

Open Innovation Test Bed for nano-enabled Membranes



# Updates on the second year of activities

The Innomem Project is entering in its third year of implementation, and many are the results achieved in the frame of the first 24 months of implementation!

Read this newsletter to learn all the progresses achieved by the consortium and to discover which are the next steps.

Remember to follow Innomem on LinkedIn, Twitter and Facebook to be always updated on the latest news!

# M24 project meeting

On 4-5th May 2022 INNOMEM Consortium finally met in person at University of Duisburg-Essen, after 24 months of virtual meetings!

The meeting, led by Tecnalia – the project coordinator- explored the results of each work package and all the partners had the chance to describe the progresses made so far.

A poster session dedicated to Pilot Lines progresses has been an astonishing occasion to discuss face-to-face of the progresses made by the project up to now.

In the next months, interesting results and successful initiatives are foreseen. Stay tuned!

Thanks to Mathias Ulbricht and his team for hosting us!! See you on Month 30 in Sorrento!



# **PROGRESSES ON THE ACTIVITIES**

# Fundacion Tecnalia Research & Innovation

The upgrading and upscaling of the two advanced membrane manufacturing pilot lines of Tecnalia have been finished: PL1 - Dual Layer Mixed Matrix hollow fiber spinning system for gas separation membrane development and PL2 Electroless plating system for Pd-based membrane development onto porous tubular supports. At the moment our team is working on the synthesis of the mixed matrix hollow fiber membranes for the showcase n°3, where membranes will be evaluated for the separation of CO2/N2 and CO2/CH4 gas mixtures; and the synthesis of the palladium-based membranes for showcase n°4, where the membranes will be evaluated for 2 Nm3/h of H2 production from biogas.

# TUE

The Technical University of Eindhoven (TUE) is responsible for the creation of a virtual lab and an expert system for modelling of membranes and membrane reactors within the INNOMEM project. Within the last few months, TUE has finalized the creation of a virtual lab for modelling through collaboration between all modelling partners and has been maintaining it (see https://www.innomem.eu/virtual-lab-modelling/). This virtual lab connects potential clients that have modelling requests to capable modelling partners within the INNOMEM consortium, and it is a prerequisite to the development of an expert system on membrane and membrane reactor modelling which will be able to do preliminary calculations to provide a more rigorous and advanced user experience. As part of the modelling capabilities in the virtual lab, modelling activities are currently being conducted within TUE on describing transport mechanisms in carbon membranes with a phenomenological model. This parallel resistance model uses a pore size distribution as a weight factor to incorporate multiple transport mechanisms in parallel.

Secondly TUE is in charge of a training & services catalogue. Preliminary works are being done on the creation of an educational video that focuses on membrane reactors and membrane modelling capabilities within TUE. This will be combined with both introductory and advanced level course material provided by pilot-line owners and service providers through knowledge developed in the project. It will ultimately serve to address a wide range of interested parties (e.g. potential adopters, non-technical staff, technical experts, students).





# **CNR-ITM**



CNR-ITM is responsible of the organization of the Virtual Testing and Characterization Lab, a lab that gathers facilities and apparatus for full characterization and testing of membrane materials, membranes and membrane operations (membrane reactors, gas separation, membrane distillation, pervaporation, micro/ultra/nanofiltration, solvent filtration, electrodialysis, etc.) passing from morphological/structural characterization to mass transport properties evaluation, long-term tests, pilot applications.

The VIRTUAL lab is located at partners' premises and spread in various EU Countries. CNR-ITM coordinates and harmonizes the activities of the various partners, defining set procedures for testing and characterizing the membranes.

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Scheme of activities steps on Virtual Membrane and Characterization Lab

# VITO



VITO (Flemish Institute for Technological Research) is an independent Flemish research organisation in the area of cleantech and sustainable development, and one of the leading membrane institutes in Europe. Within INNOMEM, VITO is WP1 leader, coordinating and streamlining the upscale and upgrade actions of 14 breakthrough membrane manufacturing Pilot Lines (PLs), under a common umbrella of a Sustainable Manufacturing framework (INNOMEM-SMF).

During the first 10 months of the project, an efficient sustainability assessment methodology was defined, consistent with current quality & environment & (nano)safety good management practices and allowing assessment of the three Sustainability Dimensions (SDs) Social, Environment and Economics. The adopted SMF tool, developed by TECNALIA in a sister-OITB OASIS, allows scoring of the current and targeted sustainability situation in the 3 SDs further subdivided in 9 Sustainability Items (SIs), and definition of appropriate KPIs and improvement plans for a chosen set of SIs.

From month 10 onwards, all PL owners started working on the initial diagnosis of the SMF in their PLs, and the description of their intended sustainability improvements, correlated to their actions for PL upgrade/upscale in WP1. All actions were completed, and the results summarized by VITO in Deliverable 1.2, by month 21. We can conclude that membrane production within INNOMEM can be considered fairly sustainable at the start of the project, with a score of 41,8%. At the end of WP1 in M24, a sustainable situation is targeted with a value of 58,9%. From the Figure below, it can be deduced that sustainability is strongest in the social dimension and weakest in the economics dimension, and that the intended sustainability increase is similar in all 3 dimensions.



Comparison of INNOMEM OITB sustainability targets with current status

#### INNOMEM NEWSLETTER | ISSUE No 3

#### **UNIZAR+POLYMEM**

UNIZAR is involved in Pilot Line 9: "Modifications of Hollow Fibers by microfluidics for water treatment by Nanofiltration". U NIZAR's m ain o bjective is the preparation of thin-film composite (TFC) and thin-film nanocomposite (TFN) membranes on the inner surface of polysulfone hollow fiber (HF) supports provided from **Polymem**. Microfluidic technology was used for that purpose controlling the key process parameters. By this technology, UNIZAR is able to create membrane layers by interfacial polymerization (TFC, TFN) or continuous MOF layer membranes. Modules from 1 to 25 HFs and lengths from 10-15 cm to 52 cm with effective membrane areas up to ca. 66 cm2 were successfully prepared and tested in our nanofiltration set-up for dye removal (Rose Bengal, 974 Da) from water. Highly reproduced measurements (> 30 membrane modules tested) with 70 % of membranes with rejections of 95 % and average permeances of 0.9 kg.m-<sup>2</sup>.h-<sup>1</sup>.bar-<sup>1</sup>.

# ICL-AU-LiqTech

Showcase 5 is dedicated to innovating ceramic substrate technology, together with taking use of the latest catalyst, to develop highly compact and efficient catalytic convertor. Through the close collaboration among Imperial College London, Aston University and LigTech, the new ceramic hollow fiber substate with desired macro- and micro-structures has been fabricated, and the small, bundled monolith is also assembled for prototype test. In the meanwhile, the catalyst with lowest light-off temperature has been identified from several candidates.

# Helmholtz-Zentrum Hereon

Helmholtz Zentrum Hereon is part of the Helmholtz Association, Germany's largest scientific organization. The research center's approximately 1,100 employees make substantial contributions to clarifying major and pressing issues in key scientific, economic and social topics. Hereon's Institute of Membrane Research is developing innovative membranes and membrane processes to address global challenges as water shortages, climate change and energy transition. This involves a holistic, interdisciplinary approach: it includes developing new materials, processing these materials into membranes as well as constructing pilot plants in which these systems are made ready for implementation. Environmentally friendly manufacturing processes are at the forefront of all of our activities. All steps are accompanied by comprehensive computer simulations. With the help of these digital twins, developing modern membrane systems can be considerably accelerated. The core of Hereon's activities in INNOMEM is the improvement and operation of a pilot line for the production of thin film composite membranes for the separation of CO2 and supplying the membranes installed in advanced membrane modules to project partners. The improved membrane production infrastructure will be made assessable to interested parties via INNOMEM's Single Entry Point (text von Newsletter 2)

### Imperial College London







Universidad

Aston University

Zaragoza



LIQTECH

CERAMICS



Upgrading of the pilot line during the M24 period of Innomem includes environmental aspects like an improved enclosure of the coating device for the thin film composite membrane production. Old motors of the coating machine were changed, and electricity meters of to all machine correlating to membrane production were implemented in order to monitor the energy consumption. Furthermore, the documentation of process data and quality parameters was improved in some extend with an automatic registration. For membrane production with respect to the Innomem project membranes for n-C4H10/CH4 were fabricated in 50 m<sup>2</sup> scale. With the pilot line it was possible to upscale a CO2/CH4 separation membrane with 6FDA-6FpDA layer from 1 m<sup>2</sup> to 8 m<sup>2</sup>.

# **University of Montpellier**



European Membrane Institute (IEM) at University of Montpellier (UM) is involved in INNOMEM project and is owner of a pilot line to produce polymeric hollow fiber membranes. Thanks to close collaboration with the company Polymem, IEM has developed knowledge on monitoring of spinning line for producing surface-modified hollow fibers by co-extrusion. The IEM is also equipped of different experimental set-up for Atomic Layer Deposition (ALD). The study conducted in the frame of INNOMEM project consists on using this technique on polymeric membranes, which is non-trivial for polymeric material and this requires to determine an appropriate fiber conditioning protocol.

The surface modification of hollow fibers aimed at reducing the effect of protein fouling. Trials are conducted to characterize the properties of the obtained membranes in terms of permeability, mechanical property and morphology.



Spinning line for producing surface-modified hollow fibers

# UTWENTE

#### **UNIVERSITY OF TWENTE.**

In the industry hollow fiber (HF) membranes are produced using a continuous spinning process. Often a surface modification on nanoscale is done to enhance their performance. This is mostly done off-line and batch-wise. Within the scope of the INNOMEM project, University of Twente/EMI-Twente is upgrading their hollow fiber spinning line with an in-line electron-beam (EB) device, eliminating the need of an off-line surface modification. This will increase the productivity and reduce the amount of rinsing water and handling. The focus will lie on the modification of a new type of dense nanofiltration (NF) membrane to improve the stability and performance. This will be done in a showcase together with **NX Filtration**. In general E-beam irradiation is suitable for:

- Polymer crosslinking
- Nano structure linking
- Functional molecule and enzyme linking









Hollow fiber spinning line with an in-line electron-beam (EB) device

#### **UDF & FVONIK**

The in this project involved Chair "Technische Chemie II" (TCII), led by Prof. Ulbricht (www.uni-due.de/tech2chem), has a key position within research and teaching in the Chemistry Department of University Duisburg-Essen, and it is an active member of CENIDE and ZWU. The research of the Ulbricht group is devoted to functional polymeric materials with a focus on membranes and particular emphasis on membrane applications in water purification, bioseparation or energy technologies.

In INNOMEM we are responsible for pilot line 7, a roll-to-roll coating system (COATEMA Click&Coat®), which is used to manufacture flat sheet membranes in a continuous process. During the first 24 months of this project, the pilot line was established for the production of anion-exchange membranes and manufacturing was subsequently upscaled to achieve high production rates at low drying temperatures. To offer the pilot line as a setup that is capable of production under industrial standards, a support cleaning system, an in-line surface plasma treatment module and a laminating

COATEMA Click&Coat®

unit were designed and then implemented by a contractor. As part of showcase 9 in cooperation with **Evonik**, the lab scale synthesis of anion-exchange membranes from a novel polymer was successfully transferred to the pilot line. Furthermore, the nanoscale morphology of membranes was investigated, and new insights into the relationship between manufacturing process, morphology and membrane properties was generated.

## **RWTH & FILATECH**



Based on the knowledge and long-term experience in designing and manufacturing of spinnerets of both partners (RWTH Aachen University, FTI Filatech Innovation GmbH), a toolbox for the design of 3d printed polymeric spinnerets was generated and transferred for fabrication of metallic 3d printed versions. Tests with several different designs of polymeric spinnerets were carried out and design was optimized. Modifications of FTI's spinning line for installation of RWTH's 3d printed rotating spinneret were done to showcase the potential of novel geometries membranes.

First round trials for production of membranes were done and industrial scale module samples were prepared. Characterization at both partners is in progress.







eco-friendly electrochemical graphite exfoliation was deployed to produce GO and replace the Hummer method. The initial pilot unit was eventually upscaled from a unit treating 5 membranes with a specific surface area of 0.001 m2 to a module with a total membrane surface area of 0.72 m2. Finally, the NF module was integrated into a low energy demand desalination NF/FO ZLD brackish water pilot unit to produce irrigation and fertigation water.

(polyethyleneimine) -functionalized Graphene Oxides (GOs).

In the activities devoted to Showcase #10, the CVD and ALD systems were upscaled to a system capable of treating ceramic and stainless-steel membrane tubes of a length of 0.5 m. CVD modified nanoporous ceramic membranes are produced using a TEOS/ozone system to tailor the nanopore size by depositing SiO2. In addition, an ALD system has been included to modify membranes by deposition of metals. An in-line probe-molecule permeation system was used to monitor in-line the evolution of the nanopore size during the CVD or ALD processes. Finally, in-situ Helium gas relative permeability and differential permeability has been included to evaluate the evolution of the nanostructure.

#### DBI

At DBI, membrane tests for connected Pilot lines PL 5# (HEREON) and PL 6 # (FHG) were further conducted. The experiments are focused on carbon dioxide and n-butane separation from natural gas.

The upscaling of membrane test facility is almost finished. The experimental setup facilitates membrane tests with feed pressure up to 60 bar and feed flow up to 1 m3/h. Additional typical trace impurities like hydrogen sulphide can be added to study degradation phenomena. The gas stream can also be loaded with water via saturator to investigate the influence of water content on separation performance.

#### RKV

Rauschert Kloster Veilsdorf (RKV) has developed single channel tubes with and without caps. An alumina material is used with a very particular dimension, according to the parameters defined together with Tecnalia (TEC). The outer diameter was defined with 14 mm and the inner diameter with 7 mm. The length of the tube is 500 mm. Finally, the active layer is 100nm  $\alpha$  – Al2O3 on the outer part of the support. By M24, 4 batches with a total value of 150 – 200 tubes will have been delivered, which comply with the agreed limit values for N2 - permeation. In order to achieve these desired properties a special coating technology was developed. Attempts to optimize this technology and minimize the rejects are currently being carried out. A Two-third reduction in rejects is within the realm of possibility and would result in a 10% reduction in the final price.

# **DFMOK**



In addition, cost-effective and



Gas- und Umwelttechnik GmbH



# PNO



Up to month 24 PNO has been primarily supporting the SEP value proposition. Two deliverables in WP2 composed a comprehensive market analysis, which explored the membranes market and provided a specific analysis to understand what kind of customers the SEP could be engaged by. The analysis included both a desktop search and a survey. The customer characterization revealed a large percentage of SMEs, many in the pre-revenue stage, which adds complexity to the business to be set up. In parallel, the service catalogue was assessed and published. From Month 18 and on, PNO has kick-started both WP4 (Open Call) and WP7 showcases business plans. The activities have been defined, on one hand, to prepare and manage the Open Call in WP4, defining their criteria and the selection process, and on the other hand to support the showcases in clearly explaining to the world their value and develop their business cases.

Finally, PNO also supports the Dissemination and Communication actions of Innomem, maintaining the project website and social media channels alive with updates related to the progresses and results achieved in the frame of the initiative.

## H2SITE & ENGIE & RKV & TECNALIA & TUE



In the frame of the validation of an advanced membrane reactor for pure hydrogen production and purification (showcase 4), many different progresses have been achieved in the last 24 months.

RAUSCHERT KLOSTER VEILSDORF GMBH (RKV) has been working on the reduction of rejected asymmetric tubular ceramic supports aiming to meet the quality targets in terms of nitrogen flow at room temperature and bubble point through different coating and drying position studies. FUNDACION TECNALIA RESEARCH & INNOVATION (Tecnalia) is being focused on palladium membrane pilot line validation. In total 7 batches (8 membranes each) have been fabricated and different fabrication control parameters have been changed looking for optimization. TECHNISCHE UNIVERSITEIT EINDHOVEN (TU/e)'s contributions are related to process design and modelling of the reactor and complete system which helped H2SITE in its reactor upgrading and the material and equipment definition. Also, different tests for hydrogen cleanup with commercial sorbents were carried out. HYDROGEN ONSITE, SL (H2SITE), has been focused on preliminary factory acceptance tests of H2 production with few integrated Pd-membranes (before the integration of final Pd-membranes can be done) where different issues were detected, and some pilot parts were changed. ENGIE (ENGIE) has been evaluating the testing site for the validation of the hydrogen production pilot using biogas as feedstock. Different issues such as pilot supervision, proper H2 release system, installation, and availability according to project schedule led to select ENGIE Lab CRIGEN as the validation site.

# **TECNALIA VENTURES**

The main objective of VNTRS activities is to establish the operation of the SEP. This includes the way it is going to create and provide value; and the organisation structure, appropriate legal entity and governance rules it will have.

During the first 13 months the Preliminary SEP Catalogue Market Analysis, the Preliminary SEP Definition & Business Model, the Final Market Analysis with focus on demand and the Final SEP Definition and business model were produced.

The other 12 months were aimed to develop the Qualification scheme and SLA and the SEP Governance and financial model. As a result of the activities which have been mentioned previously, Work Package 2 has been completed at month 24.

#### UNE

UNE, the Spanish Association for Standardization, is in charge of the standardization activities within INNOMEM project. Standardization is becoming more and more important as part of research and innovation projects since it is a useful tool to disseminate results to the industry, society and public administrations, providing compatibility, confidence and facilitating project developments acceptance.

An initial analysis of the standardization landscape has been performed, identifying existing standards and standardization technical committees related to INNOMEM research. Appropriate training has been provided to raise awareness among partners on the benefits and objectives of standardization tasks within the project and further analysis has been carried out to better identify the standardization needs. Strategy to follow in under development with proper consideration of the topics identified.

Due coordination with other European Test Beds is also taking place: a joint Workshop for test bed users has been organized and a standardization guidance document is being drafted with the aim of providing information, guidance and motivation for using standards and standardisation to future Test Bed users.



# **INNOMEM Industrial Workshop**

The INNOMEM project is about to present its best results to Industrial stakeholders and R&D experts during the first INNOMEM Workshop. It will be organized in Sorrento by CNR-ITM on November 17-18, 2022, as linked event to Euromembrane 2022!

Keep in touch to know what is at stake and who are the challengers.



Ready to come in Sorrento for Euromembrane2022 to celebrate the 40th EMS anniversary?

The abstract submission is now open, check our website:

#### http://euromembrane2022.eu

The deadline for abstract submission is June 10th, 2022.



# **INNOMEM** Consortium



#### STAY IN TOUCH

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