids are removed as waste gypsum cake contaminated with heavy metals that could not be sold as a pure product. Also, arsenic can be precipitated as calcium arsenate ( $\text{Ca}_3(\text{AsO}_4)_2$ ) in gypsum precipitation reactors. However, ferric arsenate ( $\text{AsFeO}_4$ ) precipitation is preferred to calcium arsenate for arsenic removal because of its high arsenic removal efficiency and the higher stability of arsenic cake. Ettringite ( $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$ ) precipitation can be also used for sulfate removal which is more efficient than gypsum precipitation. The concentration of remaining impurities after precipitation and solids removal might meet some of the environmental regulations but not most of them in recent years.

So, the final treatment stages like Reverse Osmosis (RO), evaporator, and crystallizers are needed to meet the incoming tough environmental regulations. The circular economy in acidic effluent treatment plants can be achieved by producing sellable products like sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) cake.

# Future Trends in Material Handling Systems for Minerals Processing

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As mineral processing continues to advance, future trends in material handling systems are expected to drive efficiency, sustainability, and automation. There is a growing emphasis on sustainability, with a shift towards eco-friendly and energy-efficient solutions. This includes the use of renewable energy sources, such as solar or wind, to power material handling equipment, as well as the implementation of recycling and waste reduction strategies. Furthermore, the adoption of digitalization and data analytics will play a crucial role in optimizing material flow, enabling real-time monitoring, predictive maintenance, and intelligent decision-making. Also, the integration of virtual and augmented reality (VR/AR) technologies may enhance training programs, allowing operators to simulate material handling scenarios and improve safety. Overall, future material handling systems in minerals processing will continue to embrace technological advancements, automation, and data-driven optimization to enhance productivity, sustainability, and safety.

Through collaboration with industry partners, continuous research and development, and a commitment to innovation, Laitex is at the forefront of addressing the future trends in material handling systems for minerals processing. By embracing automation, sustainability, digitalization, and advanced technologies, Laitex aims to provide cutting-edge solutions that enhance productivity, reduce environmental impact, and optimize overall operations in the minerals processing industry.

## Battery recycle filtration, case Roxia

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# Process development of a continuous ion exchange process in battery metals recycling: From single column experiments towards the simulated moving bed configuration

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## Keywords:

Battery metals; Separation; Ion exchange; Process development; SMB process

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Lithium ion batteries (LIBs) provide advantages like high energy density and find application in various fields e.g. as power source in mobile devices or electric vehicles (EVs). The demand for LIBs is growing and thus, the demand for LIB metals is increasing, too. Spent LIBs create a raw material potential and interest in recycling grows. Researchers reviewed LIB recycling and new processes are suggested and developed.

In recovery of LIB metals, ion exchange (IX) plays a minor role. Our research group developed a process to produce Li/Co/Ni containing battery grade raffinate from synthetic lithium-ion battery waste leachate (LIBWL). Chelating resin Lewatit TP 260 with aminomethyl phosphonic acid (AMPA) functional group was used. The AMPA group shows low affinity for the target metals (Li, Co and Ni) but desirable high affinity towards the impurity metals (Cu, Mn, Fe and Al).

Aiming towards a continuous operating multicolumn process (likewise simulated moving bed (SMB)), separation experiments in single IX laboratory scale columns were conducted to develop the continuous IX process. In the single column experiments, the focus was on the second desorption step to desorb Fe and Al with oxalate solution from the resin. Additionally, the flow rates were adjusted to achieve acceptable switch times in an SMB configuration. The larger particle size of "standard" Lewatit TP 260 (effective diameter:  $0.55 \pm 0.05$  mm) limits its application at higher flowrates due to mass transfer resistances. The IX resin was replaced by Lewatit MDS TP 260 exhibiting a smaller particle diameter ( $d_{50}$ :  $0.4 \pm 0.04$  mm) and a narrower particle size distribution resulting in good separation performance even at higher flowrates. The LIBWL feed concentration was also varied to mimic the dilution of fresh feed in an SMB configuration. The obtained results were used to develop an IX column operation strategy and to suggest an initial SMB design. The results of the initial design of the continuous multi column set up show promising results to continuously produce high purity Li/Co/Ni containing raffinate (> 99.5 %). Nevertheless, further experiments and process development are needed to improve the initial design and solve remaining challenges like the Co loss to the extract.



# Membrane distillation for lithium recovery from brine

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## Keywords:

Membrane distillation; Lithium recovery; Brines; Pretreatment

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Lithium is a crucial element used in the rechargeable batteries and, therefore, its sustainable supply is vital to move forward on the road to electrification. Conventional technologies for lithium recovery, however, are developed for its viable production from only certain resources located in a few regions across the globe. Thus, its supply remains vulnerable to the global geopolitics. The current study presents the application of membrane distillation for lithium recovery from different brine compositions. Membrane distillation is a thermally driven membrane process which can concentrate the liquid solutions to their saturation level by using waste-grade heat from natural (e.g., geothermal and sun) and industrial resources. The work carried out using single-salts lithium chloride solution indicates that shape of the recovered crystals could be tuned through the operative temperature and flow rates. For synthetic lithium brines, it was observed that the operation of membrane distillation was hampered due to the formation of calcium scaling on the membrane surface. Application of nanofiltration as pretreatment to remove divalent ions was effective in increasing the concentration of lithium from 100 to 1200 ppm in artificial brine. It was also demonstrated that the application of membrane distillation has the potential to significantly reduce the footprint, processing time, chemical consumption, and the weather vulnerability of conventional solar evaporation-based process. Thus, the application of membrane distillation adds sustainability to the lithium extraction process and opens the door to extract lithium from brines with even very small lithium concentration.

## Solvent extraction in metal recycling: Focus on phase separation

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**Keywords:**

Phase disengagement; Settling; Entrainment; Crud; Solvent recovery

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Phase disengagement is of fundamental importance in solvent extraction. Common problems with phase separation include too slow settling rates, entrainment of organic droplets in the aqueous phase (and vice versa), formation of a third liquid phase, emulsification, and crud formation. Entrainment negatively affects the purity of the target metals and results in losses of valuable metals and the solvent. Moreover, entrainment often has detrimental effects on the downstream processing, and it can have negative impacts on safety of a solvent extraction process. Third phase formation and emulsification may render the liquid–liquid system inoperable in a given liquid–liquid contactor.

Phase separation problems can sometimes be avoided by controlling parameters of the solvent extraction process. The rate and extent of phase settling depends on temperature, mixing intensity, residence time, phase ratio, and type of mixing (phase continuity, hydrodynamics). Coalescence rate is eventually determined by physical properties of the liquid–liquid system, i.e., the chemical composition. Therefore, controlling the feed solution composition is highly relevant because any surface active materials (e.g., fine solids, soluble impurities, or additives) present in the feed may significantly slow down phase disengagement and promote crud formation.

Ritcey [1] has defined crud as “the material resulting from the agitation of an organic phase, an aqueous phase, and fine solid particles, that form a stable mixture.” Crud is typically deposited at the phase interface, and it captures part of the solvent phase (solvent loss). Additionally, it may reduce stripping efficiency and regeneration of the solvent. Crud formation can be initiated by various causes and crud characteristics are unique at each plant [1]. Three-phase centrifugation [2], skimming, filtration, and acid/alkaline treatment are options for processing cruds. Careful pre-treatment of the feed solution (e.g., settling, filtration, adsorption) can inhibit crud formation, whereas coalescence filters are useful for entrainment removal from raffinates [1].

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[1] Ritcey, G. M., Crud in solvent extraction processing – a review of causes and treatment, *Hydrometallurgy* 5 (1980), 97–101.

[2] Gentis, E., Three-phase centrifuge technology tackles copper crud, *Engineering & Mining Journal: E & MJ* (2022), January, 50–51.

# Inorganic scale prevention for the Bipolar Membrane ElectroDialysis

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## Keywords:

Bipolar Membrane ElectroDialysis (BMED); PtX; Water Dissociation (WD); Anti-scale; Inorganic scale prevention

As Power-to-X (PtX) draws increasing amount of attention for direct utilization of green energy (e.g., energy from windmills), multipurpose technologies such as Bipolar Membrane ElectroDialysis (BMED) (Fig.1) have regained popularity. Bipolar Membrane (BPM) consists of both anion- and cation-exchange membrane, and when current is applied in a backward direction (ion-migration away from the BPM), water dissociation (WD) into protons ( $H^+$ ) and hydroxyl ( $OH^-$ ) will occur;  $H_2O \rightarrow H^+ + OH^-$ . This simultaneous production combined with the ElectroDialysis (ED) properties (i.e., separation by ion-migration across an ion-exchange membrane), gives the BMED multiple purposes in either resource recovery (e.g., phosphor and heavy metal recovery from leachate), green chemical production (e.g., hydrochloric acid (HCl) and sodium hydroxide (NaOH) from brine solution), or carbon dioxide ( $CO_2$ ) recovery.

For any of these cases, the BMED still suffer heavily to inorganic scale formation (ISF) due to the elevated pH from the WD reaction. Therefore, we have studied different solutions for mitigating the ISF when continuously producing green chemicals directly from synthetic seawater (SSW). The solutions performance was compared by; (I) the pressure build-up within the system; (II) comparing the Faradaic efficiency calculated from applied current, flow rate and molar concentration; and (III) comparing base-line pressure after flushing the membrane. It was found that comparing different kinds of anti-scalants (e.g., chelating agents and polymer solutions), traditional chelating agents such as Ethylenediaminetetraacetic acid (EDTA) outperformed any of the other anti-scalant tested. The EDTA solution (40-80 mg L<sup>-1</sup>) showed promising results in prolonging the pressure build-up within the system with higher rates of recovery after flushing the membrane. Though, when increasing the produced molar concentration from 0.01 – 0.1 mol L<sup>-1</sup>, the EDTA solution could not prevent a full system clogging after

30 minutes of experiment time. It was found that the polymer solutions were to increase the Faradaic efficiency, owing the increased conductivity. As no ISF could be found in the acid compartment, and with a negligible amount in the feed compartment, easily removed by re-injecting of the produced acid, a solution would be to remove ions from the base compartment. Demineralized (DI) water were tested as feed in the base compartment to prevent ISF, resulting in no pressure build-up within the base compartment, even though SSW were used as feed water for the other compartment. Therefore, by removing ions which precipitate at elevated pH (e.g., magnesium (Mg) and calcium (Ca)), the ISF can be significantly hindered in the BMED.

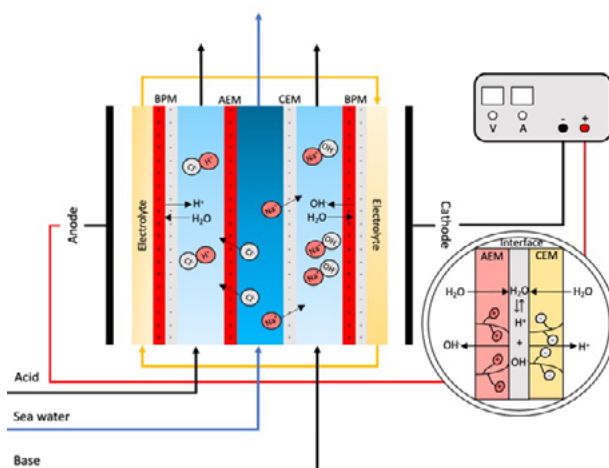


Fig.1 showing a schematic of the BMED

## Perspectives of membrane fouling monitoring by $3\omega$ sensing

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**Keywords:** Crossflow; Cake formation; Monitoring

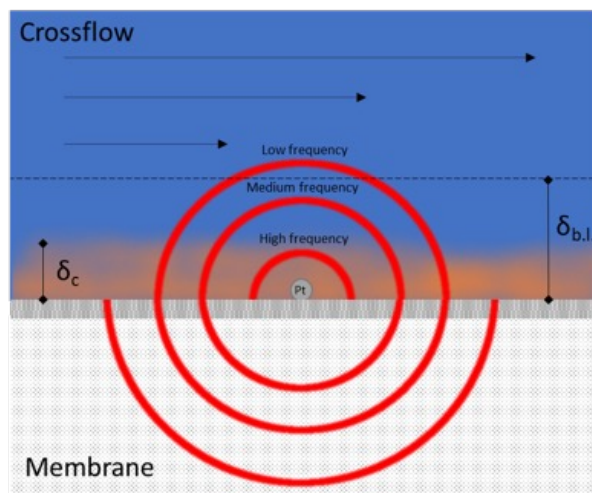
Fouling remains a challenge for efficient membrane operation. The optimization of performance is limited by the low resolution and indirect measure of fouling through flux and pressure measurements, calling for direct, local in situ fouling measurement techniques. The  $3\omega$  method was initially developed for measurement of thermal conductivity but can, nonetheless, also be used to measure deposit thicknesses and has potential for membrane fouling monitoring. In this project, 20  $\mu\text{m}$  thick platinum wires are integrated into membranes and an alternating current (AC) is applied to heat the wire. The measured oscillating voltage contains the fundamental and a third harmonic component. The amplitude of the third harmonic wave, i.e. the  $U_{3\omega}$  signal, is inversely proportional to the heat conductance, which decreases as the fouling layer builds up.

Crossflow filtration tests demonstrate that accurate measurement of the  $U_{3\omega}$  signal can reveal changes in crossflow velocity along the membrane, as an increasing crossflow velocity enhances heat convection from the Joule heated platinum wire. This effect increases with lower AC frequency, caused by a larger and frequency penetration depth of the thermal wave out from the stagnant boundary layer, as illustrated in Fig. 1.

$U_{3\omega}$  signals measured on membranes coated with layers of acrylic varnish to form an artificial fouling layer show increasing amplitude with increasing layer thickness due to the lower thermal conductivity, i.e. insulating effect, of acrylic compared to surrounding water. By measuring  $U_{3\omega}$  signals at high frequencies, the signal will reflect the thermal conductivity of the material, while by reducing the AC frequency, i.e. increasing penetration depths, the signal will reflect also the layer thickness. This can

serve to distinguish between type and amount of fouling. The same tendency is observed by in-situ measurements of  $U_{3\omega}$  signal during fouling of an ultrafiltration membrane by filtration of a dilute milk suspension. Here, the signal amplitude increases as the membrane is fouled, but is reduced again after membrane cleaning.

Hence, measurements of  $U_{3\omega}$  signals can serve as a valuable tool to monitor membrane fouling and cleaning locally during membrane filtration. In the ongoing project collaboration, it will be investigated how the method can be used to monitor different types of fouling, while investigating how the method can feasibly be implemented in industrial membrane modules.



**Fig. 1. Conceptual illustration of the effect of measuring  $U_{3\omega}$  signal at varying frequency within the fouling layer and stagnant boundary layer.**

# Effect of fouling on recovery of recombinant protein by membrane perfusion filtration

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## Keywords:

Membrane separation; ATF; Perfusion; Critical flux; CHO

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Most of therapeutic proteins are currently produced using recombinant hosts (mainly mammalian cell lines) that are cultivated in large scale bioreactors. Either at the end of the cultivation (fed-batch cultures) or continuously throughout the process (perfusion cultures), the cells need to be separated from the cultivation broth, where the product has been secreted. For this process different unit operations can be used, such as centrifugation or membrane filtration. Filtration has some great advantages, as it is possible to make this process continuously. However, one of the largest challenges of filtration, is that fouling can be difficult to control, which can make the process less effective.

In this study the focus was to investigate fouling phenomena during a cultivation process with Chinese Hamster Ovary (CHO) cells, producing a recombinant protein used in the treatment of Hemophilia. The filtration used was a microfiltration operated in Alternating Tangential Flow filtration mode. The process ran as a perfusion process, which besides minimizing buildup of toxic compounds in the bioreactor, also allowed the continuous processing of the product. In this study the short-term fouling was investigated using the improved critical flux stepping method. This method was applied both on clean media prior to inoculation, and was repeated later in the cultivation where the maximum cell density was reached. Both of these measurements show a negligible increase in Trans Membrane Pressure (TMP) when varying the flux between 0.5-67 LMH.

The long term effect of fouling was followed by monitoring the TMP, until the fouling of the membrane did not allow to maintain the set point for harvest established during the upstream process.

From the study of the long term fouling effect it was found that the fouling rate increased with increasing cell density, as a jump in TMP was observed. It was further observed that the transmission of the product remains similar throughout the run, even though the TMP has increased from 0.025 bar to 0.95 bar.



# Membrane processes in the times of the protein-shift

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## Keywords:

Pressure-driven membrane processes; Animal-based proteins;  
Plant-based proteins; Membrane fouling

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The utilization of membrane technology in the food industry is directly tied to its success in protein recovery processes. Membrane technology was first introduced in the food industry for the extraction of proteins from whey, a by-product of cheese production that was difficult to dispose of due to its low solids content and high biological oxygen demand. By using ultrafiltration, whey proteins were concentrated and desalted, leading to the production of whey protein concentrates and isolates. This breakthrough paved the way for the adoption of membrane processes in the concentration of other animal protein-rich products such as animal blood plasma, fish proteins, and albumen.

As the need for a more sustainable food system has become more apparent, a shift towards plant proteins has emerged – the so-called protein shift. Membrane technology can play a significant role in this trend by optimizing the use of vegetable raw materials as sources for vegetable proteins. Industrial-scale recovery of wheat and sunflower proteins has already been achieved through membrane processes, while the recovery of rapeseed proteins from the rapeseed oil production is still in the developmental stage.

Since rapeseed is one of the key agricultural products in the South Sweden, our research in recent years focused on the integration and optimization of membrane processes for the recovery of rapeseed protein from the press cake of the rapeseed oil production. Microfiltration has been tested for the removal of finer particles, fat, and microbes from the protein extract, while ultrafiltration was used for the final concentration and purification of the proteins. Particular focus was on the fouling and cleaning of the microfiltration membranes which was studied in depth with X-ray microtomography.

Overall, this presentation highlights the potential of membrane processes for protein production from both animal and plant sources, with examples from industry and ongoing developments.

## Acknowledgements

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# Recovery of food- and feed-grade proteins from fresh grass juice using membrane separation

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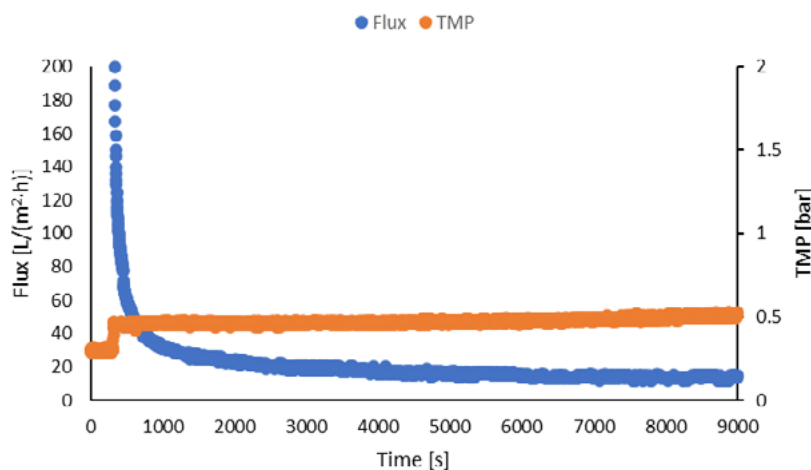
## Keywords:

Grass; Protein; Biorefining; Membrane separation

The efficient utilization of renewable resources is essential in the development of a more sustainable society. Green plant material is expected to play a central role in this development and this work focuses on the recovery of protein from fresh grass extracts utilizing micro- and ultrafiltration separation techniques. The aim is to recover both food- and feed-grade protein and improve techno-economic viability of grass biorefining. Development of grass based biorefineries has the potential to considerably lessen environmental impact and resource demand during protein production. In this study a small pilot demonstrator was constructed to allow for batches of 10 to 25 kg of fresh grass to be processed. For each individual batch, fresh grass juice was extracted using a screw press and after various treatments separated in a two-stage, crossflow membrane filtration process followed by a diafiltration step. The first membrane stage aims to retain e.g., chlorophyll and green protein which are not desirable for food applications, while the second stage allows for removal of low molecular weight compounds as well as for the concentration of the food product stream. Retentates from both membrane steps were collected, freeze dried, and characterized.

It was found that a retentate which had a green colour and grassy sensory attributes could be collected from the first crossflow filtration step while the retentate from the downstream ultrafiltration lacked any grassy smell or taste. The protein powder that was produced from the second retentate (after diafiltration) had a high protein content (50-70% based on nitrogen analysis) with high aqueous solubility, also after drying. This makes the second retentate very interesting for potential food applications, while the retentate from the first crossflow filtration stage would be of interest for feed production as this

also had a significant protein content. During the process, significant fouling was observed especially during the first crossflow filtration step (Fig. 1), and this was identified as one of the main challenges in further process development.



**Fig. 1.** Flux and transmembrane pressure (TMP) during crossflow microfiltration of grass juice.

# The application of ultrafiltration for the extraction of food grade proteins from grass

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**Keywords:** Grass; Ultrafiltration; Proteins; Food; Biorefining

Green biorefining is an increasingly prevalent area of interest as society aims to become more sustainable. Especially the food industry has a growing interest in finding alternate sources of plant-ingredients with relevant techno-functionalities. One such source of plant ingredients is legume grasses, which are rich in the protein RuBisCO, a protein of high nutritional quality and with good techno-functional properties. To retain desired properties of the recovered protein, crossflow membrane filtration may be used as a gentle processing method during biorefining, rather than conventional protein separation techniques, such as acid or heat precipitation, which can lead to denaturation and loss of functionality.

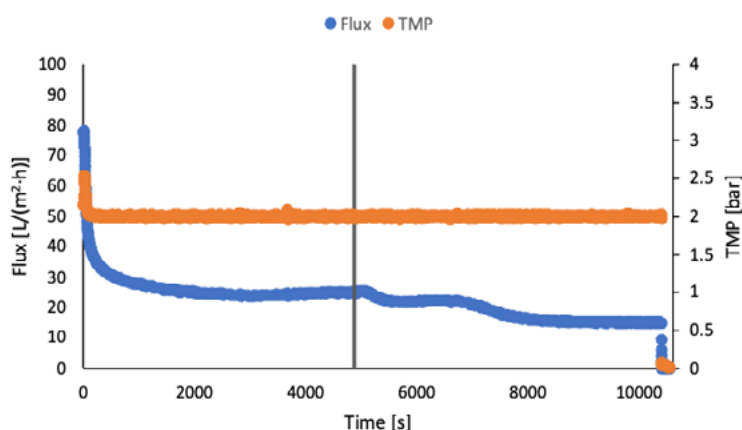
During recovery of this food-intended protein, grass extracts are fractionated in various upstream processes. Through these, larger species, such as particles, chloroplasts, and green protein, are separated from the food-applicable protein stream. This results in a protein stream free from green colour and grassy sensory attributes. This stream can subsequently be concentrated and purified in an ultrafiltration (UF) and diafiltration (DF) stage.

The aim of this study is to demonstrate and investigate the up-concentration and purification of grass protein extracts using crossflow UF and DF. This was done in a demonstrator system, where batches of 10 to 25 kg of freshly cut legume grass were treated. Following upstream processing, UF and thereafter continuous DF were both performed using a polymeric 10 kDa membrane. The retentate from this UF/DF process was found to be protein rich, containing 50-70% protein (dry matter basis), based on crude protein nitrogen analysis.

The UF process was found to be stable after an initial flux decline (Fig. 1). The DF step was less stable, with various changes in flux observed over the different experiments. During the pilot-scale experiments, a concentration factor of up to 13.75 was achieved (limited by equipment dead volume), while still operating under the relatively stable conditions, indicating that it may be possible to push the

concentration further. Additionally, indications of significant irreversible fouling were not observed by scanning electron microscopy or energy-dispersive X-ray spectroscopy.

The final retentate was freeze dried and was found to retain desired techno-functionalities, including a high water solubility. The process was therefore able to produce a protein rich powder with promising possibilities as an alternative protein source in the food industry.



**Fig. 1.** Flux and transmembrane pressure (TMP) during crossflow UF and DF of grass extracts. The black line indicates the transition from UF to DF.

# Development of nanofiltration membranes for offshore recovery of H<sub>2</sub>S scavenger chemicals

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## Keywords:

Nanofiltration; H<sub>2</sub>S scavenger; Offshore water treatment; Oil and gas production; Triazines

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Hydrogen sulfide (H<sub>2</sub>S) is a highly toxic and corrosive species, produced during offshore oil and gas operation which poses serious operational, health, safety, and environmental concerns. H<sub>2</sub>S is removed from the natural gas stream by injecting H<sub>2</sub>S scavengers (MEA-triazine) into the gas stream at offshore platforms, which converts H<sub>2</sub>S into far less hazardous and corrosive species (MEA and DTZ). In addition, MEA-triazine is generally utilized in large stoichiometric excess to increase the rate of H<sub>2</sub>S removal, which is expensive and represents > 50% of the total cost of production chemicals. The main idea of this project is the synthesis of tailor-made thin film composite (TFC) nanofiltration membranes for the separation of unspent (MEA-triazine) and spent (DTZ) H<sub>2</sub>S scavengers obtained from oil and gas wastewater from an offshore installation in the North Sea. Polyamine thin film composite (TFC) membranes are prepared by interfacial polymerization. Modifying the membrane thickness showed an effect on the separation of HET and MEA from the DTZ scavenging reaction product. The TFC membranes had a pure water permeability of [20 L m<sup>-2</sup> h<sup>-1</sup> bar<sup>-1</sup>] compared to the commercial NF270 membrane [10 L m<sup>-2</sup> h<sup>-1</sup> bar<sup>-1</sup>] attributed to their improved surface hydrophilicity and altered structural properties. The TOC of SUS wastewater was reduced by 48% and 37% for the synthesized TFC and NF270 membrane, respectively. At 50% permeate recovery, the rejections for MEA-triazine and monoethanolamine are 62% and 82%, respectively, with zero rejection for DTZ.

The results indicate that the synthesized TFC is a promising strategy for recovering MEA-triazine, thus reducing costs for offshore oil and gas operators while reducing the environmental impact associated to the discharge of this wastewater.

# Cellulose membranes in treatment of Spent Deep Eutectic Solvent used to extract lignin from hardwood

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## Keywords:

Cellulose membrane; Biomass treatment; Ultrafiltration; Organic solvent filtration; Lignin recovery

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Deep Eutectic Solvents are state-of-the-art green solvents with high efficiency, low toxicity and a wide range of applications. Great dissolving ability of DES is caused by lattice energy depression due to hydrogen bond formation between constituents. Depending on the choice of constituents and their ratio, DES can exhibit different properties which make them very tunable. DES consisting of Choline Chloride and Lactic Acid in molar ratio 1:10 is a popular solvent for lignin extraction from wood. After delignification with DES, lignin is separated from the spent DES by precipitation with the addition of water. If DES is to be reused, water is evaporated from DES. This method is commonly used in laboratory scale. It requires big volumes of water and, thus, it is not sustainable. Utilization of membrane filtration in purification of the spent DES for reuse could enable the diminishing of the required water volume. Moreover, membrane treatment could produce tailored lignin fractions for further use. In our research, cellulose membranes were chosen to be used due to insolubility of cellulose in DES (Choline Chloride : Lactic Acid 1:10) at temperatures below 60°C.

Two commercial cellulose membranes of different cut-off values – RC70PP (Alfa Laval, cut-off 10 kDa) and 5kD Disc (Millipore, cut-off 5 kDa) – were tested in series for spent DES purification and lignin fractionation. Due to high viscosity of spent DES, filtration was enabled by ethanol addition. As a result, 95% of the water insoluble compounds in the spent DES were removed, and lignin was fractionated into two fractions – above and below 10kDa. Having the concept of using cellulose membranes for spent DES purification from lignin successfully proved, we focused on the stability of the membranes in spent DES with ethanol during long-term periods. Latest results suggest that the studied membranes demonstrate stable performance of Pure Water Permeability and PEG retention up to 4 weeks of exposure to spent DES. However, the exposure leads to some changes on the membrane surface, which might influence significantly on the membrane performance in the long run. This will be discussed in the presentation.



# Exploring novel universal 2D carbon-based materials for H<sub>2</sub> gas separation from mixtures and further potential applications

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## Keywords:

Graphene oxide; Gas separation; Inorganic; Membrane; Carbon material

This work explored the effects of combining two major promising carbon-based materials, graphene oxide (GO) and single-walled carbon nanotubes (SWCNT), with various inorganic additives. Further, we deposited electrochemically a ultra-thin layer of polyaniline to improve the conductivity of the membrane surface. Fabricated GO-SWCNT composite membranes were tested for effective H<sub>2</sub> separation from permanent gases. The GO-SWCNT composites benefit from unlimited permeability-selectivity performance [1], while polymer-based commercial organic membranes usually suffer from the trade-off effect. However, the better separation performance of inorganic membranes is usually offset by their problematic scaling-up, fragility, and complicated preparation. To mitigate unwanted issues, such as fragility and lack of reproducibility, the influence of the SWCNT as support and modification of the GO skin layer by adding selected polyvalent metal ions on the overall separation properties was investigated. The modification was performed by adding selected polyvalent metal ions into the GO solution before filtration to evoke recently reported interactions of ions with the GO structure [2]. Doped GO sheets connect via intermolecular bonds, making the material more ordered and stable [3]. The gas permeability and selectivity of the membranes were tested for single and binary gas mixtures using a permeameter connected to a GC. Combining bivalent metal ions with a graphene oxide provided enormous H<sub>2</sub> permeability > 35000 Barrer with excellent H<sub>2</sub>/CO<sub>2</sub> and H<sub>2</sub>/N<sub>2</sub> selectivity (above 6.4 or 3.8, respectively), surpassing the corresponding Robeson upper bounds. A rough cost analysis was also performed to compare the material cost with the commercially available polymeric membranes. Further on, this work will also discuss various applications, such as water purification, membrane distillation, or sorption materials. The achieved results demonstrate the uniqueness of the prepared composite materials as they exceed the vast majority of published membrane materials. They further explicit the possibility of molecular engineering level tailoring of the GO layer thickness that leads to tuning the permeability.

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## NO<sub>3</sub> recovered from an industrial side stream

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Nitrate (NO<sub>3</sub><sup>-</sup>) contamination to nature mainly occurs by run-offs of fertilizers and explosives. There is a great concern about nitrate emissions, which is regulated by nitrates directive. Most of the inorganic nitrates has a great solubility in water, and high-performance and cost-effective removal and recovery technologies requires to take that into account. In this research, explosives originated nitrates were recovered from a pit water by membrane-based concept. The water contained potassium nitrate, sodium nitrate and ammonium nitrate as valuables, chloride as an impurity, and sparingly soluble salts, such as calcium sulphate, as scalants. It had, however, low concentration of inorganic aluminium, ferrous and manganese foulants or heavy metals to be concentrated by membranes.

As a result, 91% purified water with low impurity content was produced by 2-stage membrane concept. Nanofiltration (NF) using NF270 membrane was used as pre-treatment to reverse osmosis (RO) with BW30 membrane. Nitrate permeated NF270 membrane well, and even negative rejection occurred, whereas divalent cations and sulphate rejected very well. Nitrate concentrated 35 times by this membrane concept, from 110 to 3900 mg/l. High water recovery, WR 96%, in RO was obtained, partly due to osmotic pressure decrease by NF pre-treatment. However, final concentration and purification into a product needs to be carried out using other technologies.

# Hydrophilic cellulose membranes in the treatment of wool scouring effluents

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## Keywords:

Wool scouring; Water purification; Ultrafiltration, cellulose membranes; Textile industry

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Sheep wool fibers are natural, renewable and biodegradable raw material for clothes. They have recently gained increased interest among consumers due to the sustainability trend. The interest to use sheep wool in knitting has recently increased so much that there has been occasionally lack of the popular knitting yarns. The environmentally friendly image of sheep wool might at least partly originate from that we link the ball of yarn to the sheeps, which we see out on green pastures. The production of the balls of yarn from sheep wool needs, however, intensive processing. The main step of this process is called scouring, which means cleaning of the wool from impurities, such as dirt, grease, suint and vegetable matter. This process step uses huge amounts of water and produces significant amounts of wastewater, which has been said to be one of the most concentrated and polluting effluent of the textile industry [1]. Thus, in this study the focus is in the treatment of wool scouring effluents with hydrophilic ultrafiltration membranes to enable the water re-use and to decrease the environmental load caused by the scouring. The study was implemented with the Ultracel UF discs (1, 3 and 5 kDa, Millipore) and the RC70PP (10 kDa, AlfaLaval) in lab-scale. The results revealed that all the membranes could purify the scouring effluent efficiently. The best filtration capacity was measured with the loosest membrane (RC70PP), as expected. Fouling of the RC70PP was negligible. Based on the results the permeate of the RC70PP (turbidity < 1 NTU, colour < 100 APHA units and COD < 200 mg/L, measured at VR 80%) could be possible to re-use in the scouring process. As a conclusion of the experiments done by so far it can be said that the cellulose ultrafiltration membranes are a promising option for purification of wool scouring effluents.

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# Membrane modification strategies for high performance enzymatic membrane reactors

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## Keywords:

Enzymatic membrane reactor; Membrane modification;  
Electrospinning; Polyelectrolytes; Graphene oxide

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Enzymatic membrane reactors (EMR) combines enzymatic catalysis and membrane separation and allows in-situ separation of the products. The EMR can achieve high efficient downstream bioprocesses and contribute to avoid side reactions that have a negative impact on the product quality. Membranes in EMR can be used as supports for enzyme immobilization that enable reuse of enzymes and improve the overall productivity of the process. However, there are several challenges that limit their upscale and use in commercialization of bioproducts. One of them is the limited surface area of the membrane, that hampers high enzyme loading which leads to low immobilized activity. Substrates can also have problems accessing the active sites of the immobilized enzymes, which in turn decreases the activity. Moreover, enzyme denaturation during the immobilization also cause deactivation of the enzymes. In this work, we aim to address these challenges by defining novel enzyme immobilization strategies to retain high immobilized enzyme activity and meanwhile improve the selection performance of EMRs.

2D and 3D Materials that have unique chemo-physical properties were applied on the membrane to improve the enzyme immobilization. Surface modification with the 2D materials (e.g. polyelectrolytes and graphene oxide) was carried out to introduce functional groups and control the charge density on the membrane surface, thus the enzymes can attach to the membrane via non-covalent binding. High enzyme activity and good stability were observed on the 2D material-modified membranes because the non-covalent immobilization preserved the native structures of the enzymes. 3D materials such as electrospun fibers or self-assembled nanoparticles (e.g. polydopamine, tannic acid and (3-Aminopropyl) triethoxysilane) increased the surface morphology of the membranes thus allowed higher capacity of enzyme immobilization. The enzyme activity tests suggested that the porous configuration of the EMR significantly increased the enzyme activity by reducing the steric hindrance of the substrate transfer

# Sustainable power generation from salinity gradients via Pressure Retarded Osmosis (PRO): Membrane modifications for improved mechanical stability

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## Keywords:

Pressure Retarded Osmosis; Graphene oxide; Zeolites; Green energy

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Global energy supply is currently dominated by fossil fuel combustion. One of the emerging technologies that aims to minimize the overall emissions is Pressure Retarded Osmosis (PRO), which consists of harvesting the Gibbs energy resulting from mixing fresh and salt water and converting it into power. A membrane that is able to deliver high water permeability and good salt rejection, as well as to operate in the saturated salt gradient - which leads to 200 bars of pressure-, is required. Since commercial Reverse Osmosis (RO) membranes available in the market can only handle a maximum of 100 bars, membrane modification is a promising option for enhancing the resistance to pressure of such PRO membranes. Polydopamine (PDA) is one of the most popular membrane modifiers that does not only provide increased hydrophilicity but also facilitates the binding of other compounds to its surface. Zeolites, composed of microporous, crystalline aluminosilicate particles, are also promising materials for this purpose. They consist of high porosity and hydrophilic 3D structures which confer and extremely high mechanical stability. Graphene Oxide (GO) made of carbon, oxygen and hydrogen, also possesses increased hydrophilicity, excellent high surface-area-to-volume ratio and unprecedented elevated mechanical stability.

In this work, we carried out the modification of commercial thin film composite (TFC) RO membranes with different modifiers. The mechanical stability of the membranes was tested in terms of tensile strength and elongation. To our knowledge, this is the first time that zeolites are used for the PRO application.

It was observed, that after selected modifications, water permeability increased and salt rejection did not change in comparison to the pristine membrane. In addition, the membranes gained unprecedented level of mechanical stability even at low concentration of modifiers.



# Sustainable solutions for future mining

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Downstream operations and end users are increasing demands into long lists of compliance requirements. Local requirements are getting tighter and permitting is nearly impossible for the raw material operations. Meanwhile the demand for primary raw materials is heavily increasing – especially those related to carbon neutrality and e-mobility. And the regulation is adding its own flavor with various instruments such as recycled content requirement, battery passport, and carbon border adjustment mechanism. Security of supply is topical and there is potential mismatch between demand and production. This puzzle is not going to solve itself – not without new technologies and adaptation by the industry.

# Fractionation of battery metals from recycled lib waste by using membrane filtration

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## Keywords:

Nanofiltration; Discontinuous diafiltration; Lithium; Cobalt; Separation

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The development of efficient recycling processes for the lithium battery waste material is very important from point of view of sustainable economy. In this research membrane processes were applied to fractionate the metals of recycled lithium battery (LIB) waste. Selection of suitable nanofiltration membrane was made as a first step. The four different nanofiltration membranes such as Desal KH, Desal 5DL, AMS 3012 and AMS 3014 were tested with synthetic LIB leachate solution. All nanofiltration membranes studied were characterized by the lowest retention of monovalent metals such as lithium and sodium. The Desal KH membrane was selected for further trials based on results presented as flux and retention. Moreover, this membrane is very stable in the wide range of pH values. Later, the effects of operating parameter (i.e., pressure) and solution property (i.e., dilution factor) on fractionation of LIB metals were studied in the constant flow filtration mode. It was found that retention of all metals increased with an increase of pressure and an increase of dilution factor. Permeate flow, also known as flux, was strongly affected by the concentration of LIB solution. Decrease in flux with an increase of concentration was explained in terms of increase of osmotic pressure. Due to its high initial concentration cobalt is usually concomitant metal determining the purity of the lithium-containing permeate. Also, discontinuous diafiltration experiments were conducted. Diafiltration made by an addition of small portions of pure water allowed to control a flux. The results showed that the membrane fractionation of lithium from LIB waste metals could be optimized by using three parameters: purity of Li, yield of Li, and permeability of membrane. Membrane fractionation seems to be a very promising method that can be used to separate lithium from multivalent metals of LIB waste.

# Natural Phenolic Compounds as Membrane Modifiers to Decrease Biofouling Problems

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## Keywords:

Phenolic compounds; Antibacterial; Biofouling; Membrane modification

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Membrane fouling is a critical issue as it greatly comprises the efficiency of membrane-based processes and water purification and plays an important role in reducing the actual performance of membranes. To ensure cost-effective and energy-efficient operations of membrane processes and wastewater treatment, it is paramount to develop strategies to prevent the fouling formation.

Our aim is to identify novel antibacterial phenolic compounds and to develop novel modification routes to produce membranes with antibiofouling characteristics. The study combines computational modeling and screening, membrane modification and antimicrobial testing. Natural phenolic compounds from plant extracts and industrial side streams can be used to modify the polymeric membranes to disturb the formation of biofilm. To identify novel natural phenolic compounds, in silico shape and phase screenings from large commercial natural products library were performed. Based on virtual screening results several phenolic compounds were selected for experimental testing of antibacterial activity. Because quercetin has shown potential antibacterial properties, it was chosen to be used in an adsorptive modification of membrane samples. The filtration and surface properties of the modified membranes were tested by dead-end Amicon filtration and rejection measurements, and characterization of membrane samples was conducted by infrared spectroscopy and contact angle analysis. In addition, antibacterial activity of the modified membranes was tested with *E. coli* bacteria and Petrifilm count plates.

The information from the experimental results can be utilized in advanced antibiofouling strategies to prevent biofilm formation in wastewater treatment applications as well as in other applications and locations, at which biofilms tend to form and cause problems. The results of this work can support the transition of the use of membrane processes in wastewater treatment to the next level and significantly facilitate the possibility to reuse water in bigger volumes than what is done today.

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# Layer-by-Layer deposition of cellulose nanofibers on cellulosic ultrafiltration membranes

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## Keywords:

Layer-by-Layer; Nanocellulose; Coating; Surface modification

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Nanocelluloses have been gaining interest as, for instance, antibacterial coating materials and adsorptive water purification products. In terms of membrane technology, nanocelluloses have been used for improving fouling resistance, wettability and retention of specific compounds. Over time, the coating will eventually reach the end of its lifetime and lose its desired properties. At this point the coating material needs to be disposed, hence it would be ideal if the fibers could be easily detached after usage, and furthermore if they were biodegradable. Several instances of nanocellulose membrane coatings have been reported, but as far as we know, most have had poor durability, or they are non-biodegradable mixtures of polyelectrolytes and cellulose.

In this work, we present a layer-by-layer (LbL) strategy for coating a cellulosic UF membrane with cellulose nanofibers (CNFs). The CNFs are prepared using "green" deep eutectic solvent (DES) and furthermore they are modified to possess either cationic or anionic surfaces. The nanocellulose layers are deposited onto negatively charged membrane surface layer-by-layer, utilizing the ionic charges. The effect of charge on the durability of the coating is shown with agitation tests. Materials are characterized and filtration performance is assessed.

When the fibers and membrane possess opposite charges, the adhesion is stronger and the coating can withstand rather high cross-flow velocities. In this case, the substrate membrane had much tighter structure than the formed cellulose nanofiber multilayer, and thus, the neutral compound rejections did not improve. However, by coating the membrane, flux reduction during BSA filtration could be diminished, indicating anti-fouling properties. To the best of our knowledge, this is the first reported instance of coating membranes with cellulose nanofibers using the LbL methodology with no additional polyelectrolytes or cross-linkers that would hinder the coating material disposal.

# Stormwater purification with construction and demolition waste (StoPWa)

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**Keywords:** Stormwater; Filtration; Construction and demolition waste; Circular economy

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Stormwater runoff picks up nutrients, microplastics and other pollutants along its path and pushes them into rivers, lakes and seas. To reduce such pollution runoff, LUT is developing a new filtration system out of construction and demolition waste (CDW) to purify stormwater runoffs in a new three-year project funded by EU Interreg Central Baltic Programme.

Statistics Finland states that Finland produces roughly 1.6 million tons of construction and demolition waste a year. CDW is still rather moderately utilised because it contains materials that are difficult to recycle or reuse and thus recycling and utilisation rate of CDW in Finland was less than 60 per cent in 2020 even though the EU requires a recycling rate of 70 per cent. Moreover, CDW is not valorised to its full potential as e.g. crushed concrete is mainly utilised for earthworks.

In StoPWa -project different CDW fractions including difficultly recyclable brick and insulation wool, are first characterised and tested for their retention capabilities of harmful substances. The appropriate dimensions and mixtures are defined and then the most promising selected filter media are tested in field tests on semi-pilot scale in Soil Research Center Soilia (<https://www.soilia.fi/en>) located in Lahti, Finland. The best performing filter media are selected based on the field tests.

Simultaneously, different filter structures to carry the filter media are being developed. The structures can be e.g. cages, cassettes or 3D printed complicated structures. In case of 3D printing, CDW based alkali activated geopolymer composite materials are utilised.

In the last phase of StoPWa -project, the developed storm water filter systems are installed and tested in real life dammed ponds in Lahti, Estonia, and Latvia.

All through the project, the environmental impacts of the filtration system including filter media, filter material and construction, filter installation, maintenance and possible reuse/recycling are evaluated, and the results are readily embedded in the development work.

Moreover, the evaluation of the business potential of the storm water filtering system, as well as the development of the business ecosystem around it in Finland, Estonia, and Latvia, are included in the project. The aim is to create a storm water filtering concept that can easily be multiplied in Baltic countries.

The three-year development project has started in April 2023 and is a collaborative effort by the City of Lahti, LUT University, University of Helsinki, Tallinn University of Technology, the Union of Harju County Municipalities in Estonia, and Smiltene Municipality in Latvia.

Interreg



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StopWa

## Studying the effects of single and mixed algae strains on performance of algae-sludge membrane bioreactors

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To increase wastewater treatment efficiency and biofuel production, five microalgae were collected from Finnish water bodies and mixed with activated sludge and cultivated in local municipal wastewater; and tested in batch trials (phase I). *Chlorella vulgaris* and *Euglena gracilis*, well-known wastewater algae strains, were compared to these local strains for efficacy. Based on batch results, two microalgae (*Chlamydomonas* and *Selenastrum*) and their mixture were inoculated into conventional-membrane-bioreactors (CMBRs) to evaluate effects of monospecific and mixed-algae culture on the performance of algae-sludge-MBRs. The best nutrient removal, highest chlorophyll-a, and lowest membrane fouling were achieved by the mixed-algae membrane bioreactor. In comparison to activated sludge, the algae-sludge mixture had fivefold higher lipid contents during batch experiments. Additionally, using confocal microscopy, autofluorescence and staining were combined to distinguish algae from bacteria on membrane surfaces, revealing a greater role for bacteria in membrane fouling. Furthermore, sequencing analysis showed that the microbial community (e.g. *Nitrospira* and *Falvobacterium*) changed by inoculating algae which benefits CMBRs. Additionally, mixed algae-MBRs had a greater diversity of bacteria than solo algae MBRs. Consequently, the stimulation or inhibition of different species might be the reason that the mixed-algae-MBR achieves superior performance compared to CMBR and single-algae-MBRs.

## Analytical Plasma Chemistry in a Mini-pilot Scale Hydrometallurgical Laboratory

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A new hydrometallurgical laboratory has been founded in the University of Jyväskylä (JYU), Department of chemistry two years ago. The laboratory operates in a larger scale than a conventional scientific laboratory, in the so-called mini-pilot or bench scale, having reactor unit volumes of approximately 5-10 liters. The laboratory is part of the Center of Expertise for Circular Economy (CECE) infrastructure serving both academic and industrial collaboration needs and is furnished with semi-modular state-of-the art equipment. These include, but are not limited to various pumps, filter press units, lamella separators, mixer-settlers, an electrolysis cell, and a cutting mill. In its first year, research in the laboratory has revolved around a commercial joint venture involving local Finnish enterprises (ALVA, Elker, Tapojärvi) and JYU. A pre-commercial pilot plant has been planned from the ground up to meet the demands of waste electric and electronic equipment (WEEE) recycling and valuable metal recovery from printed circuit boards (PCB), a ludicrous and fast growing secondary raw material. The venture is currently in its business model and feasibility assessment step, since a proven process concept to extract pure metals (Cu, Au, Ag, Pd) from PCBs has been successfully designed and tested in the mini-pilot scale. Experimental studies have been widely changing from one process step to another, but one specific need of elemental analysis, especially metal analysis from aqueous samples, has carried through the entire project. Metal analysis involving inductively coupled plasma - optical emission spectrometry (ICP-OES) has been the workhorse of our hydrometallurgical laboratory. Despite the complex nature of PCB, elemental determinations from different process steps have been done with high throughput and sufficient accuracy, including Cu and impurity element survey in an acid leaching process followed by electrowinning, Au content determination in liquid-liquid extraction and subsequent reduction steps, and heavy metal content assessment in waste streams.