



Deliverable 2.3

Selection and validation of stack materials

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Deliverable due date: 31.8.2021

Deliverable submission date: 16.3.2022

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This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking under grant agreement No 871967. This Joint Undertaking receives support from the European Union's Horizon 2020 research and innovation programme, Hydrogen Europe and Hydrogen Europe research.



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1. Summary of scope of report

The objective of this Delivery 2.3 is the characterization of commercial membrane electrode assembly (MEAs) in small single cells from different suppliers, namely BASF, Danish Power Systems, Advent, Fumatech (In-House) and the selection of the most suitable for the project. The characterization of the MEAs involve electrochemical impedance spectroscopy (EIS) and polarization curves at different operating conditions.

2. Membrane electrode assembly (MEAs)

2.1. Advent TPS

2.1.1. Experimental conditions

The membrane electrode assembly (MEAs) used were Advent TPS, with an active area of 25 cm². This MEA is based on phosphoric acid doped PBI membrane, with total thickness of 900 μm. The MEA was placed in a single cell composed by two bipolar plate with double-serpentine geometry. PTFE gaskets with thickness of 300 μm were used to assure the fuel cell sealing; they provided a MEA compression of ca.15 %. The HT-PEMFC activation was performed at 0.63 V and 433 K with a gas flow rate adjusted for a current density of 1.0 A·cm⁻²(with λ_{Air} of 2 and λ_{H_2} of 1.2); the anode was fed with pure hydrogen and the cathode with air. The characterization was performed at temperatures of 433 K and 453 K, pressure between 1.0 bar and using a gas stoichiometry of $\lambda_{\text{Air}}=2$ and $\lambda_{\text{H}_2}=1.2$.

The HT-PEMFC was characterized by electrochemical impedance spectroscopy (EIS) and polarization curves using Zahner-elektrik/P241 Potentiostat. Nyquist spectra were obtained at 0.2 A·cm⁻², between 100 kHz and 100 mHz with a perturbation amplitude of 5 mV. Polarization curves were obtained galvanostatically between 0.05 A·cm⁻² and 0.6 A·cm⁻².

2.1.2. Experimental results

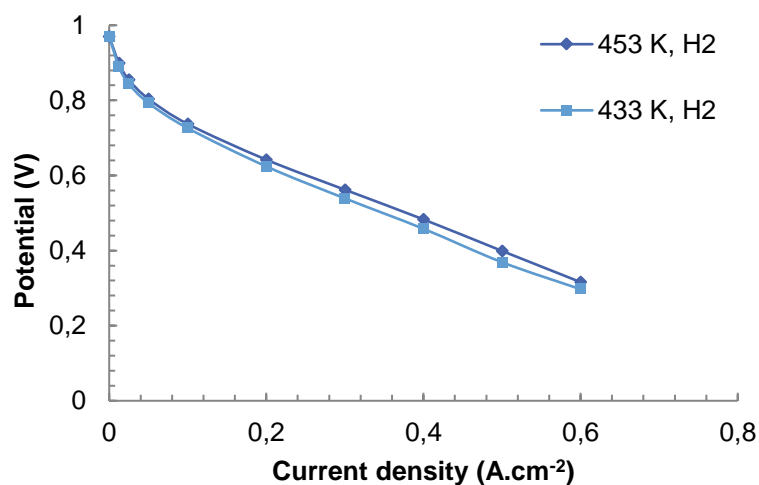


Fig. 1 – Electric potential difference as function of the current density at temperatures of 433 K and 453 K using Advent TPS MEA– lines were added for readability.

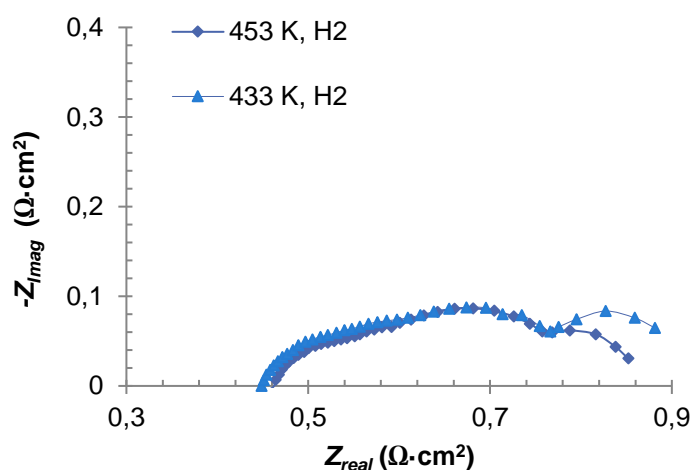


Fig. 2 – Nyquist plot at 0.2 A·cm⁻² immediately after the activation with hydrogen at temperatures of 433 K and 453 K using Advent TPS MEA – lines were added for readability.

This MEAs provides a current density of 0.6 A·cm⁻² at 0.3 V at 433 K, lower than the observed for Celtec P2200N. The ohmic resistances for this MEAs was ca. 0.44 Ω·cm². Long term stability tests for Advent MEAs at 453 K with reformat at constant current density of 0.3 A·cm⁻², are reported in the literature showing a potential loss between 300 μV·h⁻¹ to 600 μV·h⁻¹ K [6]

2.2. In-house MEA

2.2.1. Experimental conditions

A PBI membrane from Fumatech with thickness of 30 μm was doped in 85% H_3PO_4 in a closed vessel at 140 $^\circ\text{C}$ for 24 h. The doping level based on a dry membrane was fixed at 185 wt%. The MEA was assembled using Gas Diffusion Electrode loaded with 1 mgPt cm^{-2} (mass fraction of 20 % Pt/Carbon from Johnson Matthey). The MEA was placed in a single cell of 25 cm^2 , composed by two bipolar plate with double-serpentine geometry. PTFE gaskets with thickness of 250 μm were used to assure the fuel cell sealing; they provided a MEA compression of ca.15 %.

The HT-PEMFC activation was performed at 0.63 V and 433 K with a gas flow rate adjusted for a current density of 1.0 $\text{A}\cdot\text{cm}^{-2}$ (with λ_{Air} of 2 and λ_{H_2} of 1.2); the anode was fed with pure hydrogen and the cathode with air. The characterization was performed at temperatures of 433 K and 453 K, pressure between 1.0 bar and using a gas stoichiometry of $\lambda_{\text{Air}} = 2$ and $\lambda_{\text{H}_2}=1.2$.

The HT-PEMFC was characterized by electrochemical impedance spectroscopy (EIS) and polarization curves using Zahner-elektrik/P241 Potentiostat. Nyquist spectra were obtained at 0.2 $\text{A}\cdot\text{cm}^{-2}$, between 100 kHz and 100 mHz with a perturbation amplitude of 5 mV. Polarization curves were obtained galvanostatically between 0.05 $\text{A}\cdot\text{cm}^{-2}$ and 0.8 $\text{A}\cdot\text{cm}^{-2}$.

2.2.2. Experimental results

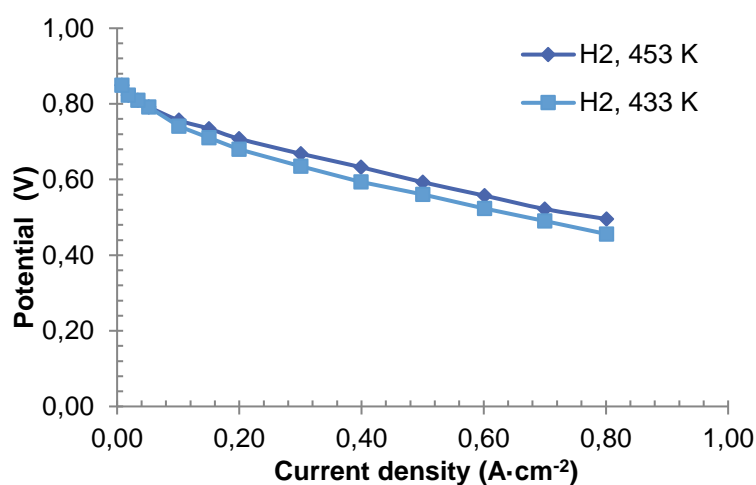


Fig. 3 – Electric potential difference as function of the current density at temperatures at 433 K and 453 K using in-house MEA – lines were added for readability.

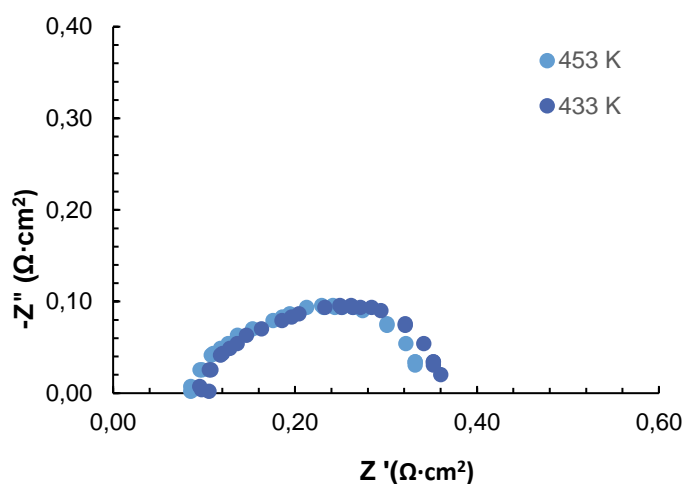


Fig. 4 – Nyquist plot at $0.2 \text{ A}\cdot\text{cm}^{-2}$ immediately after the activation with hydrogen at temperatures of 453 K using in-house MEA – lines were added for readability.

This MEAs provides a high current density of $0.6 \text{ A}\cdot\text{cm}^{-2}$ at 0.524 V at 433 K. The ohmic resistances for this MEAs was ca. $0.115 \text{ }\Omega\cdot\text{cm}^2$. The high doping level provided quite high performance, yet affecting the long term stability. Previous experiments pointed to a potential loss, at constant current density of $0.2 \text{ A}\cdot\text{cm}^{-2}$, of ca. $6.3 \text{ }\mu\text{V}\cdot\text{h}^{-1}$ at 433 K increasing up to $60 \text{ }\mu\text{V}\cdot\text{h}^{-1}$ at 463 K [7].

2.3. Blue World Technology

2.3.1. Experimental conditions

The membrane electrode assembly (MEA) from Blue World Technology has an active area of 21 cm^2 with an electrolyte based on phosphoric acid (PA) doped PBI membrane. The MEA was tested at UPorto using a testing cell from Pragma Industries composed of two bipolar plates with double-serpentine geometry. The $150 \text{ }\mu\text{m}$ thick polytetrafluoroethylene (PTFE) gaskets were used to ensure the gas-tight sealing and providing a cell compression of ca. 15-20 %.

The HT-PEMFC activation was performed during 22 h at 0.63 V and 433 K with a gas flow rate adjusted for a current density of $1.0 \text{ A}\cdot\text{cm}^{-2}$ ($\lambda_{\text{Air}} = 2.0$, $\lambda_{\text{H}_2} = 1.2$). The anode was fed with pure hydrogen and the cathode with air. The characterization was performed at temperatures of 413 K and 433 K, pressure between 1.0 bar and 1.5 bar, and gas stoichiometry of $\lambda_{\text{Air}} = 2.0$ and $\lambda_{\text{H}_2} = 1.2$.

The HT-PEMFC was characterized by electrochemical impedance spectroscopy (EIS) and polarization curves using Zahner-elektrik/P241 Potentiostat. Nyquist spectra were obtained at $0.2 \text{ A}\cdot\text{cm}^{-2}$, between 100 kHz and 100 mHz with a perturbation amplitude of 5 mV. Polarization curves were obtained galvanostatically between $0.05 \text{ A}\cdot\text{cm}^{-2}$ and $0.8 \text{ A}\cdot\text{cm}^{-2}$.

For comparison purposes these MEAs were tested at Blue World Technology facilities using two different testing cells on one from Baltic [8] with compression control and one developed in-house using $150 \mu\text{m}$ thick polytetrafluoroethylene (PTFE) gaskets to ensure the gas-tight sealing and cell compression (ca. 15-20 %).

2.3.2. Experimental results

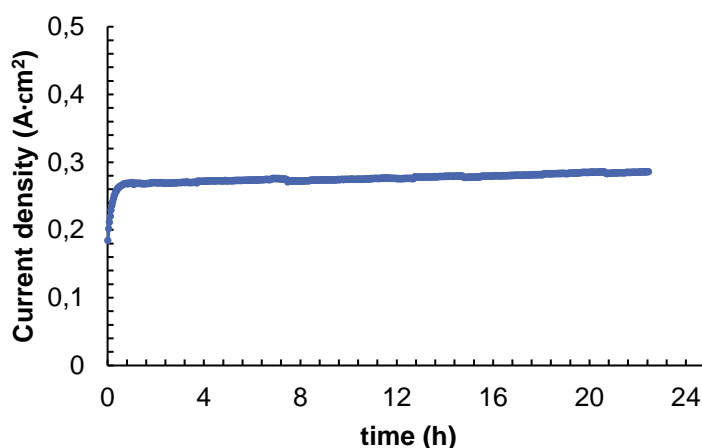


Fig. 5 – MEA activation at 0.63 V.

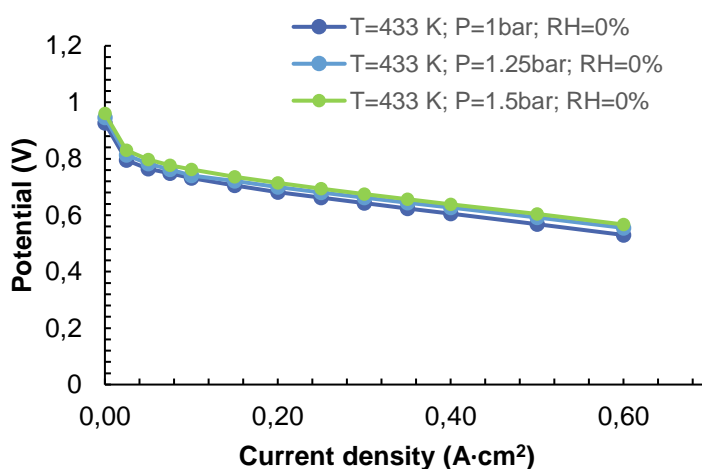


Fig. 6 – Electric potential difference as function of the current density at temperatures of 433 K using Blue World Technology MEA – lines were added for readability.

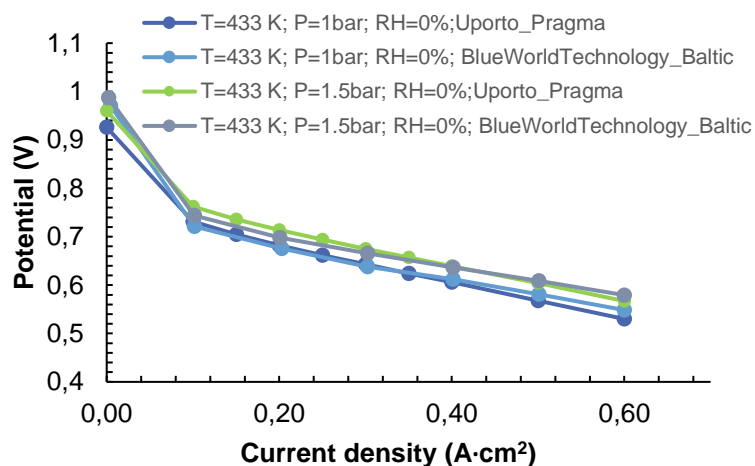


Fig. 7 – Performance evaluation using different testing stations and cells. Electric potential difference as function of the current density at temperatures of 433 K using Blue World Technology MEA.

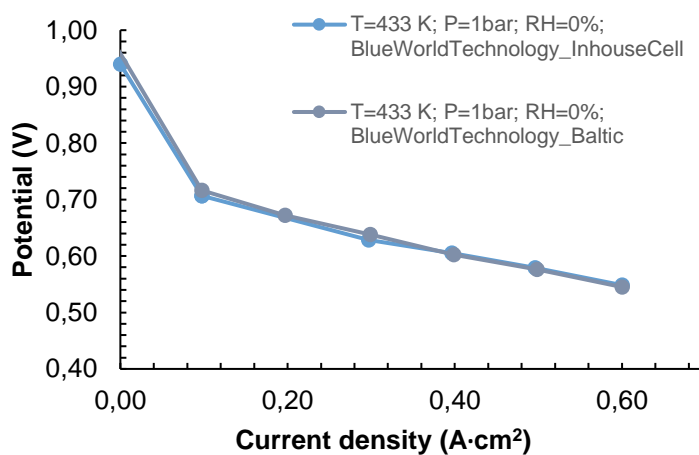


Fig. 8 – Performance evaluation using different testing cells. Electric potential difference as function of the current density at temperatures of 433 K using Blue World Technology MEA.

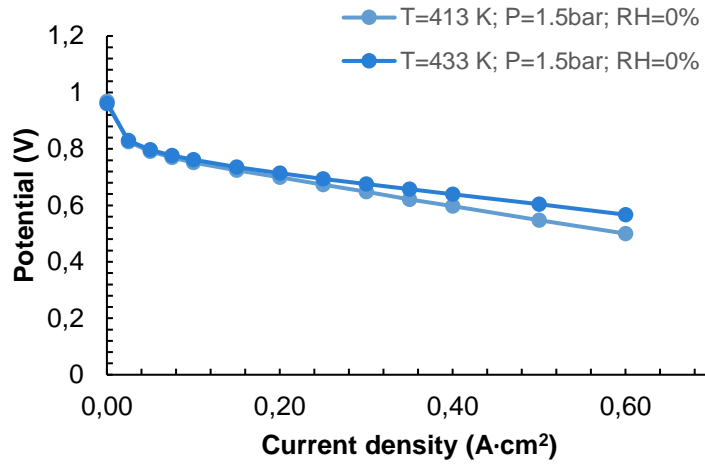


Fig. 9 – Electric potential difference as function of the current density at temperatures of 413 K and 433 K using Blue World Technology MEA. Tests at UPorto using Pragma testing cell.

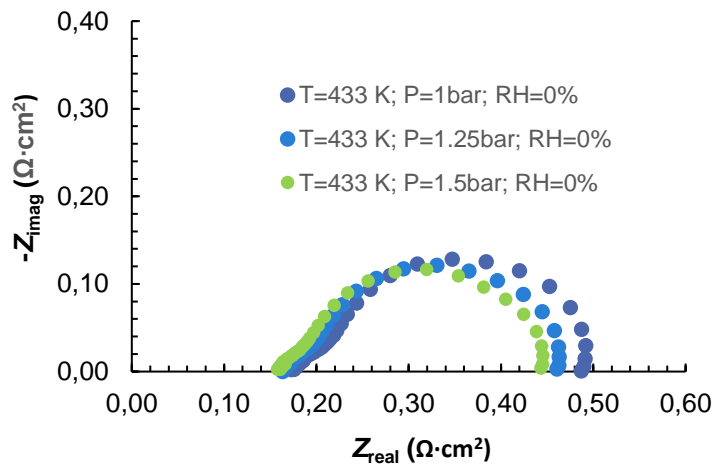


Fig. 10 – Nyquist plot at 0.2 A·cm⁻² immediately after the activation at temperatures of 433 K and different pressures. Tests at UPorto using Pragma testing cell.

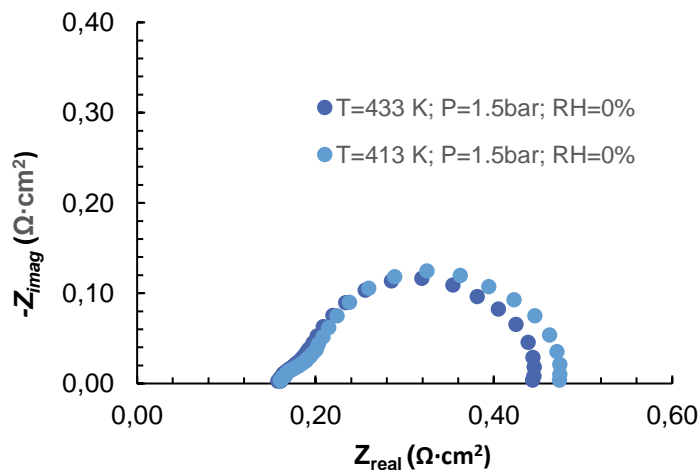


Fig. 11 – Nyquist plot at 0.2 A·cm⁻² and temperatures of 413 K and 433 K. Tests at UPorto using Pragma testing cell.

Although this MEA generally activates up to 200 h, the activation was carried out during ca. 24 h, to keep the testing procedure comparable to the one used for the other MEAs. The activation process was, therefore, interrupted and the cell characterization was initialized. This MEAs provides a high current density of $0.6 \text{ A}\cdot\text{cm}^{-2}$ at 0.53 V at 433 K and 1 bar . The ohmic resistances for this MEAs was ca. $0.158 \text{ }\Omega\cdot\text{cm}^2$. This MEAs showed high performance, yet slightly lower than the Celtec P2200N. The production process of this MEA, resulting in a strong phosphoric acid retention in the membrane, points out to higher cell durability [8].

2.4. BASF Celtec P2200N

2.4.1. Experimental conditions

The membrane electrode assembly (MEAs) used were Celtec P2200N, from BASF, with an active area of 45 cm^2 . This MEA is based on phosphoric acid doped PBI membrane, with total thickness of $852 \text{ }\mu\text{m}$. The catalyst loading was $1 \text{ mg Pt}\cdot\text{cm}^{-2}$ and $0.85 \text{ mg Pt}\cdot\text{cm}^{-2}$ on the anode and cathode, respectively. The MEA was placed in a single cell composed by two bipolar plate with hexa-serpentine geometry. PTFE gaskets with thickness of $300 \text{ }\mu\text{m}$ were used to assure the fuel cell sealing; they provided a MEA compression of ca. 18 %. The HT-PEMFC activation was performed during at 0.63 V and 433 K with a gas flow rate adjusted for a current density of $1.0 \text{ A}\cdot\text{cm}^{-2}$ (with λ_{Air} of 2 and λ_{H_2} of 1.2); the anode was fed with pure hydrogen, synthetic reformat and methanol reformat, and the cathode with air. The characterization was performed at temperatures of 433 K and 453 K , pressure of 1.0 bar and using a gas stoichiometry of $\lambda_{\text{Air}} = 2$ and $\lambda_{\text{H}_2} = 1.2$. The HT-PEMFC was characterized by electrochemical impedance spectroscopy (EIS) and polarization curves using Zahner-elektrik/P241 Potentiostat. Nyquist spectra were obtained at $0.2 \text{ A}\cdot\text{cm}^{-2}$, between 100 kHz and 100 mHz with a perturbation amplitude of 5 mV . Polarization curves were obtained galvanostatically between $0.05 \text{ A}\cdot\text{cm}^{-2}$ and $0.6 \text{ A}\cdot\text{cm}^{-2}$.

2.4.2. Experimental results

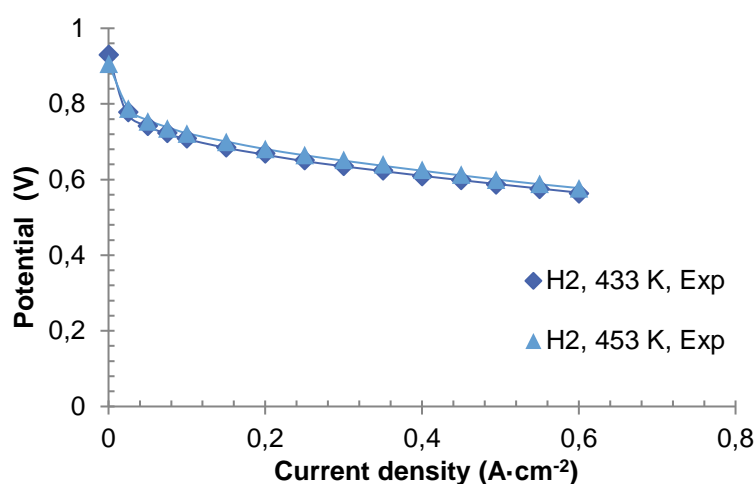


Fig. 12 – Electric potential difference as function of the current density at temperatures of 433 K and 453 K using Celtec P2200N MEA – lines were added for readability.

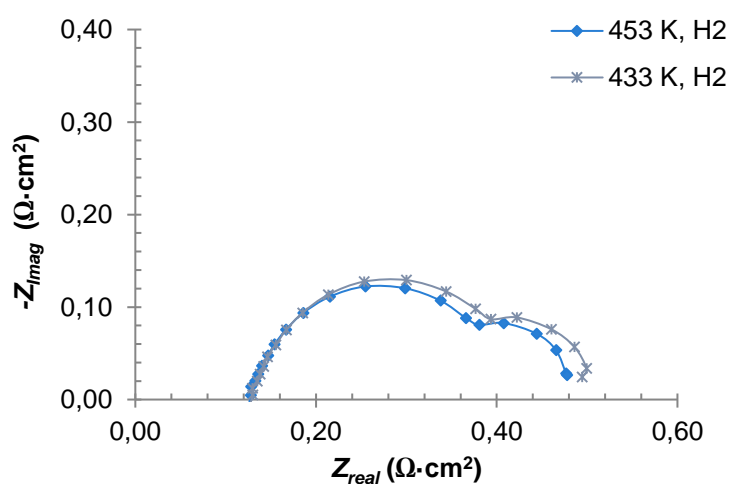


Fig. 13 – Nyquist plot at 0.2 A·cm⁻² immediately after the activation with hydrogen at temperatures of 433 K and 453 K using Celtec P2200N MEA – lines were added for readability.

This Celtec®-P 1000 MEAs present a quite high performance, providing a current density of 0.6 A·cm⁻² at 0.57 V at 433 K. The ohmic resistances for this MEAs was ca. 0.12 Ω·cm². Should be noticed, that the ohmic resistances of the setup (cables, testing cell) are 3.6 mΩ (cell area 25 cm²) and are included in all the results presented in this work.

The Celtec®-P 1000, as the MEA with the best performance, were also evaluated using synthetic reformat and methanol reformat as fuel at 453 K. The results showed very small differences in the cell performance between for the different fuels (Figure 14). In terms of EIS

slight differences are observed at low frequencies, normally related to mass transfer resistances (Figure 15).

Long term stability tests for Celtec®-P 1000 MEA at 433 K and constant current density of $0.2 \text{ A}\cdot\text{cm}^{-2}$, are already reported in the literature showing a potential loss of ca. $5 \mu\text{V}\cdot\text{h}^{-1}$ [1, 2, 3] and $25 \mu\text{V}\cdot\text{h}^{-1}$ at 453 K [4, 5].

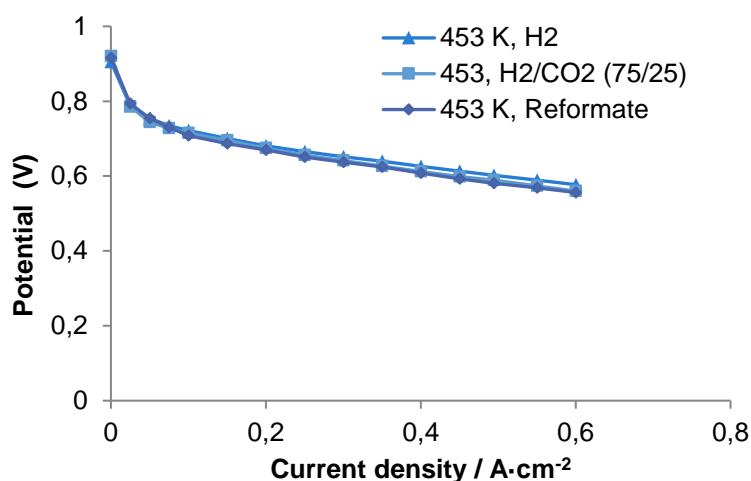


Fig. 14 – Electric potential difference as function of the current density using hydrogen, synthetic reformate and methanol reformate at temperatures of 453 K.

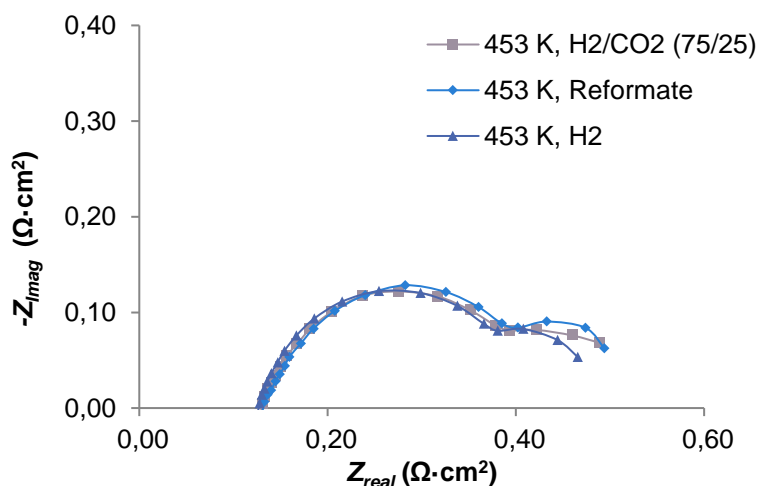


Fig. 15 – Nyquist plot at $0.2 \text{ A}\cdot\text{cm}^{-2}$ using hydrogen, synthetic reformate and methanol reformate at temperatures of 453 K.

3. Conclusions

The experimental results show that the Celtec P2200N from BASF has the highest performance among the tested MEAs. Using methanol reformate has minor effect on the fuel cell performance. The Blue World Technology MEAs also provide quite high performance, with a higher cell durability due to strong PA immobilization within the PBI membrane matrix. The operating pressure has noticeable effect on the fuel cells performance, demonstrating the importance of operating above the atmospheric pressure. During the MEAs evaluation using different testing stations and testing cells, was noticeable the effect of the parasitic losses, namely ohmic resistances from cables and electrical contact between materials, on the cell performance, which will be studied and minimized in the final prototype. The Blue World Technology MEAs will be used in the final prototype due to practical issues, such as availability.

4. References

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