Advancements in Electrocatalysts for Low-Temperature CO2 Electrolysis: Progress and Insights from the ECO2Fuel Project

245th ECS Meeting

26th May, 2024, San Francisco

Presenter: Faria Huq, DLR

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No [101037389]

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Agenda

1. Introduction
2. Project description
3. Gas Diffusion electrode development
4. Anion exchange membrane development
5. Experimental setups
6. Electrochemical characterization
7. Summary and Conclusion

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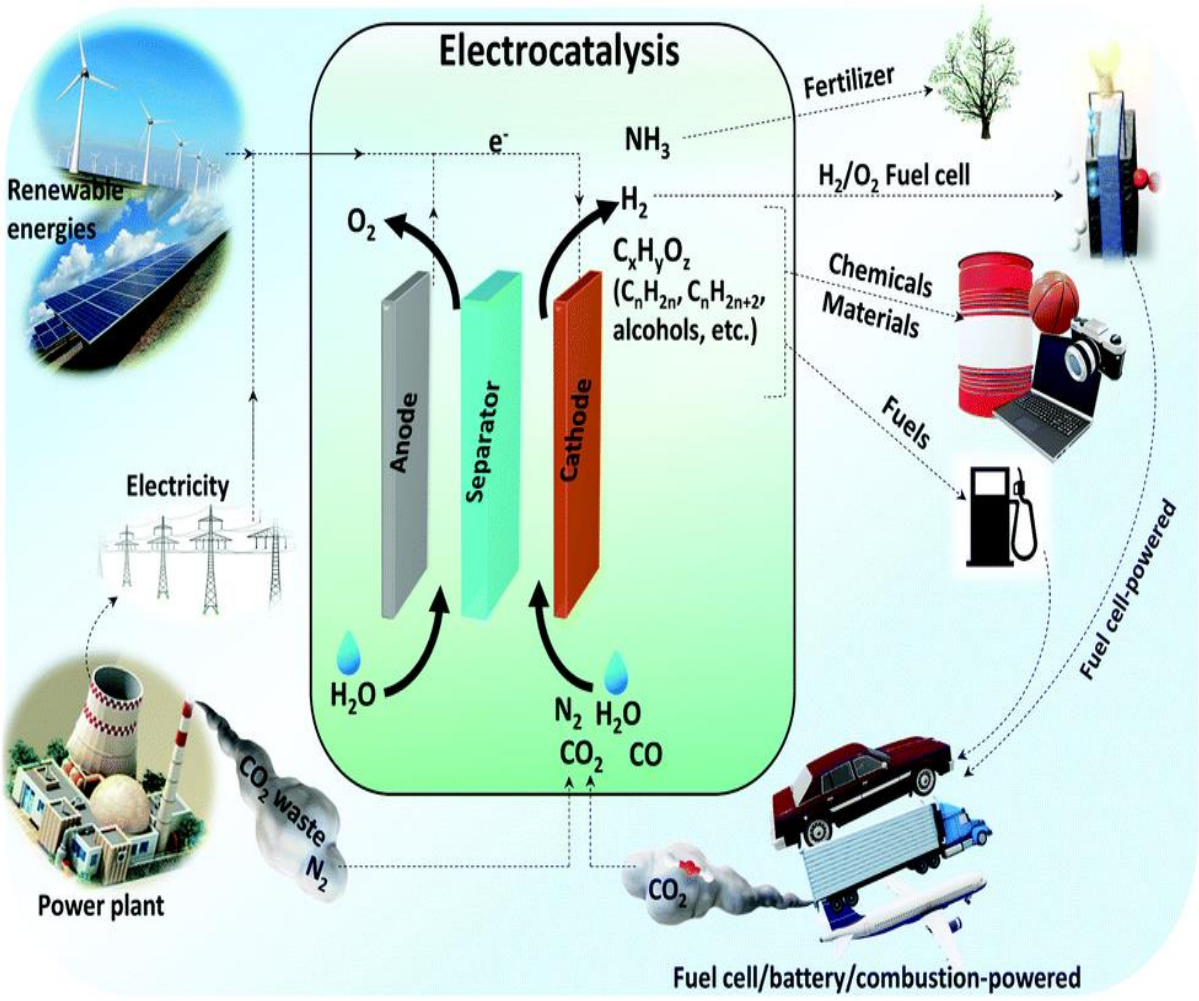
Introduction



- Renewable energy sources - Electrochemical reduction of CO_2 (CO_2RR)
  - ✓ Combines water splitting and hydrogenation
  - ✓ Produces valuable fuels and chemicals
  - ✓ CO_2 as carbon source independent of fossil fuels
- Challenges
  - ✓ CO_2 is planar thereby stable molecule
  - ✓ Multi step proton coupled electron transfer process
  - ✓ Mild operation conditions (Temperature, Pressure)

H. Rabiee, L. Ge, X. Zhang, S. Hu, M. Li, Z. Yuan, *Energy Environ. Sci.* **2021**, *14*, 1959

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Introduction

Catalyst Requirement: Activity, selectivity, and stability

Overall Reaction

$$CO_2 + H_2O + \text{Energy} \rightarrow C_xH_yO_Z + O_2$$

Water Oxidation (Anode)

$$2H_2O \rightarrow O_2 + 4(H^+ + e^-) \qquad 1.23\ V$$

CO_2 Reduction (Cathode)

$$CO_2 + m(H^+ + e^-) \rightarrow C_xH_yO_Z + nH_2O \qquad \sim 0\ V$$

Overpotential: -1V

Fuels + Chemicals

Hydrogen Evolution

$$2(H^+ + e^-) \rightarrow H_2 \qquad 0\ V$$

CO_2 reduction is a difficult reaction and requires extra voltage of -1V referred to as overpotential

HER takes place at 0V and kinetically more favored compared to hydrogenation reaction

H. Rabiee, L. Ge, X. Zhang, S. Hu, M. Li, Z. Yuan, *Energy Environ. Sci.* **2021**, *14*, 1959

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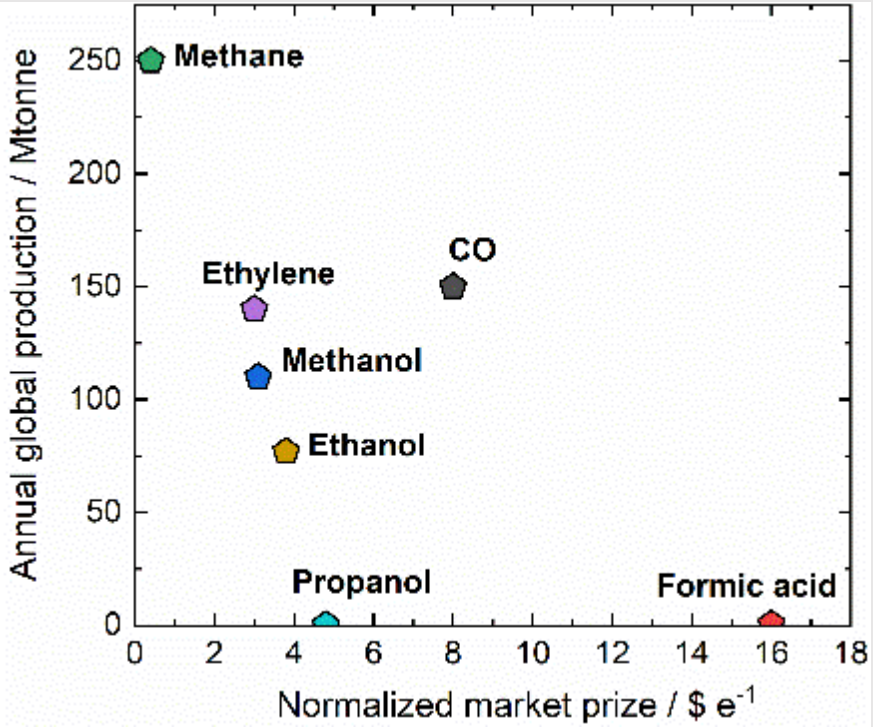
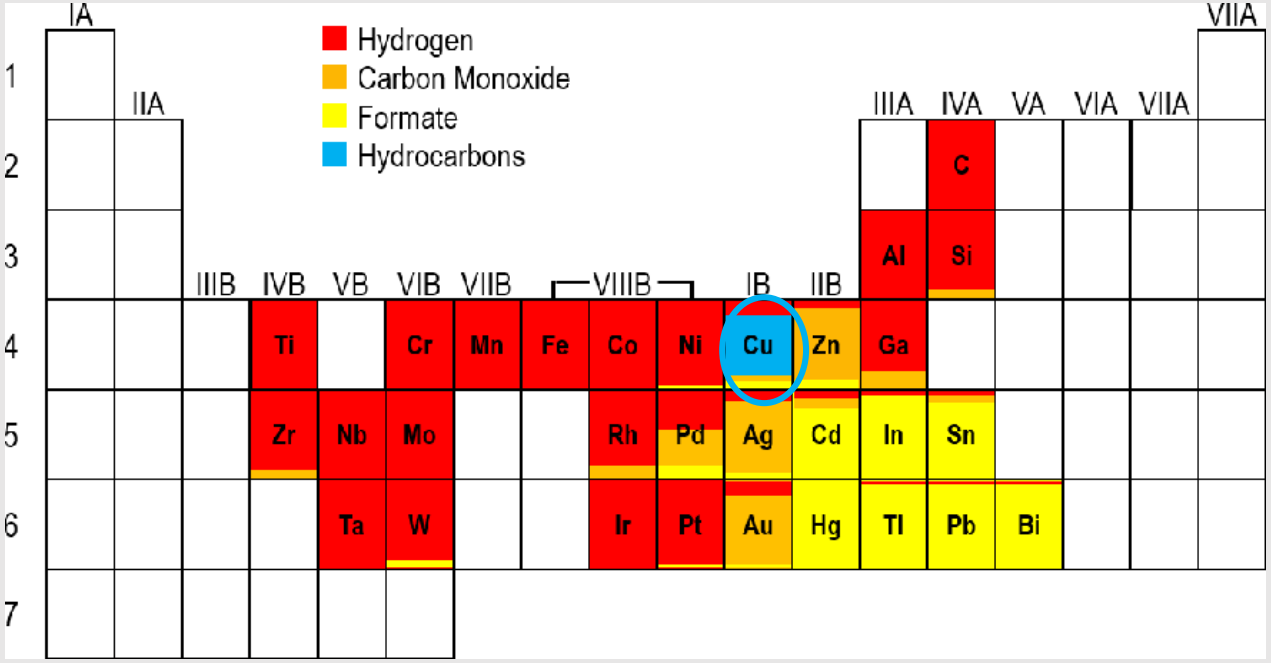
Introduction

Target products – ethanol, propanol

Cu – most selective towards C_{2+} products





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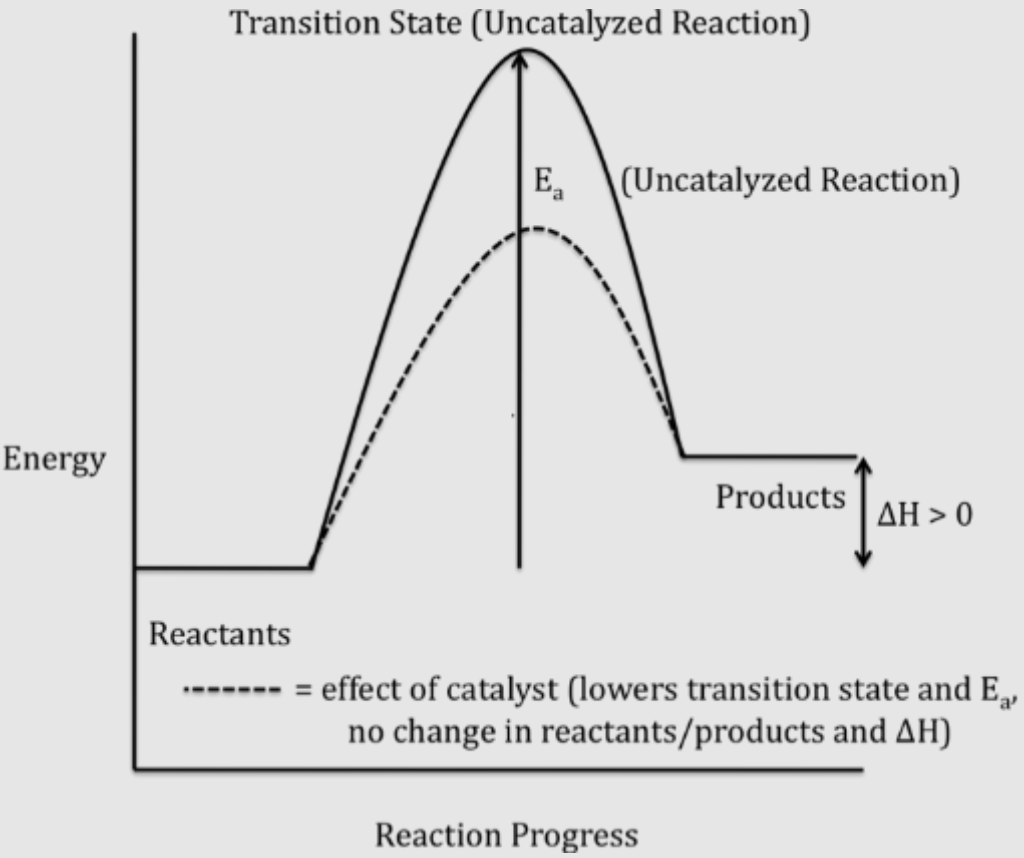
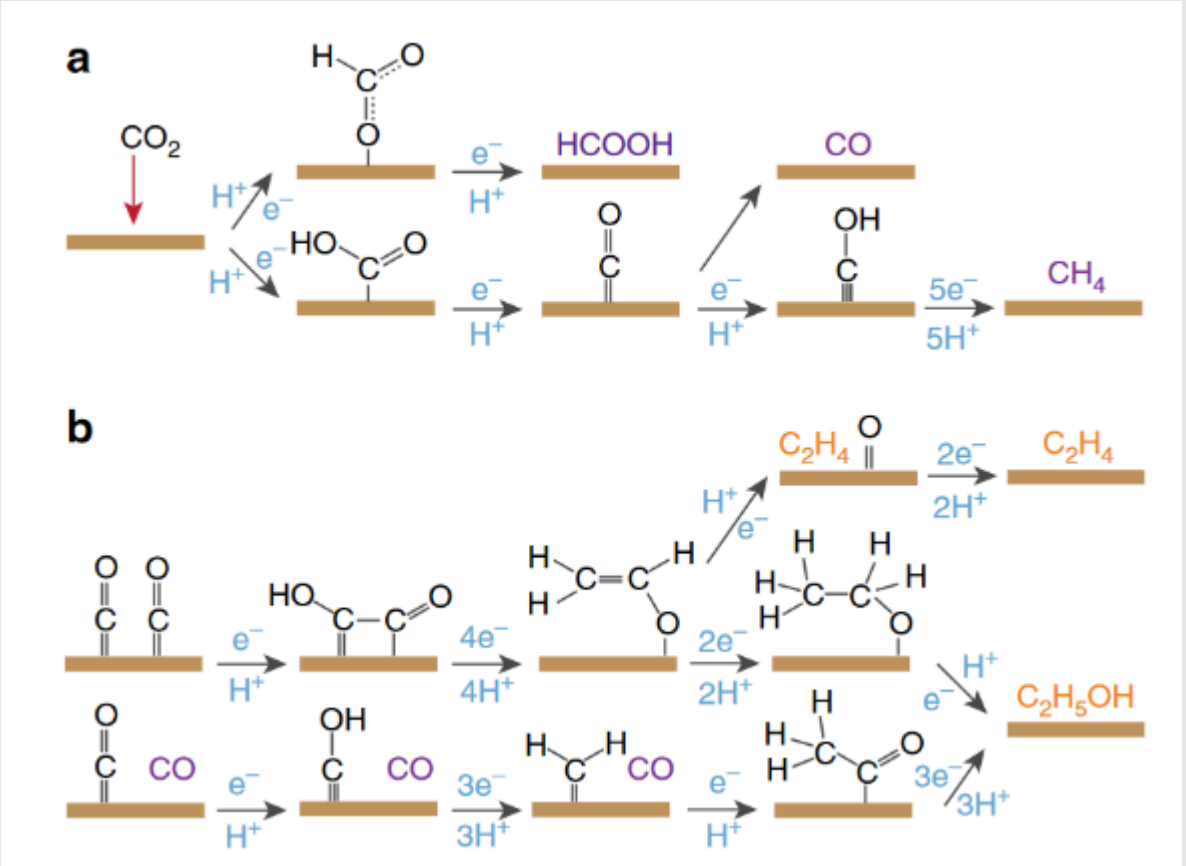
Introduction

Reaction path-

- Dimerization of CO intermediates (b) leads to C_{2+} products





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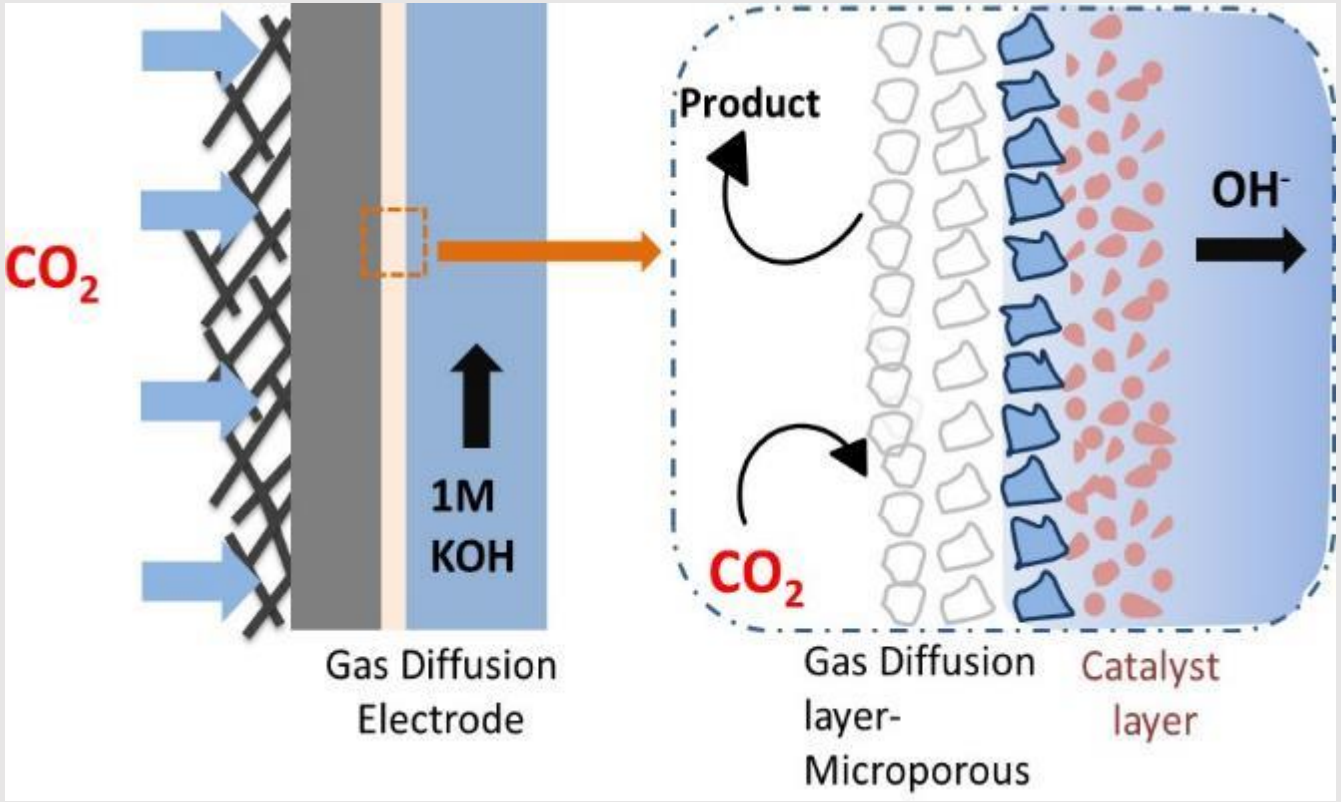
Introduction - Gas Diffusion Electrode



- Gas Diffusion electrode
  - Adhesion layer
  - Catalyst layer
  - Gas Diffusion Layer – Carbon Cloth

- Gas Diffusion Layer – Carbon Cloth
  - Microporous
  - Hydrophobic – prevents electrolyte flooding
  - Allow gaseous CO_2 access to the catalyst layer

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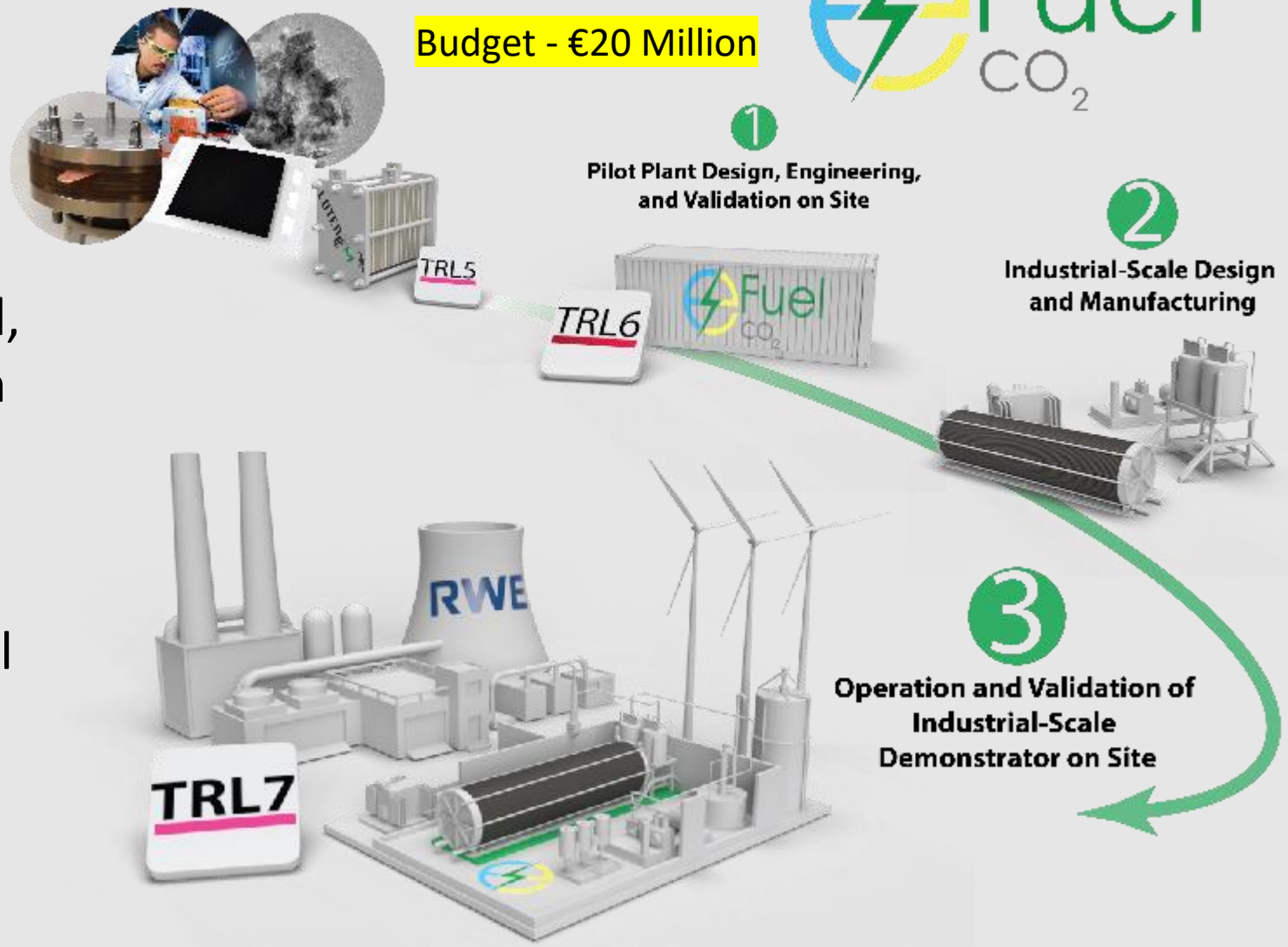
Objectives

- 1 MW demonstrator
- Sustainable catalytic systems
- Highly ion conductive durable and cost-effective membrane separators
- Use advanced strategies for material, component and system optimization and upscale
- Closing the gap between research and commercialization
- Demonstrate the coverage of the full value chain

Oct. 2021
-
Sep. 2026

Budget - €20 Million



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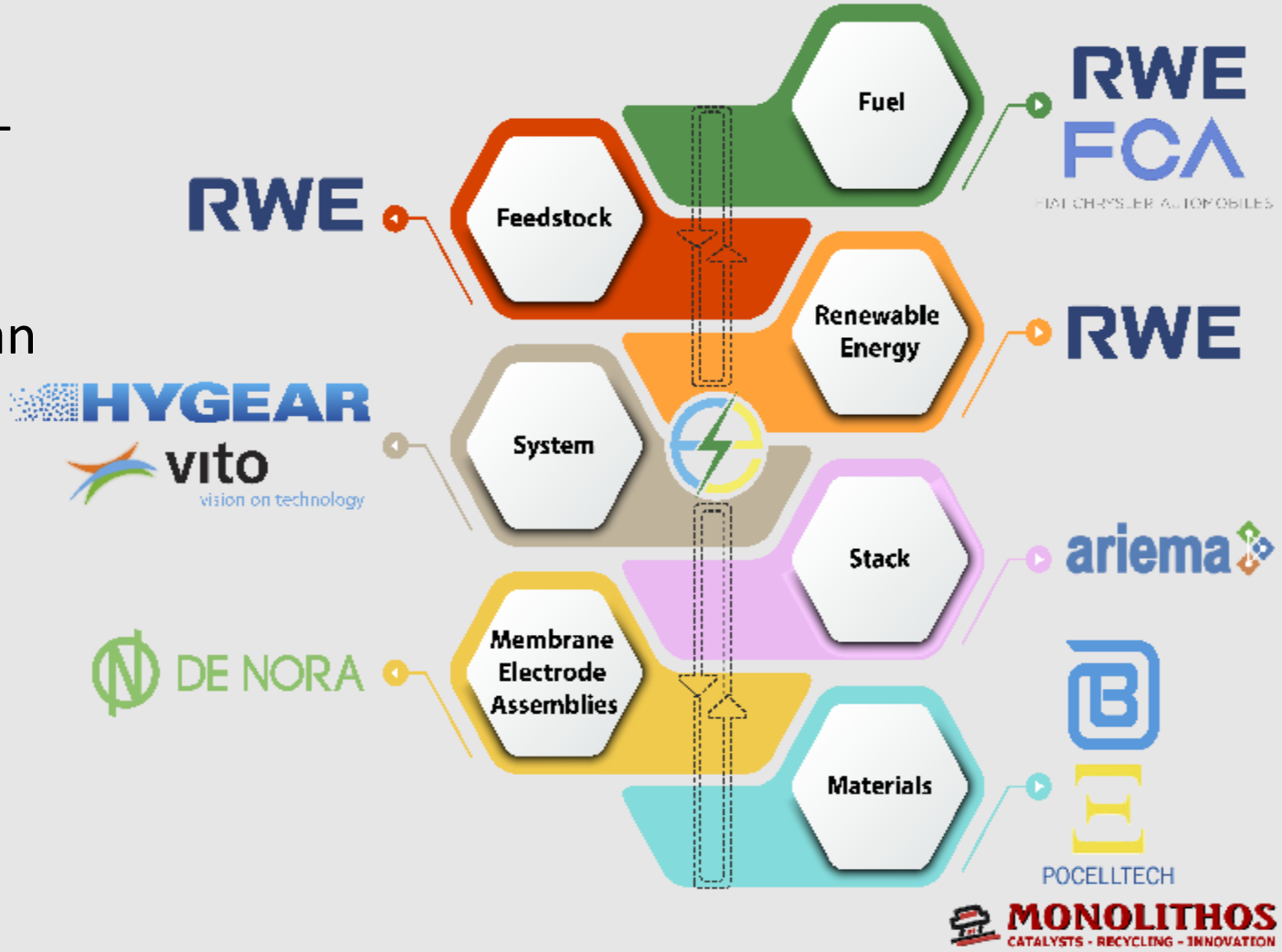
Objectives

- Validate the economic feasibility of the ECO2Fuel technology through a techno-economic analysis
- Address the regulatory aspects and public acceptance to plan for a European wide large-scale diffusion of the ECO2Fuel technology.
- Demonstrate the specific economic target of having a CAPEX of 400-600 €/kW at system level



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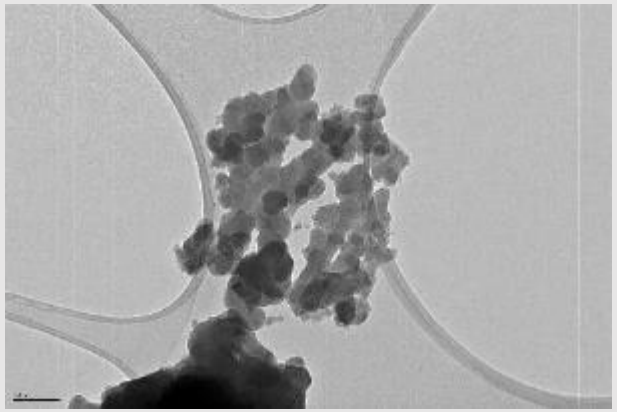
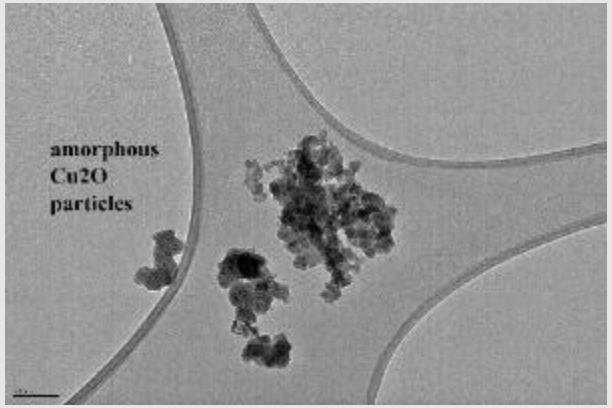
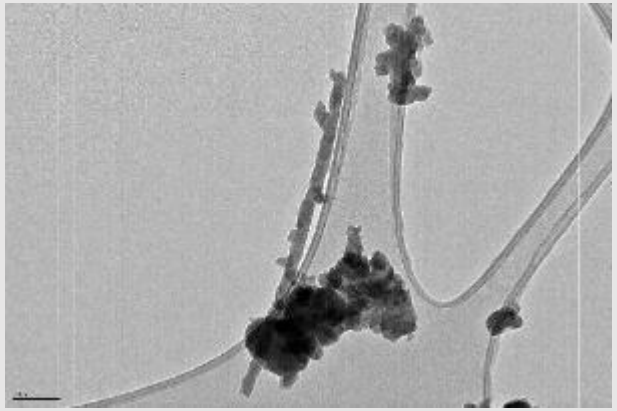
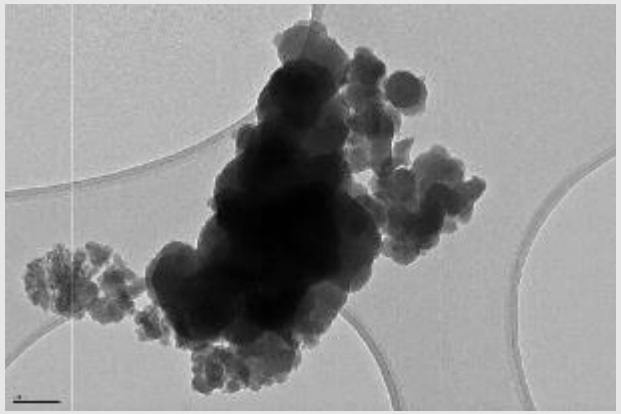
Optimization of catalyst synthesis and characterization in semi-pilot scale (2 kg level)

TEM/Cathodic electrocatalyst









✓Homogeneous particles size and shape distribution

BET surface area of Cu_2O (lab scale, MON): 26.5 m^2/g
BET surface area of Cu_2O (US Nanomaterials, Commercial): 7.44 m^2/g

- Our materials show higher BET surface area and lower pore size than the commercial one.

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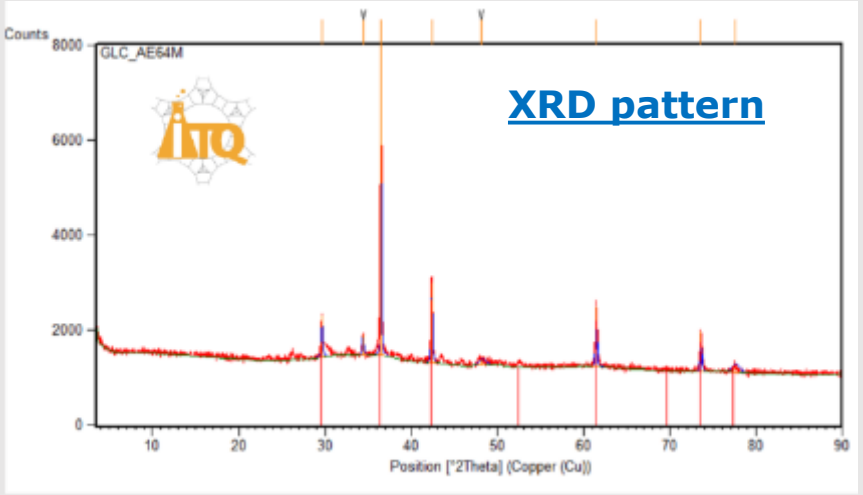
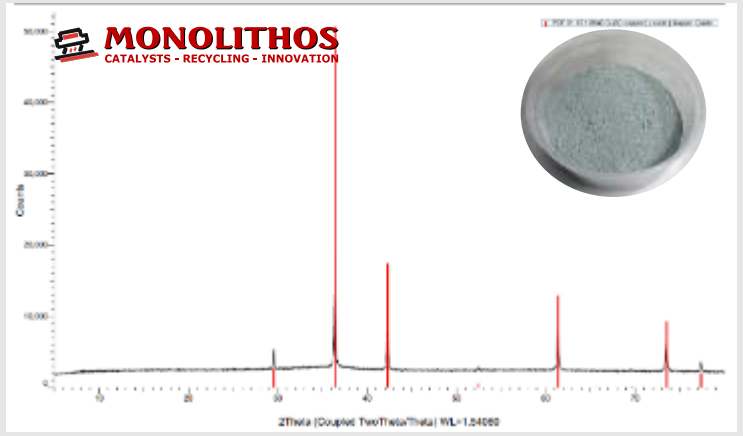
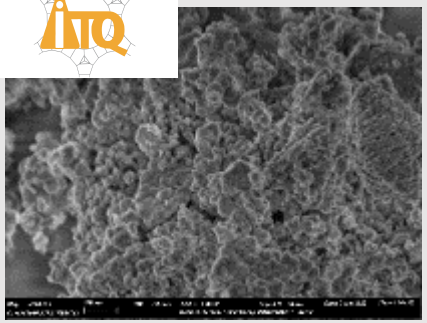
Optimization of catalyst synthesis and characterization in semi-pilot scale (2 kg level)

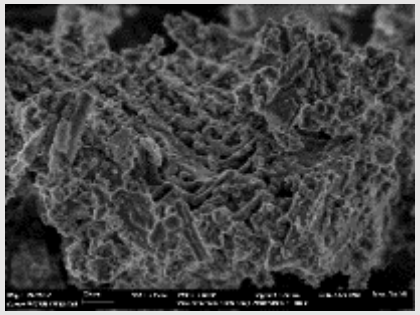
XRD pattern – SEM/EDS /Cathodic electrocatalyst

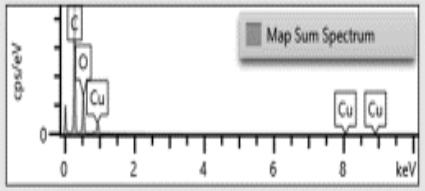


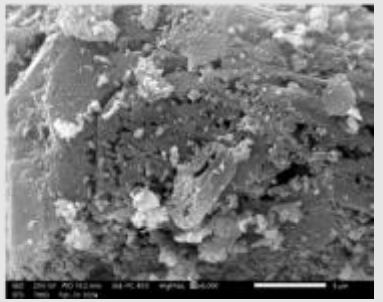


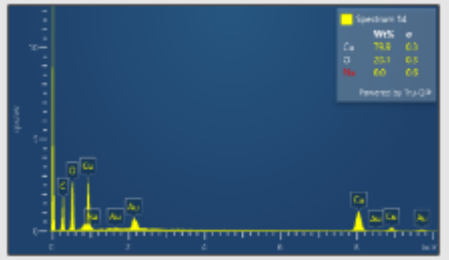
C33
Cu_2O development (lab scale, 95-100°C)











MONOLITHOS
CATALYSTS - RECYCLING - INNOVATION

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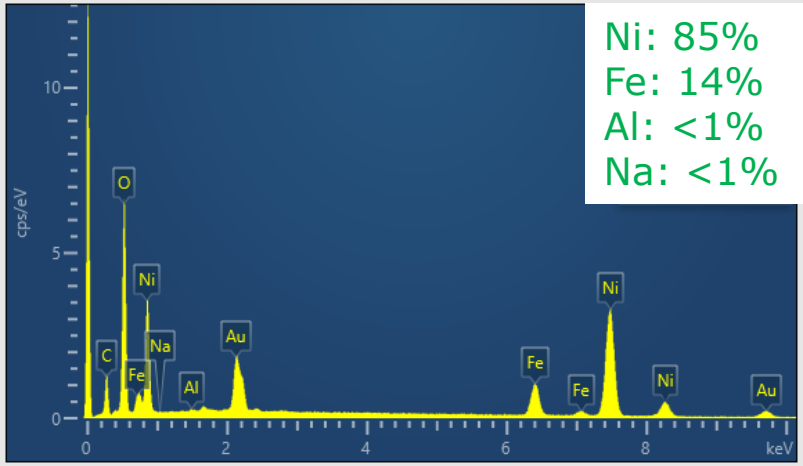
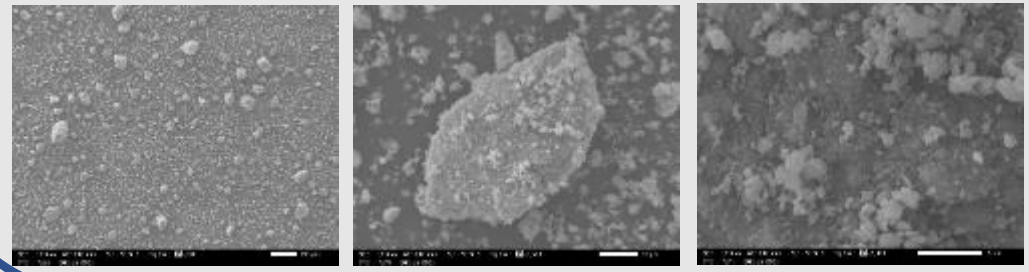
Optimization of catalyst synthesis and characterization in semi-pilot scale (2 kg level)

SEM & BET analysis / Anodic electrocatalyst

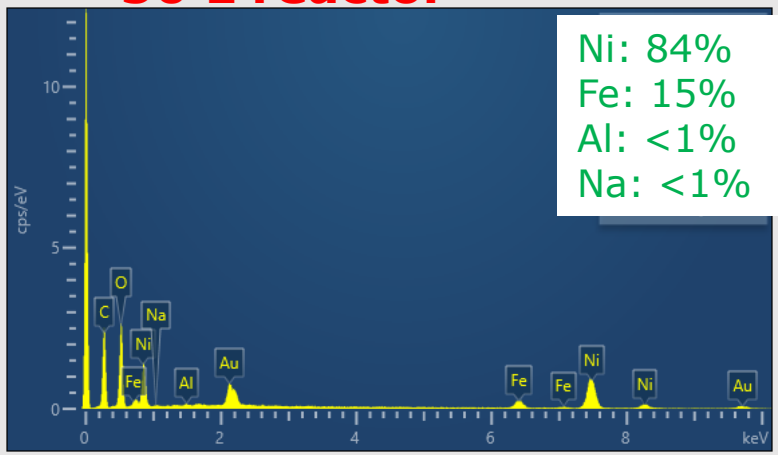
20 L reactor



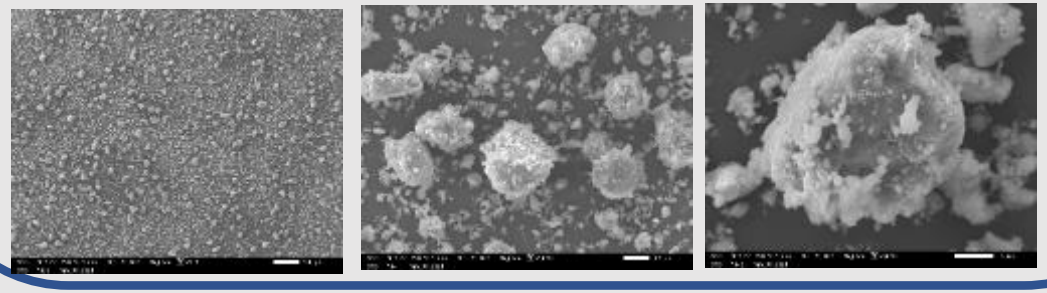
BET surface area: 173 m²/g



50 L reactor



BET surface area: 168 m²/g



Scaling up of the process: no significant effect on electrocatalyst composition/morphology

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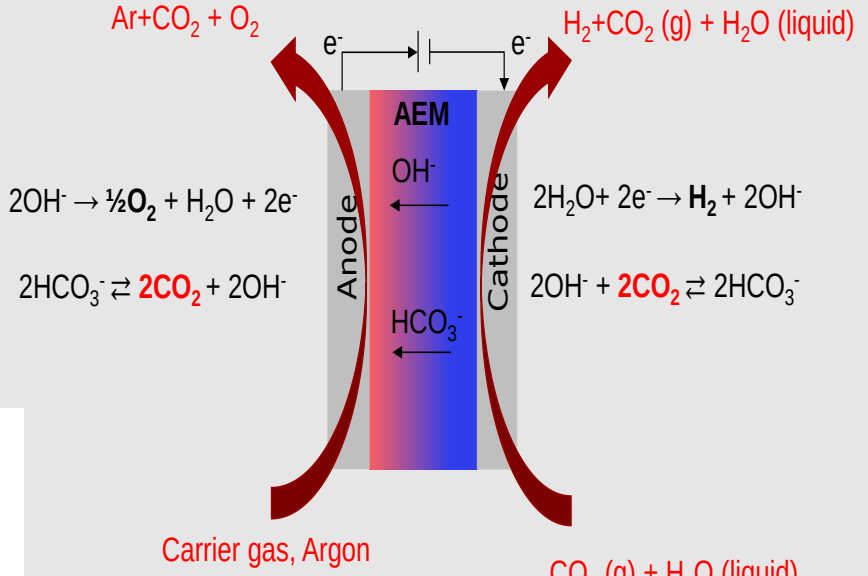
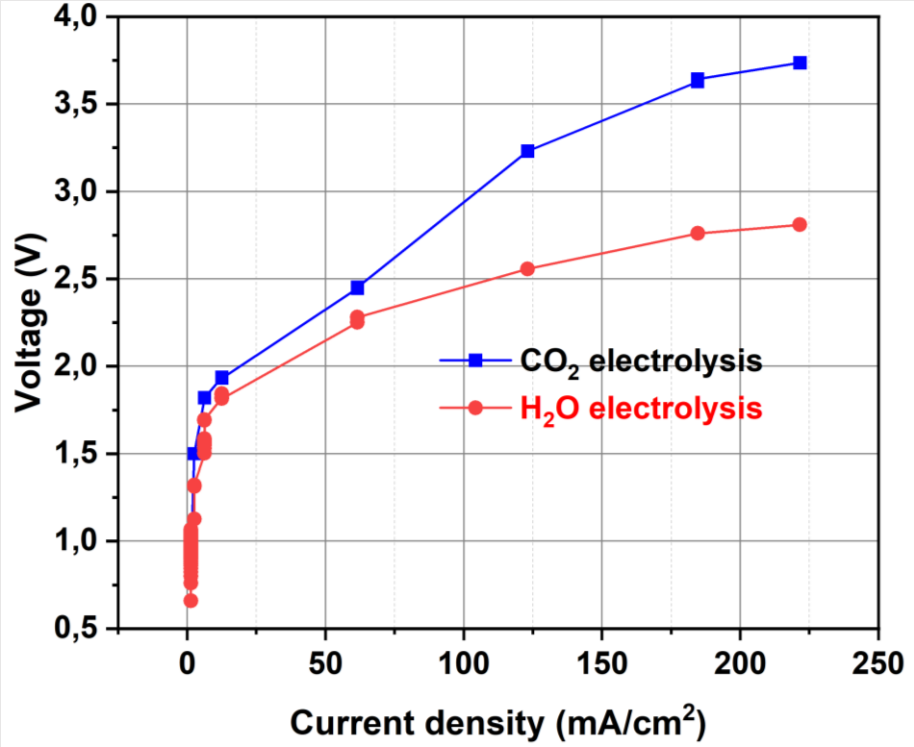
CO_2 crossover measurement using Micro GC

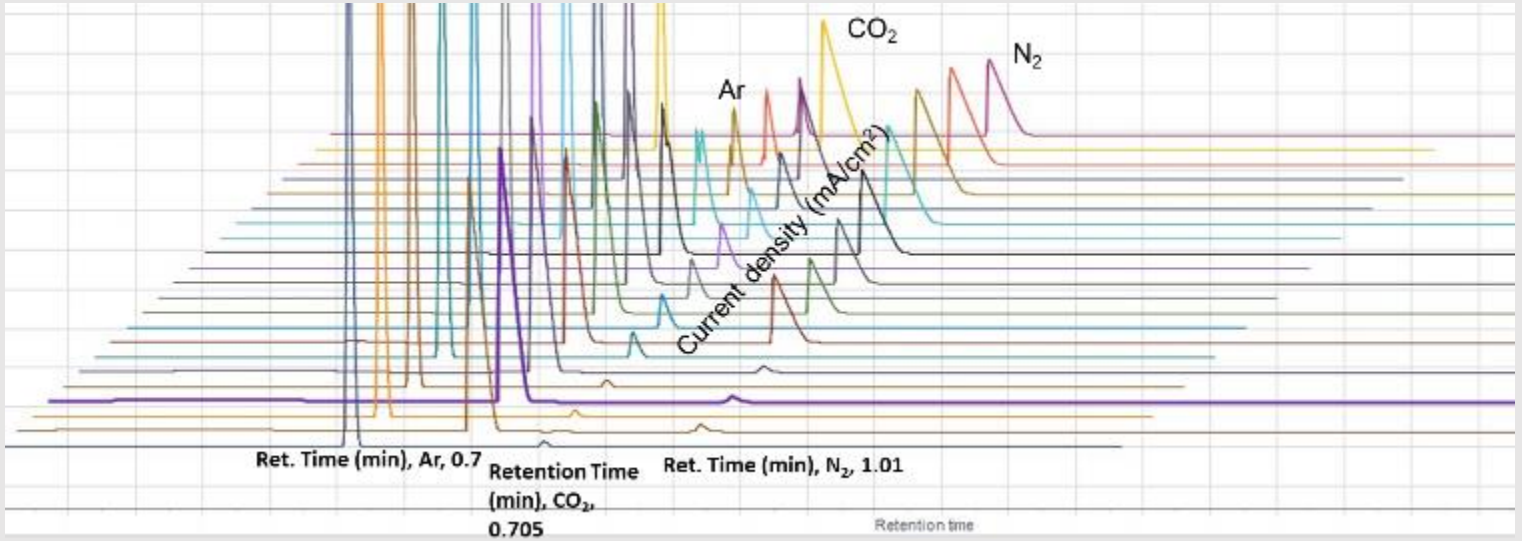
Water electrolyzer

- FAA 3-50_HCO_3^- membrane
  - Pt/C cathode
    - IrO_2 anode
    - Active area: 8 cm^2



- CO_2 crossover under various current densities for a 100% CO_2 feed
- When CO_2 is applied to the cathode, the cell voltage increases and at the same time conductivity decreases.





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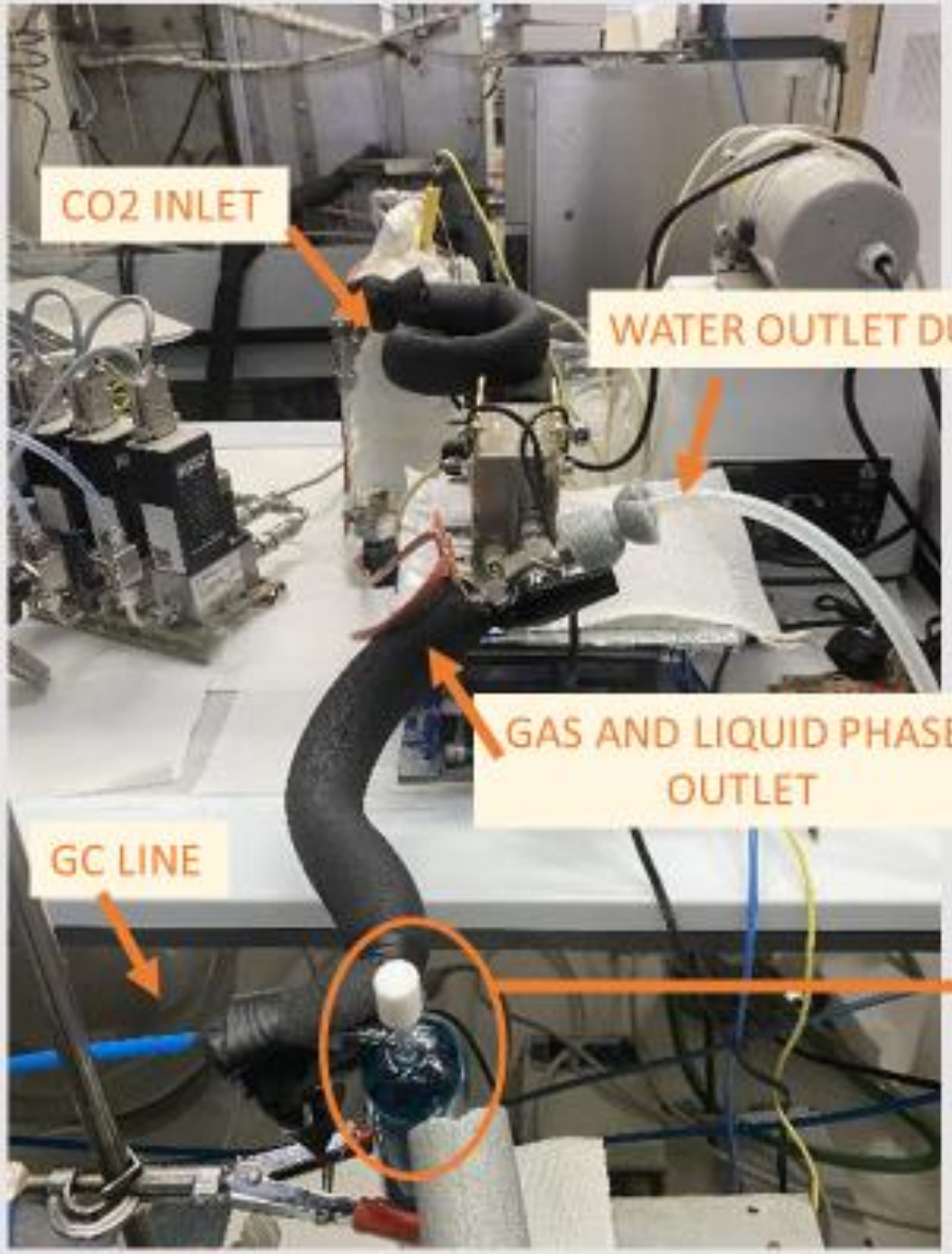
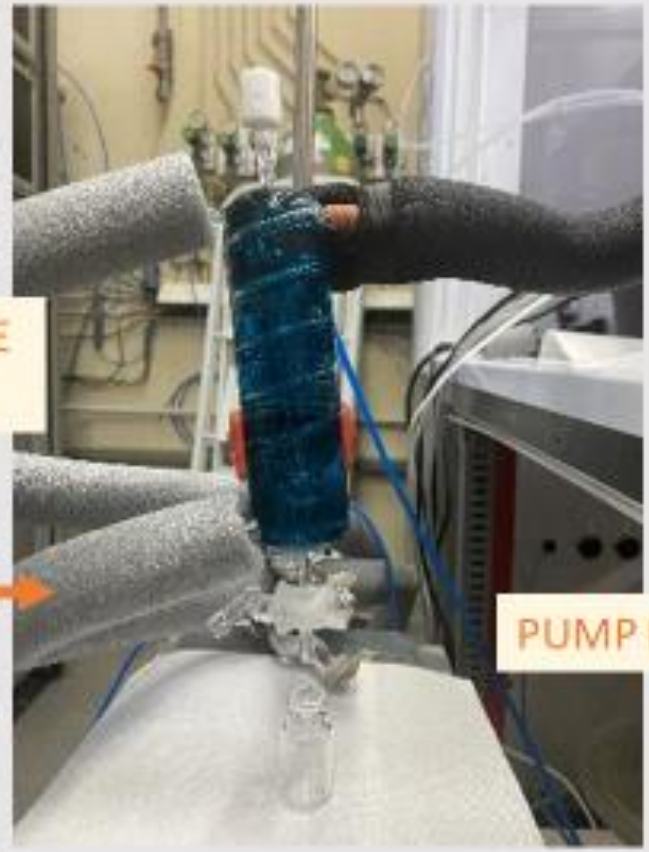
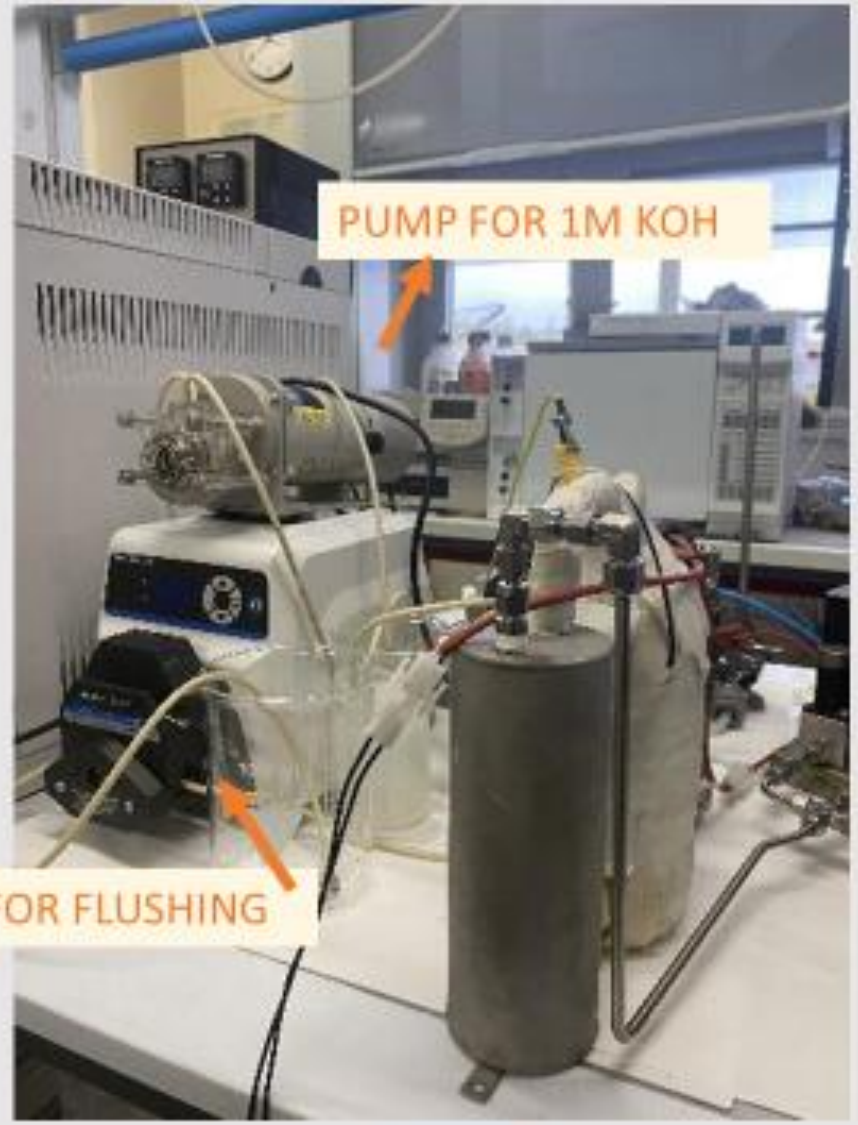
CO2 Reduction Testing Plant







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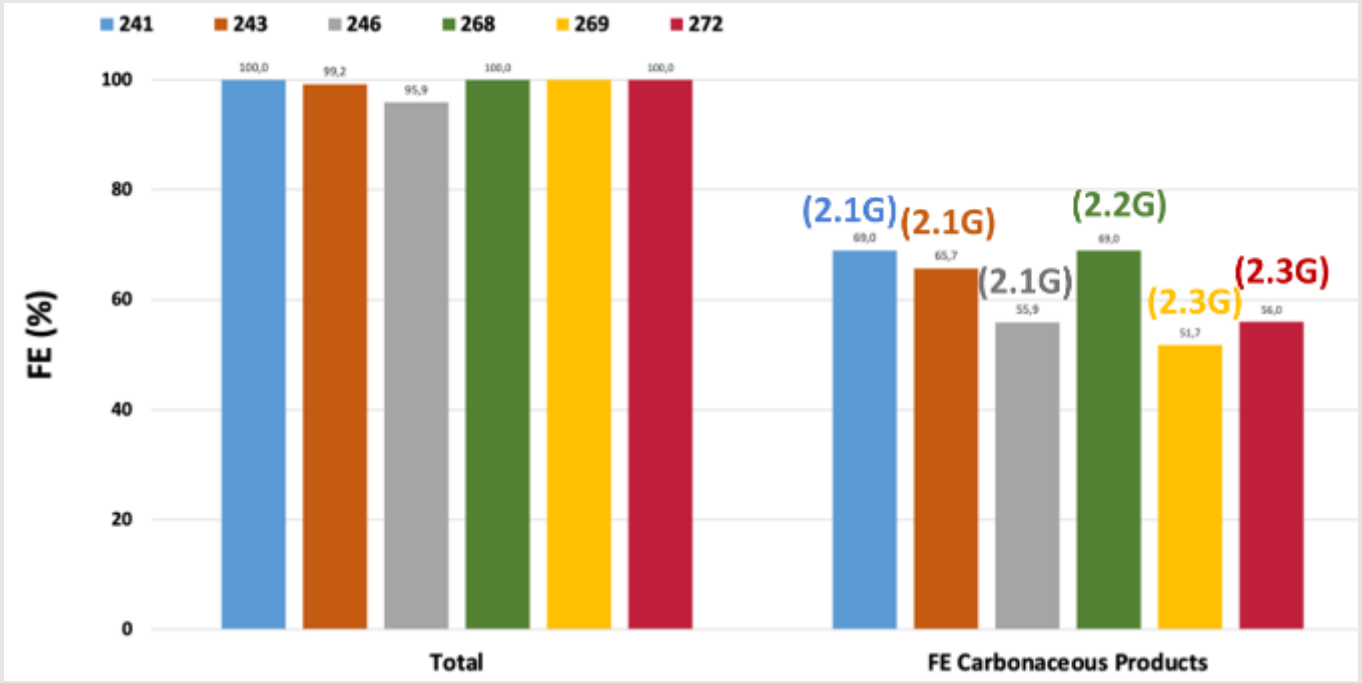
MEA test and characterization



Temperature: 50 °C

Cathode: Denora electrodes

Catholyte: CO_2 humid (T: 50 °C), Anode: NiFeOx (CNR)

Anolyte:1 M KOH, recircle. 80 mL/min

Membrane: Hydrolite (pretreated in 1M KOH)

MEA 241: CuO_UPV - Hydrolite (2.1G) - NiFe CNR

MEA 243: CuO+Ag+ionomer- Hydrolite (2.1G) - NiFe CNR

MEA 246: CuO+Ag (10:1)+ionomer- (2.1G) - NiFe CNR

MEA 268: CuO_UPV +ionomer (RB 96/104) - Hydrolite (2.2G) - NiFe CNR

MEA 269: CuO UPV +Ag (RB 93/60)Denora_Hydrolite (2.3G) _NiFe CNR

MEA 272: CuO_UPV +ionomer (RB 96/104) - Hydrolite (2.3G) - NiFe CNR

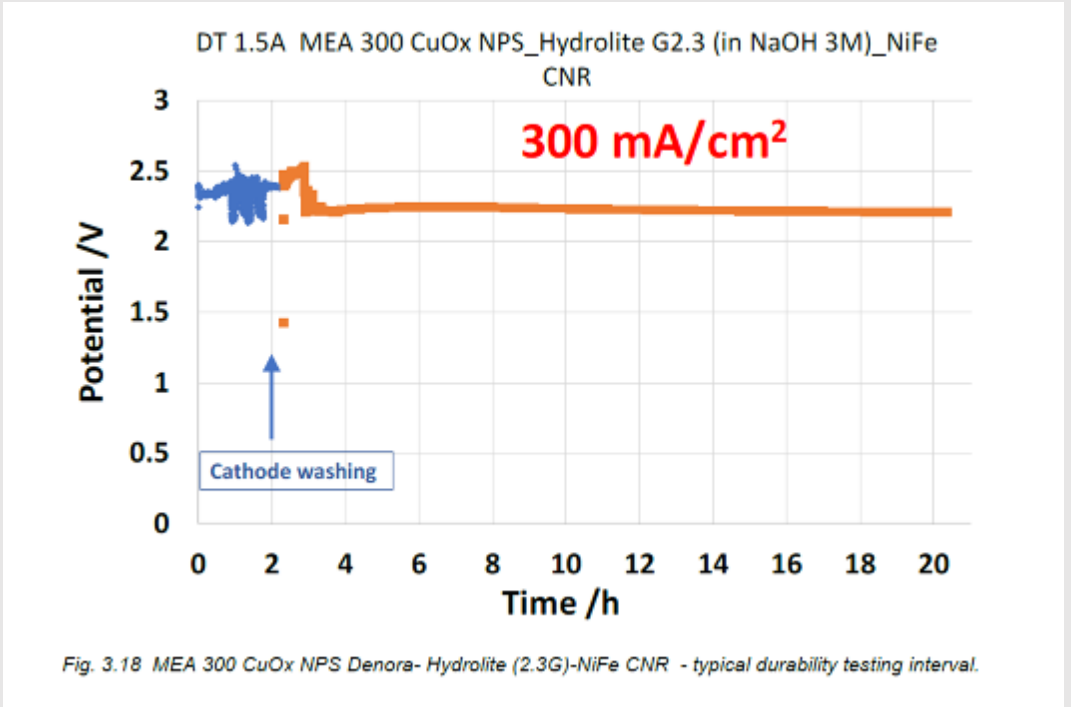


Fig. 3.18 MEA 300 CuOx NPS Denora- Hydrolite (2.3G)-NiFe CNR - typical durability testing interval.

- Selectivity and durability test

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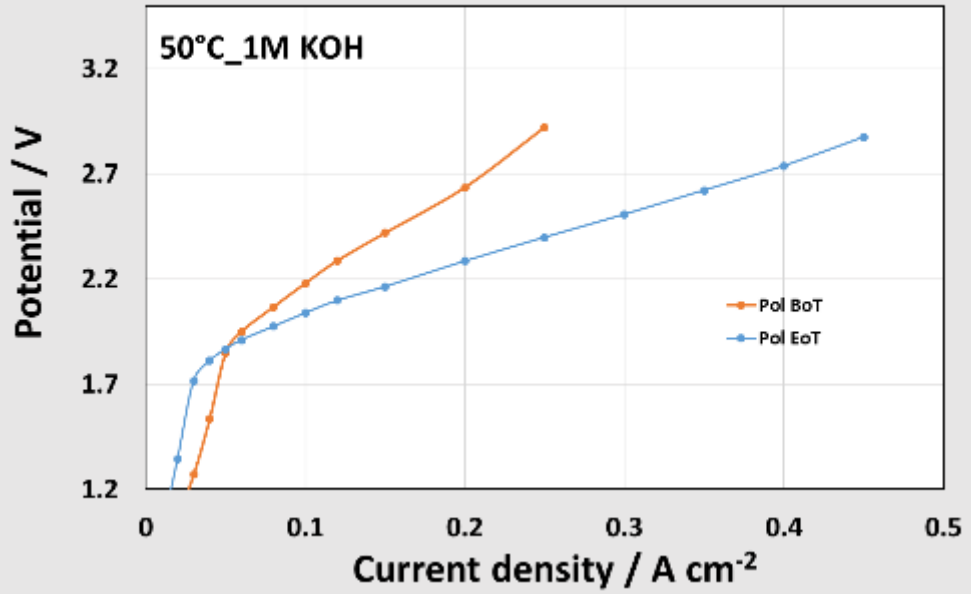
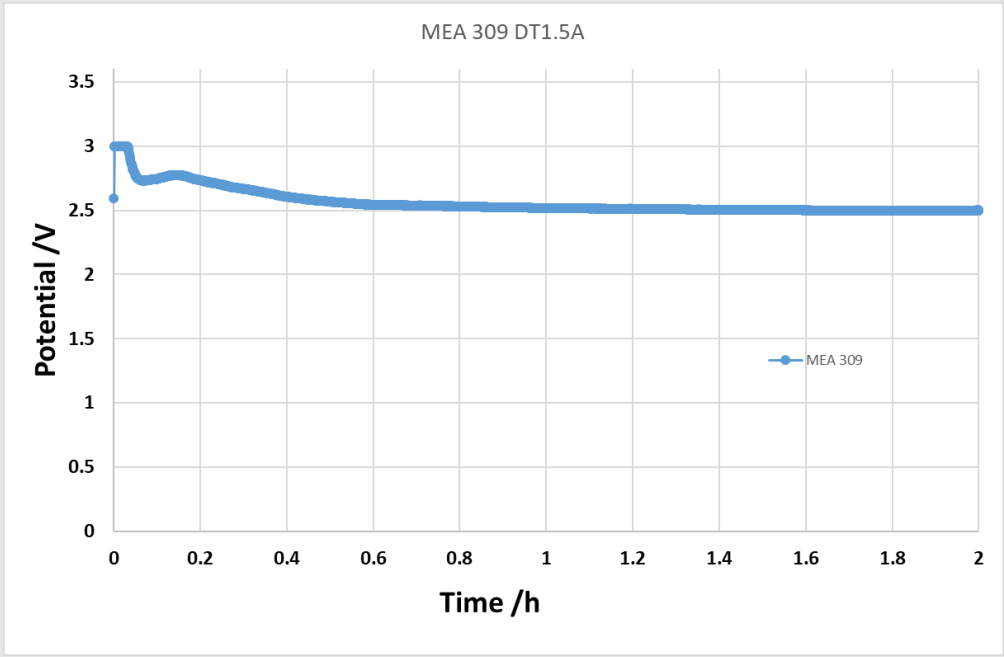
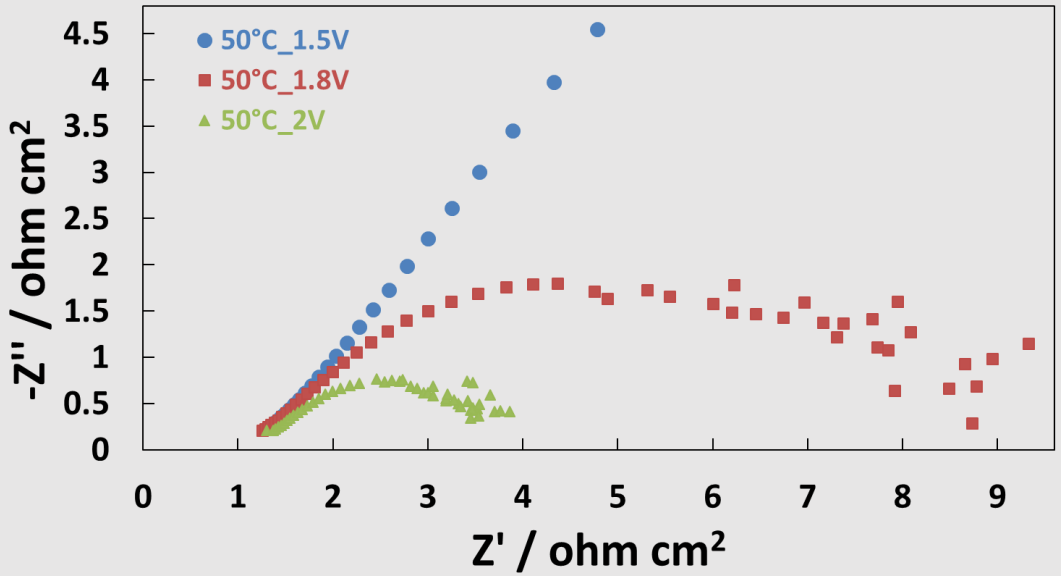
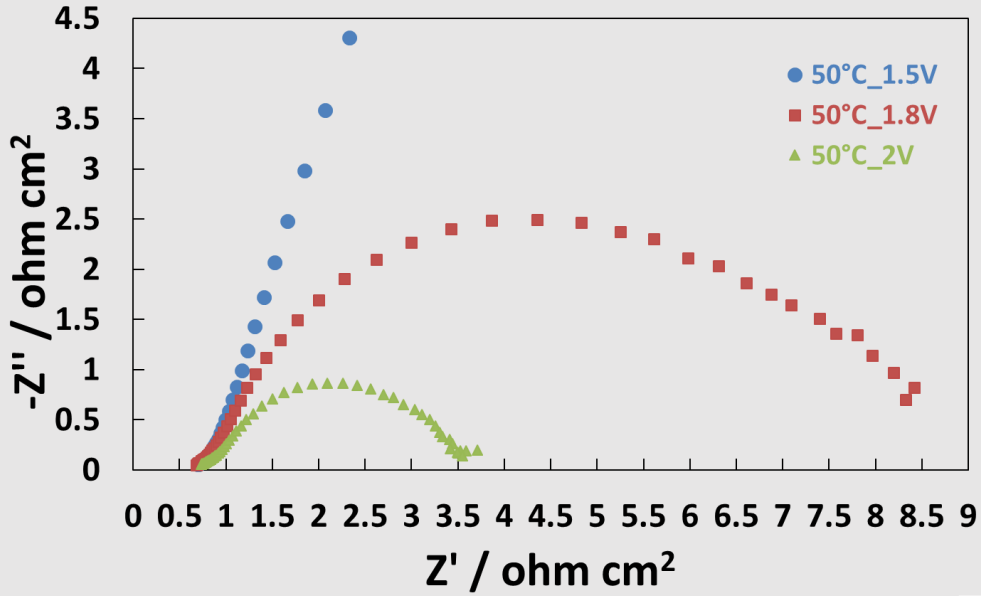
MEA 309

MEA 309: Cu2O UPV_Hydrolite G 2.3 improved_NiFe DN-CNR (DeNora)









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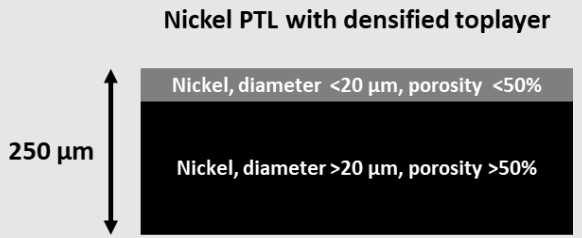
Summary and Conclusion

- Commissioning of 50kW electrolyzer
- Modified method for preparing electrocatalysts with high performance (**20 kg for anode and 20 kg for cathode catalyst**)
- Manufacturing of cathodes with production system (Coatema)
- Membrane -Improve process for better thickness uniformity and prevention of waviness
- Manufacturing of large Ni PTL anode sheets (58 cm x 30 cm)



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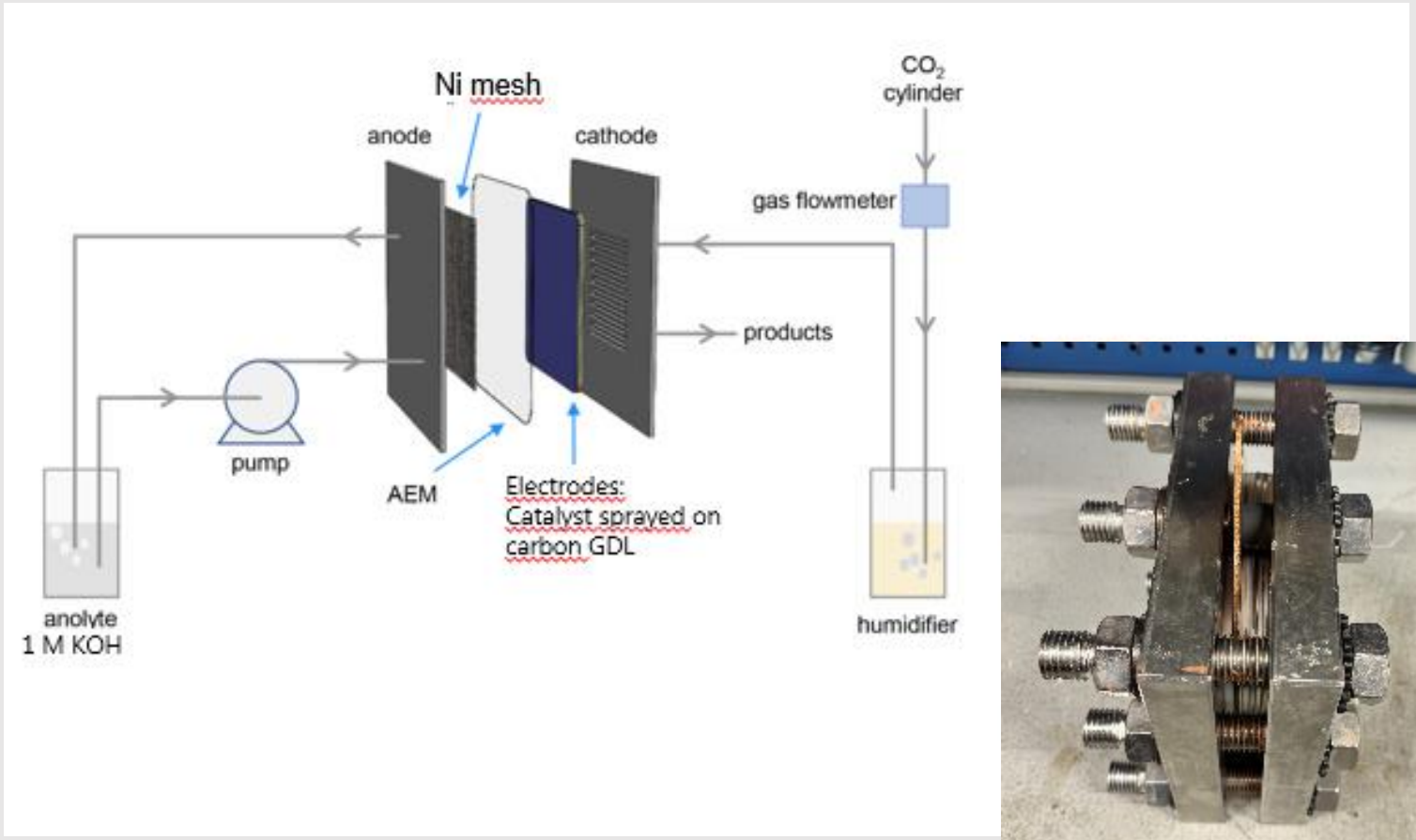
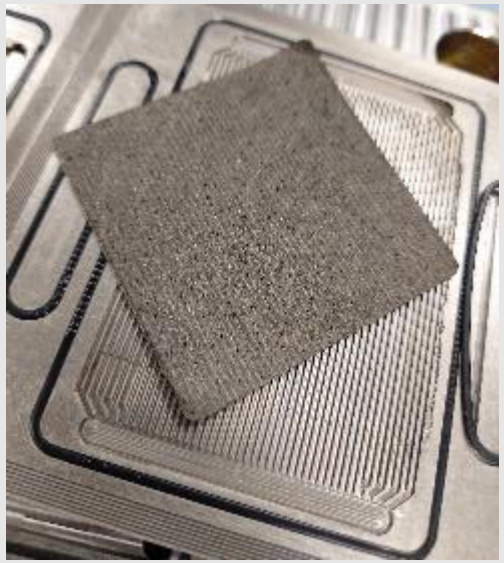
Experimental Setup Zero-Gap-Cell





Temperature: 50 °C

Cathode: Denora electrodes

Catholyte: CO_2 humid (T: 50 °C), 80 mL/min

Anode: NiFeOx (CNR)

Anolyte:1 M KOH, recircle. 80 mL/min

Membrane: Hydrolite (pretreated in 1M KOH)

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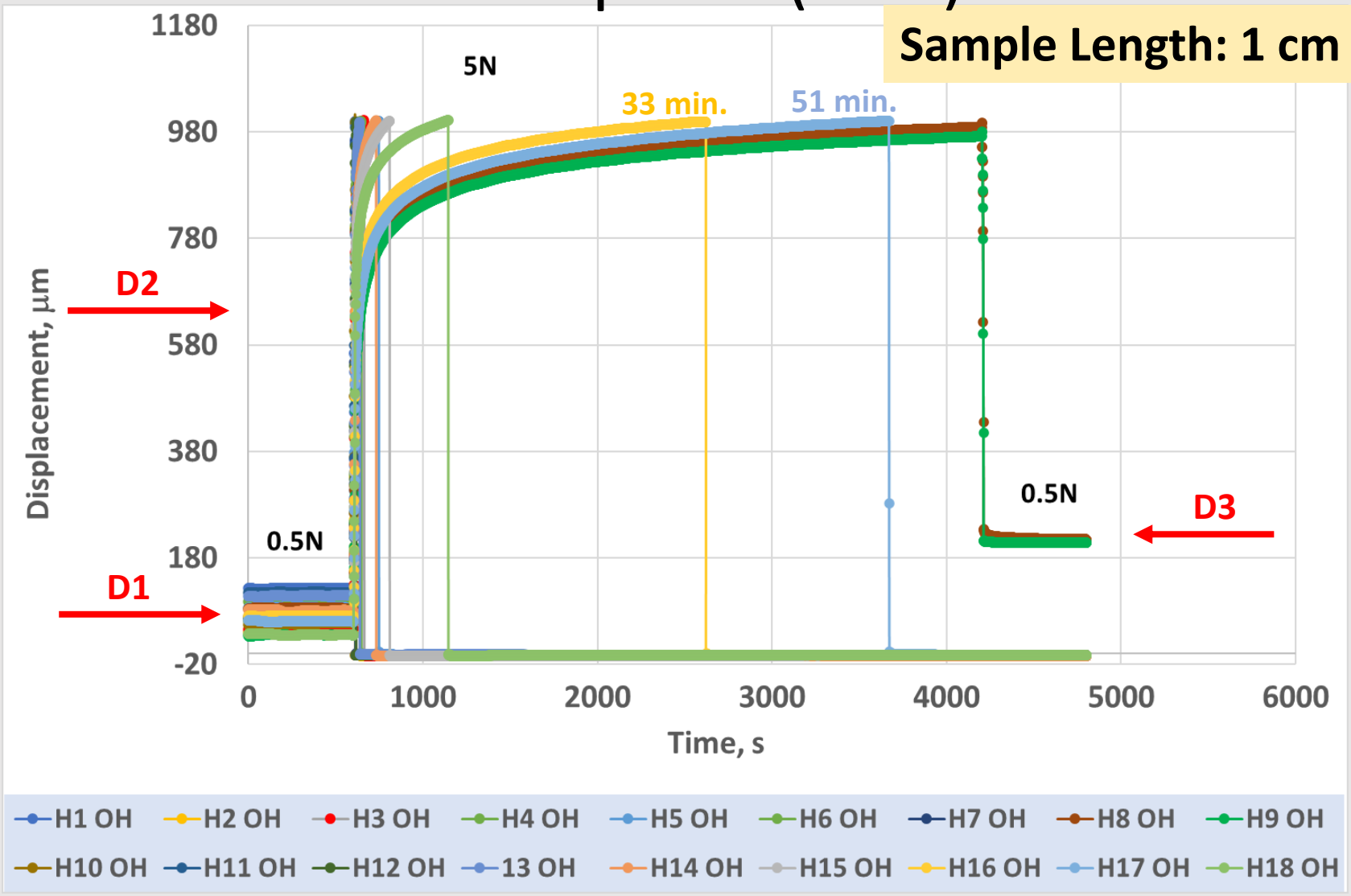
Anion exchange membrane development
Creep test (CNR)



- Successful at 5N: **H8** and **H9**
- **H16** and **H17** displacement of 1mm before 1 h at 5N

Max elastic def
(D2-D1/L)*100

Remaining def
(D3-D1/L)*100

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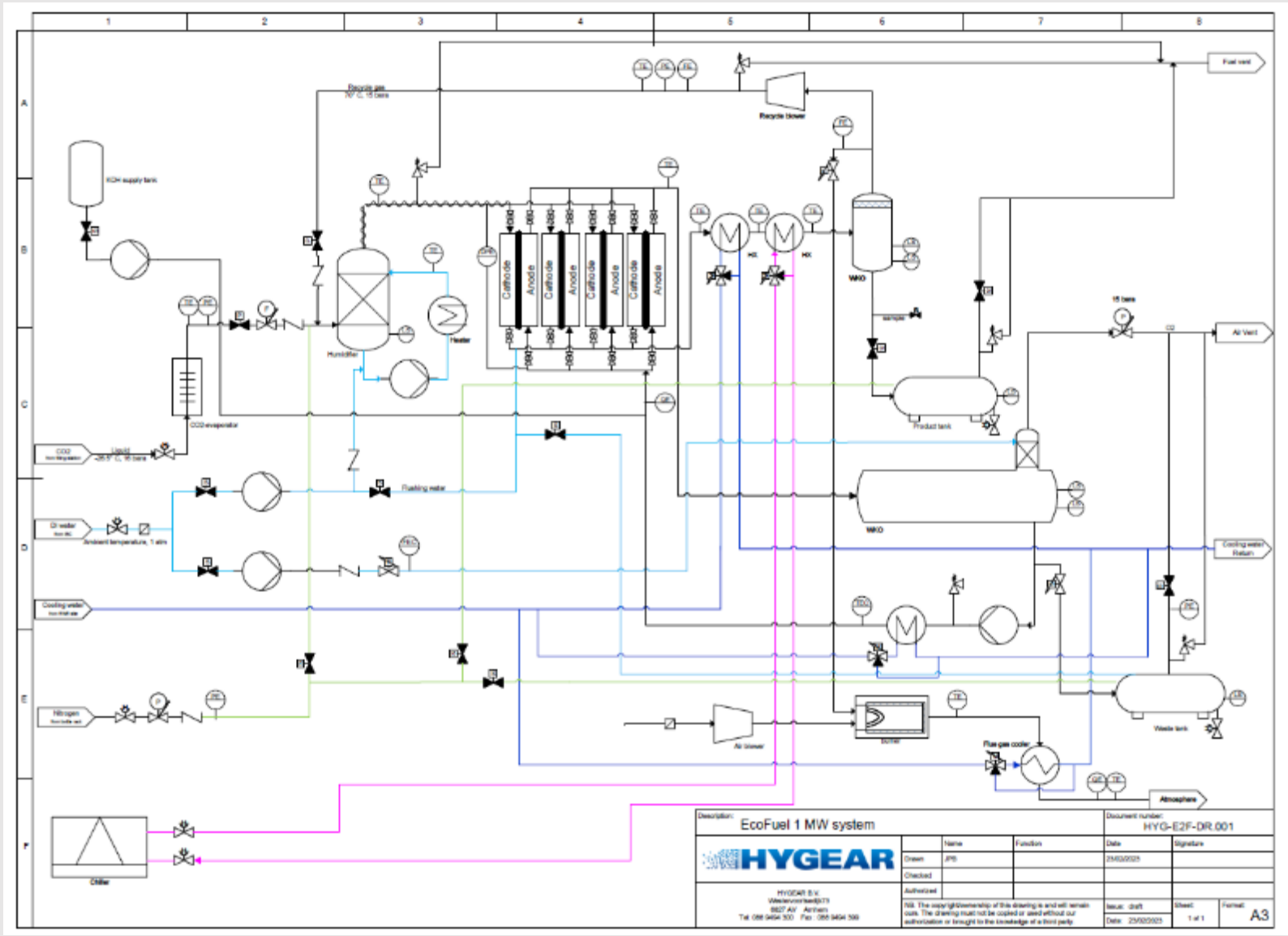
Process Flow Diagram



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