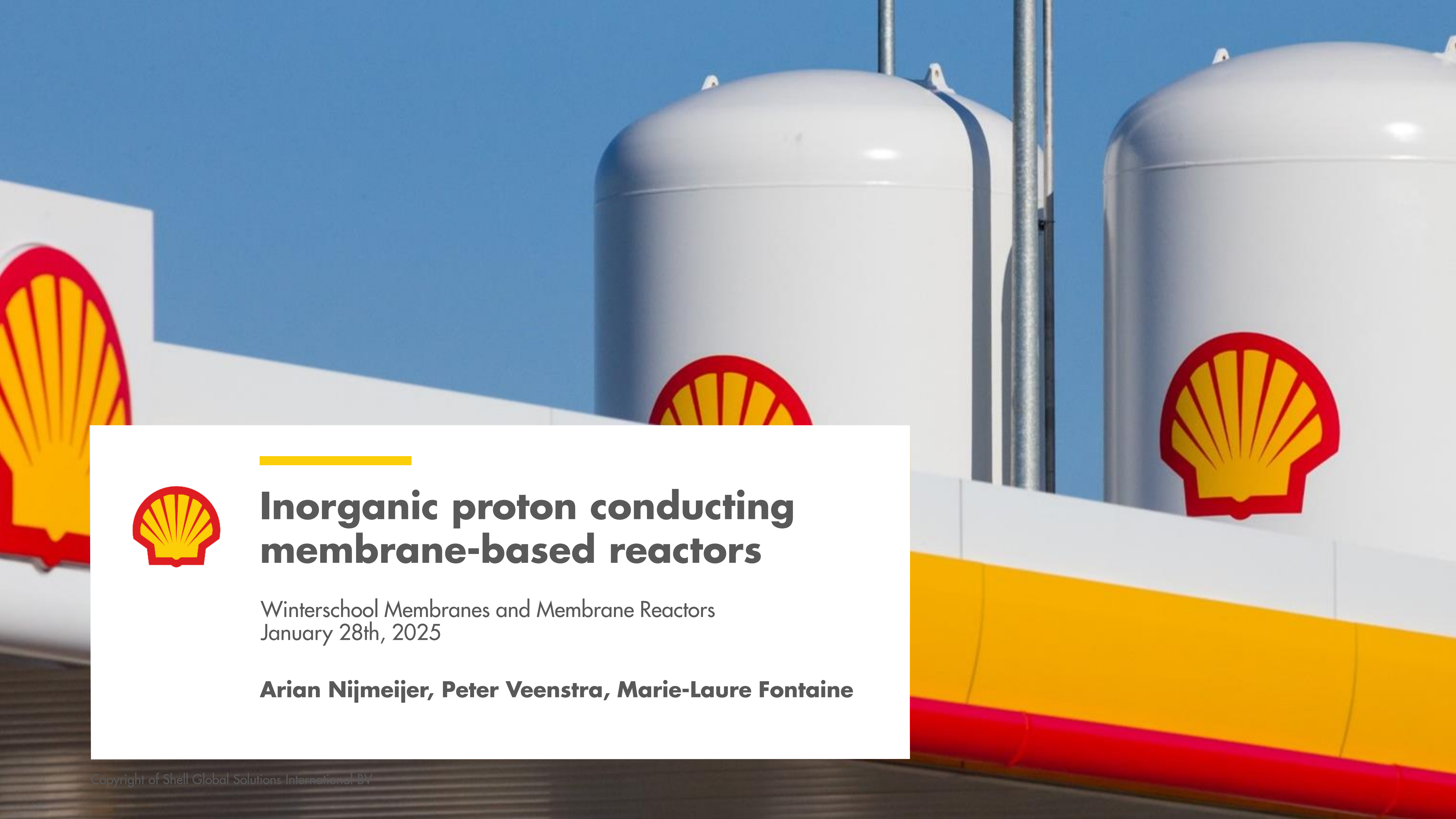


# Inorganic proton conducting membrane-based reactors

Winterschool Membranes and Membrane Reactors
January 28th, 2025

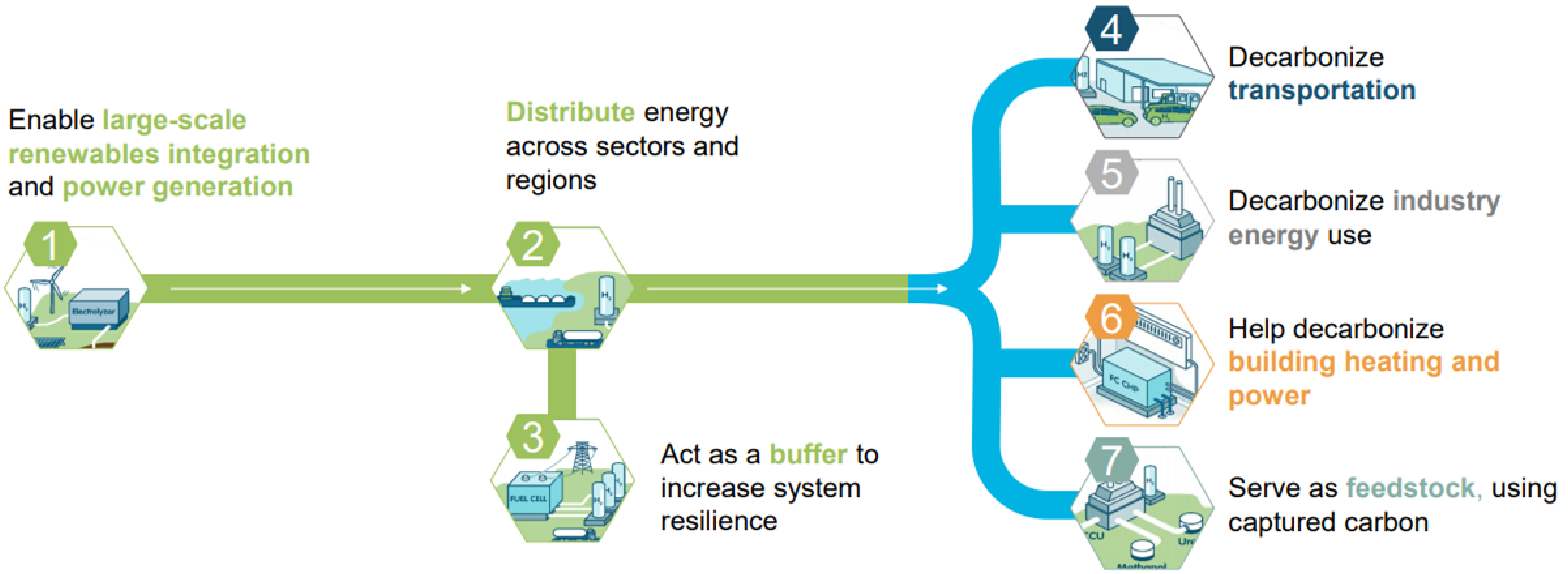
**Arian Nijmeijer, Peter Veenstra, Marie-Laure Fontaine**


# Hydrogen

A fuel for transport and industry

# Hydrogen is the only molecular zero carbon vector and sits at the intersection of three systems: mobility, industrial and energy

Enable the renewable energy system → Decarbonize end uses

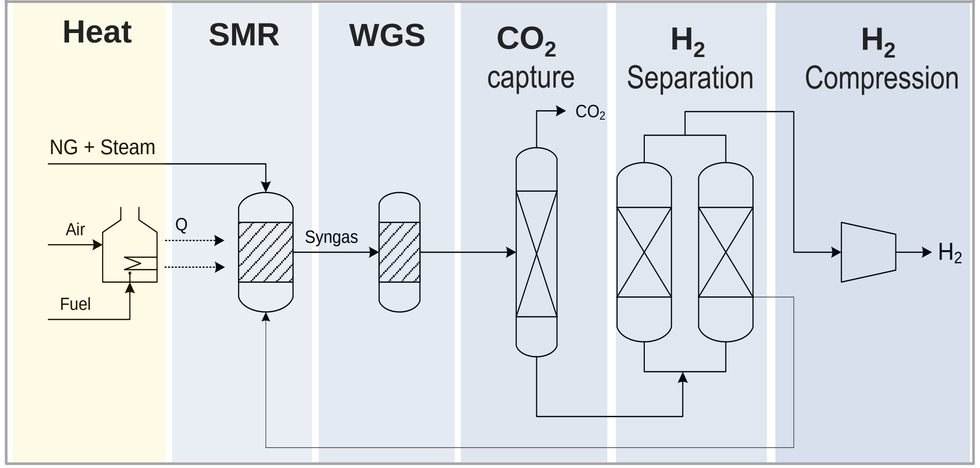



# PROTONIC

# Solution
## Process intensification with electrochemical ceramic membranes

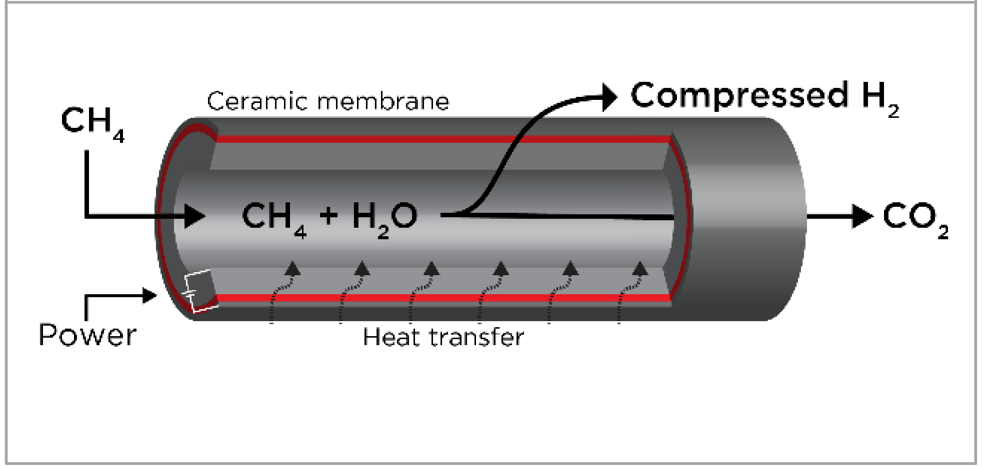
CoorsTek MEMBRANE SCIENCES

**Steam Methane Reforming w. CCS (6 steps)**




**Process step reduction**

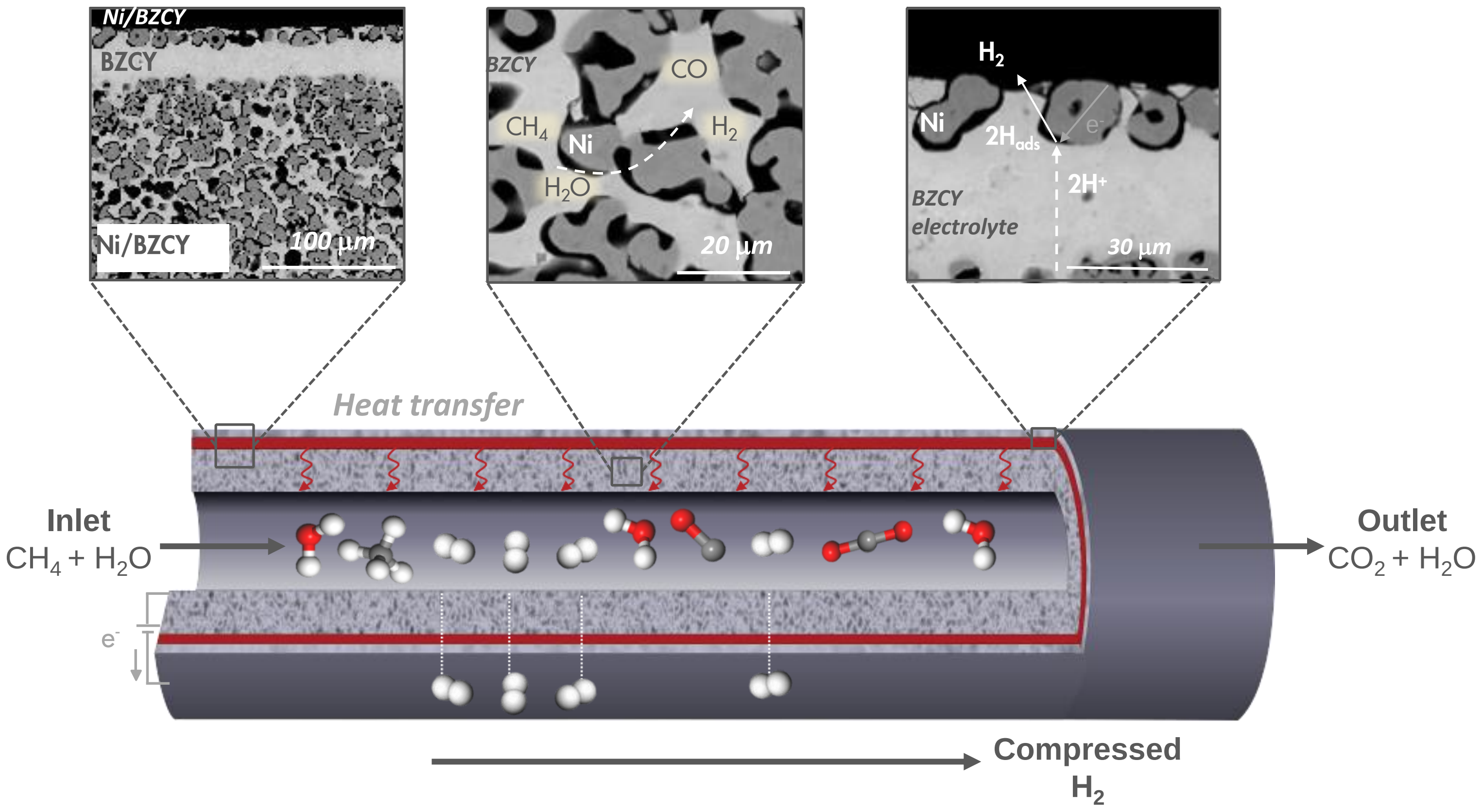
**Protonic Membrane Reforming (1 step)**

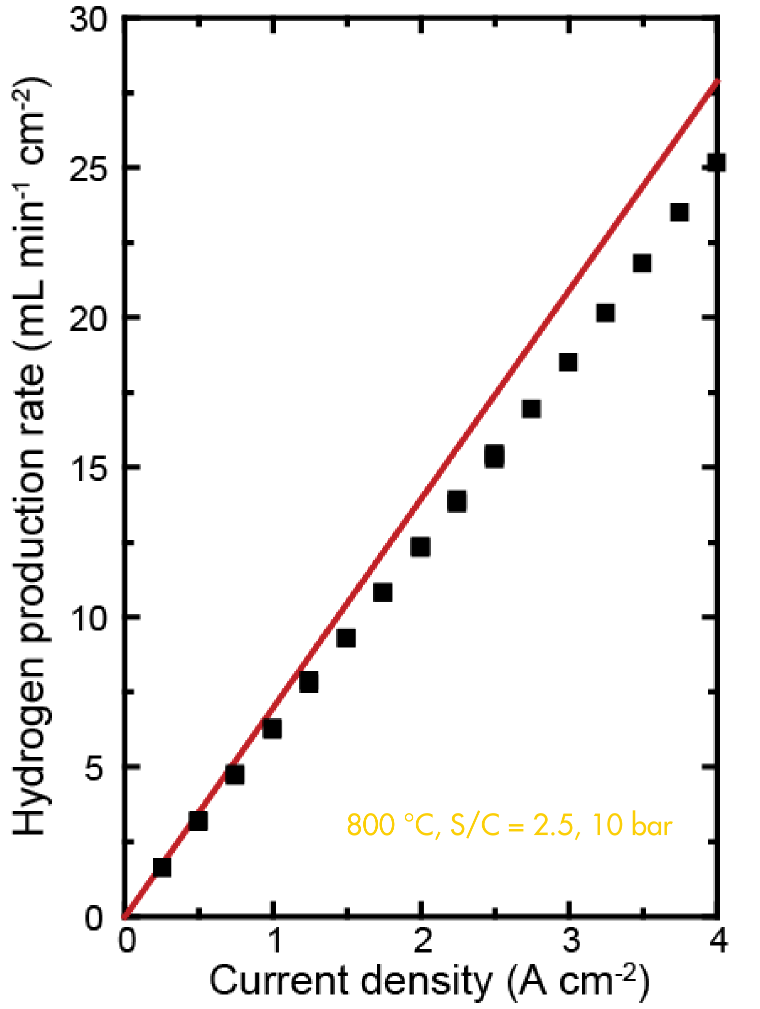



**Background and techno-economics:** Malerød-Fjeld et al., ***Nature Energy***, Thermo-electrochemical production of compressed hydrogen from methane with near-zero energy loss, **https://www.nature.com/articles/s41560-017-0029-4**

# Protonic Membrane Reformer (PMR)

Selective extraction of hydrogen



# Scaling up protonic ceramics for hydrogen production

Single cell (15 cm$^2$)

First stack design (~190 cm$^2$)

Proton ceramic electrochemical reactor (PCER) stack (584 cm$^2$)

PCER Panel (2920 cm$^2$)


Modular on-site $H_2$
1000 kg/day
250 kg/day
$H_2$
COORSTEK
MEMBRANE SCIENCES
Copyright of Shell Global Solutions International BV

GAMER

# Game changer in high temperature steam electrolysers

GOAL: Demonstrate high temperature (600 °C) steam electrolysis with novel tubular cells integrated in a 10 kW module for dry pressurized hydrogen production (30 bar)

- Novel electrolyser concept
- Mass manufacturing of cells
- Efficient thermal management by coupling of electrolyser system with
  - Renewable or waste heat sources
  - Steam
  - Renewable electricity

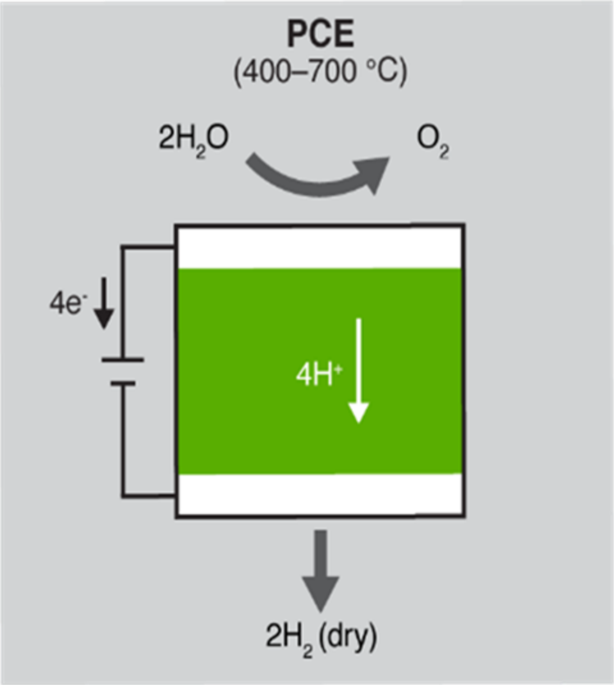
Methanol Plant
Refineries
Ammonia plant

| Partners | Country |
| --- | --- |
| SINTEF (coordinator) | Norway |
| Carbon Recycling International | Iceland |
| CSIC-ITQ | Spain |
| Coorstek Membrane Science AS | Norway |
| University of Oslo | Norway |
| MC2 Ingenieria y Sistemas SL | Spain |
| Shell Global Solutions International BV | Netherlands |

*Advisors: YARA and Air Liquide*

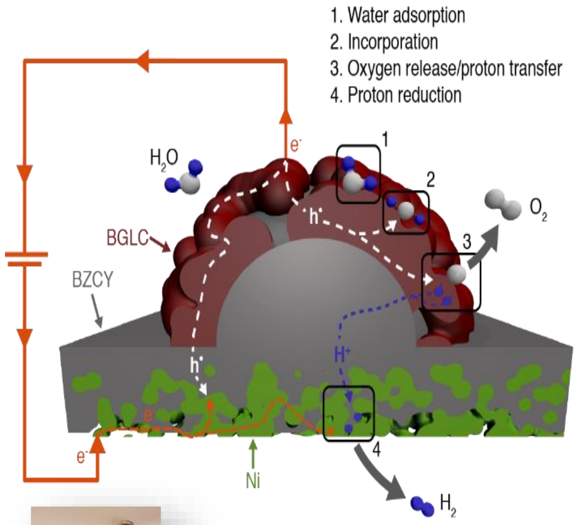
GAMER is a European research project co-financed by the European Union's Horizon 2020 research and innovation program and the Fuel Cells and Hydrogen Joint undertaking under grant 779486

# GAMER focus




P = 30 bar

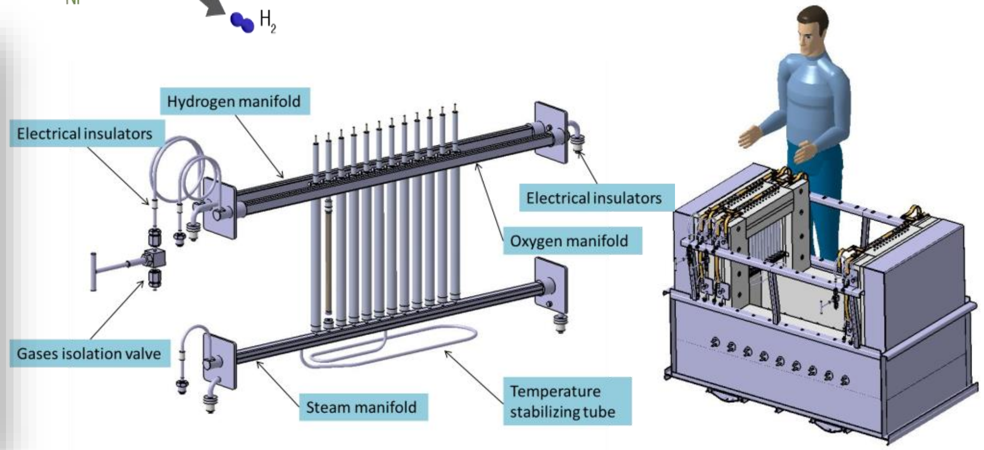
Electrodes




"*Mixed proton and electron conducting double perovskite anodes for stable and efficient tubular proton ceramic electrolysers*", Nature Materials (2019);

Cell architectures

10 kW system with BoP




Efficiency & LCA

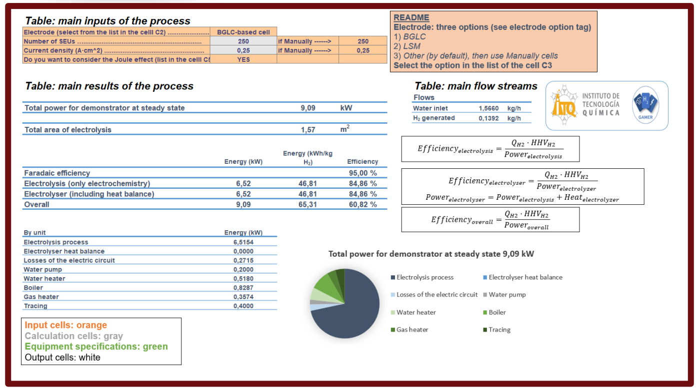


*Table: main inputs of the process*

| | | | |
|---|---|---|---|
| Electrode (select from the list in the celll C2) ..................... | BGLC-based cell | | |
| Number of SEUs ........................................................... | 250 | if Manually ------> | 250 |
| Current density (A·cm^2) ................................................ | 0,25 | if Manually ------> | 0,25 |
| Do you want to consider the Joule effect (list in the celll C5 | YES | | |

**README**
**Electrode: three options (see electrode option tag)**
1) *BGLC*
2) *LSM*
3) *Other (by default), then use Manually cells*
**Select the option in the list of the cell C3**

*Table: main results of the process*

| | | |
|---|---|---|
| Total power for demonstrator at steady state | 9,09 | kW |
| Total area of electrolysis | 1,57 | $m^2$ |

| | Energy (kW) | Energy (kWh/kg $H_2$) | Efficiency |
|---|---|---|---|
| Faradaic efficiency | | | 95,00 % |
| Electrolysis (only electrochemistry) | 6,52 | 46,81 | 84,86 % |
| Electrolyser (including heat balance) | 6,52 | 46,81 | 84,86 % |
| Overall | 9,09 | 65,31 | 60,82 % |

| By unit | Energy (kW) |
|---|---|
| Electrolysis process | 6,5154 |
| Electrolyser heat balance | 0,0000 |
| Losses of the electric circuit | 0,2715 |
| Water pump | 0,2000 |
| Water heater | 0,5180 |
| Boiler | 0,8287 |
| Gas heater | 0,3574 |
| Tracing | 0,4000 |

*Table: main flow streams*

| Flows | | |
|---|---|---|
| Water inlet | 1,5660 | kg/h |
| $H_2$ generated | 0,1392 | kg/h |

$$Efficiency_{electrolysis} = \frac{Q_{H2} \cdot HHV_{H2}}{Power_{electrolysis}}$$

$$Efficiency_{electrolyser} = \frac{Q_{H2} \cdot HHV_{H2}}{Power_{electrolyzer}}$$

$$Power_{electrolyser} = Power_{electrolysis} + Heat_{electrolyzer}$$

$$Efficiency_{overall} = \frac{Q_{H2} \cdot HHV_{H2}}{Power_{overall}}$$

Total power for demonstrator at steady state 9,09 kW

Electrolysis process, Electrolyser heat balance, Losses of the electric circuit, Water pump, Water heater, Boiler, Gas heater, Tracing

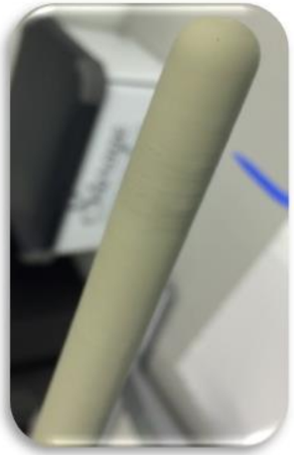
Input cells: orange
Calculation cells: gray
Equipment specifications: green
Output cells: white

"*Multiscale multiphysic modelling tools GES.VI*"

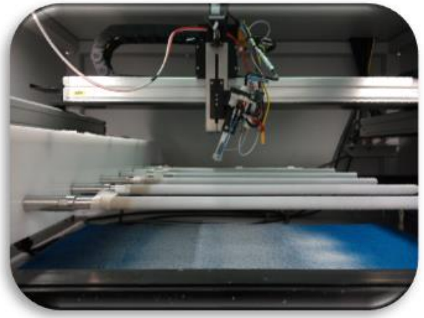
For more information about GAMER: https://www.sintef.no/projectweb/gamer/

# Manufacturing routes

1. Extrusion with 40 ton automatic extruder (capping/cutting)



2. Spray-coating of SSRS based electrolyte with automatic spray-coater



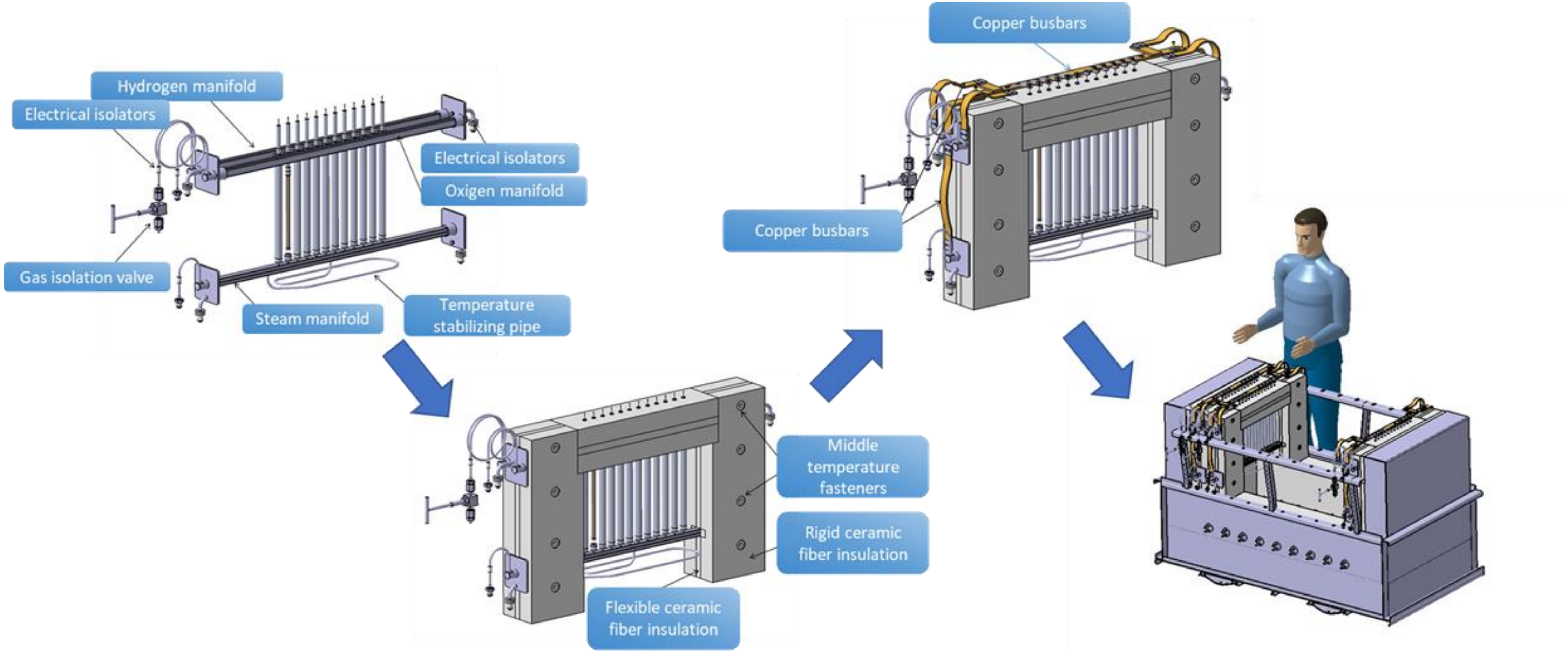
3. Co-sintering of electrode/electrolyte

4. Dip-coating of electrode with semi-automatic dip-coater and firing

*100 $m^2$ clean room class 7*

This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 779486. This Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe Research.

# Electrolyser design

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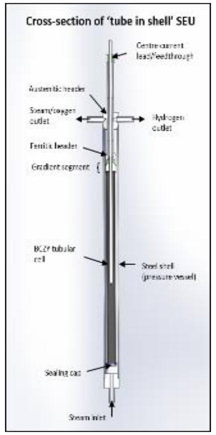
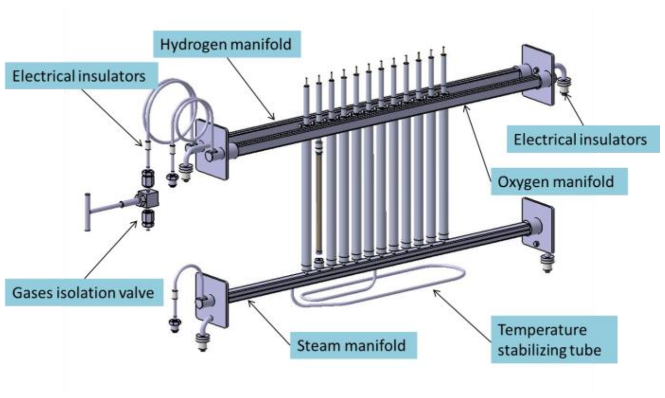
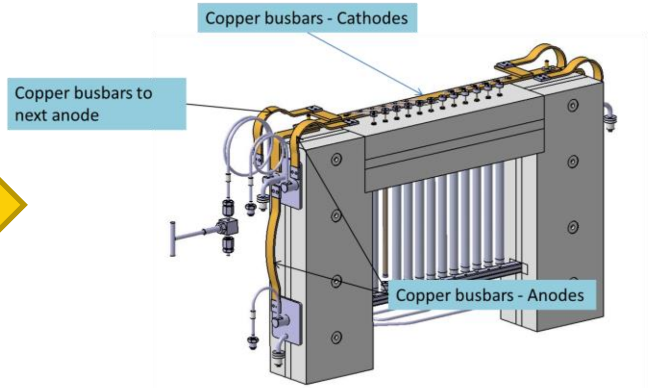
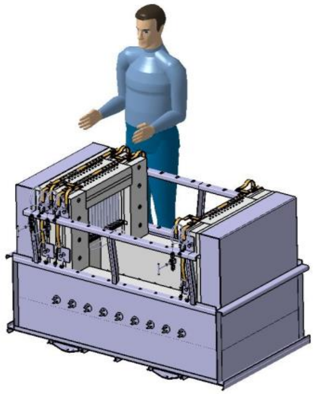
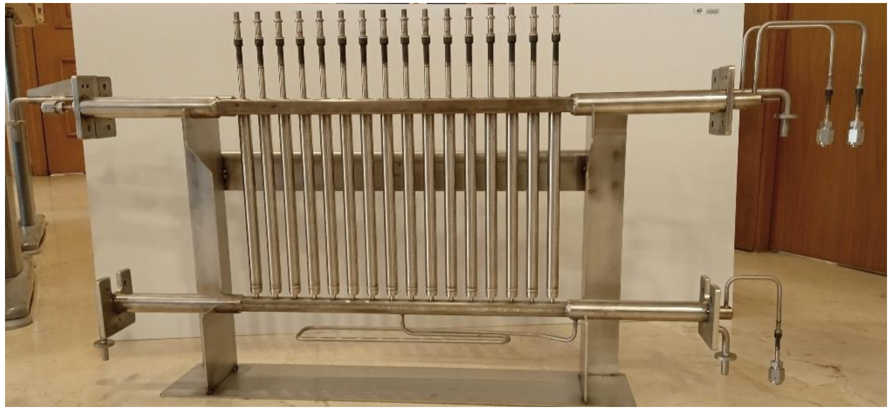
# From SEU to multi-SEUs in hot box





*Tested at RT at 41 bar*

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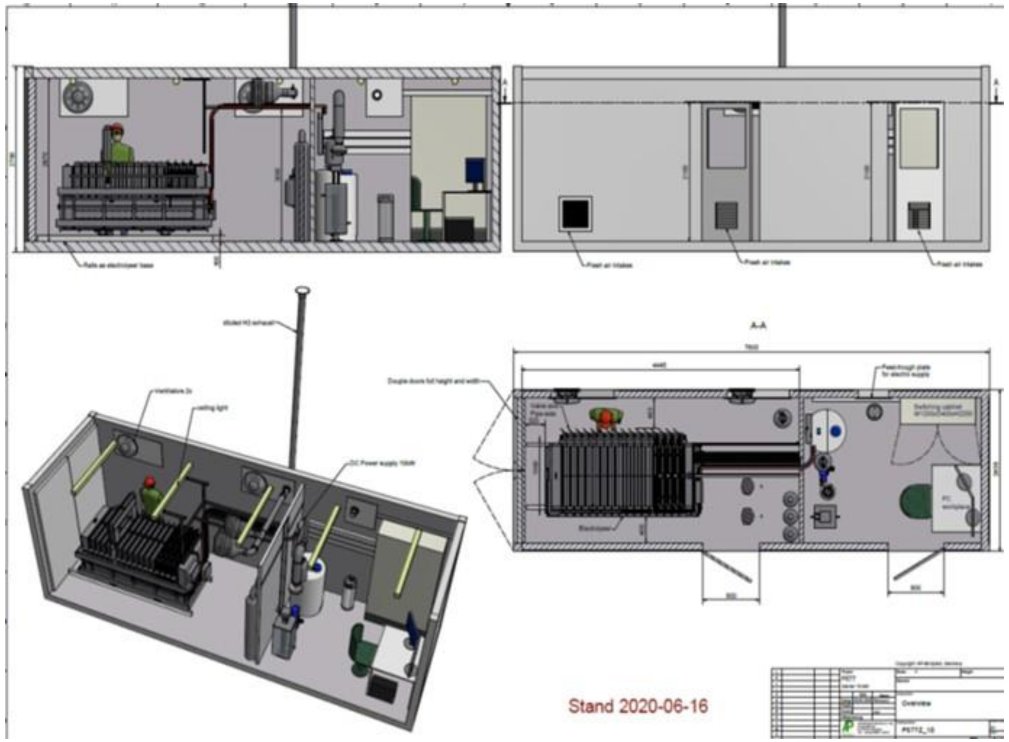
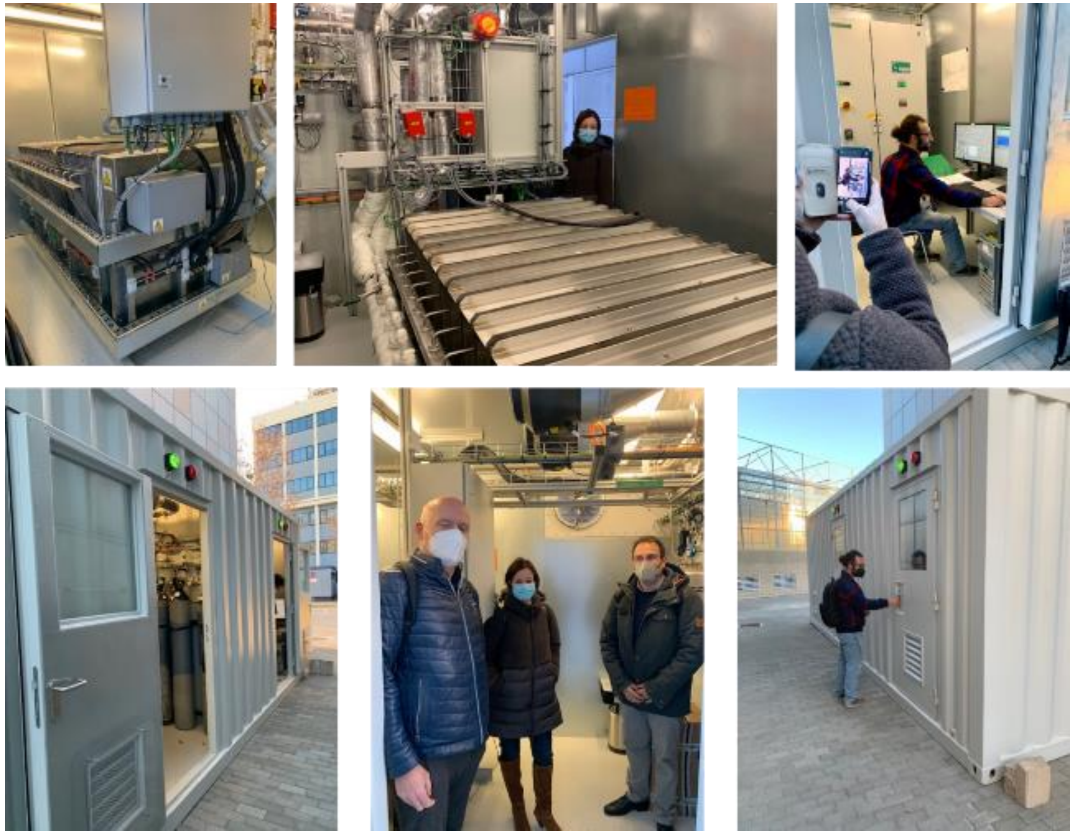
# Plant so far in commissioning phase



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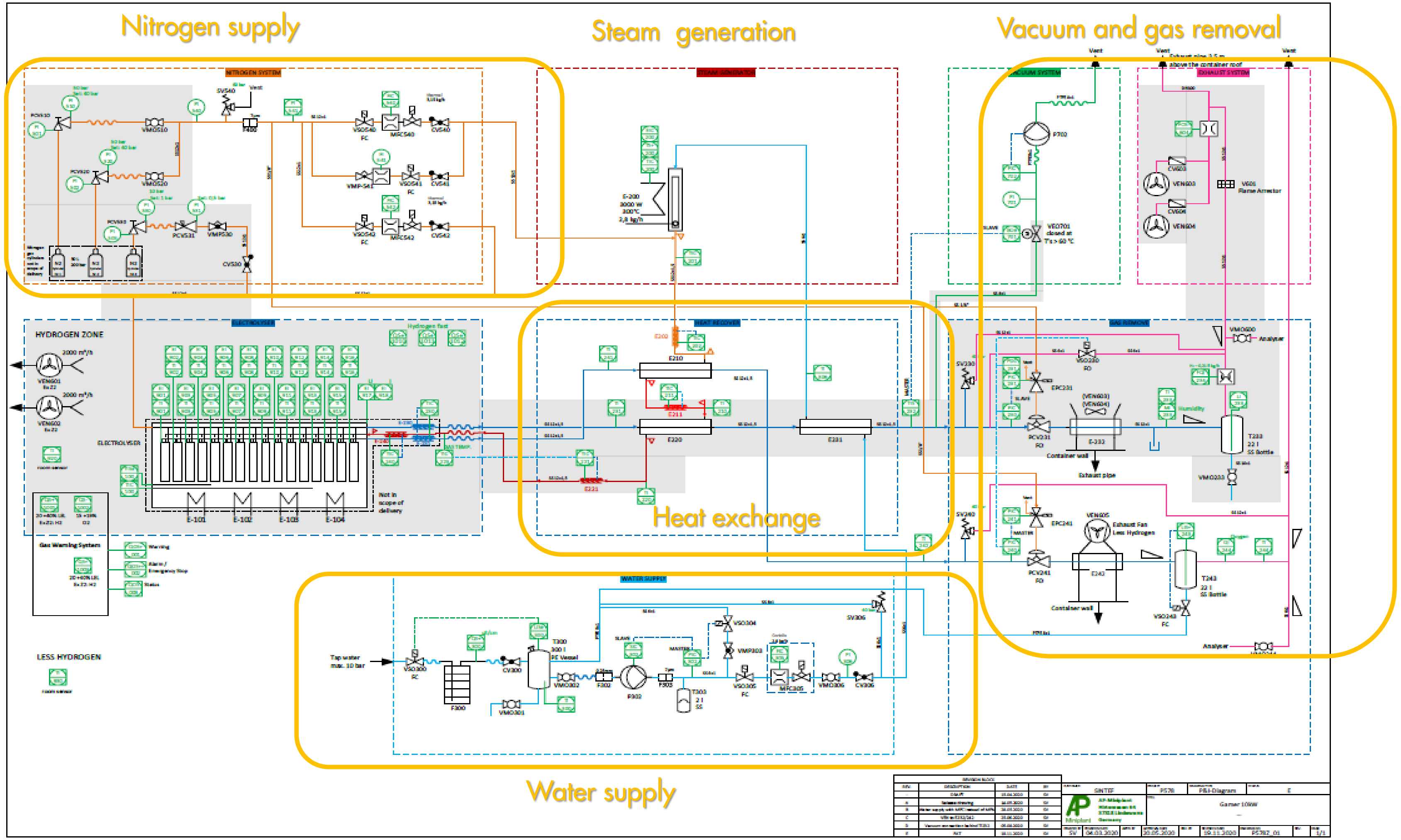
# Balance of Plant

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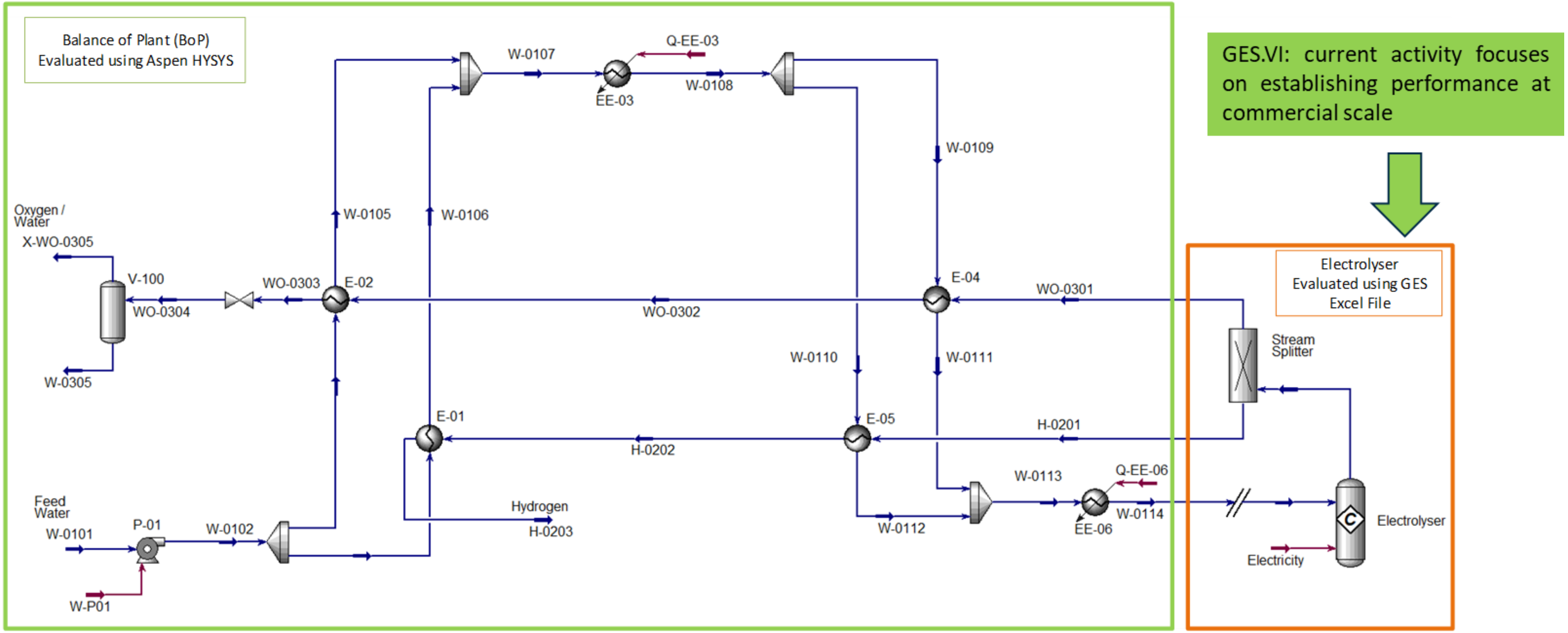




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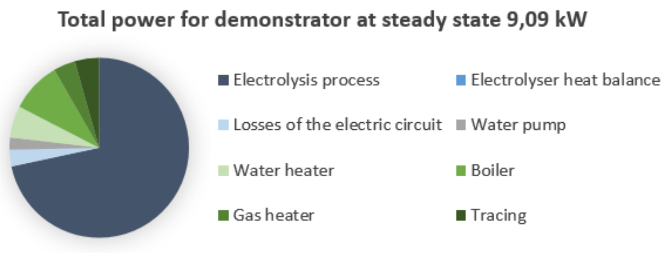


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PROTOSTACK

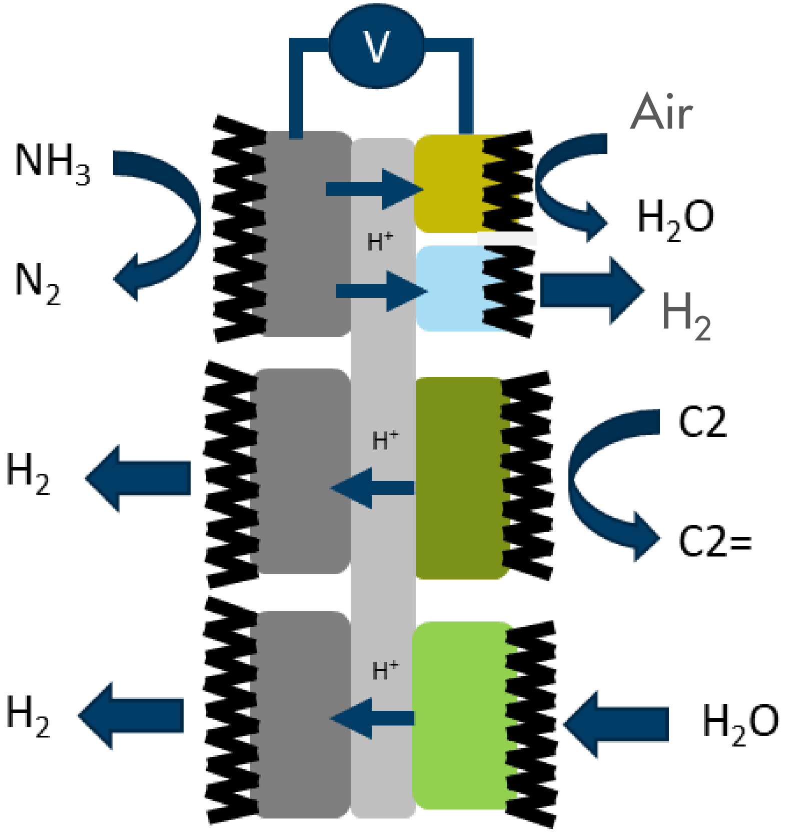
# Cell/stack design


The project is supported by the Clean Hydrogen Partnership and its members Hydrogen Europe and Hydrogen Europe Research

# World class Innovative Novel Nanoscale optimized electrodes and electrolytes for Electrochemical Reactions

WINNER

# Applications



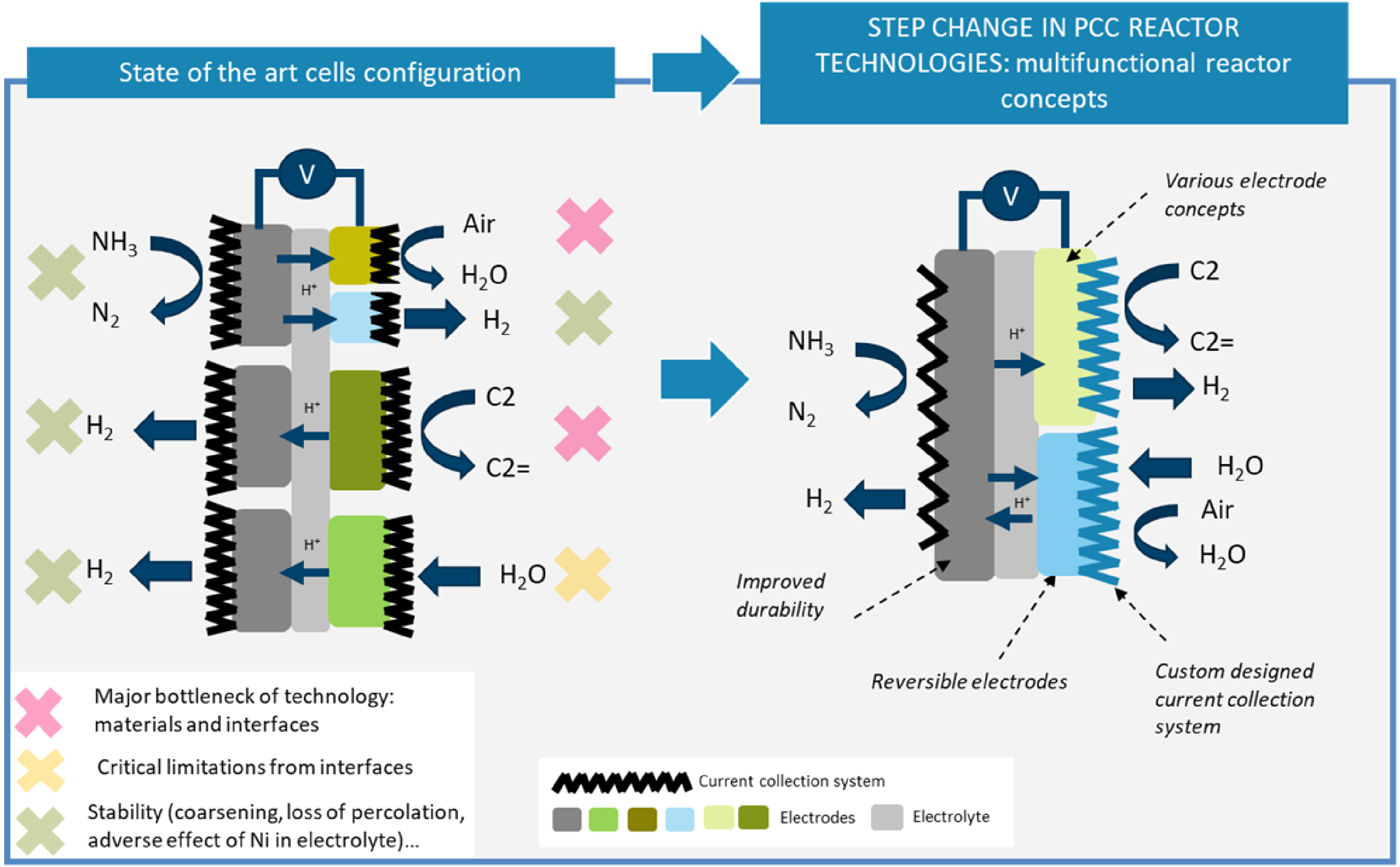

**PCC based technologies for:**

- **Cracking of ammonia to pressurized hydrogen or power**
- **Dehydrogenation of ethane to produce ethylene and pressurized hydrogen**
- **Reversible steam electrolysis**



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007165. This Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe Research.

# Applications

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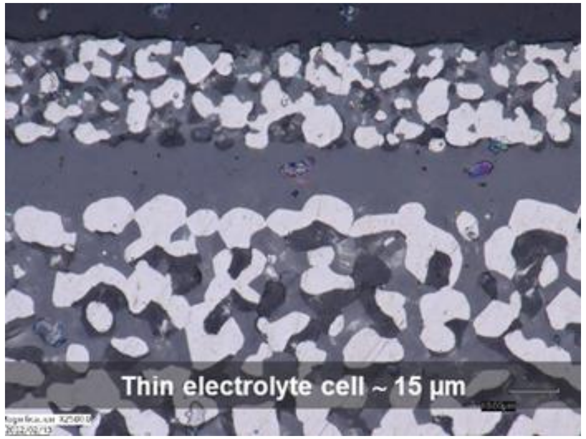
# Development of innovative architectures and cells

GOAL - Providing performing tubular cells for

- Reversible electrolysis – selecting for demonstrator scale-up
- Ammonia to power/hydrogen
- Ethane dehydrogenation

HOW

- 50% reduction of electrolyte thickness
- Development of new architecture with new electrolyte and adjustment of the fabrication steps
- Innovative electrode microstructure

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# Manufacturing line




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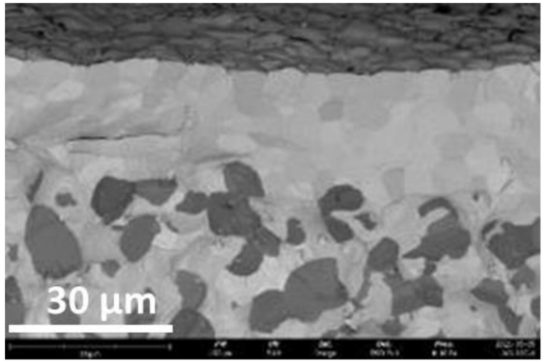
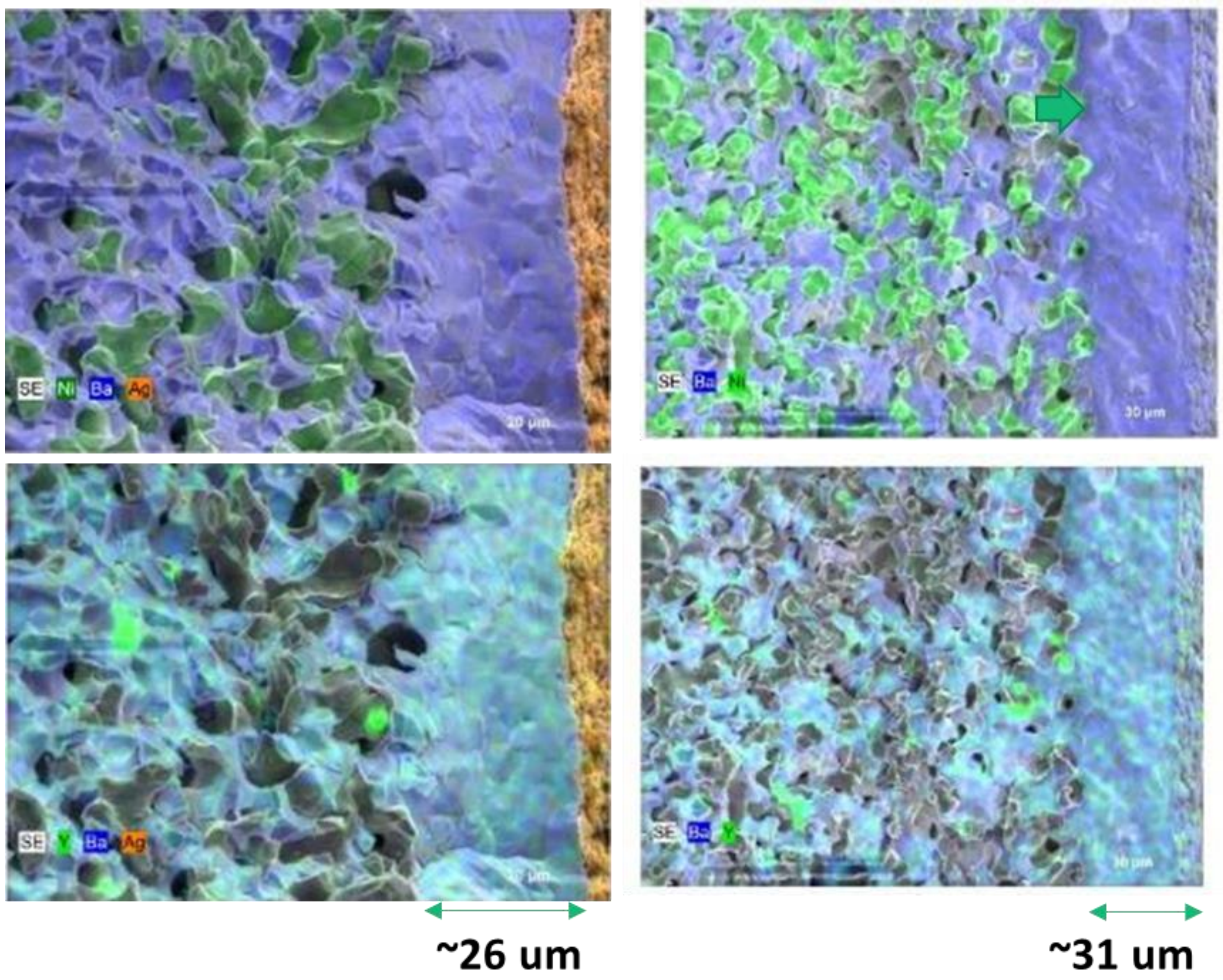
# Versatile manufacturing

## Solid State Reactive Sintering (NiO)

- BZCYYb4411: $BaZr_{0.4}Ce_{0.4}Y_{0.1}Yb_{0.1}O_{3-x}$
- BZCY532: $BaZr_{0.5}Ce_{0.3}Y_{0.2}O_{3-x}$
- BZCY442: $BaZr_{0.4}Ce_{0.4}Y_{0.2}O_{3-x}$
- BZCY721: $BaZr_{0.7}Ce_{0.2}Y_{0.1}O_{3-x}$



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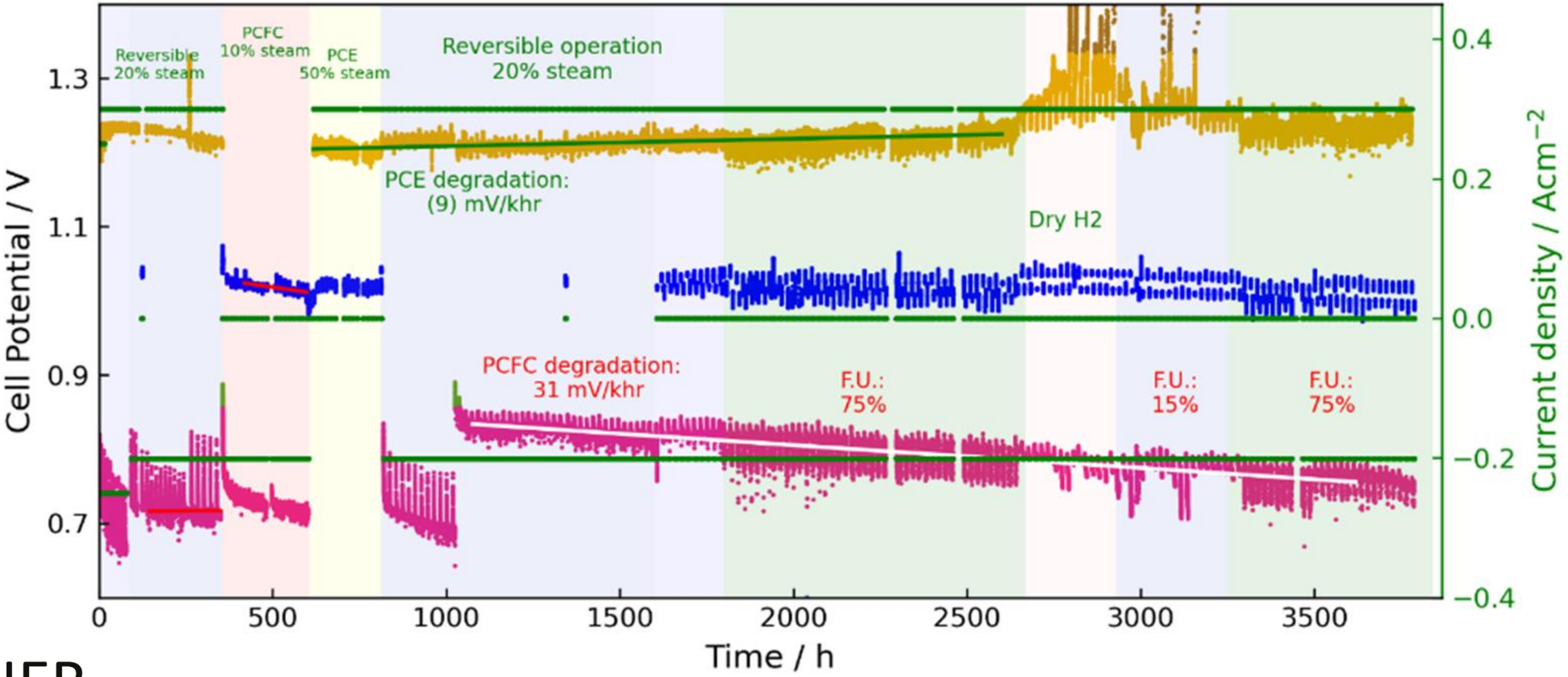
# 12 cm² cell in pressurized alumina vessel

Tested at 4 bar total pressure in reversible fuel cell and electrolysis mode




WINNER



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# Multi-scale multi-physics modelling platform

### Atomic

**Ea**
**$A^0$**
μ - chemical potential (i)
φ - electrical potential,
ΔG, ΔH, ΔS

### Electrode

U (I) - Cell voltage / current
η - Overpotential
Ea – Activation energy
$A^0$ - Pre-exponetial value
P - partial pressures (n, m)
S - Geometrical factor

### Cell

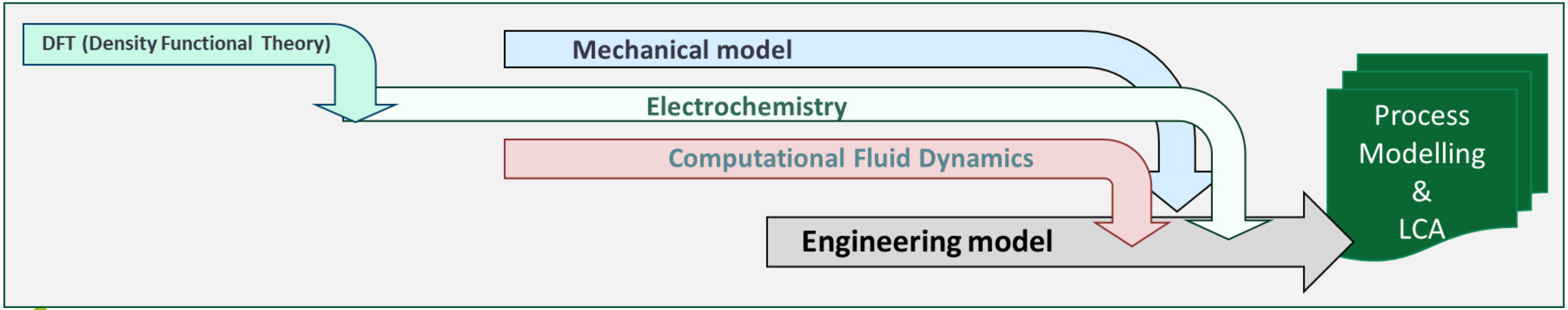
Diameter of cell
Length of cell
Efficiency per cell
ASR of cell
Power per cell
Flux per cell
Specific power per cell
Pressure rating

### Stack/reactor

Pressure Rating
Temperature rating
Reactor Diameter
Reactor Length
Number of cells per reactor
Number of reactors
Efficiency per reactor
Power per reactor
Voltage
Specific power (W/m³, W/kg)
Space Time Velocity

### System (BoP)

Throughput
Process efficiency
Pressure
Temperature
Product quality




WINNER



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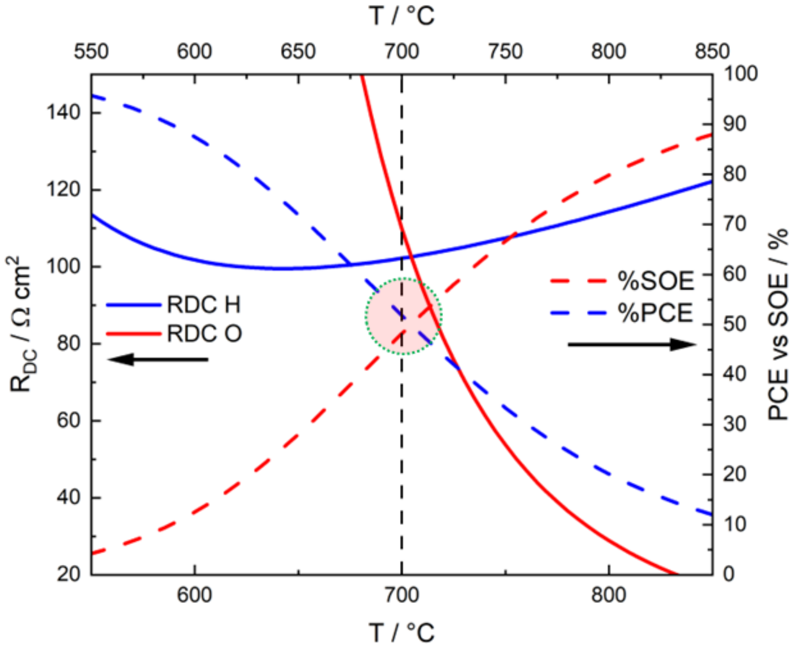
# Electrochemistry & kinetics

- Kinetic model has been developed and implemented:

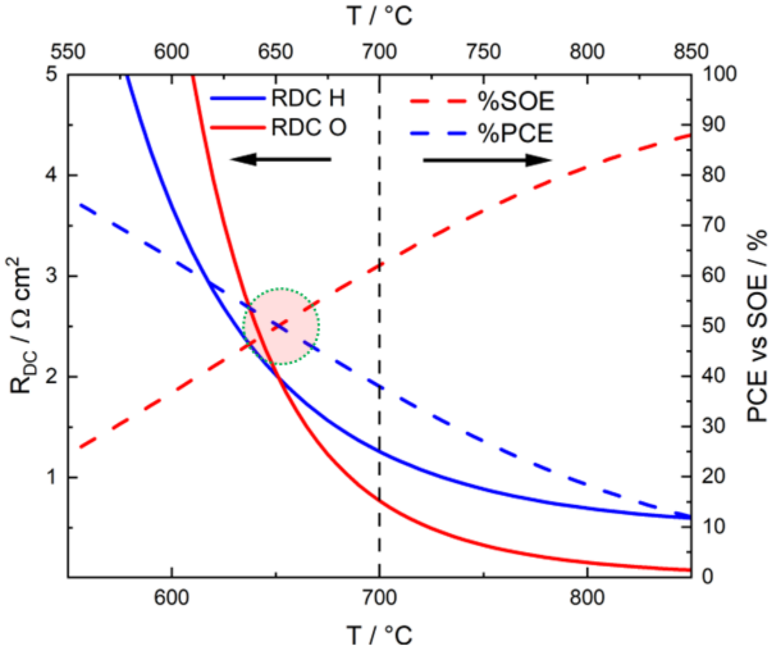
$$\frac{1}{R_{p,i,j}} = nFpO_2^{n(i,j)}pH_2O^{m(i,j)}A^0_{,i,j}S_{x,y,z}exp\left(-\frac{E_{A,i,j}}{RT}\right)$$

- Application to realistic application: Selectivity of electrolyte and electrodes for protons and oxide ions

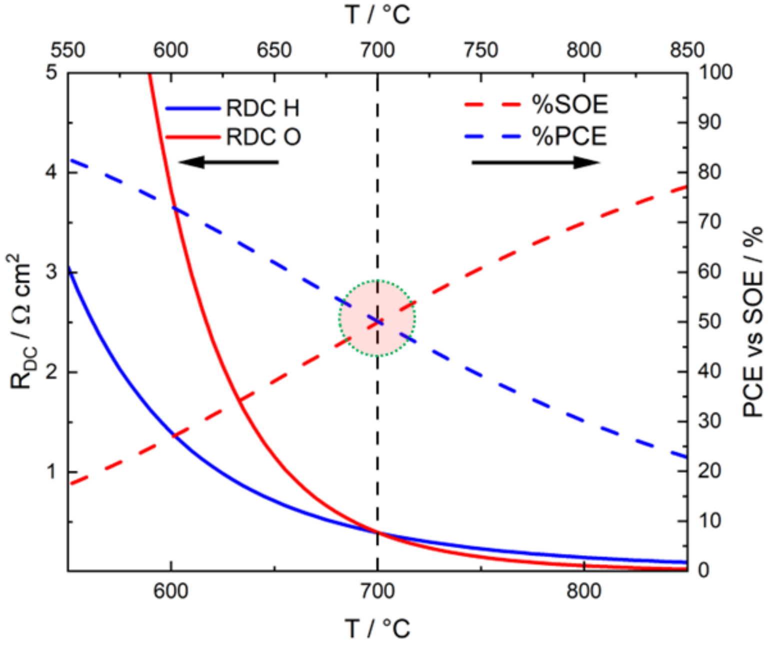
**1 cm** electrolyte, ***p*$H_2O$: 0.026 atm**




**30 µm** electrolyte, ***p*$H_2O$: 0.026 atm**




**30 µm** electrolyte, ***p*$H_2O$: 0.5 atm**



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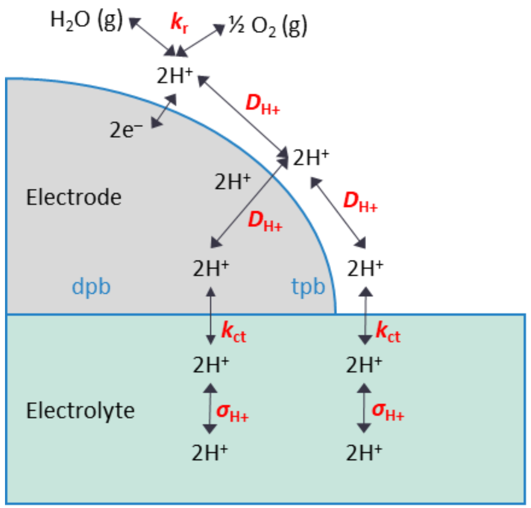
# Atomistic modelling




**Electrode/gas interface:** $2H_2O \rightleftarrows 4H^+ + O_2(g) + 4e^-$

| | |
|---|---|
| $2H_2O + *_b \rightleftarrows 2 * H_2O_b$ | water adsorption |
| $2 * H_2O_b + *_O \rightleftarrows 2 * OH_b^- + 2 * H_O^+$ | water dissociation |
| $2 * OH_b^- + 4 *_O \rightleftarrows 2 * O_O^0 + 2 * H_O^+ + *_b + 4e^-$ | hydroxide dissociation |
| $2 * O_O \rightleftarrows * O_2(TS) \rightleftarrows O_2(g) + 2 *_O$ | oxygen association |

**Diffusion on/in the electrode:**

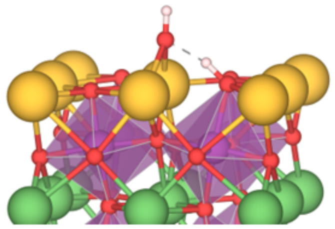
| | |
|---|---|
| $* H_O^+ + *_O \rightleftarrows *_O + * H_O^+$ | H+ surface diffusion |
| $OH_O^+ + O_O \rightleftarrows O_O + OH_O^+$ | H+ bulk diffusion |

**Charge transfer across the electrode/electrolyte interface:**

| | |
|---|---|
| $* H_O^+ + O_{O,BZCY} \rightleftarrows *_O + OH_{O,BZCY}^+$ | charge transfer at tpb |
| $OH_O^+ + O_{O,BZCY} \rightleftarrows O_O + OH_{O,BZCY}^+$ | charge transfer at dpb |

**Water dissociation**

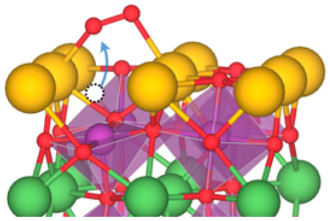
$* OH_b^- + * OH_O^+$



$E_{ads}$ = −1.21 eV

**Oxygen association**

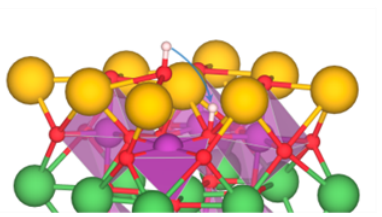
$* O_O + *_O \rightleftarrows * O_2 + V_O^{2+} + 2e^-$



$E_a$ = ~1.4 eV

**Surface diffusion**

$* H_O^+ + *_O \rightleftarrows * H_O^+ + *_O$



$E_a$ = 1.29 eV

## Kinetics from DFT

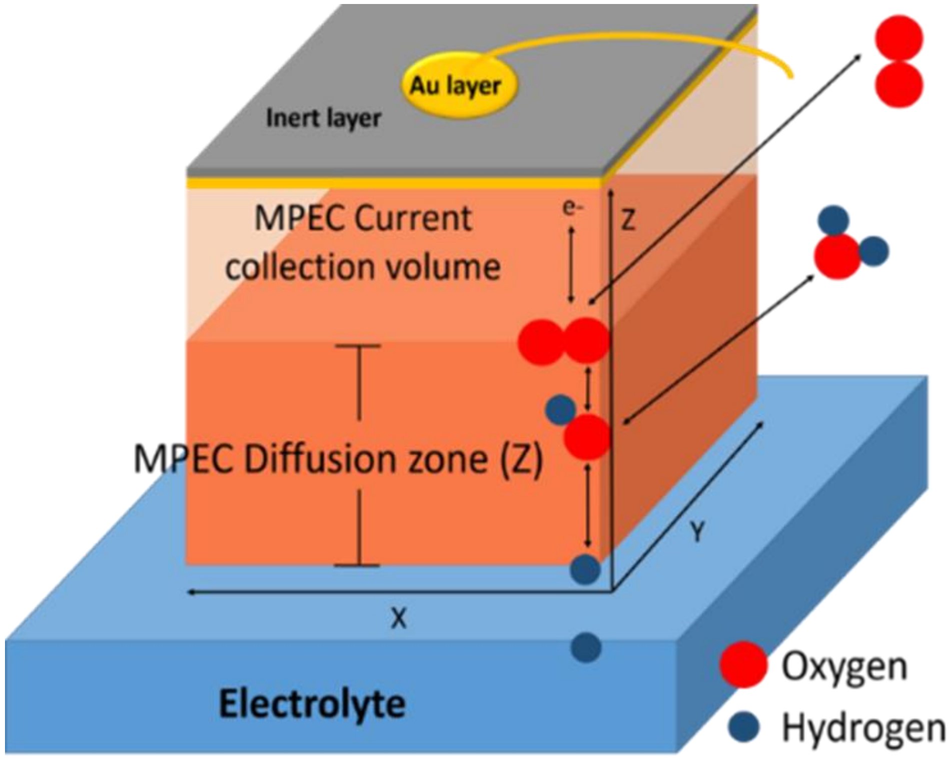
$$A_i^0 = \frac{k_B T}{h} \frac{\sum_j^N \left(1-\exp\left(-h\nu_j/k_B T\right)\right)}{\sum_j^{N-1}\left(1-\exp\left(-h\nu_j^*/k_B T\right)\right)}$$ pre-exponential

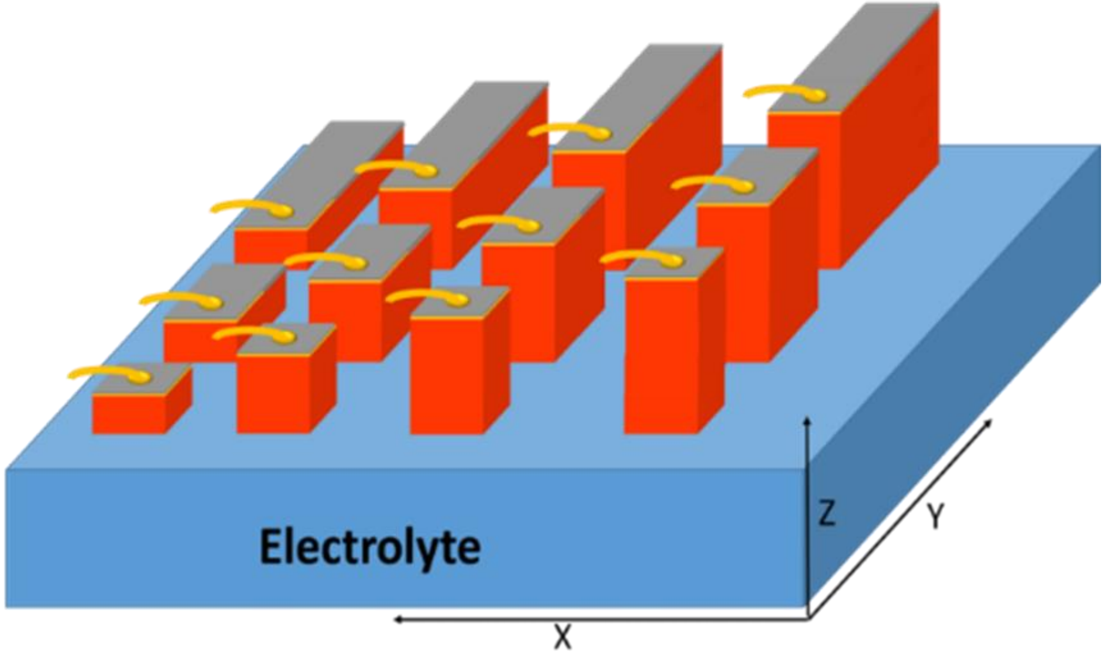
$$\Delta G_{a,i} = \Delta H_{a,i} - T\Delta S_{a,i}$$ activation energy

**Input for the electrochemistry model!**


This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007165. This Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe Research.

# Electrochemical modelling



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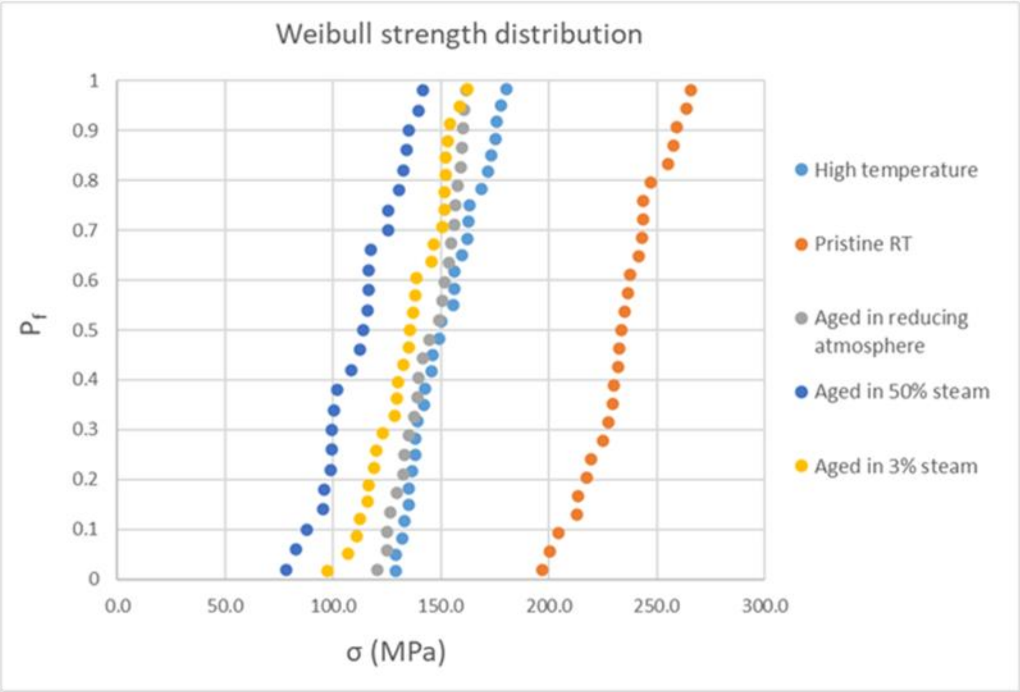
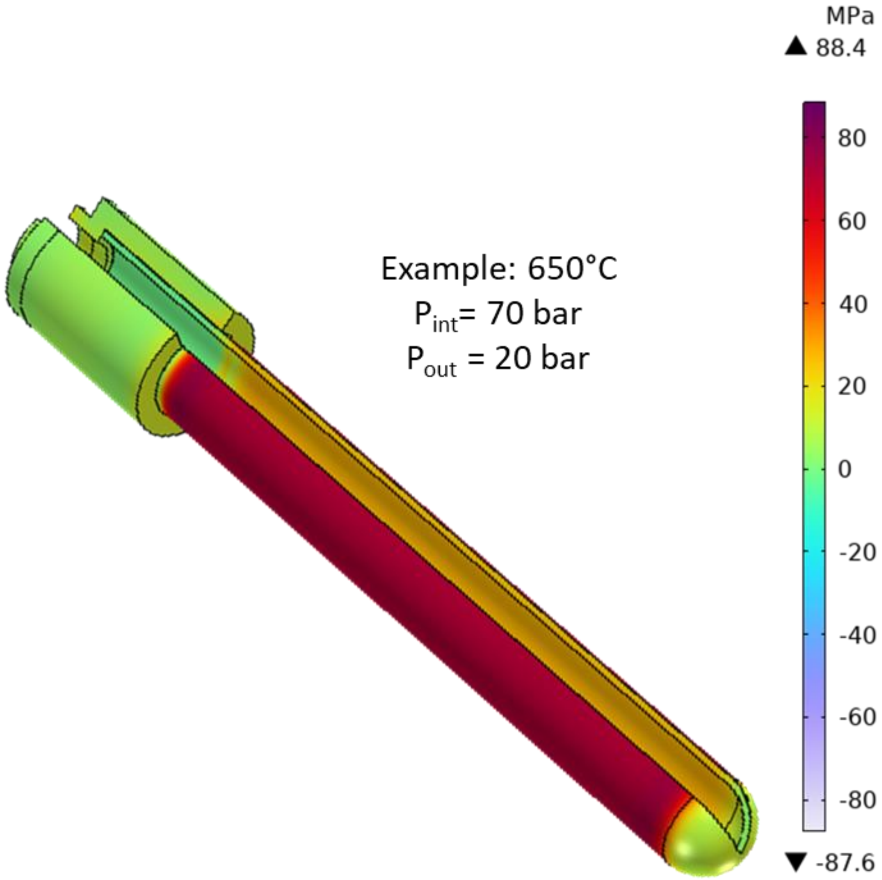
# Mechanical model




Glass $CaO/MgO/SiO_2$
Coorstek Patent PCT/EP2018/085958



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# Engineering model

1. **Ammonia cracking**

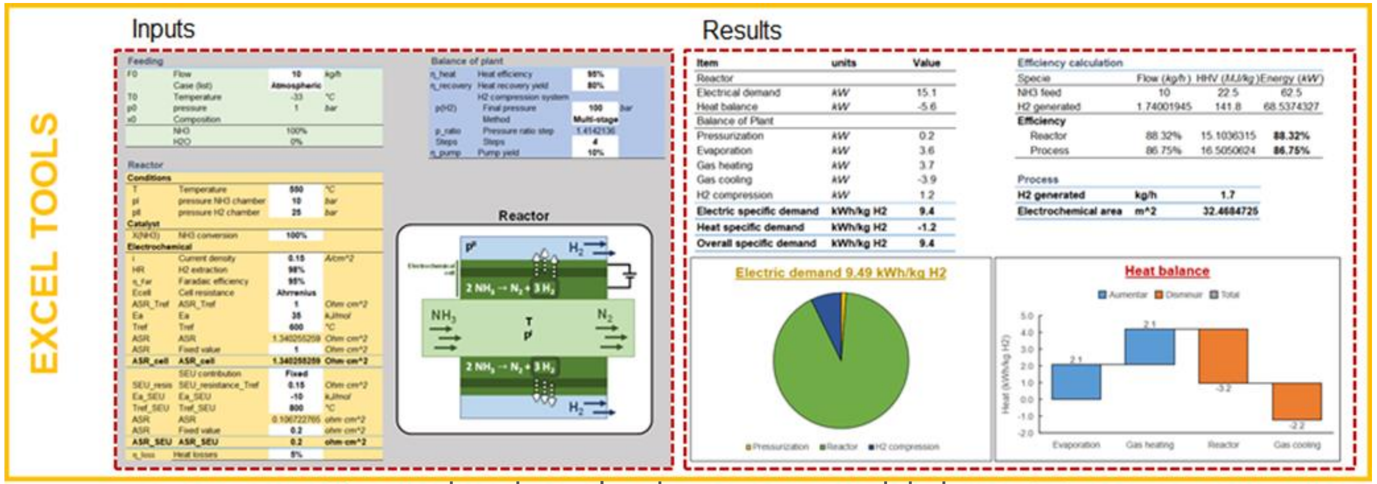
$$2\,NH_3 \rightarrow N_2 + 3\,H_2$$

2. **Ethane dehydrogenation**

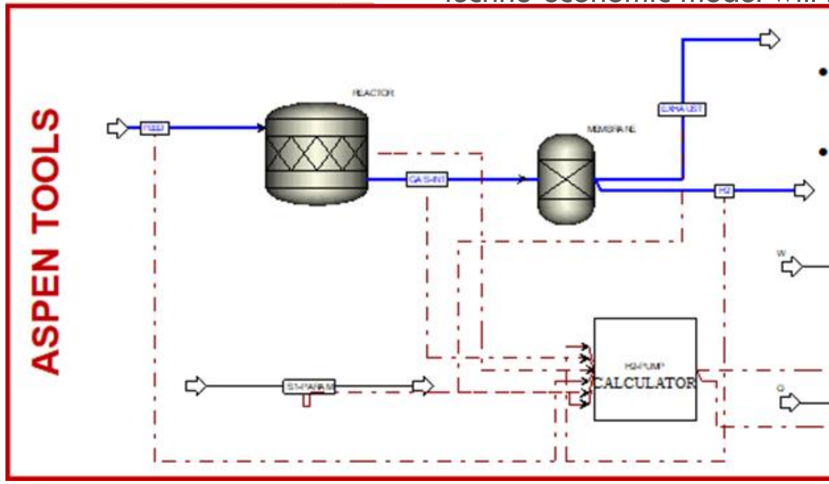
$$C_2H_6 \rightleftarrows C_2H_4 + H_2$$

3. **Reversible electrolysis**

$$2\,H_2O \rightarrow O_2 + 2\,H_2$$




Adapt the tool to the program in whih the techno-economic model will be made




- Adapting the different parameters to the program
- Electrolysis calculated using a built block



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# Engineering model

## EXCEL TOOL

### Diagram of the process

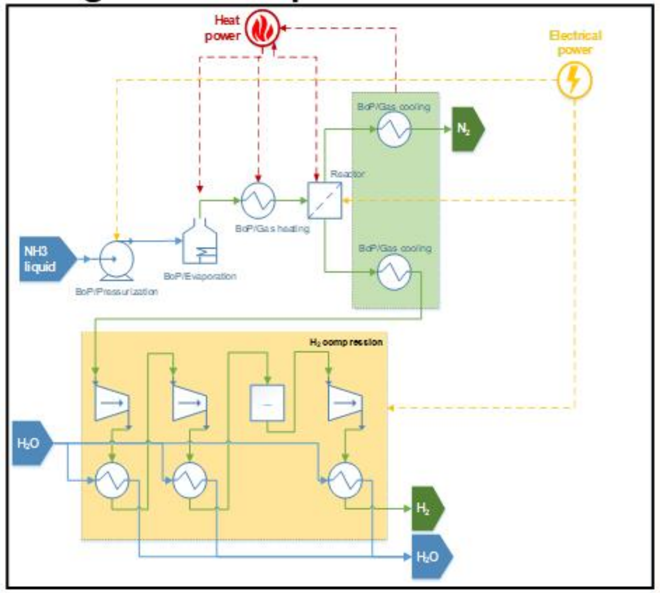
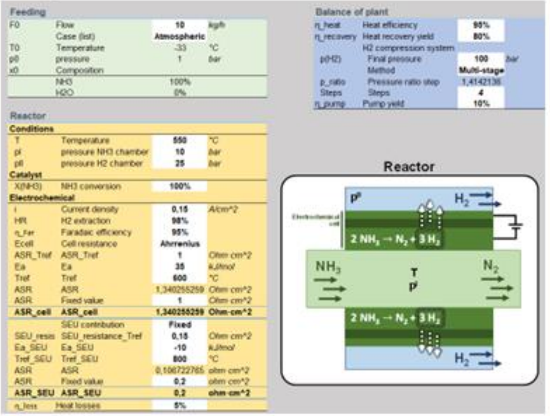



**Diagram validated for the partners**

### Input sheet




Introduce the inputs of each section:

- Feed stream
- Reactor conditions
- Electrochemical performance
- BoP properties

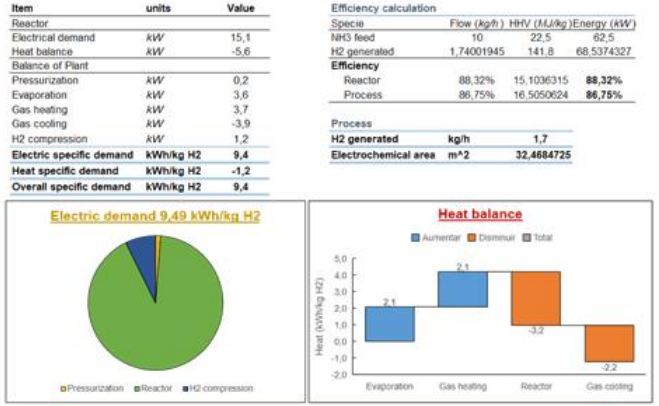
### Calculation sheet

| Electrochemistry | unit | Value |
|---|---|---|
| pH2-NH3 chamber | bar | 2,6886792 |
| pH2-H2 chamber | bar | 25 |
| Reversible potential | V | 0,0790806 |
| Current density | A·cm^2 | 0,15 |
| Cell resistance | Ohm·cm^2 | 1,3402553 |
| SEU resistance | Ohm·cm^2 | 0,2 |
| Overall resistance | Ohm·cm^2 | 1,5402553 |
| Cell potential | V | 0,3101189 |
| Ionic current | A | 46267,573 |
| Faradaic efficiency | % | 0,95 |
| Total current applied | A | 48702,709 |
| Electric power applied | kW | 15,103631 |
| **Heat released by electrochemistry** | **kW** | **15,103631** |
| **Electrochemical area** | **m^2** | **32,468473** |

| Heat balance | | |
|---|---|---|
| Reaction enthalpy | kJ/mol | 53,813071 |
| Reaction rate | mol/s | 0,1631059 |
| Reaction heat | kW | 8,7772277 |
| Electrochemical heat | kW | -15,10363 |
| Heat losses | % | 0,05 |
| **Overal heat balance** | **kW** | **-5,571222** |

**Excel tool shared with partners to be validated since March 2022**

### Main results sheet




WP6 develops tecno-economic studies using Aspen

**Excel tools** → Work adapted and shared in November 2022 → **Aspen tools**



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# eCOCO2 Spire project

**AIM:** Set-up a technology for direct synthesis of carbon-neutral jet fuels from $CO_2$ using renewable energy and electrochemical catalytic membrane reactors. Bench-testing targets a 500 W multi-tubular system.

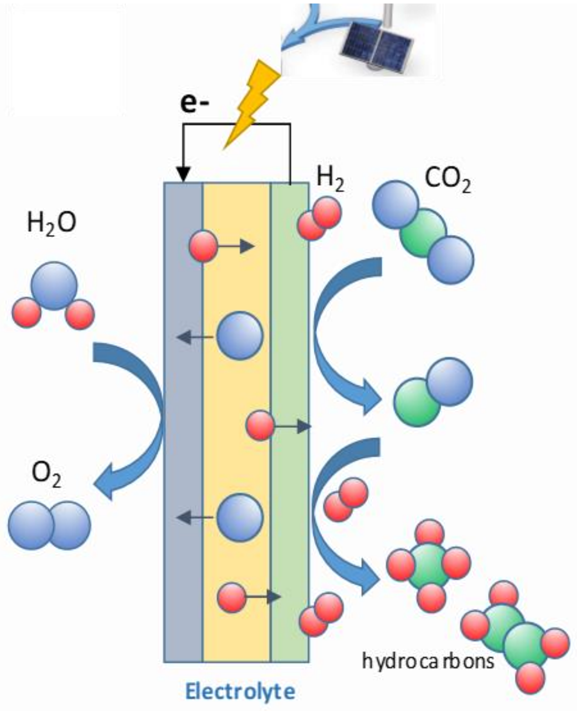
- Single-step electrolysis and one-pot catalytic conversion.
- Operating conditions:
  T = 350-450 °C and > 25 bar.

Product:
Jet fuel

Efficiency:
> 85%

Full integration:
compact sized reactor

Final TRL:
5




Co-Electrolysis Reactor

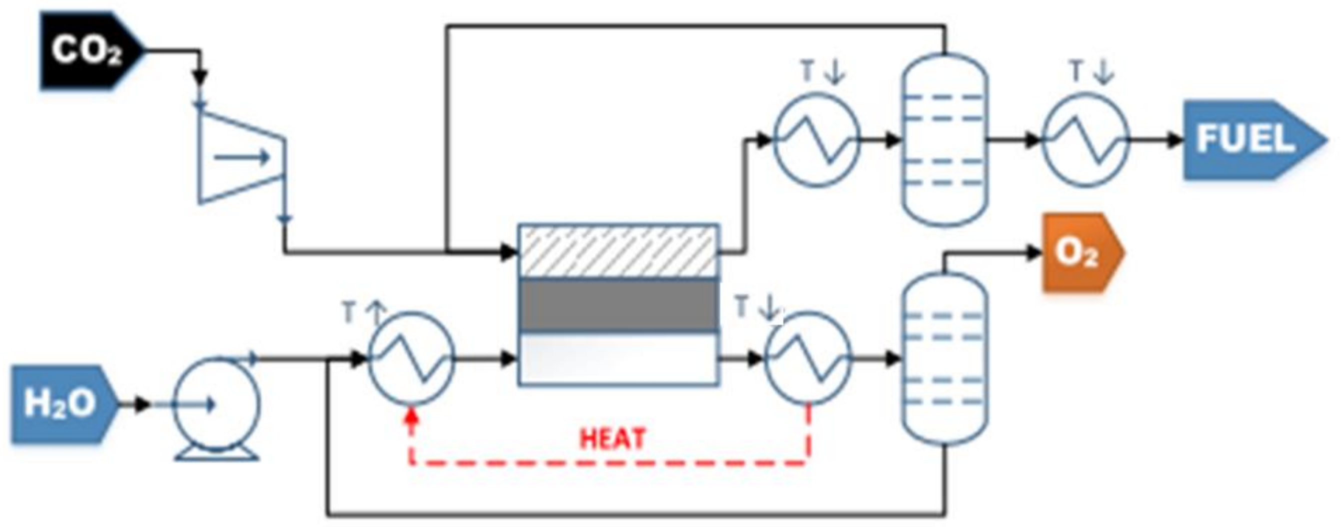
**PARTNERS**

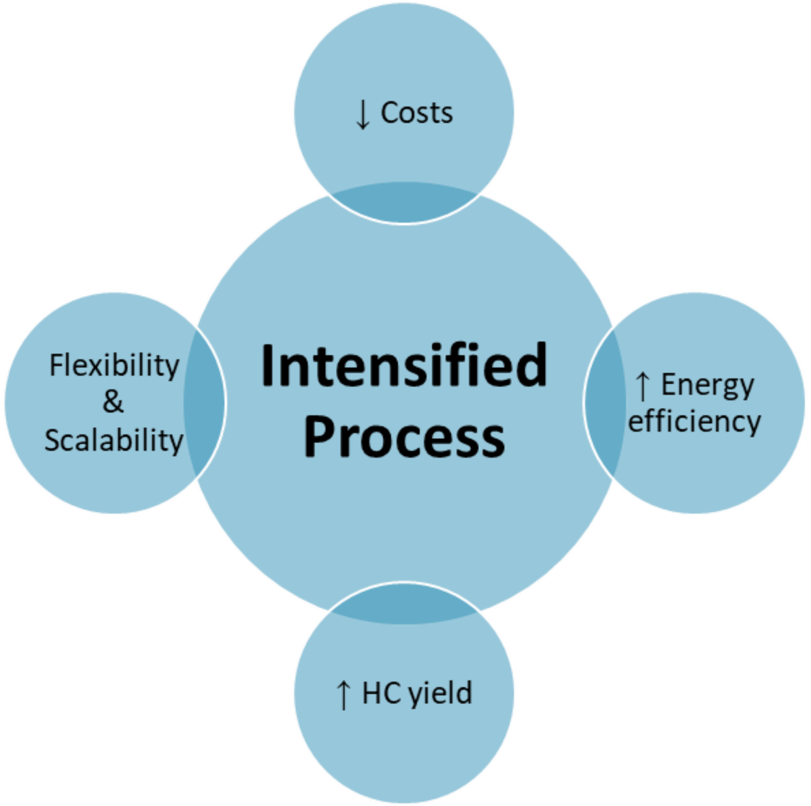
CSIC, CoorsTek Membrane Sciences, Instituto de Tecnología Química (ITQ), RWTH Aachen University, Universitas Osloensis, Universitat Politècnica de València, HERA, Shell, ArcelorMittal, SINTEF, CEMEX, 九州大学 Kyushu University, Universitas Amoiensis

**H2020-LC-SC3-2018-NZE-CC** | Duration: May 2019 – May 2023 | **EC funding**: 3.9 M€

This project has received European Union's Horizon 2020 research and innovation funding under grant agreement № 838077.

# eCOCO2 concept




**H2020-LC-SC3-2018-NZE-CC** | Duration: May 2019 – May 2023 | **EC funding**: 3.9 M€

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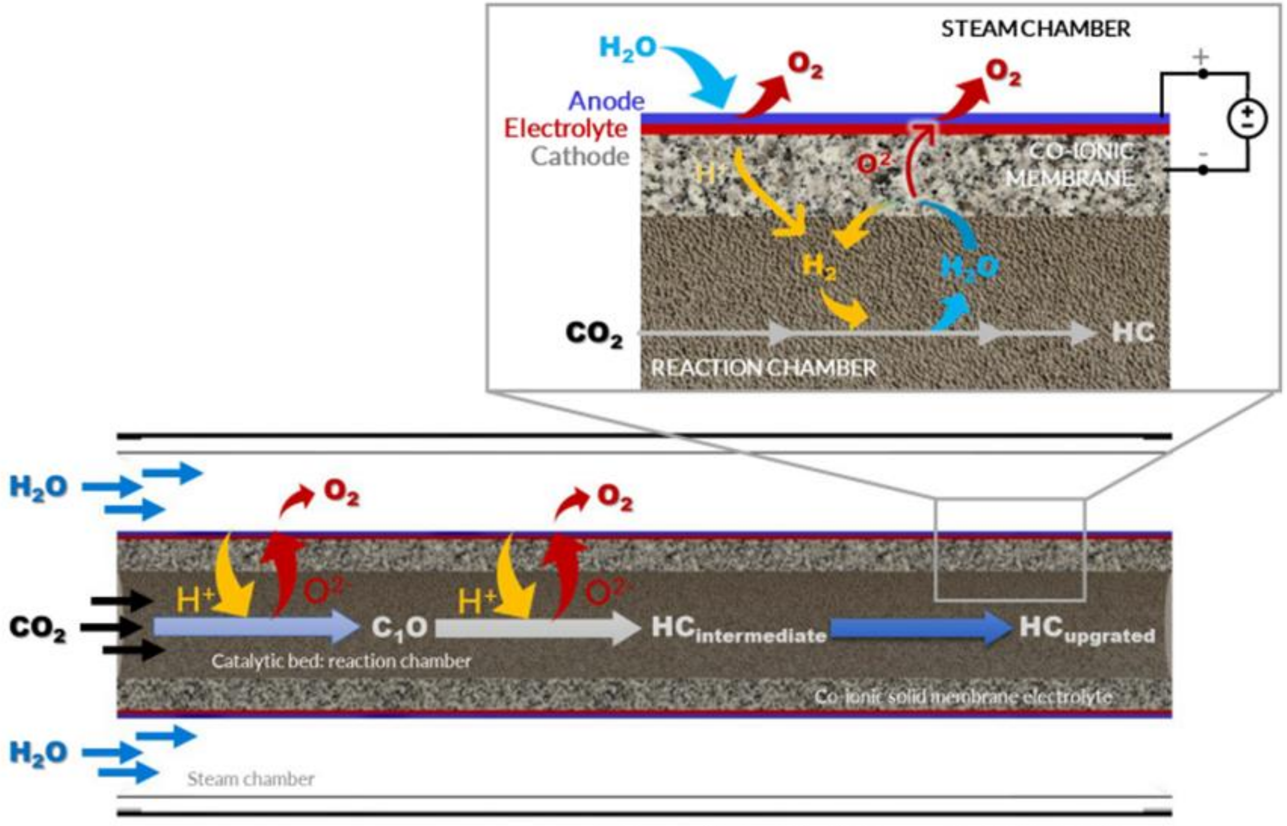
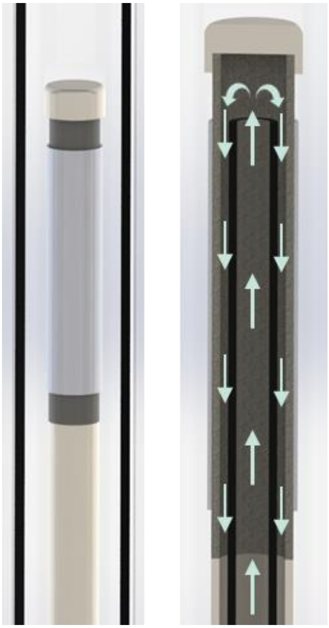
# eCOCO2 concept




(intermediate: C2-C10; upgrated: C8-C16)



- Membrane with adequate $H^+/O^{2-}$ conductivity
- Chemically stable under reaction condition: 350-450 ºC, 20-30 bar, high steam content
- Multifunctional catalyst based on iron oxides & zeolites

**H2020-LC-SC3-2018-NZE-CC** | Duration: May 2019 – May 2023 | **EC funding**: 3.9 M€

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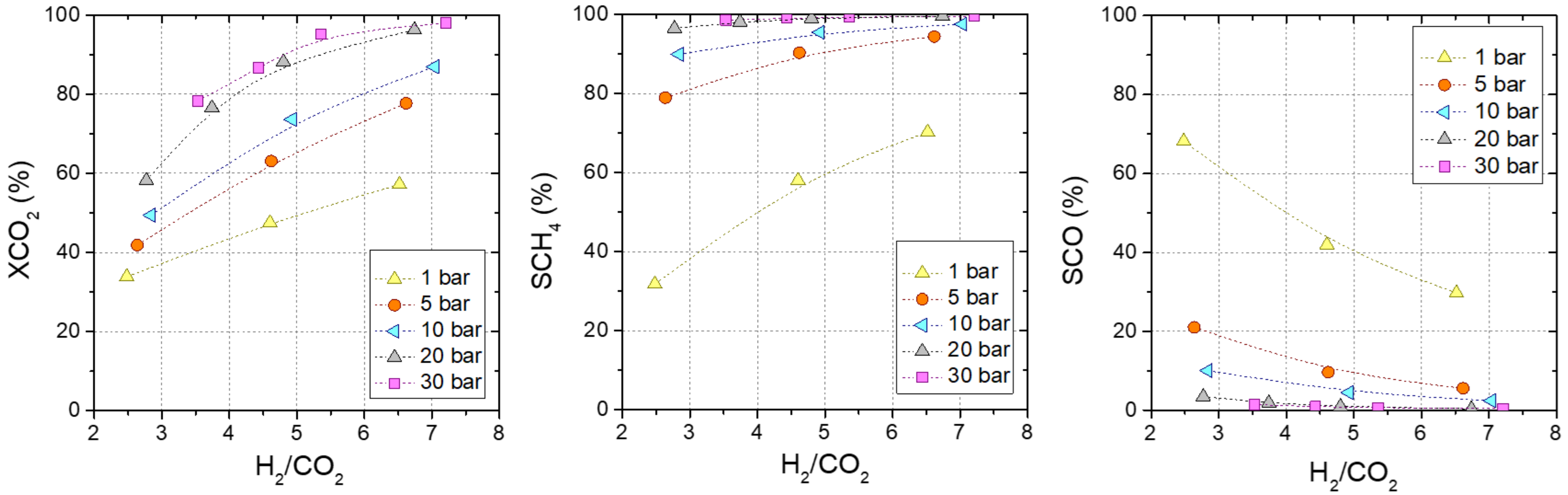
# Methanation

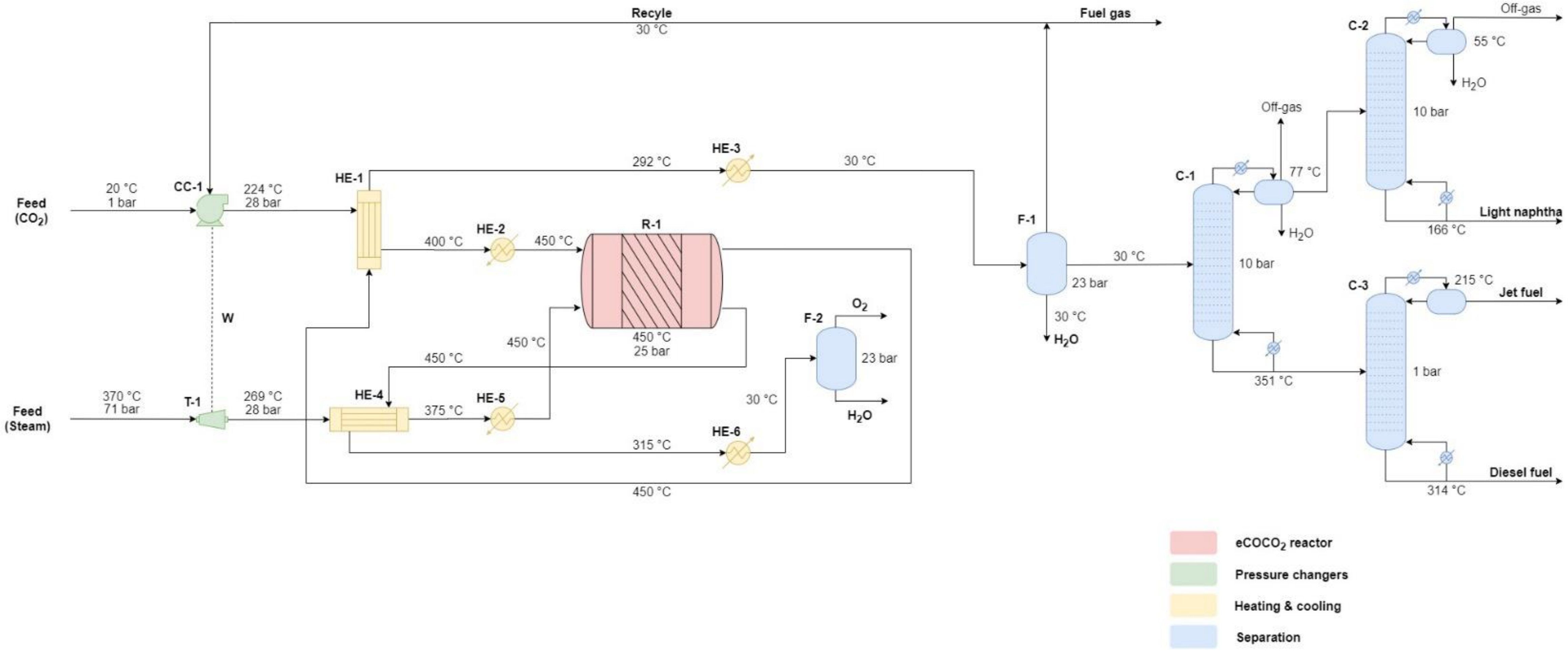



Performance improves with pressure ↑$CH_4$ selectivity & ↓CO selectivity

**H2020-LC-SC3-2018-NZE-CC** | Duration: May 2019 – May 2023 | **EC funding**: 3.9 M€

This project has received European Union's Horizon 2020 research and innovation funding under grant agreement Nº 838077.

# eCOCO2 process integration




**H2020-LC-SC3-2018-NZE-CC** | Duration: May 2019 – May 2023 | **EC funding**: 3.9 M€

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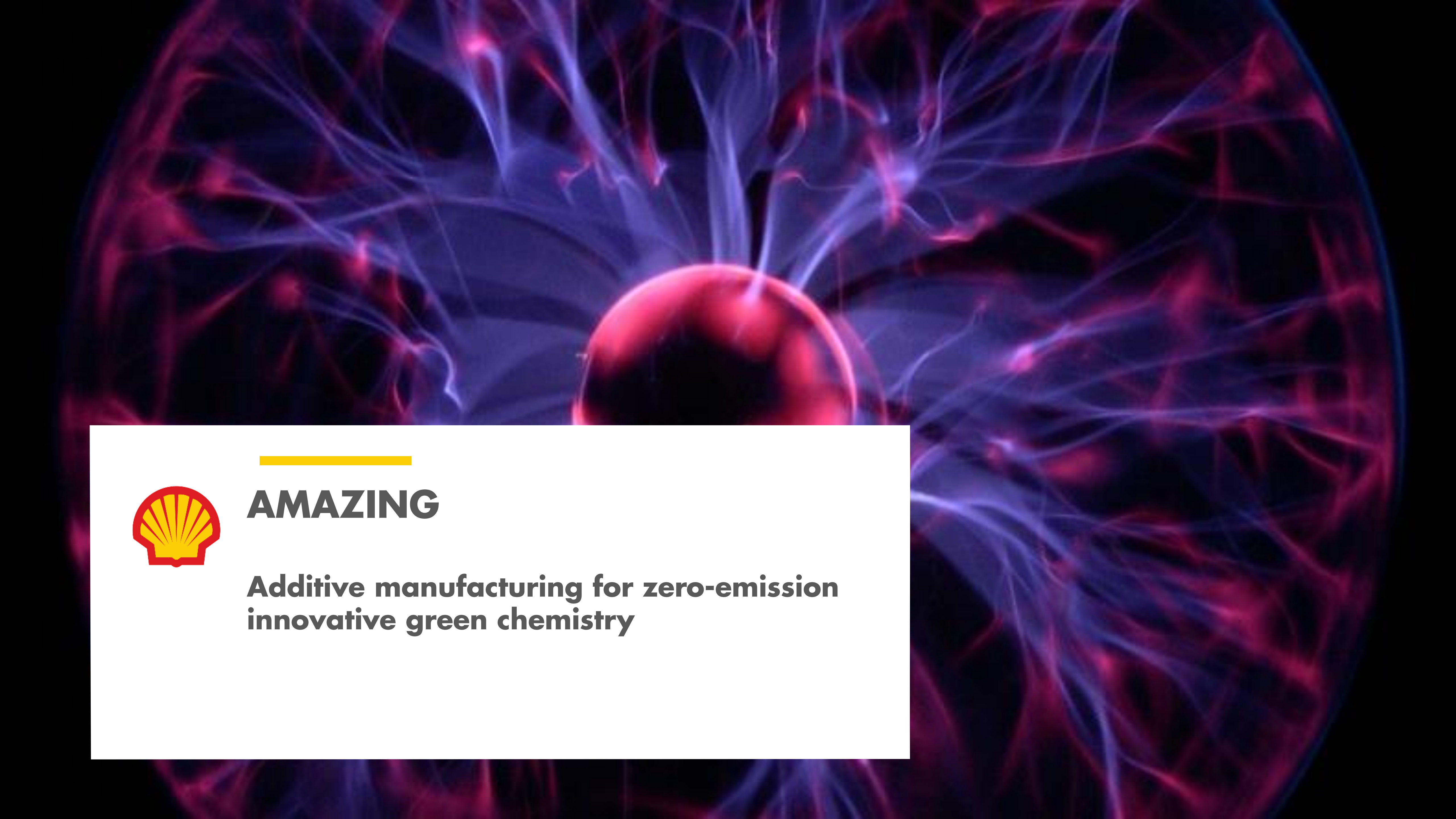
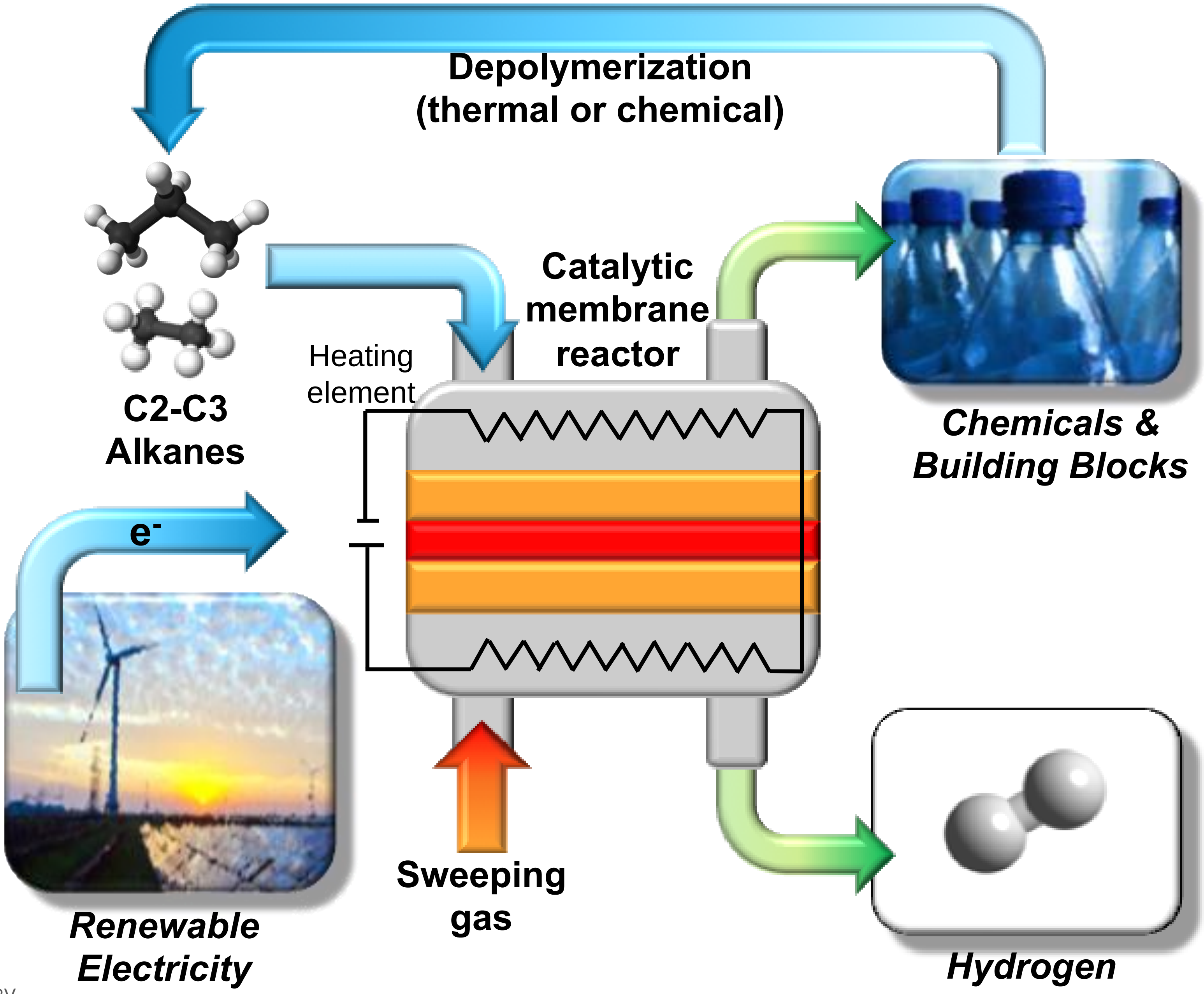


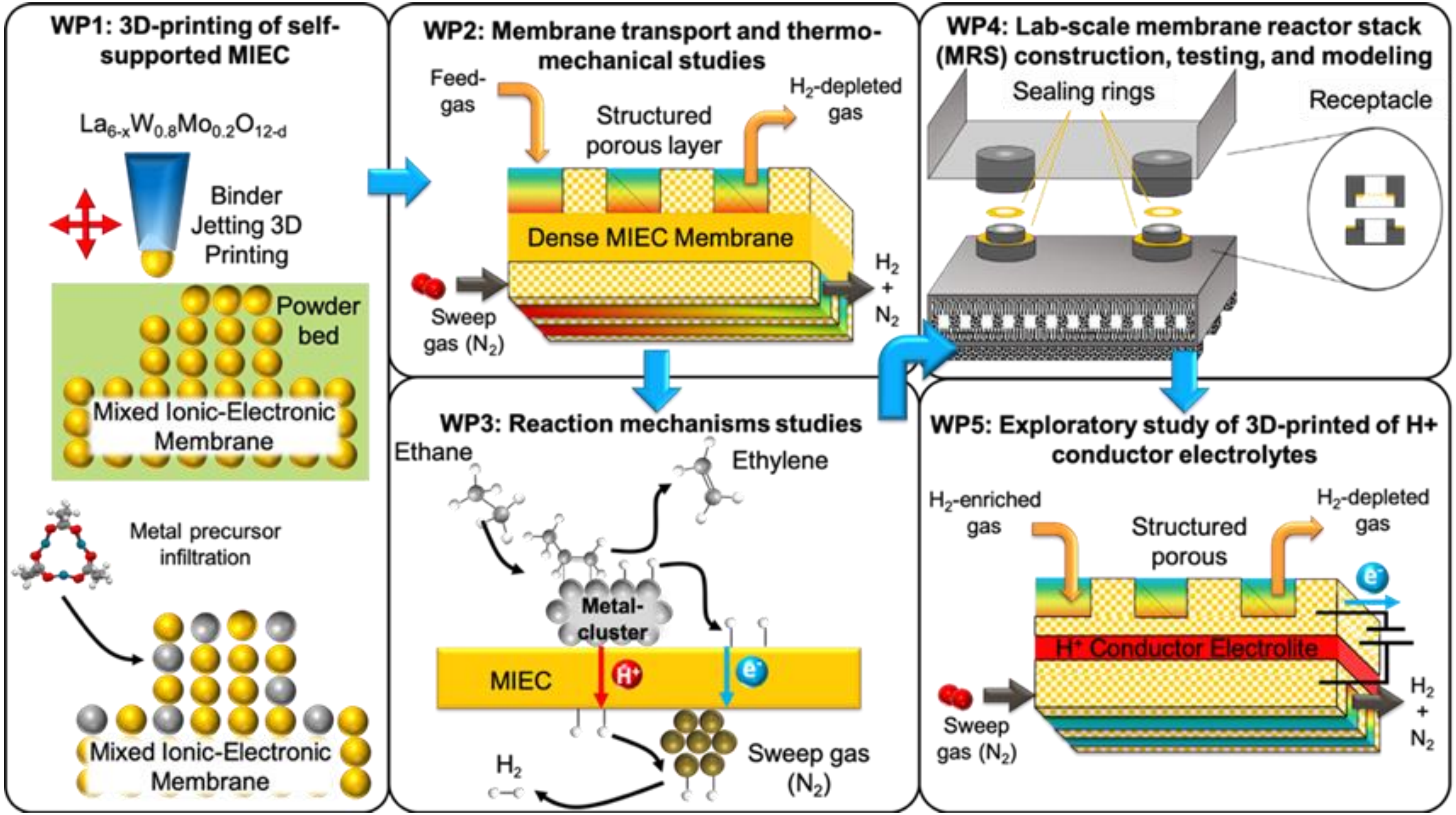
# AMAZING

## Additive manufacturing for zero-emission innovative green chemistry

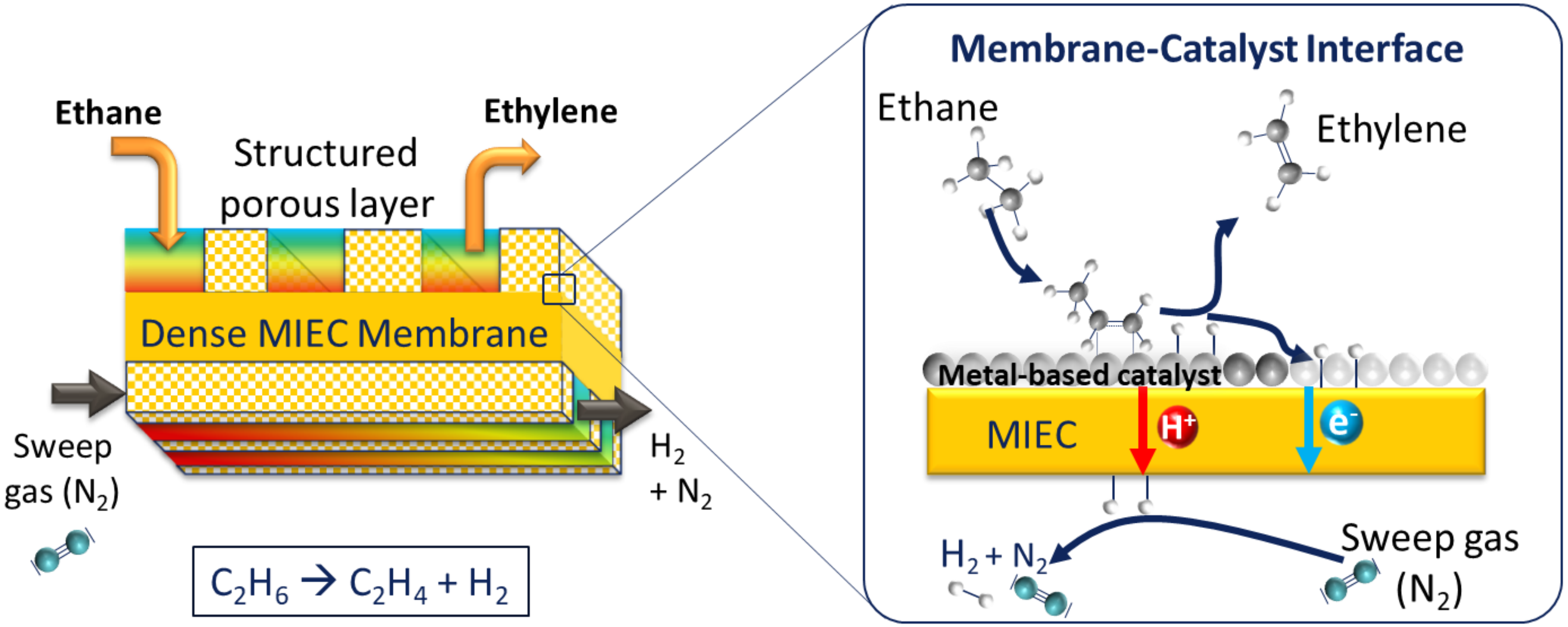
# Concept


Depolymerization
(thermal or chemical)
C2-C3
Alkanes
Heating
element
Catalytic
membrane
reactor
Chemicals &
Building Blocks
e-
Renewable
Electricity
Sweeping
gas
Hydrogen

## *Project structure*



# The MIEC membrane



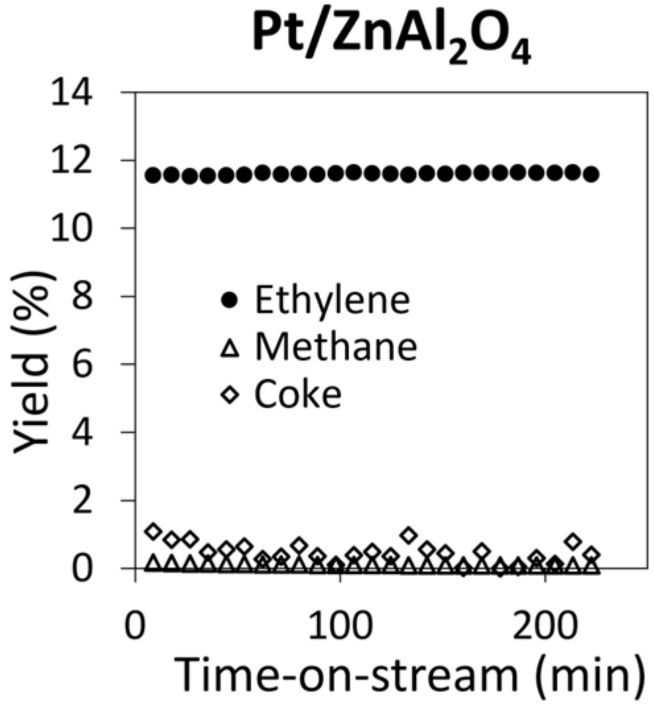

Promising MIEC membrane material:
**Lanthanum tungstate**

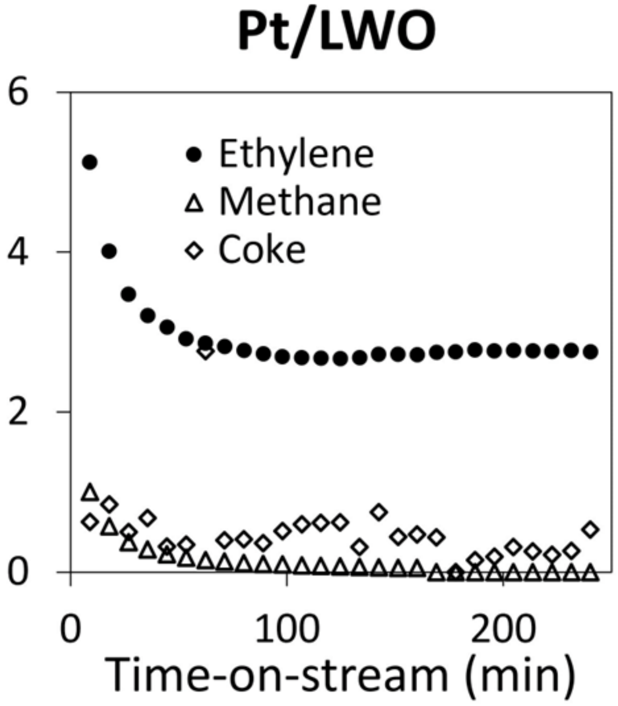
*Pros*:
- Highly stable under reducing conditions
- Easy to prepare

*Con*:
- Low electronic conductivity

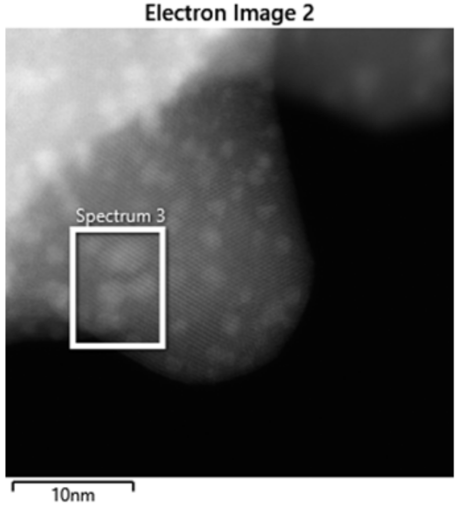
The low electronic conductivity is a drawback for the application as a hydrogen-permeation membrane. Doping by e. g. Mo enhances the electronic conductivity under reducing conditions.

# Pt functionalization of proton conducting materials



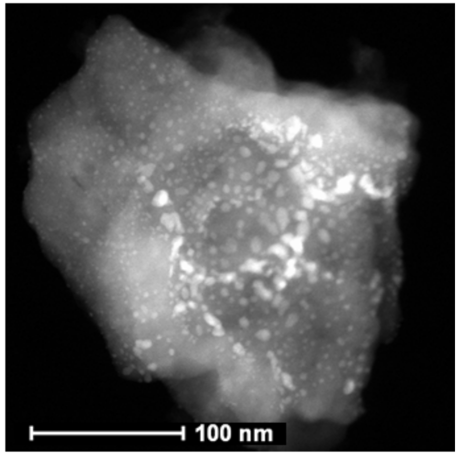




**Fresh Pt/LWO**




$d_p$ = 1.4 ± 0.3 nm

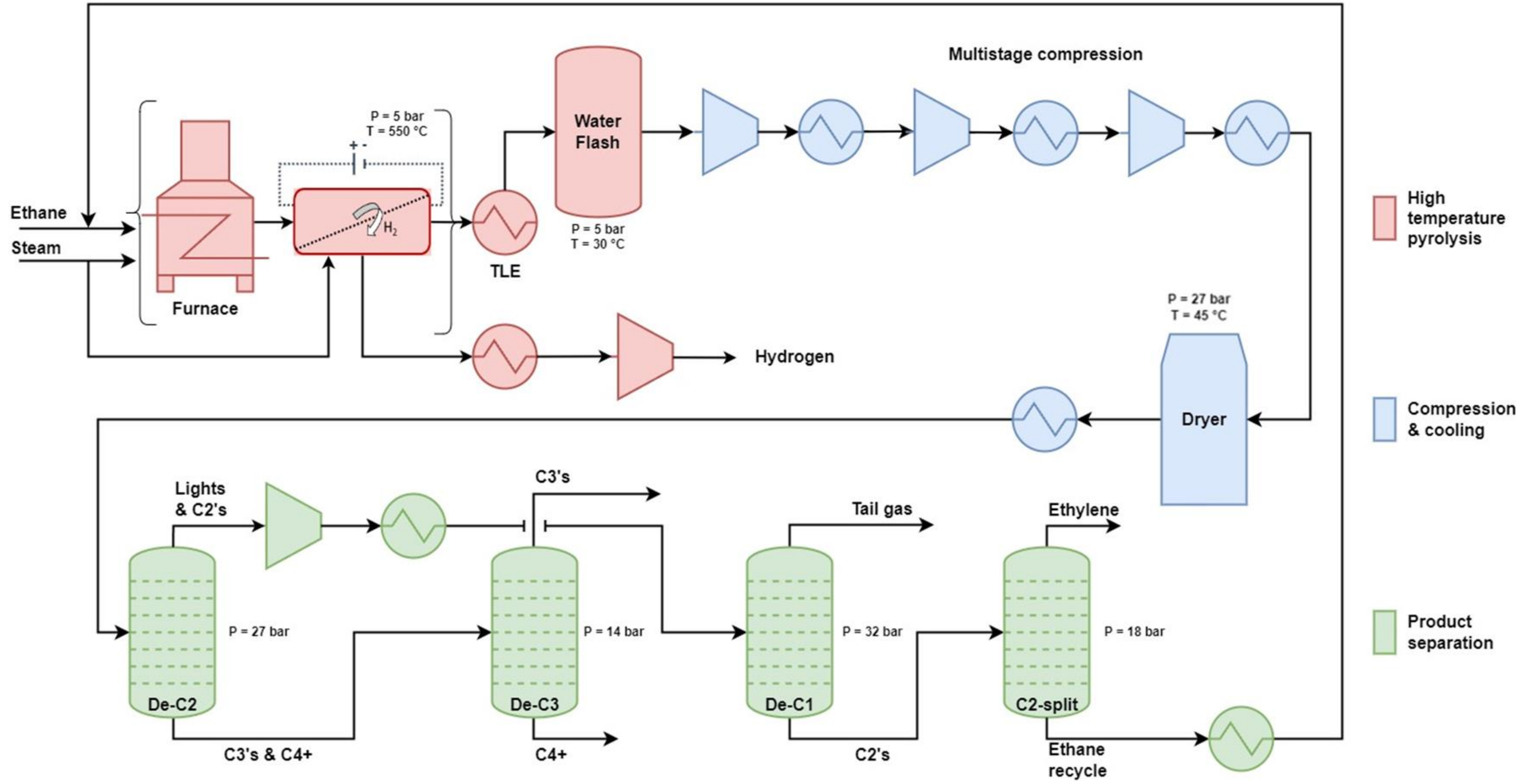
**Used Pt/LWO**




$d_p$ = 4.8 ± 4.0 nm

**Pt sintering**

# Process scheme for ethane dehydrogenation



# Main take aways

1. Integrated approach shows beneficial for both industries and academia
2. Agreed definitions and scale helps to show credible outcomes
3. Various disciplines are needed for an end to end solution.
4. The electrochemical reactor is only a (minor) part of the full process
5. Don’t over optimize on materials, keep an eye on the overall process
6. Stay positive and think in solutions, not in problems!

# Questions and Answers

Q&A