



Open Innovation Test Bed
for nano-enabled Membranes



INNOMEM OITB Continues as a Commercial Entity!

Starting from November, the INNOMEM Open Innovation Test Bed (OITB) will operate as an independent commercial entity, no longer relying on European funds. With 8 leading partners – **EMI Twente BV** (acting as coordinator), **Fundación Tecnalia Research & Innovation**, **VITO NV**, **Helmholtz-Zentrum Hereon GmbH**, **Fraunhofer Gesellschaft**, **PolyMem**, **Ciaotech Srl**, and **Innovative nanosolutions HTF SA (HTF)** – the OITB will continue to foster the deployment and scale-up of innovative nano-enabled membranes and their derived products.

EMI Twente will manage the **Single Entry Point (SEP)**, making it easy for businesses to access the OITB's wide range of expertise and services.

What does the OITB offer?

The OITB offers a comprehensive range of both technical and non-technical services. These services include **characterization of membrane properties**, **R&D development and scale-up**, and **software development**, as well as full **techno-economic market analysis** and **validation**. Interested parties can explore the updated service catalogue and submit project requests via the INNOMEM website (www.innomem.eu). Project contracts will be established directly between clients and service providers, ensuring flexibility and tailored solutions. One of the key benefits of the OITB is the ability to easily offer complementary services from multiple partners.

For more information, please contact the OITB coordinator, **Dr. Kristianne Tempelman-Bolt** (info@innomem.eu).

Let's continue innovating together!



Keep in touch with INNOMEM!

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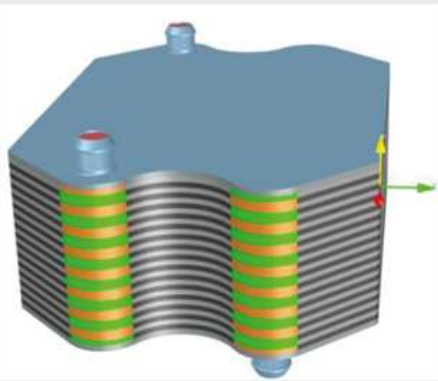
 [#innomem-project](https://www.linkedin.com/company/innomem-project)

 [@innomemP](https://twitter.com/innomemP)

MINIMAL Modelling of an Air Humidifier at the Cathode Inlet

The Fuel Cell Electric Vehicle (FCEV) uses electricity to generate enough power to drive a vehicle. On the cathode side of the fuel cell, the membrane might dry out depending on the water content of the air supplied to the system. Thus, placing a humidifier before the cathode inlet provides water vapour to the air and protects the fuel cell membrane. Different types of humidifiers (flat or hollow fibre membranes) are used in automotive applications. However, little is known about the implementation of humidifiers in vehicles.

Thus, Kayser Automotive Systems reached out the Innomem OITB, to build up a model for the humidifier to get a better understanding of the parameters like the air flow configuration, surface size area of the membrane, pressures, and temperatures.



Develop 3D model of a concept for the humidifier



Experiment set up for characterisation of the membrane (left) and tested membrane (right)

The project was successful in increasing the knowledge about important parameters like water transfer rate, membrane surface area, temperature & pressure to construct an air humidifier for the PEM fuel cell. From the gained data in the project A. Kayser Automotive Systems was able to decide which concept should be further developed. Thus, of the tested membrane the one with the best performance characteristic was chosen to be used further.

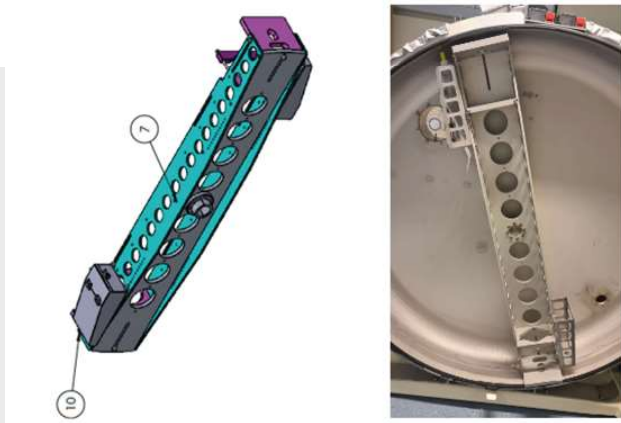
By using knowledge obtained from simulations and experiments done by the Innomem partners, University of Twente and EMI Twente, it was possible to determine the main characteristics for the humidifier and the concept to be further considered for the prototype manufacturing.

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IMCEPO Development new potting technology for gas separation hollow fiber membranes



Parker is a manufacturer of PPO-based hollow fiber membrane products which are used for air separation applications in aerospace and industrial areas. The objective of the project is to develop a new potting technology for one of our largest products to allow expanding its operating conditions. A new epoxy adhesive formulation and potting process needs to be found that allows the manufacturing of hollow fiber gas separation modules with a housing of at least 150 mm diameter. The final tube sheet needs to have sufficient mechanical strength and glass transition temperature to allow operation at 65°C and 13 barg. The fibers need to be potted using a centrifuge to prevent wicking effects which typically cause fiber breakages upon pressurization of the module.



Design new potting fixture and mounted in centrifuge.

Various suppliers were approached for suitable epoxy adhesive systems based on the defined specification, but only a few responded and could offer a suitable system. Especially the high Tg required made the selection difficult for suppliers (without having to develop something new). Despite these difficulties, the IMCEPO project did result in a selection of systems that based on their datasheet fulfil most of the requirements. The systems that were obtained were screened for their reactivity (exotherm), thermal and mechanical properties. A

method was developed to analyze the reaction kinetics using DSC. Furthermore, a silicone-based mould was developed to prepare sample bars for testing the mechanical properties of the various epoxies using an Instron tensile tester. Based on the results, the most suitable epoxy system was selected for further trials with hollow fiber membranes. A potting fixture was designed and built and mounted to the existing centrifuge to allow potting first 6" scale prototypes with the selected epoxy adhesive system. The module consisted of 3D-printed potting caps, that were glued to a straight thin-walled stainless steel tube.

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The production of membranes for sustainable and environmentally friendly applications is based on processes that use water-polluting, harmful and carcinogenic chemicals to process membrane materials that are difficult to recycle. In the project SusMem, the University of Twente and the RWTH Aachen university investigated the potential of the newly developed polymer PRIOLON as membrane material. The focus was to produce hollow fiber membranes from the new polymer and different types of green solvents.

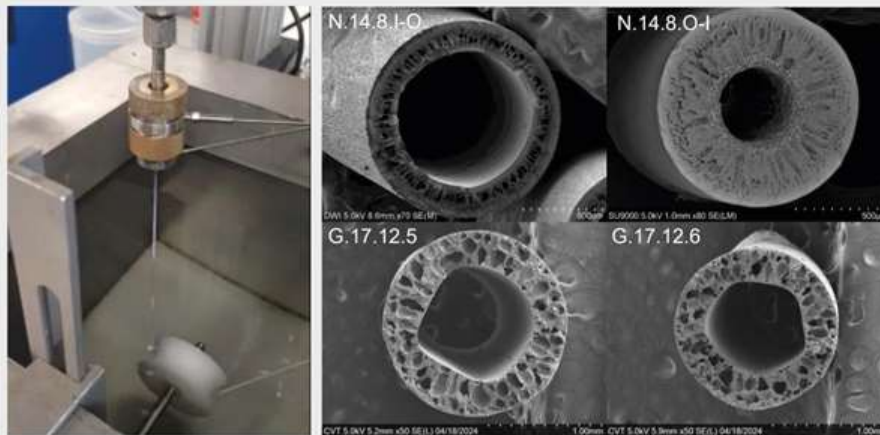


Figure 3 Left: Hollow fiber spinning. Right: Hollow fiber membranes produced with NMP (top) and GLV (bottom)

PRIOLON Hollow fiber membranes were successfully fabricated with NMP and the green solvent GVL. The results obtained demonstrate the suitability of PRIOLON as membrane material. The combination of the sustainable membrane material and a green solvent, opens the door for the production of more environmentally friendly membranes. In the remaining project time, the partners focused on testing other different types of green solvents and improving spinning conditions.

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CaBeMem Characterization and benchmarking of membranes

With growing demands to reduce water usage and recycle wastewater, the EU's "Zero Pollution Action Plan" is expected to introduce new rules on water pollution. A key goal is to cut harmful emissions and improve urban wastewater treatment. We believe membrane technology will be crucial in this effort. Through constant innovation of membrane technology, we at the Pentair X-Flow RD department strive to tackle the challenges of maintaining safe and healthy water.

In the INNOMEM project "CaBeMem", the University of Twente and Pentair X-Flow collaborated to test and compare our innovative membranes with commercially available membranes for removing various harmful substances.



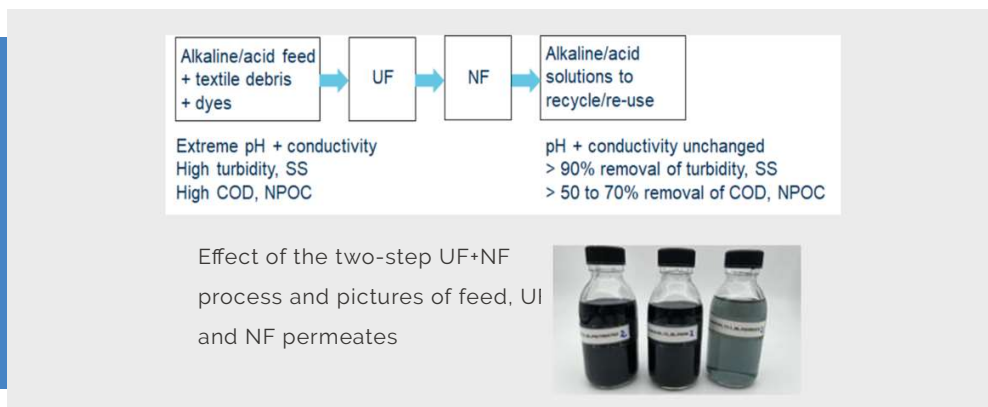
Overview of different techniques used by the Department of Membrane Science & Technology (University of Twente) for characterization of membranes

Within the "CaBeMem" project, our products were characterized, compared, and benchmarked against commercially available membranes by the Department of Membrane Science & Technology of the University of Twente. Through an initial screening, the most promising membranes for different scenarios were identified. The performance of the selected membranes will be further evaluated in a real wastewater effluent study.

We thank the European Union for giving this great opportunity for industries and universities to share their knowledge and resources in the INNOMEM framework. At Pentair X-Flow, we continue to work to achieve the zero pollution goals and improve water - life's most essential resource - for our future generations.

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Textile industry creates about 10% of the greenhouse gas emissions, and is responsible for very high-water consumption (~2700 L per cotton shirt). Moreover, a huge amount of textile waste - about 92 million tons - is produced annually. Currently, only 1% of the textile is recycled, and more than 50% is incinerated or ends up in a landfill. To incentivize a more sustainable textile industry, worldwide different legislative and technological initiatives are taken. In Europe, the EU directive 2018/851 mandates separate collection and recycling by 2025. Furthermore, a multitude of technology development projects are funded, searching for energy and resource efficient recycling processes. One of them is a Danish project partnering Textile Change (<https://textilechange.com/>) and B4C. Typically, sustainable textile recycling asks for a complex combination of mechanical and chemical processes, taking into account the circular resource optimization of all auxiliaries required. Efficient recovery of water and chemicals typically used for fibre dissolution is paramount to create a fully circular and sustainable process.



Three relevant process streams were selected: one alkaline stream, one oxidizing alkali stream, and one organic acid stream. All streams were thoroughly analysed. This showed that all streams have extreme pH and correlated high conductivity, high turbidity and correlated high suspended solids (SS) due to the textile debris, and high organic carbon (COD + NPOC non-purgeable organic carbon) originating from dyes and other impurities.

Subsequently, the 30 nm TiO₂ UF membranes, and the 0,9 nm TiO₂ NF membranes, commercially available from partner Rauscher, were chosen for first screening tests with the 3 solutions. The results showed that turbidity and SS are partly removed by the UF step with further polishing by the NF step. Organic carbon is only partly removed by the NF step. pH and conductivity remain unchanged during the whole process, allowing direct recycling of water and chemicals. Subsequent Proof of Concept testing with multichannel membranes (0.1 m² membrane area) showed the feasibility to recycle at least 80% of the purified alkaline/acid solutions, with relative low flux decline during the concentration process. For the UF process optimal process parameters were defined, and efficient processes to clean the membranes investigated.

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BIOMETH

Membrane intensified processes for Biogas upgrading to Biomethane and Methanol



In the BIOMETH service project, highly innovative membranes are used to upgrade the biogas produced by MESOPOTAMIA ENERGY SA by separating it into biomethane and CO₂. In a second stage, the CO₂ is converted via hydrogenation into readily transportable methanol. The recovered methane, which is 82 times more potent than CO₂ in terms of global warming potential, is fed into the natural gas grid.

To achieve this goal highly innovative membranes are being developed, namely: oriented graphene oxide hollow fibre membranes with high CO₂ permeance >700GPU and highly innovative water-selective Molecular Sieving (MS) dehydration membranes.

The execution of the BIOMETH democase service has focused on two major issues to assist MESOPOTAMIA ENERGY SA in selecting the most appropriate technologies to upgrade the biogas produced, namely: (a) The proposed two-stage, readily monitored and controlled polymeric membrane system for biogas separation into biomethane and CO₂ is more suitable than conventional amine technologies and should be selected by MESOPOTAMIA ENERGY SA for the production of biomethane. (b) The proposed molecular sieving membranes, with high H₂O/H₂ selectivity and high water permeance, outperform zeolitic and other molecular sieving membranes. They can be readily upscaled and are the best option to tackle the issue of disposing of pure CO₂ after biomethane removal.

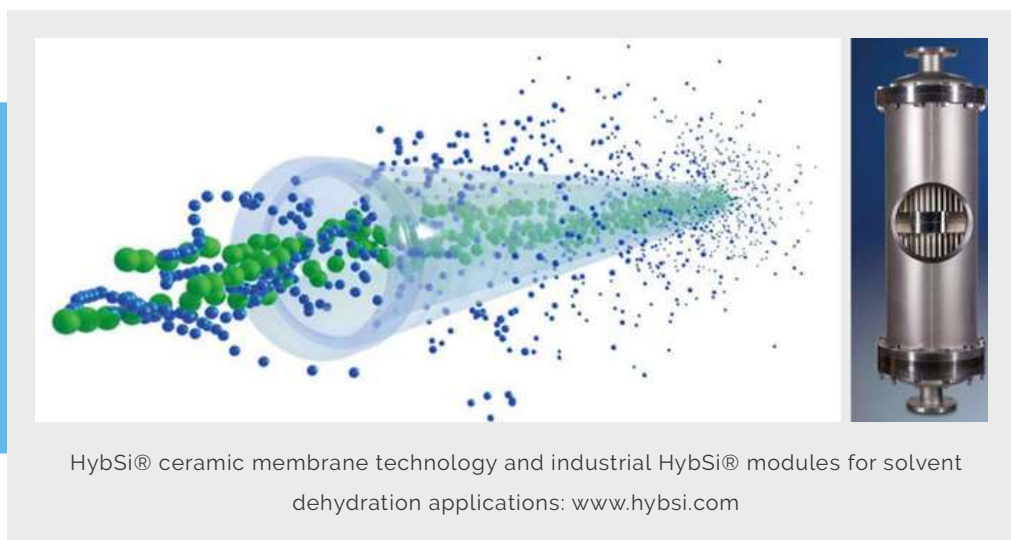
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TEA HybSi® Techno-economic evaluation of solvent dehydration by HybSi® pervaporation membranes



With growing demands to reduce water usage and recycle wastewater, the EU's "Zero Pollution Action Plan" is expected to introduce new rules on water pollution. A key goal is to cut harmful emissions and improve urban wastewater treatment. We believe membrane technology will be crucial in this effort. Through constant innovation of membrane technology, we at the Pentair X-Flow RD department strive to tackle the challenges of maintaining safe and healthy water.

In the INNOMEM project "CaBeMem", the University of Twente and Pentair X-Flow collaborated to test and compare our innovative membranes with commercially available membranes for removing various harmful substances.



TEA analysis was successfully performed for the dehydration of five industrially relevant feed mixtures, and showed that:

- To dehydrate solvents as IPA or acetonitrile, pure pervaporation or hybrid distillation/pervaporation with the HybSi® membranes of Pervatech leads to much lower CAPEX and separation costs compared to the benchmark azeotropic distillation (> 45%, KPI1)
- CO2 footprint is reduced in all cases (up to 86%, KPI2)
- The purified solvent yield and thus solvent recovery for all pervaporation process is high (> 90%, KPI3) • The pervaporation process with HybSi® is very selective for water in all cases: water content > 99% (KPI4)
- For dehydration of high boiling solvents as NMP and acetic acid, pervaporation is not the most economical option, but offers the possibility to work at lower temperatures and for lower feed flows.

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BioMime Biorefinery of Microalgae by Membrane technology for the production of bioactive ingredients



There is an increasing demand for drugs produced with active ingredients of natural origin, known as phytochemicals compounds (PC). The EU faces a shortage of active ingredients due to the COVID-19 pandemic and the geopolitical situation. These factors are pushing European companies to diversify imports and/or bring production back to Europe. ISGREEN is a small enterprise active in developing bioderived products based on Spirulina microalgae cultivated in photobioreactors under controlled conditions (absence of contamination). Microalgae Spirulina is a source of active ingredients, among them the most important in terms of quantity is phycocyanin. The Global Spirulina Market was valued at \$393,6 million in 2019 and is projected to reach over \$900 million by 2027. Extraction of phycocyanin, purification, and preservation of its bioactivity is still a challenge. Within BioMiMe, CNR-ITM in close cooperation with ISGREEN, advanced knowledge on how to overcome current challenges in the extraction of phycocyanin, purification, and preservation of its bioactivity by using membrane technology.

Through membrane processes, pure phycocyanin was extracted from Spirulina Algae:

- Purity: $A_{615}/A_{280} = 1.4$
- Suitable for food grade
- High-protein conc. solution



- Brilliant blue appearance



- Fluorescence under light

The following results were obtained:

- Identification of the pre-treatment to clarify the cell lysate suspension
- Identification of membrane material, pore size, and process conditions for MF and UF to purify and concentrate phycocyanin and maintain its bioactivity
- Achievement of 5 fractions: 1) Spirulina cells harvested and purified

from the broth (the broth can be reused in the photobioreactor) 2) MF retentate with cell fragments, 50 (\pm 5) % of phycocyanin (strategy 1) and 25 (\pm 5) % of phycocyanin (strategy 2) 3) MF permeate with phycocyanin and chlorophylls A and B 4) UF retentate with food-grade phycocyanin 5) UF permeate with purified water (that can be reused in the process/ photobioreactor). The Key performance indicators were achieved:

- A pressure-normalized flux higher than 50 L m⁻² h⁻¹ bar⁻¹ using permeate of MF as feed of UF membranes
- A rejection of phycocyanin through UF membranes higher than 90
- A fouling index for MF membranes of 0.1. Overall, the results and KPI are very promising and well beyond the state-of-the-art.

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Ucaneo Biotech GmbH develops pioneering biomimetic direct air capture technology, featuring an electrochemical direct air capture (e-dac) system powered by renewable energy. Their cutting-edge approach combines bio/catalytic solvents with electrochemistry and proprietary components like solvent and cell stacks. The fully electrical, modular technology operates without (waste) heat and can be paired with renewable energy.

While electrodialysis for carbon removal is novel with limited industrial-scale knowledge, Ucaneo's benchtop tests show promising energy efficiency. An external study predicts energy use could drop to 1000 kWh/t at scale. This project aims to reduce scale-up risks and optimize processes through empirical data and simulations.

This project provides qualitative data to minimise scale up risks and provides insights into process optimization possibilities at large scale regarding membrane compatibility and process performance both empirically validated and through simulation.

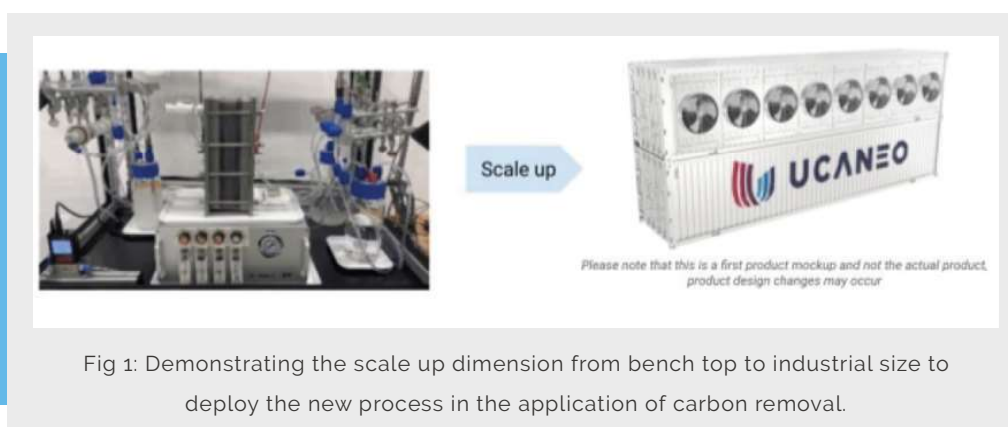


Fig 1: Demonstrating the scale up dimension from bench top to industrial size to deploy the new process in the application of carbon removal.

A suitable membrane for scale up has been identified. The standardised testing protocol was developed and showed significant performance difference between different bipolar membrane suppliers.

The simulation showed no limitation in conductivity depletion within the process when compared to empiric process performance at small scale.

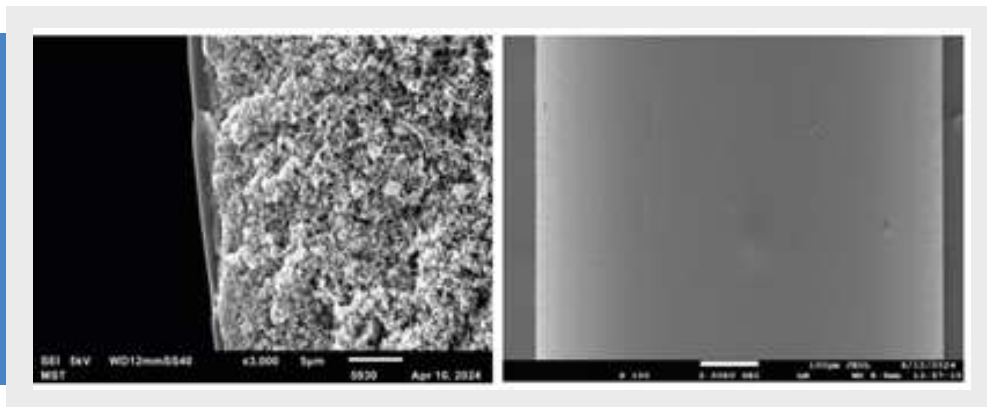
CO₂ bubble formation within the solvent does not pose a risk for scaling up, however current density limitation showed larger impact than expected. Further investigation is required to follow up on the unexpected current density limitation which is likely due to the process itself, but was out of scope of this project.

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ULTRAMEM

Translating Dip-Coating into a Continuous Technique for Thin Film Composite Production

Thin Film Composite (TFC) hollow fiber membranes are essential for applications such as gas separation, water treatment, and wastewater purification. The increasing industrial demand for longer fibers necessitates a shift from laboratory-scale dip-coating methods to continuous coating techniques. The continuous coating process is crucial for achieving consistent and scalable application for both gutter and selective layers. Transition from dip-coating to continuous coating can be achieved through the fine-tuning of critical parameters such as withdrawal speed, solution viscosity, and coating time, ensuring optimal coating uniformity and performance maintaining an optimal balance between permeance and selectivity to meet industrial requirements.



Transition from dip coating to continuous coating has a success rate of approximately 90% in terms of the gas permeance performance.

- A defect-free layer is achieved, resulting in a perfectly uniform coating that varies from 200 nm to 4 µm depending on the required application and coating conditions

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NANOGOM Study for the optimum production of Magnesium-Calcium Enriched



Drinking Water and Lithium recovery from spent batteries

In the NANOGOM service project, it is proposed to use a layer-by-layer developed nanofiltration (NF) membrane to address mineral depletion in desalination plant product water, which often results in water product that lacks essential salts and minerals that are crucial for consumption. To this end, a hybrid nanofiltration (NF)-reverse osmosis (RO) desalination system is proposed to increase the magnesium content of the desalinated water. Through a combination of pilot-scale NF experimentation and RO simulations, the NANOGOM service demonstrated the practicality and economic feasibility of this innovative treatment approach. Furthermore, in the NANOGOM service project, it is proposed to improve nanofiltration membranes using the layer-by-layer technique for the development of systems that are suitable for the cost-effective recovery of lithium from spent lithium batteries.

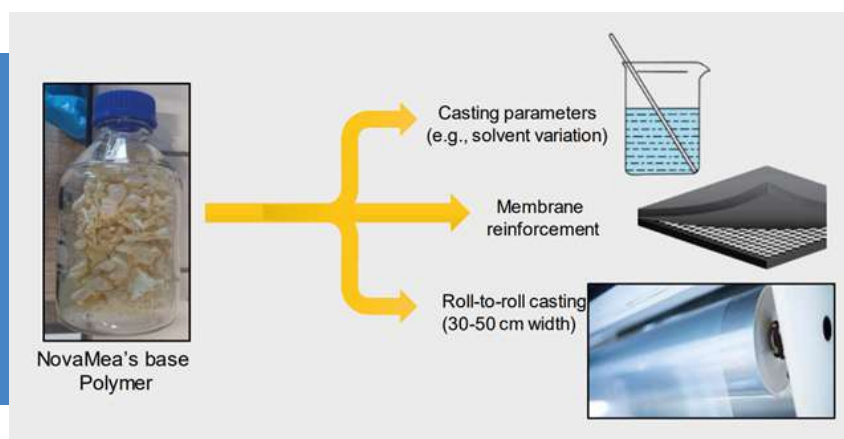
- The preliminary results show that it is possible to achieve the targeted Mg and Li ions rejection with membrane-based plants.
- Simply modified membrane systems are not able to comply with the requested rejection levels.
- Advanced treatment with addition of several carbon-based layers provided most promising solutions.
- The techno-economical study through the flowsheeting modeling conducted provided feasibility support for the scale-up of the method.
- More advanced techno-economical studies are required for each particular case as the selection of the optimum solution varies depending on several factors such as feed composition and scale.

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SUMMIT

Scaled-Up Manufacture of Membranes and Ionomers for the energy Transition

The SUMMIT democase centered around the development of high-performance anion exchange membranes (AEM) for water electrolysis, which plays a crucial role in the production of green hydrogen. NovaMea, a cleantech start-up spun out from the École Polytechnique Fédérale de Lausanne (EPFL), in collaboration with the membrane group of Prof. Dr. Mathias Ulbricht from UDE, aimed to scale up membrane production processes from lab-scale to industrial levels. NovaMea's innovative AEM technology holds significant potential to reduce reliance on expensive, precious metal catalysts while enabling high current density and compatibility with intermittent renewable energy sources such as solar and wind power. At the heart of the project is the challenge of transitioning from small-scale, sheet-to-sheet membrane casting methods to a continuous roll-to-roll production process. Currently, NovaMea's membranes are produced at relatively small areas, but to make the technology commercially viable, large-scale production could be achieved without compromising the membrane's favorable electrochemical properties. To this end, the INNOMEM project provided access to pilot-line facilities and expert resources, allowing for the necessary scale-up.



The project made significant progress across its phases despite initial delays. We successfully completed lab-scale casting experiments, with optimizing key parameters such as polymer concentration, casting speed and drying conditions. Furthermore, reinforcement strategies were established, with a porous support identified as best choice. The optimized conditions for casting unsupported membranes and feasible conditions for casting supported membranes were used for pilot-line roll-to-roll casting trials, though delayed and limited in terms of size of resulting samples, due to limited amount of polymer. Nevertheless, in pilot-line trials, the influence of varied process parameters such as cast thickness, casting speed, and drying conditions was evaluated. Membrane performance metrics like ion-exchange capacity, membrane conductivity, tensile strength indicate that the resulting materials are competitive with state-of-the-art anion-exchange membranes for alkaline water electrolysis.

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SOLFROTUBES

Solvent-free production of low-fouling, chemically stable tubular filtration membranes

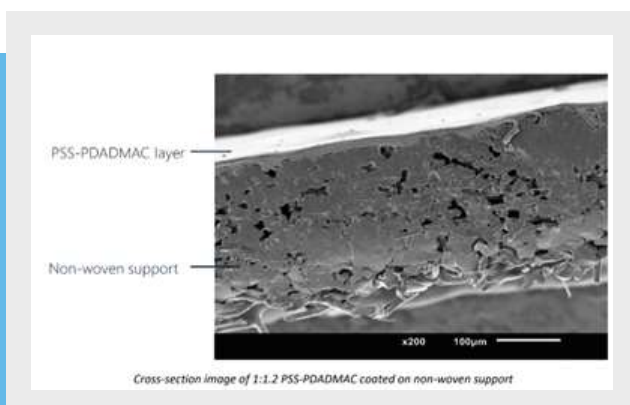
Berghof Membrane Technology GmbH offers a full line of tubular membranes for filtration and separation of industrial effluent and process streams. These membranes deliver the resilience and robustness necessary to handle challenging treatment applications in various industries including dairy, food and beverage, chemical, pharmaceutical, mining, landfills and petrochemicals.

Berghof Membranes applied for a demo-case, because we see the necessity to develop tubular membranes by means of salt-dilution induced phase-separation (SIPS) to provide chemically stable low fouling polyelectrolyte complex (PEC) based membranes. The SIPS technique provides the opportunity of water-based production membranes. There are four objectives for this project:

1. Solvent-free production: remarkable reduction in use of solvents is expected.
2. Low-fouling: this is beneficial to the end users and decreases the environmental footprint.
3. Chemical stability: long-term application of these robust membranes by end users. Optimized cleaning is possible, which decreases chemicals as well as energy consumption.
4. PFAS-free: there is a clear need to reduced fluoropolymers and similar substances

THE RESULTS AND CONCLUSION

The non-woven support has a smooth and a rough side of which the rough side was chosen for the coatings. From the SEM images it is quite evident that the coating is well integrated on the support.



Polyelectrolyte solutions with PSS and PDADMAC in the stoichiometric ratio of 1:1.2 was prepared and additionally to these solutions additives like PEGs were also added. Pure water permeability tests were carried out for these membranes and the permeability for these membranes varied in the ranges of 5-11 LMH/bar. Salt retention tests were carried out with $MgSO_4$ and the retentions were less than 5%. Molecular weight cut-off was carried out with PEG. The results showed a MWCO higher than 35000 Da.

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COHY Coke Oven gas and Blast Furnace Gas to HYdrogen

In the COHY project, three membrane-intensified processes have been recommended: (a) hybrid two-stage membrane/VPSA systems for the cost-effective recovery of CO₂ from KARDOKMAK's emissions, (b) high hydrogen-selectivity MS membranes for the cost-effective recovery of hydrogen from KARDOKMAK's COG emissions, and (c) water-selective molecular sieving (MS) membranes for the cost-effective hydrogenation of CO₂ to methanol.

The hydrogen-selective Molecular Sieving (MS) membrane offer superior H₂/CO₂ selectivity and hydrogen permeance compared to conventional Pd membranes. Based on this, they can be used for producing hydrogen from KADOKMAK's COG emissions, instead of burning it. The two-stage membrane-intensified unit is superior to conventional amine absorption technologies, being 50% less expensive for recovering CO₂ from BFG emissions. Water-selective Molecular Sieving (MS) membranes are superior to conventional zeolitic and other membranes at high temperatures for shifting the equilibrium of CO₂ conversion and reducing the cost of producing readily transportable methanol.

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Per- and polyfluoroalkyl substances (PFAS) constitute a group of synthetic chemicals widely utilized in various products for their water- and grease-resistant properties. The persistent nature of PFAS in the environment and potential health concerns have sparked a growing interest in devising effective methods for their removal from water sources. Current technologies for treatment are mainly IEX or adsorption-based technologies. The efficiency of these processes depends largely on the wastewater matrix and the type of PFAS which needs to be removed. Membrane technologies are also considered as a treatment possibility for PFAS. The required performance and choice of membrane will depend on the re-use water quality requirements, the wastewater matrix and the PFAS profile in the wastewater. When implemented as a pre-treatment, membranes can play a part in reconditioning the wastewater by altering the wastewater matrix. Compounds which inhibit possible post-treatments can be separated from the target PFAS compounds, which improves the overall process efficiency.



Typical PFAS molecule and NXFiltration membranes efficient
in PFAS removal (www.nxfiltration.com)

The PFASELECT project was successful and the following conclusions can be drawn:

- The 7 selected polymeric and ceramic membranes show relatively high PFAS retentions, even for the ultrashort PFAS species (< 200 Da). Retentions vary between 50 and 95% depending on membrane, PFAS chain length, and PFAS distribution in the feed.
- Several membranes show retentions for short-chain PFAS > 80%
- Especially Grignard grafted ceramic NF membranes show separation of ions and PFAS species: PFAS are well retained, while ions typically inhibiting post-treatments are transported. Recovery of purified PFAS > 80% is feasible
- Proof of Concept testing has shown the robustness of PFAS concentration and purification process over several days

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Despite the shift towards more eco-friendly technologies in recent years, the membrane manufacturing industry still uses toxic solvents like DMAC, DMF, and NMP to produce synthetic membranes. The main goal of the EcoSolventCycle project is to substantially enhance the sustainability of membrane fabrication by investigating the feasibility of process water recycling. Therefore, the challenge is to separate the (green) solvents and polymeric additives (glycerol, PVP) from the wastewater of the manufacturing process. The project partners studied the removal and possible recycling of various organic solvents with membrane technology, as the current industrial solvent recycling relies heavily on thermal separation approaches, which are energy-intensive and account for 80% of the industrial energy use. On the other hand, membrane separations can selectively distinguish molecules based on a nuanced interplay of molecular size, shape, and physicochemical interactions and uses up to 90% less energy than conventional distillation. Within this project, various membrane processes have been explored, including ultrafiltration (UF), nanofiltration (NF), organic solvent nanofiltration (OSN), pervaporation (PV), and membrane distillation (MD). The project EcoSolventCycle aims to screen a large variety of membranes, identify the optimal membrane processes, and propose an industrial separation cascade. Furthermore, a first techno-economic assessment (TEA) allows to define the next steps for possible piloting and upscaling of the proposed process.



Figure 2. Small bench-top unit (1 L) for membrane filtration screening (left), membrane distillation unit (middle), and pervaporation unit (right)

The EcoSolventCycle project was successful leading to the following conclusions:

- Commercial, open polymeric membranes can successfully remove the PVP with a solvent recovery of over 90% (KPI1).
- Both pervaporation (PV) and membrane distillation (MD) provide excellent dewatering with an NMP and glycerol rejection over 99%. Over 90% of the water can be recovered (KPI2).
- Liquid-liquid extraction (LLE) can be used in combination with distillation to recover over 75% of the solvent mixture (KPI3).
- A first techno-economic assessment (TEA) revealed that the MD-based process costs significantly less than the current burning and a benchmark distillation train.

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The demand for carbon-rich bio-based raw materials is increasing sharply in the chemical sector. Sugars play an important role in this transition because they can serve as a replacement for fossil raw materials for production of chemicals such as acetic acid or (bio)plastics such as PLA and PHA. Recell has developed an innovative technology for recovery of tertiary cellulose from residual waste(water) streams with the aim of processing the cellulose present into high-quality glucose that can be used as a raw material for the chemical industry (so-called third generation glucose). A pilot plant (TRL 7) is currently in operation in Leek, The Netherlands. Recell's ambition is to have a facility to produce 50 kt sugars in 2025. The recovery and purification of the sugar-rich streams from Recell's process, studied in PureGluc, is crucial to reach market demand. Obtaining generic semi-finished glucose products with standardized quality is considered essential for the development of the value chain and application as renewable feedstock for high added value bio-based molecules in tomorrow's chemical industry.



Renewable third generation cellulose (left) and glucose (middle) obtained from residual streams, hydrolysate before and permeate after membrane filtration (right)

The PureGluc project aims to develop and demonstrate an efficient membrane-based process for recovery of high-purity glucose from a hydrolysate of a complex residual wastewater stream obtained through Recell's Chem process. In the framework of a previous project (funded by the Dutch government), membrane filtration was shown to provide a promising technology option for downstream purification. An efficient post-treatment process is envisioned with the aim to purify and concentrate glucose to the highest standards. The experimental results of this project allow to perform a first techno-economic evaluation and estimate glucose recovery costs, while providing robust data for industrial process design or further research.

The following promising conclusions can be drawn from the PureGluc project:

- All selected UF membranes exhibit high raw materials retentions > 99% (KPI 1) in combination with a high recovery of glucose in the permeate.
- Several NF membranes show high glucose retentions > 95%, however maximal glucose concentration and yield in longer-term concentration tests are relatively low.
- However, when applying other techniques on UF permeate, targets on glucose concentration > 200 g/L (KPI 2) and glucose recovery > 85% (KPI 3) are met.
- Alkaline membrane cleaning allows for process flux recovery > 80% in UF (KPI 4).

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A-membranes is a start-up company which has acquired the rights to the technology to graft ceramic membranes using organometal (Grignard) chemistry, partially owned by INNOMEM partner Vito. A-membranes is currently investing in the development and implementation of an industrial process for the grafting at high MRL level. The benefits of Grignard grafting organic moieties to the surface of ceramic membranes has been demonstrated in numerous academic publications and in industrial demonstrations as well. However, the technology has limitations. The current state of Grignard modifications only grafts organic moieties on TiO₂ or ZrO₂ filtration layers. No moieties are attached on standard Al₂O₃ supports keeping their original polar behaviour. This leads to relatively low, non-economical flux levels in applications with less-polar solvents as e.g., n-hexane or petroleum-derivates, caused by the lack of affinity of the unmodified and polar Al₂O₃ support material, working as a “brake” to the flux.

Fraunhofer IKTS has developed tubular titania ceramic membrane supports and nanofiltration (NF) and ultrafiltration (UF) membranes derived from them. In the TNT Demo case Project partner VITO grafted these innovative full TiO₂ membranes “Through & Through”, i.e., not just the outer filtration layers but the interior macro-porous TiO₂ as well, and this when using the Grignard grafting technology developed at VITO and transferred to A-membranes. With this project, A-membranes seeks to compare filtration results of these grafted mono membranes to the results obtained in some well-documented cases on comparable grafted Al₂O₃/TiO₂ membranes. We anticipate seeing increased flux levels for lower polarity solvents. This undoubtedly increases both the technical and economic application window for the technology. Tests have been performed both on UF and NF membranes, to understand also if the narrowing of the pore size could have an impact on the efficiency of this “support grafting”, due to e.g., steric effects.



Full TiO₂ ceramic membranes (left) and grafting Pilot Line (right) used in the TNT project.

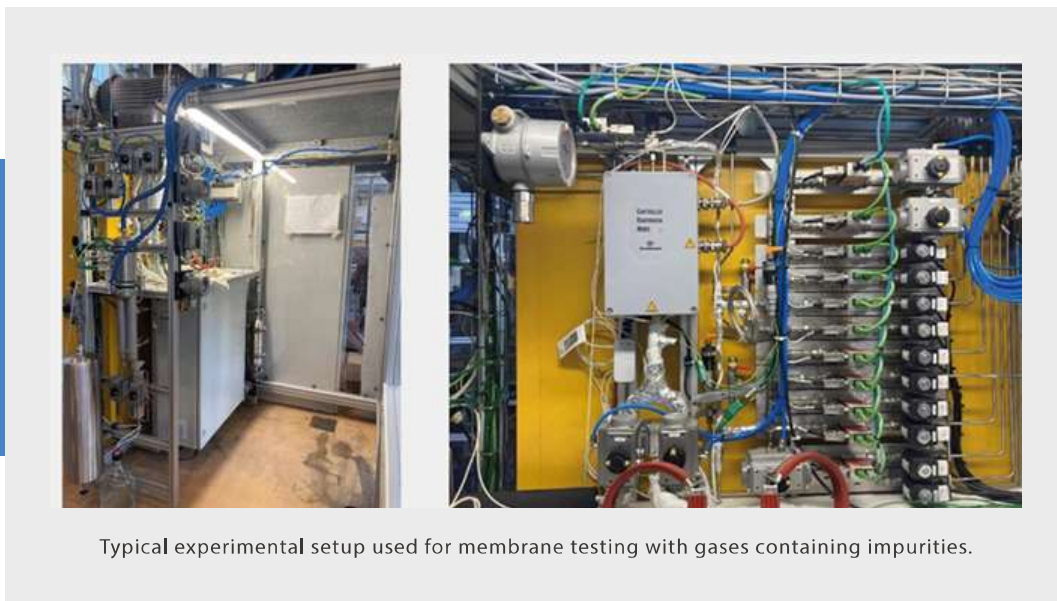
The following promising conclusions can be drawn from the TNT project:

- TiO₂ supported ultrafiltration and nanofiltration membranes can be successfully grafted “through and through”, i.e., not just the outer filtration layers (top layers) but the interior macro-porous TiO₂ support as well.
- SEM-EDX analysis of the top layers and the support show clearly increased carbon contents after methyl Grignard grafting
- First filtration results with apolar solvent-based model mixtures, mimicking a variety of industrially relevant applications, show increased fluxes for TNT grafted membranes
- This will allow A-membranes to increase their market for grafted membranes to e.g., the edible oil market, pyrolysis oil from plastics, petrochemical applications, food and beverage market etc.

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Membrane models are available at different scales but used especially in academia. These models are generally ad-hoc models and are not available in commercial tools. MODELTA, has created commercial tools that are integrated in simulation software like ASPEN. However, these models need a large array of experimental data to be able to validate the models in different conditions and for different membranes. This requires production (or in general acquisition) of different membranes and testing is several conditions and a great knowledge of all the parameters used in the testing along with physico-chemical characterization of these membranes. This is why it is often difficult to validate models with literature data, as those are generally not complete or critical info are missing. MEMODEV project has helped MODELTA acquiring the experimental data needed for their model validation using dedicated experimental setups available at TUE.

Several membranes, carbon and Pd based, have been produced and tested for permeation. The results have been obtained for several conditions including pure gases, mixtures of binary gases and more complex mixtures and created in tables that can be used by MODELTA for their model validation. Membranes have been tested at high pressure and in the conditions required by the company. Also, commercial membranes have been tested, especially polymeric membranes.

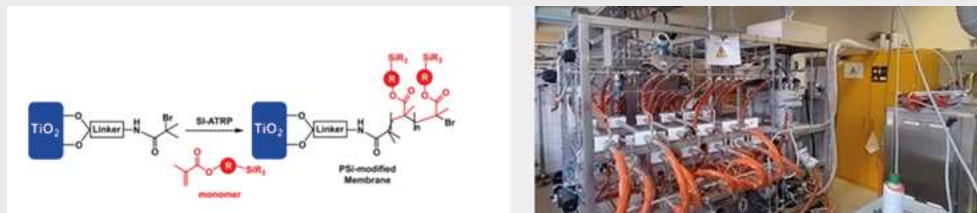


Typical experimental setup used for membrane testing with gases containing impurities.

Compared with the situation before the project, MODELTA has now access to a large array of experimental data on different types of membranes. While data are available, characterization of the membrane also help MODELTA to validate their model, thus TUE will continue support the validation also beyond the project. Membranes have been tested in the conditions required for validation

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Milk whey is one of the main by-products of the dairy industry, having excellent nutritional properties. Current demineralization processes based on ion exchange (IEX) resins are becoming more and more uneconomical due to increasing resin regeneration costs. Partial demineralization of milk whey by nanofiltration prior to IEX would allow for a significant reduction of these regeneration costs (chemicals, water), while also enabling a flexible capacity enhancement. Membrane filtration can be considered a “natural” technology choice for food processing thanks to its clean and energy-efficient character, gentle processing conditions, flexibility, scalability, and complementarity with electrification trends. Despite these features and the clear opportunities for CO₂-savings, implementation of membrane filtration is still relatively scarce and hampered by technical challenges such as lack of robust membranes in the market. Building further on a previous collaborative project, the Mem4SusDiary project aimed to demonstrate an efficient NF process using modified ceramic membranes to pre-concentrate and partially demineralize milk whey before the IEX step. This NF process is intended to replace an RO process at a specific FrieslandCampina production site, in such a way that the demineralized whey output per batch can be increased, and the chemical consumption and wastewater generation associated with IEX can be reduced, resulting in significantly lower production costs per kg of product. Experimental development included systematic testing of a variety of modified ceramic membranes synthesised in house at VITO on a representative milk whey mixture, and benchmarking against commercial polymeric NF membranes. After these screening trials, the best performing modified ceramic membrane was scaled up to multi-channel modules, and proof-of-concept-trials were carried out under industrially relevant conditions.



Schematic representation of modified ceramic membrane (left) and picture of modification pilot installation (right) as used in Mem4SusDiary project.

The following promising conclusions can be drawn from the Mem4SusDairy project:

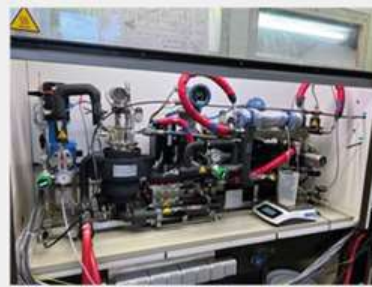
- Various modified ceramic membranes show low and sharp cut-off, combining high lactose retention >98% and low salt retention, allowing for efficient recovery of lactose and milk proteins (>90%, KPI 1), hence efficient demineralization of milk whey (>50%, KPI 2), competitive to polymeric reference membranes.
- Best performing modified membrane (HyMem) was successfully scaled up to 19-channel geometry for the first time.
- Compared to small scale screening trials, multi-channel HyMem membrane exhibited somewhat lower lactose yield and flux performance (approx. 1.3 L/hm²bar at start of concentration, KPI 3), however flux recovery after repeated filtration-cleaning cycles was excellent (>85%, KPI 4).

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Liquid-ion Battery (LIB) manufacturers are using large amounts of solvents. Especially N Methyl-pyrrolidone (NMP) is an indispensable auxiliary material for manufacturing of cathodes. Indaver has developed a process to purify NMP from LIB manufacturing waste till battery grade specification using dedicated distillation units. Due to the very stringent specifications, especially on metal contaminations (typically 10 ppb), transport of purified NMP from these dedicated units to the LIB factory poses an additional challenge, with a significant risk of metal (re-)contamination. Therefore, Indaver is looking for alternative downstream purification technologies, capable of removing metal contaminations arising during LIB manufacturing, as well as during solvent processing and transport.



Schematic LIB manufacturing process



Membrane filtration installation at VITO

The Mem4Bat project leverages on the successful developments and insights gained in the former EU project SOLVER where VITO together with several solvent re-refiners studied membranes for trace metal removal from electrochemical grade solvents, mainly alcohols. In this latter project, organic solvent nanofiltration (OSN) proved to be capable of removing 35 key trace metals till (sub-)ppb levels, which was demonstrated up to pilot scale. However, purification of spent NMP poses significant challenges, as few membranes, even dedicated OSN membranes and modules, provide long-term stability in such aggressive polar aprotic solvents which are commonly used for membrane manufacturing as well. Only a small number of commercial membranes, including ceramic membranes, are robust enough without significant swelling, which is essential for selective removal of very small metal contaminations.

The Mem4Bat project allows to draw the following promising conclusions:

- Analysis of metal traces at ppb levels proved to be very challenging, requiring more efforts and taking far more time than expected, however significant progress was made by an external lab, and a suitable method for evaluation of the membrane trials was developed.
- Direct processing of a highly contaminated crude spent NMP sample using UF/NF membranes was not successful.
- A clean pretreated NMP sample was sourced and various membrane technologies, incl. OSN, PV and MD have been tested on it, with encouraging results regarding removal of metals and water.

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Innomem Final Workshop

Discover the opportunities of the 1st European single entry point for all your membrane related questions!

10 September 2024, 14:00 – 18:00,
Prague Congress Centre – Chamber Hall, Prague (CZ)

www.innomem.eu



For more information: a.brunetti@itm.cnr.it

On 10 September 2024, the INNOMEM Project held its final workshop as part of the EUROMEMBRANE 2024 conference at the Prague Congress Centre. The event, which took place in the Chamber Hall, gathered leading figures from academia and industry to discuss the latest advancements in membrane technology.

Attendees had the opportunity to dive into cutting-edge innovations in membrane processes and engage in thought-provoking discussions with experts. The workshop fostered a collaborative environment for networking and the exchange of ideas among professionals from various sectors.

The esteemed speaker line-up included:

- **Jon Zuñiga** – TECNALIA Research & Innovation
- **Mathias Ulbricht** – University of Duisburg-Essen (UDE)
- **Lidietta Giorno** – CNR-ITM
- **Jon Meléndez Rey** – H2SITE
- **Carla Glass** – University of Twente / UCANEO
- Efecan Pakkaner – VITO
- **Torsten Brinkmann** – Helmholtz-Zentrum Hereon
- **Patrick de Wit** – EMI Twente BV

This event, open to all registered participants of EUROMEMBRANE 2024, highlighted INNOMEM's role as Europe's premier single-entry point for membrane-related inquiries. The workshop served as a platform for sharing innovation and insights, advancing the field of membrane technology through collaboration.



INNOMEM Consortium



Project details

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Duration:	48 months
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