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MEMBRANE SCALE-UP FOR CHEMICAL INDUSTRIES

MEASURED aims at developing and demonstrating advanced membrane materials for pervaporation (PV), membrane distillation (MD) and gas separation (GS) technologies applied to acrylic ester production, membrane manufacturing and gas separation from a carbon capture & utilization (CCU) stream.

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WP1 – Exploitation and Business Model

M1 – M12

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WP 1- Goals

- To obtain a comprehensive view of European market and external players (stakeholders and potential customers) involved and KERs Expansion
- to determine a clear Value Proposition and a roadmap for the market uptake
- Initial Value Map (Business Model Canvas) for 3 business case and figure out the interactions with the market
- Characterise the markets, the stakeholders and their needs.
- Assess the outstanding features of our exploitable results.
- Identify proper business models for each result and set a plan for commercialization & market uptake.
- Characterize the different risks and identify solutions to mitigate them.

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Goals of the Market and Stakeholder analysis

Goals

- Identification and Analysis of Supply and Demand Markets,Their Segments, Trends and Forecasts, Barriers and Drivers
- Study of the Technology and Innovations Landscape
- Identification and Analysis of Relevant Key Players and Their Positioning (Collaboration Partners, Competitors, End-Users)

Analysis

- Data-driven Analysis carried out using owner and public databases for different types of analyses: Public and Private Funding, Patenting, Scientific Articles, Commercial Projects, Market Outlook
- Internal and/or External Interviews

Context

- Markets and R&D Outlook Observed at European Level
- Analyses centred on the last 10 years (from 2014)
- Three Main Business Cases based on Membrane Technology: Pervaporation (PV), Gas Separation (GS) and Membrane Distillation (MD)

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Market and Stakeholder Analysis Main Outcomes

TIMELINE & WORKFLOW

JAN-APR (M1-M4)

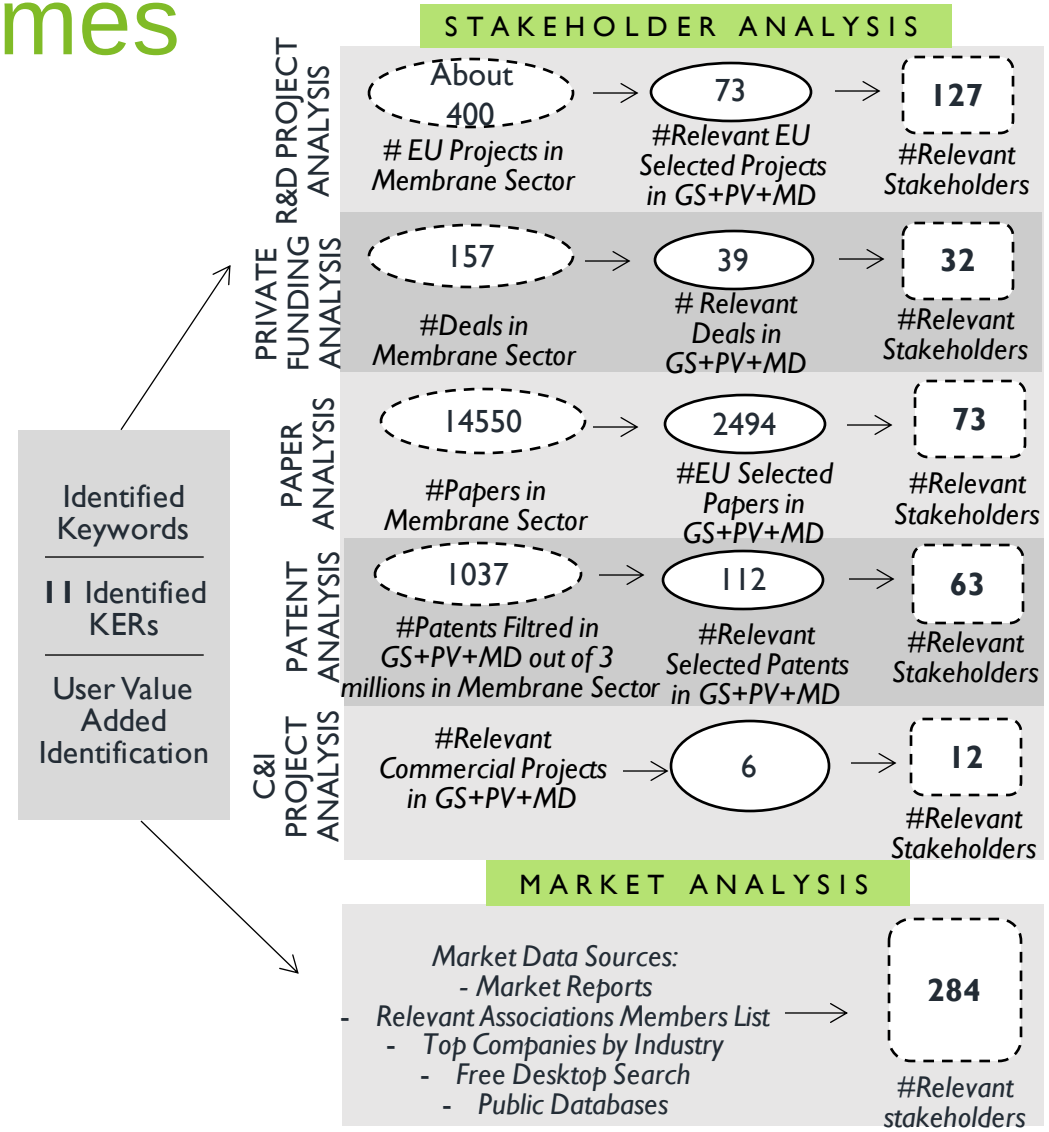
- Stakeholders and Market Analysis starts from preliminary talks to key partners, keywords and KERs /business cases study- The target is to capture the key market dynamics, barriers and key-players / competitors or potential customers /users

MAY – SEP (M5-M9)

- Defining the business plan ToC
- Desktop market analysis: segments, trends, products benchmark
- Stakeholder Analysis / Network Analysis to find R&D and industry leaders, list of potential research partners and connections for the project
- List of potential relevant companies /PNO's Market & Innovation Positioning Map

OCT – DEC (M10-M12)

- Interviews results to be integrated into Analysis
- Updated list of priority stakeholders
- Updating Stakeholder and Market Analysis
- D1.1 release



(a) Market and Innovation Trends Outlook

(b) List, Rank and Mapping of Relevant stakeholders: (**541**)

(1) Geographical Map by players' country

(2) Radars by role

(3) Value Chain Positioning Map

(4) Competitors and Potential End-Users Mapping

(5) Network Analysis

(6) Market and Innovation positioning map

(c) 6 Interviews performed

DELIVERABLE D1.1 RELEASED

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Value Proposition

The following information have been collected and fully described within the Business Model deliverable:

- 1) Technology state of art and comparison with the technologies in the market
- 2) Potential Market Uptake and market needs
- 3) Technological Key points and drawbacks

MD	*Membrane Distillation (MD) is a novel process where a difference in partial pressure serves as the driving force, and the presence of a hydrophobic membrane ensures high water quality regardless of feedstock parameters. Hot-side temperatures under 90 °C are suitable; hence this process is ideal for exploiting waste heat or solar thermal resources.*
PV	*Pervaporation (PV), a contraction of permeation and evaporation, can be considered as a selective evaporation process using a membrane. The technology is an alternative for distillation for some industrial sectors.*
GS	*Power-to-Methane (PtM) pathway aims to produce synthetic natural gas (SNG) via the CO_2-methanation reaction. [$CO_2 + 4H_2 \leftrightarrow CH_4 + 2\ H_2O$] CO_2-methanation involves green hydrogen (produced by electrolysis fed with renewable electricity) and a carbon source like biogenic CO_2 (by-product of anaerobic digestion) or atmospheric CO_2 (Direct Air capture).*

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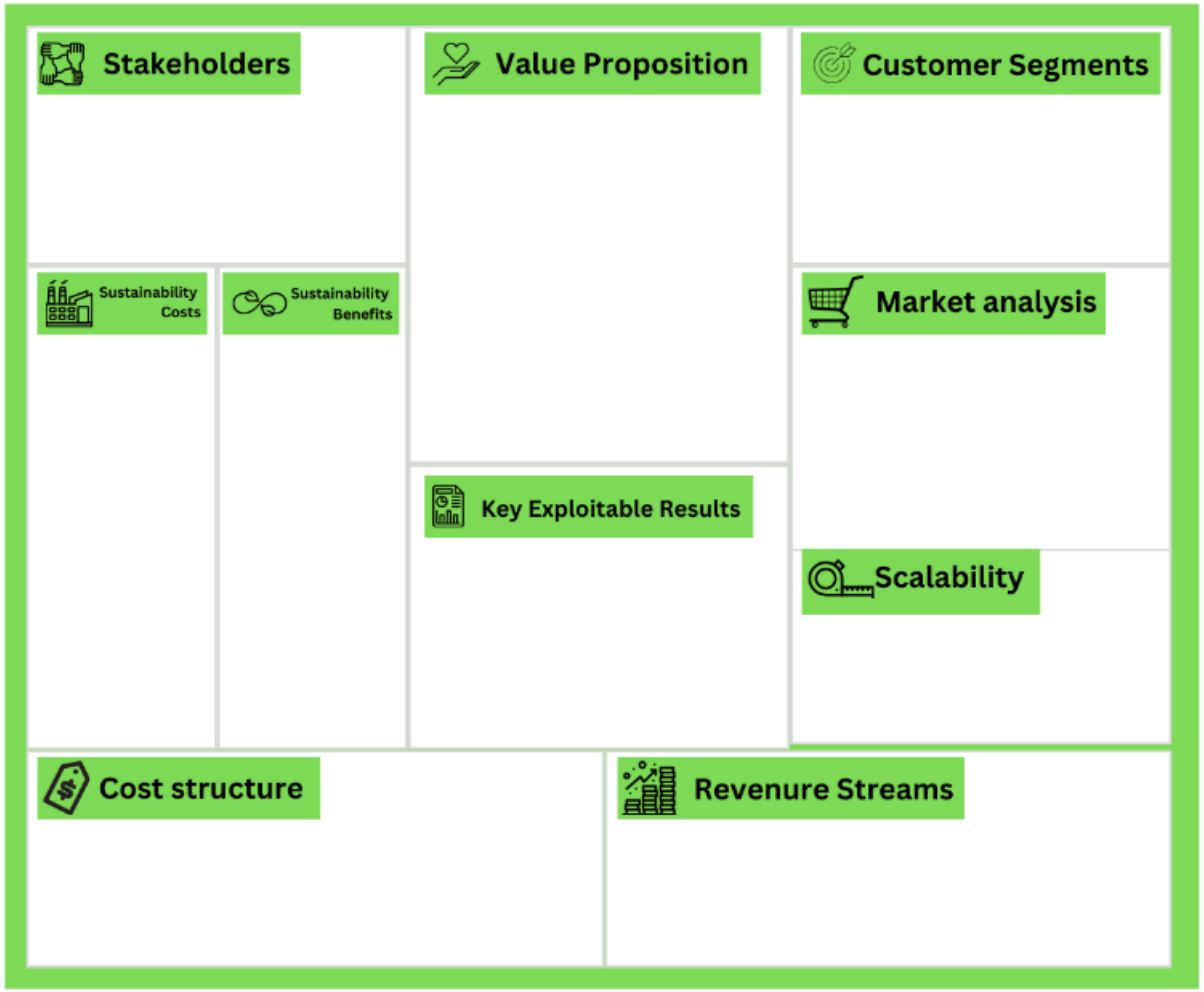
Value Proposition and Business Model



Taking into account the widely used Business Model canvas, an adapted scheme has been structured. In order to identify the key element required for this project, the boxes added have been:

- Stakeholder, substituting the key partners, to include also subject not related to the market.
- Sustainability costs and benefits
- Market analysis and KERs section are essential to include the information provided by Ciaotech and discussed in the previous points
- Scalability, that allows to identify lots of element of the other section and should be included in the value proposition.

Once set up this scheme and interviewd the project partner, three Preliminary Business Model have been set up.

- Membrane Distillation Business Model
- Pervaporation Business Model
- Gas Separation Business Model

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WP3 – Membrane development and Scale up

M1 – M12

CONTACT: YVONNE VAN DELFT

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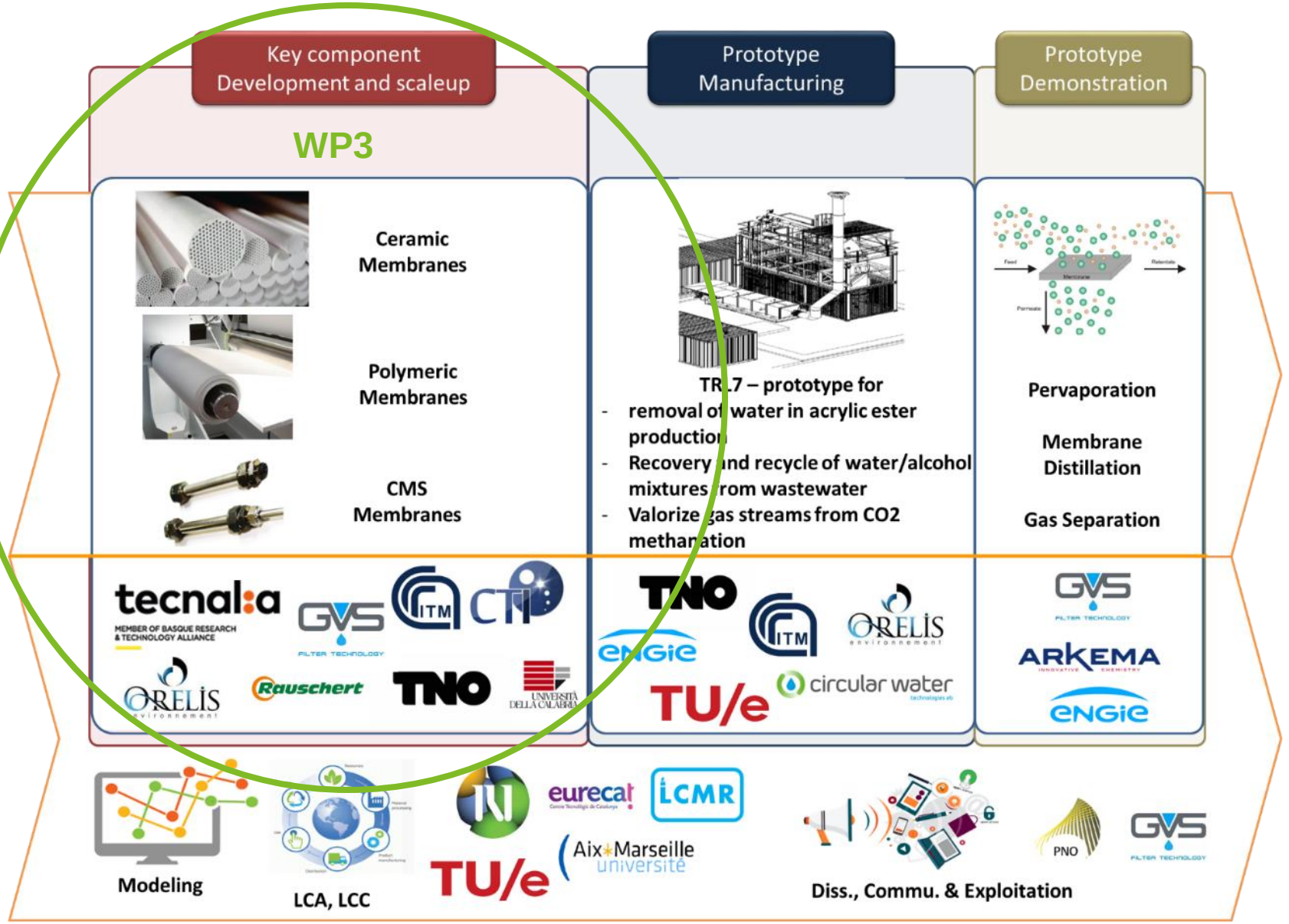

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WP3 objectives:

- Preparation of custom-made new generation of membranes with outstanding separation performance and/or superior properties essential to achieve the goals of the European Green Deal.
- Characterization of the performance of membranes under industrial conditions to enable design of field-tests and validate the models.
- Up-scaling the preparation of membranes (1-1.2 m^2) for pilot demonstration (WP5).

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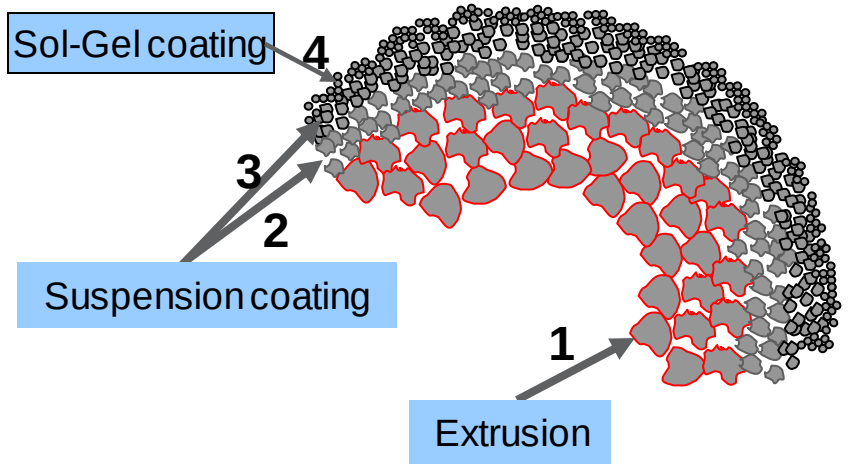
Development of HybSi membranes for pervaporation

- HybSi is a robust ceramic membrane
- High chemical stability in acidic condition
- High thermal stability up to 200°C
- Up-scalable to multi-channel membranes



Lab scale HybSi® membrane and stainless steel module

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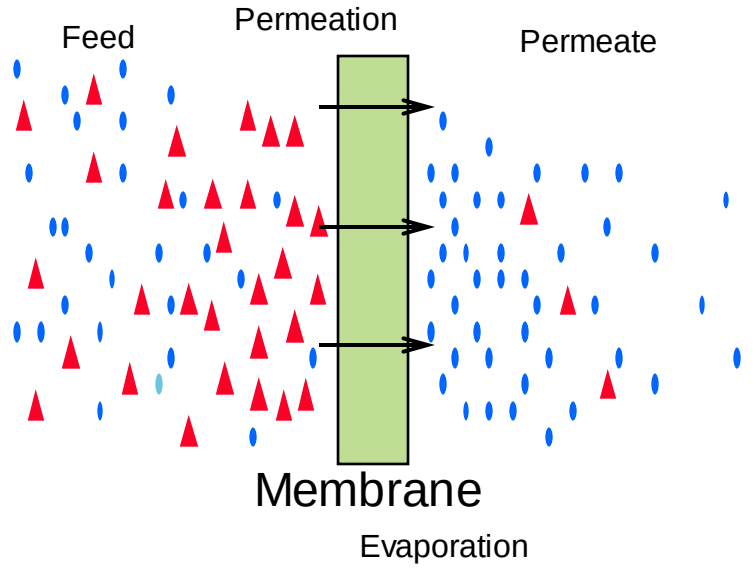
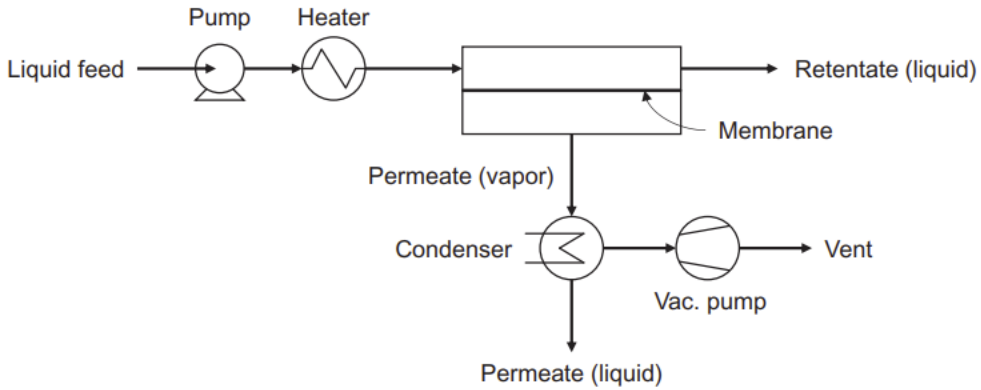
Pervaporation using HybSi membranes

- **Pervaporation = Permeation + Evaporation**
  - selective evaporation of water via membrane
  - more energy efficient than distillation

- Pervaporation temperature: 80°C lower than distillation
- Membrane performance:
  permeate flux> 1 kg/m^{2}h water with >98 wt.% water in permeate
- 30% lower operational cost (OPEX)





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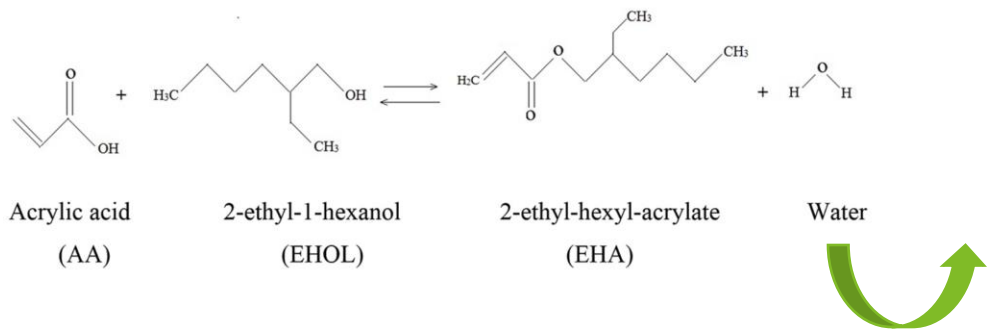
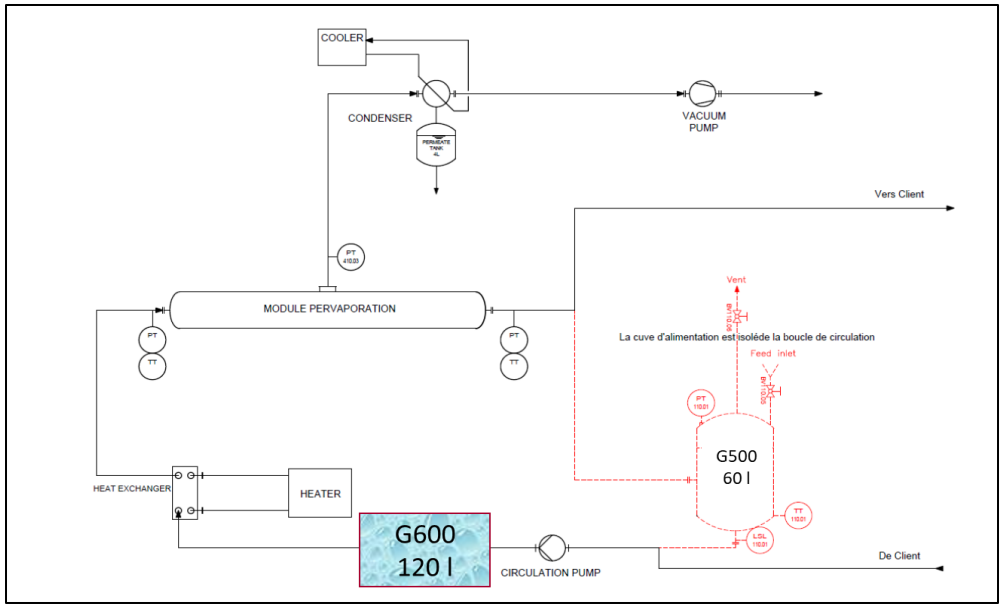
Application of HybSi membranes in esterification reactions

- Esterification reaction for production of 2-ethyl-hexyl-acrylate





- Reaction temperature: 80-100°C,
- Water content: 1-2 wt.% , acrylic acid content: 5-10 wt.%, pH=4
- Boiling point of the mixture = 160°C
- Potential advantages pervaporation:
  - Lower temperature => decrease polymerization and blocking of equipment
  - Faster water removal => decrease undesired byproducts and higher feedstock efficiencies

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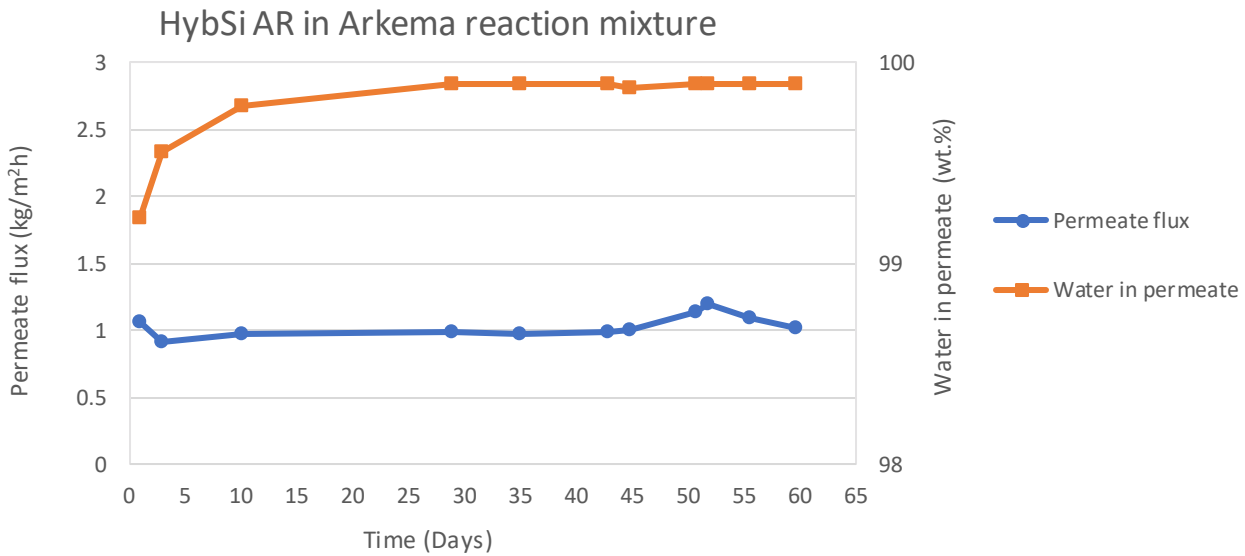
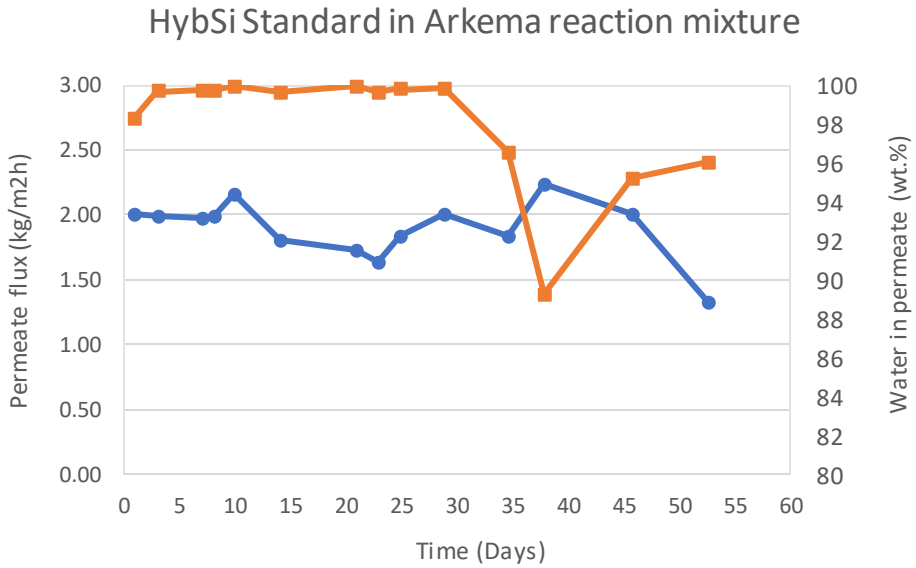
Highlights of HybSi membrane development

- Two versions of HybSi membranes were prepared:
  - HybSi Standard
  - HybSi Acid Resistant (AR) ➡ Stable in the reaction mixture in long term ➡ Will be upscaled



HybSi AR showed a stable performance for 2 months



HybSi Standard lost its selectivity after a month

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Development of PVDF membranes for membrane distillation

Dope solution preparation

PVDF polymer with sugar-based solvent (GS)

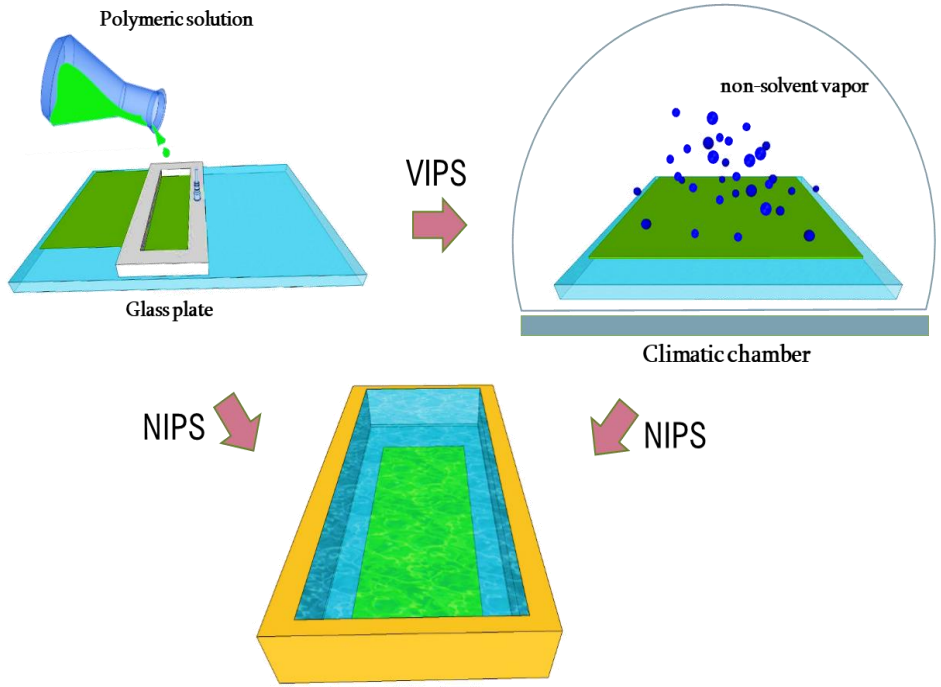
Preparation of PVDF membranes using green solvent => different variables were investigated to optimize the composition of the dope solution and casting conditions

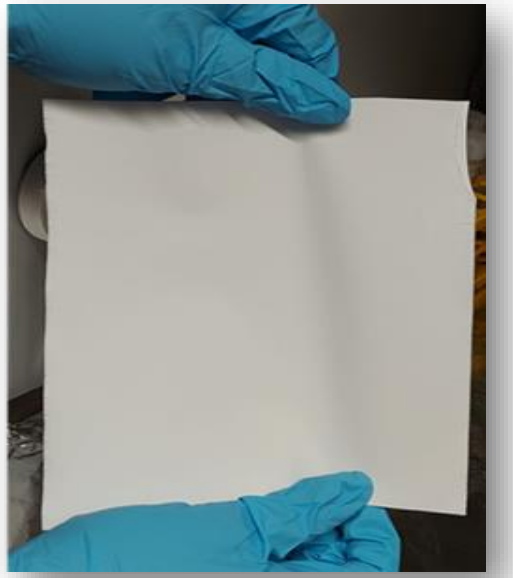
GS is considered as **Green Solvent**

GS is a potential alternative for **Polar Aprotic Solvents (PAS)**

GS is totally **water miscible**

PVDF Membrane preparation*





PVDF Membrane

*F. Russo *et al.*, "Tamisolve® NxG as an Alternative Non-Toxic Solvent for the Preparation of Porous Poly (Vinylidene Fluoride) Membranes," *Polymers (Basel).*, vol. 13, no. 15, p. 2579, 2021.

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Coating preparation

The AFAS coating is designed as a hybrid organic-inorganic layer capable of producing hierarchically organized coatings with advanced water repellency. This hybrid coating consists of an inorganic, porous layer based on ceramic (Al_2O_3) nanoparticles obtained via sol-gel, while the organic layer is composed of fluoroalkylsilane molecules.

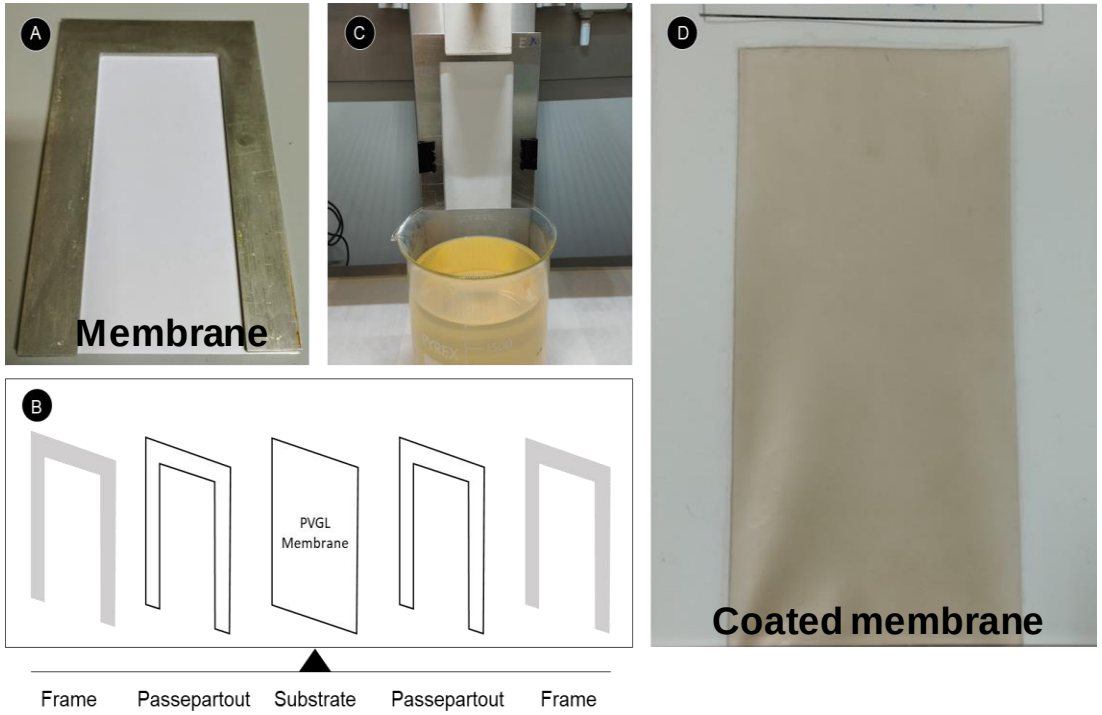
AFAS coating solution was used to cover the **Advanced PVDF Membranes** and **commercial PVDF membrane from GVS Company** for MD with the pore size of 0.2 µm , 0.45 µm and 0.8 µm.

Hybrid coating formulations*

Label	Inorganic Layer	Organic Layer	Coating Typology
AFAS	Al_2O_3	Fluoroalkylsilane	Dual-layer inorganic-organic[1]

[1] two-step deposition: i) one for the inorganic layer and ii) for the organic layer.

Hybrid coating preparation



* M. Raimondo, F. Veronesi, G. Boveri, G. Guarini, A. Motta, and R. Zanoni, "Superhydrophobic properties induced by sol-gel routes on copper surfaces," *Appl. Surf. Sci.*, vol. 422, pp. 1022–1029, 2017, doi: 10.1016/j.apsusc.2017.05.257.

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Membrane Characterization

Thickness of the PVDF membranes: ~ 120 µm
Thickness of the GVS membranes : ~ 160 µm

Porosity

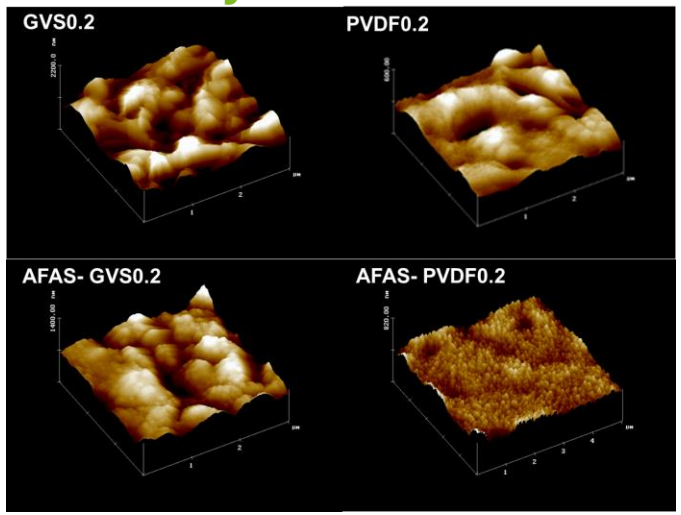
Before coating

PVDF membranes: 80-87 % **AFAS-PVDF membranes:** 36-79 %
GVS membranes : 63-65 % **AFAS-GVS membranes :** 50-64 %

Water Contact angle of PVDF membranes:
~ 100 °
Water Contact angle of GVS membranes :
~ 130 °

The presence of coatings resulted in a significant enhancement of the hydrophobicity (contact angle around **170° with the AFAS coating**), together with an improvement of the repellency of the industrial **"Rinsing" wastewater** provided by GVS (γ=32 mN/m), for which the AFAS coating showed a contact angle of **about 140°** .

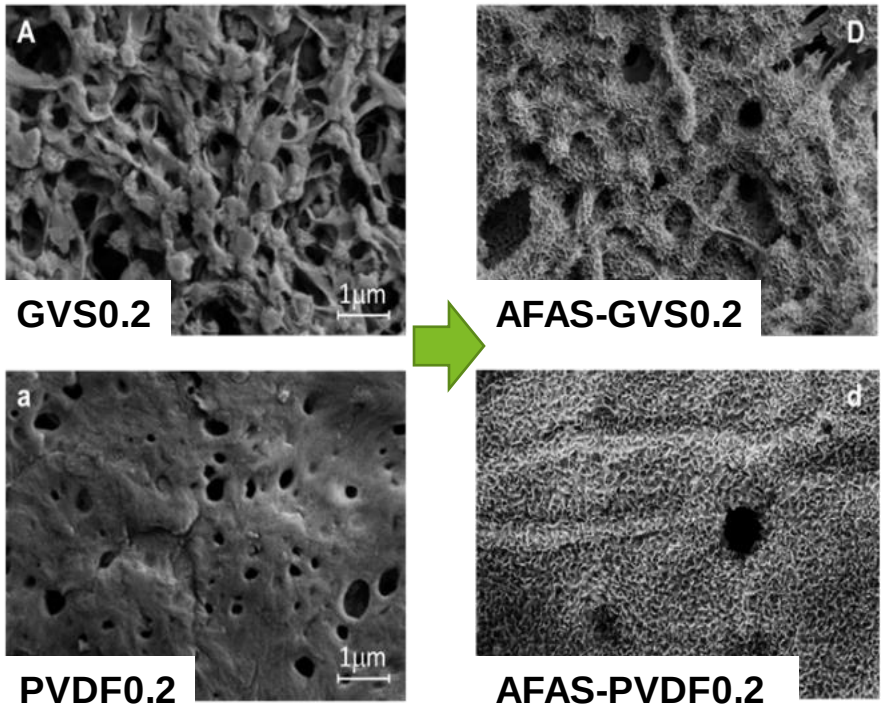
AFM analysis



The AFAS-GVS membrane showed a grainy surface, but less regular than the AFAS-PVDF.

SEM analysis of TOP SURFACE

Before coating **After coating**



The alumina nanoparticles of the AFAS coating leaded to the formation of the "flower-like" structure with a thickness of few nanometers.

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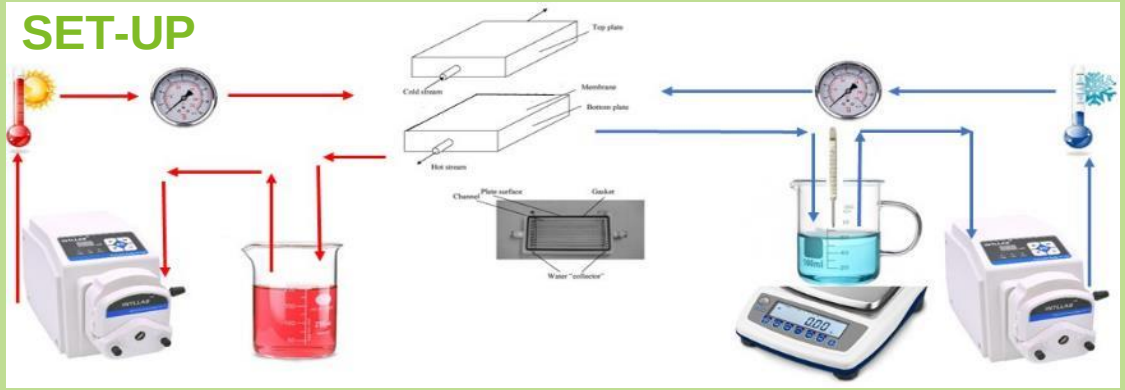
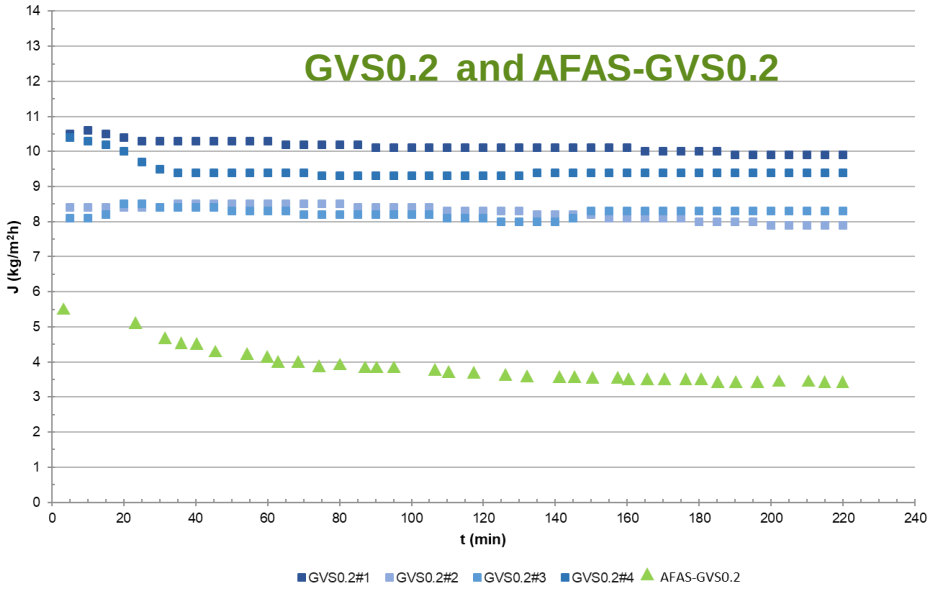
Direct contact membrane distillation (DCMD)

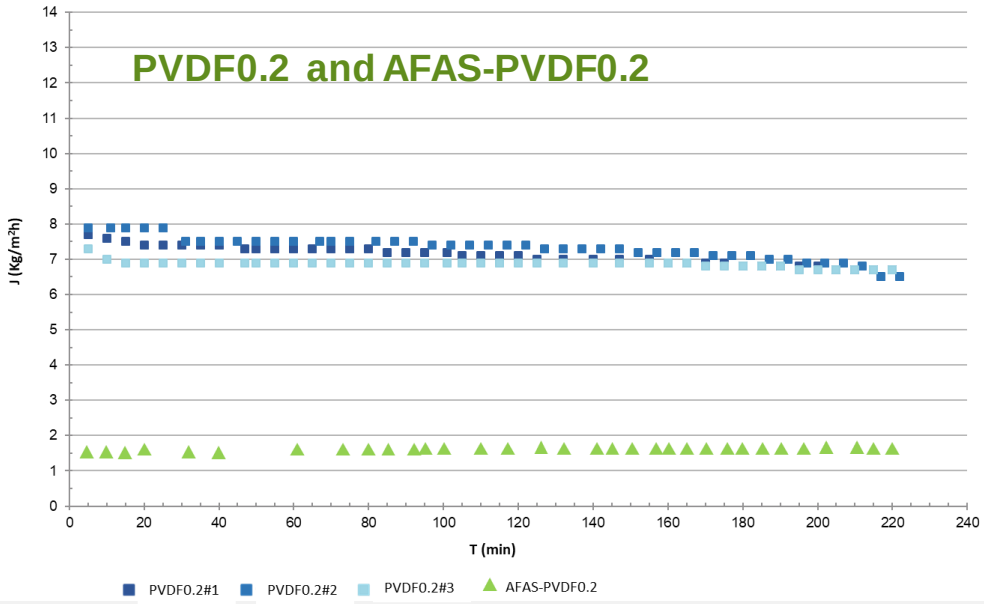
SET-UP



T_{feed}=60°C; Q_{feed}=66 L/h; T_{dist}=20°C; Q_{dist}=42 L/h

Permeate fluxes for 0.2 μm GVS and PVDF membranes





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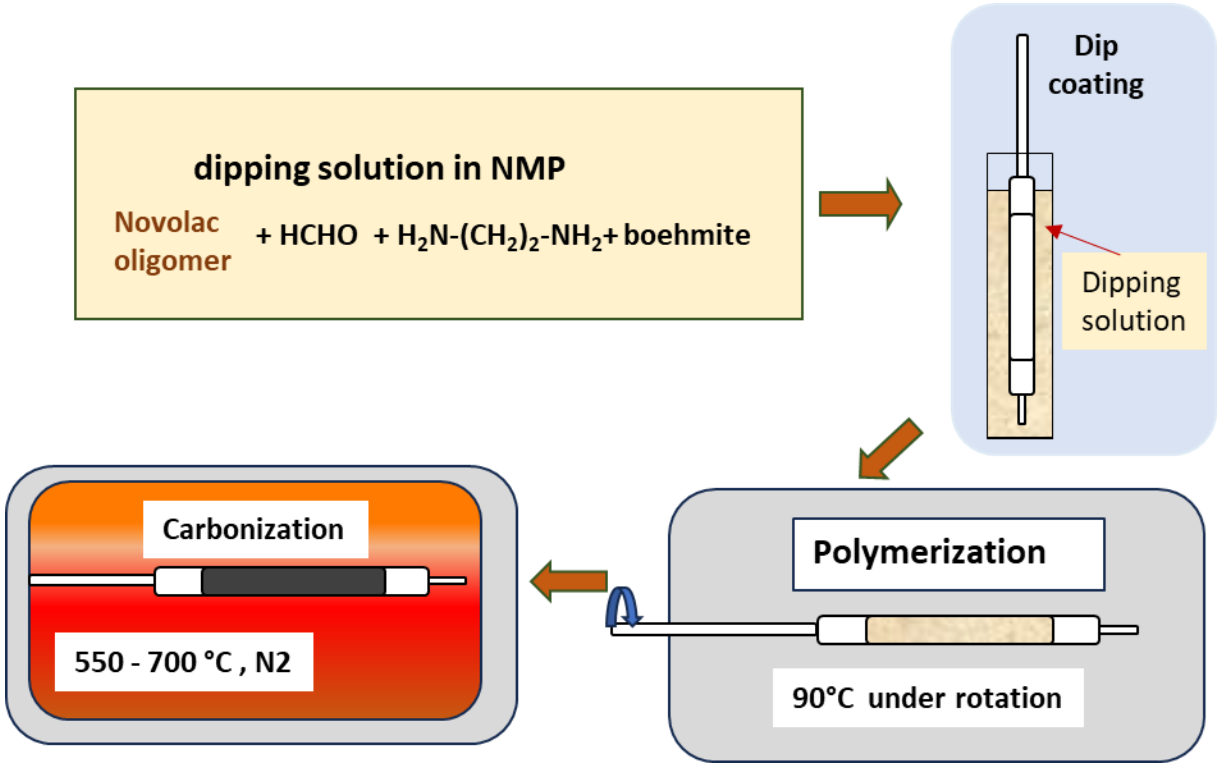
Development of CMS membranes for gas separation

Preparation of the carbon molecular sieve membrane (CMSM)



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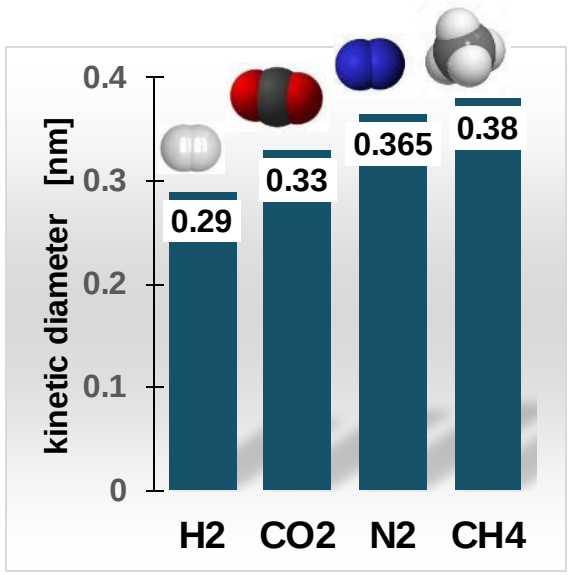
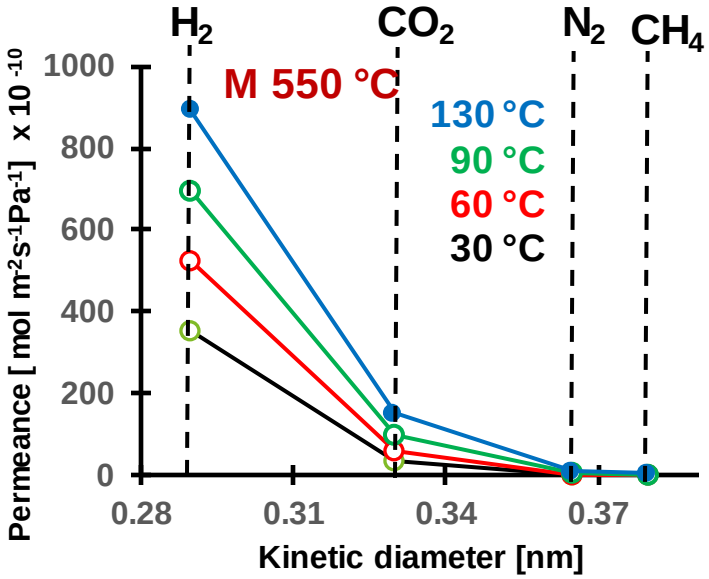
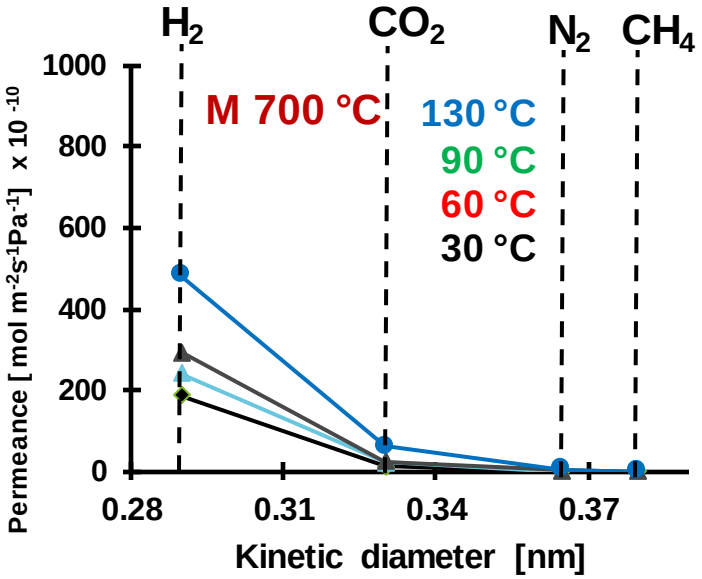
CMSM performance







Gas permeance at different temperatures for different kinetic diameters of the gases

=> Higher temperature give smaller pores and a more hydrophobic membrane.

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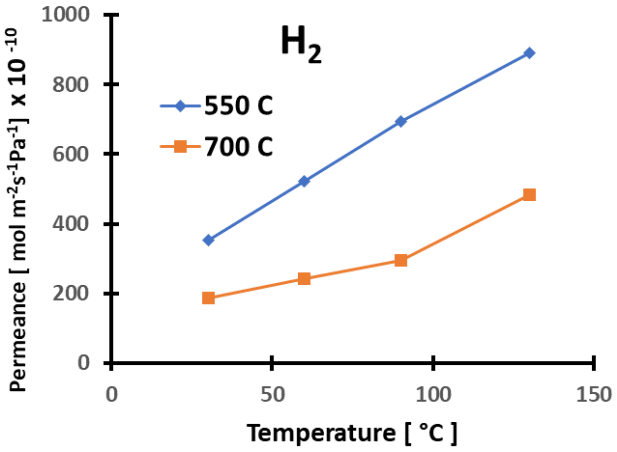
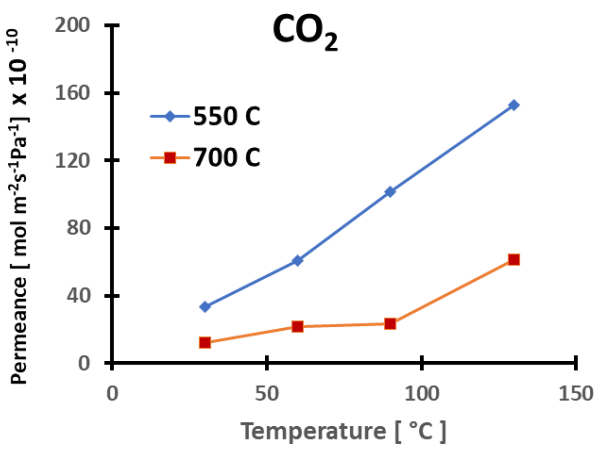
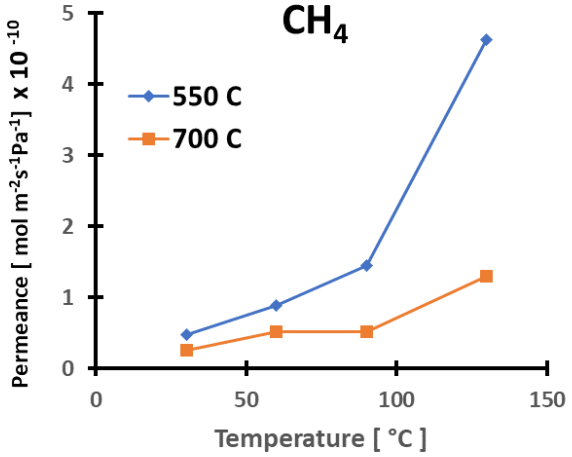
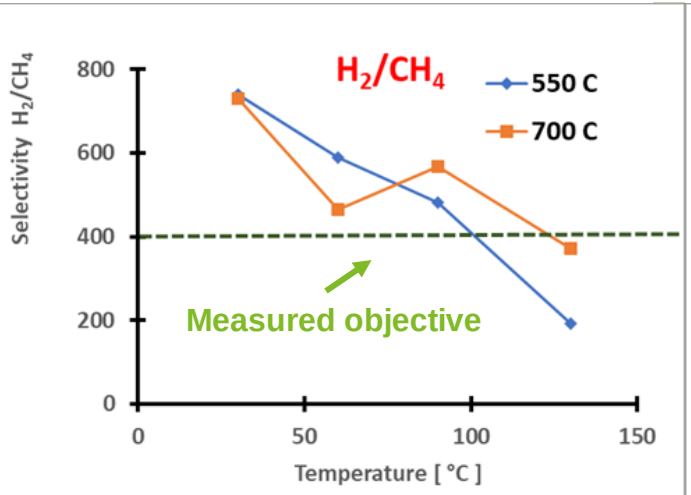
CMSM performance

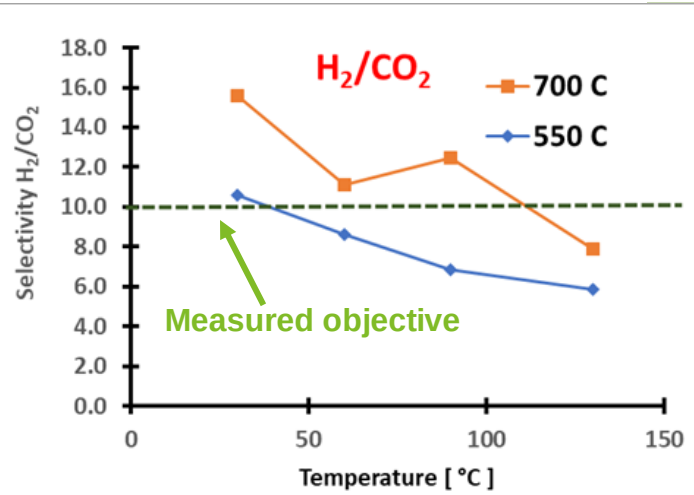
Gas permeance at different permeation temperatures

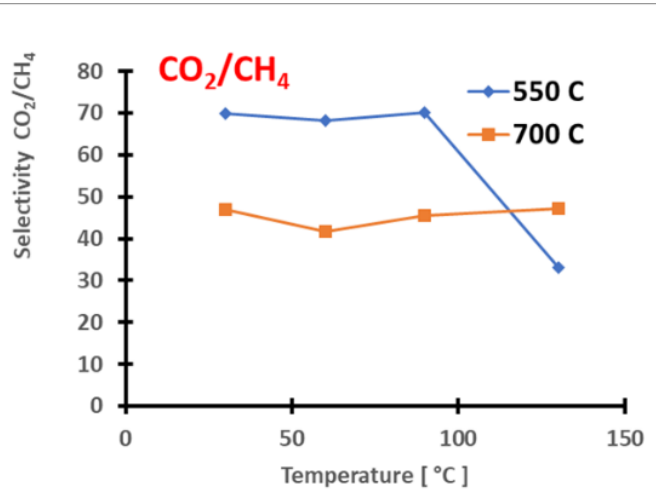












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CMSM performance

Permeance at ambient temperature

	Carb*	Permeance					Selectivity	
		mol m^{-2} s^{-1} Pa^{-1} x 10^{-10}				GPU		
	°C	H_2	CO_2	N_2	CH_4	H_2	H_2/CH_4	H_2/CO_2
MEA-1	500	215		0.7	0.36	61	650	
MEA-2	550	611		2.37		176		
MEA-3	550	437	52.3	1.1	0.56	126	794	8.4
MEA-4	550	754	102	2.6	0.97	217	775	7.4
MEA-5	700	187	12	0.3	0.26	54	730	47
MEA-6	550	354	33	1.3	0.48	102	740	70

*Carbonization temperature

MEASURED objective: H_2 permeance 320 GPU

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WP4 – e Engineering and Construction

M1 – M12

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Gas Separation

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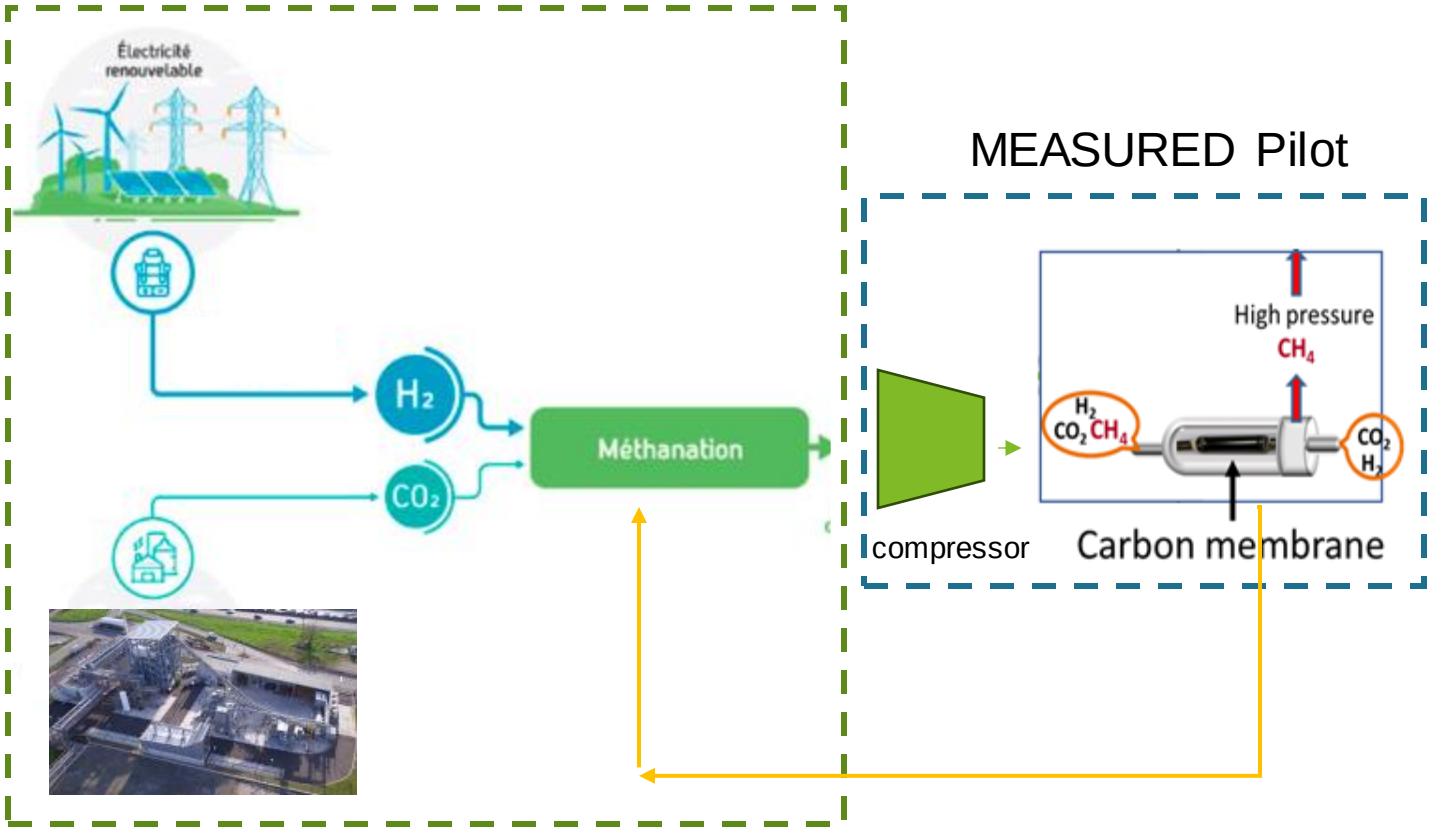
Gas Separation pilot principle



Gaya R&D Platform - Engie

At the inlet of the Gas Separator ; a mixture of H2/CO2/CH4

Objectives of the « Gas Separation » pilot: develop a **one-step process of SNG purification from CO_2 methanation process**

➔ Obtain at the outlet a gas compatible with the natural gas network specifications

Operating conditions for the Gas Separation pilot:

- Temperature: around 40-50°C
- Pressure : around 10-15 bar to allow separation through the membrane

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Gas Separation Pilot will be adapted from an existing pilot from Engie

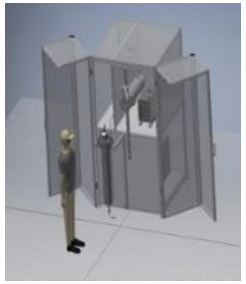
Engie Lab CRIGEN currently operates a pilot working with Pd membrane for H2/NG separation.

After the H2/NG gas separation tests at Engie Lab CRIGEN, the pilot will be adapted for MEASURED project

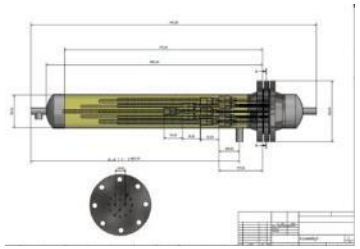
Innovative CMSM (Carbon Molecular Sieve Membrane) will be developed and scaled-up in the framework of MEASURED project by TU/e and Tecnalia and will be integrated into the pilot in replacement of the current Pd membranes.

The demonstration will be based on already developed alumina-CMSM and patented aluminum single atom-CMSM by Tecnalia and TU/e for gas separation.

The pilot will be designed to accommodate modules with 1,2 m² of membrane



H2/NG pilot at Engie Lab CRIGEN / H2 Factory



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Pervaporation

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Pervaporation pilot

Development of a pervaporation pilot by Orelia that will be connected with an existing Arkema synthesis pilot using **Advanced ceramic hybrid silica membrane (HybSi)** materials for acrylic ester production to be used as a raw material in paint, adhesives, acrylic rubbers, and acrylic fibers.

Pervaporation membranes will replace distillation equipment:

➔ Lower CAPEX: - »0% compared to current cost

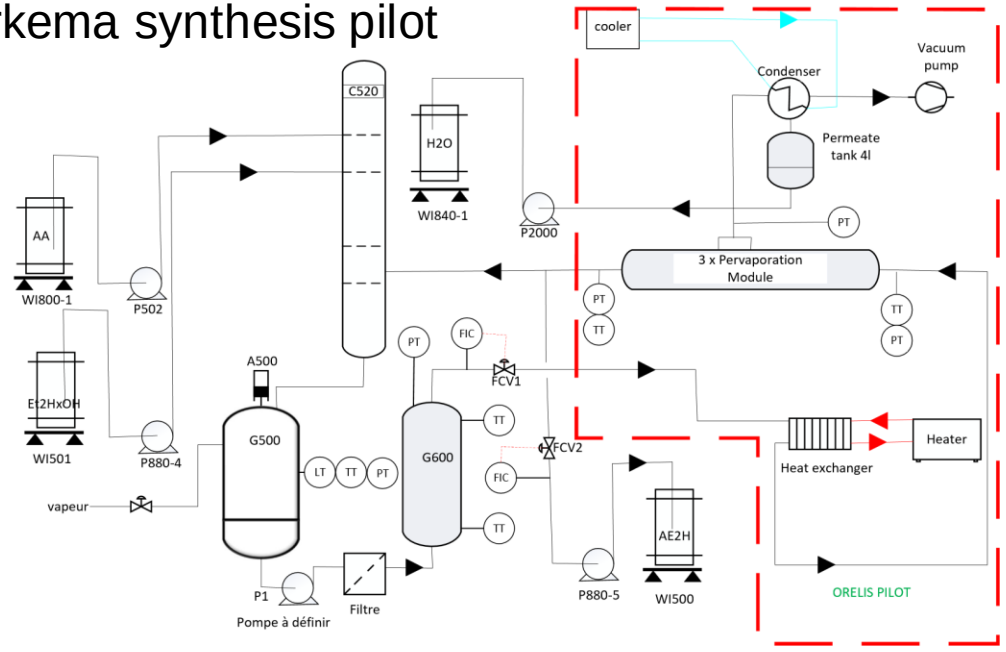
➔ Energy saving up to 50%

➔ Better quality of the final product

Operating conditions :

- Temperature: 80 – 100 °C
- Pressure: 2 bar
- %water in feed: from 1 to 0,4%.

Arkema synthesis pilot



Orelis pervaporation pilot

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Membrane Distillation

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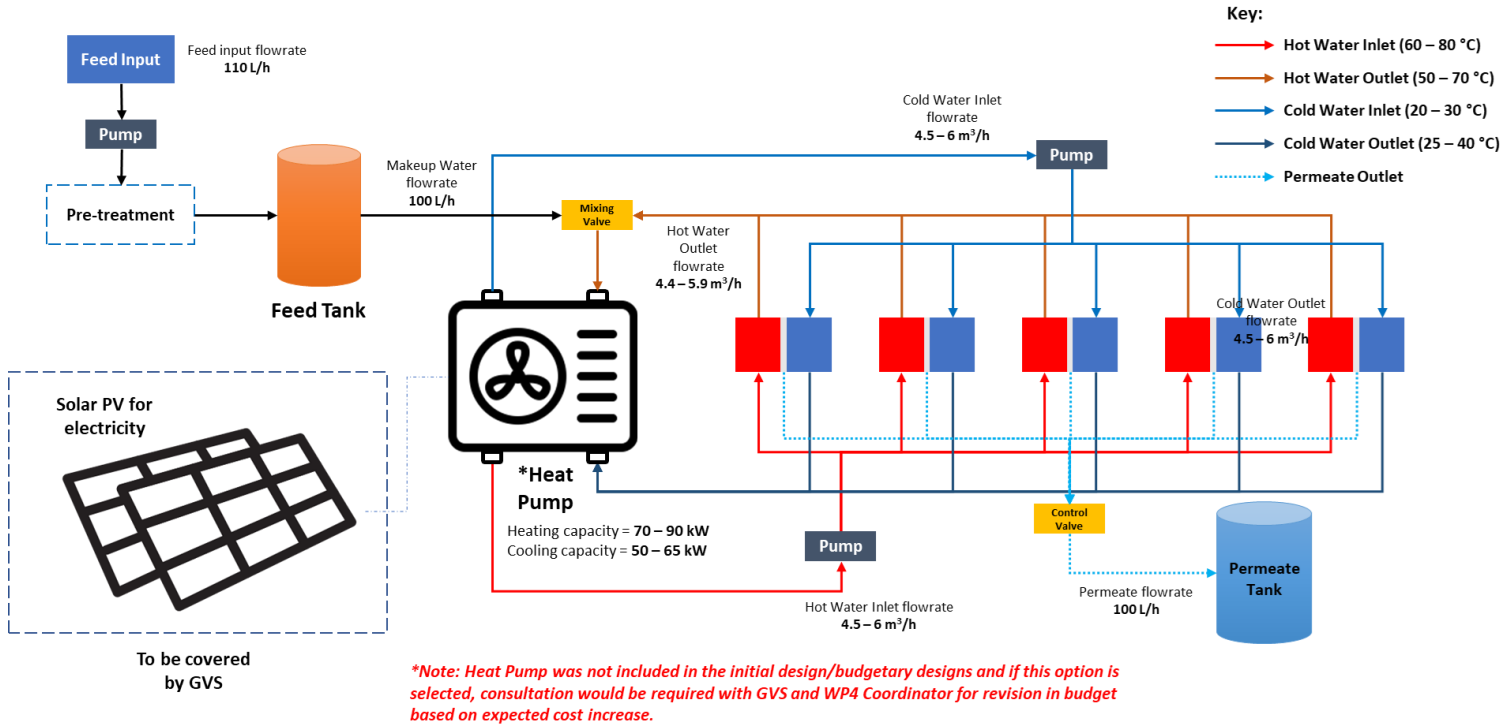
Membrane Distillation

Application : Membrane Distillation (MD) treatment of wastewater discharged from phase inversion membrane manufacturing plant.

This pilot will demonstrate innovative PVDF membranes showing
➔ higher chemical resistance (> 10%),
➔ 25% reduction of water footprint
➔ permeability of reused MD for Microfiltration > 500 L/m^{2}h·bar

Membrane Distillation pilot flowsheet



*Note: Heat Pump was not included in the initial design/budgetary designs and if this option is selected, consultation would be required with GVS and WP4 Coordinator for revision in budget based on expected cost increase.

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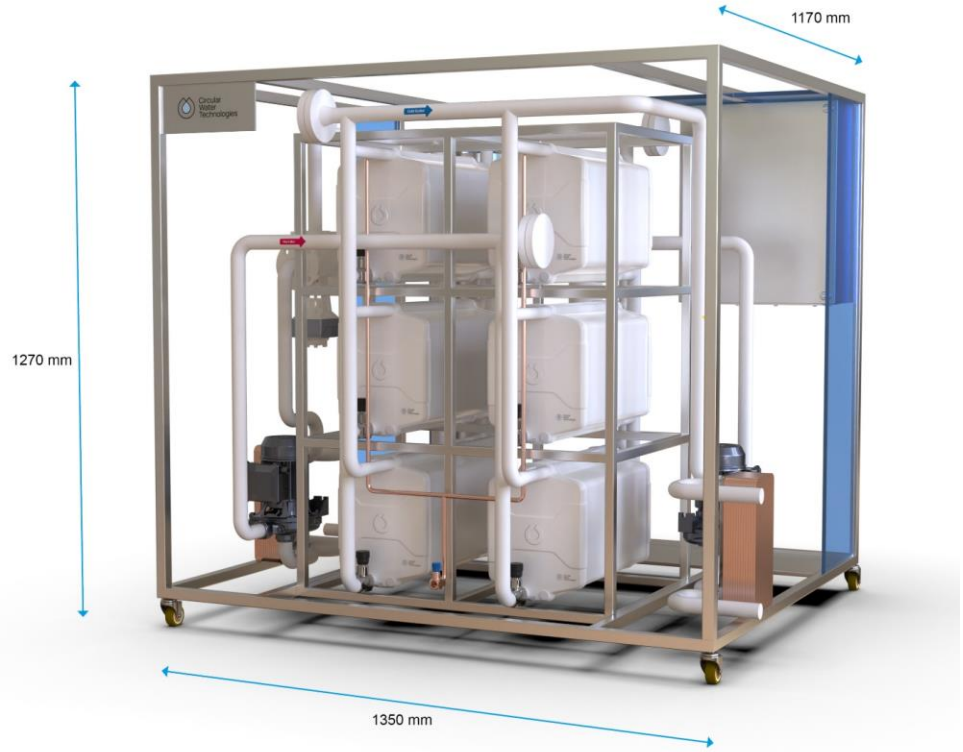
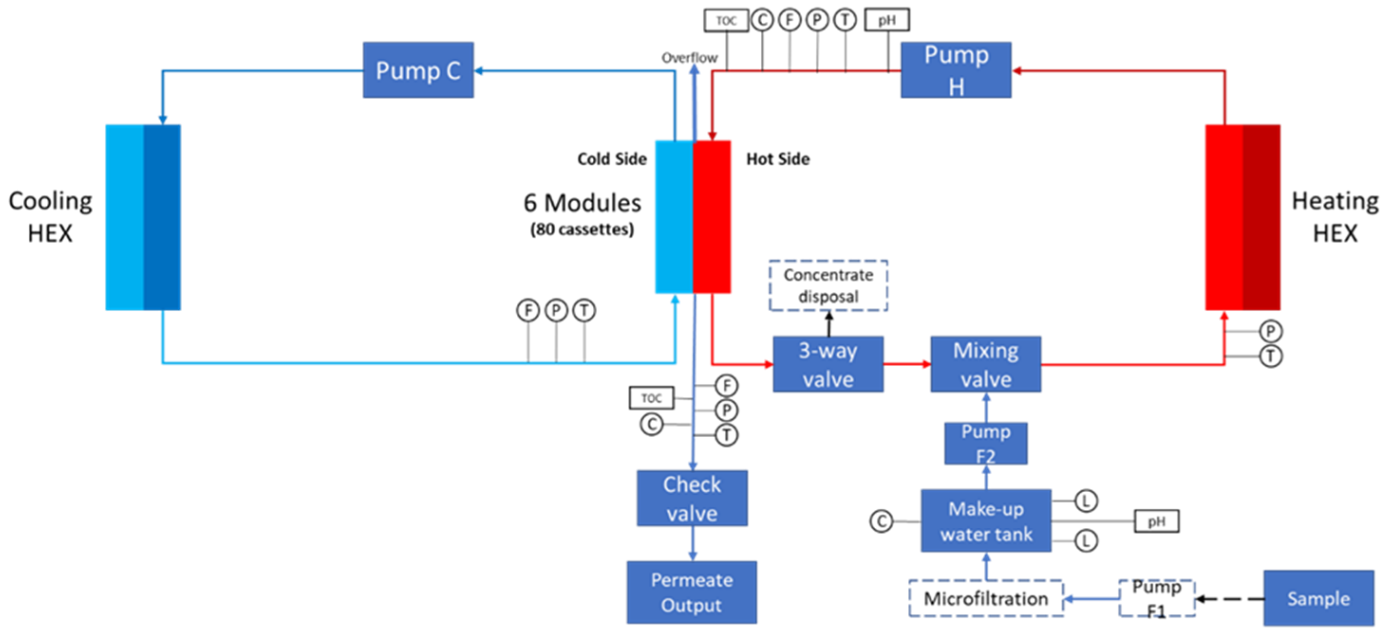
Membrane Distillation

3D Scheme



Flowsheet



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WP5 – Technology demonstration

M1 – M12

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Gas Separation

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Experimental: GAYA Platform, a cutting-edge demonstration site for the GS Pilot

- ✓ A **semi-industrial environment available for start-ups** to test, de-risk and validate the performance of their innovative solutions on real gas
  - *Raw bioSNG treatment solution*
  - *Syngas cleaning technologies…*
- ✓ An **experimental platform available for future research projects**
  - *Waste gasification for instance*
  - *Production of H_2 or CH_4*
- ✓ **An analytical expertise center** dedicated to green gases (tests on new sensors, validation of analyzers, on site experimental campaign…)
- ✓ **A training and expertise center** to support industrialization at bigger scale.

- More than 3,000 sensors
- Highly automatized with a DCS
- With flexible processes and, by design, modular on purification and on upgrading parts

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Experimental: GAYA Platform, a cutting-edge demonstration site for the GS Pilot

- The GS pilot will be tested at the GAYA facility operated by ENGIE. Part of the off-gas of the methanation unit will be sent to the membrane module
- Catalytic methanation reactor
- Technology: Isothermal fluidized bed
- Total Capacity: 500 kW_{LHV} / 50 m3(n)/h SNG
- Modifications to connect the GS unit
- *Objective = in one step obtain: a) retentate = SNG (methane) at Natural Gas Grid quality for injection. b) permeate = upgraded stream that can be recycled to the inlet of the methanation reactor enhancing conversion efficiency*

GAYA
R&D demonstration plant
- St Fons/Lyon – France

Methanation reactor installed at the GAYA platform

ENGIE will test the GS Pilot in GAYA platform, working on CO_2-methanation mode during at least 100 continuous hours

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Pervaporation

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Experimental: Arkema Pilot, a cutting-edge demonstration site for the Acrylic Process Development

- ✓ A **semi-industrial environment available for start-ups** to test, de-risk and validate the performance of their innovative solutions on new process
- ✓ An **experimental platform available for future research projects**
  - *New routes for Acrylic Acid and Esters synthesis*
  - *Process Technology Evaluation*
- ✓ **An analytical expertise to support Pilot Activities**
- ✓ **A deep collaboration** with the Acrylic Research and Process Center located on the same industrial site
- ✓ **An Open Pilot facility for other Partners within Arkema or for other Companies**.

- Highly automatized with a DCS
- With flexible processes and, by design, modular on purification and on upgrading parts

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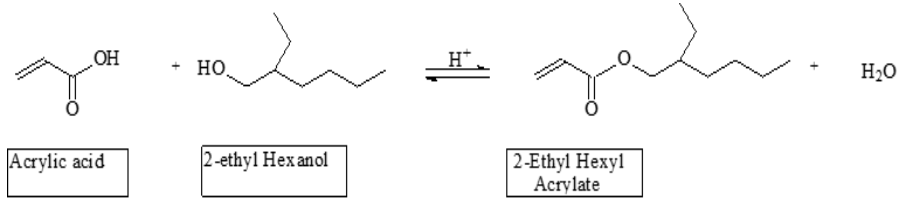
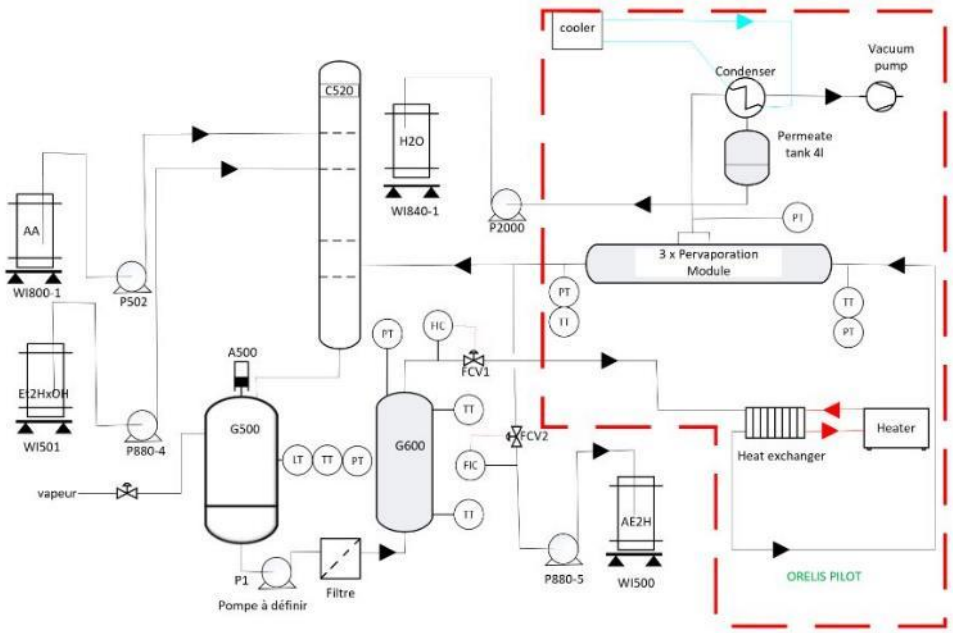
Experimental: Arkema Pilot, a cutting-edge demonstration site for the Acrylic Process Development

- The Pervaporation skid will be tested at the Arkema Facility in connection with a fixed bed reactor working on an esterification Process
- Heterogeneous Catalytic reactor
- Technology: Fixed bed
- Pervaporation Membrane Area : 1-3 m^2
- *Objective = Obtain high yield of ester by the shift of the equilibrium state thanks to water withdrawal through the pervaporation membrane*





ARKEMA will test the Pervaporation Pilot , working on Esterification Process

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Membrane Distillation

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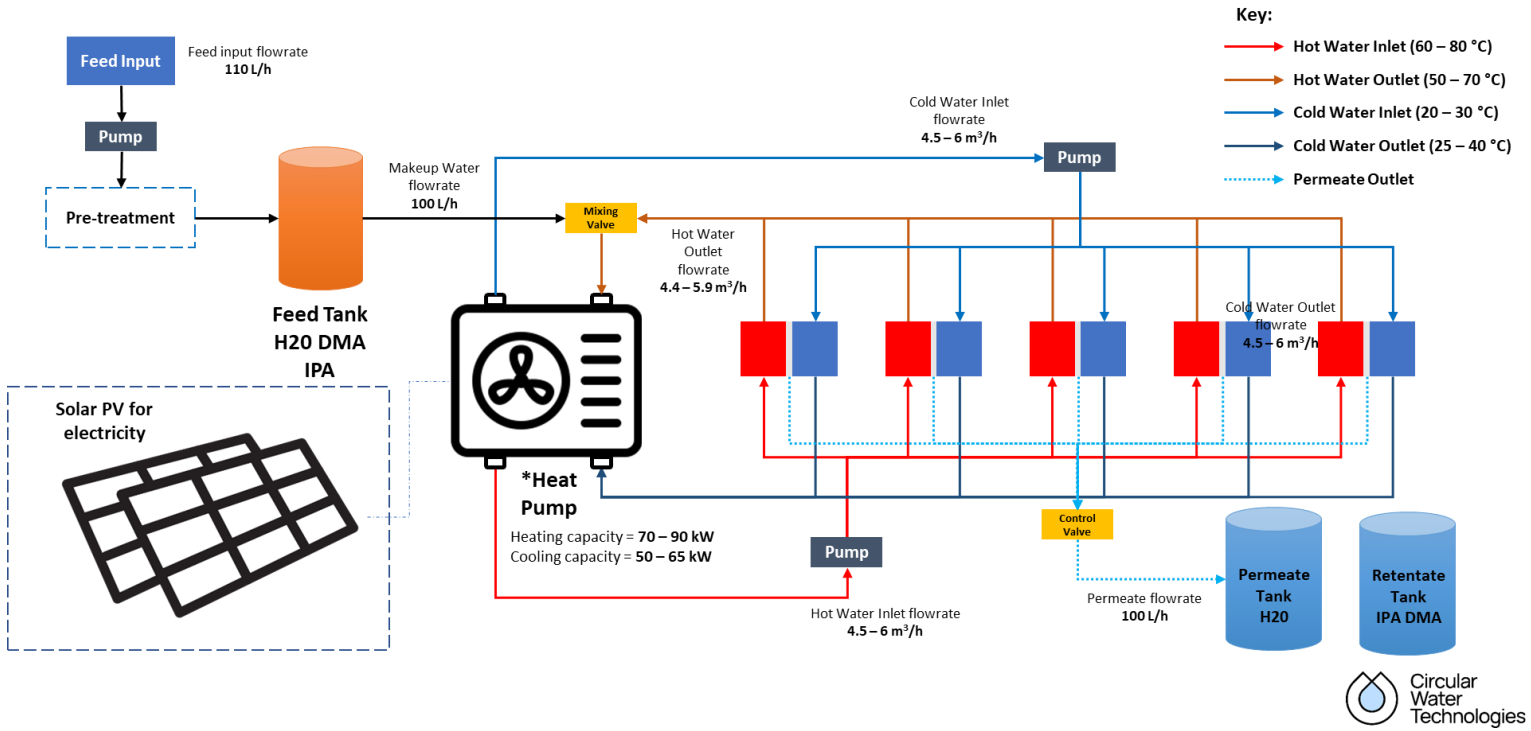
Experimental: GVS Pilot, a cutting-edge demonstration site for the wastewater treatment from polymeric membrane manufacturing process

- ✓ A **semi-industrial environment available for start-ups** to test, de-risk and validate the performance of their innovative solutions on new process
- ✓ An **Experimental Platform** to test the **feasibility of Membrane Distillation** process for a new industrial sector.
- ✓ Membrane production process needs **high-boiling solvent** that need **to be recovered**
- ✓ **A deep collaboration** with CWT and Italian National Research Council (CNR located on the same industrial site



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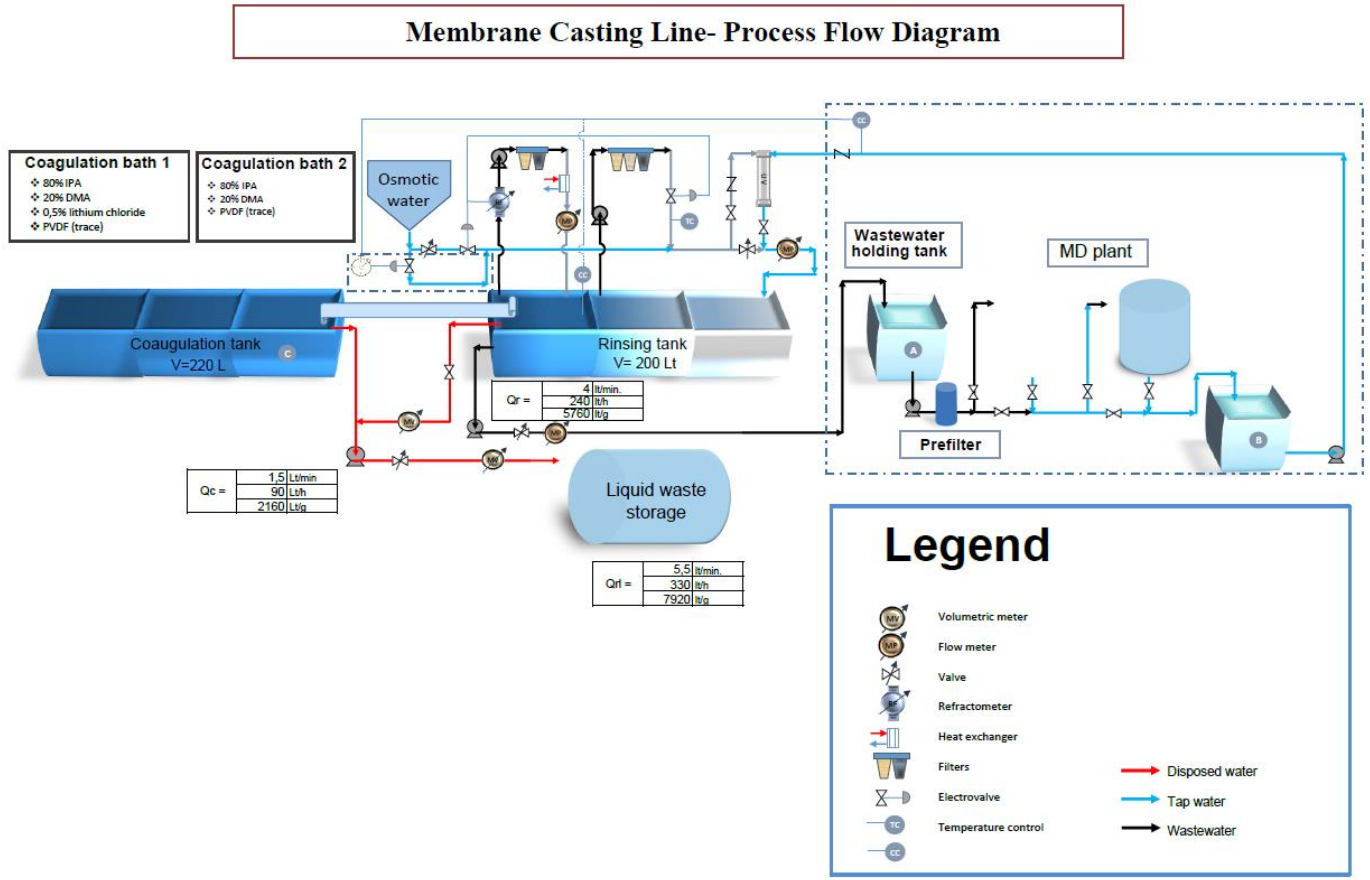
Experimental: GVS Pilot, a cutting-edge demonstration site for the wastewater treatment from polymeric membrane manufacturing process

- The Membrane Distillation (MD) skid will be tested at the GVS SpA Facility downstream to polymeric membrane casting line
- Solar Panel (105 Kw) and Heat Pump will be used for the energetic demand of the MD plant
- MD Membrane modules consists of PVDF membrane produced with a green formulation
- *Objective = To recover the high-boiling solvent (DMAc) and Isopropyl alcohol from the wastewater effluents*



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WP6 – Modelling

M1 – M12

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WP6 - MODELLING

MEASURED

European Commission

WP1 EXPLOITATION AND BUSINESS PLAN (**GVS**)

WP2 COORDINATION (**TUE**)

INNOMEM

MACBETH
Membranes And Catalysts Beyond Economic and Technological Hurdles

WP3 MEMBRANES DEVELOPMENT AND SCALEUP (**TNO**)

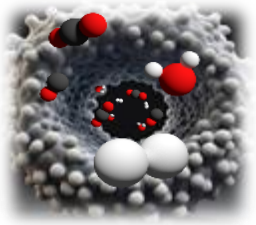
WP4 ENGINEERING AND CONSTRUCTION (**ENGIE**)

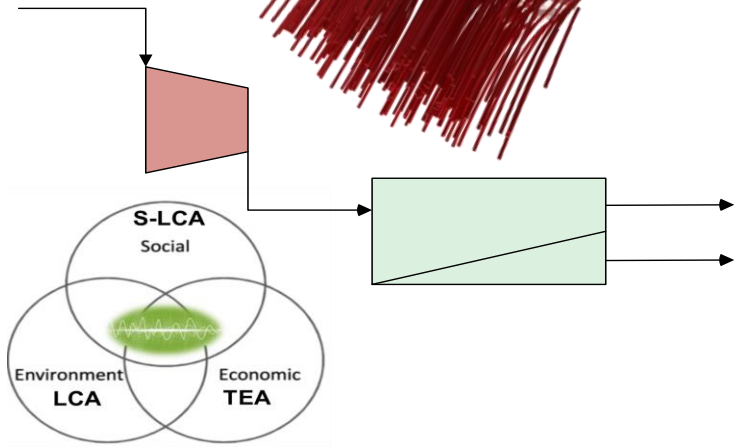
WP5 TECHNOLOGY DEMONSTRATION (**ARK**)

WP6 MODELLING (**MODELTA**)

WP7 DISSEMINATION AND COMMUNICATION (**PNO**)

multi scale modelling





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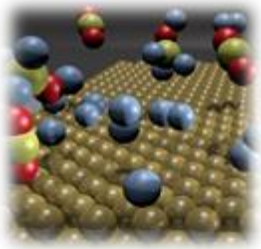
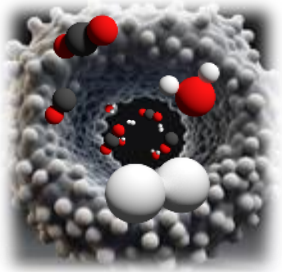
multi-scale modelling



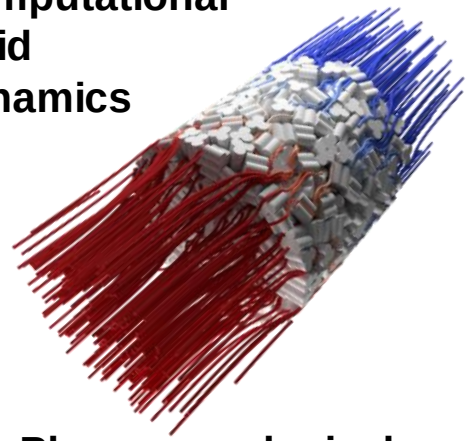
Density Functional Theory

Molecular Dynamics

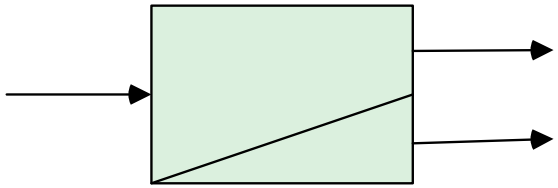


Atomic & molecular scale

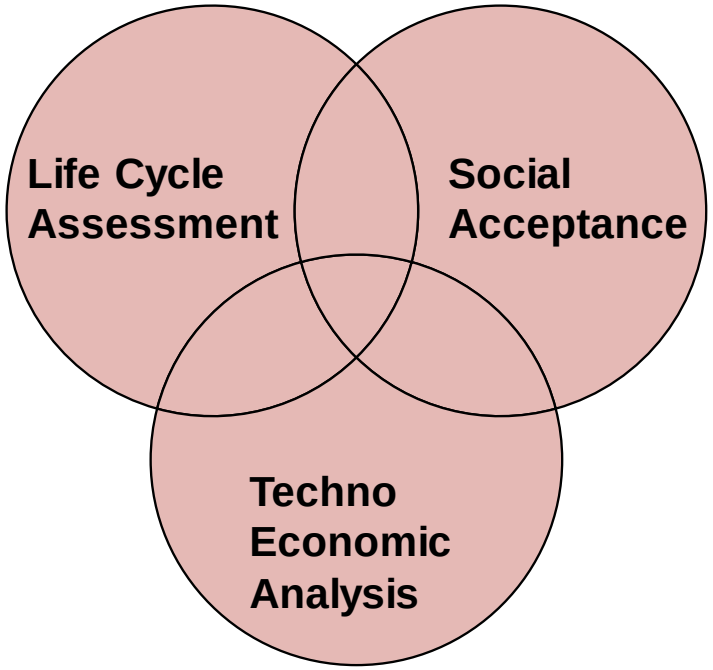
Computational Fluid Dynamics



Phenomenological



Module scale



Process scale

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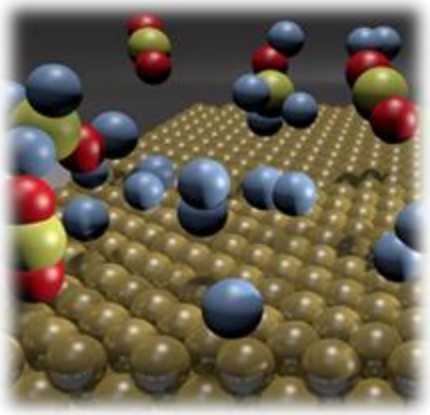
Atomic/molecular scale & module scale

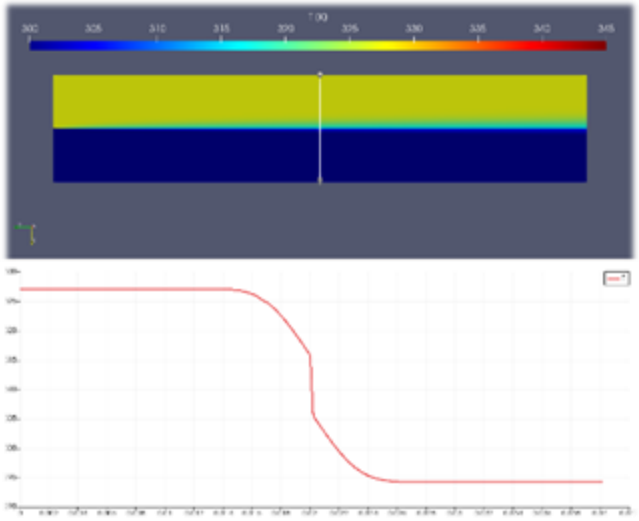
DFT & MD

Analysis at atomic and molecular scale to **define permeation mechanism** and **material properties**, to optimize membrane pore structure and coating composition.



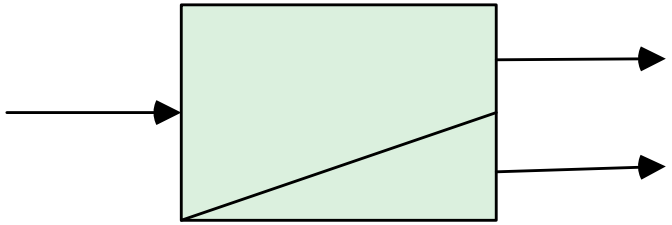
CFD

Computational Fluid Dynamics is performed to **optimize module design** and to study relevant phenomena as **concentration and temperature polarization**.



Phenomenological Model

Phenomenological models are simple 1D model that are based on **material and energy balances** and where the permeation is estimated through a defined flux expression. They are **computationally simple** and include the relevant phenomena underlined from CFD.



$$J^K = K_0 \frac{\bar{v}}{RT} \frac{(P_f - P_p)}{\delta}$$

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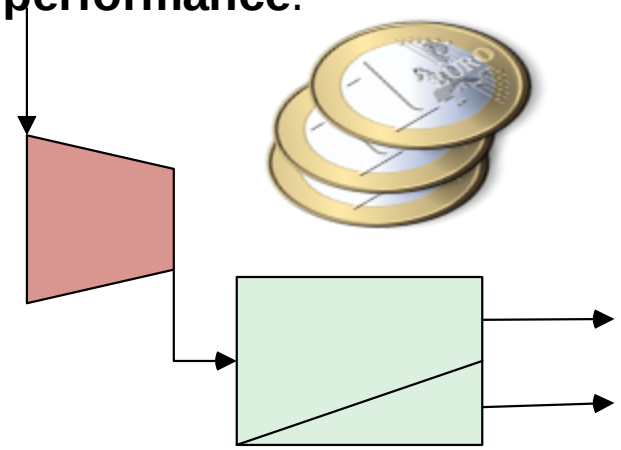
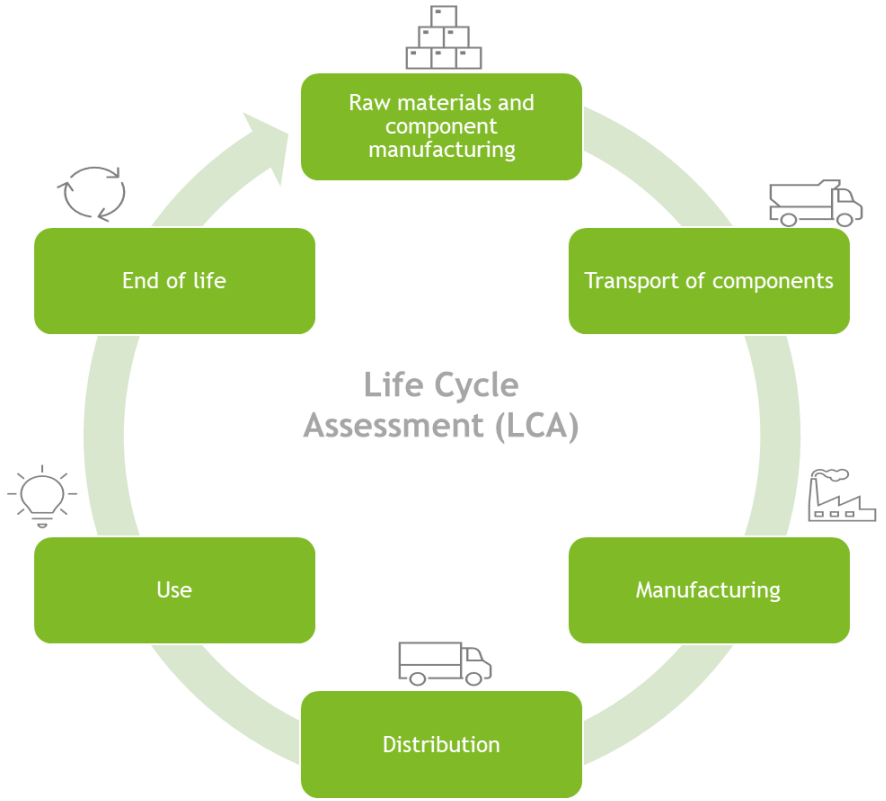
Process scale

Techno Economic Analysis

Techno-economic analysis allows the **optimization of the process** through the **evaluation of the costs and performance**.



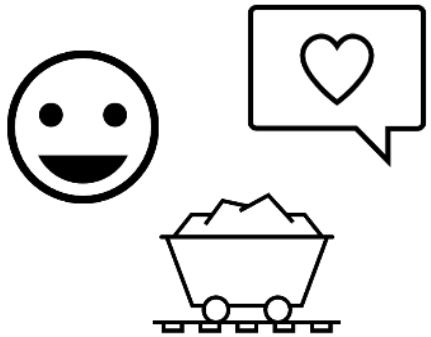
Life Cycle Assessment



LCA allows the **environmental impacts** of products associated with a product or activity to be assessed through **all stages of the life cycle**.

Social Acceptance

Social acceptance is mostly related with the definition of the **critical raw materials**, with proposal to for their reduction and substitution.



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WP7 – Dissemination & Communication

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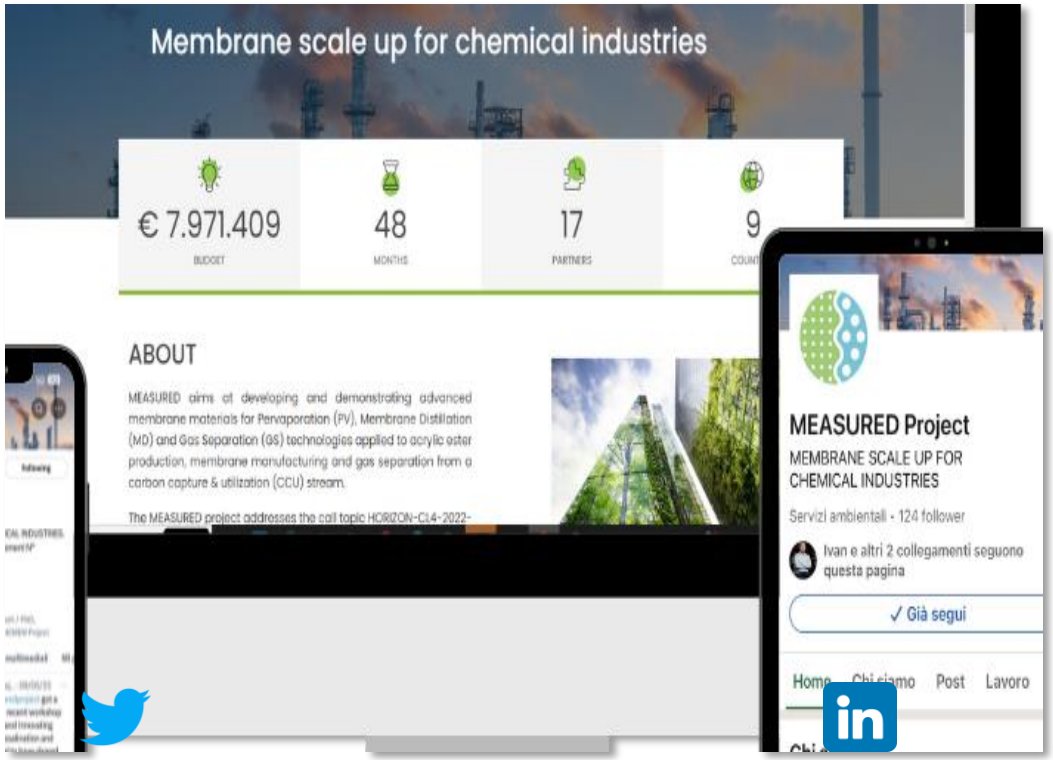

Visit the MEASURED website and
follow the LinkedIn and Twitter account to be always updated!

https://www.measured-project.eu/

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Want to discover more about MEASURED?

Read the MEASURED newsletters to learn more about the project activities!

☞ **NEWSLETTER ISSUE #1**
To learn more about the project aims and goals and the partners involved in the initiative.

☞ **NEWSLETTER ISSUE #2**
To discover the results achieved in the first year of project implementation.

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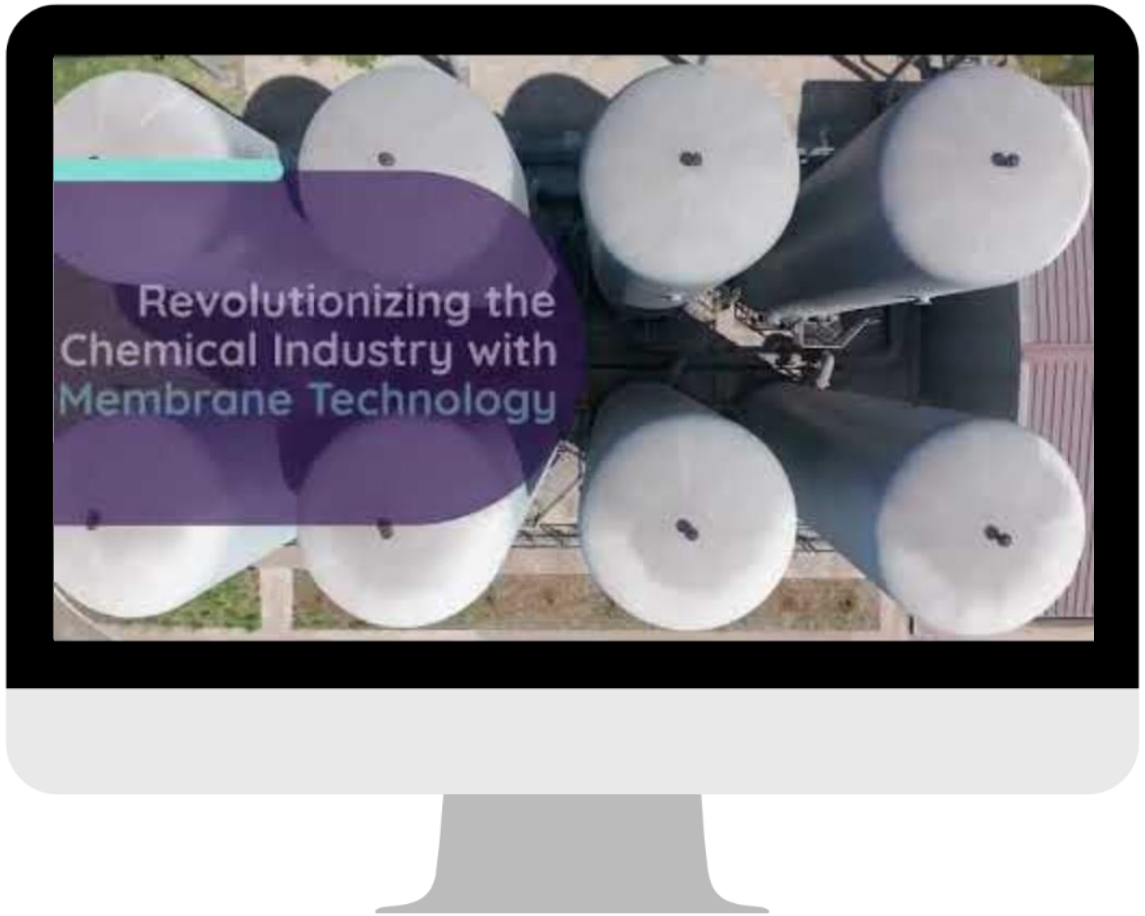
Want to discover more about MEASURED?



Watch the MEASURED project video

to delve deeper into the project's primary goals, aspirations, and the consortium partners who are actively engaged.

Available on:

https://www.youtube.com/watch?v=GQtUz-Ny0LI

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Consortium

MEASURED

TU/e EINDHOVEN UNIVERSITY OF TECHNOLOGY

tecnalia MEMBER OF BASQUE RESEARCH & TECHNOLOGY ALLIANCE

TNO innovation for life

NATIONAL INSTITUTE OF CHEMISTRY

UNIVERSITÀ DELLA CALABRIA DIPARTIMENTO DI INGEGNERIA DELL'AMBIENTE DIAM

Rauschert

Consiglio Nazionale delle Ricerche

GVS

ENGIE

ARKEMA

ORELIS environnement

ALSYS Group

CiaoTech

PNO

MODELTA

CTI

ALSYS Group

Aix*Marseille université Sociolement engagée

eurecat

Circular Water Technologies

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