MEMBER

ADVANCED **MEMB**RANES AND MEMBRANE ASSISTED PROC**E**SSES FOR PRE- AND POST-COMBUSTION CO_2 CAPTU**R**E

H2020 GRANT AGREEMENT NUMBER: 760944

Start date of project: 01/01/2018 **Duration: 4 years**

WP08 - Dissemination and communication

Workshop on "Advanced Membranes and Membrane assisted processes for pre- and post-combustion CO_2 capture" Booklet

Topic: NMBP-20-2017: High-performance materials for optimizing carbon dioxide capture
Funding scheme: Innovation action t
Call identifier: H2020-NMBP-2016-2017

Due date of deliverable:	Actual submission date:	Reference period:
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v11	30-06-2022	Final version	TECNALIA	J.L. Viviente

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Dissemination Level		
PU	Public	X
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	
CON	Confidential, only for members of the Consortium	

(*) for generating such code please refer to the Quality Management Plan, also to be included in the header of the following pages

(**) indicate the acronym of the partner that prepared the document

Content

1. EXECUTIVE SUMMARY

1.1. Description of the deliverable content and purpose

The present document includes the presentation of the finql[t] workshop organised by the project MEMBER. The workshop on “Advanced Membranes and Membrane assisted processes for pre- and post-combustion CO2 capture” was hosted by IFE on June 23^{th}, 2022. The agenda is shown in the figure hereafter.

Workshop on Advanced Membranes and Membrane assisted processes for pre- and post combustion CO_2 capture

Kunnskapsbyen Conference Center, June 23^{rd}, 2022

(Gunnar Randers Vei 24, 2007 Kjeller, Norway)

Link to attend the meeting online: Click here to join the meeting

Agenda

9:30 – 9:50	Introduction to the MEMBER project **José Luis Viviente (TECNALIA)**
9:50 – 10:10	Developing sustainable and economic scale-up routes for metal organic frameworks **Adam Deacon (JM)**
10:10 – 10:30	Mixed Matrix Membranes production scaling -up. **William Marechal (POLYMEM)**
10:30 – 10:50	Pre- and Post-combustion CO_2 capture with MMM systems **Hans ten Dam (HYGEAR)**
10:50 – 11:10	Pd-based membranes production **José Luis Viviente (TECNALIA)**
11:10 – 11:25	**Coffee break** **WE WILL START AT 11h45**
11:45 – 12:05	High temperature sorbent and catalyst for the MA-SER process - Upscaling and performance **Julien Meyer (IFE)**
12:05 – 12:25	Sorption Enhanced Reforming **Arnstein Norheim (ZEG-POWER)**
12:25 – 12:45	MA-SER reactor for H_2 production with CO_2 capture **Luca di Felice (TU/e)**
12:45 – 13:05	Market analysis and techno-economic assessment of MA-SER system **Vittoria Cosentino (KT)**
13:05 – 13:25	Environmental Life Cycle Assessment and Life Cycle Costing of the MEMBER systems **Alexander Borsch (QUANTIS)**
13:25 – 13:30	Final remarks and closure **Jose Luis Viviente**

Figure 1. Agenda of the final public workshop organised by MEMBER.

2. Presentations

2.1. Introduction to the MEMBER project (José Luis Viviente – TECNALIA)

Advanced MEMBranes and membrane assisted procEsses for pre- and post- combustion CO_2 capture (MEMBER)

https://member-co2.com/

Speaker: joseluis.viviente@tecnalia.com

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 760944

Duration: 4.5 years. *Starting date: 01 January 2018*

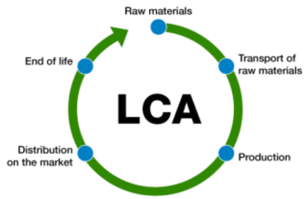
Budget: € 9 596 541,50 *EU contribution: €7 918 901*

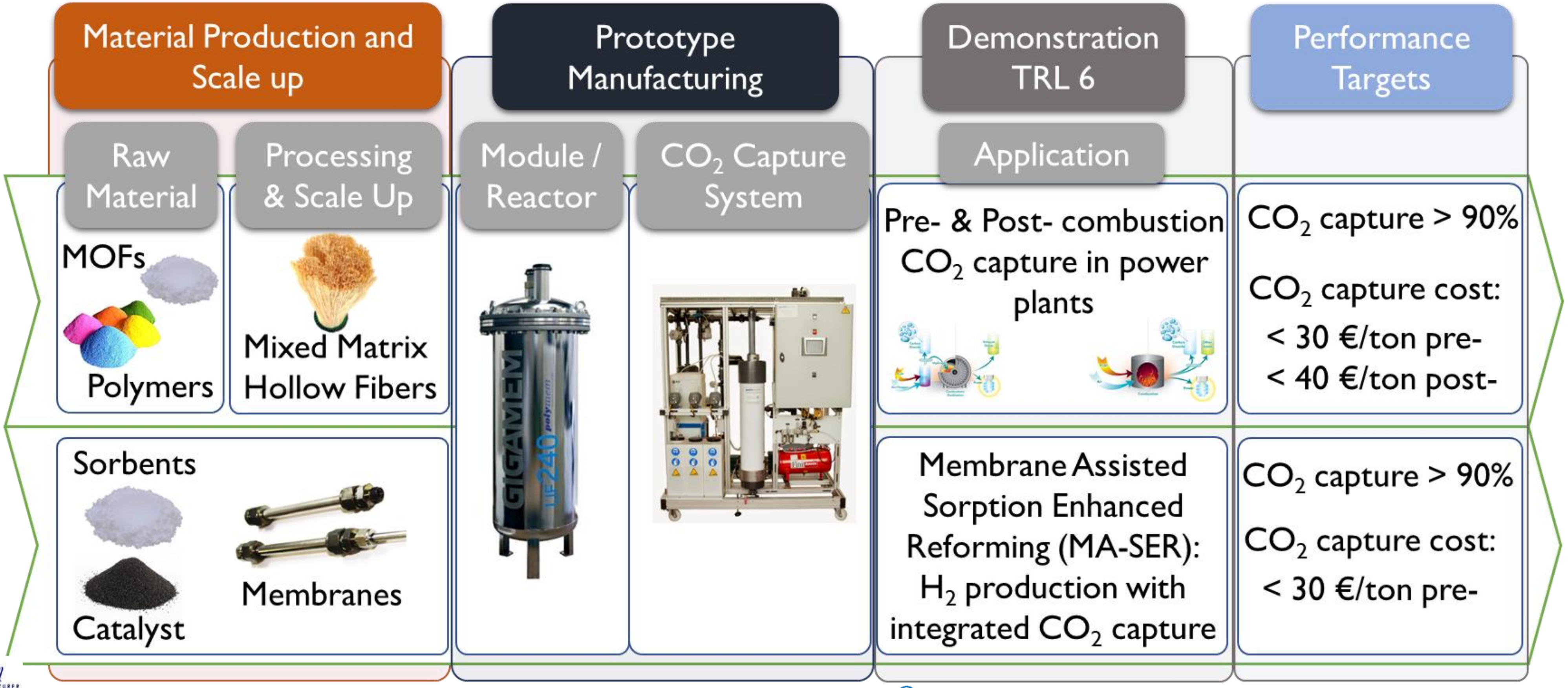
Outline

1. General approach
2. Industrial upscaling
3. Demonstration
4. Expected impacts

1. General approach

MEMBER project aims to reduce the cost of the Carbon Dioxide capture technologies by scaling-up and manufacturing advance materials (membranes, catalysts and sorbents) to develop membrane-based technologies that outperform current technology for pre- and post-combustion CO_2 capture in power plants as well as H_2 generation with integrated CO_2 capture.





tecnalia · TU/e Eindhoven University of Technology · TU Delft · IFE Institutt for energiteknikk · Universidad Zaragoza · CENER · Marion Technologies · C&CS catalysts & chemical specialties · galp · polymem · HYGEAR · ECO Recycling · ZEG Power · Quantis · KT Kinetics Technology · ARKEMA · Johnson Matthey

2. Industrial upscaling

Scale-up the manufacturing processes of materials and membranes for the CO_2 capture prototypes

Prototype A	Prototype B	Prototype C
Pre-combustion CO_2 capture	Post-combustion CO_2 capture	Pure H_2 production with integrated CO_2 capture
MMM hollow fiber membranes	MMM hollow fiber membranes	Pd-based membranes

Recycling of critical membrane materials and membrane modules

2. Industrial upscaling

Increase the manufacturing development stage (from MRL 4-5 to MRL 6) a portfolio of materials & membranes

MMMs for Pre- and Post combustion CO_2 capture

Material / product	Starting TRL/MRL	Targeted / Achieved TRL / MRL	Industrial scale production for cost estimation
MOF: ZIF-8 & ZIF-94	MRL4: 100 gr scale	MRL6 :1 kg each MOF for prototype	10-20 tonnes
Polymers	Commercial		
MMMs for Pre- and Post-combustion CO_2 capture	Up to 100 HF(1 m long) Membrane area: 0.1 m^2	> 10,000 HF (1 m long); Membrane area: 10 m^2	Industrial process production

Pre-combustion CO_2 capture

Post-combustion CO_2 capture

2. Industrial upscaling

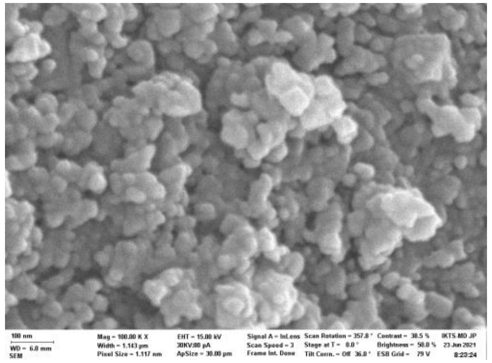
Increase the manufacturing development stage (from MRL 4-5 to MRL 6) a portfolio of materials & membranes

MA-SER: Pure H_2 production with integrated CO_2 capture

Material / product	Starting TRL/MRL	Targeted / Achieved TRL / MRL	Industrial scale production for cost estimation
Sorbent	MRL4: < 1 kg/day	MRL6: 50 kg/day (250 kg for prototype)	500 kg/day
Catalyst	MRL4: gr scale	MRL6: 5 kg per batch (78 kg for prototype)	2 tonnes per day
Pd-based membranes	Single membrane per batch Membrane area: ~ 5 m^2	8 membranes (50 cm long); Membrane area: >55 m^2	Semi-industrial process production

Sorbent

Catalyst



Pd-based membranes

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3. Demonstration

Targets

Prototype A

Pre-combustion capture in power plants using MMMs at HYGEAR reforming equipment.

CCR	Capture Cost
> 90%	**< 30 €/ton**

Prototype B

Post-combustion capture in power plants using MMMs at the 8.8 MW CHP facilities of Agroger (GALP, Portugal).

CCR	Capture Cost
> 90%	**< 40 €/ton**

Prototype C

Pure hydrogen production with integrated CO_2 capture using MA-SER at the IFE-HyNor (Norway) under the supervision of ZEG POWER.

CCR	Capture Cost
> 90%	**< 30 €/ton**

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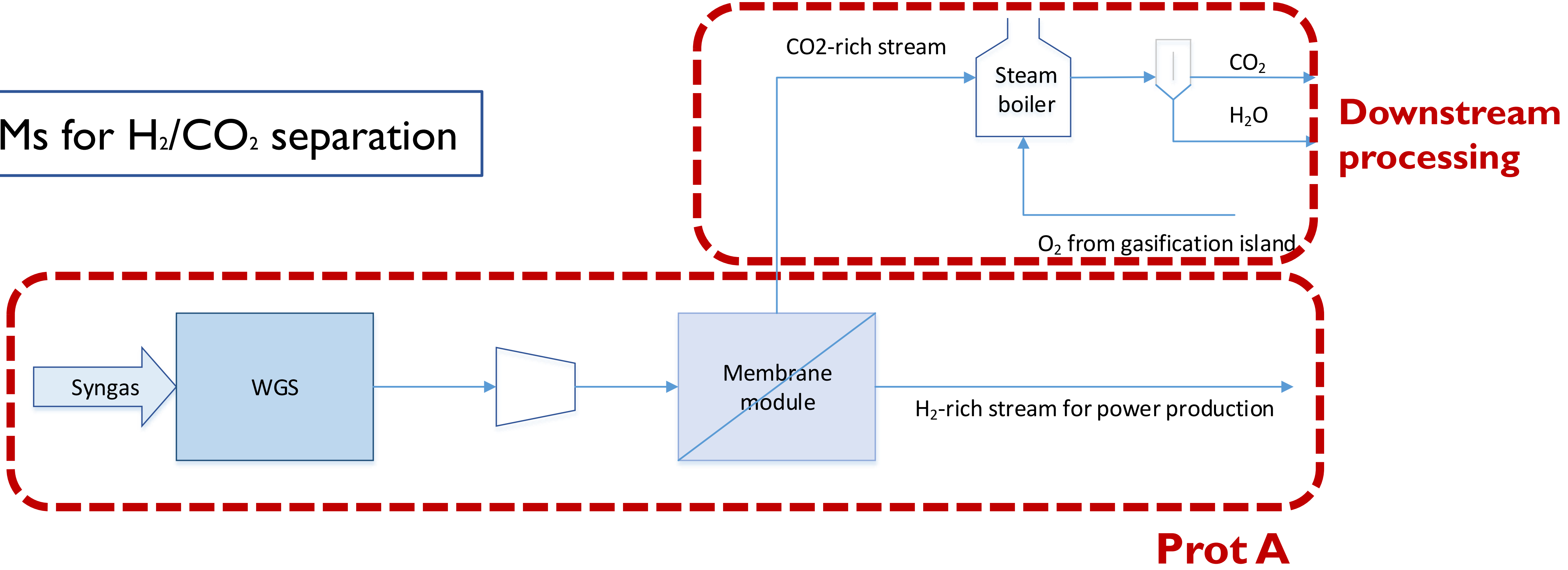
3. Demonstration: prototype design and testing

Pre-combustion capture – Prototype A

MMMs for H_2/CO_2 separation



- CO_2 separation from the shifted syngas after Water Gas Shift reactor
- The heating value contained in the original feedstock is re-allocated in a "decarbonized" fuel → Hydrogen

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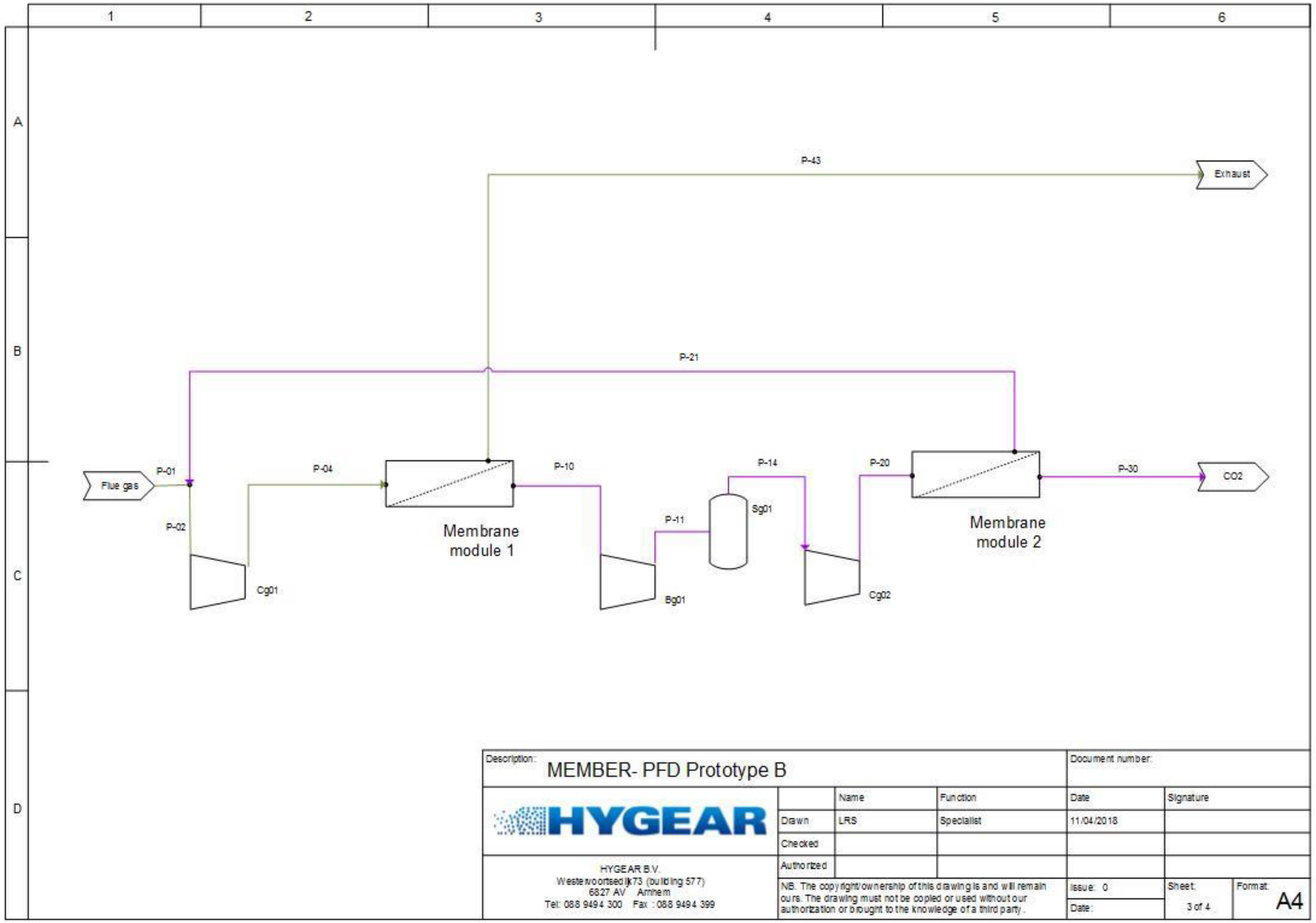
3. Demonstration: prototype design and testing

Post-combustion capture – Prototype B

MMMs for CO_2/N_2 separation

- CO_2 is separated from the N_2-rich flue gases from a combustion process
- Two membrane modules in series, module 1 larger than module 2



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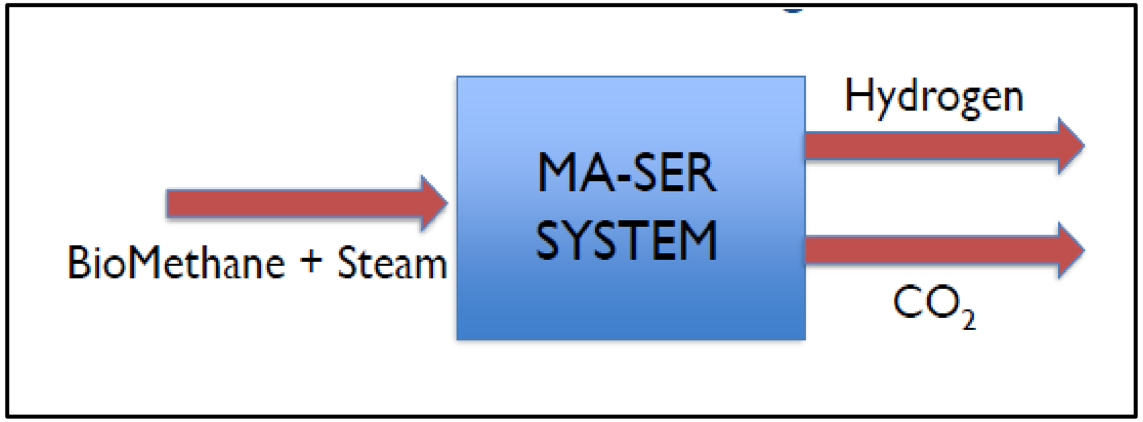
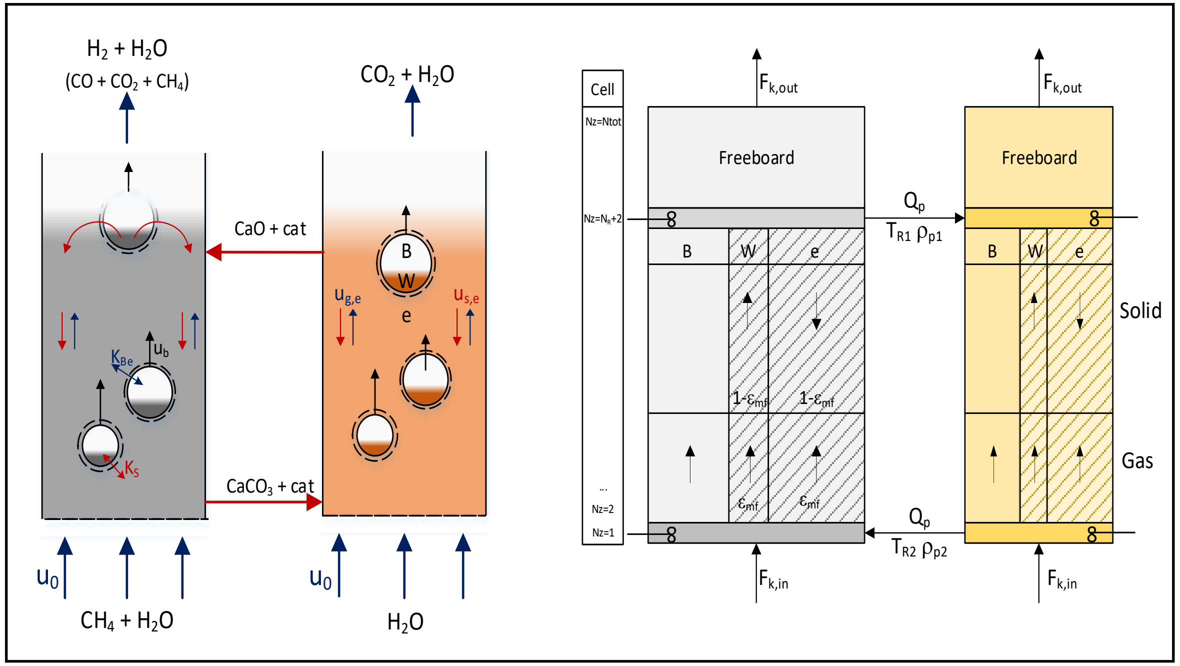
3. Demonstration: prototype design and testing

Hydrogen production integrated with CO_2 capture – Prototype C

Combination of metallic membranes, catalyst and sorbents





- A combination of metallic H_2 membranes, reforming catalyst and CO_2 sorbent into an advanced Membrane Assisted Sorption Enhanced Reforming (MA-SER) process.

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4. Expected impact

Category	#	Main exploitation product/ technologies/ others
PROCESS	1	MMM based system for pre-combustion CO_2 capture
PROCESS	2	MMM based system for post-combustion CO_2 capture
PROCESS	3	MA-SER system for pure H_2 production with integrated CO_2 capture
MATERIALS	4	Advanced polymers for post-combustion MMMs
MATERIALS	5	Advanced MOFs for pre- and post-combustion MMMs
MATERIALS	6	Advanced MMMs for pre- and post-combustion
MATERIALS	7	Advanced sorbents for MA-SER
MATERIALS	8	Advanced catalysts for MA-SER
MATERIALS	9	Advanced Pd-based H_2 membranes for MA-SER
SERVICE TOOLS	10	Software tool for Membrane reactor and SER design. Membrane separation modules
SERVICE TOOLS	11	Consulting services on LCA of CO_2 capture

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Thank you for your attention

https://member-co2.com/

Contact:

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Acknowledgement: For the CO2 molecule used in the logo:The original uploader was Frederic Marbach at French Wikipedia [GFDL (http://www.gnu.org/copyleft/fdl.html)

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2.2. Developing sustainable and economic scale-up routes for metal organic frameworks (Adam Deacon – JM)

JM

Johnson Matthey
Inspiring science, enhancing life

Developing efficient scale-up routes for MOFs

Adam Deacon

MEMBER workshop – Kjeller 23-06-2022

Strong credentials

Strong brand
200+ year history

Technology leadership
#1 or 2
in chosen markets

2021/22 sales*
£3.9 billion

2021/22 underlying operating profit
£556 million

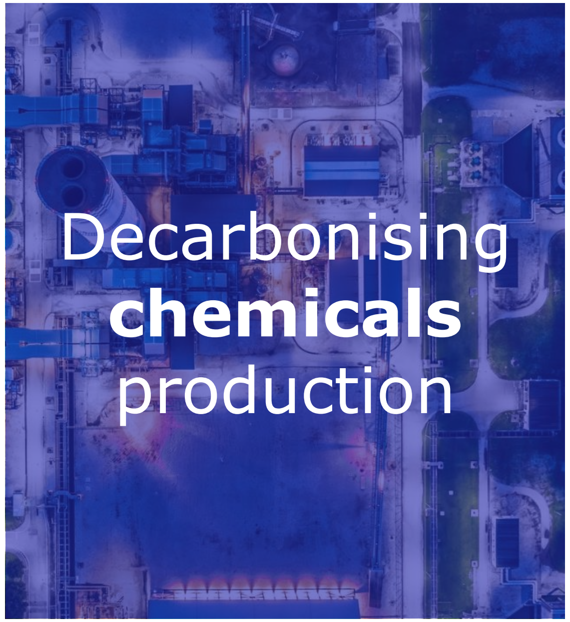
JM

*Sales excluding precious metals

Accelerating the transition to a cleaner, healthier world

Our vision is for a world that's cleaner and healthier. And so we are making it our business to help address the four essential transitions the world needs for a sustainable future.

Using world class science and technology to solve complex problems



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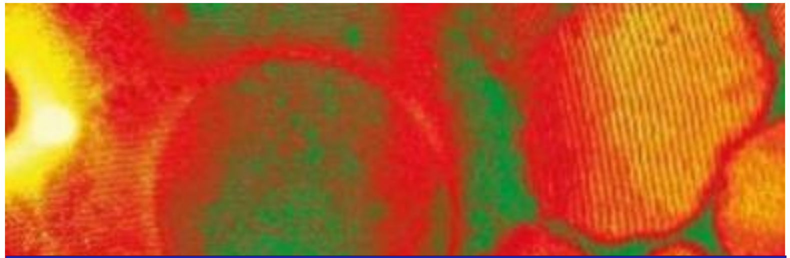
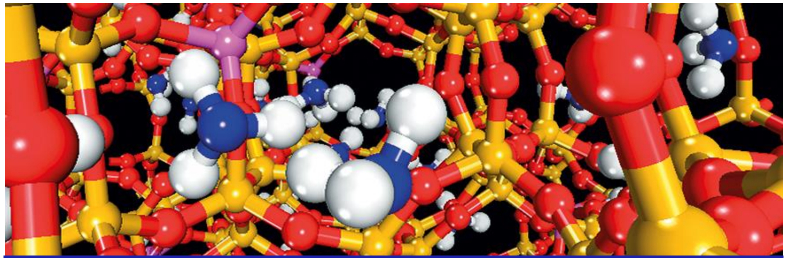
World class science and technology expertise

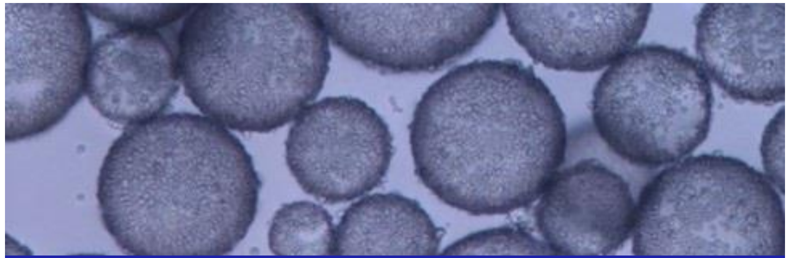


Characterisation and modelling

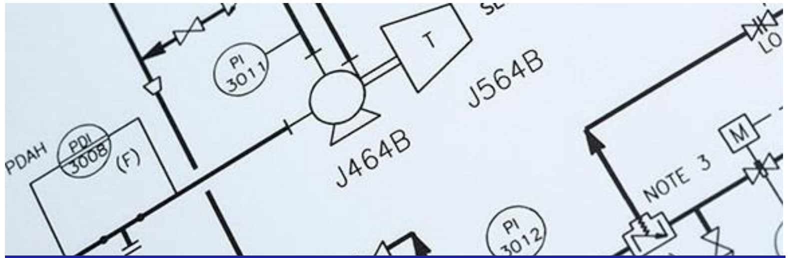
Chemical synthesis



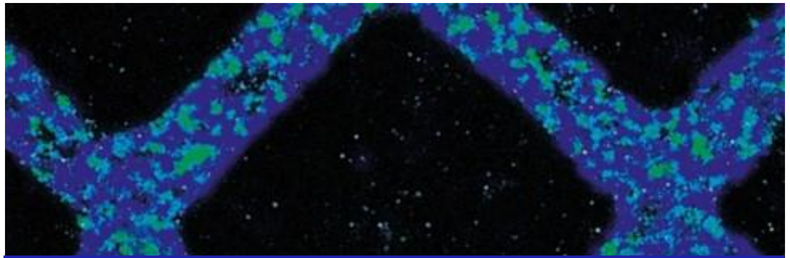
Material design and engineering



Product formulation



Process optimisation



Surface chemistry and coatings

Pgm chemistry and metallurgy

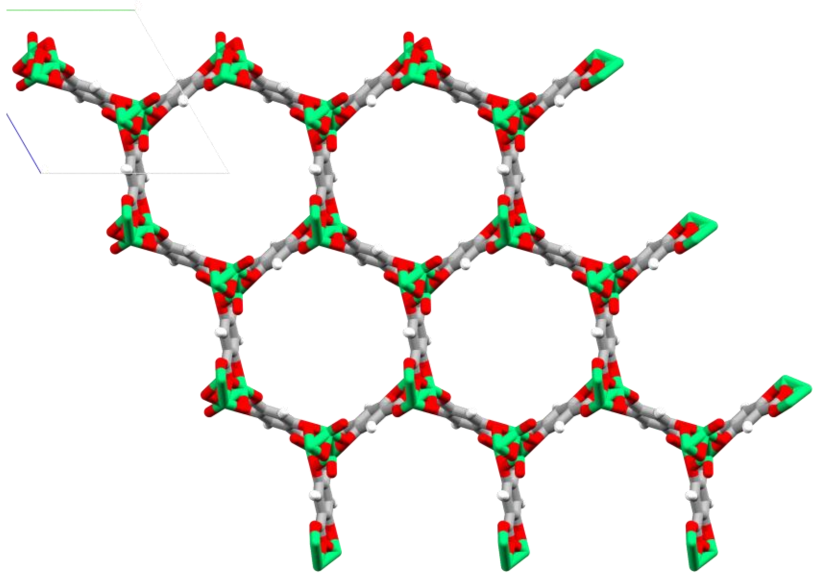
Catalysis and advanced materials

Electrochemistry

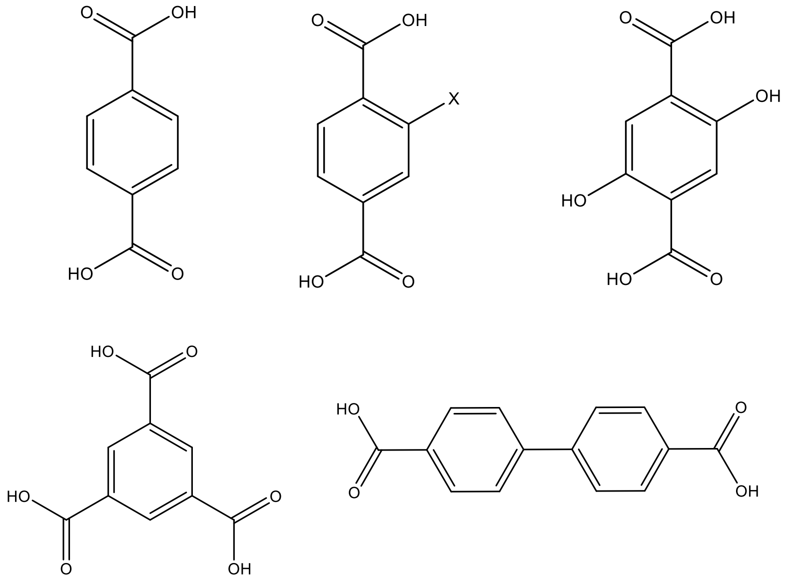
JM

What are Metal-Organic Frameworks (MOFs)?

Functional hybrid materials consisting of metal nodes connected by organic linkers.



- **High surface areas**
  - 1 g of material possessing the same surface area as a football pitch
- Huge number of possible structures with ~70 k reported [1].
- Functionality arises from:
  - **Porosity**, **pore structure**, metal nodes & linker functional groups
- Certain MOFs can be **stable under harsh conditions**
- Lots of academic interest over the last ~30 years
- Few products using MOFs now exist
  - TruPick™ & ION-X



Need to develop large scale, cost effective scale-up routes to make these application a reality

JM

[1] P. Z. Moghadam, et al. Chem. Mater. 2017, 29, 2618−2625

Scale-up considerations

Chemical

- Concentration
- Temperature/pressure
- Solvent

Physical

- Mixing
- Separation
- Washing
- Waste
- Product performance

Solvent	Safety Score	Health Score	Env. Score	Ranking
H_2O	1	1	1	Recommended
EtOH	4	3	3	Recommended
MeOH	4	7	5	Problematic
THF	6	7	5	Problematic
DMF	3	9	5	Hazardous
Sulfolane	1	9	7	Hazardous

D. Prat, et al., *Green Chem.*, 2016, **18**, 288-296

Reduction of raw materials is key for MOF scale-up

JM

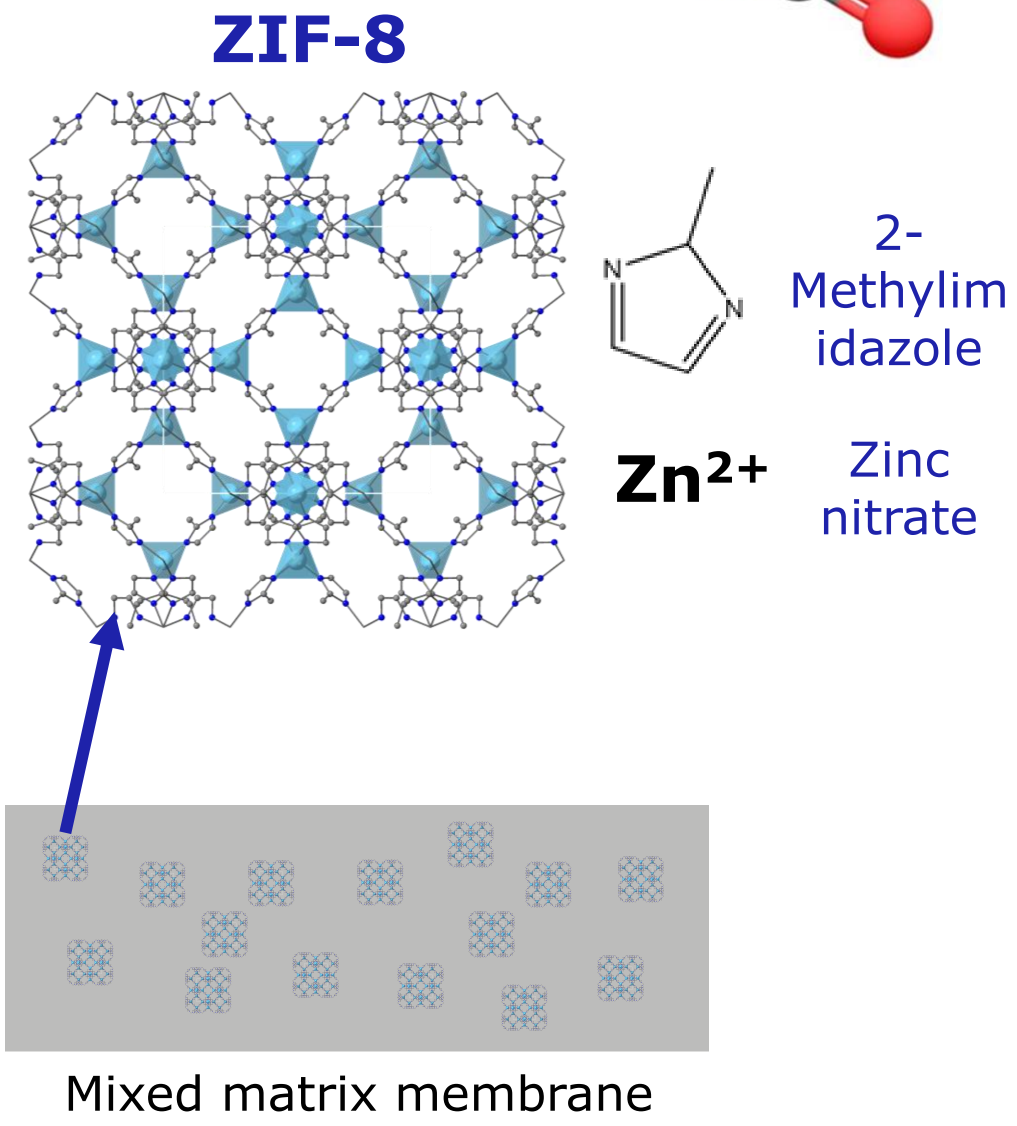
Nano ZIF-8 scale-up case study

Properties

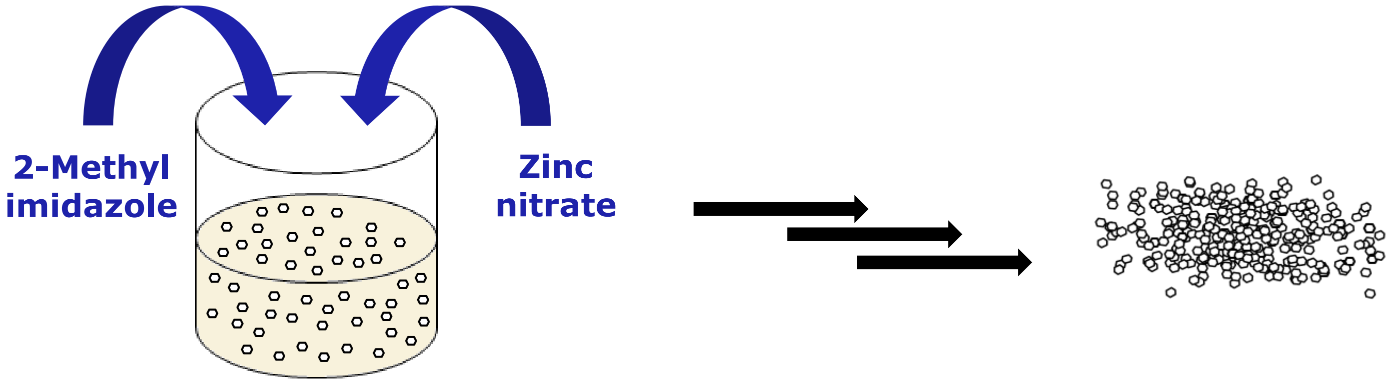
- Very high surface area ~ 1600 m^2g^{-1}
- High thermal stability – stable 400 ^oC
- Pore aperture – 3.4 Å

Application

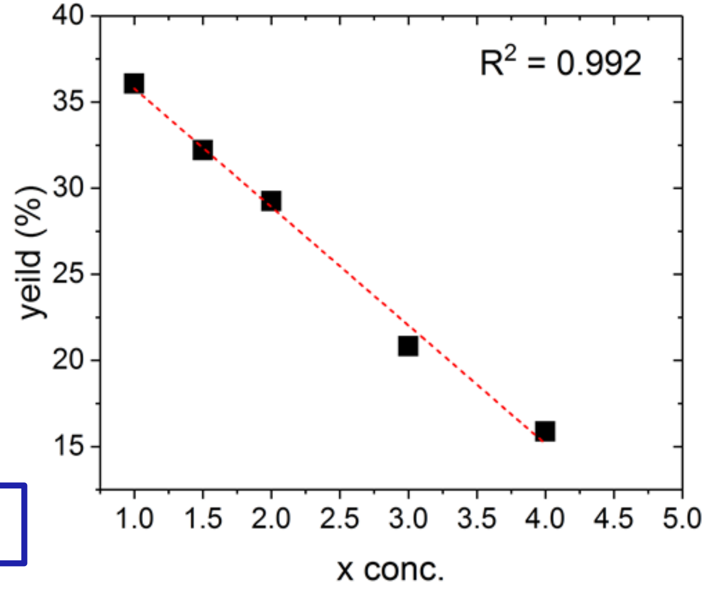
- Used in pre-combustion application - separation of H_2/CO_2
- Nano sized needed for membrane applications



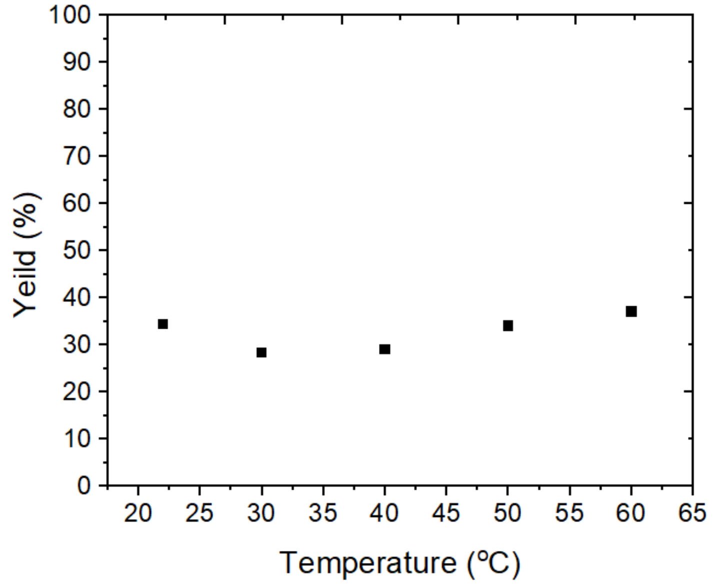
Original nano ZIF-8 route



$$Zn(NO_3)_2.6H_2O + 2C_4H_6N_2 \rightarrow Zn(C_4H_5N_2)_2 + 2H^+ + 2NO_3^- + 6H_2O$$



- Dilute conditions needed
  - Large quantities of Methanol used ~ 5 L MeOH needed for 5 g of nano ZIF-8

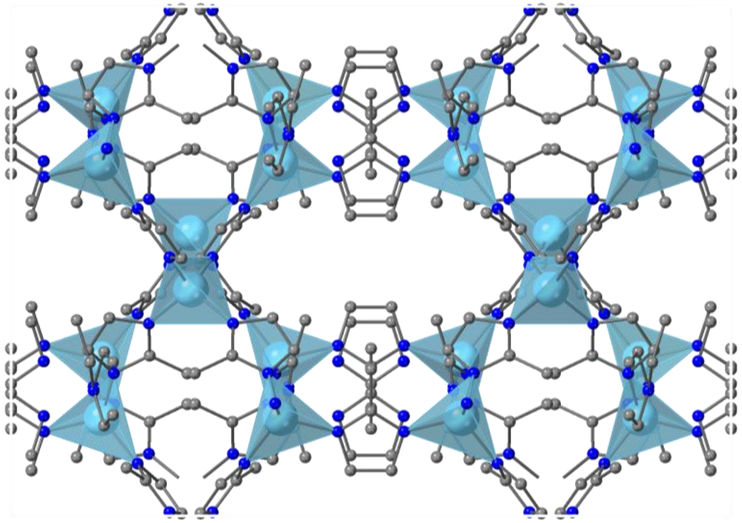


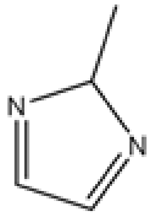
Parameter study showed its difficult to improve the original synthesis.

ZIF-L as an alternative route to nano ZIF-8

Properties

- ZIF-L is a dense phase polymorph of ZIF-8
- Consists of same raw materials as ZIF-8
- 2D material connected by linker molecules – leaf shape
- Low porosity – 92 m^2g^{-1}

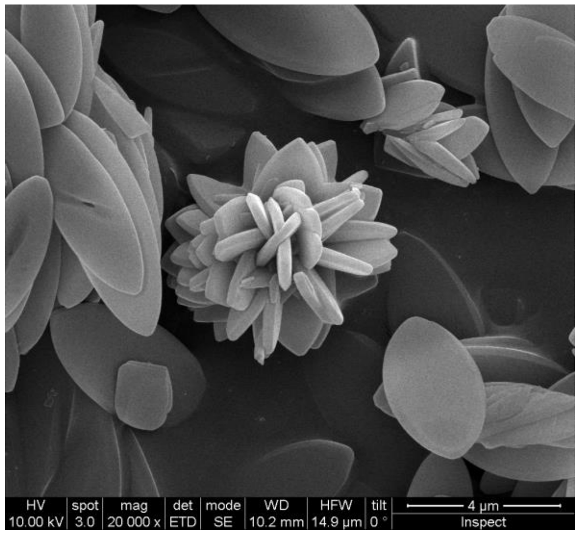




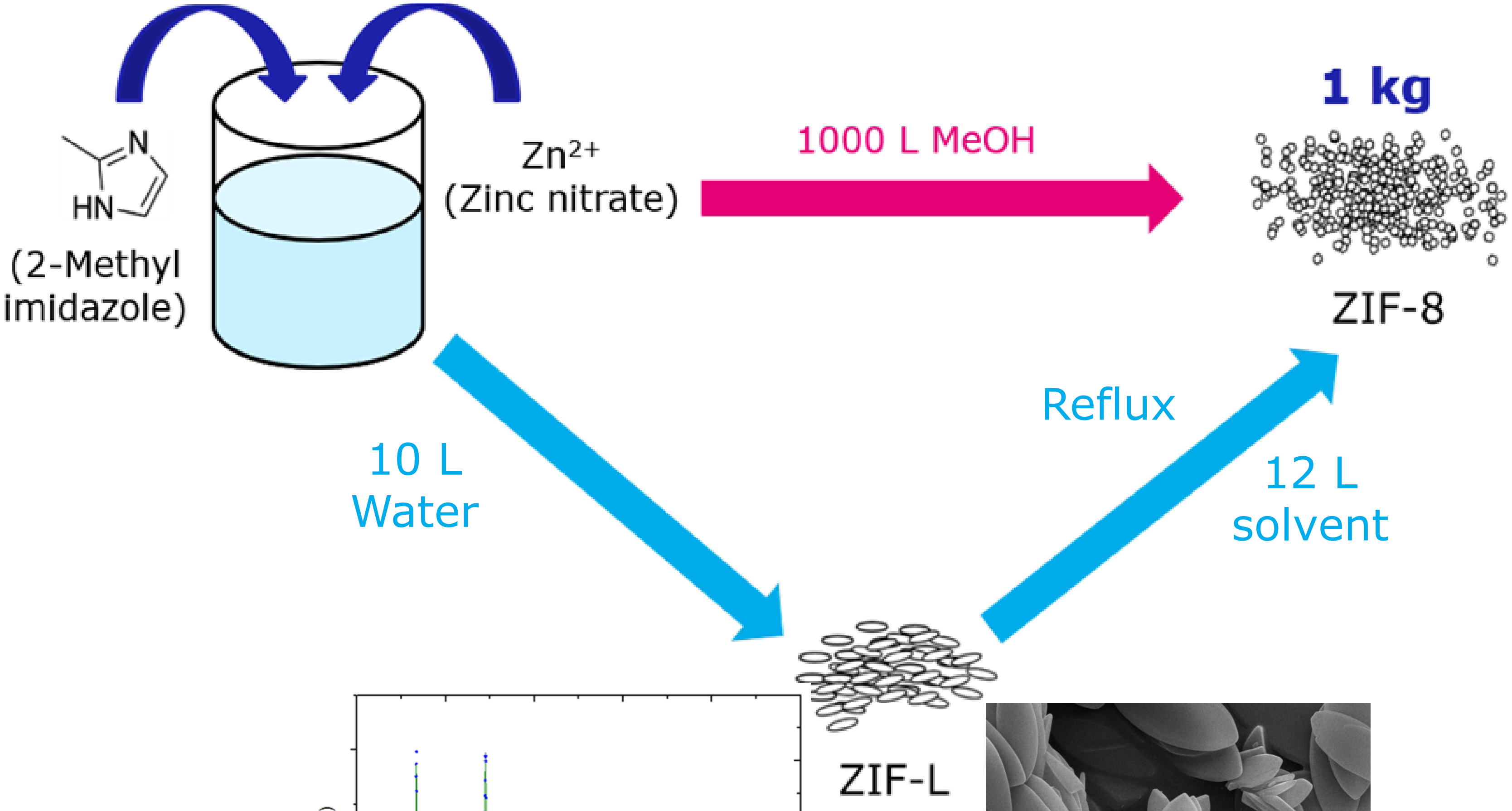
2-Methylimidazole

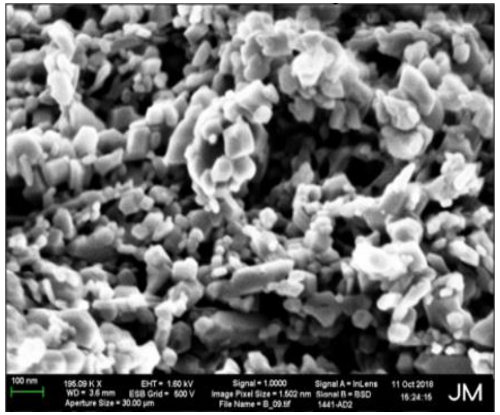
Zn^{2+} Zinc nitrate

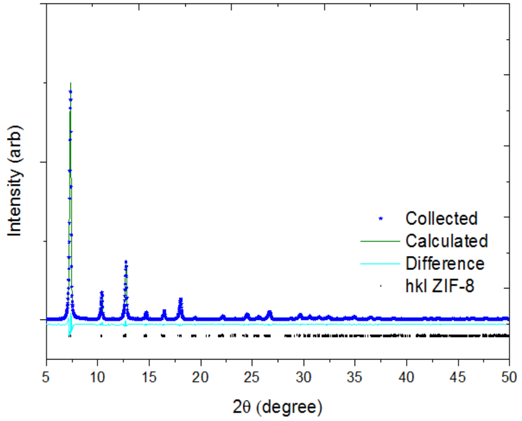
ZIF-L



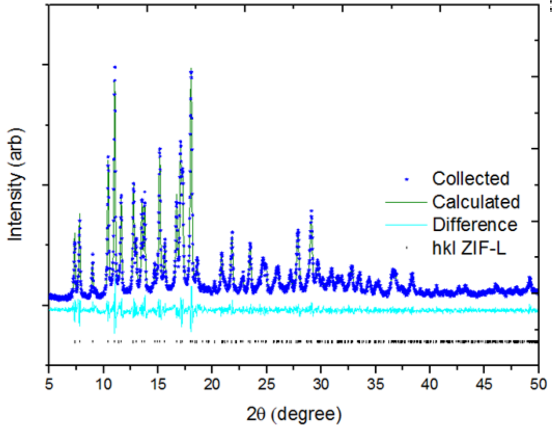
ZIF-L as an alternative route to nano ZIF-8

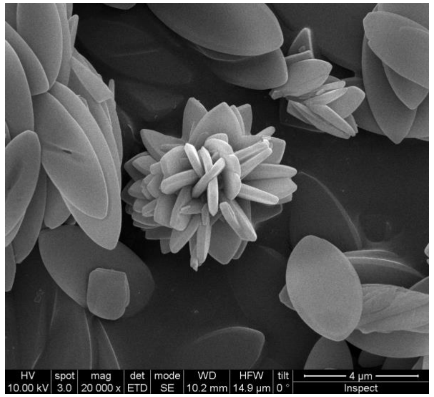






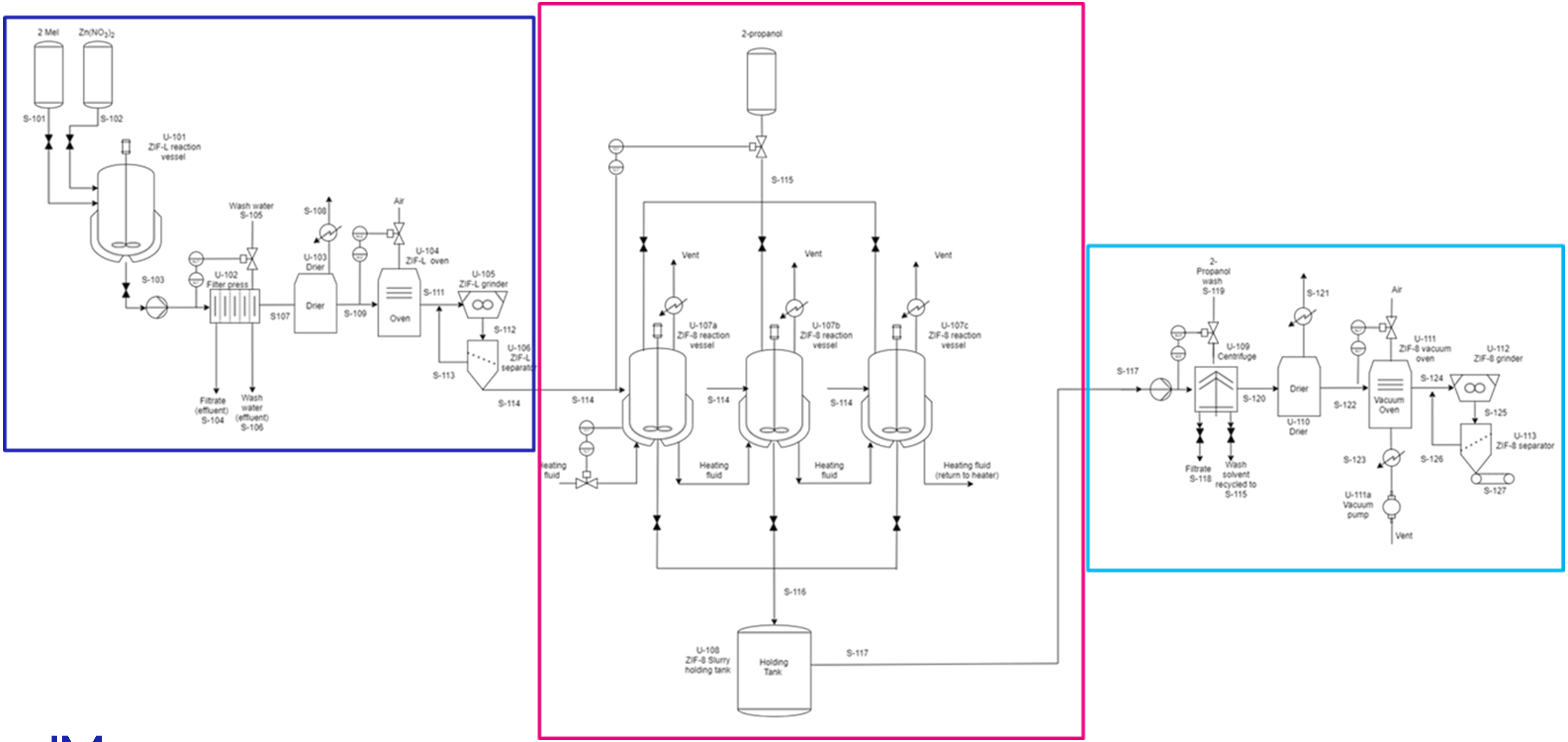
ZIF-8 BET surface area ~1600 m^2g^{-1}





ZIF-L BET surface area 92 m^2g^{-1}

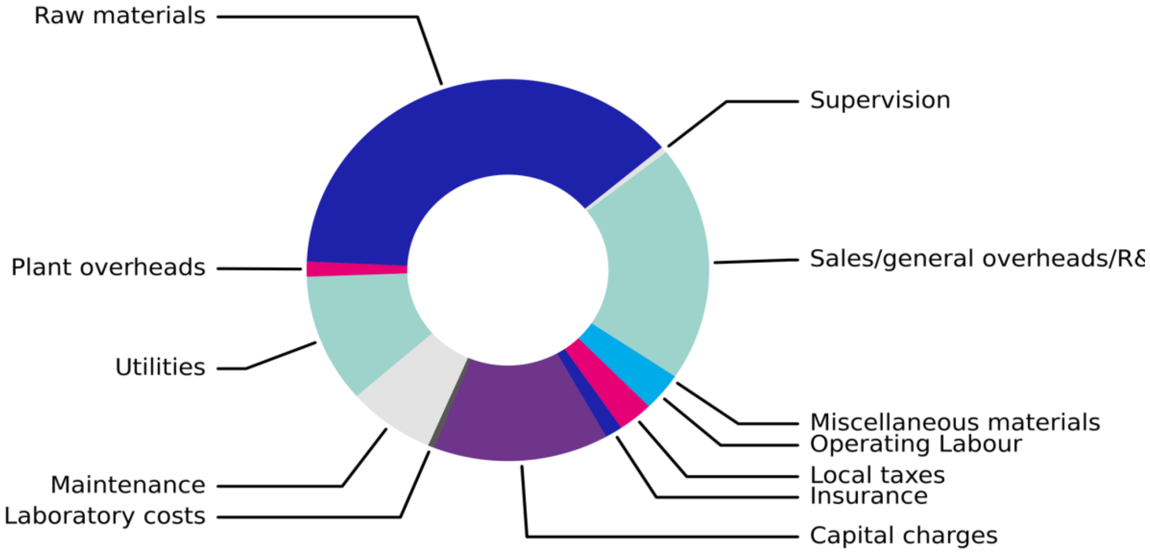
Industrial scale concept for nano ZIF-8



Nano-ZIF-8 case study summary

Developed scalable route

- Two order of magnitude solvent reduction
- Increased space time yield by factor of 10
- Replaced methanol with non-toxic solvent
- Industrial scale concept for nano ZIF-8 designed.



7x reduction in cost to produce

JM

Other scale-up examples

Scale-up: Fe-BTC

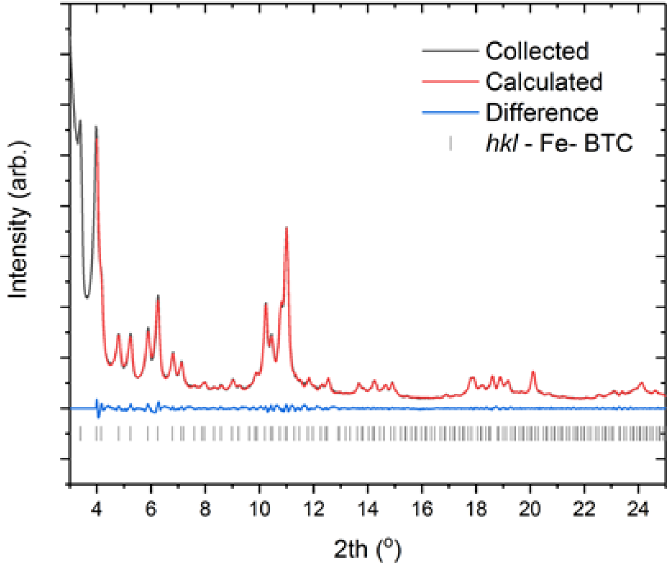
- 60 L batch reaction vessel
- Washed in purpose built setup
- 15 kg MOF produced
- BET surface area ~1500 m^2/g

Scale-up: CPO-27-Ni

- 10 kg CPO-27-Ni

JM

- Used in heat pump and



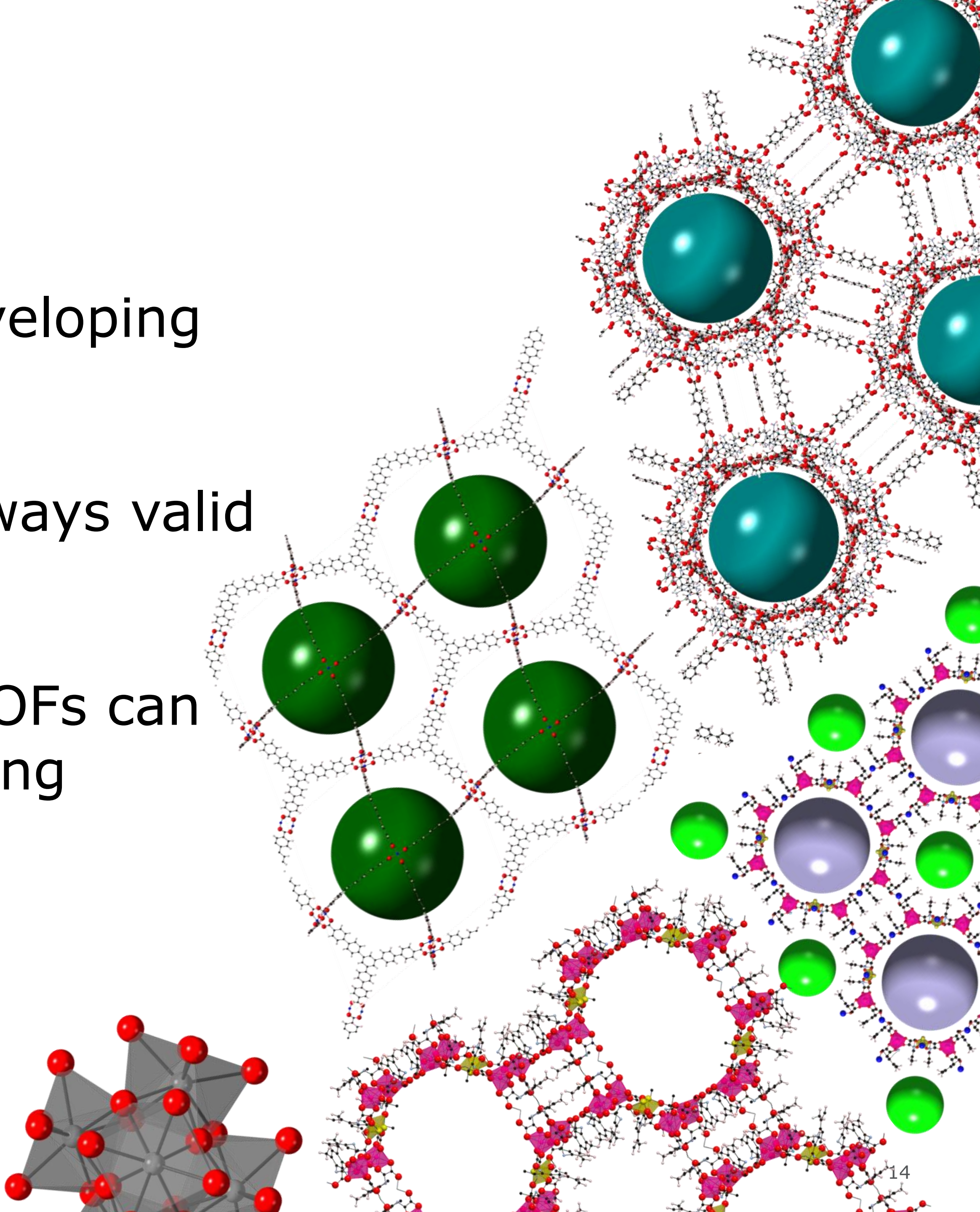
ProDIA

Summary

- Reducing raw materials cost key to developing large scale synthesis

- Conventional scale-up methods not always valid
  - Chemistry of MOFs is important

- Commercial large scale synthesis of MOFs can be achieved with the right understanding



JM

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JM

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Catalyst Research Group – Chilton

UNIZAR

Joaquin Coronas

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2.3. Mixed Matrix Membranes production scaling -up (William Marechal – POLYMEM)

Workshop on Advanced Membranes and Membrane assisted processes for pre- and post combustion CO_2 capture

Kunnskapsbyen Conference Center, June 23rd , 2022

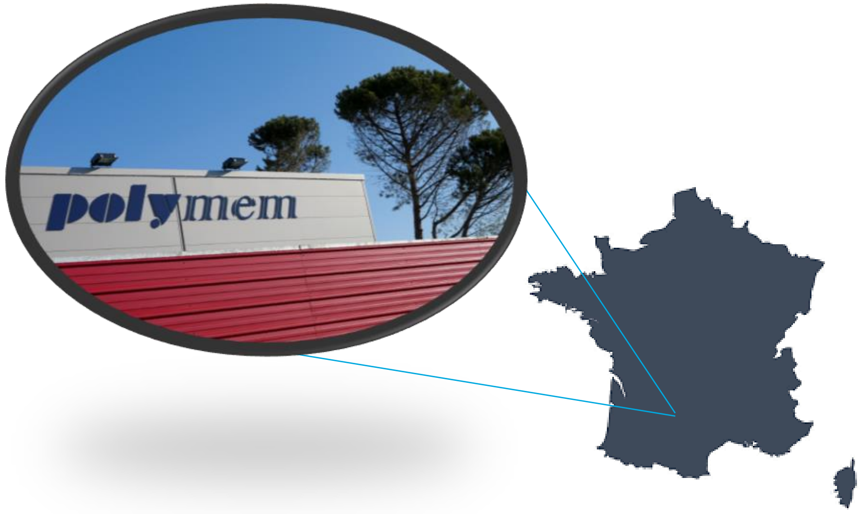
Mixed Matrix Membrane production scaling-up

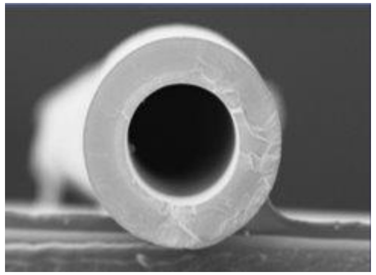
William MARECHAL
Principal Engineer for Membranes Development
w.marechal@polymem.fr

polymem | A REPLIGEN COMPANY

Our company
French hollow fibers membranes manufacturer

- Founded in 1997 and part of Repligen group since 2021
- Based in Castanet-Tolosan (Toulouse), France
- More than 80 people : General management, HR, QHSE, Sales & Marketing, R&D, Technical services
- Membrane technology knowledge and expertise for water treatment and biotechnologies applications
- **From the HF membrane to the system fabrication**

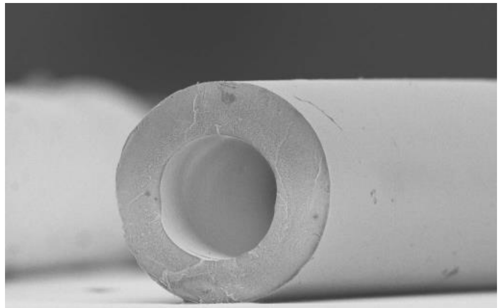
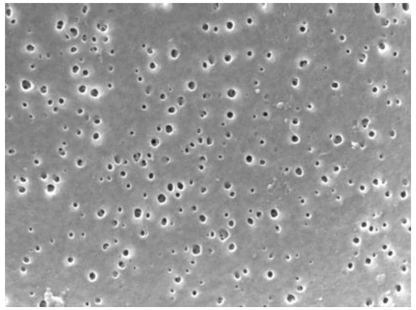
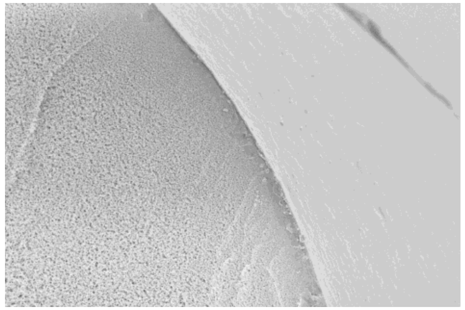
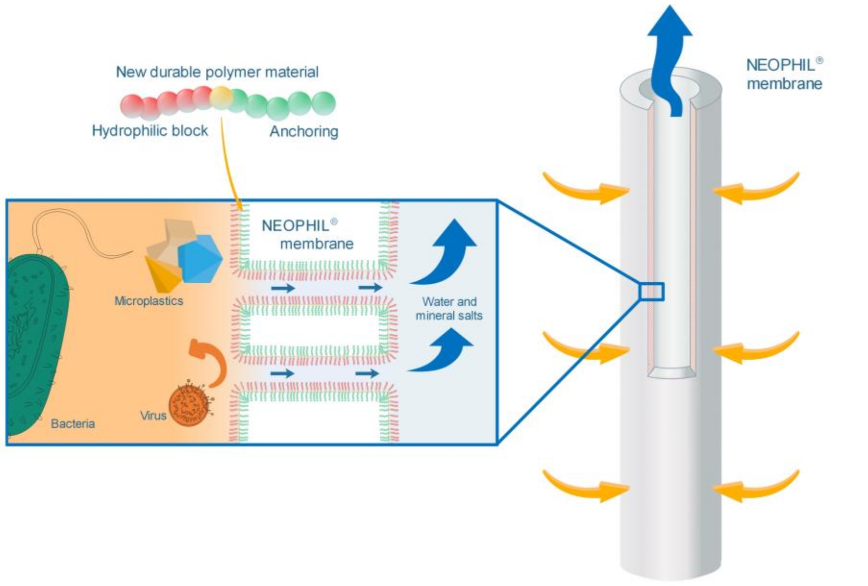




Hollow fiber membrane manufacturer

Manufacturing a various range of hollow fiber membrane

- Various dimensions: from 0,5 mm to 2 mm
- Various cut-off: from microfiltration to ultrafiltration
- Various porosity: with filtration skin inside and/or outside
- Various materials: Neophil ®, Polysulfone, Polyethersulfone

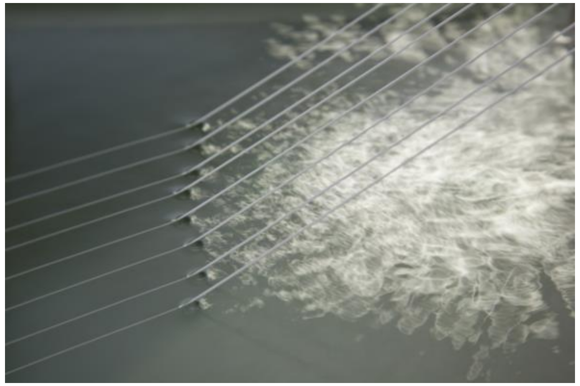
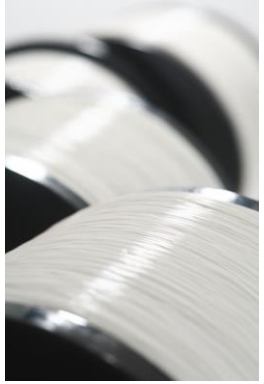





To provide the best filtration solution

Hollow fiber membrane spinning process

NIPS process : Non-solvent Induced Phase Separation



3 industrial production lines

1 pilot line: parametric study and pilot scale production

1 laboratory line: material screening

Other core business

- Laboratories for R&D and quality control
  - Membrane characterization, expertise
- Module fabrication
  - Wide range from 0.5 m² to 582 m², custom made modules
- System design and integration
  - Industrial unit, containerized unit
- Workshop: fabrication of laboratory tools up to production spinning line
  - Industrial spinning line, pilot and industrial unit

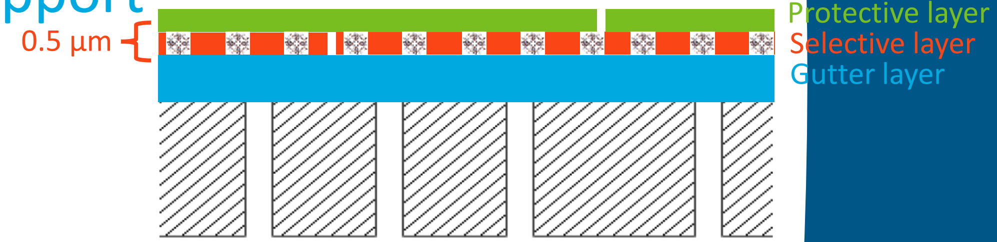
Mixed Matrix Membrane fabrication scaling-up

Optimization and scaling-up of a MMMs prepared by coating for CO_2 separation

MEMBER Project

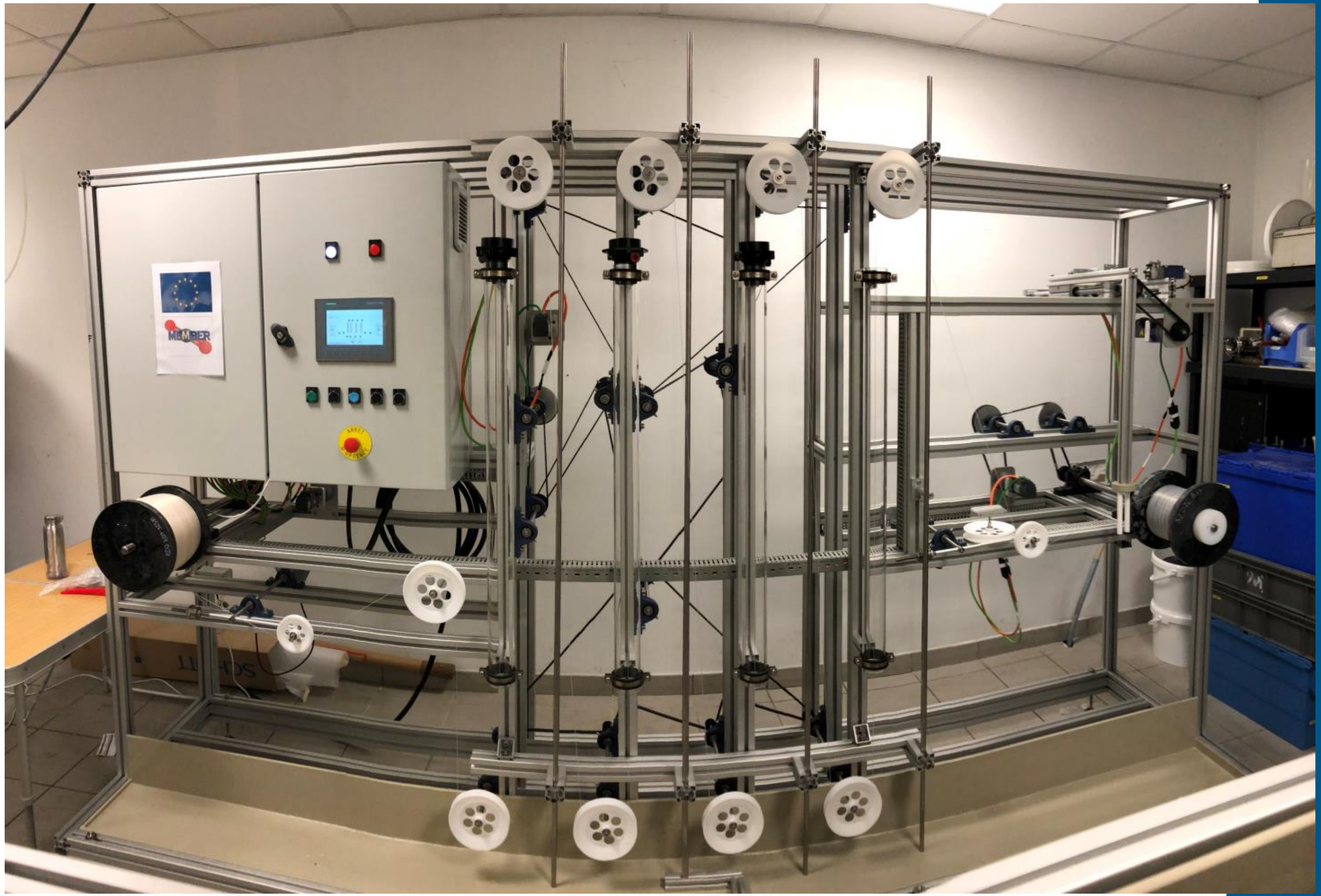
Objectives

- Fabrication of composite membrane with dense layer incorporating MOF for enhanced gas separation performances
  - Multilayer coating of dense layers on porous support
  - MOF incorporation
- Optimization of the fabrication recipe
  - Increase productivity by switching from batch to continuous process
  - Fabrication scaling-up to produce several 1,000 m batch
- Fabrication of 10 m² and 0.54 m² modules for demonstration on pilot installation

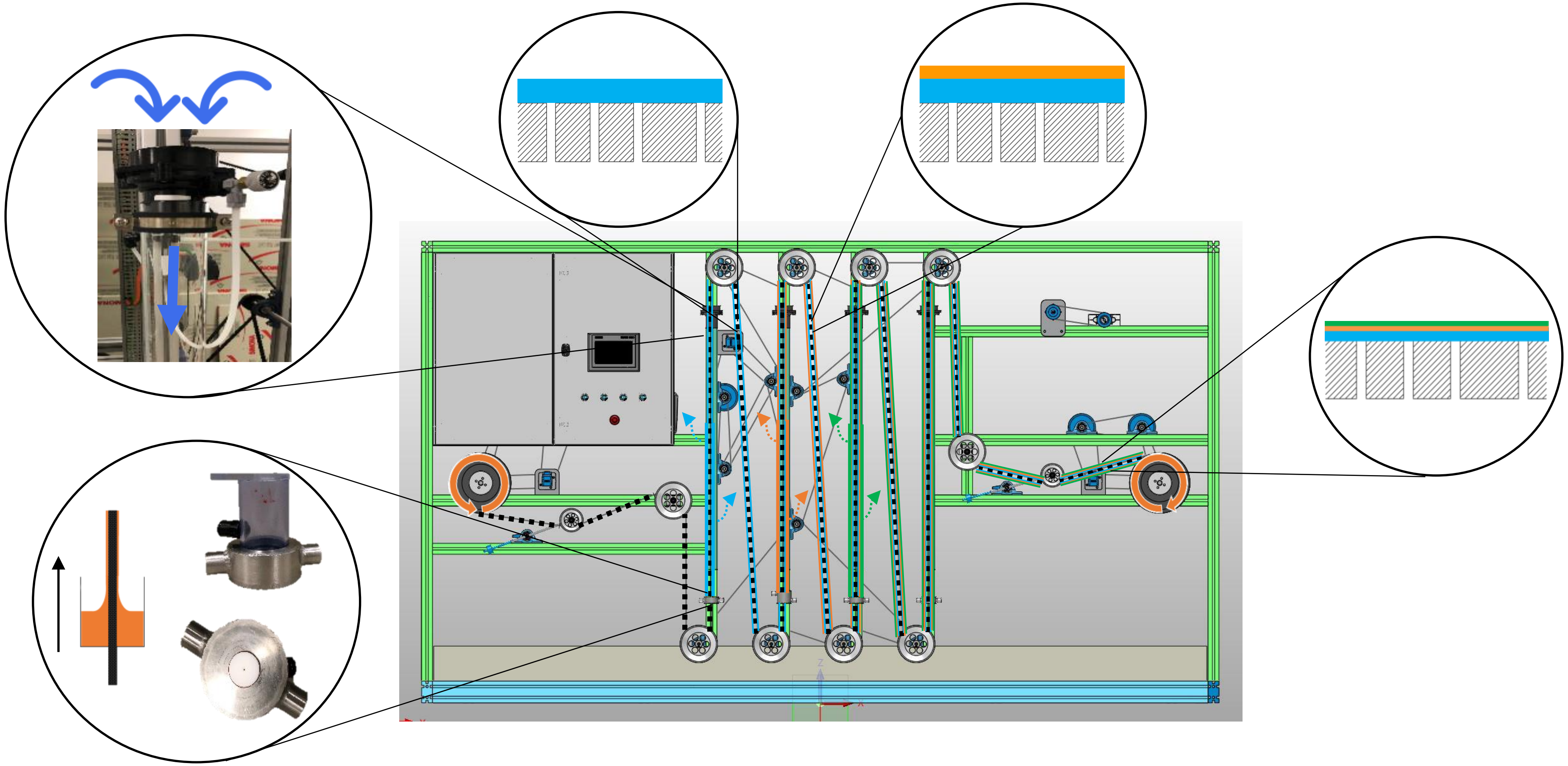


Continuous coating process for composite membrane fabrication

- Design and fabrication of an innovative pilot
- Built by our workshop
- Roll to roll machine
- Continuous coating of multiple layers: up to 4 different layers



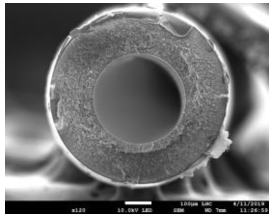
Continuous coating process for composite membrane fabrication

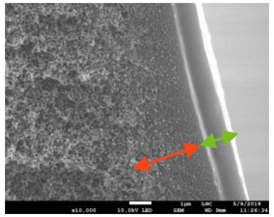


Road map to successfully produce at large scale HF MMMs

Year 1

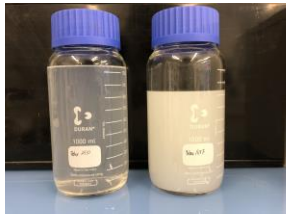
- **Start-up of installation**
- Validation of characterization tools
- **Demonstration of feasibility**
- Improvement of the coating line
- Optimization of the coating conditions





Year 2

- Definition of optimal coating materials: Fast curing PDMS and new grade of Pebax
- Increase of productivity x50



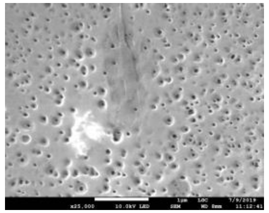
Year 3

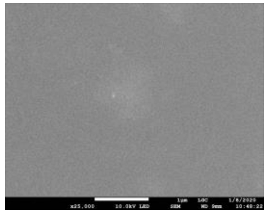
- Fine tuning of porous support porosity
- Parametric study for pure polymer coating optimization
- Fabrication scaling-up
- **1st membrane with good results reproducible over 100 m fabrication**

PCO_2 = 130 GPU ; α(CO_2/N_2) = 23

- **Improvement**

PCO_2 = 200 GPU ; α(CO_2/N_2) = 23





Year 4

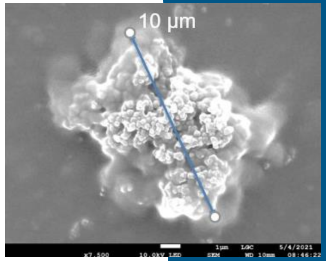
- Optimization of MOF incorporation
- Parametric study for MMMs coating optimization
- Scale-up :

6000 m of MMMs batch with quality control

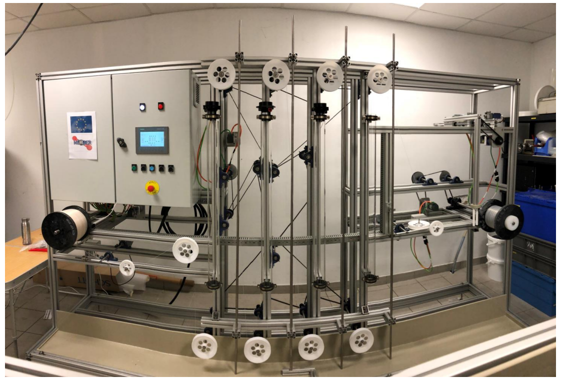
PCO_2 = 245 GPU ; α(CO_2/N_2) = 15,3

- Modules fabrication:

2 modules fabricated of 10 m² and 0.54 m²

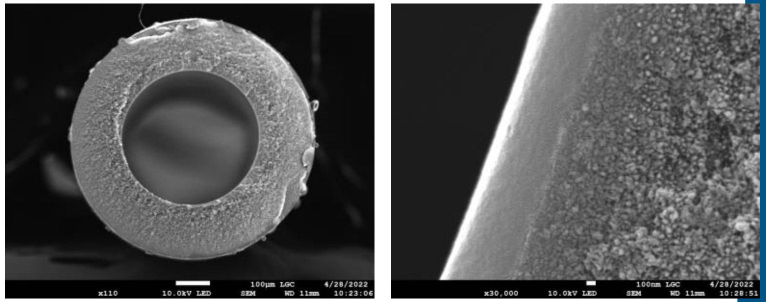


Main achievements



- Fabrication of an innovative coating line
  - Continuous process
  - Multilayer coating in one pass
- Taking over a new membrane fabrication process
  - Proof of feasibility
  - Parametric study
  - Optimization
- Large scale fabrication of a MMM :
  - 3 layers composite membrane with MOF incorporated
  - **Increased productivity x 50**
  - 6,000 m produced and validated by quality control

PCO_2 = 245 GPU ; PN_2 = 16,0 ; $\alpha(CO_2/N_2)$ = 15,3



Main achievements

- 10 m² and 0.54 m² modules fabrication for demonstration in a relevant environment:
  - Integrated in a skid unit fabricated by Hygear
  - Skid should have been installed in a Galp plant for flue gases CO_2 capture

From TRL 3 to TRL 6

Thank you for your attention…

… and a thanks to the contributors from Polymem : Olivier Lorain (head of research), Renan Bienassis (R&D technician), Lucie Laulhe (R&D technician), Patrick Santalo (Development manager), Jean-Michel Espenan (President)

MEMBER has received from the European Union's Horizon 2020 Research and Innovation Program under grant agreement n°760944

2.4. Pre- and Post-combustion CO_2 capture with MMM systems (Hans ten Dam – HYGEAR)

Membrane and system modelling

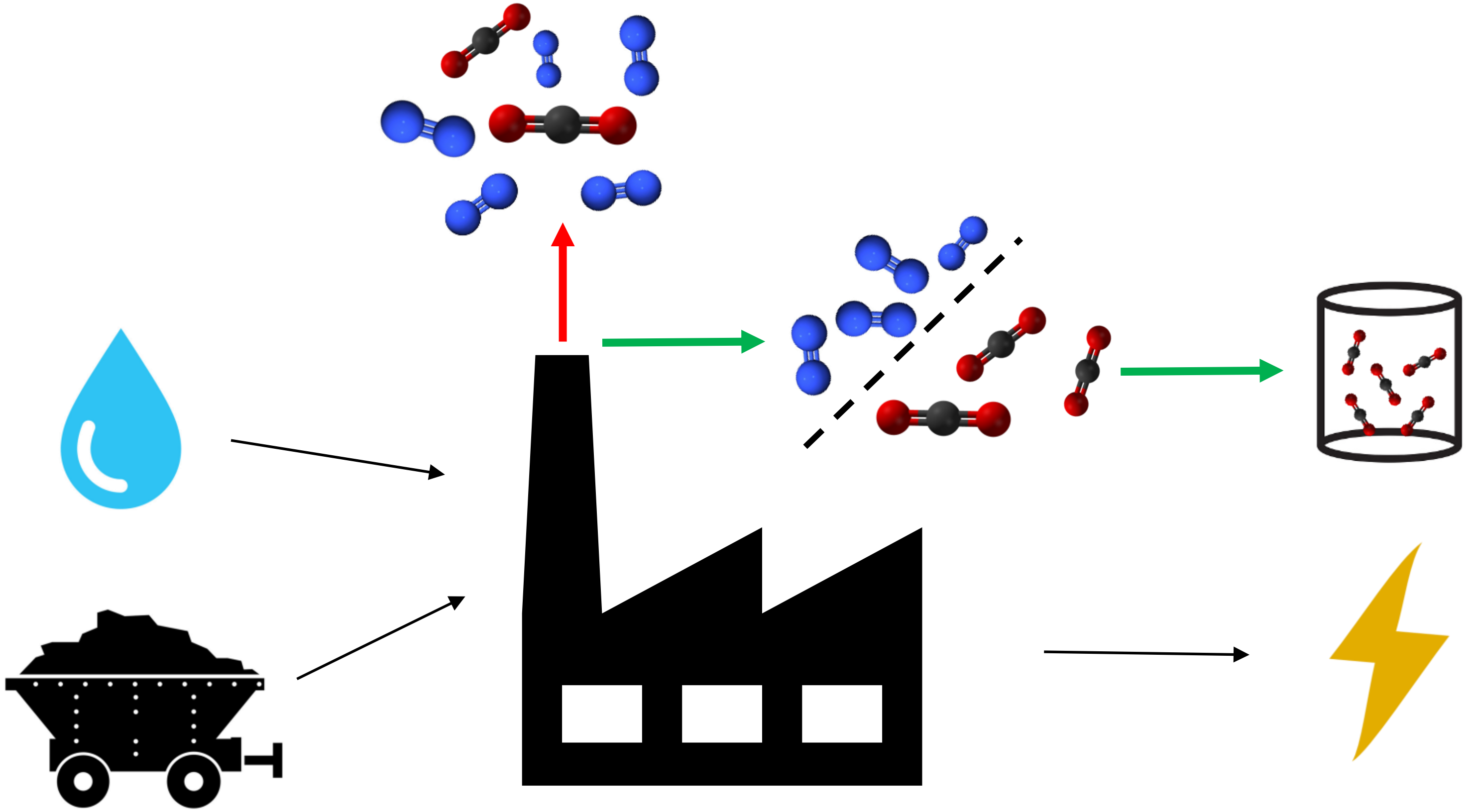
Pre-combustion and post-combustion CO_2 separation technologies with MMM

Hans ten Dam

Disclaimer: This presentation reflects the author's view and the Commission is not responsible for any use that may be made of the information it contains.
This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 760944

Workshop IFE
23-06-2022

Introduction – post combustion



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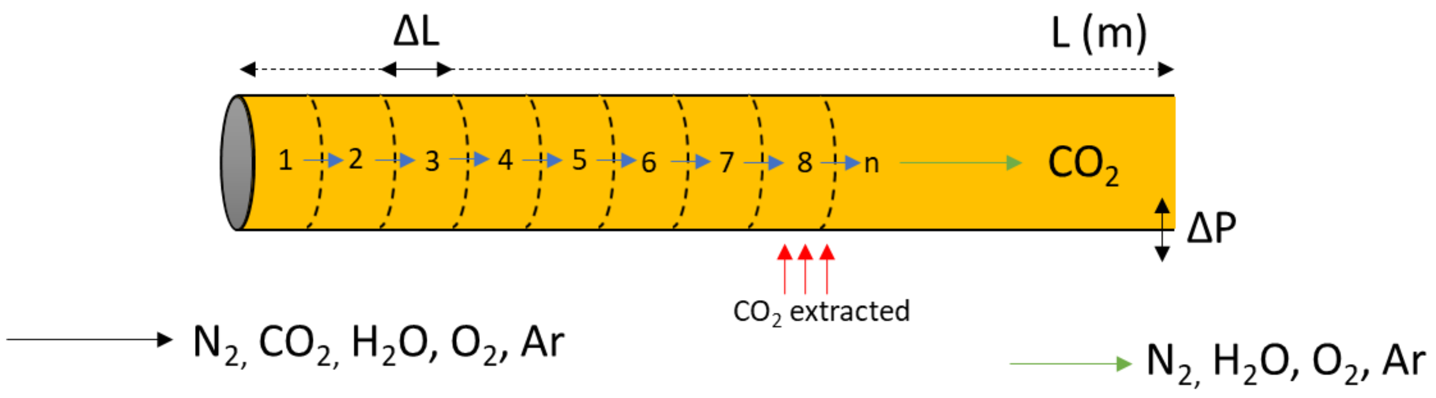
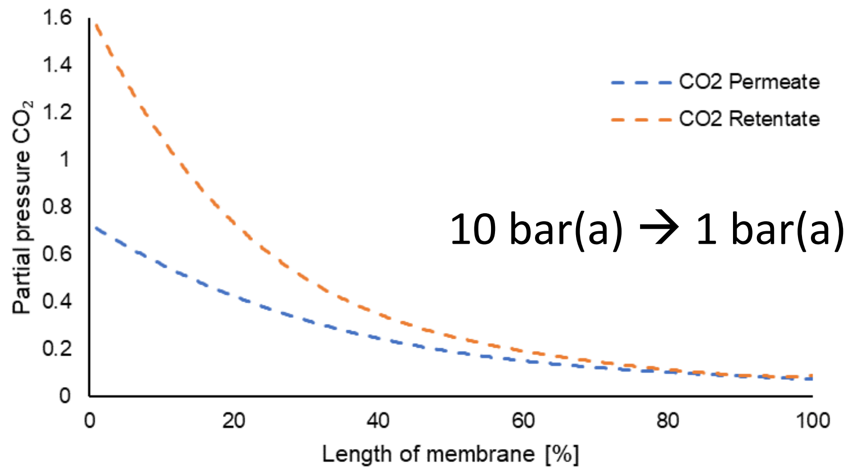
Workshop IFE
23-06-2022

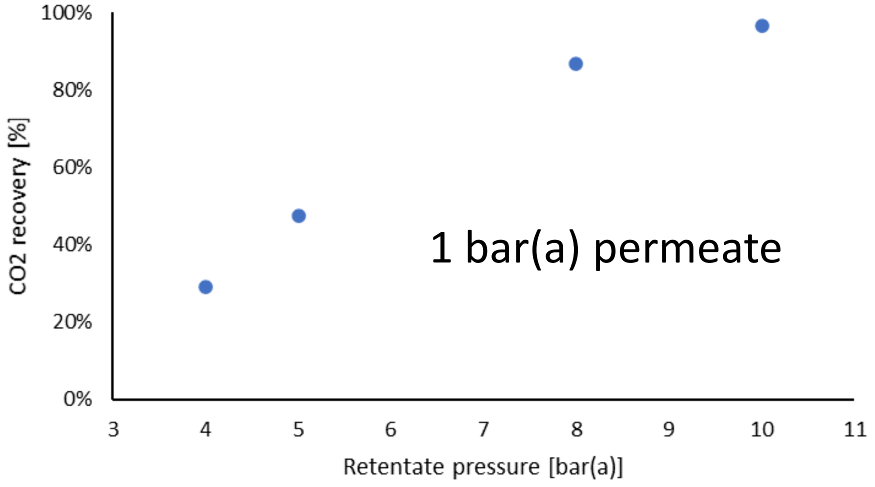
Hollow fiber membranes modelling





- Selectivity
- Permeability
- Length
- Pressure difference

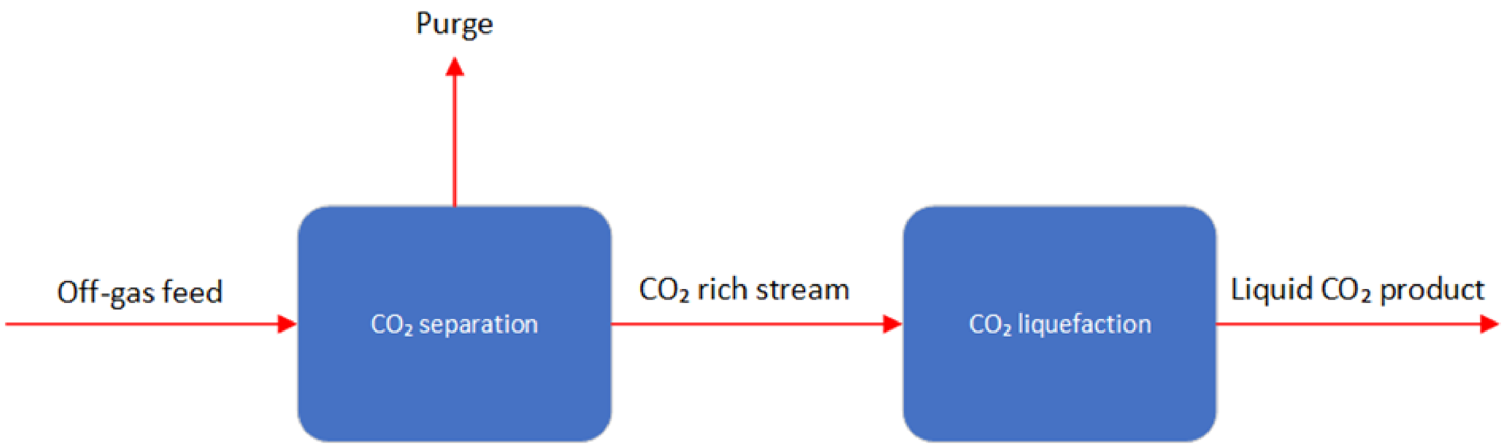
→ Membrane area



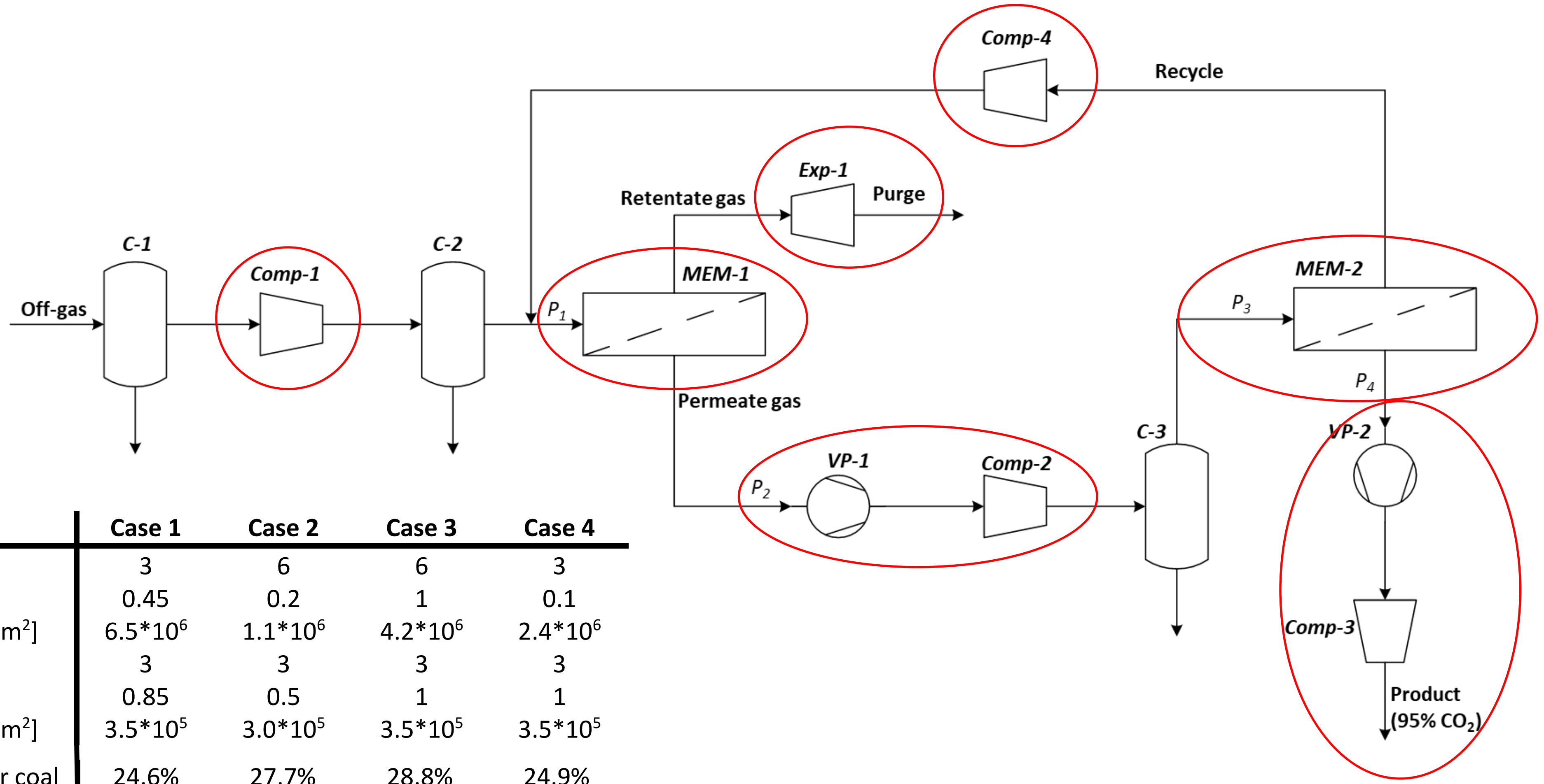
Process functional diagram

- Basis of design
  - MMM in coal fired power plant with net power production 550 MWe
  - 90 % CO_2 recovery & 95 % CO_2 purity

Component	Composition mol %
CO_2	13.5
N_2	68.1
O_2	2.4
H_2O	15.2
Ar	0.8



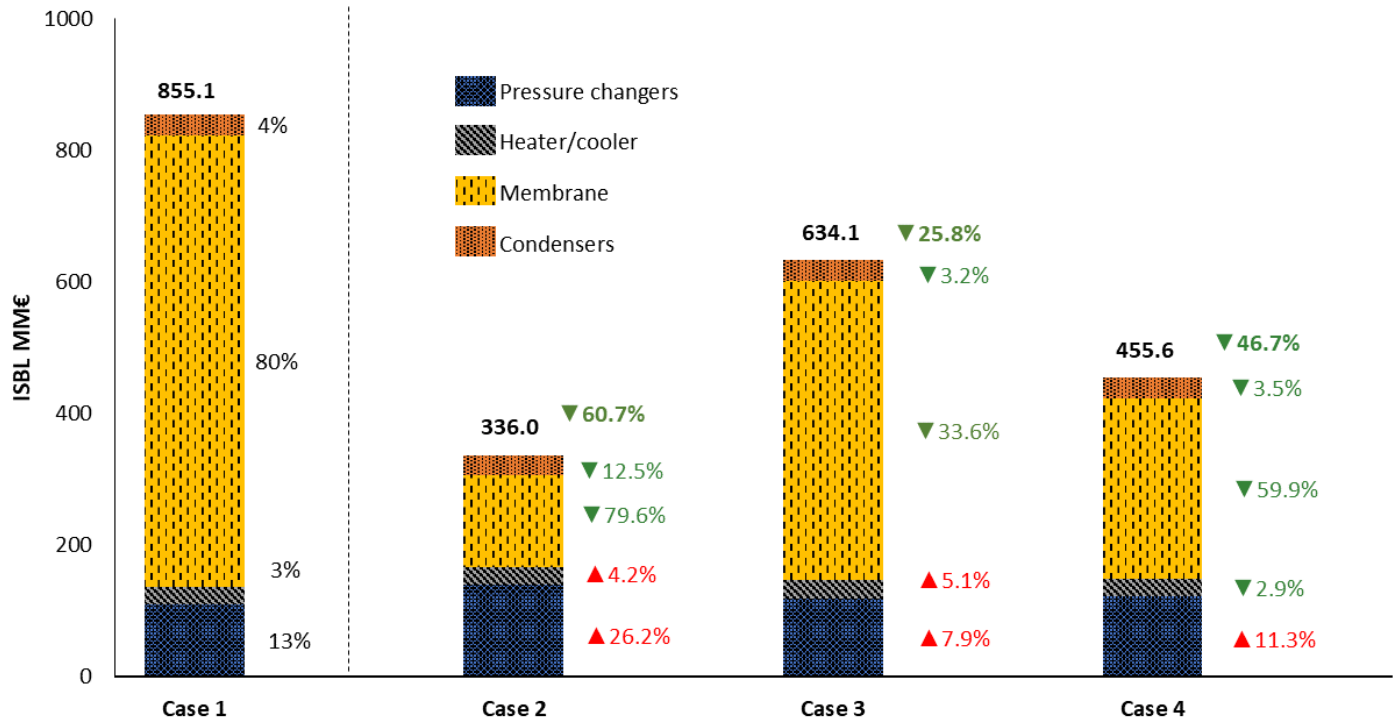
Process flow diagram



Parameter	Case 1	Case 2	Case 3	Case 4
P_1 [bar_a]	3	6	6	3
P_2 [bar_a]	0.45	0.2	1	0.1
Area MEM-1 [m^2]	$6.5*10^6$	$1.1*10^6$	$4.2*10^6$	$2.4*10^6$
P_3 [bar_a]	3	3	3	3
P_4 [bar_a]	0.85	0.5	1	1
Area MEM-2 [m^2]	$3.5*10^5$	$3.0*10^5$	$3.5*10^5$	$3.5*10^5$
E %-net power coal plant	24.6%	27.7%	28.8%	24.9%

Simulation results (CAPEX)

- Membrane cost → 100 €/m^2
- Membrane length → 0.5 m



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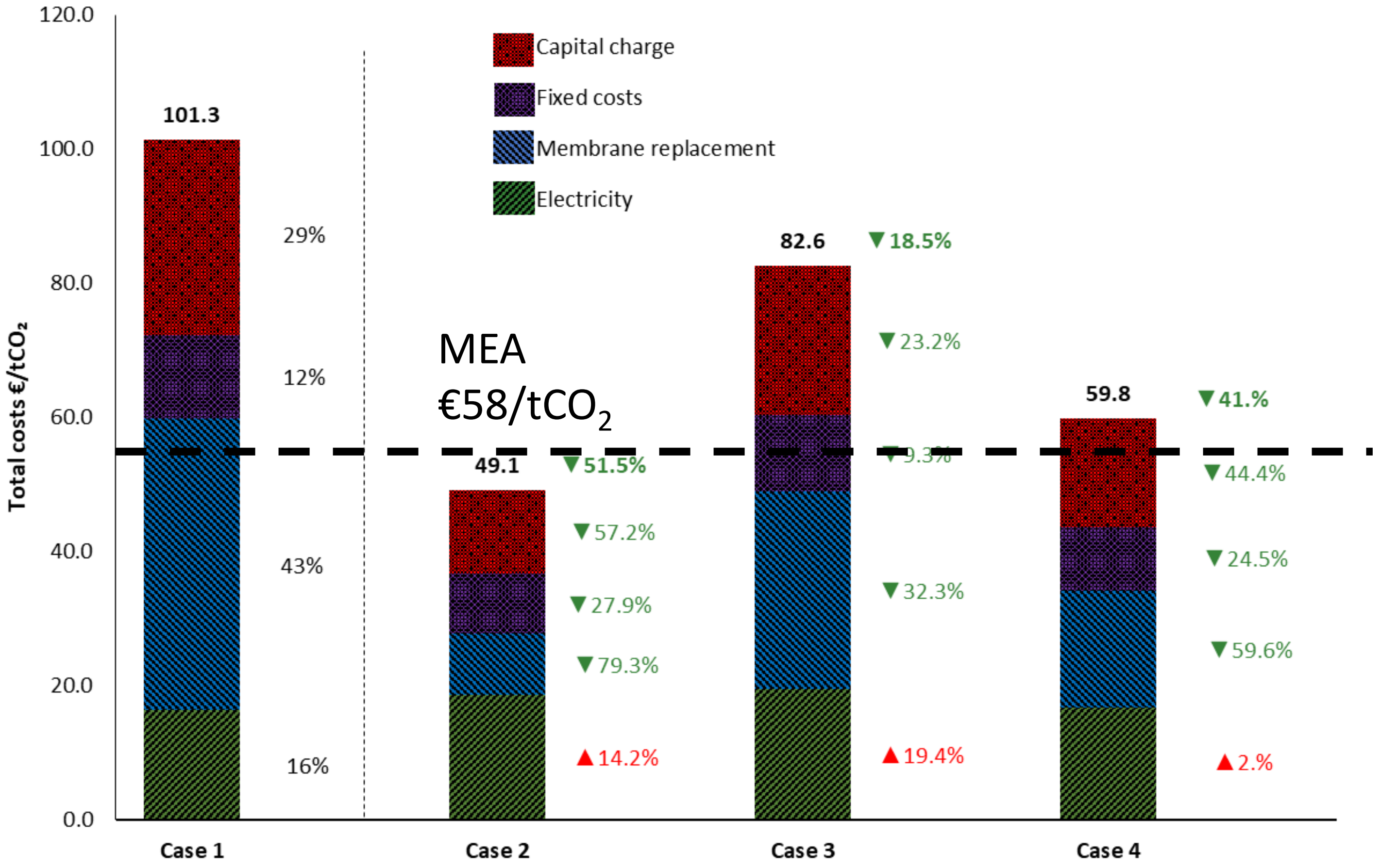
Simulation results (CAPEX + OPEX)

- Replacement → 5 yr
- Electricity → €52/MWh
- Operation → 8000 hr



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23-06-2022

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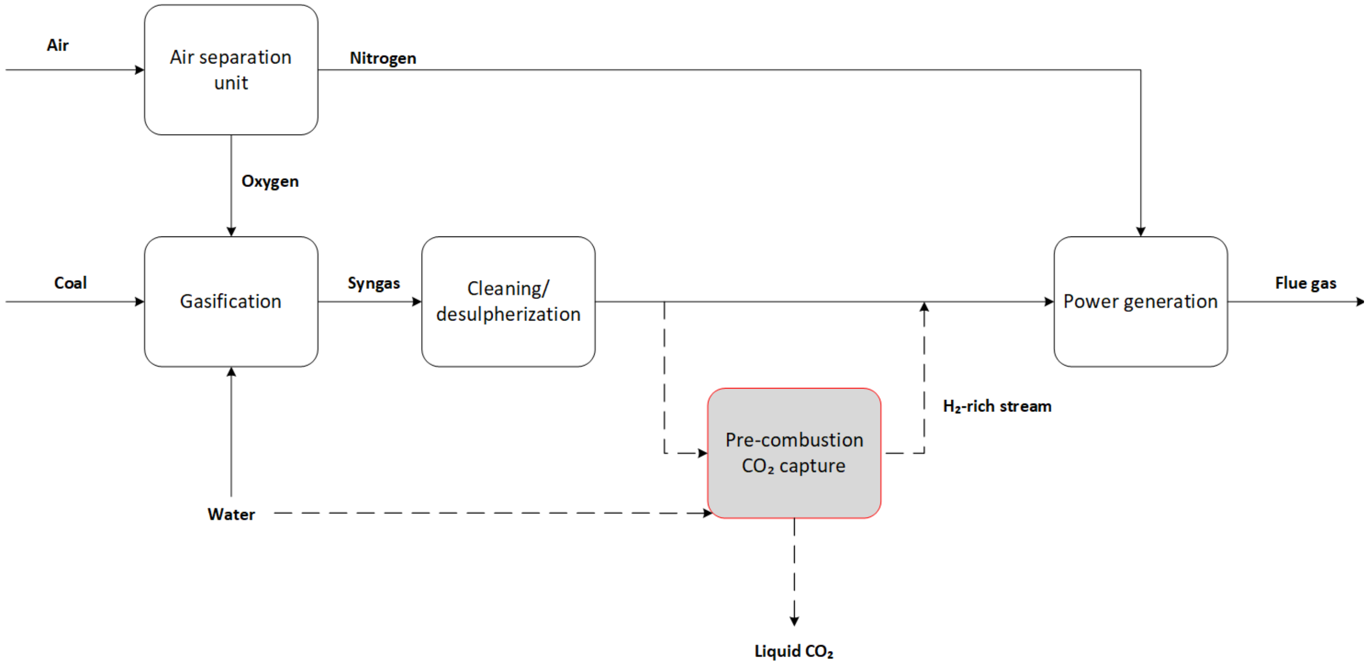
Conclusions and outlook for post-combustion carbon capture

- Cost-competitive with MEA
- Large membrane area

- Module design
- Decreasing membrane area
- Potentially interesting purge stream

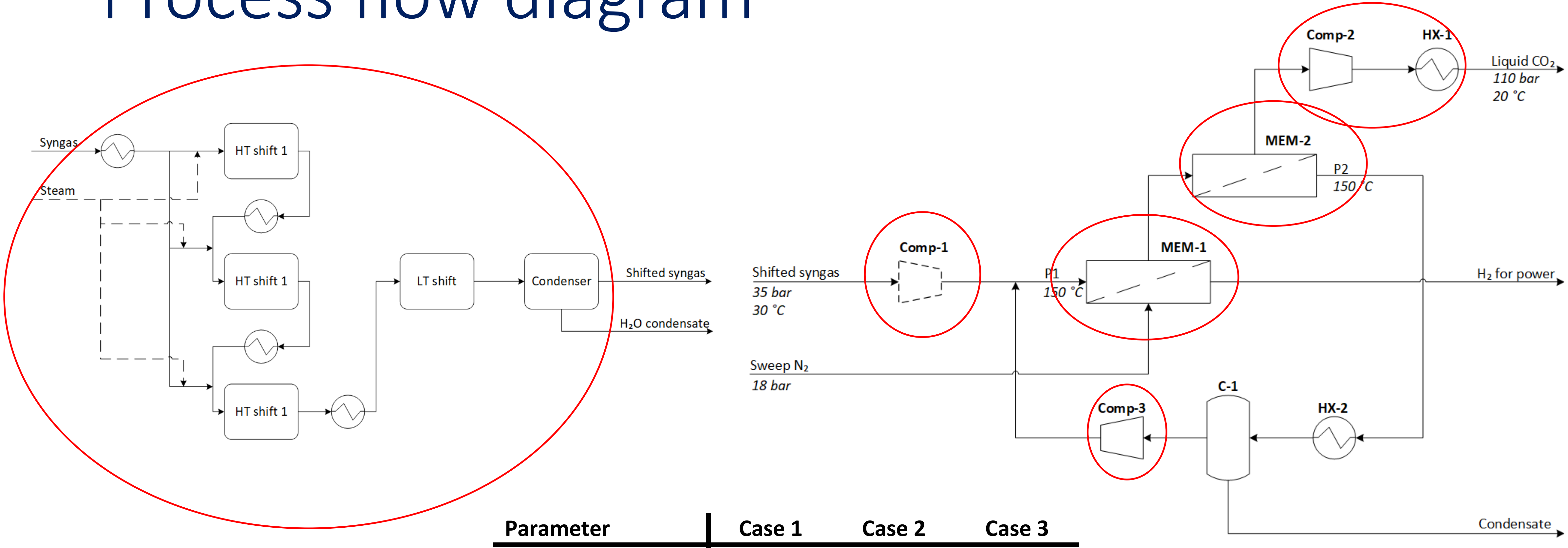
Pre-combustion process

- Basis of design
  - MMM in coal fired IGCC with net power production 536 MWe
  - 90 % CO_2 recovery & 95 % CO_2 purity



HYGEAR Member of the xebec group

Process flow diagram



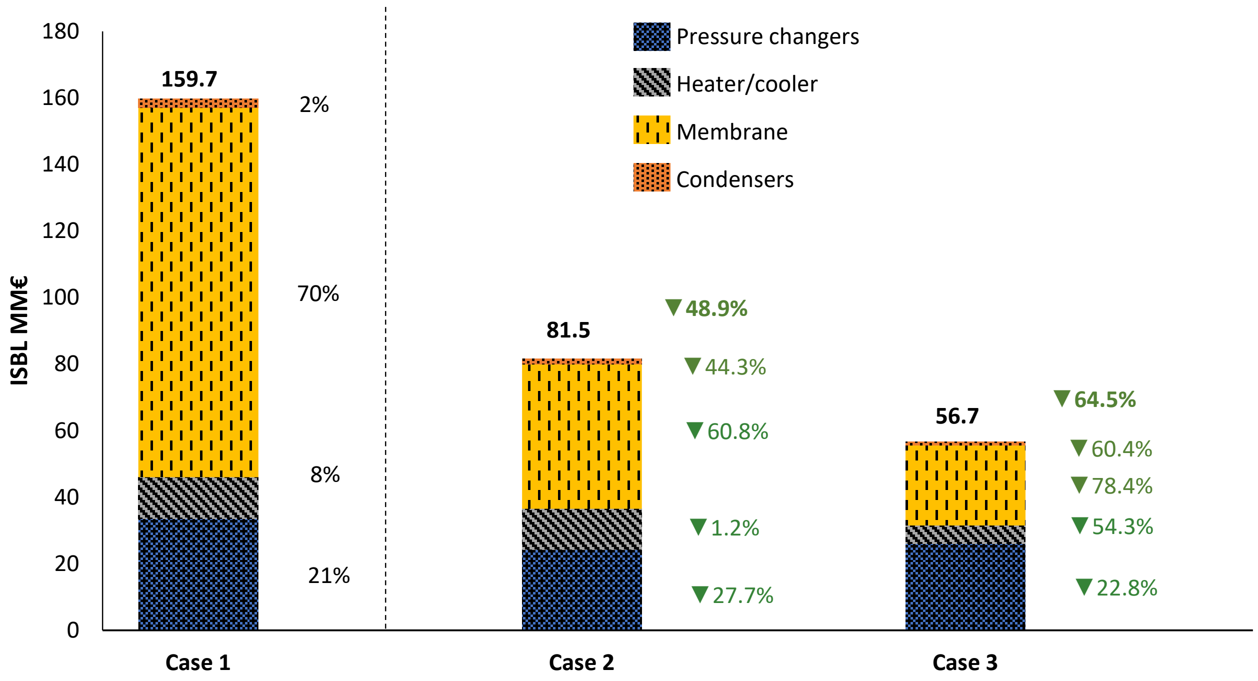
Parameter	Case 1	Case 2	Case 3
P_1 [bar_a]	35	70	110
P_2 [bar_a]	5	10	15
Area MEM-1 [m^2]	110000	35000	20000
Area MEM-2 [m^2]	260000	110000	60000
E %-net power plant	12.4%	9.1%	9.3%

(Confidential & Proprietary)

Workshop IFE
23-06-2022

Simulation results (CAPEX)

- Membrane cost → 150 €/m²
- Membrane length → 0.5 m



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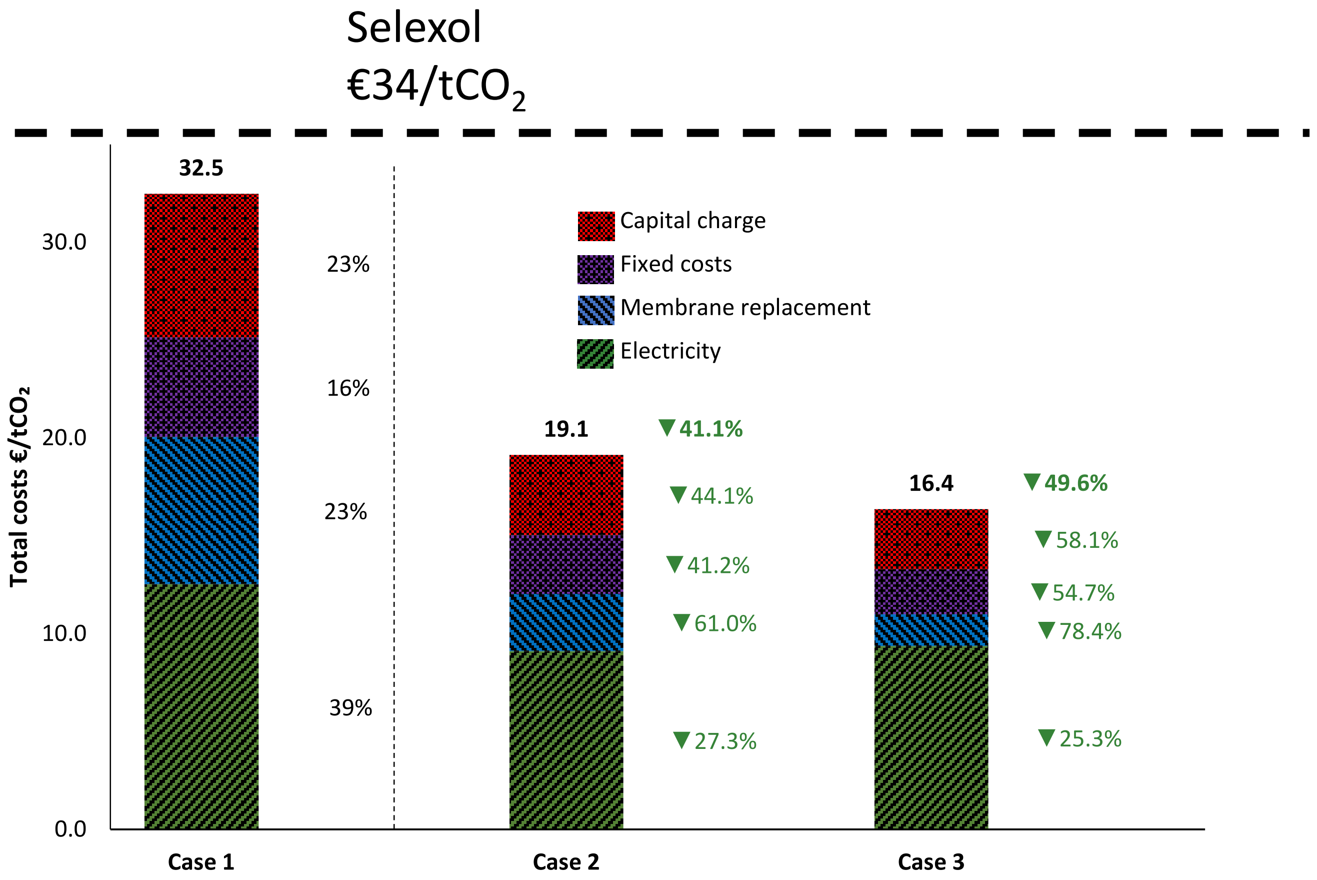
Simulation results (CAPEX+OPEX)

- Replacement → 2 yr
- Electricity → €68.8/MWh
- Operation → 8000 hr



Conclusions and outlook for pre-combustion carbon capture

- Higher feasibility than conventional Selexol
- Relatively low energy consumption

- Module design
- High pressure membranes
- Potential for blue hydrogen production

Overall conclusion

- The use of MMM seems a good fit for pre-combustion carbon capture
- Post-combustion MMM need further development
- Large scale module designs are necessary before further development

Prototype – post combustion CCS

(Confidential & Proprietary)

Prototype – pre-combustion CCS

Questions

hans.ten.dam@hygear.com

2.5. Pd-based membranes production (José Luis Viviente – TECNALIA)

Workshop on Advanced Membranes and Membrane assisted processes for pre- and post combustion CO_2 capture

Kunnskapsbyen Conference Center, June 23rd, 2022

(Gunnar Randers Vei 24, 2007 Kjeller, Norway)

Pd-based membranes production

https://member-co2.com/

Speaker: joseluis.viviente@tecnalia.com

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 760944

Duration: 4.5 years. *Starting date: 01 January 2018*

Budget: € 9 596 541,50 *EU contribution: €7 918 901*

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Outline

1. Membranes for H_2 separation
2. Properties
3. Membrane preparation
4. Membrane performance
5. Membrane production in MEMBER
6. Cost Analysis
7. Who we are
8. H2SITE

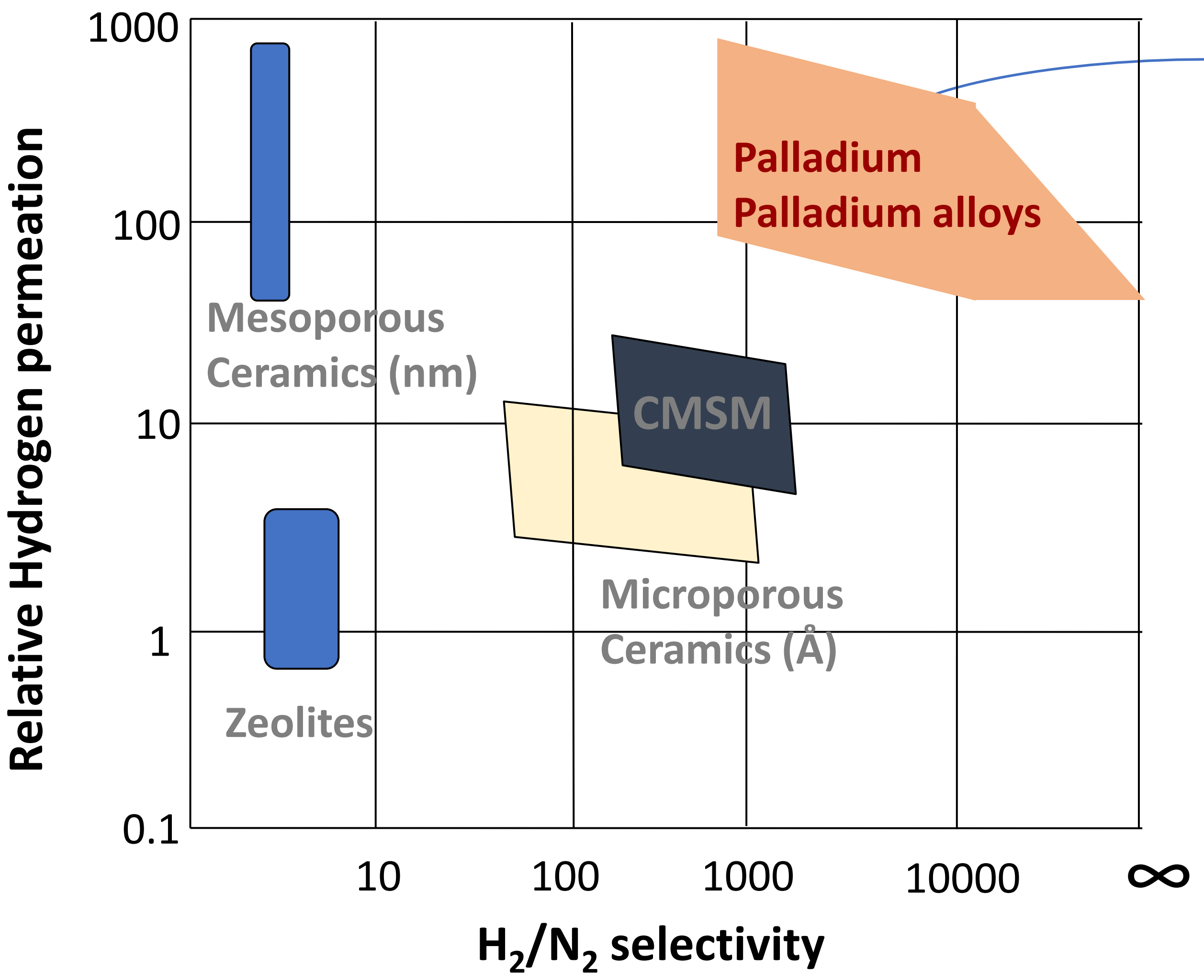
1. Membranes for H_2 separation

Membrane classification:

- Membrane classification
  - Nature
    - Synthetic
      - Inorganic
        - Ceramic, metallics, carbon, zeolites, glass etc.
      - Organic
    - Biological
  - Structure
    - Symmetric
    - Asymmetric
  - Geometry
    - Tubular
    - Hollow fiber
    - Flat sheet
  - Transport mechanism
    - Dense
      - Solution diffusion
      - Facilitated transport
    - Porous
      - Knudsen, molecular sieving, Surface Flow etc.

Chapter 2: Pd-based Selective Membrane. State-of-the-Art, A. Basile et al.
M. De Falco et al. (eds.), Membrane Reactors for Hydrogen Production Processes,
DOI: 10.1007/978-0-85729-151-6_2, Springer-Verlag London Limited 2011

1. Membranes for H_2 separation



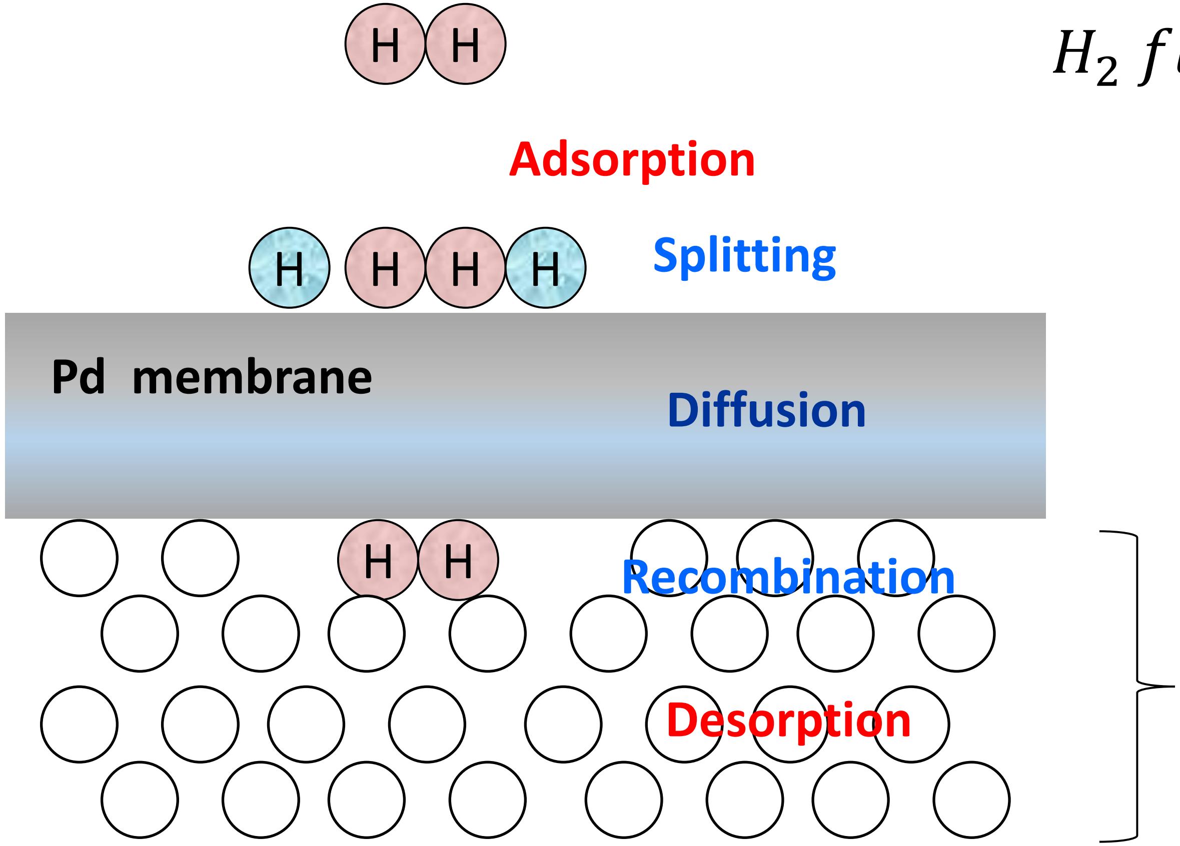
Why Palladium?

Defect free Pd membrane:

∞ selective to H_2

2. Properties

Diffusion mechanism: Solution-diffusion



$$H_2\ flux = J_{H_2} = \frac{P_e^0}{\delta} e^{-\frac{E_a}{RT}} \left(P_{ret}^n - P_{perm}^n\right)$$

P_e^0 : Pre-exponential factor of H_2 permeability (mol m^{-1} s^{-1} Pa^{-n})

δ: Membrane thickness (m)

n: n-value f(limiting step)

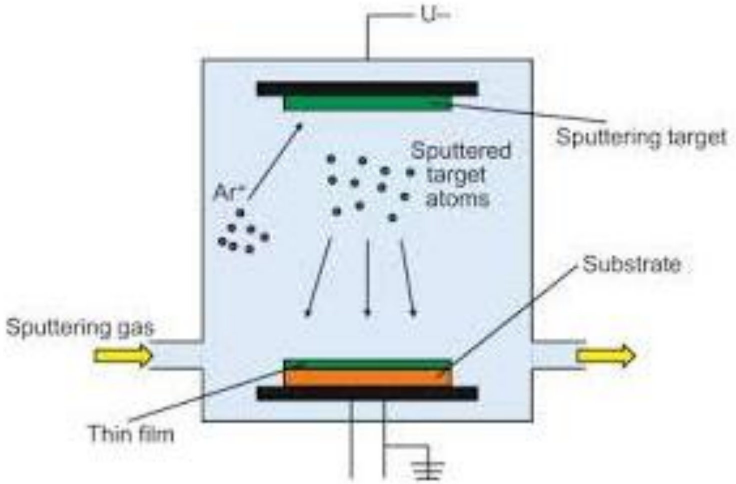
n= 0.5 (Bulk)

n= 1 (Surface)

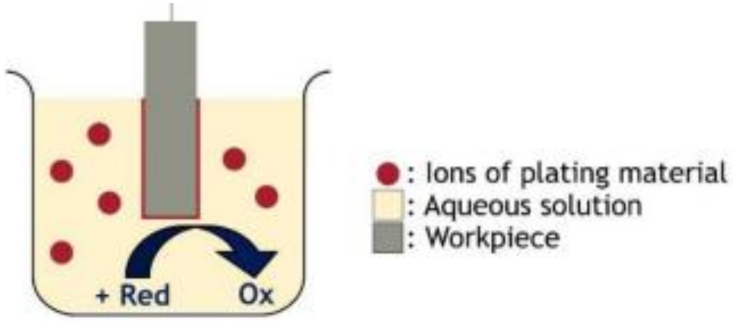
Defect free Pd membrane:
∞ selective to H_2

Fabrication techniques (supported membranes)

Dry techniques
- **PVD** (Plasma vapor deposition)
- **CVD** (Chemical vapor deposition)
- **Spray pirolysis**



Wet techniques
- **ELP** (Electroless plating)
- **EP** (Electroplating)



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3. Membrane preparation

Fabrication techniques (supported membranes)

Technique	Pros	Cons
PVD	• Used for many metals • High deposition rate • Control of thickness and composition of alloys • No liquid wastes	• Expensive equipment • Influence of support geometry (shadowing)
CVD	• Complex geometries	• Low deposition rate • Toxic reactants • Small-sclae (complex to scale-up)
Electroless plating	• High deposition rate • Complex geometries • Cheap equipment • Simple operation • Ease of scale up	• For limited number of metals • Limited number of elements in the alloy (ternary alloy difficult)
Electroplating	• High deposition rate	• Support must be conductive • Need of electricity • Mainly used for pure metal (not alloys)

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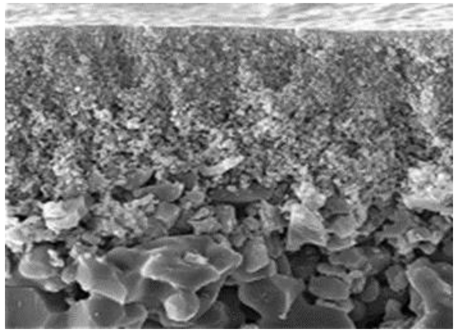
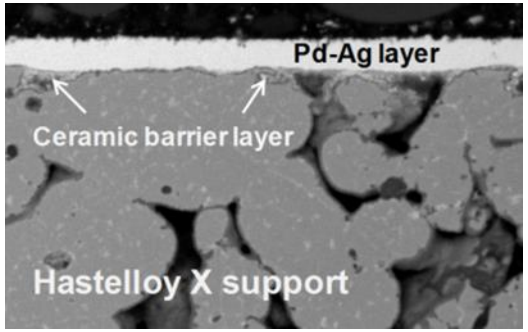
3. Membrane preparation

Importance of the support

	Support material (asymmetric)	
	Ceramic	**Metallic**
Pros	• Low resistance to gas permeation • Small por size • Smooth surface • Less expensive than metallic supports	• Low resistance to gas permeation • Mechanically strong • No problem with sealing • Easy to connect to a reactor • Mechanically strong





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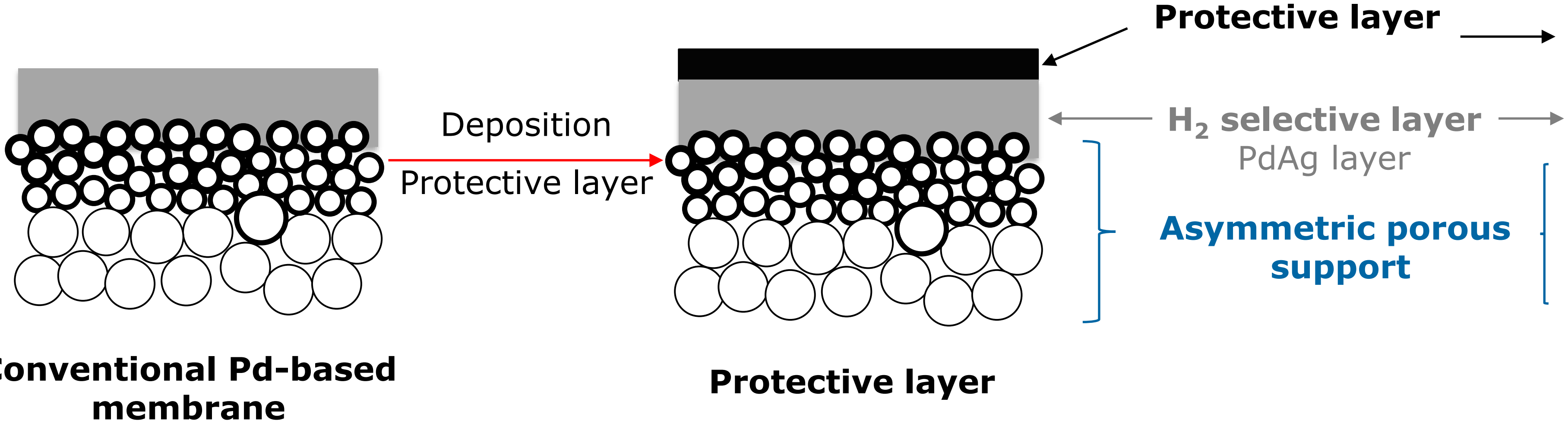
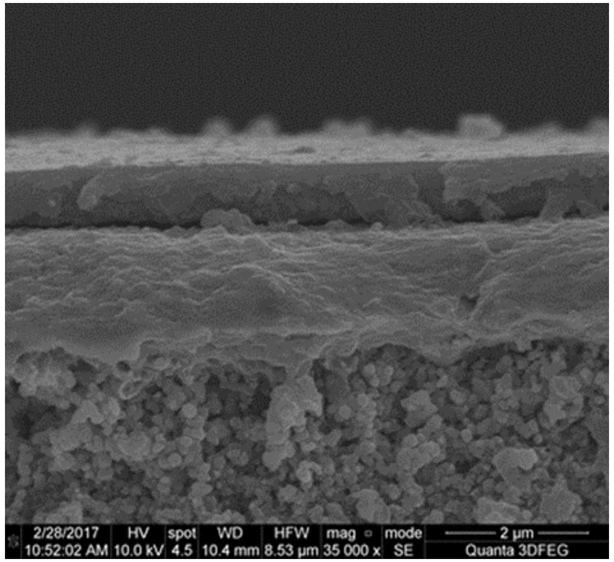
3. Membrane preparation

Thin Pd-based double-skinned (DS) membranes

- high H_2 permeation, selectivity and attrition-resistant



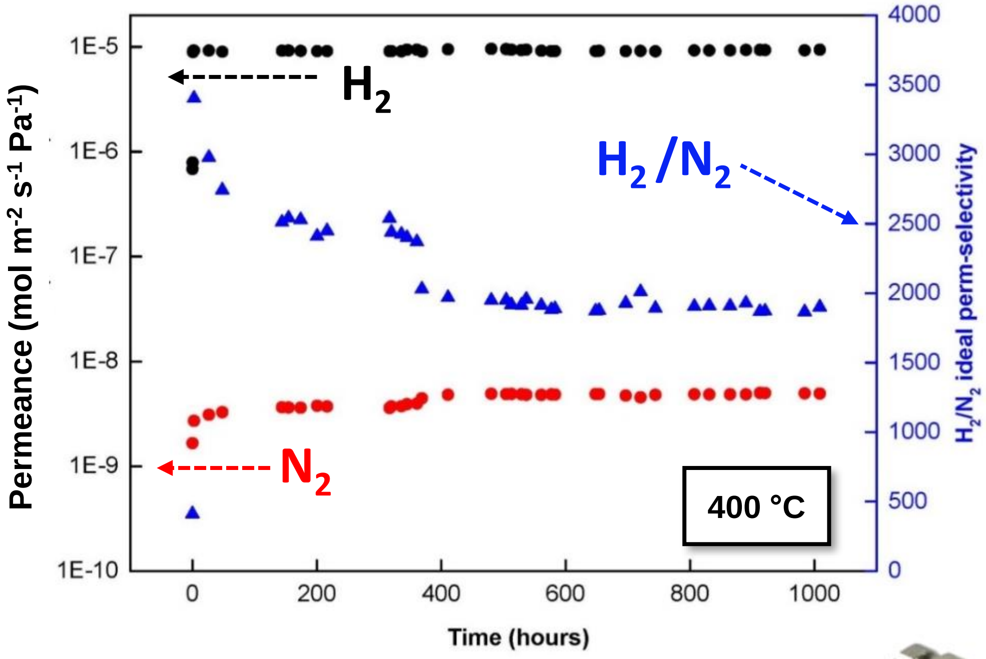


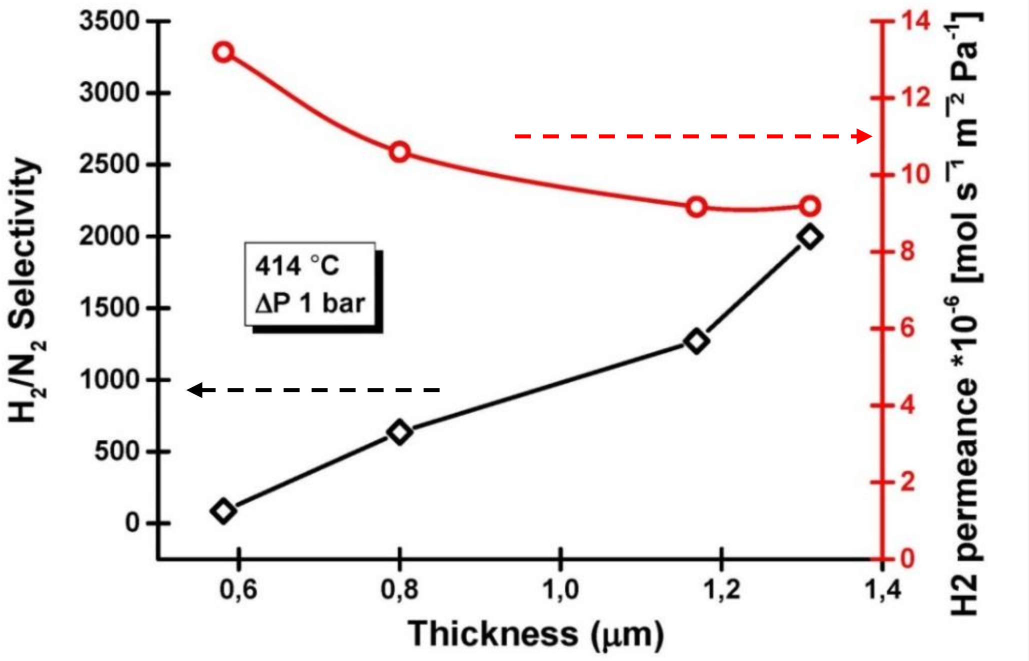
SEM image in cross section of **Pd-based DS membrane**

Alba Arratibel et al. Journal of Membrane Science 550 (2018) 536-544

4. Membrane performance

- Ultra-thin (≤1 μm thick) Pd-Ag membranes (ceramic support)





1.3 μm thick Pd-Ag membrane

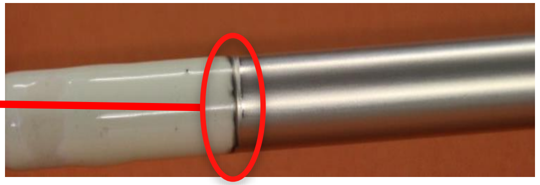
J. Melendez et al., J. Membr. Sci 528 (2017) 12-23

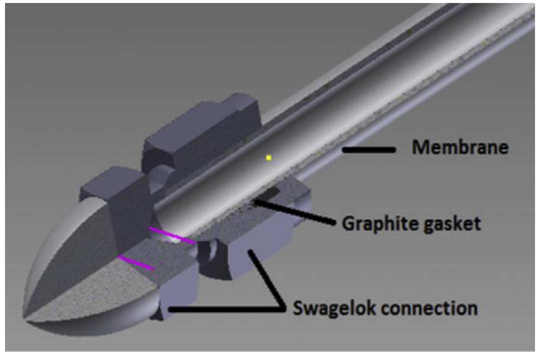
4. Membrane performance

➢ Thin (4-5 µm thick) Pd-Ag membranes (metallic support)

Ceramic support

Leak zone

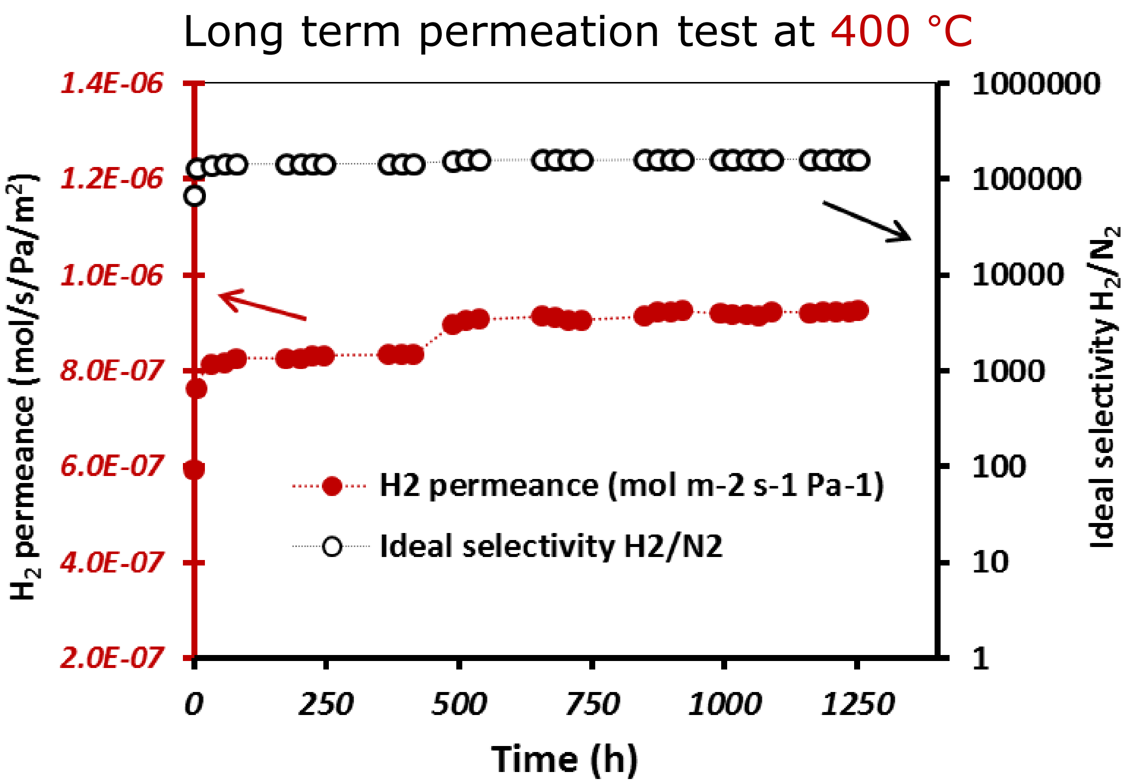




No Leak

No Leak

Metallic support



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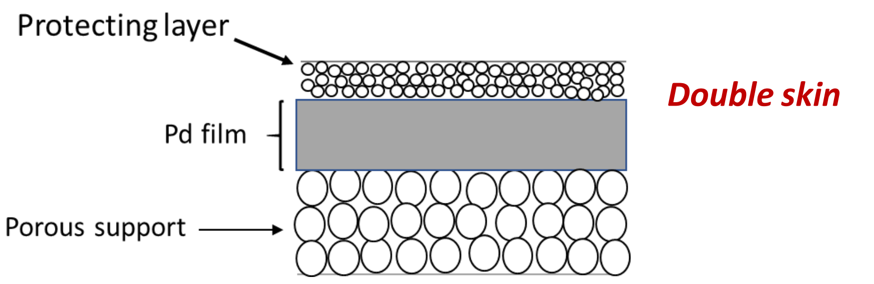
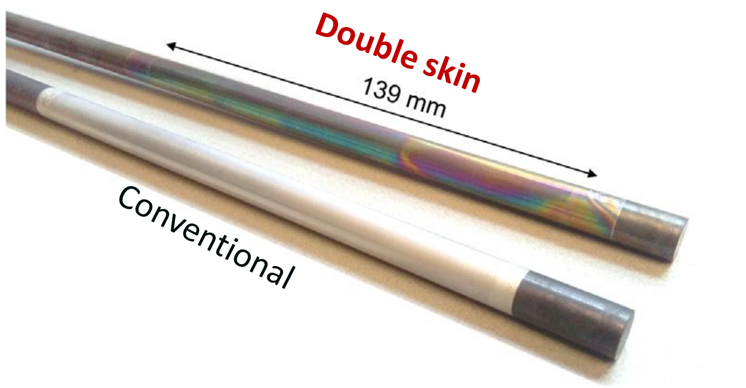
4. Membrane performance

- Double-skin Pd-based membranes

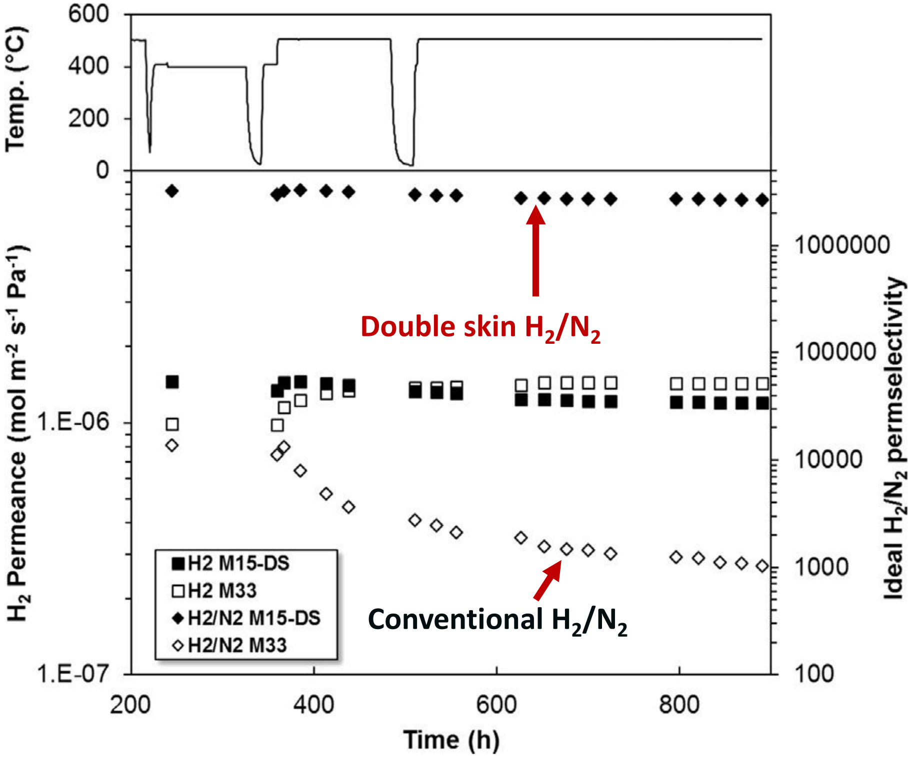
Attrition resistant membranes for fluidized bed Pd MR





H_2 permeation under fluidization

Rh on alumina (100-300 μm size)



A. Arratibel et al., J. Membr. Sci. 563 (2018) 419

5. Membrane production in MEMBER

Optimization of process parameters and scaling-up the manufacturing of supported Pd-based membranes

- Several metallic supported membranes have been prepared by TECNALIA. However, the permeation properties obtained for these membranes were below the defined targets.

- Therefore, in M24 meeting it was decided to manufacture ceramic supported Pd-based membranes for Prototype C. The process parameters for ceramic supported Pd membranes were optimized and the prepared membranes showed suitable permeation properties

5. Membrane production in MEMBER

Manufacturing the Pd-based membranes for the MA-SER prototype reactor and Process control & quality assessment of Pd-Ag membranes.

- 55 sealed Pd-Ag membranes (50 membranes for being integrated in the prototype + 5 spare membranes) have been successfully manufactured by TECNALIA.
- All the membranes showed a N2 leakage limit lower than the defined limit (<2E-10 mol m-2 s-1 Pa-1) and the average leakage was 4.92E-11 mol m-2 s-1 Pa-1.
- The finger parts of the membranes were cut and replaced with metal caps in the case of finger parts with defects. membrane with defects on the finger part.
- **The 55 membranes delivered to TUE**

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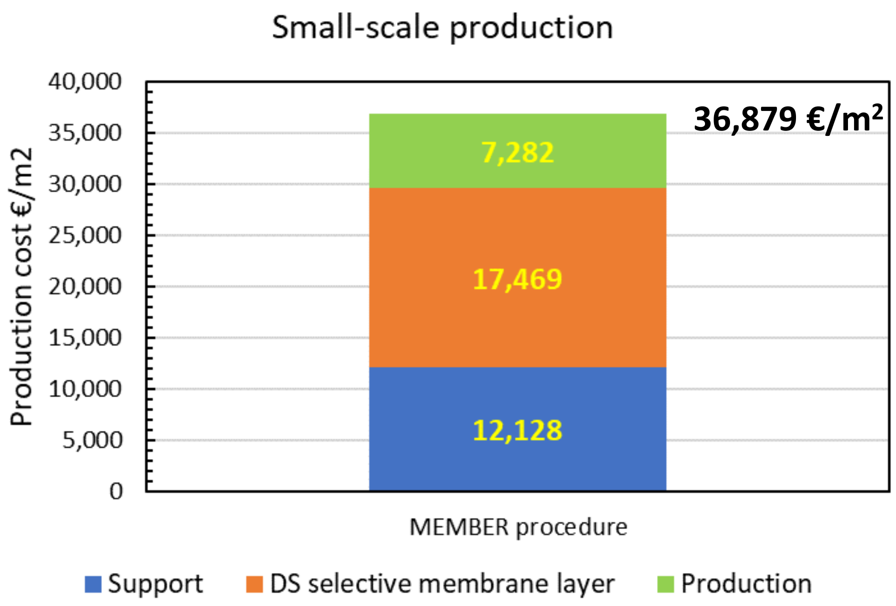
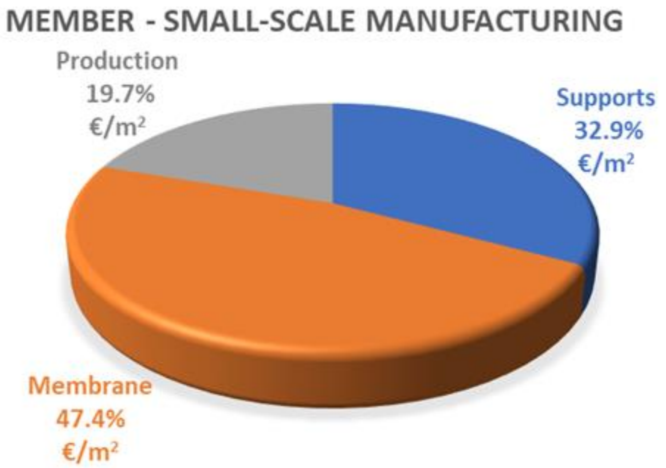
6. Cost Analysis

Small-scale production:

- Porous ceramic support manufacturing:
  - Price per 50 cm long support: 200 €/unit (10,000 €/m²)
  - Sealing connection: Swagelok with graphite ferrule.
- Pd-Ag membrane deposition onto support:
  - Electroless plating (ELP): 1 membrane per bath.
  - 45 cm long effective membrane after sealing.
  - Thickness of the Pd based layer: 5 μm.
  - Personnel cost.
  - Electricity cost.
  - Equipment cost depreciation.
  - Waste management cost.
  - Rejection considered in the calculation.
- Recycling of the membranes (Pd-Ag and supports) and baths have not been considered.





6. Cost Analysis

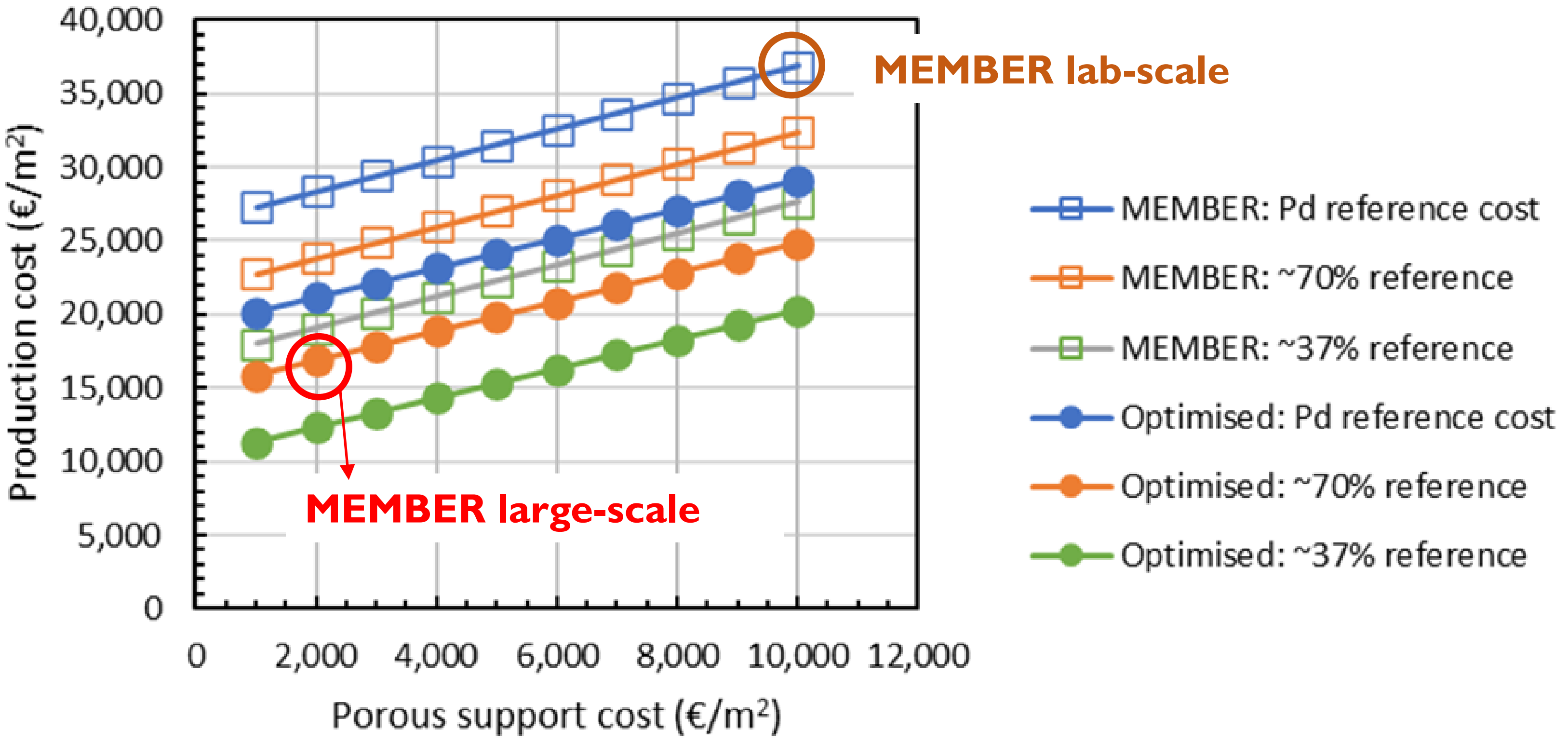
- When applying an optimized procedures at lab-scale (developed in the frame of INNOMEM project) the total cost is decreased to 29,096 €/m^2. This is around 79% of the cost in MEMBER.

6. Cost Analysis

Large-scale production:

- Membrane production cost is mainly depending on the costs of the support and Pd compound used in the ELP process. Both are around 90% of their respective total cost group reported before.
- The large-scale production cost calculated (Oct. 2021) considering the impact of the cost of the porous support, Pd compound and process optimization.

Dependence of the membrane production cost on the porous support and Pd compound costs



- large-scale production cost : 16,855 €/m^2 (46% of the lab-scale production cost)
- Lowest value: 11,319 €/m^2 (31% of the lab-scale production cost)

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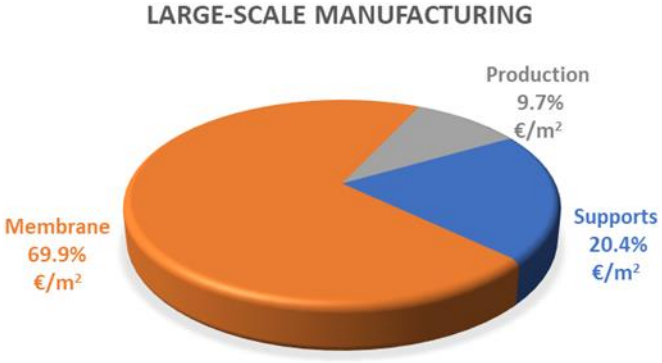
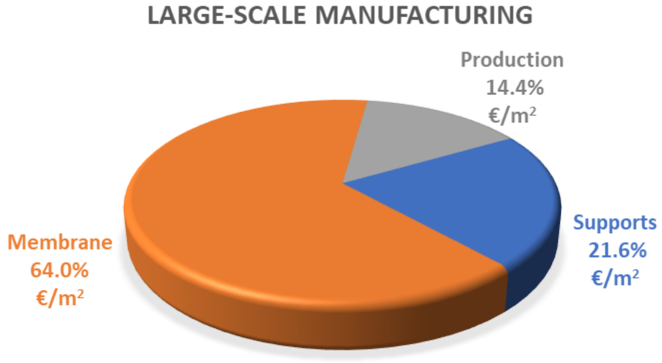
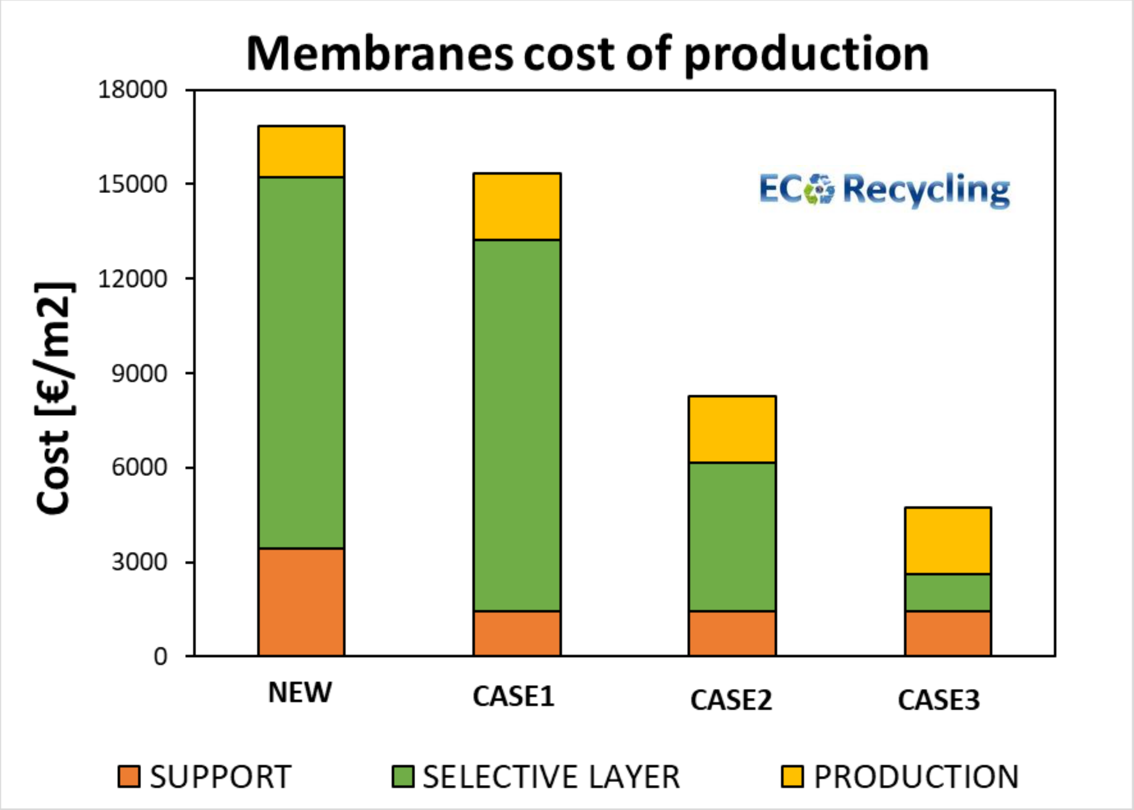
Large-scale production:

- Recycling of the membranes (Pd-Ag and supports) and baths should be considered (90.3% or 85.6% of the total costs)







7. Who we are

Membranes Technology and process intensification. What you need?

- TUe and Tecnalia form a unique team that takes advantage of synergistical effect between membranes and reactors.
- Together, we share a team of more than 25 researchers that cover the entire value chain around process intensification through membrane reactors: Process design & optimization, membrane and reactor development and scale up, prototype development and validation and Techno-economic analysis.
- The team gathers more than 11 years collaborating together in the frame of 14 European projects as well as uncountable private initiatives. We own several patents, more than 50 common publications in peer reviewed journals as well as several book chapters.

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8. H2SITE. The origins

H2SITE was born after +10y of **European R&D with over 70M€ invested in the development of advanced membrane reactors**

TECNALIA holds unique *know-how* on membranes, **TUe** holds unique *know-how* on integrated reactors

H2SITE's founders, **TECNALIA and TUe, spearheaded most of this R&D**


First prototypes

H2 SITE
Membrane reactors for H2 generation

Creation

engie

Major EU partner investment

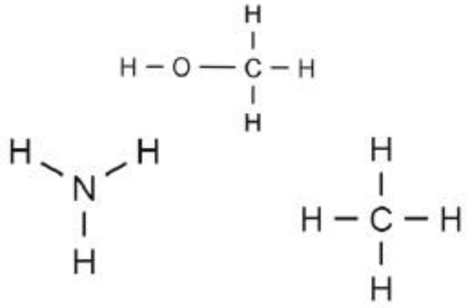
2012 – 2014 – 2016 – 2018 – 2019 – 2020

tecnalia TU/e

DEMCAMER Catalytic Membrane Reactors · FERRET · BIONICO · RE4CELL · MEMERE O2 · PROMECA · Fluid CELL · C2FUEL · HyGrid · MEMBER · MACBETH

8. H2SITE. Solutions to transport H2: carriers and pipelines

1



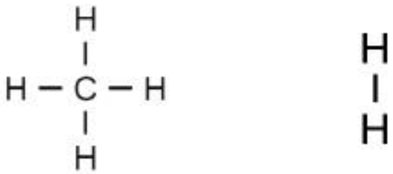
Hydrogen carriers

Ammonia, ethanol, methanol
biogas, formic Acid…

Membrane reactors

Catalytic, fixed or fluidized bed
integrated membrane reactors

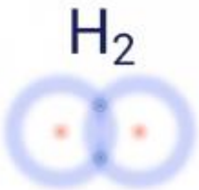
2



Hydrogen blend

As low as 5% H2 – 95% CH4

Membrane separator



Fuel cell grade H2

8. H2SITE. Mission and vision

H2 SITE
Membrane reactors for H2 generation

H2SITE produces **cost-efficient, on-site, renewable** H2 for small and medium consumers in industry and mobility segments using **feedstock-versatile** membrane reactors.

https://www.h2site.eu/en/

tecnalia
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Thank you for your attention

https://member-co2.com/

Contact:
joseluis.viviente@tecnalia.com

Acknowledgement: For the CO2 molecule used in the logo:The original uploader was Frederic Marbach at French Wikipedia [GFDL (http://www.gnu.org/copyleft/fdl.html)

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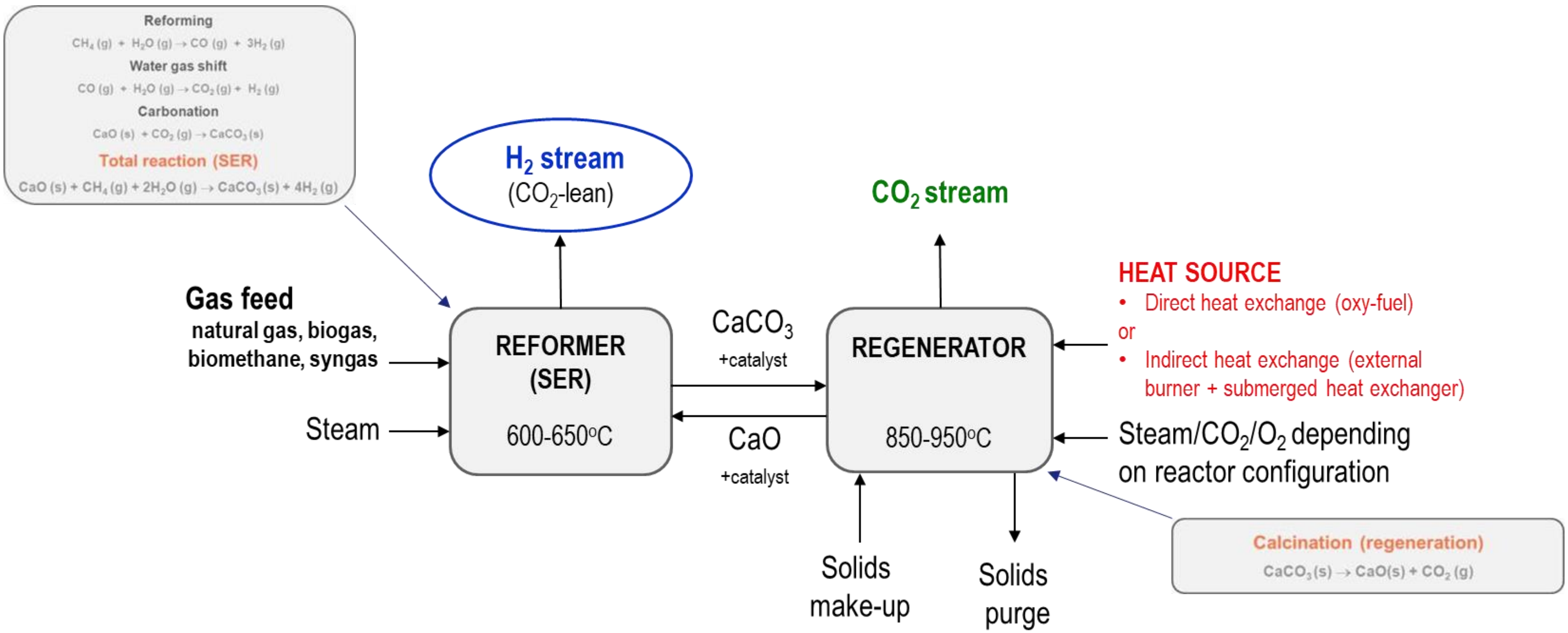
2.6. High temperature sorbent and catalyst for the MA-SER process - Upscaling and performance (Julien Meyer – IFE)

High temperature sorbent and catalyst for the MA-SER process - Upscaling and performance

MEMBER final workshop
23-06-2022

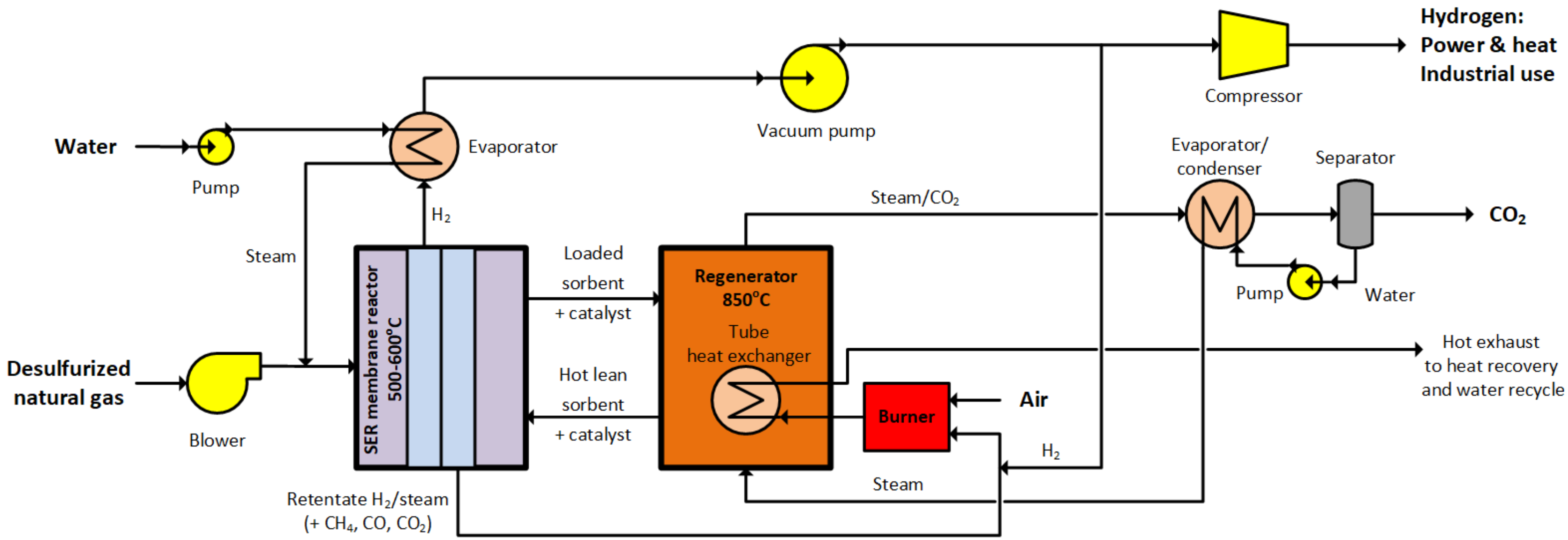
Julien Meyer (IFE), Christophe Voisin (MTEC), Manfred Nacken (C&CS)
Julien.Meyer@ife.no

Sorption-Enhanced Reforming (SER)



- 2 reactors instead of 6 in the conventional reforming process with CO_2 capture
- Heat supplied to the reformer by hot solids and carbonation reaction
- Suited for continuous operation in fluidized bed reactor system

Membrane-Assisted Sorption-Enhanced Reforming (MA-SER)



- Integration of high temperature hydrogen membranes in the reformer
- High-purity hydrogen (>99.99 %)
- No need for downstream purification
- Maximized process intensification

MA-SER
Material requirements

- High temperature CO_2 sorbent
  - CaO-based
  - High sorption capacity
  - High reaction rate
  - High chemical and mechanical stability
  - Suitable for fluidized bed operation (fluidizable)

- Tailor made SER reforming catalyst
  - Ni-based
  - High catalytic activity
  - High reaction rate
  - High chemical and mechanical stability
  - Suitable for fluidized bed operation (fluidizable)

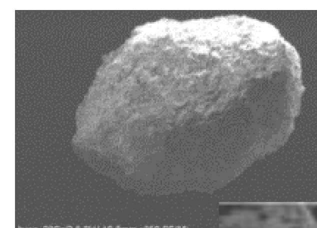
- High temperature hydrogen membranes
  - Dense Pd-Ag based membranes
  - High selectivity
  - Should withstand sorbent and catalyst dust

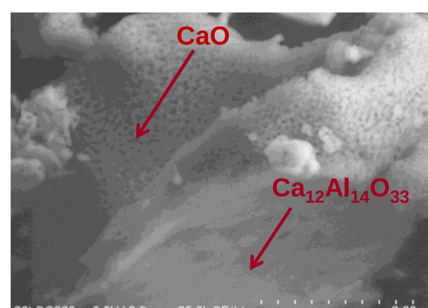
Lab-scale material development and tests towards upscaling

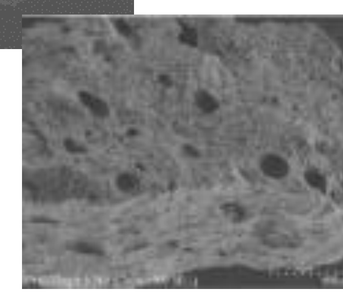
HTSORB CO_2 sorbent (IFE-MTEC)

- CO_2 sorbent using a micro-porous mayenite-based support ($Ca_{12}Al_{14}O_{33}$)
- Low-cost calcium hydroxide and aluminium hydroxide precursors
- Hydrothermal synthesis method to produce a micro-powder
- High-shear granulation to produce granules
- Thermal treatment to remove binder, provide support stability, disperse CaO, and give mechanical strength

- Development work
  - Optimisation of the synthesis parameters
    - Precursor ratio, liquid to water ratio, temperature, reaction time
  - Optimisation of the granulation parameters
    - Binder type and concentration, binder addition, mixing speed and time
  - Thermal treatment
    - Treatment steps, temperatures and time
  - Test methods
    - XRD, BET, SEM, TGA, fluidized bed reactor

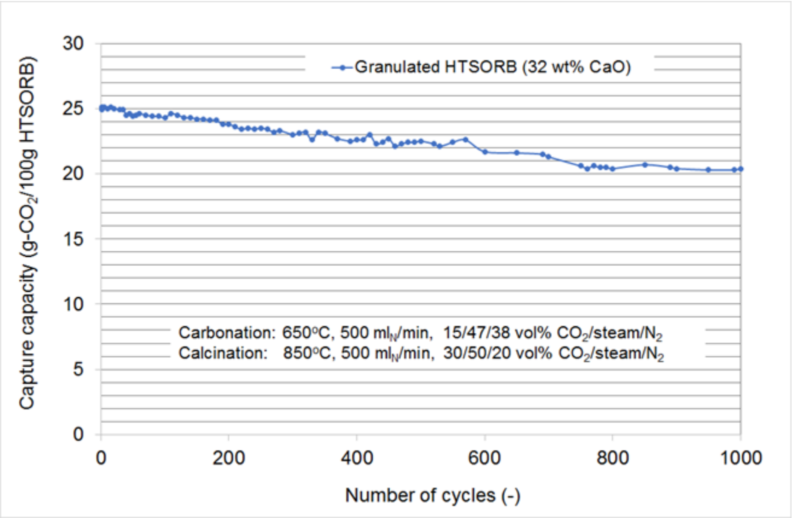


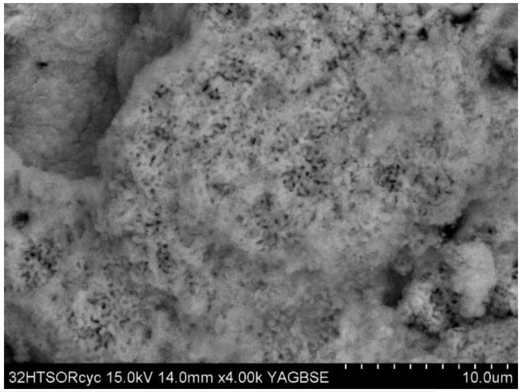


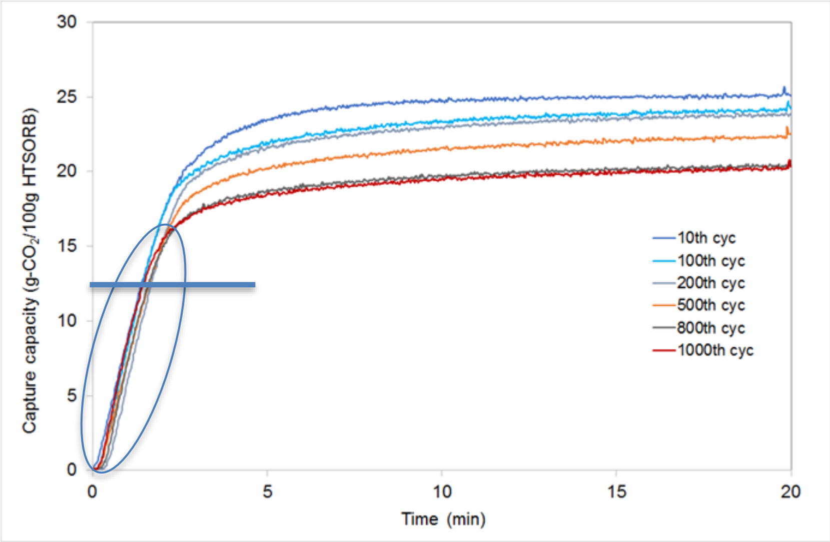


HTSORB CO_2 sorbent (IFE-MTEC)

- Long-term chemical and physical stability test
  - Stable capacity of 0.20 g-CO_2/g HTSORB after 1000 cycles
  - Maximum operating capacity of 0.125 g-CO_2/g HTSORB for fast kinetics
  - Stable reaction kinetics during multi-cycling
  - Very limited sintering





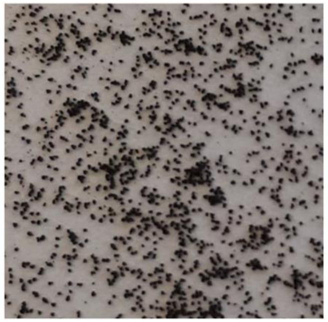


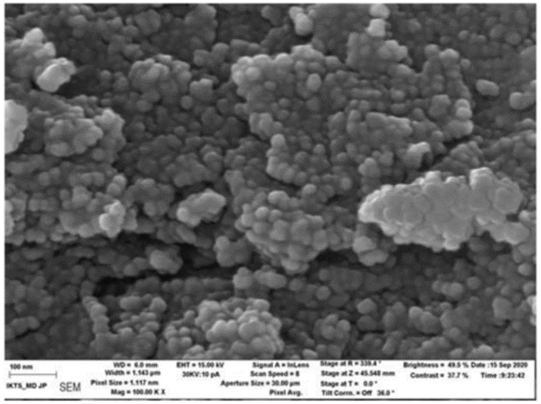
C&CS #1005-Mod2 catalyst

- Magnesium-aluminium mixed oxides support
- Chemical modification of support materials by impregnation method
- Support calcination and sieving
- Incipient wetness impregnation method with Ni precursor
- Calcination and sieving of catalyst material

- Development work
  - Optimisation of support material
  - Optimisation of Ni content
  - Optimisation of sieving yields
  - Test methods
    - XRD, BET, TPR, SEM, TGA, catalytic packed bed reactor & fluidized bed reactor

European Patent application
EP 3805 149 B1



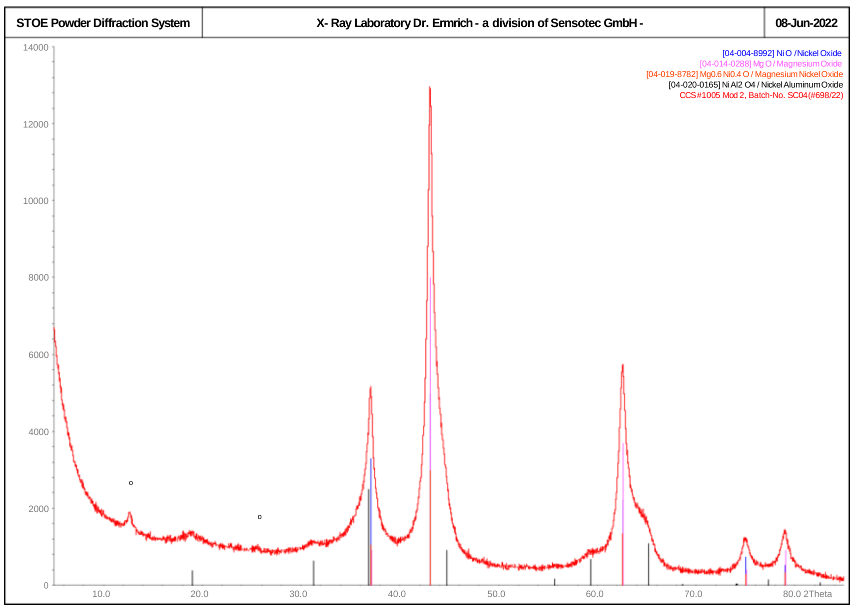


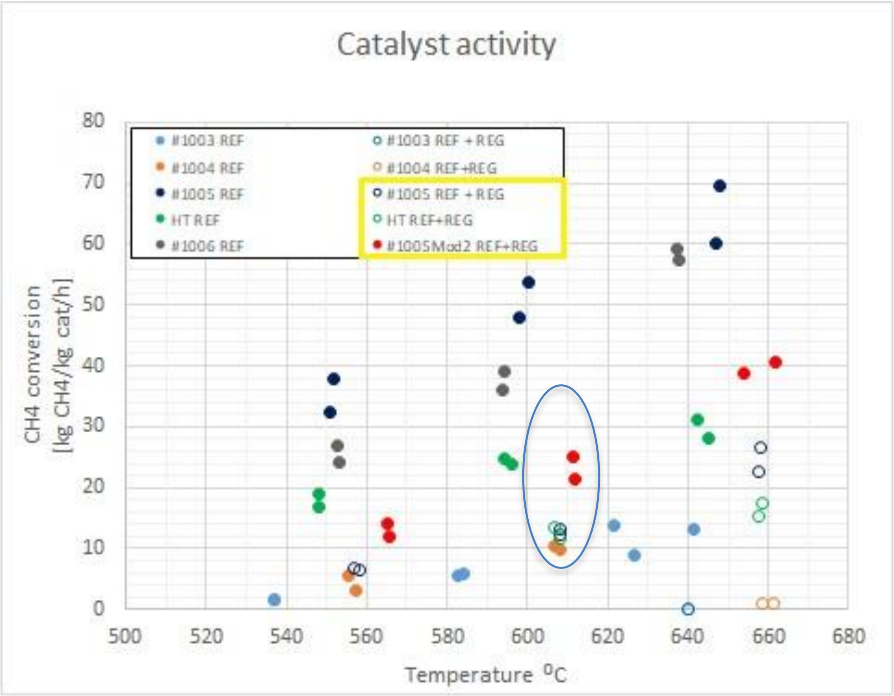
IFE Marion Technologie Nanomatériaux Matériaux Nanostructurés & Poudres Céramiques C&CS catalysts & chemical specialties

C&CS #1005-Mod2 catalyst

- Stable chemical phases during upscaling
  - NiO, MgO, $Mg_{0.6}Ni_{0.4}O$ and $NiAl_2O_4$

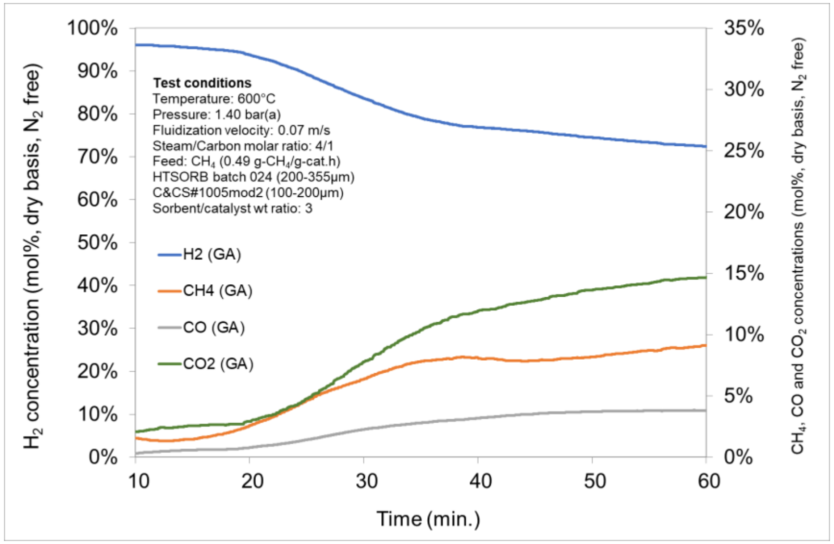
- Satisfactory catalytic activity in relevant cyclic SER conditions

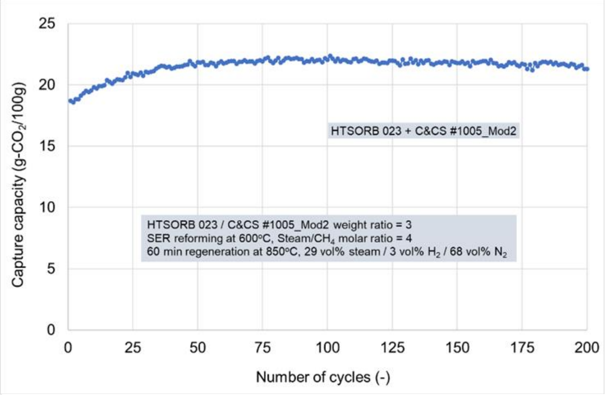


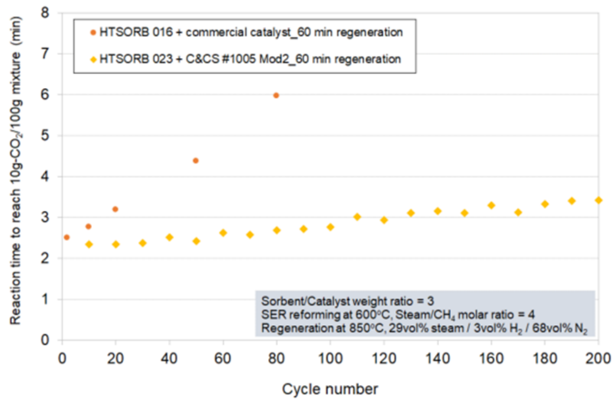


SER performance of HTSORB and C&CS catalyst

- Separate aging test of #1005_Mod2 during 100 h in fluidized bed relevant regeneration conditions
- Followed by SER-FBR test in lab-scale FBR mixed with HTSORB 024
  - 96-94 mol% H_2 during the pre-breakthrough period after 100 h aging
- Satisfactory chemical stability of both HTSORB and #1005_Mod2 in SER-TGA multicycle







Attrition performance of HTSORB and C&CS catalyst

- Air Jet attrition test (ASTM D5757) performed in non-reactive atmosphere (nitrogen) at 850°C
- AJI index lower than 10 wt% for both HTSORB and C&CS catalyst

	AJI 5h wt%
HTSORB batch # 024 (high-shear granulated)	6.4
C&CS #1005 Mod2	9.9
Commercial crushed/sieved reforming catalyst	6.1
Kaolin CP758 (FCC reference material)	4.2
Zeolite/Kaolin JM61 (FCC reference material)	6.4

Upscaled production of HTSORB (MTEC)

$Ca(OH)_2$ precursor

$Al(OH)_3$ precursor

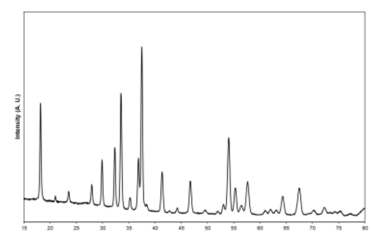
$m(Ca(OH)_2) / m(Al(OH)_3) \sim 1.7$

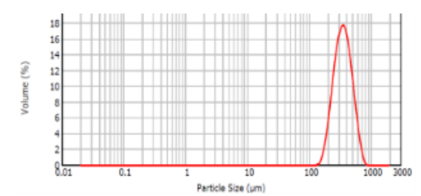
- Mixing in autoclave in demineralized water (120°C / 3h)
  Liquid to Solid ratio: 3
- Centrifugation
- Drying at 100°C
- $Ca(OH)_2$-Hydrogarnet powder

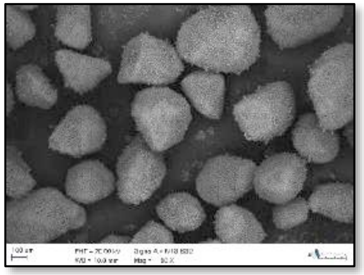
$Ca(OH)_2$-Hydrogarnet powder

- Granulation
  0.145 kg binder/kg powder
- Drying at 100°C
- Sieving
- Thermal Treatment
  1000°C / 1h
- HTSORB granulated powder

- **Optimized granulation parameters:**
  - Temperature: 70°C
  - Blade rotation speed: 100 rpm
  - Binder: Polyvinyl alcohol
  - 14 wt% PVA in binder solution
  - Binder to solid sorbent weight ratio: 2%
  - 0.145 kg binder solution/kg powder

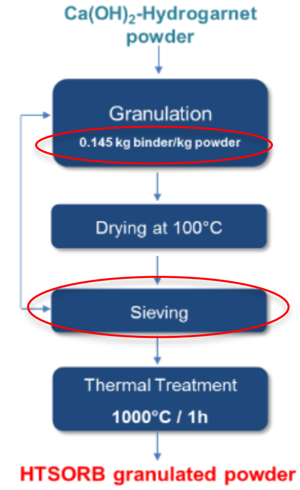






250 kg (HTSORB BATCH 025) delivered for MEMBER test campaign

Process optimization for a larger scale-up of HTSORB



Actual process: Granulation yield **~50%** → not suitable at industrial scale

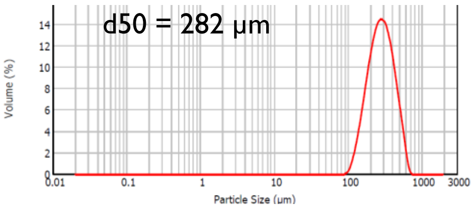
Necessity to increase this yield ➔ **Solution: Re-granulation of the fines particles**

- Re-granulation of the fines particles with **only water**
- Re-granulation of the fines particles with **binder quantity divided by 2:** 10g binder/kg powder (Binder to solid sorbent weight ratio: 1%)

No cohesion between the particles

- Re-granulation of the fines particles with the **same binder quantity:** 20 g binder/kg powder (Binder to solid sorbent weight ratio: 2%)

Obtention of **dense granules (d ~2,7g/cm³)** with the desired particles size distribution



- Next steps for further upscale of HTSORB manufacturing
  - Building of a pilot plant (MTEC, on-going)

Upscaled production of #1005Mod2 (C&CS)

- The production method of C&CS #1005Mod2 at 5 kg scale was applied for the production of 50 kg catalyst for the MEMBER test campaign

Raw material → Chemical modification by impregnation → Drying (Support) → Calcination (Support)

↓ 255 kg

Sieving (including crushing) (0.1 – 0.25 mm) → Incipient wetness Impregnation with NiO precursor → Drying → Calcination

58 kg ↓

Final sieving (0.1 – 0.25 mm) → Quality control (BET, XRD)

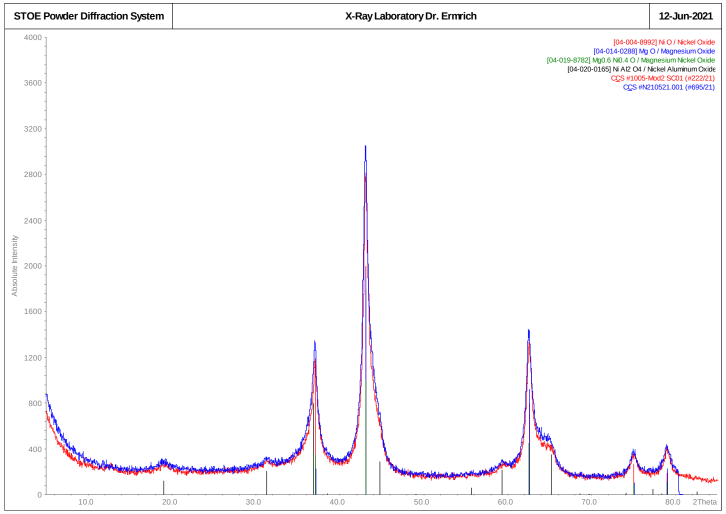
Sieving yield of about 86%

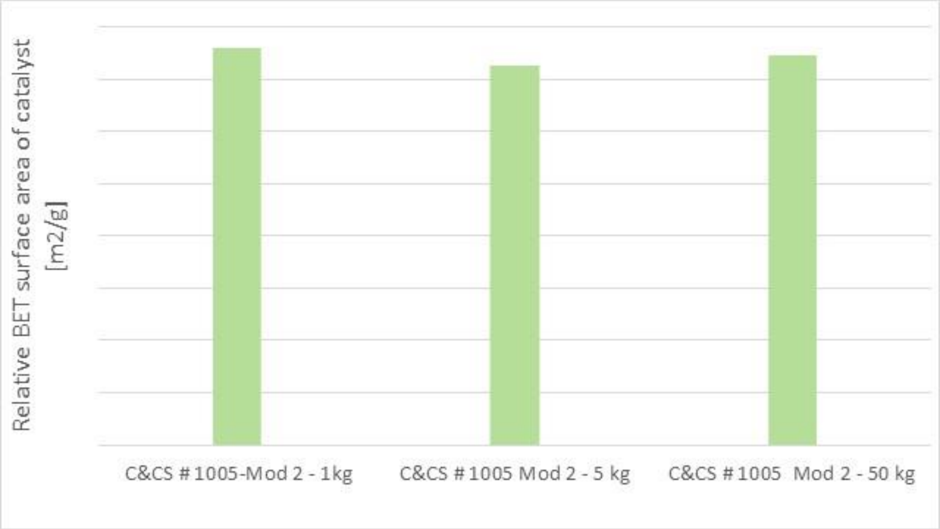
More details are given in the European Patent application EP 3805 149 B1

Quality control #1005Mod2 (C&CS)

- Similar chemical phases identified on 5 kg and 50 kg scales
  - NiO, MgO, $Mg_{0.6}Ni_{0.4}O$ and $NiAl_2O_4$ crystalline phases detected

- Pretty similar BET surface area measurements on 1, 5 and 50 kg scales





- Next steps for further upscale of C&CS catalyst manufacturing

  - Transfer of production method to a lean manufacturer (C&CS, on-going)

Advanced MEMBranes and membrane assisted procEsses for pre- and post- combustion CO_2 captuRe

https://member-co2.com/

Thank you for your attention

Contact:
joseluis.viviente@tecnalia.com

2.7. Sorption Enhanced Reforming (Arnstein Norheim – ZEG-POWER)

Z·E·G

ZEG Power

Clean hydrogen from gas – Sorption Enhanced Reforming

MEMBER Workshop - June 23rd 2022

Arnstein Norheim, CTO

Z·E·G

ZEG delivers solutions for clean hydrogen production using the novel ZEG ICC™ Technology

Company

Technology developed through 20 years by top experts in the field

ZEG has received strong industrial and public support

Initial R&D and Technology Development | **Industrialization and Commercialization** | **Scale-up**

- **2001**: Initial research on technology initiated by IFE and CMR
- **2005**: ZEG power established as a Joint Venture by IFE and CMR
- **2010**: ZEG pilot plant (30kW) established at IFE Hynor, a Hydrogen Technology Test Center in Lillestrøm, Norway. Technology and system integration tested and verified
- **2010**: Stratel and Abelsnes private investment
- **2016**
- **2017**
- **2018**: StartupLab and private Investments incl. CEO, CFO, chair
- **2019**: Strategic Partnership Agreement w/CCB
- **2020**: NOK 130m (USD 15m) Capital raise
- **2020**: Plant configuration and solids development
- **2021**: NOK 77m Enova grant
- **2021**: Hynor pilot plant for operational testing
- **2022**: ZEG H1 upscaling project
- **2023**: ZEG H15 upscaling project
- **2023**: ZEG H1 commissioning of first commercial plant at CCB Energy Park
- **2024**: First ZEG H15 sale expected
- **2025**: First ZEG H3 sale expected
- **→**

Executed | In progress

Proven potential of technology

ZEG ICC™ Technology verified in pilot plant

Proven feasibility of larger plant

Z·E·G

ZEG is supported by global investors financing the energy transition

AP Ventures and SPARX are backed by global hydrogen frontrunners

15%

APVentures
ADVANCE & PIONEER

- One of the world's largest hydrogen-focused venture capital funds
- Manages VC funds which invest in advanced technology companies aiming to tackle global environmental and sustainable challenges
- Has a portfolio of investments with a focus on the hydrogen value chain from production through to end use

Selected investments

hydrogenious LOHC TECHNOLOGIES
hystar
HRS
Starfire Energy
PLUG POWER

Key LP's

- **Anglo American:** co-developing the world's largest hydrogen-powered mine haul truck
- **Sumitomo:** promotes hydrogen related businesses
- **Mitsubishi:** offers world's first standard packages for green hydrogen integration
- **Equinor, Yara and Nysnø**

11%

SPARX

- General partner to the Mirai Creation Fund I & II
- Sparx Mirai Creation Fund targets private start-ups across intelligent technologies, robotics, technologies that promote hydrogen economy, electrification and new materials

Selected investments

Exergy Power Systems
FLOSFIA
Japan H2 Mobility
MEIJO NANO CARBON

Key LP's

- **Toyota Motor:** one of the world's largest FCEV manufactures, with a production goal of over 30k cars in 2021
- **Sumitomo Mitsui Banking:** provides leasing of hydrogen facilities such as hydrogen refueling stations

21%

IFE

- Highly recognized research institute within energy technology, and the founder of ZEG Power
- IFE Invest AS is IFE's venture arm, with the purpose of commercializing spin-off companies from IFE through active ownership follow-up

Selected Investments

visavi
GIAMAG TECHNOLOGIES
SUNPHADE
wirescan

12%

nysnø Climate Investments

- 100% state-owned investment company, focused on renewable energy, resource efficiency, enabling technologies, sustainable demand and circular economy
- Nysnø invests in companies that provide profitable and smart solutions to the challenges of climate change

Selected Investments

TORGHATTEN
OTOVO
eSmart SYSTEMS
DISRUPTIVE TECHNOLOGIES
NorSun

Other key investors

Danske Invest
ccb energy holding
STARTUP LAB
STRATEL
CX CO2 MANAGEMENT AS
Nordea ASSET MANAGEMENT
ZEG Employees

Recent news flow

Shareholding (fully diluted)

Our Team

With extensive track-record of building profitable deep tech businesses

ZEG organisation

- 18 employees
- 4 PhDs
- 12 MSc
- Broad experience from the energy business

Key management team

Arild Selvig
CEO

Technip FMC, Hydro, FMC

Kathrine K. Ryengen
CCO

Norsk Titanium, Scatec, Accenture

Alexandra Shabasheva
Director Legal & Contract

Technip FMC, ENFLOW

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CTO

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IFE, Visavi

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SASOL

Sustainability is the core of ZEG Power

The UN Sustainable Development Goals are the blueprint to achieve a better and more sustainable future for all

- ZEG unlocks the energy of natural gas
  – Goal 7: affordable and clean energy
- ZEG is based on patented technology for clean hydrogen production with integrated CO_2 capture
  – Goal 9: industry, innovation and infrastructure
- ZEG uses natural sorbent to capture CO_2 with no toxic emissions to air or water
  – Goal 12: responsible consumption and production
- ZEG works to achieve carbon removal, using biogas as feedstock combined with CCS
  – Goal 13: climate action

Technology

Z·E·G is a pure-play clean hydrogen company

Vision: To empower the world with clean energy

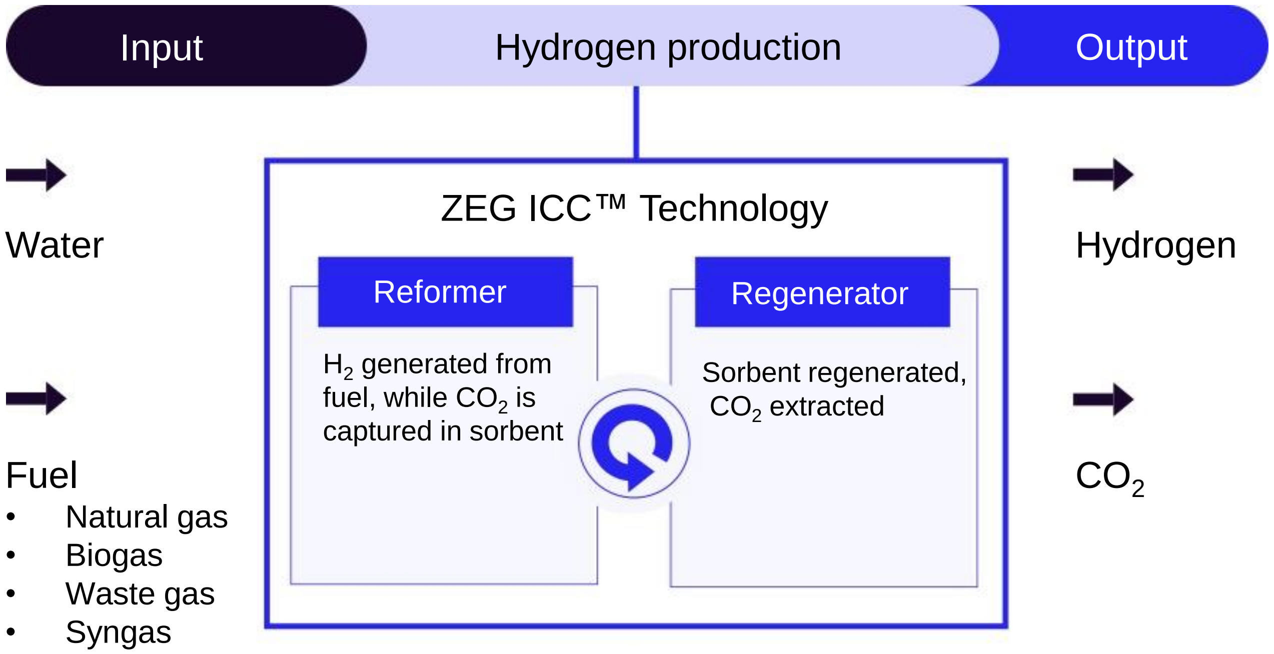
90-99%	<$1,5/kg	NOK 210m (USD 24m)
CO_2 capture[1]	levelized cost of hydrogen[2]	R&D since 2001

- ZEG provides systems to produce clean hydrogen from hydrocarbon gas with proprietary integrated carbon capture technology
- ZEG's target is to provide hydrogen with the lowest cost and carbon intensity to the market, enabled by the ZEG ICC™ Technology
- The **ZEG ICC™ Technology**
  - IP protected across seven approved patent families
  - high thermal efficiency
  - verified at pilot plant scale
  - first commercial sale secured
  - roadmap to industrial scale established
  - EU taxonomy compliant clean hydrogen

[1] Includes CO_2 from steam generation, regenerator and reformer. Dependent on plant configuration.
[2] 2020 estimates - at industrial scale

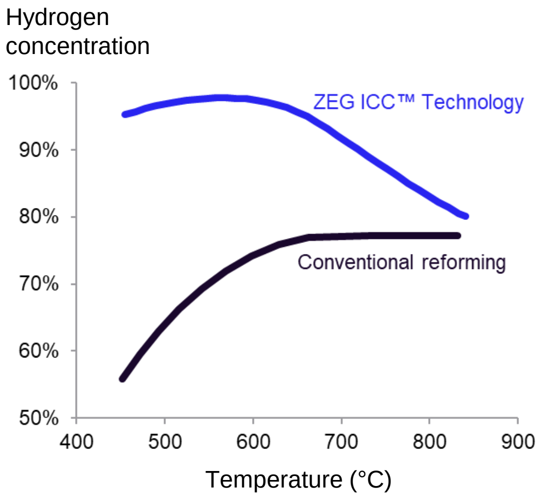
ZEG offers a very competitive route to clean hydrogen

High yield hydrogen - integrated CO_2 capture



Uniqueness of the ZEG ICC™ Technology

- Captures the CO_2 inside the reformer where the CO_2 concentration is the highest, enables high CO_2 capture rate
- Increases the yield of hydrogen
- Enables high thermal efficiency
- Eliminates the need for Water Gas Shift needed in traditional reforming, creating a step change reduction in footprint, driving size, CAPEX and OPEX down



Z·E·G

ZEG ICC™ Technology vs. conventional blue hydrogen

Eliminates four process steps compared with steam methane reforming with amine-based carbon capture

Process flow

Steam methane reforming w/carbon capture

Treated feedstock including fuel and water →

1. **Pre-reformer** – Any hydrocarbons present in feedstock converted to methane
2. **Reformer** – Gas reformed at high temp. to produce hydrogen and mixture of CO, CO_2 and H_2O
3. **HT shift reactor** – High temperature shift to convert CO to CO_2 and hydrogen by excess steam
4. **LT shift reactor** – Low temperature shift to convert remaining CO to CO_2 and hydrogen
5. **CO_2 absorber (MDEA)[1]** – CO_2 capture using amine-based solvent
6. **CO_2 stripper** – CO_2 release and amine regeneration
7. **Pressure swing absorber** – Hydrogen purification to 99.999% hydrogen

→ Hydrogen
→ CO_2

ZEG ICC™ Technology

Treated feedstock including fuel and water →

1. **Reformer** – Hydrogen production with Integrated carbon capture enhancing hydrogen yield
2. **Regenerator** – CO_2 release and solid sorbent regeneration
3. **Pressure swing absorber** – Hydrogen purification to 99.999% hydrogen

→ Hydrogen
→ CO_2

- High yield production
- Clean hydrogen production
- Attractive cost of hydrogen
- Scalable and flexible

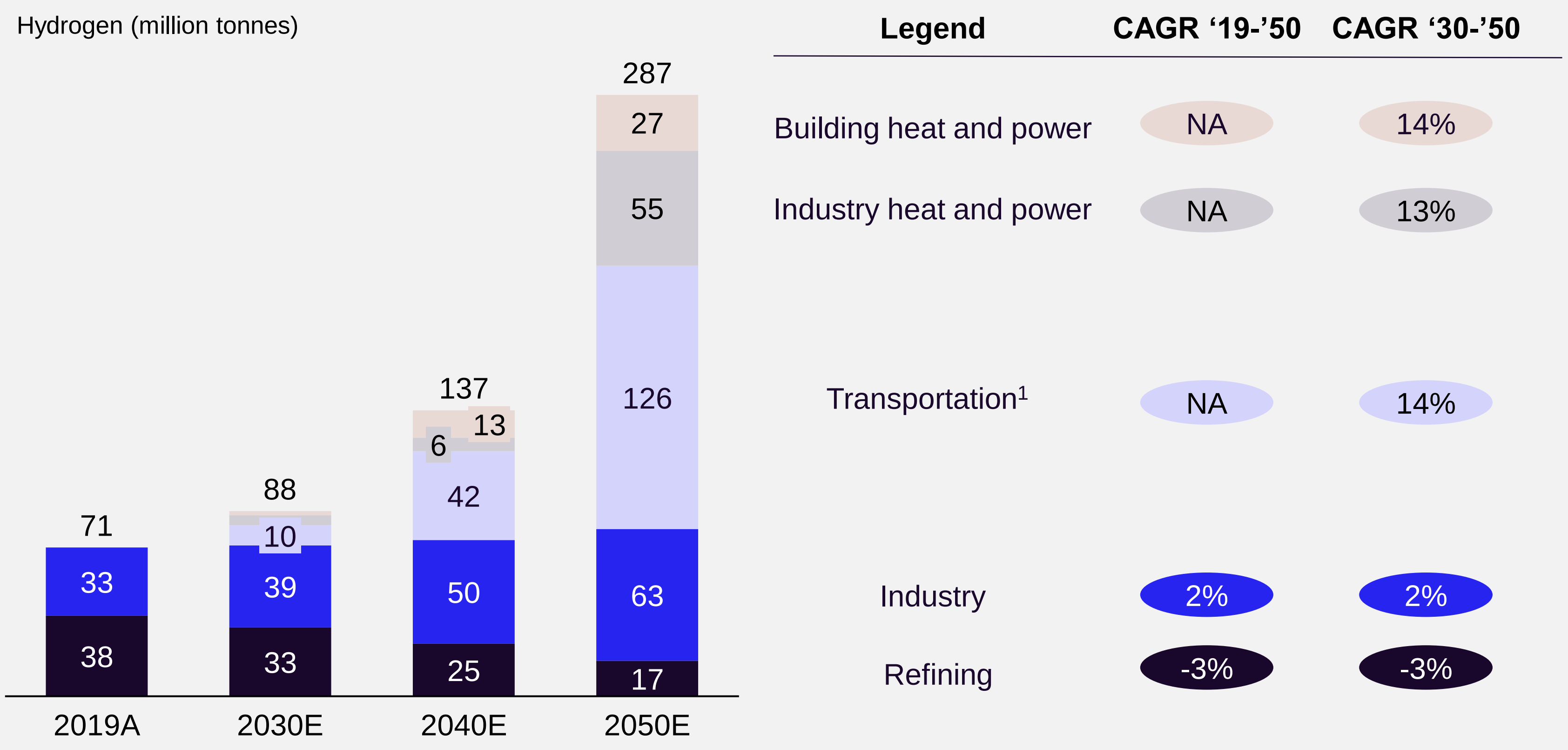
Z·E·G

Market

Hydrogen demand quadruples as economy is decarbonised

Incremental growth expected in Industry, major growth in Transportation and Heat & Power

Global hydrogen demand by application



Source: IEA 2020

1) Transport expected to account for 44% of hydrogen demand in 2050, including hydrogen used in fuel cell electric vehicles, synthetic fuel and ammonia for ship fuel

Project

First EU taxonomy-compliant blue hydrogen plant

- First customer – H2 Production AS, a subsidiary of CCB Energy Holding AS - a Norwegian clean industry hub developer
- The ZEG H1 plant has ~1 ton/day hydrogen production capacity
- NOK 77m of Enova grant funding awarded to the project
- Construction underway with EPC partner Zeton
- The ZEG H1 plant will be production ready in Q4 2022 and commissioned in early 2023
- Letter of intent signed for further expansion on same site with a ZEG H15 plant with ~15 ton/day hydrogen production capacity

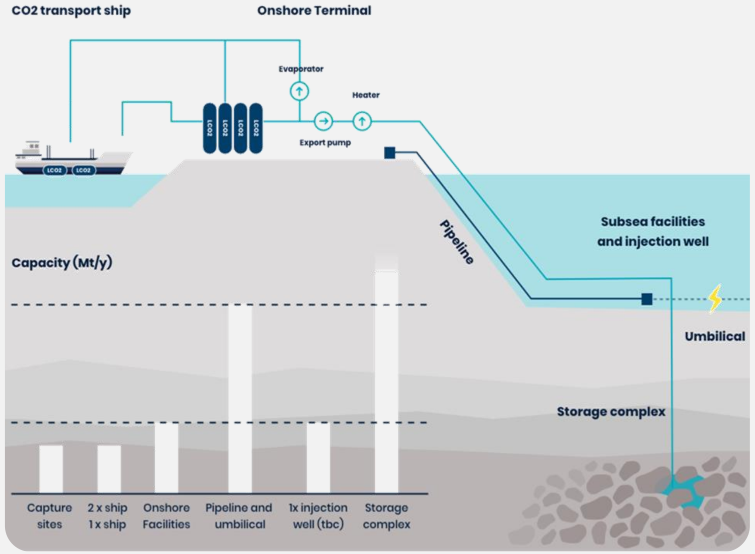
H2 Production AS Customer of first ZEG H1	**Q4 2022** Production ready
ZETON Under construction	**NOK 77m** (USD 8.85m) Supported by Enova grant

Z·E·G

First commercial ZEG plant located at a sweet spot for blue hydrogen

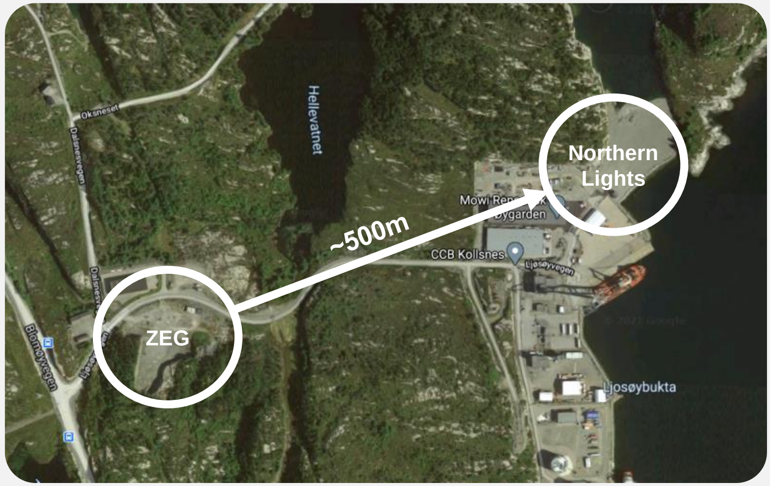
Proximity to Northern Lights CO_2 storage, natural gas terminal and local hydrogen market

Northern lights project



CCB Energy Park, Kollsnes is also the location for a large-scale CO_2 storage ("Northern lights") to be operational from 2024, a highly strategic national, full-scale CCS led by Equinor, Shell and Total and the Norwegian Government

Proximity to CO_2 offtake



The CO_2 will be captured and stored through the Northern Lights' CO_2 storage terminal, located only ~500m away from the ZEG hydrogen production site

Strong local hydrogen market

The combination of a strong local market for hydrogen and the opportunity to store CO_2 on site means CCB Energy Park is the unique location for clean hydrogen production

Z·E·G

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Strategy

ZEG is currently promoting the following solutions to the market

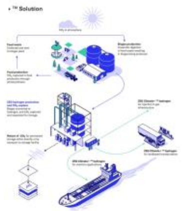
1

ZEG Clean Hydrogen Solution

Sustainably unlocking the value of natural gas

2

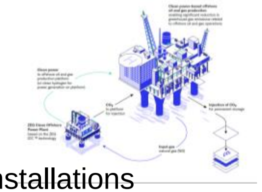
ZEG Climate+ Solution



Enabling carbon removal

3

ZEG Clean Offshore Power Solution



Enables electrification of O&G installations

4

ZEG Clean Methanol Solution

Enabling clean maritime shipping

5

ZEG Maritime Bunkering Solution

Enabling maritime bunkering of hydrogen, ammonia or methanol

6

ZEG Refinery Solution

Decarbonizing hard-to-abate industry

7

ZEG Clean Ammonia Solution

Enabling clean maritime shipping and fertilizer prod.

8

ZEG Clean Steel Solution

Decarbonizing hard-to-abate industry

- (dark blue) Partners identified/ ongoing study, tender or project
- (light blue) Searching for partner(s)

On a clear path towards larger scale …

ZEG upscaling timeline for compact clean hydrogen plants

	1 ZEG H1 – H5 product platform	2 ZEG H10 – H50 product platform
		
Capacity	1-5 metric tonnes hydrogen per day	10-50 metric tonnes hydrogen per day
Footprint	2-300 m^2 (2-3,000 sqft)	4-600 m^2 (4-6,000 sqft)
Turnkey delivery	Q4 2022 and onwards (Project in execution)	Q4 2024 and onwards (LOI entered for first-of-a-kind H15 plant, RCN grant received 2021 with partners)

Z·E·G

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Thank you

2.8. MA-SER reactor for H_2 production with CO_2 capture (Luca di Felice – TU/e)

Modelling of MA-SER reactor for H_2 production with CO_2 capture

by: Luca Di Felice (TU/e)

MEMBER

https://member-co2.com/

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 760944

Contact: *l.d.felice@tue.nl*

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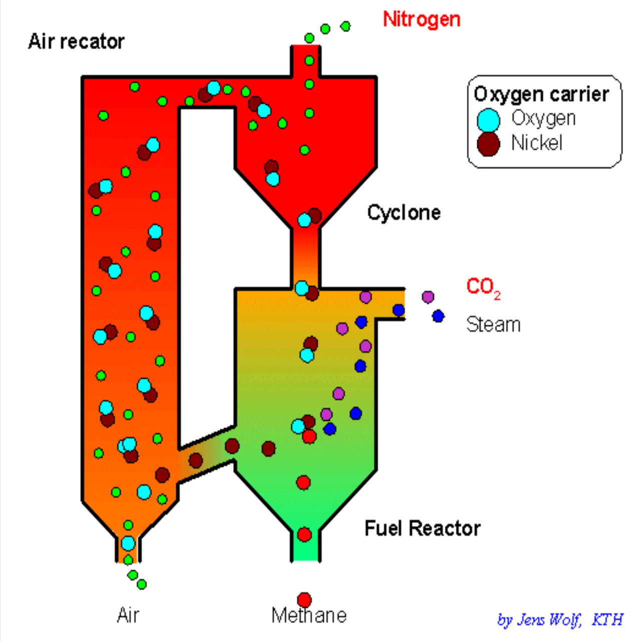
Membranes and membrane reactors group – TUE

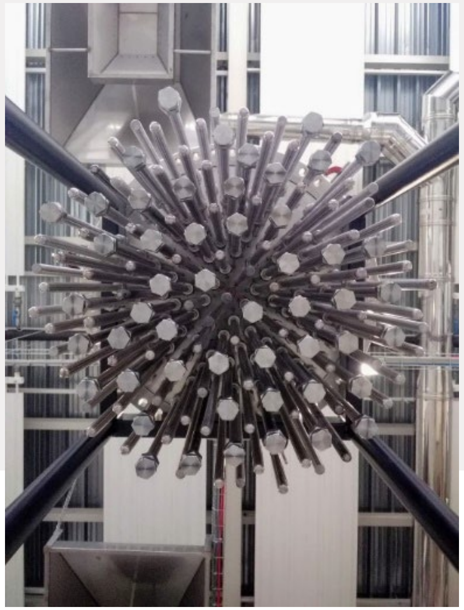
Novel intensified reactor concepts via:

- Integration of reaction and separation (membrane reactors combined with, e.g.: chemical looping concepts, ammonia decomposition, oxidative dehydrogenations, partial oxidations…)

- Integration of reaction and heat/energy management (endo/exothermic, plasma systems)

- Packed bed and fluidized bed membrane reactors: use membranes to improve fluidization and fluidization to improve membrane flux





- **Research approach:** combination experimental and modelling approach

STEPS TAKEN TILL NOW

2010
SINGLE MEMBRANE TUBE
10CM LONG

2012
MULTI TUBE MEMBRANE
15CM LONG

2014
MULTI TUBE MEMBRANE
15CM LONG
Fluid bed
<10 ppm CO

2016
MEMBRANE
25CM LONG
Fluid bed
ethanol

2018
MEMBRANE
50CM LONG
Fluid bed
>65% recovery
Biogas reforming
>99.8% purity

Design and construction
$2 m^3/h$ prototype

2019
Design and construction
$20 m^3/h$ prototype

11 EU projects
Several Spanish and Dutch projects

Content

1. **Introduction**
2. **Project Objectives (TUE)**
3. **From approach and methodology to results**
4. **Conclusion & outlook**

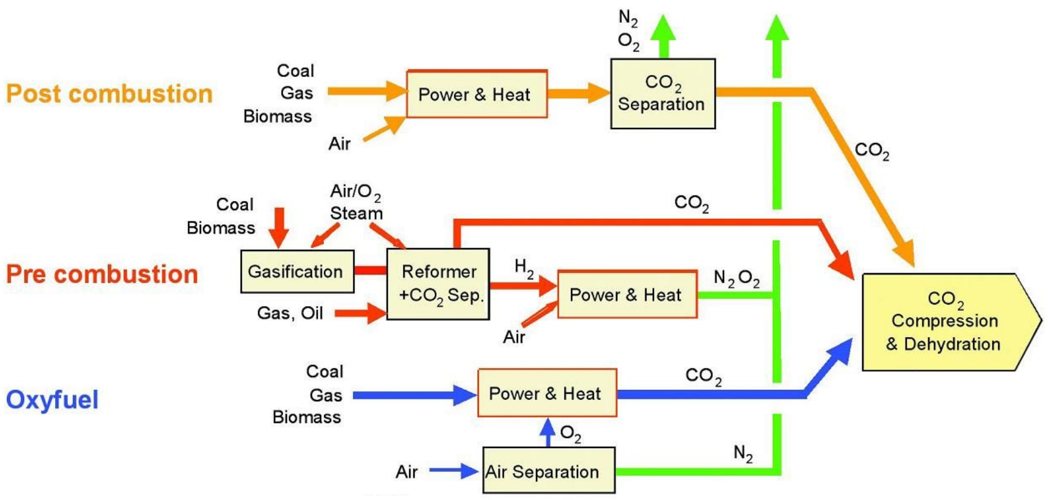
1. Introduction

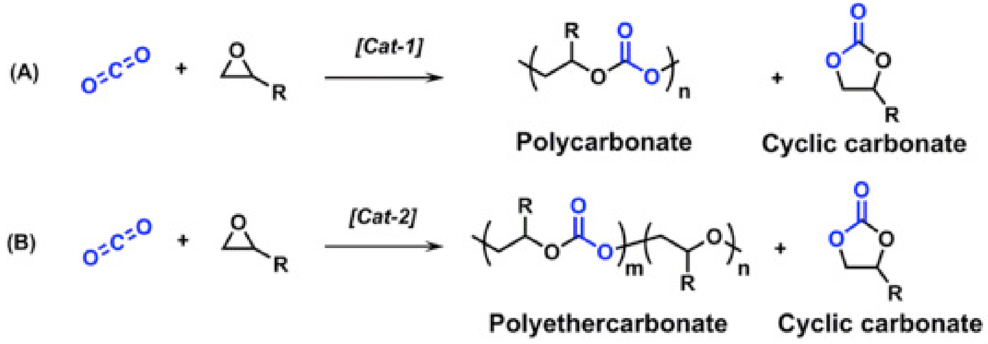
Carbon Capture technologies: [1]

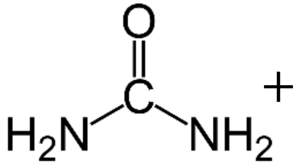
- Pre-combustion
- Post-combustion
- Oxyfuel combustion

CO2 for:

- Feedstock for chemicals
  - Fertilizers, polymers [2]
- Solvent extraction
- Carbonation beverages
- Storage





$CO_2 + 2NH_3 \rightarrow$  $+H_2O$

$CO_2(g) + XO \rightarrow XCO_3(s)$

[1]: Based on Overview of CO2 capture processes and systems (IPCC, 2005)
[2]: Polymers from carbon dioxide: Polycarbonates, polyurethanes; S.Lui,X Wang (2017)

1. Introduction

Targets

Prototype A

Pre-combustion capture in power plants using MMMs at HYGEAR reforming equipment.

CCR	Capture Cost
> 90%	**< 30 €/ton**

Prototype B

Post-combustion capture in power plants using MMMs at the 8.8 MW CHP facilities of Agroger (GALP, Portugal).

CCR	Capture Cost
> 90%	**< 40 €/ton**

Prototype C

Pure hydrogen production with integrated CO_2 capture using MA-SER at the IFE-HyNor (Norway) under the supervision of ZEG POWER.

CCR	Capture Cost
> 90%	**< 30 €/ton**

2. Project objectives for TUE

Objective: Modelling of the MA-SER system to optimize the performance of the reactor with respect to H_2 production, CO_2 capture and material utilization for up-scale process design

3. Approach & methodology

- Process description
- Define performance indicators
- Material modelling
  - Catalyst
  - Sorbent
  - Membrane
- MA-SER reactor modelling

MA-SER process description

1) Reforming of CH_4 to reformate using catalyst

$CH_4 + H_2O \leftrightarrow CO + 3H_2$

$CO + H_2O \leftrightarrow CO_2 + H_2$

2) Absorption of CO2 using sorbent

$CO_2 + CaO \rightarrow CaCO_3$

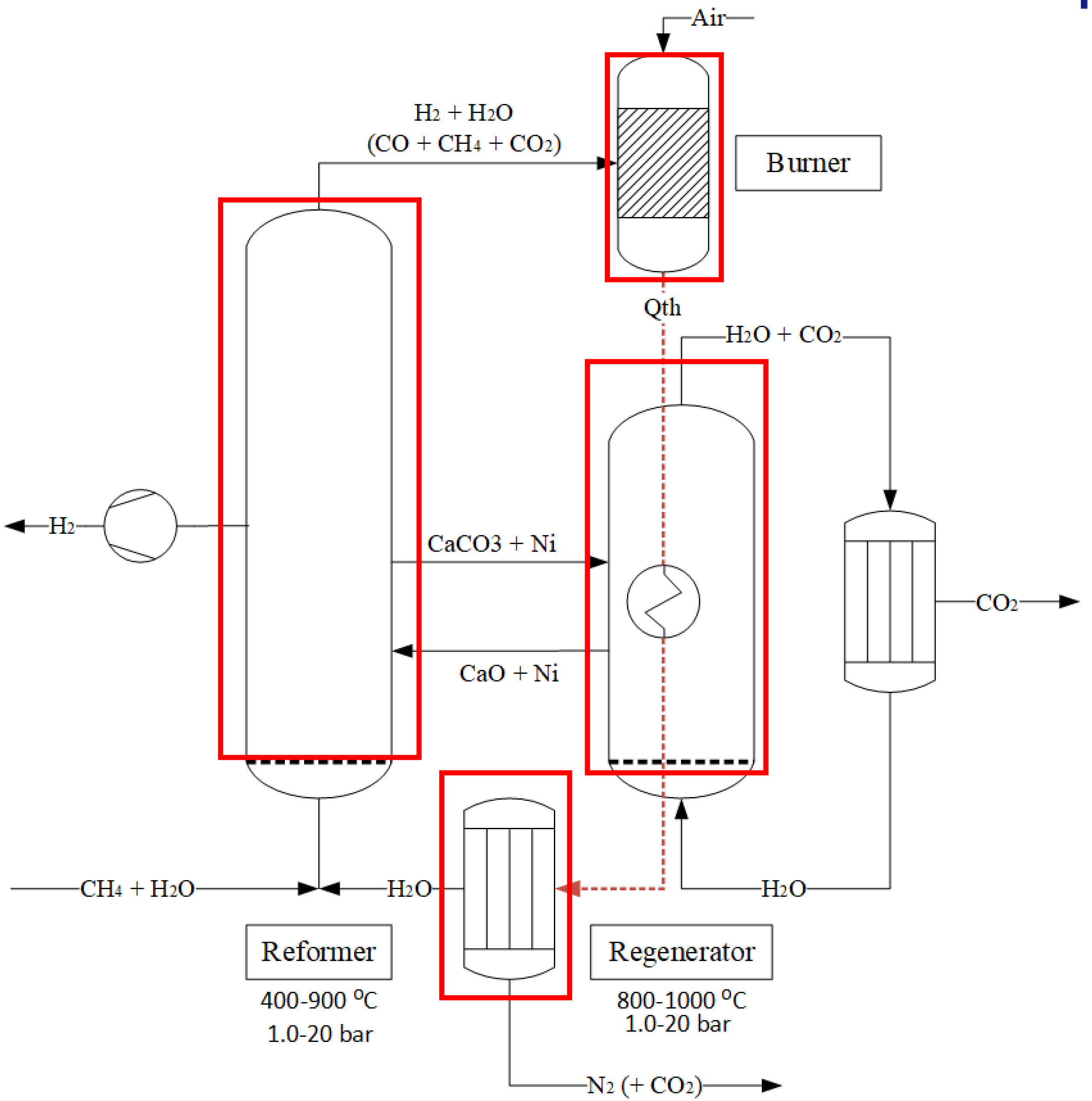
3) Removal of H_2 product using membranes

4) Saturated sorbent send to generator

$CaCO_3 \rightarrow CaO + CO_2$

5) Carbon lean stream combusted to supply energy for calcination reaction regenerator

6) Excess steam recovered by condensation

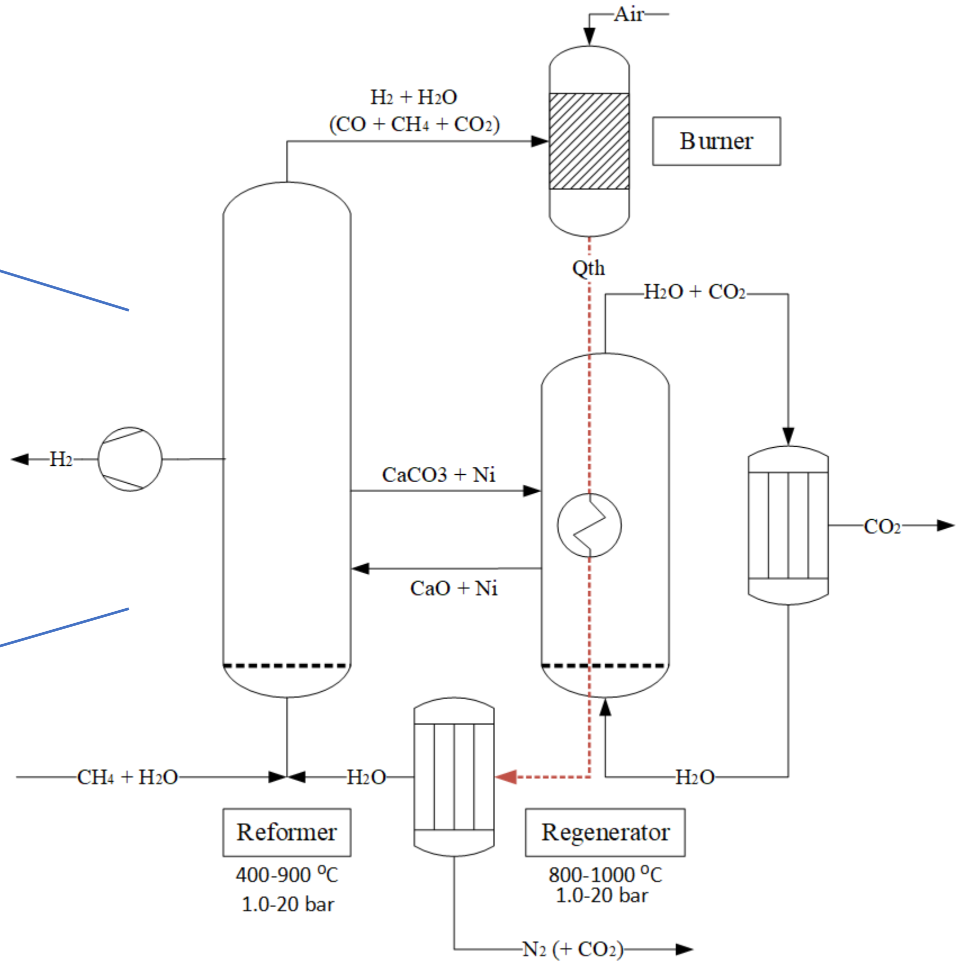


Key performance indicators

Performance indicators

- CH_4 feedstock conversion $$X_{CH4} = 1 - \frac{F_{CH4}|_{Rout}}{F_{CH4}|_{Rin}}$$
- CO_2 capture recovery $$CCR = 1 - \frac{F_{CO2}|_{REG}}{F_{CH4}|_{Rin}}$$
- H_2 product yield $$HRF = \frac{F_{H2}|_{mem}}{4 \cdot F_{CH4}|_{Rin}}$$

Materials relevant for modelling



MEMBER

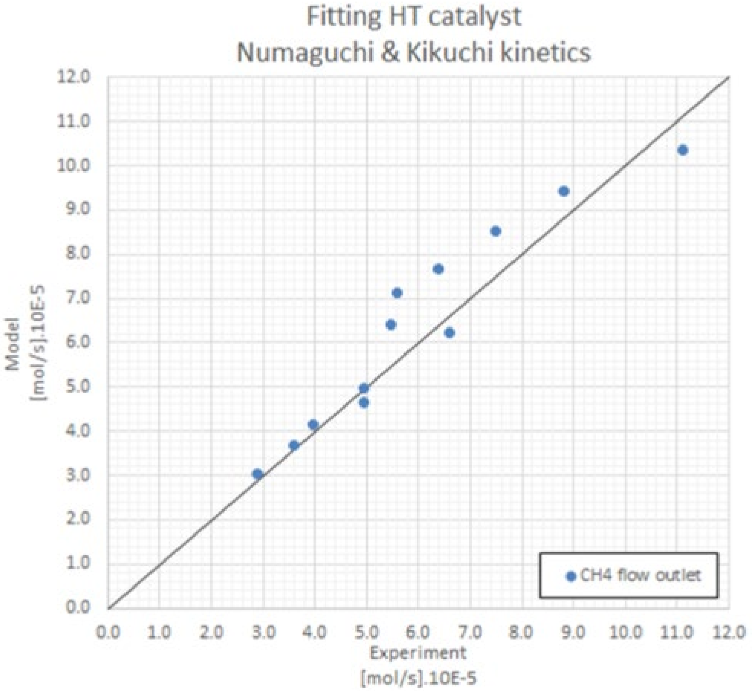
Reforming catalyst

Nickel based catalyst

Kinetics of reforming CH_4 from literature

Researchers	Article	Abr.	H_2O/CH_4	T_R	p_R
			[mol/mol]	[°C]	[bar]
Xu and Froment	1989	XF	3.0 – 5.0	300 – 575	3.0 – 15
Numaguchi and Kikuchi	1988	NK	1.44 – 4.50	400 – 887	1.2 – 25.5
Hou and Hughes	2001	HH	4.0 – 7.0	400 – 550	12 – 60

Experiments C&CS catalyst



Reaction rate expression
fitted using micro fixed bed reactor

Reaction rate SMR, NK $$r_{SMR} = \frac{k_{SMR,NK}}{p_{H2O}^{1.596}}\left[p_{CH4}p_{H2O} - \frac{p_{H2}^{3}p_{CO}}{K_{eq,SMR}}\right] \quad \left[\frac{mol_{CH4}}{kg_{Ni}\cdot s}\right]$$

Reaction rate WGS, XF $$r_{WGS} = \frac{k_{WGS,NK}}{p_{H2O}}\left[p_{CH4}p_{H2O} - \frac{p_{H2}^{3}p_{CO}}{K_{eq,WGS}}\right] \quad \left[\frac{mol_{CH4}}{kg_{Ni}\cdot s}\right]$$

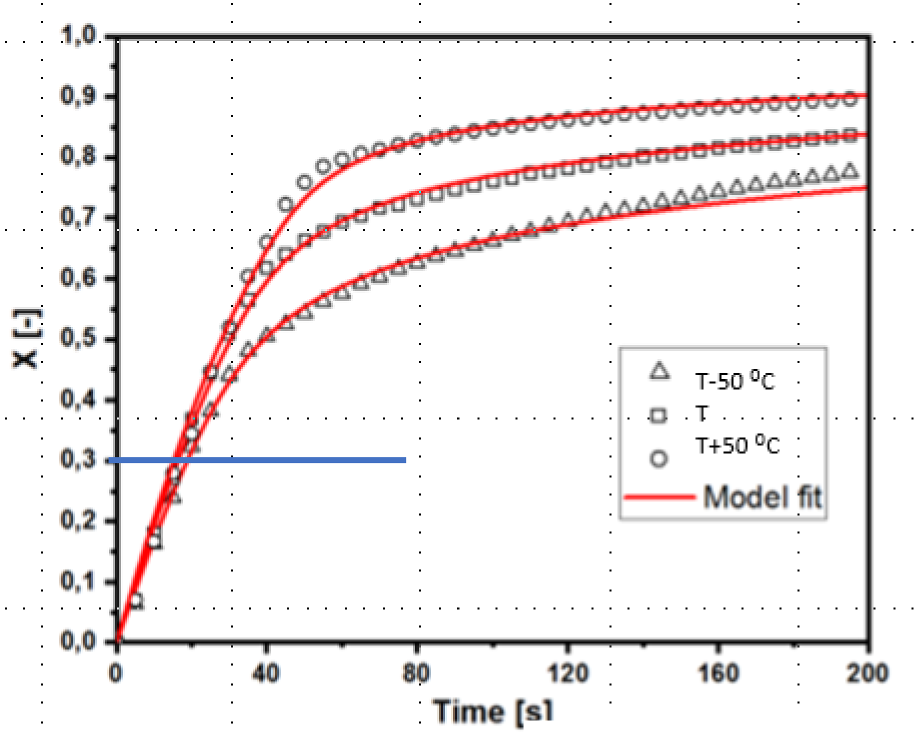
Sorbent - Carbonation kinetics

- Sorbent – CaO based
- Sorbent carbonation kinetics, described by particle model with transition kinetic-ion diffusion limiting regimes.
- Kinetics are fitted after restructuring of CaO grains (> 5 cycles)

$$\frac{dX}{dt} = \frac{k_s \sigma^0_{CaO}(1-X)^{2/3}}{1 + \frac{N^0_{CaO} k_s}{2D_{PL}} \delta^0_{CaO} \sqrt[3]{1-X}} \frac{\left(P_{CO_2} - P^{eq}_{CO_2}\right)}{RT}$$

$$D_{PL}(X,T) = D^0_{PL} \exp\left(-aX^{(bT+c)}\right)$$

- Only interested in kinetic limited regime (X_{CaO} < 0.3)



Workshop on Advanced Membranes and Membrane assisted processes for pre- and post combustion CO2 capture

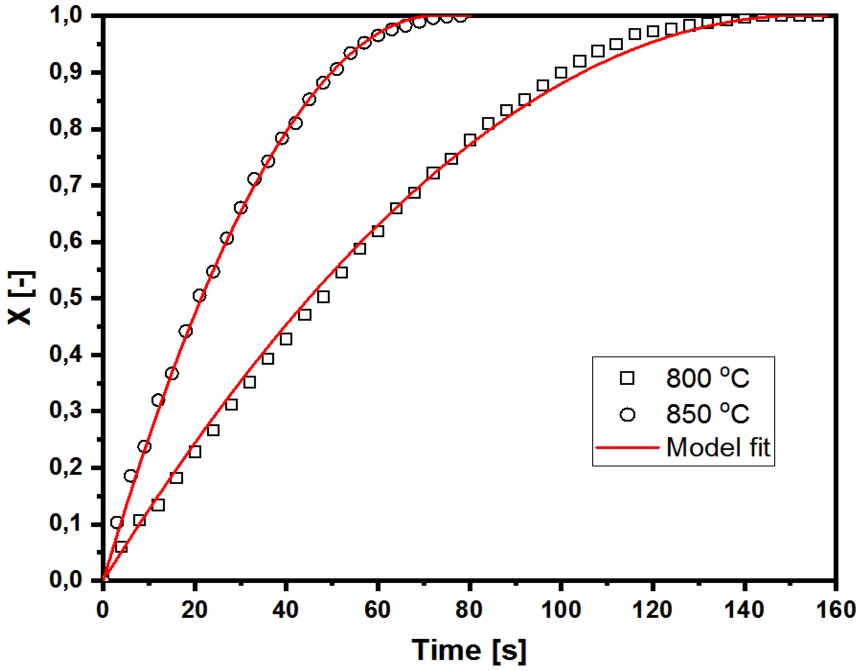
Sorbent - Calcination kinetics

➢ Sorbent calcination kinetics, described by shrinking core particle model.

➢ Kinetics are fitted after restructuring of CaO grains (> 5 cycles)

$$\frac{dX}{dt} = k_s^{cal}(1 - X)^{0.5}$$

$$k_s^{cal} = k_{s,0}^{cal} \exp\left(-\frac{E_a^{cal}}{RT_R}\right)$$



➢ In calciner performance not limited by kinetics, dominated by thermodynamics

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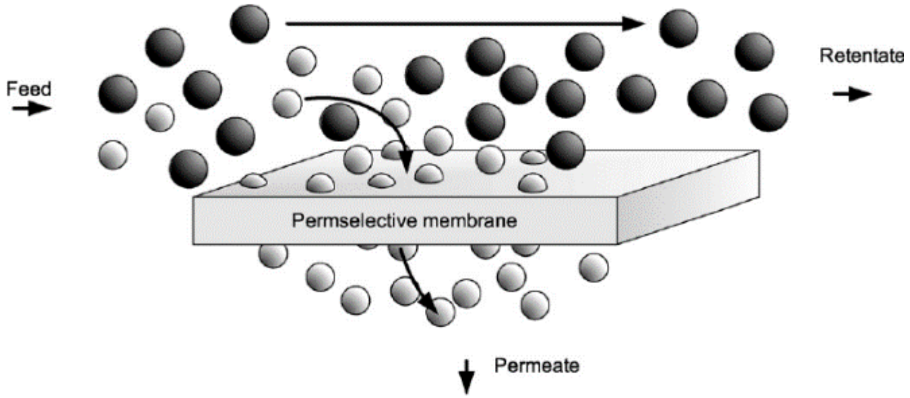
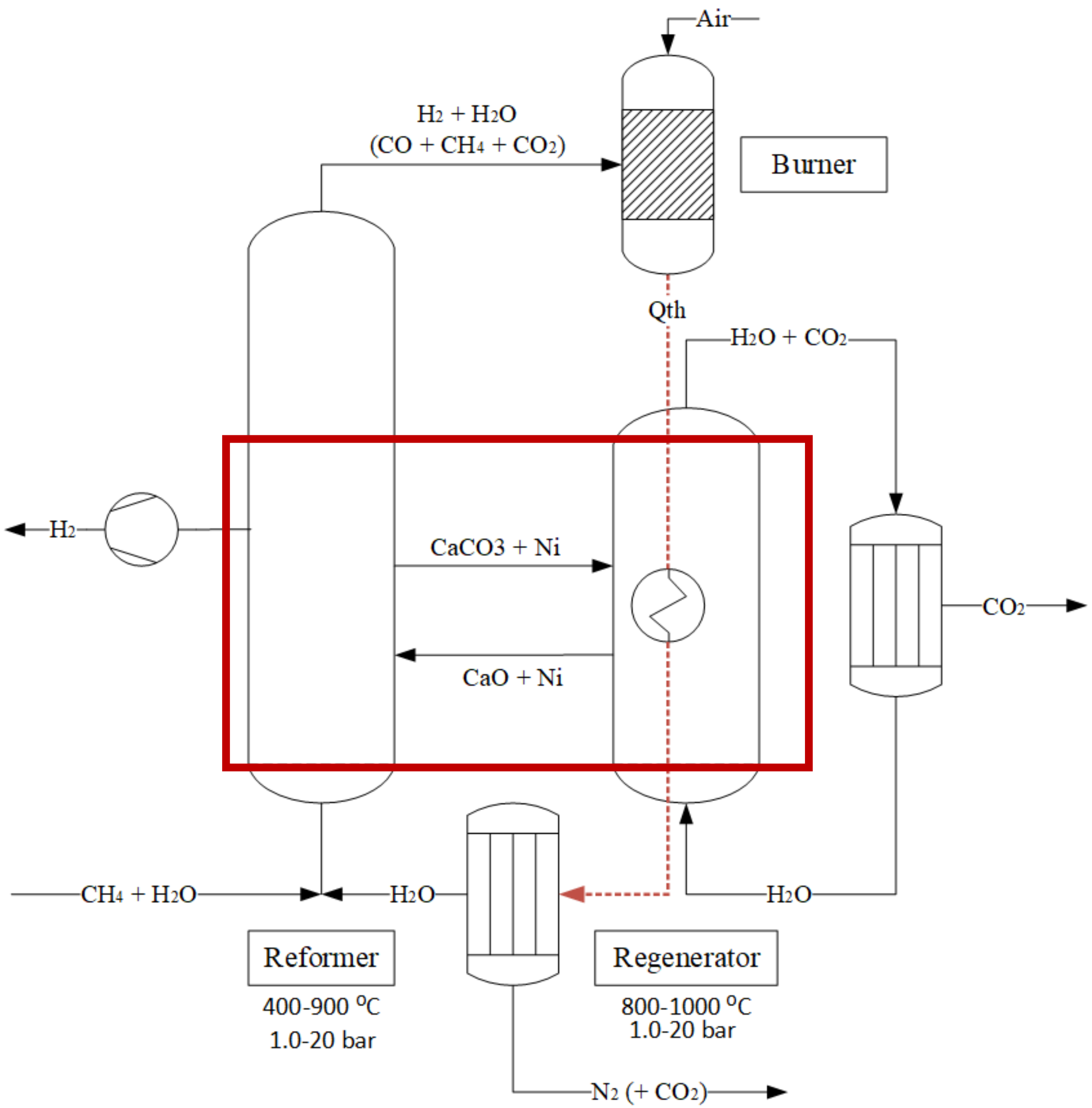
Membrane – H_2 recovery

- Hydrogen permeation rate
  - Permeation rate determined by:
    - Surface activity
    - Membrane selectivity
    - External mass transfer limitations



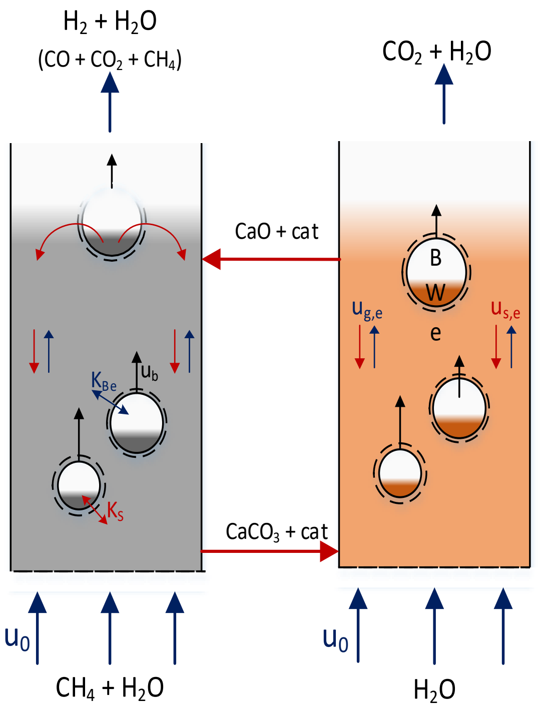
Sherwood correlation in triangular pitch [20]	$Sh_k = \sqrt{f^2 + g^2 Gz^{2/3}}$ $f = \frac{8.92(1 + 2.82\phi)}{1 + 6.86\phi^{5/3}}$; $g = \frac{2.34(1 + 24\phi)}{(1 + 36.5\phi^{5/4})[3.464\phi^2 - \pi]^{1/3}}$	[−]
External mass transfer flux	$N_{H2}^{ext} = -\frac{p_R}{RT_R}\frac{ShD_{H2}}{d_h}\frac{\langle y_{H2}\rangle - y_{H2,ret}}{1 + \frac{\langle y_{H2}\rangle + y_{H2,ret}}{2}}$	$\left[\frac{mole}{m_{mem}^2 \cdot s}\right]$
Membrane flux	$N_{H2}^{mem} = \frac{P_{H2}}{\delta_{mem}\left[1 + \ln\left(\frac{r_{sup} + \delta_{mem}}{r_{sup}}\right)\right]}[p_{H2,ret}^n - p_{H2,perm}^n]$	$\left[\frac{mole}{m_{mem}^2 \cdot s}\right]$
Steady state assumption	$N_{H2}^{mem} = N_{H2}^{ext}$	$\left[\frac{mole}{m_{mem}^2 \cdot s}\right]$

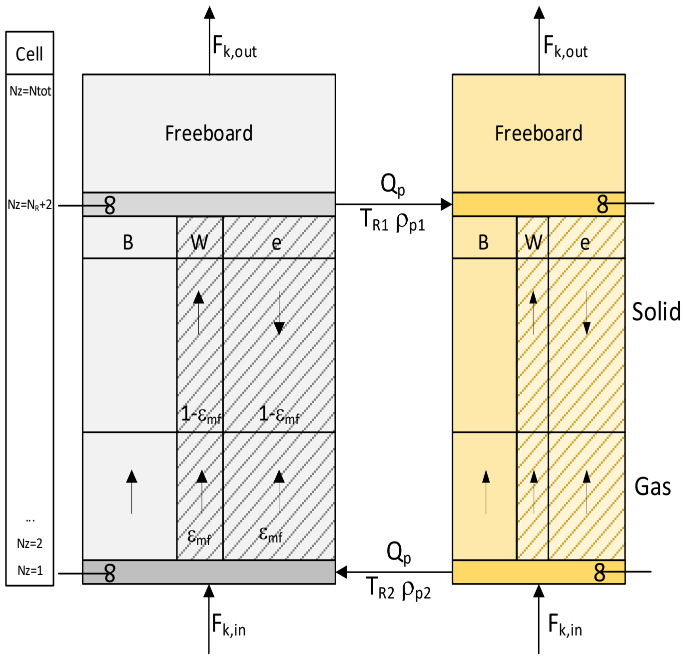
Reformer + regenerator modelling



Modelling of MA-SER reactor

Mathematical description





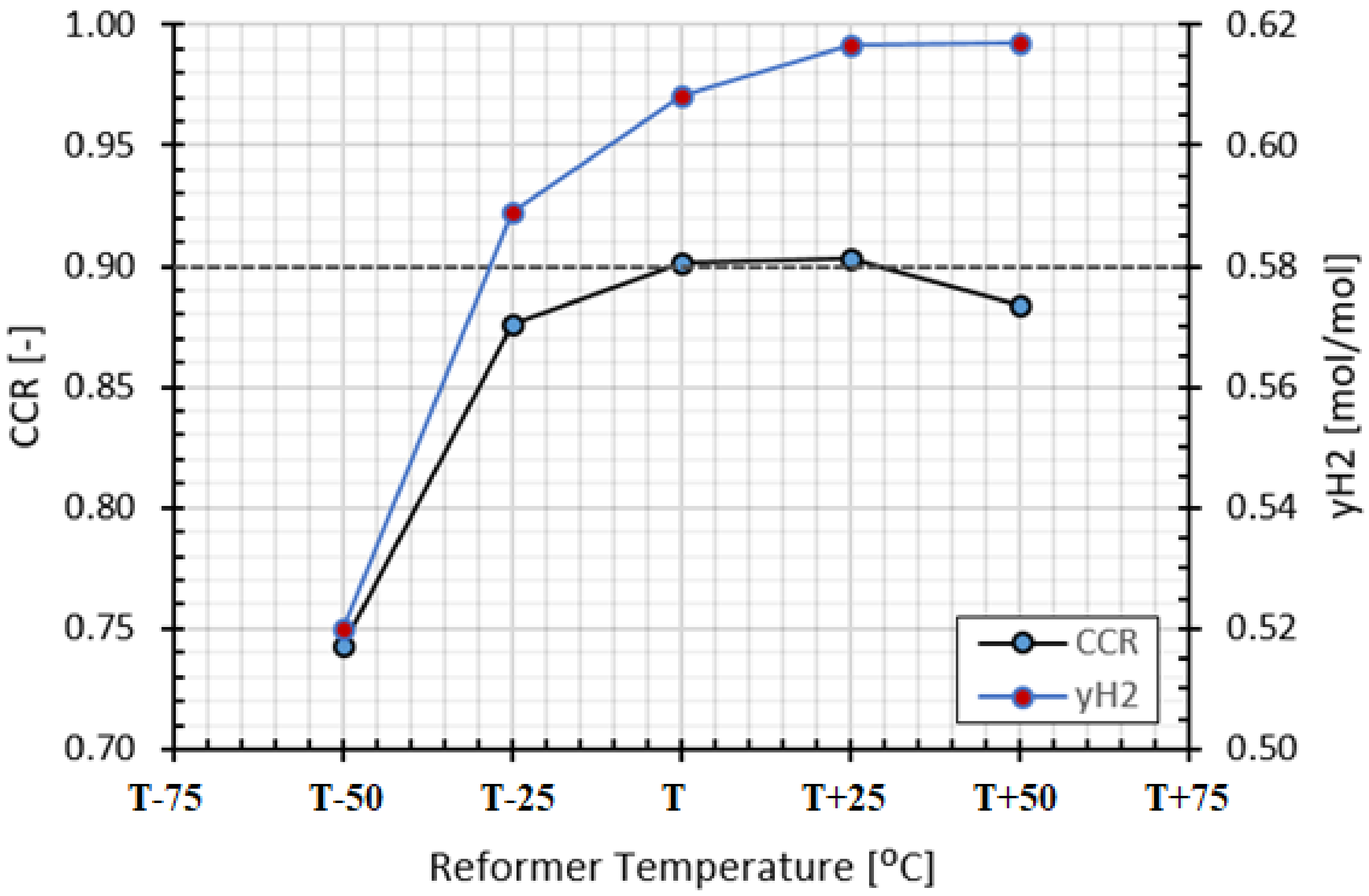
- Modelled the MA-SER reactor system using 1D phenomenological model
- Kunii and Levenspiel 3-phase fluidized bed reactor model

Parametric study on MA-SER reactor

Effect of reformer temperature on KPI

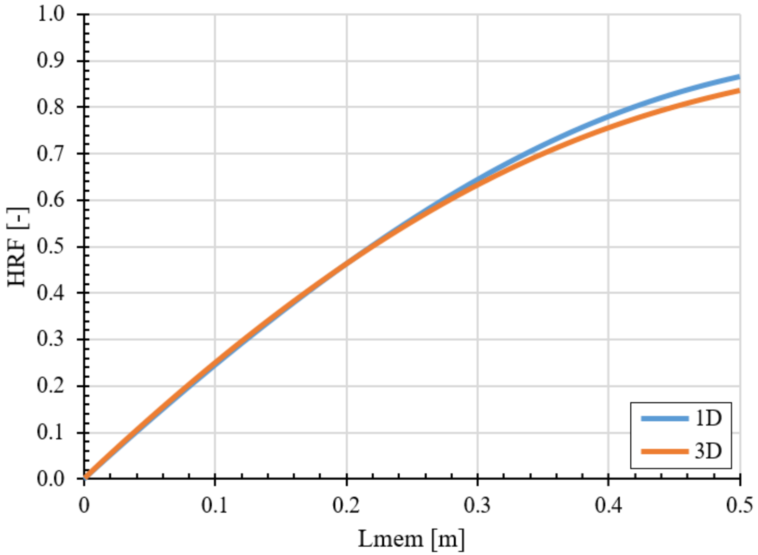
Target performance

CCR > 90%
yH2 > 0.6

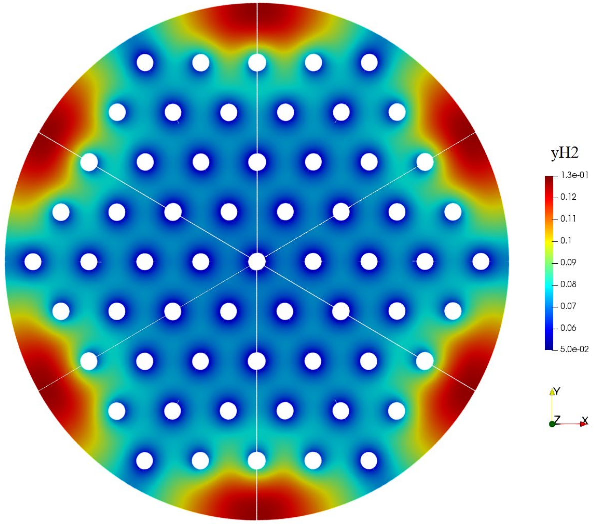


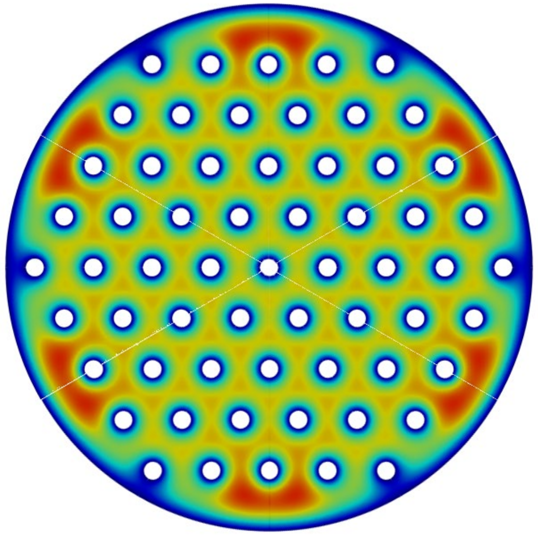
Parametric study on membrane module

- Membrane module design
  - Full 3D simulation of membrane module
  - Comparison with prediction 1D model



  - Hydrogen distribution through module
    - Positioning of membrane important due to concentration polarization





4. Conclusion & outlook

We presented the modelling of the MA-SER system to optimize the performance of the reactor with respect to H2 production, CO2 capture and material utilization for up-scale reactor design

- Dual fluidized bed reactor at bubbling fluidization conditions modelled using 1D phenomenological model
  - Implemented kinetics derived for individual material characterization
- Evaluation of model results
  - Process performance limited by sorbent kinetics
- Evaluation of membrane module
  - 1D model can predict the 3D full scale model CFD simulation
  - Positioning membranes are key for optimal performance

- Model can be used for up-scale reactor design of high-purity hydrogen

Thank you for your attention

https://member-co2.com/

Contact:

joseluis.viviente@tecnalia.com

Acknowledgement: For the CO2 molecule used in the logo:The original uploader was Frederic Marbach at French Wikipedia [GFDL (http://www.gnu.org/copyleft/fdl.html)

2.9. Market analysis and techno-economic assessment of MA-SER system (Vittoria Cosentino – KT)

Market analysis and techno-economic assessment of MA-SER system

Final Workshop MEMBER Project
Kjeller, 23-06-2022

Vittoria Cosentino
v.cosentino@kt-met.it

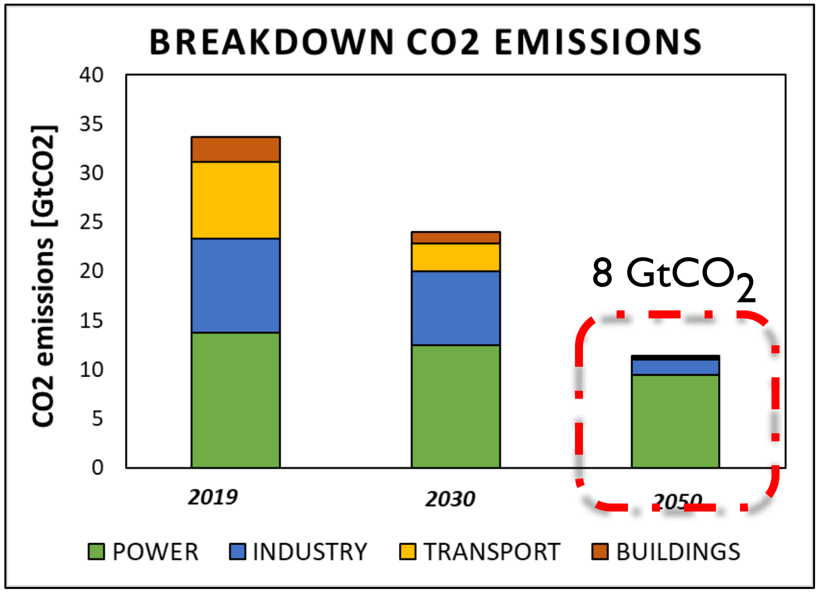
Outline

- Introduction on CO_2 emissions and H_2 market
- MA-SER technology
  - System description
  - H2 cost of production
  - Sensitivities analysis
- Conclusions and Future Perspectives

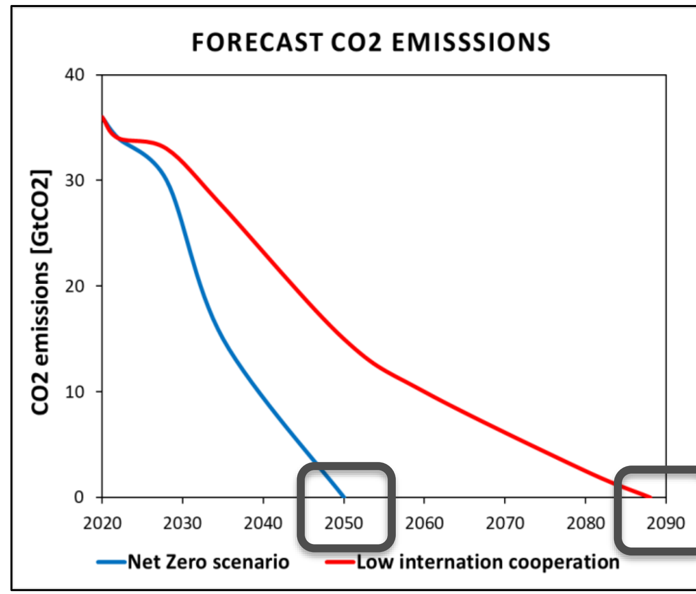
Introduction

➢ Goal of the European Union in Green Deal [1] :

Turning the EU into THE FIRST CLIMATE NEUTRAL CONTINENT by 2050. → Reduce emissions by at least 55% by 2030, compared to 1990 levels. → Capture at least 75 tons of CO_2 by 2035 / Produce less CO_2



Forecast of global CO_2 emissions if energy and industrial assets are left as they are until the end of their technical life[2]



Global CO_2 emissions in the Net Zero scenario and Low International cooperation scenario [2]

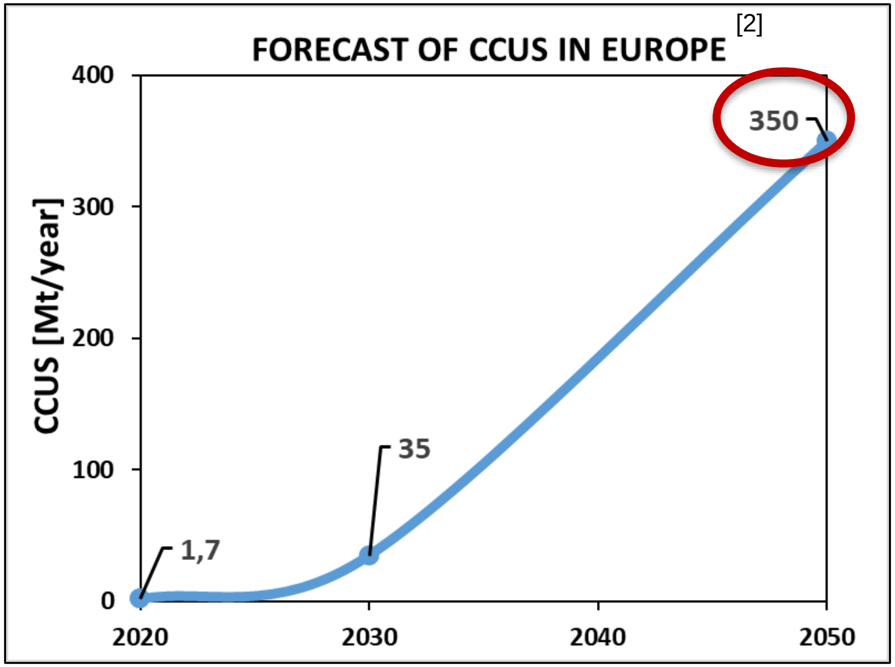
[1] Delivering the European Green Deal | European Commission (europa.eu)
[2] IEA, "Energy technology Perspective-Special Report on Carbon Capture Utilisation and Storage CCUS in clean energy transitions," 2020.

Introduction

CO_2 Capture

Three different application for Carbon Capture:

- **CCS** - Carbon capture and storage
- **CCU** - Carbon capture and utilization or CO_2 use
- **CCUS** - Carbon capture, utilization and storage



To push CO_2 capture →

CO_2 as resource and not waste

Categories of CO_2-derived products and services [3]:

- CO_2-derived fuels
- CO_2-derived chemicals
- Building materials from minerals and CO_2
- Building materials from waste and CO_2
- Crop yield boosting with CO_2

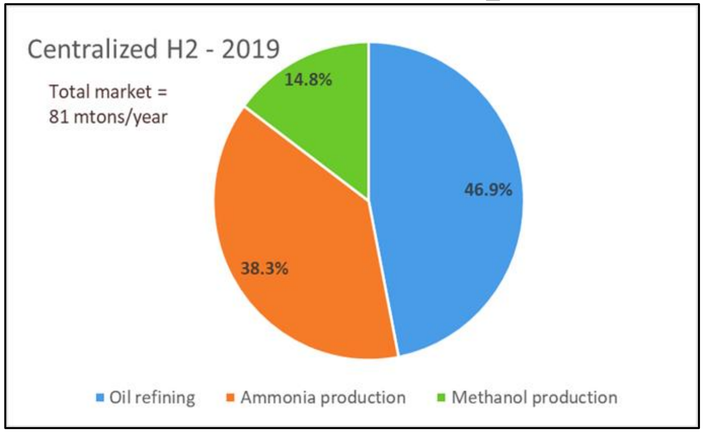
[2] IEA, “Energy technology Perspective-Special Report on Carbon Capture Utilisation and Storage CCUS in clean energy transitions,” 2020.
[3] IEA, “Putting CO2 to Use,” 2019.

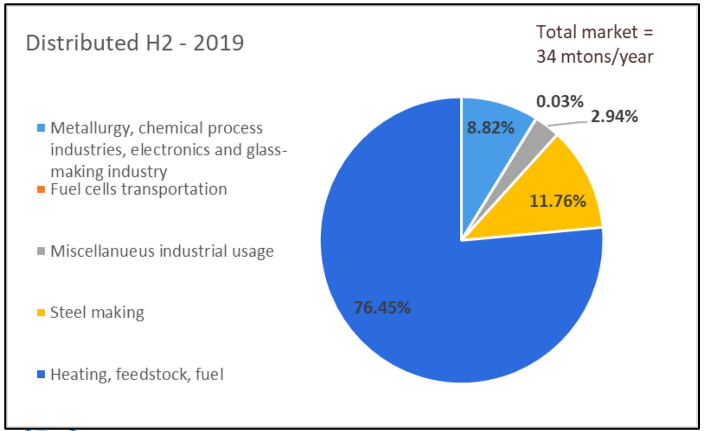
Introduction

H_2 market (1/2)

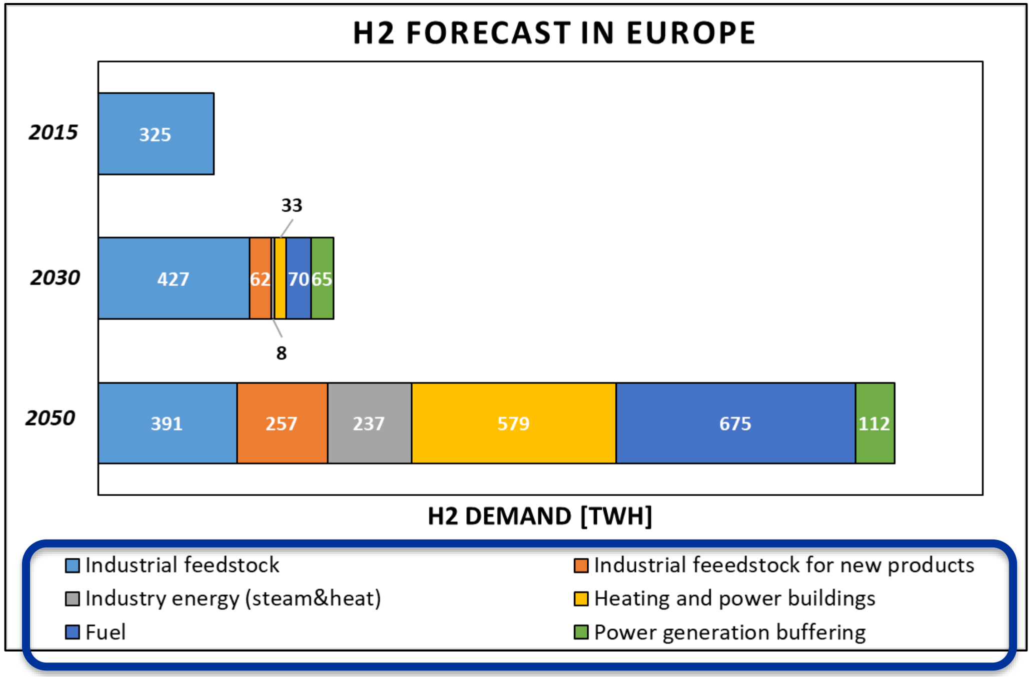
- **Hydrogen** → important piece of the puzzle to decarbonize Europe

Breakdown of market for centralized and distributed H_2 [4,5]





Forecast of split of H_2 demand for Net Zero Scenario in 2030 and 2050[6]

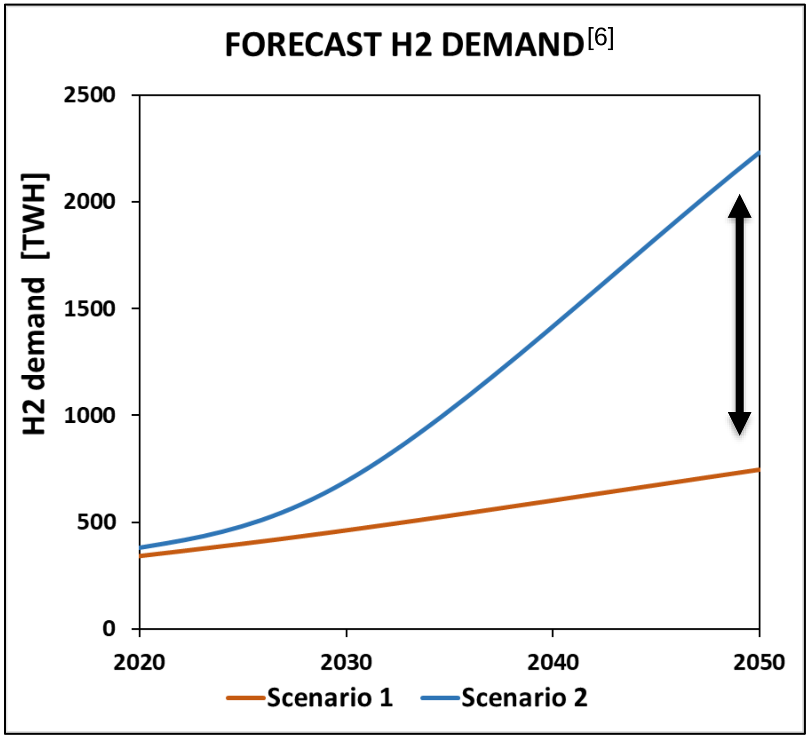


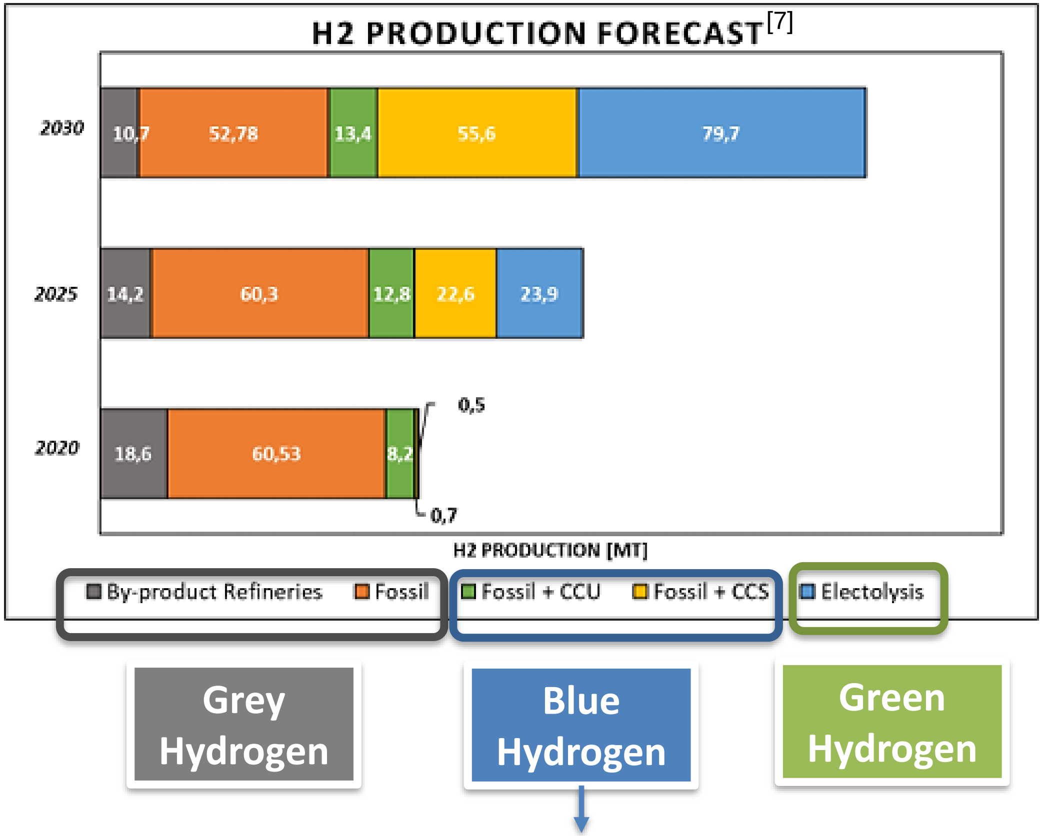
[4] G. C. Institute, "The European Green Deal: New opportunities to scale up carbon capture and storage," 2020.
[5] IEA. The Future of Hydrogen. 2019.
[6] FCH&EU, Hydrogen Roadmap Europe: a sustainable pathway for the European energy transition, 2019.

Introduction

H_2 market (2/2)

- H_2 can be used with high efficiency in many applications without emitting CO_2
- All these considerations are valid and interesting if also its production, and not only the use, is CO_2-free!





MA-SER technology

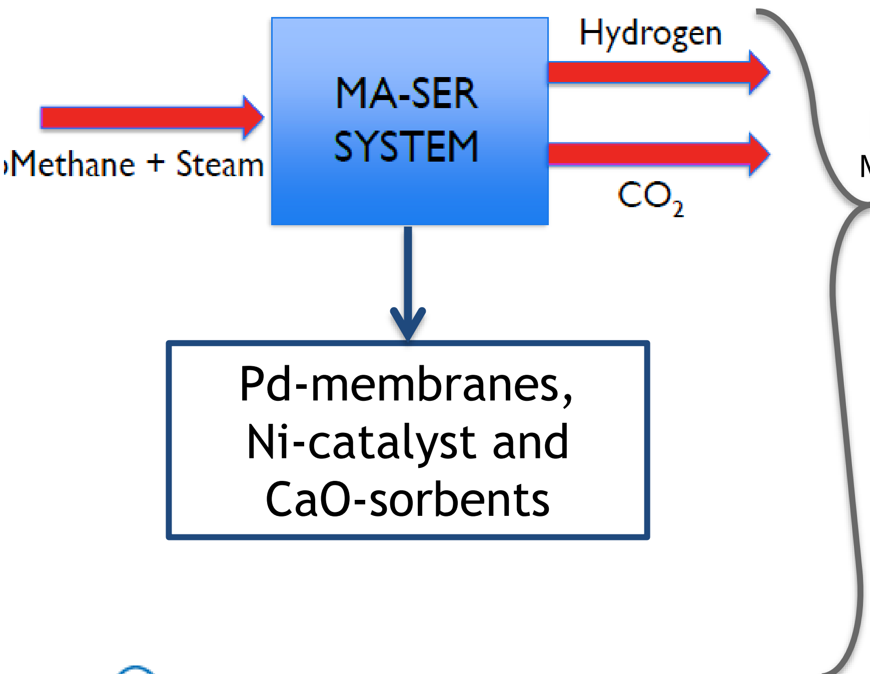
[6] FCH&EU, Hydrogen Roadmap Europe: a sustainable pathway for the European energy transition, 2019.
[7] IEA, Hydrogen, November 2021

MA-SER technology
System description (1/2)

- Pure Hydrogen production with integrated CO_2 capture

- Combination of H_2 membranes, reforming catalyst and CO_2 sorbent into an advanced Membrane Assisted Sorption Enhanced Reforming (MA-SER) process

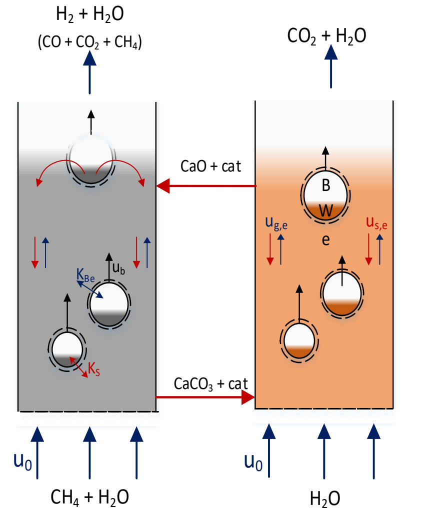
System to be compared to

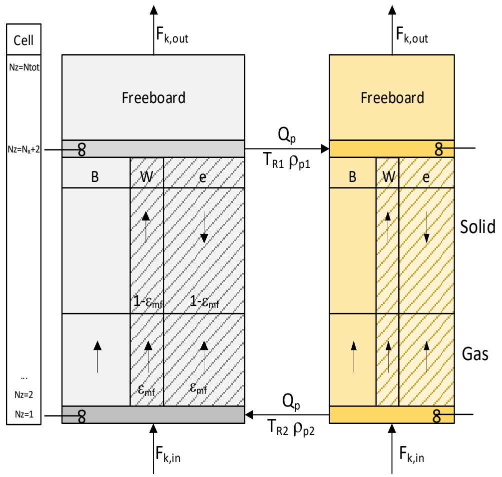


H_2 purity	**vol%**	**≥99.9**
CCR	**%**	**≥90**

MA-SER technology
System description (2/2)

- Dual bubbling fluidized bed reactor system





- New MEMBER materials for MA-SER line

Pd-based membranes

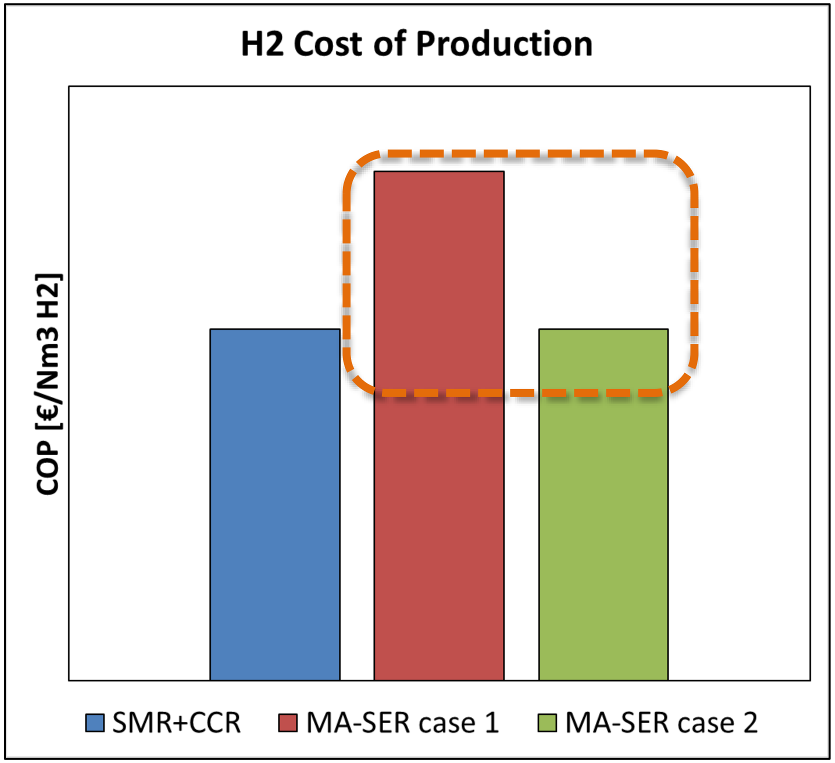
Sorbent

Catalyst

MA-SER technology

H2 cost of production

- H2 cost of production: Depreciation + OPEX + Maintenance & Operation
  - Plant maintenance
  - Solids make-up for attrition rate
  - Substitution membranes after 3 life years



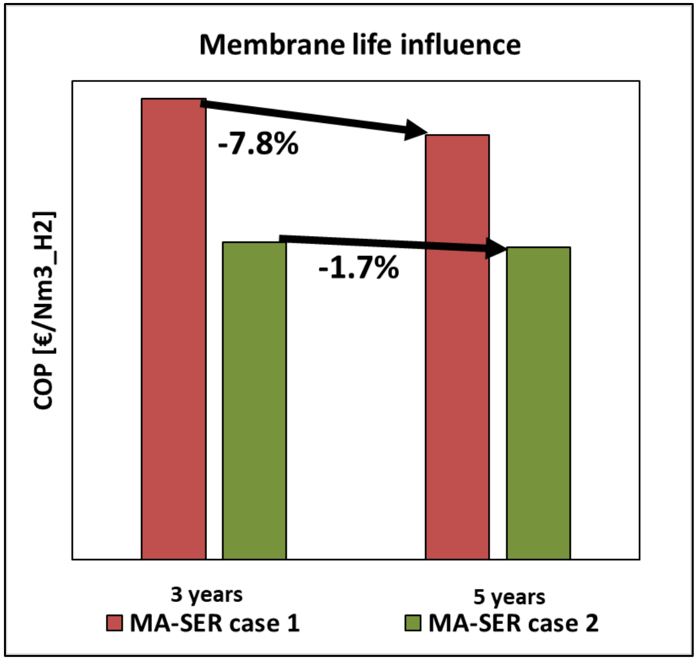
Consumptions & OPEX	Respect to SMR+CCR
Total NG	About -9%
Total electrical energy	About -49%
CO2 fossil	About -25%

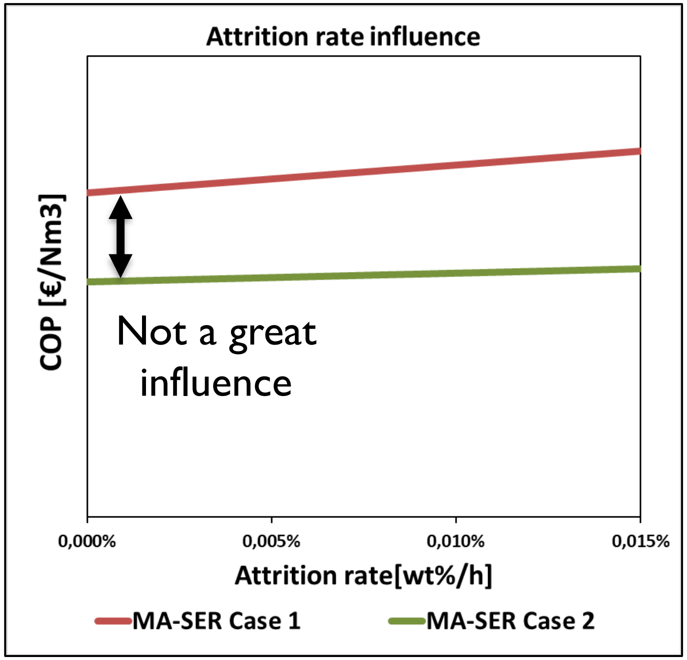
- Case 1: Actual membranes, sorbent and catalyst costs
- Case 2: Estimation membranes, sorbent and catalyst costs → 80% less than case 1

MA-SER technology

Sensitivities analysis - Materials costs and stability

- Materials stability





- Main influence is **membranes, sorbent and catalyst costs**!
  - ✓ Further optimization of manufacturing process
  - ✓ Improvement of solids mechanical stability due to attrition rate
  - ✓ Evaluation of different selective layer for membranes
  - ✓ **Membrane recycle**

Conclusions and Future Perspectives

- The main purpose of the MEMBER project is the development of technologies that exceed the state of the art in hydrogen production with pre/post-combustion
- Advanced materials (Pd-membranes, catalyst and sorbents) are being developed to this aim.
- A techno-economic assessment of the MA-SER processes at an industrial scale shows a strong dependence on materials cost and stability.
- Further optimization on materials manufacturing should be carried out before the technology can enter the market

Market analysis and techno-economic assessment of MA-SER system

Final Workshop MEMBER Project
Kjeller, 23-06-2022

Thank you for your attention

https://member-co2.com/

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Acknowledgement: The research leading to these results has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement nº760944.

Acknowledgement: For the CO2 molecule used in the logo:The original uploader was Frederic Marbach at French Wikipedia [GFDL (http://www.gnu.org/copyleft/fdl.html)

2.10. Environmental Life Cycle Assessment and Life Cycle Costing of the MEMBER systems (Alexander Borsch – QUANTIS)

Environmental Life Cycle Assessment (LCA)

Final MEMBER workshop

Alexander Borsch, Mireille Faist, Quantis

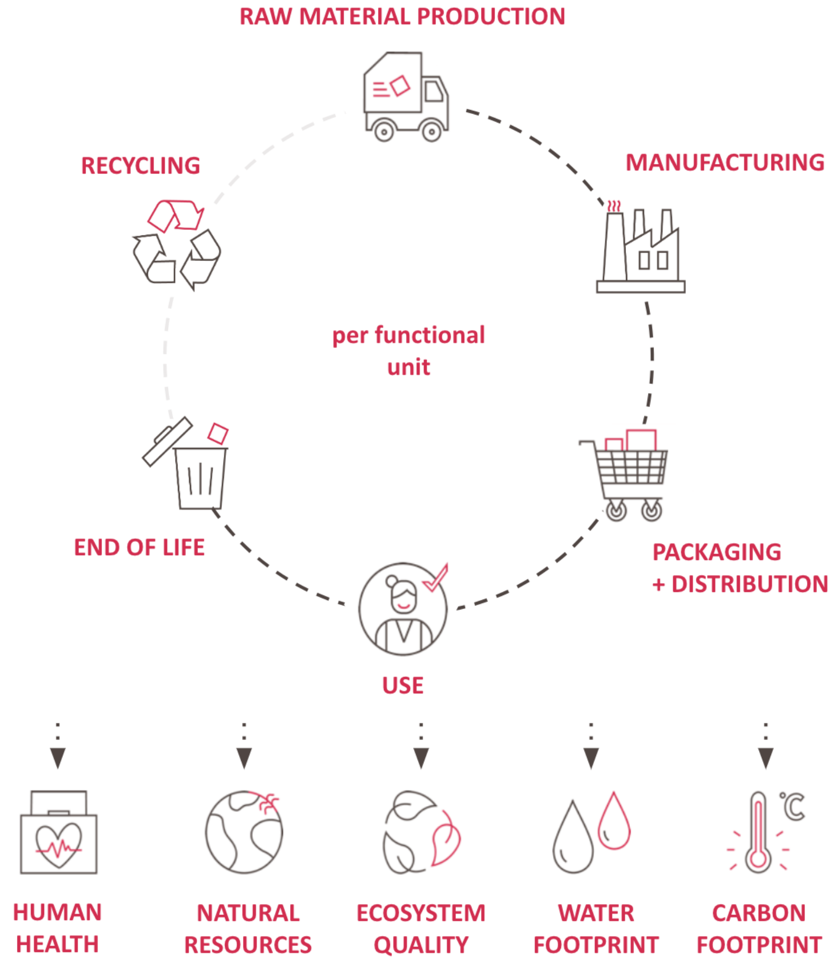
Outline

- LCA introduction
- Method – data collection, FU, system boundaries
- Life Cycle Impact Assessment (LCIA)
  - pre- and post-combustion CO_2 capture
  - H_2 production from natural gas
- Summary and key take-aways

LCA introduction

Life Cycle Assessment is recognized as the leading methodology for environmental impact evaluation. The main strengths of this method are the following:

- **Life-cycle oriented,** allowing users to consider various product stages, to highlight potential 'burden shifting', or unintended consequences.
- **Metrics-based** approach, allowing impact evaluations and/or comparisons to be made on a quantified and credible scientific basis.
- **Multi-criteria**: it covers a multiplicity of indicators in the assessment (including water use, ecotoxicity, ozone depletion, etc.)



Quantis

LCA introduction

Goal and scope

The functional unit

The system boundaries

Inventory analysis

Tools and software

Inventory data and databases

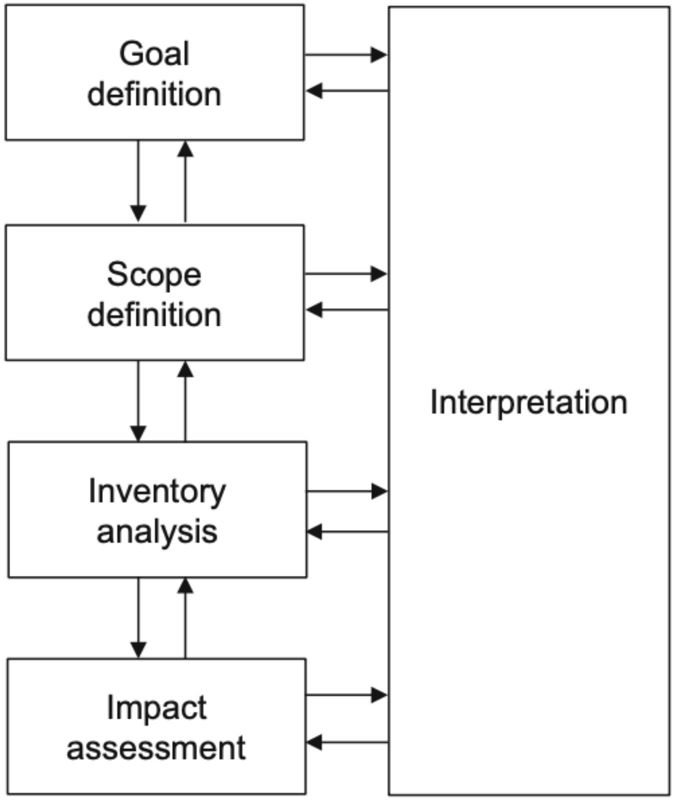
Impact assessment

The synthetic nature of impact assessment

Avoiding tradeoffs (e.g., single indicator)

Interpretation

Numbers are not enough



International Organization for Standardization ISO NORMS 14 040 + 14 044 (2006) FOR LCA

Method – data collection

- Key materials and parameters (membranes, catalysts and sorbents and system parameters) were part of the MEMBER development → primary data

- System parameters are based on prototype testing and scaled up to an industrial level

Method – Functional Unit (FU)

The functional unit quantifies the performance of a product system and is used as a reference unit for which the life cycle assessment study is performed.

The FUs for the pre- and post-combustion CO_2 capture were chosen:

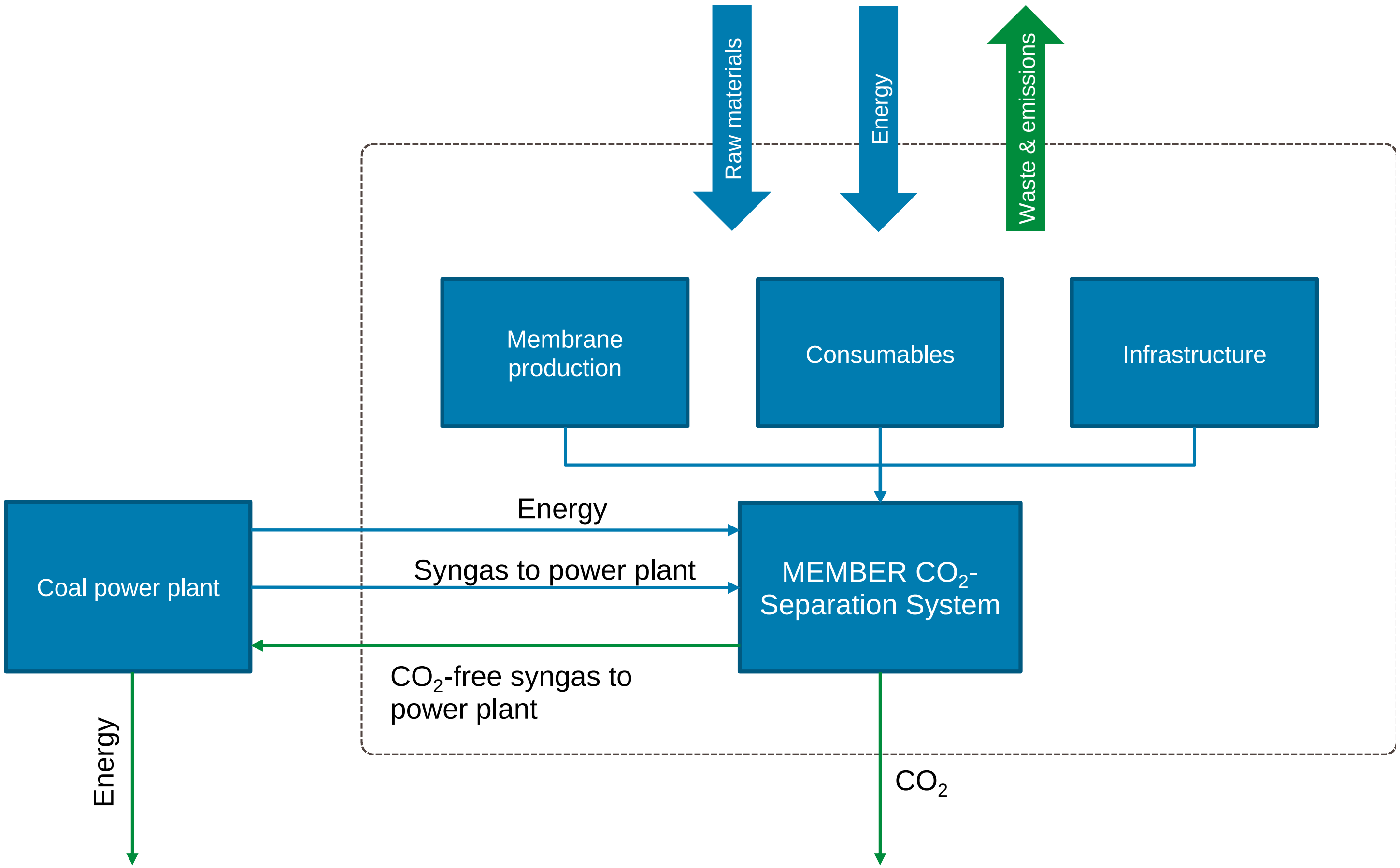
- **1 kg of CO_2** captured and stored for the pre-combustion, post-combustion systems using Mixed Matrix Membranes (MMM)

The FU for the H_2 production with CO_2 capture was chosen:

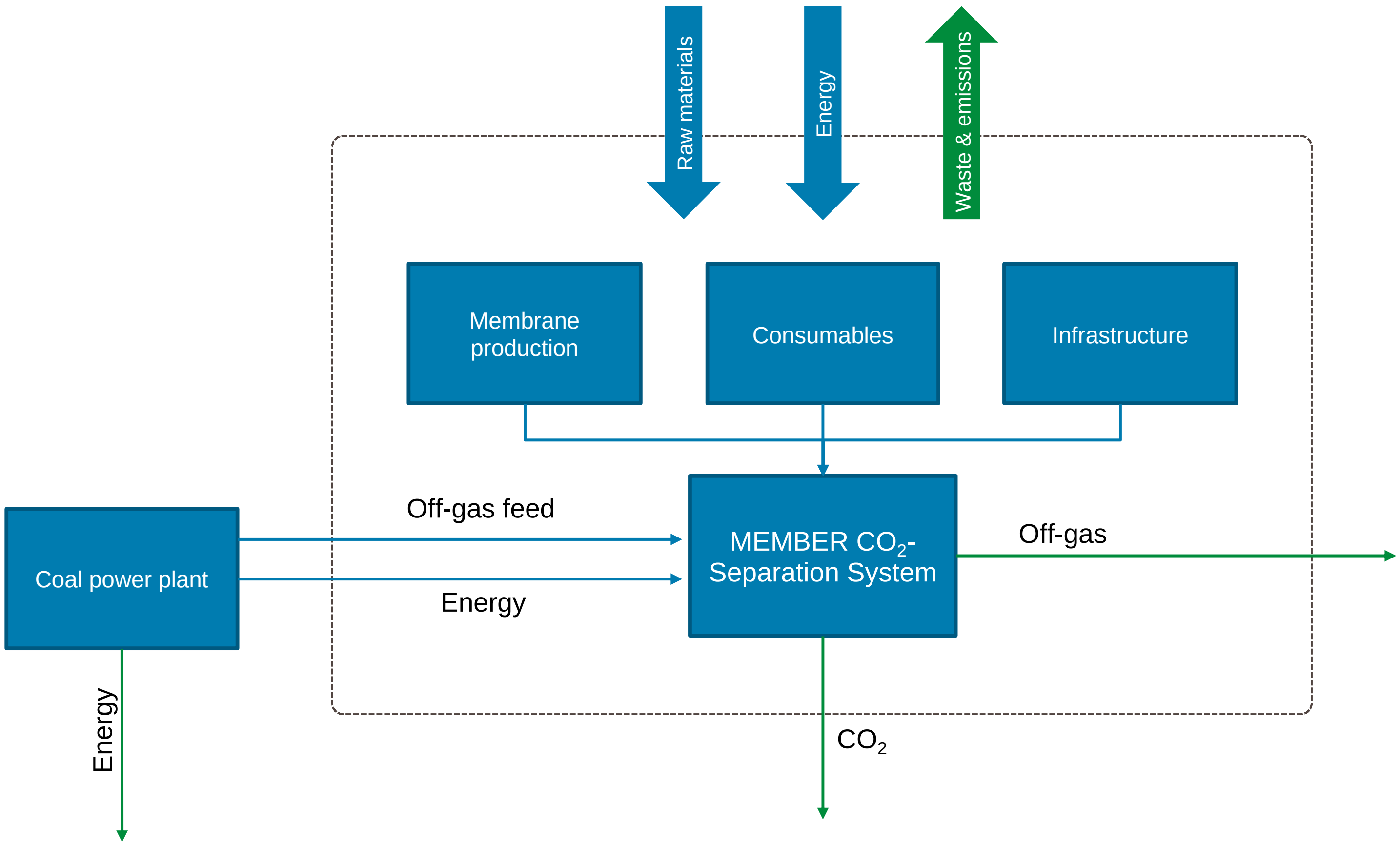
- **1 m^3 of H_2** produced in the hydrogen production system using MA-SER's with a carbon capture rate of 90%

Quantis

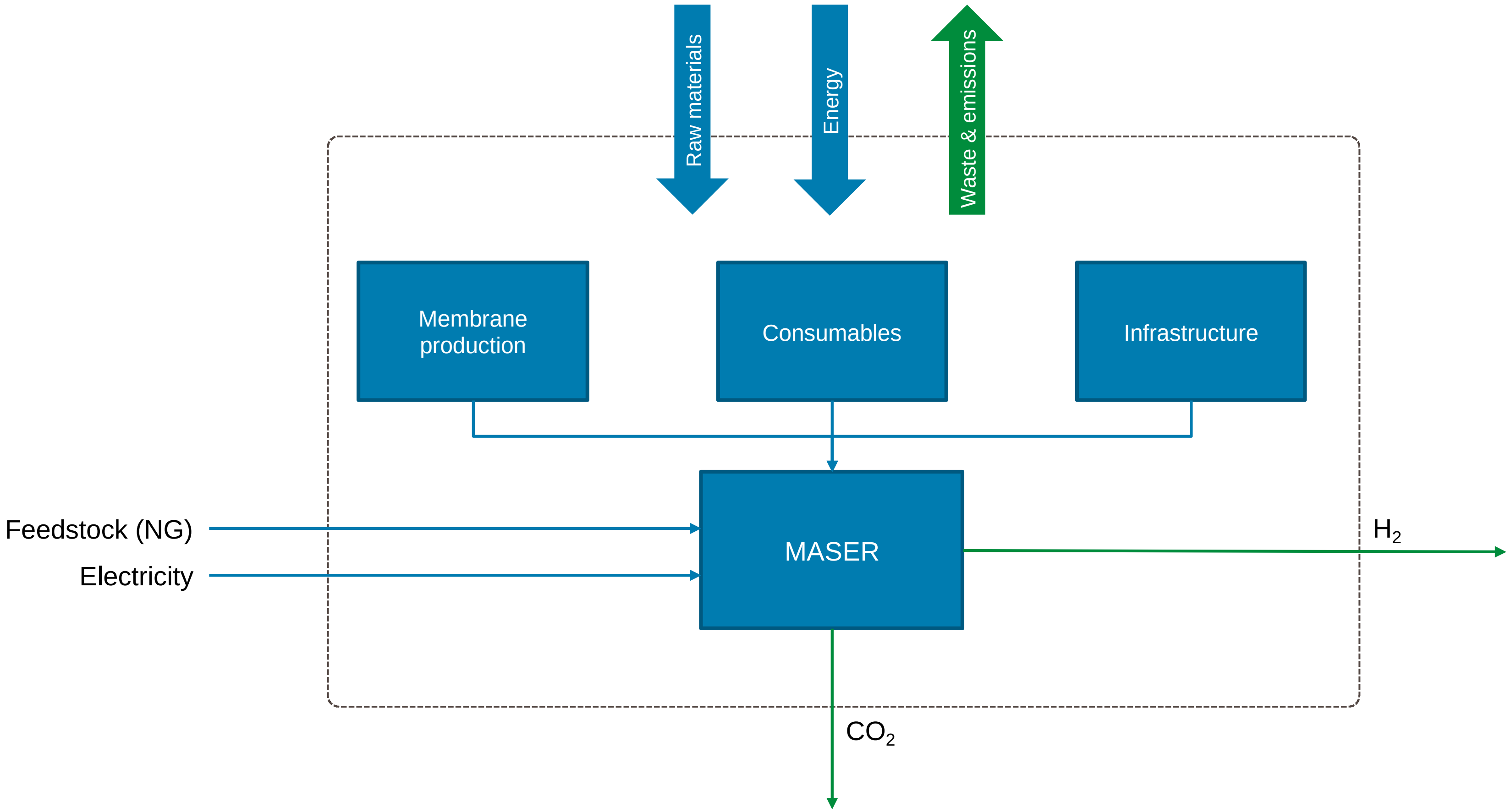
Method – system boundaries for pre-combustion CO_2-capture



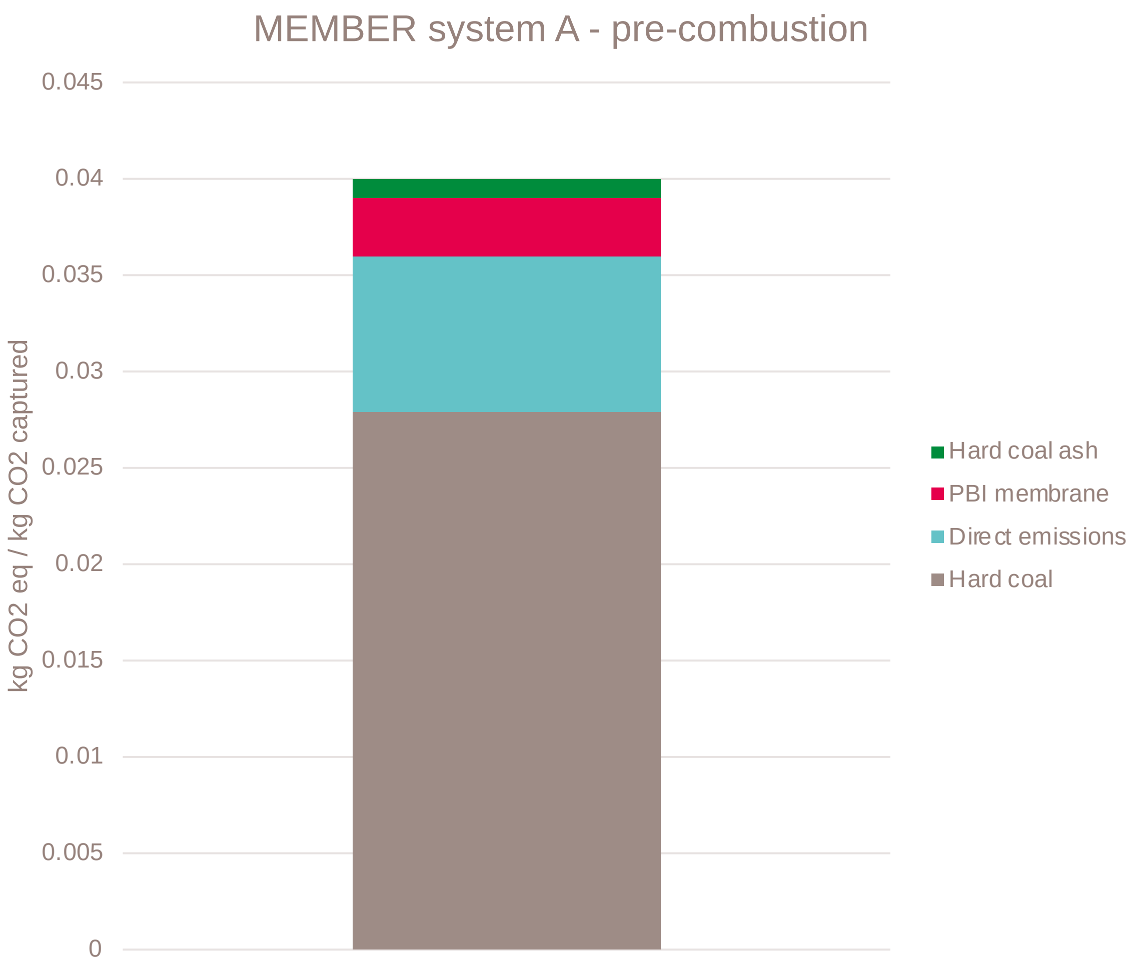
Method – system boundaries for post-combustion CO_2-capture



Method – system boundaries for H_2 production



Life Cycle Impact Assessment – for pre-combustion CO_2-capture

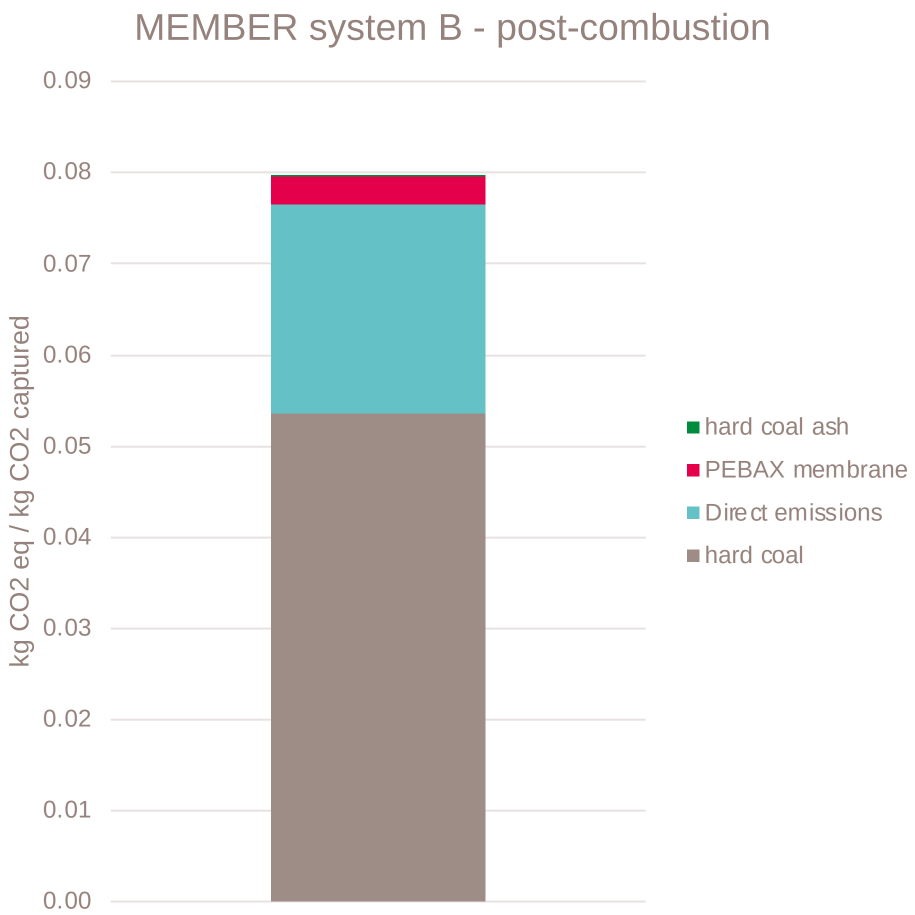


Main contributors:

- Hard coal: direct emissions during mining, particularly methane
- Direct emissions: mainly CO_2 that isn't captured (i.e. 10%)
- PBI membrane: Methanol and Hexane to wastewater
- Hard coal ash: Process specific burdens and landfill emissions

Quantis

Life Cycle Impact Assessment – for post-combustion CO_2-capture

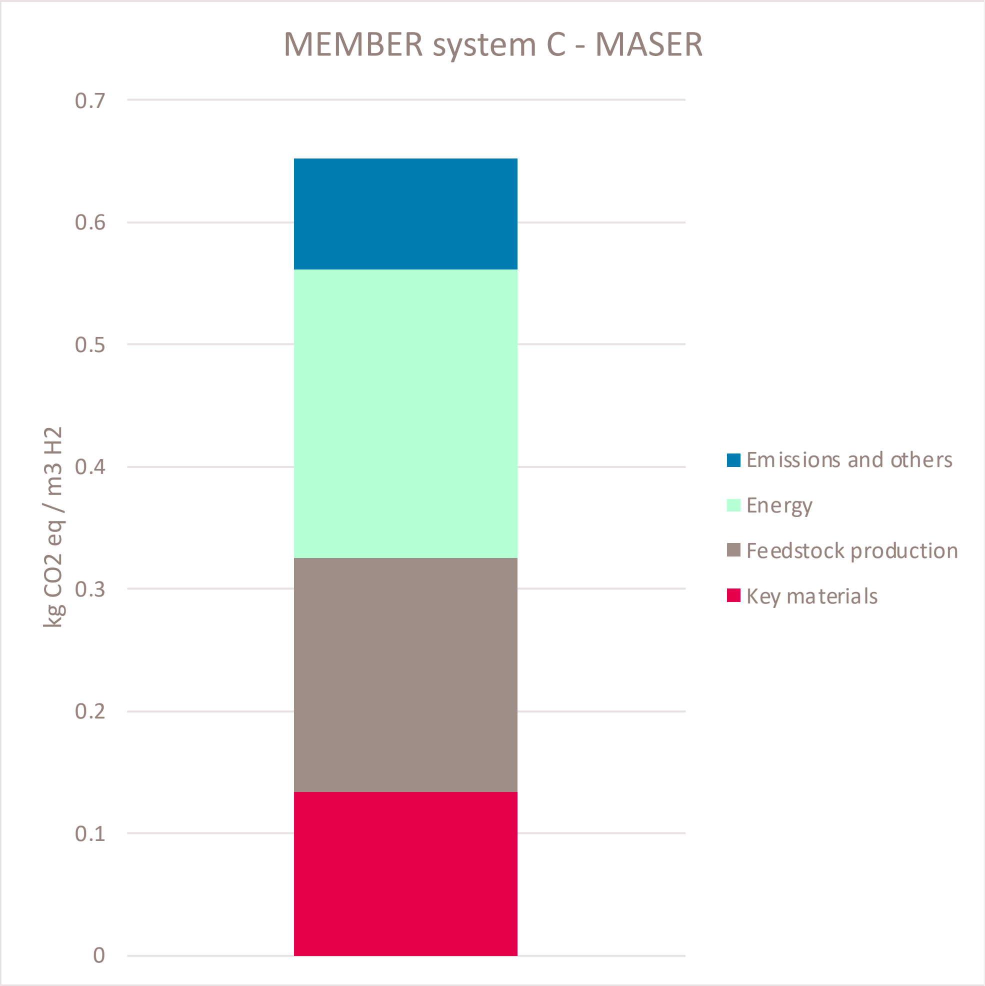


Main contributors:

- Hard coal: direct emissions during mining, particularly methane
- Direct emissions: mainly CO_2 that isn't captured (i.e. 10%)
- PEBAX membrane: 31% NMP solvent, 25% PVP pore former, 17% PSU polymer
- Hard coal ash: Process specific burdens and landfill emissions

Quantis

Life Cycle Impact Assessment – for H_2 production

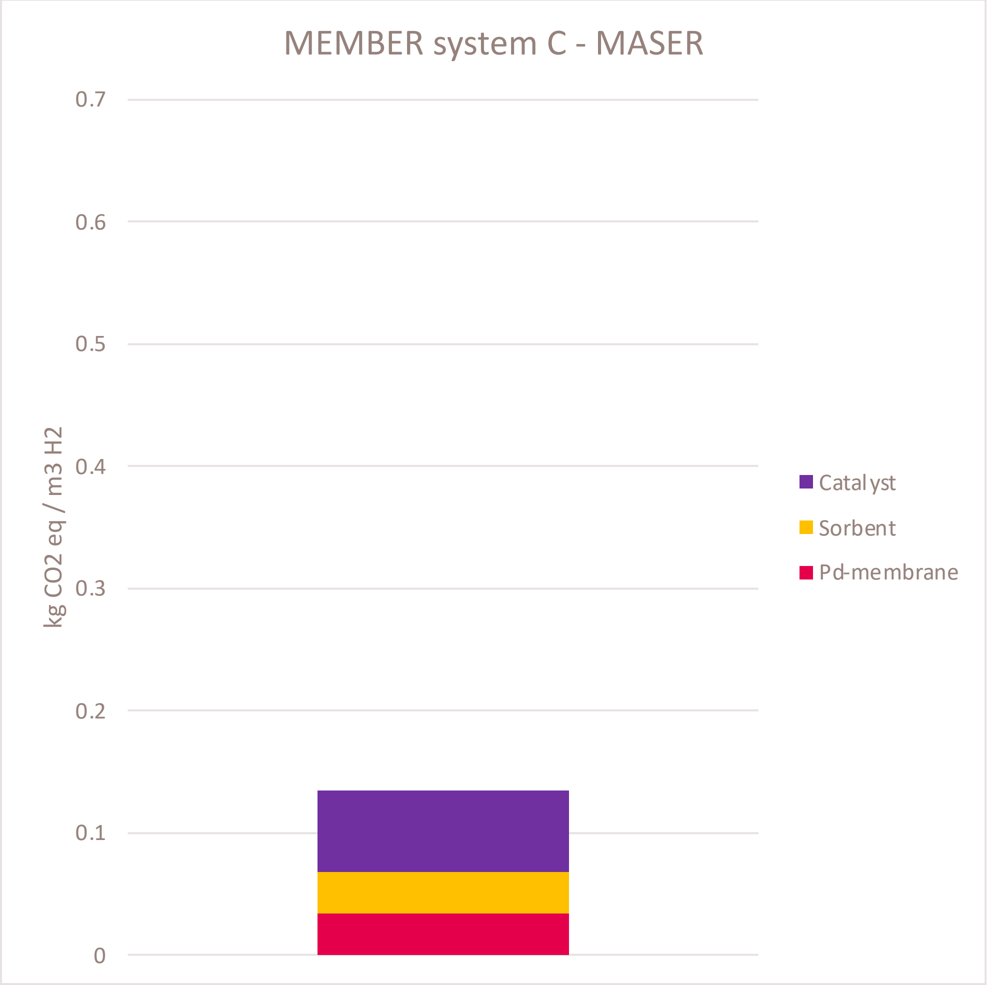


Main contributors:

- Feedstock: leakage of natural gas along the supply chain
- Energy: Fossil fuel combustion in the electricity mix
- Key materials: raw materials, energy during production and attrition of material

Quantis

Life Cycle Impact Assessment – zoom on key materials in H_2 production

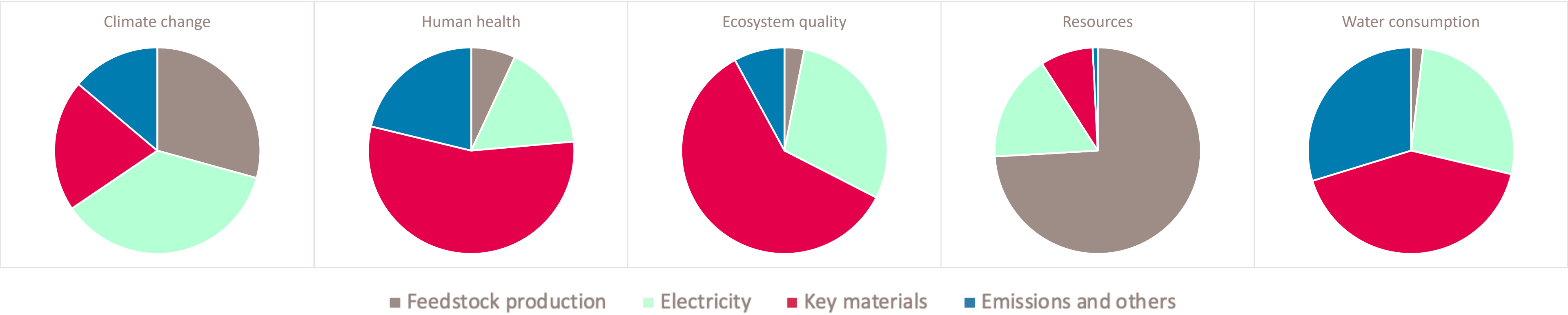


Main contributors:

- Catalyst
  - raw material input
  - attrition rate
- Sorbent
  - energy for production
  - attrition rate
- Pd-based membrane
  - raw material input
  - lifetime of membrane

Life Cycle Impact Assessment – for H_2 production

Other environmental indicators show a different distribution of hot spots.



Main contributors per impact category:

- Climate change: Feedstock production
- Human health: Emissions from mining activities for minerals
- Ecosystem quality: Emissions from mining activities for minerals and other direct emissions from combustion of feedstock
- Resources: Use of fossil resources
- Water consumption: Electricity consumption (cooling water)

Summary and key take-aways

Pre- and post-combustion CO_2-capture

- The main influencing parameter is the **energy** needed for **CO_2 capture**
- The **CO_2 capture rate** has a further impact on the systems' environmental performance

Hydrogen production

- **Electricity** and **feedstock production** have a significant influence on the environmental performance of the system
- key materials have a visible contribution with good potential for optimisation

The developed MEMBER systems show a **good environmental performance** compared to existing reference technologies with further promising improvement with higher technological maturity.

Advanced MEMBranes and membrane assisted procEsses for pre- and post- combustion CO_2 captuRe

https://member-co2.com/

Thank you for your attention

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Acknowledgement: For the CO_2 molecule used in the logo:The original uploader was Frederic Marbach at French Wikipedia [GFDL (http://www.gnu.org/copyleft/fdl.html)