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THESIS

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di Torino**

Master Thesis

**Experimental study of the effect of compression on various PEM
electrolyser cells: KPIs and EIS analysis**

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Contents

List of Figures	vi
Abstract	1
Chapter 1: Introduction	2
1.1 Global Energy Context and Role of Hydrogen	2
1.2 Hydrogen Production Technologies	2
1.2.1 Electrolysis Technologies (Advanced Methods).....	2
1.2.2 Alkaline Electrolysis	3
1.2.3 Proton Exchange Membrane (PEM) Electrolysis	3
1.2.4 Solid Oxide Electrolysis Cells (SOEC).....	4
1.2.5 Anion Exchange Membrane (AEM) Electrolysis	4
1.3 PEM Electrolyzers Structure and Components	5
1.3.1 PEM Electrolyzer Structure Overview	5
1.3.2 Proton Exchange Membrane.....	5
1.3.3 Catalyst Layers.....	6
1.3.4 Porous Transport Layer (PTL)	6
1.3.5 Gas Diffusion Layer (GDL).....	6
1.3.6 Bipolar Plates.....	7
1.4 Key Challenges in PEM Electrolysis.....	7
1.4.1 Challenges in Durability, Cost and Transport for PEM Electrolyzers.....	7
1.5 Research Gap and Thesis Objectives.....	8
1.5.1 Research Gap	8
1.5.2 Thesis Objectives.....	9
1.5.3 Thesis Structure.....	9
1.5.4 Forward Look and Reflection	10
Chapter 2. Literature Review	11
2.1 Role of Compression and GDLs in PEM Electrolyzers	11
2.1.1 Functions of GDL and PTL	11
2.1.2 Compression effect on the Electrical, Thermal, and Transport properties	12
2.1.3 Trade-offs and Innovations	13
2.1.4 Relevance to Thesis	14
2.2 Experimental Studies on Compression	14

2.2.1 Effect of the Compression on PEM Electrolyzer	15
2.2.2 Effects of Compression on Carbon-Based GDLs in PEM Electrolyzers	34
2.2.3 Effects of Compression on Titanium-Based GDLs in PEM Electrolyzers.....	35
2.2.4 Hybrid and Novel Materials	35
2.2.5 Moderate and High Compression Comparison.....	36
2.3 The Role of KPIs in Compression Effects	37
2.3.1 Criteria of the Efficiency.....	37
2.3.2 Productivity of The Hydrogen.....	38
2.3.3 Durability Criteria.....	39
2.3.4 Integration with EIS Studies	39
2.4 EIS as Diagnostic Tool in Compression Studies	40
2.4.1 EIS for Resolving Resistances	40
2.4.2 Interpretation of Nyquist and Bode Plots	41
2.5 Literature Gaps and Relevance to Present Work.....	41
2.5.1 Gaps in Compression-Related Research on PEM Electrolyzers	42
2.5.2 Insufficient Integration of Compression, KPIs, and EIS in PEM Electrolyzer Research	42
2.5.3 Novelty of the Present Work	43
Chapter 3. Experimental	44
3.1. Electrochemical test bench	44
3.1.1. Explanation of the test bench	44
3.1.2. Electrochemical cell (Test 1).....	48
3.1.3. Electrochemical cell (Test 2).....	50
3.1.4 The quickCONNECTfixture preparation of the Setup.....	52
3.1.5 Compression Pressure Definition in the quick Connect Fixture	52
3.2. Cell assembly	54
3.2.1 cleaning	54
3.2.2 Assembly procedure	54
3.2.3 Pressure regulator setup	56
Chapter 4. ECM & EIS	57
4.1 Introduction of ECM & EIS.....	57
4.2 EIS Test Equipment and Set-up	57

4.2.1 Measurement Modes in AC Impedance Spectroscopy	59
4.3 Test Procedure.....	60
4.4. Data Quality Kramers Keronig (KK) Analysis	66
4.5 Selection of the Equivalent Circuit Model	70
4.5.1 High-frequency (HF) region	71
4.5.2 Mid-frequency (MF) region.....	72
4.5.3 Low-frequency (LF) region	73
4.6 ECM Fitting Results.....	74
4.6.1 Initial Estimation of the Parameters	74
4.6.2 Model Development.....	74
4.7 Evaluation of Fitting Scenarios and Selection of the Final ECM Configuration	76
4.7.1 Fixing the Ohmic Resistance Based on Analytical Estimation (Scenario 1)	77
4.7.2 Fixing the CPE Exponent n (Scenario 2)	78
4.7.3 All Parameters Unfixed (Scenario 3).....	78
4.7.4 Python-Based Fitting Routine (Scenario 4)	79
4.7.5 Final Results	80
Chapter 5. Results.....	81
5.1 Results of the Test One	81
5.1.1 Compression Effect of the Test One	81
5.1.2 Basics of Degradation.....	81
5.1.3 Analysis of Voltage Behavior Under Different Conditions (Test 1)	81
5.1.3.1 Effect of the Degradation at Compression 3.4 bar (Test 1)	83
5.1.3.2 Effect of the Degradation at Compression 4.4 bar (Test 1)	83
5.1.3.3 Effect of the Degradation at Compression 5 bar (Test 1).....	84
5.1.3.4 Effect of the Degradation at Compression 4.4 bar descent (Test 1)	85
5.1.3.5 Effect of the Degradation at Compression 3.4 bar descent (Test 1)	86
5.1.3.6 Effect of the Degradation at Compression 2.4 bar descent (Test 1)	86
5.1.3.7 Analysis of the Degradation Effect (Test 1)	87
5.1.4 EIS Characterization at Different Compression level (Test 1)	88
5.1.4.1 Impedance Response Under Potentiostatic Operation (0 V)	89
5.1.4.2 Impedance Response Under Potentiostatic Operation (0.5 V)	91
5.1.4.3 Impedance Response Under Potentiostatic Operation (1V)	92

5.1.4.4 Impedance Response Under Potentiostatic Operation (1.3V).....	94
5.1.4.5 Impedance Response Under Potentiostatic Operation (1.5V).....	96
5.2 Results of the Test Two	101
5.2.1 EIS Analysis & Protocols of the Test Two.....	101
5.3 Validation of the experimental results.....	103
5.3.1 Polarization curves.....	103
5.4 Analysis of Voltage Behavior Under Different Conditions (Test 2)	106
5.4.1 Effect of the Degradation at Compression 2.4 bar (Test 2).....	106
5.4.2 Effect of the Degradation at Compression 3.4 bar (Test 2).....	106
5.4.3 Effect of the Degradation at Compression 4.4 bar (Test 2).....	107
5.4.4 Effect of the Degradation at Compression 4.9 bar (Test 2).....	107
5.4.5 Effect of the Degradation at Compression 4.4 bar Descent (Test 2).....	108
5.5 EIS Characterization Before and After Compression Variation	109
5.5.1 Impedance Response Under Potentiostatic Operation (1.5 V)	109
5.5.2 Impedance Response Under Potentiostatic Operation (1.3 V)	116
5.5.3 Impedance Response Under Galvanostatic Operation (1.5 A)	119
5.5.4 Impedance Response Under Galvanostatic Operation (1.3 A)	122
5.6. Analysis of Voltage Evolution via Linear Extrapolation (Test 2)	125
Chapter 6: Conclusions and Future Work.....	129
6.1 Conclusions	129
6.1.1 Conclusion of the Test One.....	129
6.1.2 Conclusion of the Test Two	129
6.2 General Discussion (both tests)	130
6.3 Future Work.....	130
Appendix: List of Tables.....	132
Bibliography.....	151

List of Figures

Figure 1 Various Hydrogen Production Methods [4].	3
Figure 2 Schematic illustration of PEM water electrolysis [4].	4
Figure 3 left: Current density at 1.8 V, 2.0 V and 2.2 V for different clamping pressures & right: Polarization curves for different clamping pressures for expanded mesh design [31].	15
Figure 4 left: Ohmic resistance from EIS at 2.0 V and polarization curves and mass transport losses from EIS at 2.0 V for expanded mesh design & right: Current density at 1.8 V, 2.0 V and 2.2 V for different clamping pressures for Freudenberg design [31].	16
Figure 5 left: Polarization curves for different clamping pressures & right: ohmic resistance from EIS at 2.0 V and polarization curves and mass transport losses from EIS at 2.0 V for Freudenberg design [31].	17
Figure 6 Example for measured impedance spectra, expanded mesh design [31].	18
Figure 7 Polarization curves of polymer electrolyte membrane water electrolyzer (PEMWE) test cells equipped with different commercially available catalyst-coated membranes (CCMs) determined at an operating temperature of 80°C and a hydraulic medium pressure level[30].	19
Figure 8 Polarization curves for all compressions at 60°C (solid) and 80°C (dashed) [37].	20
Figure 9 Impedance data at 2.30 MPa and different currents as a Nyquist plots. The marks represent the frequencies 10000Hz, 1000 Hz ● (C), 100 Hz ■, 10Hz ♦, and 1 Hz ▲. The respective model fits are shown for both 60 C (solid) and 80 C(dashed) [37].	21
Figure 10 Cell voltages and hydrogen volume fraction, data with different compression values K characterized once; B) data characterized three times, each time with an increased compression value K [34].	22
Figure 11 cell voltages and permeation current densities as a function of current density A) Data with different compression values K characterized once; B) data characterized three times, each time with an increased compression value K [34].	23
Figure 12 Polarization curve of N212 PEMWE cells with an average anodic loading under different compression levels present comparable performances at compressions of 30 and 40 % (a) The cells' HFR shows a decreasing trend for higher compression levels, going from approximately 67.5 $m\Omega cm^2$ to 50.5 $m\Omega cm^2$ at 3 A/cm ² for a compression of 20 % and 60 % respectively (b) [38].	24
Figure 13 a) Polarization curves at the beginning of life for the different coated and uncoated Clamping pressure = 5.1 MPa [39].	25
Figure 14 a) Polarization curves at the beginning of life for the Pt-coated and uncoated PTLs for different clamping pressures [39].	26
Figure 15 left: The polarization curve with PTFE gasket material at 3, 5, 10, 15, and 20 Nm clamping bolt torques & right: The polarization curves of the cell with silicon gasket material at 3, 5, 10, 15, and 20 Nm clamping bolt torques [36].	27
Figure 16 The polarization curves with EPDM gasket material at 3, 5, 10,15 and 20 Nm clamping bolt torques [36].	28

Figure 17 Performance comparisons of PEMEC with titanium thin LGDL and conventional LGDL red colour & EIS comparisons of PEMEC with titanium thin etched LGDL and conventional LGDL (red colour: titanium thin LGDL with thickness of 25 <i>lm</i> , pore size of 100 <i>lm</i> and porosity of 0.30; black colour: titanium felt LGDL with thickness of 350 <i>lm</i> , average pore size of 100 <i>lm</i> and porosity of 0.78) [40].	29
Figure 18 The measured impedance spectra, the estimated polarization and high-frequency resistances under different temperatures (a) Spectra measured at 50 A under 40 ° C, 50 ° C, and 60 ° C & The measured impedance spectra and the estimated polarization and high-frequency resistances under different cathode pressures (a) Spectra measured at 50 A under 1, 3, and 5 bar [42].	31
Figure 19 Increasing performance between 0% and 40% compression polarization curve & Increasing Ohmic and kinetic overpotential between 60% and 80% compression [29].	32
Figure 20 Nyquist plots of electrochemical impedance spectroscopy for the PEMWE single cell with the IrO ₂ /CP anode at various pressures [42].	33
Figure 21 Polarization curves of the PEMWE single cell with IrO ₂ /CP (a) at 90 and 120°C (atmospheric pressure) and (b) at various pressures from 1.0 to 2.5 bar (120°C) [42].	34
Figure 22 Environment park- HYSYLAB.	44
Figure 23 Test Bench.	45
Figure 24 P&I of test bench.	46
Figure 25 Software interface.	48
Figure 26 MEA composed by Nafion 115.	49
Figure 27 left: Titanium mesh GDL – right:H23C6 Carbon paper GDL.	49
Figure 28 MEA composed by Nafion 115.	50
Figure 29 left: Titanium Fiber Felt GDL – right:H23C6 Carbon paper GDL.	50
Figure 30 qCf FC25/125 LC 8bar.	51
Figure 31 Cell housing.	51
Figure 32 Pneumatics on the compressed air cylinder and circuit diagram.	52
Figure 33 contact pressure depending on air pressure supply.	53
Figure 34 Cleaning procedure.	54
Figure 35 Cutting procedure.	54
Figure 36 Housing closed and correctly tightened.	55
Figure 37 left: Housing placed on Test Bench - right: Housing placed on Test Bench with power supply connected.	56
Figure 38 left: Air Pressure Regulator – right: assembled Baltic Fuel Cells to the test bench.	56
Figure 39 Schematic EIS measurement configuration.	58
Figure 40 Input parameters and settings for the EIS instrument [60].	59
Figure 41 Output parameters for the EIS measurement [60].	59
Figure 42 Energy lab-XM System.	61
Figure 43 Schematic configuration of the connection between the cell and the device.	61
Figure 44 Types of experiments in the XM-studio ECS software.	62

Figure 45 Possible step types for Impedance Voltage Control.	62
Figure 46 Possible step types for Impedance Current Control.	63
Figure 47 Cell setup on the software.	63
Figure 48 Hardware requirements and cell setup.	64
Figure 49 scan setup and the impedance setup.	64
Figure 50 Impedance setup.	65
Figure 51 Impedance spectrum with noisy residuals at potentiostatic 0V (Test 1).	67
Figure 52 Impedance spectrum with noisy residuals at potentiostatic 0.5V (Test 1).	67
Figure 53 Impedance spectrum with moderate noisy residuals at potentiostatic 1V (Test 1).	67
Figure 54 Impedance spectrum with moderate noisy residuals at potentiostatic 1.3V (Test 1).	68
Figure 55 Impedance spectrum with acceptable residuals at potentiostatic 1.5V (Test 1).	68
Figure 56 Impedance spectrum with acceptable residuals at potentiostatic 1.3V (Test 2).	68
Figure 57 Impedance spectrum with acceptable residuals at potentiostatic 1.5V (Test 2).	69
Figure 58 Impedance spectrum with acceptable residuals at Galvanostatic 1.3A (Test 2).	69
Figure 59 Impedance spectrum with acceptable residuals at Galvanostatic 1.5A (Test 2).	69
Figure 60 frequency range.	70
Figure 61 CPE and resistance.	72
Figure 62 CPE & ohmic resistance.	72
Figure 63 charge transfer resistance and CPE.	72
Figure 64 Rct, Ran and Rcath.	73
Figure 65 Warburg elements in series with other elements.	73
Figure 66 fitting of the EIS graphs.	74
Figure 67 Example of the equivalent circuit.	75
Figure 68 CPE set.	75
Figure 69 Fitting options.	76
Figure 70 alternative ECM.	76
Figure 71 Initial values with Pre fitting.	77
Figure 72 Results of the Resistances with First Scenario.	78
Figure 73 Results of the Resistances with Second Scenario.	78
Figure 74 Results of the Resistances with Third Scenario.	79
Figure 75 Results of the Resistances with Python Code Scenario.	80
Figure 76 Fitting result.	80
Figure 77 Voltage behavior during the time (Test 1).	82
Figure 78 Cell Temperature behavior during the time (Test 1).	82
Figure 79 Degradation behavior at Compression 3.4 bar during the time (Test 1).	83
Figure 80 Degradation behavior at Compression 4.4 bar during the time (Test 1).	84
Figure 81 Degradation behavior at Compression 5 bar during the time (Test 1).	85
Figure 82 Degradation behavior at Compression 4.4 bar descent during the time (Test 1).	85
Figure 83 Degradation behavior at Compression 3.4 bar descent during the time (Test 1).	86
Figure 84 Degradation behavior at Compression 2.4 bar descent during the time (Test 1).	87

Figure 85 Nyquist plot Potentiostatic 60°C Different Pressures at 0 V (Test 1).	89
Figure 86 Trend of the ohmic resistance in the HF region at 60°C & 0 Volt (Test 1)	90
Figure 87 Nyquist plot Potentiostatic 60°C Different Pressures at 0.5 V (Test 1).	91
Figure 88 Trend of the ohmic resistance in the HF region at 60°C & 0.5 Volt (Test 1).	92
Figure 89 Nyquist plot Potentiostatic 60°C Different Pressures at 1 V (Test 1).	93
Figure 90 Trend of the ohmic resistance in the HF region at 60°C & 1 Volt (Test 1).	93
Figure 91 Nyquist plot Potentiostatic 60°C Different Compressions at 1.3 V (Test 1).	94
Figure 92 Trend of the ohmic resistance & the charge transfer resistance in the HF region at 60°C & 1.3 Volt (Test 1).	95
Figure 93 Trend of the capacitance in the HF region at 60°C & 1.3 Volt (Test 1).	95
Figure 94 Nyquist plot Potentiostatic 60°C Different Pressures at 1.5 V (Test 1).	96
Figure 95 Trend of the ohmic resistance, the charge transfer resistance & mass transport resistance at 60°C & 1.5 Volt (Test 1).	97
Figure 96 Trend of the capacitance in the HF region at 60°C & 1.5 Volt (Test 1).	98
Figure 97 Trend of the capacitance in the LF region at 60°C & 1.5 Volt (Test 1).	98
Figure 98 Trend of the total resistance at 60°C & 1.5 Volt (Test 1).	99
Figure 99 Trend of the ohmic resistance at different compressions & 60°C from 0 Volt to 1.5 Volt (Test 1).	100
Figure 100 Trend of the voltage at different compressions during time (Test 2).	101
Figure 101 Net voltage degradation after removing previous compression- induced degradation.	102
Figure 102 Raw & corrected voltage degradation at different compression levels.	102
Figure 103 Stabilization procedure on the cell.	103
Figure 104 Polarization curves in constant cell temperature 60°C & before different compression levels.	104
Figure 105 Polarization curves in constant cell temperature 60°C & after different compression levels.	105
Figure 106 Degradation behavior at Compression 2.4 bar during the time (Test 2).	106
Figure 107 Degradation behavior at Compression 3.4 bar during the time (Test 2).	107
Figure 108 Degradation behavior at Compression 4.4 bar during the time (Test 2).	107
Figure 109 Degradation behavior at Compression 4.9 bar during the time (Test 2).	108
Figure 110 Degradation behavior at Compression 4.4 bar descent during the time (Test 2).	108
Figure 111 Nyquist plot Potentiostatic 60°C Different Pressures at 1.5 V (Test 2).	110
Figure 112 Trend of the ohmic resistance, the charge transfer resistance & mass transport resistance at 60°C & 1.5 Volt (Test 2).	111
Figure 113 Trend of the capacitance in the HF region at 60°C & 1.5 Volt (Test 2).	111
Figure 114 Trend of the capacitance in the LF region at 60°C & 1.5 Volt (Test 2).	112
Figure 115 Trend of the total resistance at 60°C & 1.5 Volt (Test 2).	112
Figure 116 Trend of the ohmic resistance, the charge transfer resistance & mass transport resistance EIS before & after different compressions at 60°C & 1.5 Volt (Test 2).	113

Figure 117 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.5 Volt (Test 2).	114
Figure 118 Trend of the capacitance in the LF region EIS before & after different compressions at 60°C & 1.5 Volt (Test 2).	114
Figure 119 Trend of the total resistance before & after different compressions at 60°C & 1.5 Volt (Test 2).	115
Figure 120 Nyquist plot Potentiostatic 60°C Different Pressures at 1.3 V (Test 2).	116
Figure 121 Trend of the ohmic resistance & the charge transfer resistance at 60°C & 1.3 Volt (Test 2).	117
Figure 122 Trend of the capacitance in the HF region at 60°C & 1.3 Volt (Test 2).	117
Figure 123 Trend of the ohmic resistance & the charge transfer resistance for EIS before & after different compressions at 60°C & 1.3 Volt (Test 2).	118
Figure 124 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.3 Volt (Test 2).	118
Figure 125 Nyquist plot Galvanostatic 60°C Different Pressures at 1.5 A (Test 2).	119
Figure 126 Trend of the ohmic resistance & the charge transfer resistance at 60°C & 1.5 A (Test 2).	120
Figure 127 Figure 123 Trend of the capacitance in the HF region at 60°C & 1.5 A (Test 2).	120
Figure 128 Trend of the ohmic resistance & the charge transfer resistance for EIS before & after different compressions at 60°C & 1.5 A (Test 2).	121
Figure 129 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.5 A (Test 2).	121
Figure 130 Nyquist plot Galvanostatic 60°C Different Pressures at 1.3 A (Test 2).	122
Figure 131 Trend of the ohmic resistance & the charge transfer resistance at 60°C & 1.3 A (Test 2).	123
Figure 132 Figure 123 Trend of the capacitance in the HF region at 60°C & 1.3 A (Test 2).	123
Figure 133 Trend of the ohmic resistance & the charge transfer resistance for EIS before & after different compressions at 60°C & 1.3 A (Test 2).	124
Figure 134 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.3 A (Test 2).	124
Figure 135 Linear extrapolation to 1000h at compression 2.4 bar (Test 2).	126
Figure 136 Linear extrapolation to 1000h at compression 3.4 bar (Test 2).	126
Figure 137 Linear extrapolation to 1000h at compression 4.4 bar (Test 2).	127
Figure 138 Linear extrapolation to 1000h at compression 4.9 bar (Test 2).	127
Figure 139 Linear extrapolation to 1000h at compression 4.4 bar descent (Test 2).	128

Abstract

Proton Exchange Membrane (PEM) water electrolysis has emerged as a key technology for high-purity green hydrogen production. Despite significant technological progress, the mechanical assembly conditions especially the applied compression on the cell components remain one of the most important yet insufficiently characterized parameters affecting performance, durability, and electrochemical behaviour. This thesis provides an experimental investigation into the effect of mechanical compression on different PEM electrolyzer cell configurations, with a specific focus on key performance indicators (KPIs), Electrochemical Impedance Spectroscopy (EIS), and degradation behaviour. PEM cells equipped with different membrane electrode assemblies (MEAs) and gas diffusion layers (GDLs) were assembled and tested under controlled compression levels. Two experimental campaigns were conducted using two sets of cells, each operated at varied compression levels to evaluate the influence of compression on performance, degradation, and the evolution of ohmic, charge-transfer, and mass-transport resistances through polarization curves, EIS measurements, and long-term degradation tests.

The results show that mechanical compression has a decisive impact on PEM electrolyzer performance, impedance behaviour, and durability. Test 1, performed across the contact pressure levels from 2.4 to 5 bar, demonstrated that moderate compression around 3.4 bar ensured the most stable operation, yielding the lowest degradation rates and the lowest resistances. Both lower and higher compression levels led to increased resistances and faster degradation due to insufficient mechanical contact or excessive mechanical stress. Test 2 that has been done with a different set of CCM and GDLs, confirmed these trends across a wider compression range and identified that by increasing the compression the performance will increase by decreasing the degradation rate the ohmic resistance and the charge transfer resistance. As the compression 4.9 bar exhibited the lowest value in degradation rate, on the other hand, at compression 4.4 bar, where the cell exhibited the lowest polarization-curve. Overall, both tests demonstrate that optimal compression enhances interfacial contact and reduces resistive losses, whereas insufficient or excessive compression accelerates degradation. This work provides a detailed experimental characterization of compression effects in PEM electrolysis and offers practical guidelines for selecting suitable compression levels to improve PEMWE performance and durability.

Chapter 1: Introduction

1.1 Global Energy Context and Role of Hydrogen

The global energy is undergoing a deep transformation impacts of climate change and the need to transition from fossil fuels and currently account for 73% of global energy-related emissions. For global decarbonization, hydrogen is important. hydrogen acting as a flexible energy carrier to reserve renewable energy and fuel. As long as 96% of current production depends on fossil fuels, green hydrogen produced through electrolysis is vital for net zero emissions, although the needs of infrastructure and high costs remain [1].

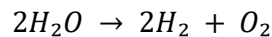
1.2 Hydrogen Production Technologies

The technologies for hydrogen production focus on extracting hydrogen from hydrocarbons, water, or biomass. There are three methods, which are steam methane reforming, gasification, and finally electrolysis that is water splitting. Production of the hydrogen through electrolysis, such as PEM, Alkaline, SOEC is scaling up to replace the conventional methods based on fossils. The production of hydrogen is a foundation for hydrogen economy, with technologies developing to meet the two important demands of efficiency and sustainability.

However, the global try for decarbonization focuses on renewable-based methods, especially electrolysis. Electrolysis splits water into hydrogen and oxygen using electricity. This section shows advanced hydrogen production methods, and it is emphasized on their mechanisms and efficiencies to green hydrogen production. Meanwhile, proton exchange membrane (PEM) electrolysis stands out for its efficiency, compactness, and dynamic response [2].

1.2.1 Electrolysis Technologies (Advanced Methods)

For splitting water into hydrogen and oxygen electrolysis uses electricity as the following formula:



According to this formula, the efficiency of electrolysis depends on the cell voltage. This value close to the theoretical equal to 1.23 V, but practical systems require 1.8-2.2 V due to overpotentials. Besides, the efficiency is calculated as:

$$\eta = (HHV \text{ of } H_2 \cdot \text{mass of } H_2) / \text{Electrical energy input}$$

The higher heating value (HHV), in this formula shows hydrogen which is equal to 141.8 MJ/kg. Electrolysis methods vary in their electrolytes, and operating conditions for renewable integration. There are four main types available in this case: alkaline, proton exchange membrane (PEM), solid oxide electrolysis cells (SOEC), and anion exchange membrane (AEM) [3].

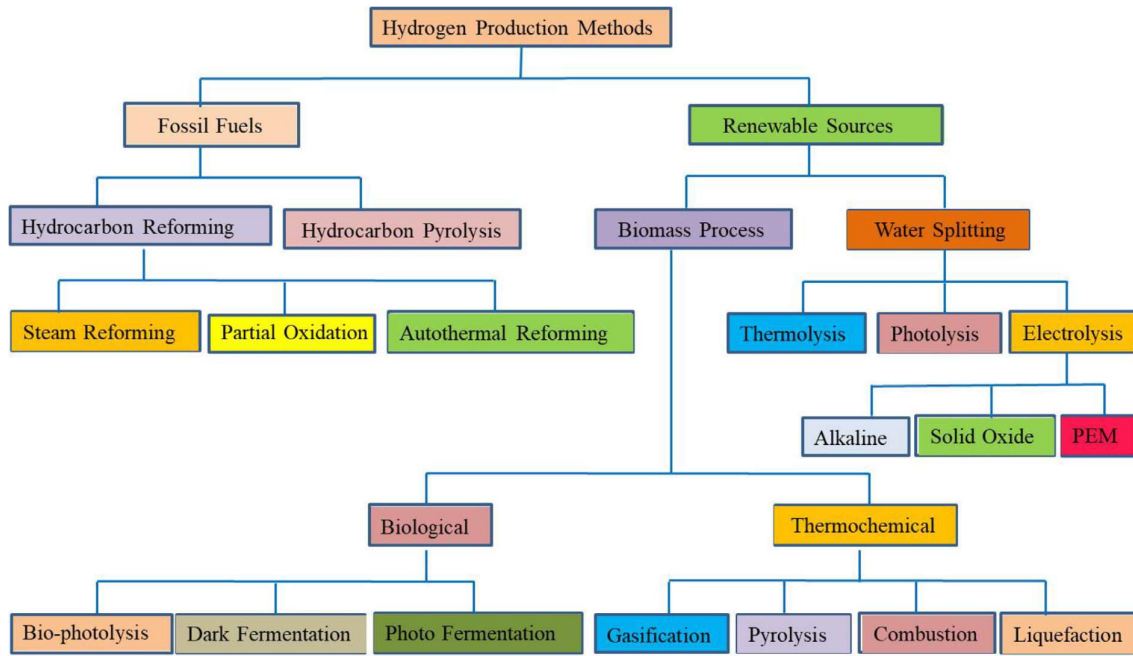
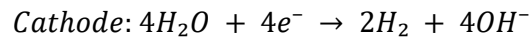
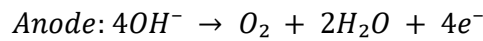


Figure 1 Various Hydrogen Production Methods [4].

1.2.2 Alkaline Electrolysis

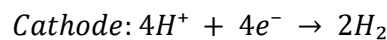
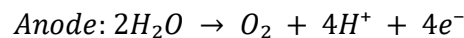
Alkaline electrolysis is one of the oldest and most mature technologies, dating back to the 19th century. This is widely used in industrial applications. It employs a liquid electrolyte, typically 20-30% potassium hydroxide (KOH), to conduct hydroxide ions (OH^-). Between electrodes separated by a porous diaphragm. There are often asbestos or polymer based. They operate at 60-80°C and 1-30 bar. The alkaline systems can achieve current densities of 0.2-0.5 A/cm² and efficiencies of 50-70% LHV (Grigoriev et al., 2020). The oxygen evolution reaction (OER) at the anode and hydrogen evolution reaction (HER) at the cathode are equal to:



Alkaline slow response to load changes like minutes and lower current density make it less suitable for coupling with variable renewable sources like wind or solar [3].

1.2.3 Proton Exchange Membrane (PEM) Electrolysis

PEM electrolysis uses a solid polymer membrane like Nafion to conduct protons (H^+), making it possible to operate at 50-80°C with high current densities at around 1-2 A/cm². The reactions are as below:



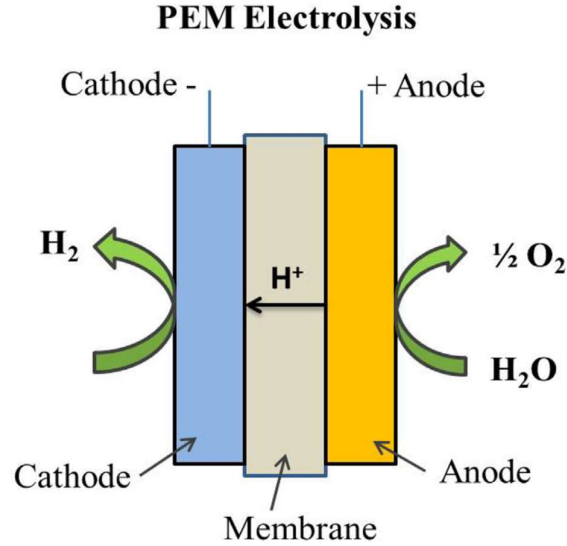


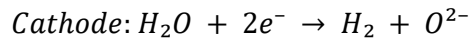
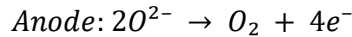
Figure 2 Schematic illustration of PEM water electrolysis [4].

The efficiencies of the PEM arrived at 60-80% LHV. PEM is Better than alkaline in dynamic applications due to its fast startup which is in seconds [5]. The compact design of PEM requiring 30-50% less space than alkaline systems and makes it ideal for decentralized production, such as integration with offshore wind farms [6], [7].

The PEM is superior in efficiency and dynamic response. The ability of the PEM is to operate at high current density reduces the number of cells needed for a given output, lowering capital costs [8]. Moreover, the fast response of the PEM to variation of the power 0-100% load in seconds, aligns with the nature of renewables, enhancing grid stability in systems [9]. This research makes PEM the focus of this study, particularly for optimizing component interactions like gas diffusion layers (GDLs) to improve two-phase transport and performance.

1.2.4 Solid Oxide Electrolysis Cells (SOEC)

SOEC operates at high temperatures around 600-900°C, using a solid ceramic electrolyte like zirconia to conduct oxygen ions (O^{2-}). The reactions are:



By leveraging thermal energy, SOEC achieves efficiencies above 90% LHV, the highest among electrolysis methods [2]. However, high temperatures cause material degradation, particularly in ceramic electrolytes and electrodes. SOEC is promising for industrial settings with waste heat sources, such as steel plants, but compared to PEM, the complexity of solid oxide electrolysis cell makes it less suitable for dynamic renewable integration [3].

1.2.5 Anion Exchange Membrane (AEM) Electrolysis

AEM electrolysis is an important technology which uses a hydroxide-conducting polymer membrane, combining with the low-cost materials of alkaline with PEM's solid electrolyte

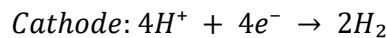
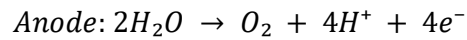
design. AEM achieves efficiencies of 50-70% LHV, Operating at 50-60°C and current densities of 0.5-1 A/cm² [3]. The membrane stability and degradation under alkaline conditions remain challenges [2].

1.3 PEM Electrolyzers Structure and Components

Proton exchange membrane (PEM) electrolyzers are one of the most advanced technologies for green hydrogen production. It is offering high efficiency, rapid dynamic response, and strong compatibility with renewable energy systems. Their performance arises from a precisely engineered assembly of components that work together to facilitate water splitting into hydrogen and oxygen. The membrane electrode assembly (MEA) is central to this system which is supported by porous transport layers (PTLs), gas diffusion layers (GDLs), and bipolar plates. All compressed within a stack structure to ensure mechanical stability and electrical continuity. These components collectively determine the electrolyzer's efficiency, durability, and suitability for large-scale deployment.

1.3.1 PEM Electrolyzer Structure Overview

A PEM electrolyzer consists of a single cell or sometimes stack of cells. Each containing an MEA sandwiched between PTLs, GDLs, and bipolar plates, all compressed within end plates to ensure electrical and mechanical stability. The core functional unit is the MEA, where water is oxidized at the anode to produce oxygen, protons, and electrons, and protons are reduced at the cathode to form hydrogen. The reactions are:



The PTLs and GDLs facilitate the transport of reactants like water and products hydrogen and oxygen gases. On the other hand, bipolar plates manage gas and water flow through integrated channels. A typical stack might contain 10-100 cells. They are delivering outputs from kilowatts to megawatts [6]. The efficiency of the system typically is around 60-80% based on the lower heating value (LHV) of hydrogen, depends on minimizing losses in mass transport, electrical resistance, and electrochemical overpotentials, which are influenced by component design [10].

1.3.2 Proton Exchange Membrane

The proton exchange membrane that conducts protons while separating hydrogen and oxygen gases [10]. The structure of the Nafion consists of a polytetrafluoroethylene (PTFE) backbone with sulfonic acid side chains, which facilitate proton transport via a hydrated network. Conductivity depends on hydration. Conductivity requiring water management to maintain performance, typically at 50-80°C and 1-30 bar. However, the performance of the Nafion drops at low humidity, prompting exploration of alternatives like Aquivion, a Perfluoro sulfonic acid (PFSA) membranes with shorter side chains that enhance conductivity under drier conditions [11].

Membrane thickness typically is around 50-180 μm and it balances conductivity and mechanical strength. Thinner membranes reduce ohmic losses but risk gas crossover, where hydrogen and oxygen mix, lowering efficiency and posing safety concerns [10]. The membrane's role is to ensuring protons flow efficiently while keeping gases apart, a delicate balance that influences overall system performance.

1.3.3 Catalyst Layers

The catalyst layers, coated onto both sides of the membrane and they drive the electrochemical reactions. The oxygen evolution reaction (OER) is catalysed by iridium oxide (IrO_2), at the anode and its stability in acidic conditions and low overpotential (0.3 V at 1 A/cm²). Where the hydrogen evolution reaction (HER) occurs in the cathode. It typically uses platinum (Pt) nanoparticles supported on carbon, benefiting from its high activity and low overpotential 0.05 V. Catalyst loading ranges are from 0.5-3 mg/cm² for IrO_2 and 0.2-0.5 mg/cm² [11].

The thickness of the catalyst layers typically 5-20 μm thick, are applied with spraying techniques to ensure uniform coverage. Their performance depends on surface area, porosity, which affect reaction kinetics and electron transfer. Degradation, such as iridium dissolution under high potentials or carbon support corrosion at the cathode, reduces efficiency over time. A challenge of this thesis indirectly addresses by optimizing adjacent components like the GDL. The catalysts are like the engines of the electrolyzer, driving reactions but requiring costly materials, a hurdle for scaling to gigawatt levels [10].

1.3.4 Porous Transport Layer (PTL)

The porous transport layer (PTL), typically titanium-based, facilitates water delivery to the anode catalyst and oxygen removal. Titanium's corrosion resistance in the acidic PEM environment is critical. But its high cost like 10-20% of stack cost has spurred exploration of carbon-based PTLs [12]. PTLs are often sintered titanium powders or meshes with porosity of 30-50% and pore sizes of 10-100 μm , optimizing two-phase flow the liquid water in, gas out. The PTL's structure influences mass transport losses, particularly at high current densities which is higher than 1 A/cm², where gas bubble accumulation can block water access, increasing overpotential [10].

Surface treatments, like titanium coatings enhance PTL performance by improving wettability and gas release. The PTL's role in this thesis is significant, as its interface with the GDL affects contact resistance and transport efficiency, areas targeted for optimization. Think of the PTL as a highway system, directing traffic of the water and gas to from the reaction site, where congestion (bubbles) can slow the process [12].

1.3.5 Gas Diffusion Layer (GDL)

Titanium or carbon felt which are the gas diffusion layer (GDL), ensures uniform gas distribution and electrical contact between the catalyst layer and bipolar plate. In PEM electrolyser, the GDL on the cathode side, removes hydrogen gas. On the other hand, on the anode side, it aids oxygen release. Its porosity of 50-70% and thickness around 200-500 μm are critical for balancing gas transport and electrical conductivity. PTFE, which is a

hydrophobic coating, prevent water flooding, while high conductivity minimizes ohmic losses. The GDL's properties directly influence two-phase transport and contact resistance, making it an important point for this thesis's experimental investigations into performance optimization [10].

The GDL's role is particularly relevant at high current densities, where gas accumulation can increase mass transport losses. This is a challenge exacerbated by poor interface design with the PTL or catalyst layer. Recent studies suggest that tailored GDL porosity and thickness can reduce these losses by up to 20% [12].

1.3.6 Bipolar Plates

The material of the bipolar plates is typically titanium to resist corrosion. They manage gas and water flow through etched channels, connecting cells in a cell. They account for 20-30% of stack weight and cost, prompting research into carbon-based alternatives. On the other hand, they offer lower costs but face durability challenges in acidic conditions [12]. Flow field designs, such as parallel or serpentine channels, optimize reactant distribution and gas removal, impacting pressure drop and efficiency [10].

Table 1: Key Components of PEM Electrolyzers

Component	Material	Function	Key Properties	Challenges	Source
Membrane	Nafion, Aquivion (PFSA)	Proton conduction, gas separation	Conductivity (~0.1 S/cm), 50-180 μm	Gas crossover, degradation	[10]
Catalyst Layers	IrO_2 (anode), Pt/C (cathode)	OER, HER catalysis	Loading 0.2-3 mg/cm^2 , high activity	Cost, dissolution	[12]
PTL	Titanium (sintered, mesh)	Water/gas transport, electron conduction	Porosity 30-50%, 10-100 μm pores	Cost, bubble accumulation	[11]
GDL	Titanium/carbon felt	Gas distribution, electrical contact	Porosity 50-70%, 200-500 μm thick	Transport losses, contact resistance	[2]
Bipolar Plates	Titanium, carbon (emerging)	Flow management, cell connection	Corrosion resistance, channel design	Cost, durability (carbon)	[11]

1.4 Key Challenges in PEM Electrolysis

1.4.1 Challenges in Durability, Cost and Transport for PEM Electrolyzers

Although cost-reduction strategies such as lowering catalyst loading or adopting carbon-based components are being explored, these often compromise durability or stability [11]. Durability remains another problem as current PEM systems typically last 20,000 to 40,000 hours well below the 50,000 to 80,000 hours required for commercial viability. It is due to degradation mechanisms including IrO_2 dissolution at high potentials, Pt agglomeration or detachment, and membrane chemical breakdown leading to fluoride release and increased gas crossover [10],

[11], [13]. These issues are intensified under high current densities and fluctuating renewable inputs, though coated stainless-steel components show emerging potential as cost-effective alternatives [13]. Additionally, water and gas transport limitations significantly impact performance, as oxygen and hydrogen bubble accumulation within PTLs around 30–50% porosity and GDLs around 50 to 70% porosity obstructs flow pathways, increasing mass transport resistance and raising cell voltage by 0.1 to 0.3 V at 2 A/cm², as described by Darcy's law [14]. Optimizing pore size, hydrophobicity, and two-phase transport properties particularly within the GDL, can reduce these losses by up to 20%, directly motivating this thesis's focus on GDL design to mitigate transport bottlenecks and improve overall PEM electrolyzer efficiency [10], [12].

1.5 Research Gap and Thesis Objectives

Despite advancements in efficiency, compactness, a significant challenge remained, particularly in cost, durability, and two-phase transport. The literature provides a robust foundation for understanding PEM components and performance, critical gaps. Besides, it tries to address dynamic operation and component interactions, especially at the gas diffusion layer (GDL) and porous transport layer (PTL) interface. This section identifies these research gaps, outlines the objectives of this thesis to solve them, and provides an overview of the thesis structure, along with the experimental focus on effect of compression on the cell and properties to enhance PEM electrolyzer performance.

1.5.1 Research Gap

The PEM electrolysis literature, discovers underexplored areas, including the effect of the compression on the cell performance [15]. In this case for example, the impact of the compression on cell performance, such as polarization curves and electrochemical impedance spectroscopy. On the other hand, its influence on ohmic resistances, charge transfer resistances and mass transport losses at high current density (>1 A/cm²) [14], [15].

Another significant gap can be worth to be considered is the degradation mechanisms. The detail catalyst and membrane degradation, the GDL's role in long-term performance is less understood. For example, compression-induced porosity reduction or hydrophobicity loss. It can worsen transport limitations over time. Few studies quantify these effects under realistic operating conditions [16]. Contact resistance at the GDL-PTL interface, which contributes 50–100 m Ω ·cm² to ohmic losses, was also reviewed, particularly in carbon-based versus titanium-based GDLs [12]. These gaps are critical, as optimizing MEA properties could improve efficiency and durability, and try to support the scalability of PEM electrolysis [8].

In top of that, there are some gaps in integrated studies combining electrochemical impedance spectroscopy (EIS) and polarization curves, offer diagnostic insights, their application to the cell performance is limited in the literature [15]. This thesis looks to mitigate these gaps by experimentally investigating the effects of compression on the cell performance to find a pathway to enhance PEM electrolyzer performance and align with global decarbonization goals.

1.5.2 Thesis Objectives

To meet the identified research gaps, this thesis pursues the following objectives:

1. **Characterize the Effects of the Compression on the Cell Performance:** The thesis Investigates the impact of the compression on the cell performance, using EIS, durability and polarization curves to quantify mass transport and contact resistance losses. The goal is to cover the gap [15].
2. **Cell Compression for Two Sets of Cell Assembly:** Investigated and tested two different MEAs and GDLs at different compression levels in order to see the performance of the cell.
3. **Assess Long-Term Durability:** To analyze cell degradation mechanisms, under different compression and analyze performance metrics to enhance lifespan [16].
4. **Analyze Compression Effect on PEMWE:** By analyzing key performance indicators (KPIs), and EIS analysis followed by degradation analysis (extrapolation to 1000h) of PEMWE under different compression perspectives about compression effect on the cell performance.

1.5.3 Thesis Structure

The structure of the thesis provides an opportunity to address these objectives, based on the foundation of the following chapters:

- **Chapter 2: Literature Review** – This chapter expands the research gaps mentioned here, synthesizing findings from [15] and [16] to use their contents in cell compression performance within PEM electrolysis advancements.
- **Chapter 3: Experimental**– In this chapter, details of the experimental setup, including MEAs, testing protocols (EIS, polarization curves, durability), and performance metrics (cell voltage) has been explored.
- **Chapter 4: ECM & EIS Analysis** – This chapter explained the basics of ECM and procedure of the fitting scenarios and the equivalent circuits which used in fitting analysis with comparing some results and decision of the final fitting scenario.
- **Chapter 5: Results and Discussion** – This chapter presents findings from cell compression performance experiments. Furthermore, analyzing impacts on, polarization curves, contact resistances, and durability, along with comparisons to baseline performance.
- **Chapter 6: Conclusions and Future Work** – This chapter provides Summarizes contributions and evaluates objective achievement. To proposes future research, such as integrating cell performance.

1.5.4 Forward Look and Reflection

It is evident that PEM electrolysis, like a complex machine, requires fine-tuning at the component level to unlock its full potential. The focus on cell compression performance in this thesis bridges theory and practice, addressing critical barriers to efficiency and scalability. This work aims to contribute to the broader goal of making green hydrogen a cornerstone of the energy transition by tackling the effects of the compression on the cell performance and interface challenges. The next chapters build on this foundation, providing the experimental and analytical framework to achieve these objectives.

Chapter 2. Literature Review

2.1 Role of Compression and GDLs in PEM Electrolyzers

In proton exchange membrane (PEM) electrolyzers, there are critical components; one of them is the MEA, gas diffusion layer and the other is the porous transport layer (PTL). Both are acting as an important interface between the catalyst layer and bipolar plates. These layers ensure efficient transport of reactants, which is water, and products, which are hydrogen and oxygen in the gas phase. On the other hand, these layers conduct electricity and provide mechanical support, which directly influences the efficiency of the system, durability, and scalability. As demonstrated in Chapter 1, the GDL and PTL manage the dynamics of two-phase flows that are complex: liquid water inflow and gas outflow. These flows are essential for reducing losses related to the mass transport, particularly at high current densities ($> 1 \text{ A/cm}^2$) [17]. During the cell assembly the compression applied further modulates their efficiency by changing properties like electrical, thermal, and transport. Compression can either increase or decrease the performance of the electrolyzer. This section considers the different aspects and the roles of the GDLs and PTLs, as well as the impact of the compression, extracting from the recent literature to provide their significance in PEM electrolysis and this thesis's focus on the optimization of the cell performance.

2.1.1 Functions of GDL and PTL

The GDL, usually made from carbon or titanium. GDL carries out three primary functions, such as simplifying gas diffusion, conducting electricity, and providing structural support. On the cathode side, the GDL ensures impressive removal of hydrogen gas to prevent accumulation that could block the sites of the catalysts for the hydrogen evolution reaction (HER), reducing the active area. On the anode side, it helps in releasing oxygen gas, keeping access for water to reach the catalyst layer for the oxygen evolution reaction (OER) [18]. The porosity (50 – 70%) and pore size (50 – 200 μm) of the gas diffusion layer are critical for permeability of the gas. Hydrophobic coatings, such as polytetrafluoroethylene (PTFE), prevent water flooding that could prohibit gas transport [17]. For instance, a study on carbon-based gas diffusion layers showed that the optimization of the porosity can increase the utilization of the catalyst by up to 55%, in comparison with 45% for coarse titanium mesh GDLs. Furthermore, titanium mesh GDLs suffer from rough contact with catalyst layer. This highlights the role of the GDLs in guaranteeing uniform distribution of the reactants, an important factor in achieving high rates of efficiency and hydrogen production [19].

The titanium-based PTL is primarily responsible for water delivery to the anode catalyst and removal of the oxygen due to its corrosion resistance in the acidic PEM environment. Titanium-based PTL also conducts electrons between the catalyst layer and bipolar plate. It requires high electrical conductivity and mechanical stability [20]. PTLs are optimized for two-phase flow and usually have a pore size of 10 – 100 μm and a porosity of 30 – 50%. Ultra-thin titanium PTLs (0.14 – 0.25 mm), which are a recent development of PTLs, are made by powder rolling, reducing material costs, on the other hand Ultra-thin titanium PTLs maintain

performance and achieve current densities up to 2 A/cm^2 with a negligible increase of voltage [21]. Another innovation like titanium hydride that is integrated into PTLs by reducing ohmic losses through suppressing the formation of the oxide layer will further increase the energy efficiency [22]. The design of the PTLs is an important matter for managing interactions of water and gas, because an inappropriate pore structure can cause bubble entrapment and increase mass transport overpotentials by $0.1 - 0.3 \text{ V}$ [21].

Regarding thermal management, both GDLs and PTLs contribute to dissipating heat generated during electrolysis (at 2 A/cm^2 and energy input up to $20 - 30\%$) to prevent hotspots that cause degradation of the components [23]. The interactions between these layers and the catalyst layer are critical; for example, new pore-graded GDLs, which are produced through spraying plasma in vacuum conditions, improve water management and contact of the catalyst, also increasing performance by $10 - 15\%$ in comparison with the conventional designs [24]. These matters show the GDL and PTL as an important part of PEM electrolysis and directly affect the ability of the system to operate efficiently under dynamic conditions.

2.1.2 Compression effect on the Electrical, Thermal, and Transport properties

During cell assembly to ensure mechanical integrity and electrical contact, a compression will be applied. This compression deeply affects the GDL's and PTL's properties, like electrical, thermal, and transport. A compression ranging from 1 to 3 MPa usually reduces interfacial contact resistance but on the other hand, it can change the structures of the pores, creating a balance between conductivity and transport efficiency [25]. For optimization of the PEM electrolyzer, it is essential to understand these effects.

2.1.2.1 Electrical Properties

Compression by increasing contact points between the GDL, PTL and catalyst layer or bipolar plates will increase electrical conductivity while reducing ohmic resistance. The formula below shows the contact resistance:

$$R_{\text{contact}} = \rho / A$$

Where (ρ) is the specific contact resistivity and (A) is the effective contact area. A compression around $1 - 2 \text{ MPa}$, which is a moderate compression, can decrease contact resistance by $50 - 100 \text{ m}\Omega \cdot \text{cm}^2$; also, at 1 A/cm^2 , it lowers cell voltage by $20 - 50 \text{ mV}$ [20]. The studies show that for the carbon-based GDLs, compression to 1.5 MPa increases electrical conductivity by 30% , but extreme compression ($> 3 \text{ MPa}$) can destroy carbon fibers, on the other hand increasing resistance because of the structural damage [26]. The hard structure of titanium PTLs benefits from compression up to 2 MPa , but additional increases in the compression cause increasing yield diminishing returns due to the pore closure [27]. According to the advanced coatings like $\text{Au} - \text{TiO}_2/\text{Ti}$ hybrids, maintaining low contact resistance under compression by preventing the growth of the oxide layer causes improved stability [28].

2.1.2.2 Thermal Properties

Compression also affects thermal conductivity. It is critical for reducing the heat generated during the process of the electrolysis. The thermal conductivity (k_{therm}) of GDLs and PTLs depends on their material and porosity. The thermal conductivity can be modelled as:

$$k_{therm} = k_{solid} \cdot (1 - \varphi) + k_{fluid} \cdot \varphi$$

where (φ) is porosity, and (k_{solid}) and (k_{fluid}) are the conductivities of the solid material and pore-filling fluid for example, water or gas. Compression increases k_{therm} by increasing the solid contact and reduces the porosity. Compression to 2 MPa for carbon-based GDLs can increase thermal conductivity from 0.5 W/m · K to 1.2 W/m · K; it helps heat dissipation [29]. However, excessive compression increases the risk of breakage in carbon-based GDLs. It creates hotspots that precipitate the degradation of the catalyst [26]. Titanium PTLs are less sensitive with a higher range of conductivity (~2 W/m · K) but under the high compression still have the problem of the reduced porosity and impacting thermal uniformity [23].

2.1.2.3 Transport Properties

Compression has the most significant impact on transport properties. Compression affects two-phase flow (water in and gas out) and it changes porosity and permeability. The Kozeny-Carman equation determines (k) which is the permeability:

$$k = (\varphi^3 d^2) / (72 (1 - \varphi)^2)$$

where (φ) is porosity and (d) is particle diameter. Compression reduces (φ), decreasing permeability and increasing pressure drop, which prevents removal of the gas and delivery of water [21]. For example, compressing a GDL with porosity from 70% to 50% can reduce permeability by an order of magnitude while increasing by 0.2 V at 2 A/cm² the mass transport overpotential [25]. Carbon-based GDLs are exclusively sensible because compression can collapse pores, and this leads to water flooding that blocks the pathway of the gas [18]. Titanium PTLs with more tough structures can better maintain the transport under a moderate compression, but the designs of the pore-graded are needed to optimize the distribution of the flow [24]. Hydrophobic coatings like PTFE on GDL or IrO₂/TiO₂ on PTLs, by increasing gas release, mitigate flooding; on the other hand, they improve performance by 5 – 10% under compression [27].

2.1.3 Trade-offs and Innovations

In the literature the exchange between the advantages of the compression (lower resistance) and disadvantages (reduced permeability) is an iterative subject. Moderate compression (1 – 2 MPa), with the losses due to the transport, will balance electrical gains and achieve the optimal performance, on the other hand, high compression (> 3 MPa) usually degrades

efficiency because of the pore closure [20]. Innovations like pore-graded GDLs are addressed by preserving transport under compression because they maintain larger pores near the catalyst layer and smaller pores near the bipolar plate [24]. Hybrid coatings like $Au - TiO_2/Ti$ similarly increase the stability of the PTL and reduce the contact resistance. Furthermore, they are maintaining porosity [28]. These improvements show the potential for the appropriate design of the GDL and PTL. It is a motivation for this thesis to focus on the experimental optimization of the GDL properties under dynamic conditions.

2.1.4 Relevance to Thesis

The roles of the GDL and PTL's in managing two-phase flows and the electrical conductivity and thermal dissipation are critical to the performance of the PEM electrolyzer. Also, compression action as a variable, which is important to consider. The literature addresses the need for design to be optimized and balance these properties. Especially according to the GDLs, porosity and hydrophobicity directly affect the contact resistance and mass transport [29]. Based on these results, the GDL's modifications to improve performance under compression conditions were experimentally studied.

According to the complexity of GDL and PTL functions, they are a system of the electrolyzer that ensures seamless flow of reactants and products. On the other hand, they are maintaining structural integrity. Compression is an important matter that must be carefully managed to prevent blocking this system.

2.2 Experimental Studies on Compression

Cell compression is a key assembly parameter that directly influences the electrochemical performance of PEM electrolyzers. Experimental studies have shown that variations in cell compression effects on the cell's polarization behavior, electrochemical impedance response, and the evolution of resistive and capacitive elements during operation. These effects arise from changes in interfacial contact quality, current distribution, and the mechanical and electrical coupling within porous transport layers and catalyst layers.

A wide range of experimental approaches has been used to quantify these performance impacts. Researchers commonly perform polarization curve measurements and EIS under controlled compression levels to evaluate changes in ohmic resistance, charge-transfer resistance, double-layer capacitance, and mass-transport-related features [30], [31], [32]. Long-term and cyclic compression tests have also been conducted to assess degradation trends, including the evolution of resistances and the stability of electrochemical interfaces under repeated mechanical loading. Complementary mechanical and electrical characterizations, such as pressure-dependent contact resistance measurements, provide additional insight into how compression modifies interfacial conduction pathways [32], [33], [34], [35]. This section reviews representative experimental work focused on the electrochemical consequences of

compression and synthesizes the common findings and open questions relevant to performance, impedance behavior, and degradation mechanisms [36], [37].

2.2.1 Effect of the Compression on PEM Electrolyzer

This study investigates the influence of clamping pressure on the performance of a PEMWE cell in a test cell that directly applies pressure (0.5 – 3.5 MPa) to the active area. Polarization curves and EIS demonstrate that pressures greater than 2.5 MPa tend to improve ohmic and mass transport losses resulting in a poor performance. The stress relaxation in the CCM falls and leads to mechanical pressure drop, which accounts for overload more than 17%. Performance for key performance indicator is best at 1.5 – 2 MPa, where the trade-off between contact resistance and mass transport is optimized. The operating temperature is around 60 – 80°C for PEMWE. The two cell designs are both mesh type with an expanded mesh as porous transport layer on the anode and cathode side, which consist of Freudenberg design plus an extra Freudenberg carbon paper layer on the cathode side. There was optimum clamping pressure between 2.0 and 3.0 MPa for the mesh design and Freudenberg design, respectively. Above this pressure ohmic and mass transport losses become too high, causing deteriorating performance [31].

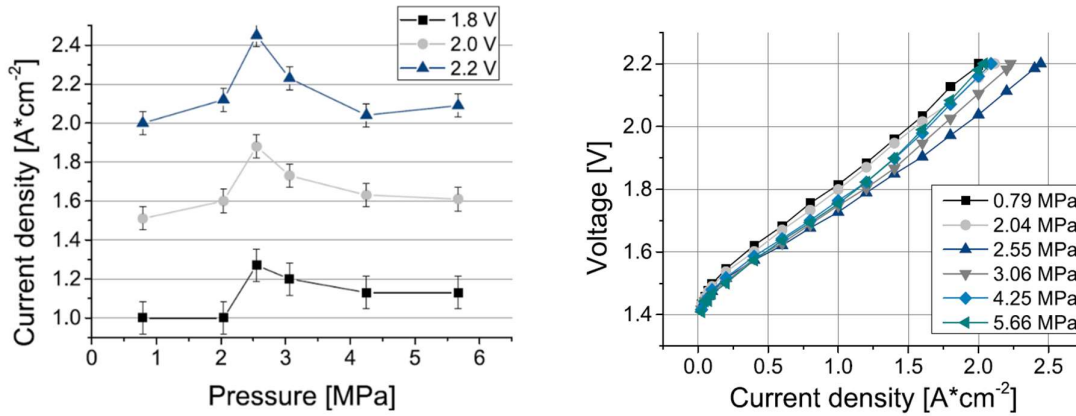


Figure 3 left: Current density at 1.8 V, 2.0 V and 2.2 V for different clamping pressures & right: Polarization curves for different clamping pressures for expanded mesh design [31].

The current density versus pressure (0 - 6 MPa) relationship for different voltages of applied electric fields of 1.8 V, 2.0 V, and 2.2 V, is illustrated on this graph as follows. In 1.8 V, at ambient pressure, approximate current density is at $1.1 A \cdot cm^{-2}$; from approximate 0 MPa to 2 MPa current density increased to approximate $1.3 A \cdot cm^{-2}$; above 2 MPa current density returned down to approximate $1.1 A \cdot cm^{-2}$ until 6 MPa. Overall, the trend showed a decrease, additionally a slight decrease with pressure increase. At 2.0 V, current density began at approximately $1.5 A \cdot cm^{-2}$, increased to approximately $1.6 A \cdot cm^{-2}$ at 2 MPa and from 2 MPa to approximately 2.5 MPa increased to approximately $1.9 A \cdot cm^{-2}$. The current density decreased again with increasing pressure even though it was increasing originally. At 2.2 V, current density began at approximately $2 A \cdot cm^{-2}$; current density caused an approximate peak

of $2.4 \text{ A} \cdot \text{cm}^{-2}$ at approximately 2.6 MPa and current density decreased from 2.6 MPa to 6 MPa steadily to $2.1 \text{ A} \cdot \text{cm}^{-2}$. It showed an overall larger increase initially in current density then more steady decreases thereafter.

This figure 3 on the right, shows voltage plotted against current density ($0\text{--}2.5 \text{ A} \cdot \text{cm}^{-2}$) at different pressures at 0.79 MPa, 2.27 MPa, 3.06 MPa, 4.25 MPa, 5.67 MPa. The pressure at 0.79 MPa indicates that voltage increases rapidly, correlating to a current density of 2.2 V at $2 \text{ A} \cdot \text{cm}^{-2}$. This suggests that the overpotentials and performance are worse at low pressure. As pressure increases for example from 2.27 MPa to 5.67 MPa, the curves continue to shift right/downward; relatively at a current density of $2 \text{ A} \cdot \text{cm}^{-2}$, the voltage decreased from 2.1 V at 0.79 MPa to 1.8 V at 5.67 MPa, again indication that as pressure increases the performance increases because more current is achieved at a lower voltage.

In summary, the data presented in this figure shows the relationship between current density with the pressure at fixed voltages, while also evaluating how the voltage changes with constant current density at the given pressure. Higher pressure seems to improve performance (lower voltage at the same current density); however, trends of current density appear to reach a peak performance around 2.55 MPa before a decrease [31].

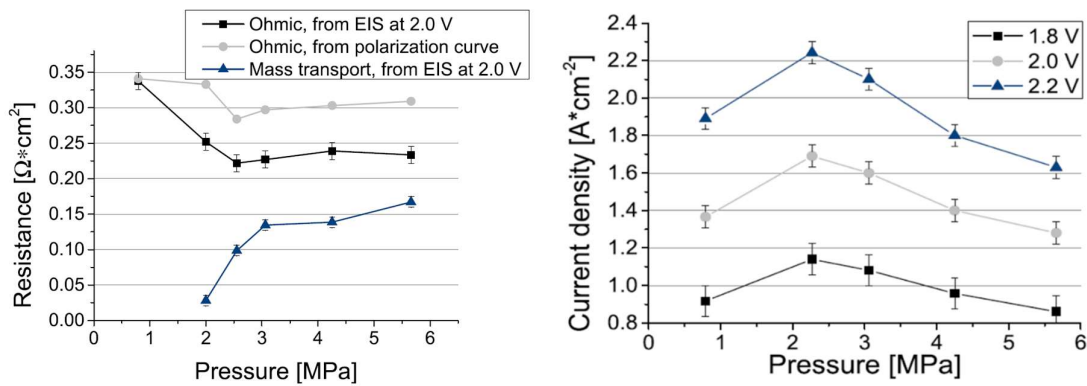


Figure 4 left: Ohmic resistance from EIS at 2.0 V and polarization curves and mass transport losses from EIS at 2.0 V for expanded mesh design & right: Current density at 1.8 V, 2.0 V and 2.2 V for different clamping pressures for Freudenberg design [31].

This figure shows resistance versus pressure (0 to 6 MPa) for the same three structural trends, ohmic from EIS, ohmic from polarization, and mass transport. This sample, or condition, could imply something different relative to the resistances presented above as the trends are not quite the same in terms of pressure. Ohmic from EIS starts off about 0.34 at 0 to 1 MPa, down to 0.23 at 2.5 MPa, and then a slight increase to 0.24 at 4.5 MPa. Significant initial drop then slight increasing behavior. Ohmic from polarization, starts relatively high at or about 0.35 or 0 to 1 MPa, whereas changing at about 2.4 MPa to 0.28, then changing to 0.31 at about 6 MPa. The trend overall suggests a U-shaped mean path, but worsening behavior at higher pressures. Mass transport starts relatively low at 0.04 at 2 MPa then rise to a peak, and most importantly, it is crucial to observe the critical peak of 0.14 at 3 MPa and subsequently rise to 0.16 at 6 MPa.

Overall, mass transport resistance seems to be increasing with increasing pressure with respect to other experimental data.

There are two panels, current density vs. pressure (0–6 MPa) for fixed voltages. Voltage vs. current density (0–2.5 $\text{A}\cdot\text{cm}^{-2}$) under varying pressures. 1.8 V starts at 0.9 $\text{A}\cdot\text{cm}^{-2}$ at between 0 and 1 MPa and rises to 1.1 at a pressure of 2.3 MPa and then declines to 0.9 at 6 MPa. Minor overall reduction that exhibits decreased performance with increased pressure. 2.0 V. Initial current density is 1.3, then increases to a peak of 1.7 at roughly 2.3 MPa, and then subsequently declines to 1.3 at 6 MPa. The overall decrease is benign, with a transient enhancement early on at increased pressure. At 2.2 V, current density rises to 2.3 $\text{A}\cdot\text{cm}^{-2}$ at 2.3 MPa and decreases to 1.6 $\text{A}\cdot\text{cm}^{-2}$ by 6 MPa. Significant initial increase but subsequent continuous decrease. Transport losses increased than decreased when increasing the pressure [31].

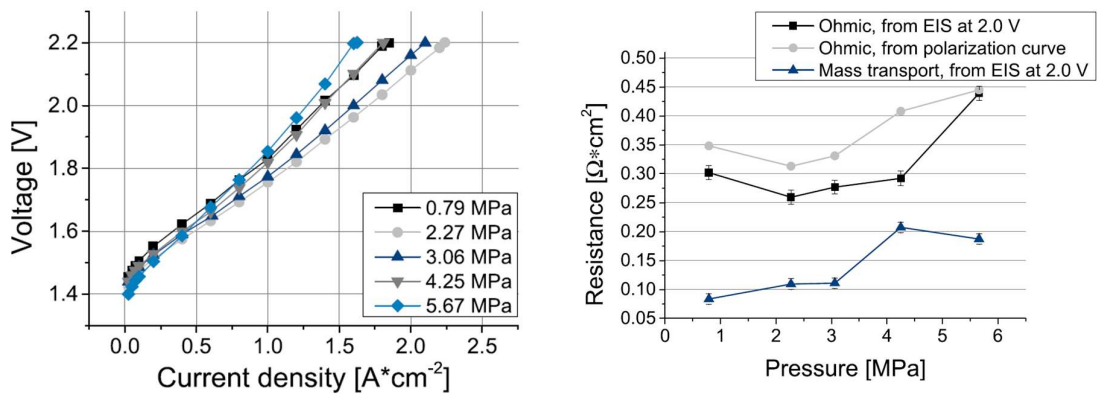


Figure 5 left: Polarization curves for different clamping pressures & right: ohmic resistance from EIS at 2.0 V and polarization curves and mass transport losses from EIS at 2.0 V for Freudenberg design [31].

Lower pressures at for example 0.79 MPa, the voltage increases sharply as current density increases, reaching a voltage of 2.2 V at 1.9 $\text{A}\cdot\text{cm}^{-2}$, indicating large overpotentials and obviously poor performance. When the pressure increases up to a maximum of 5.66 MPa, the curves shift right/down, and the voltage increases more gradually, for instance, a current density of 1.7 $\text{A}\cdot\text{cm}^{-2}$ the voltage decreases from 2.1 V at low pressure to 1.8 V at high pressure. This illustrates improved performance at higher pressure, where higher currents can be achieved at lower voltages.

The figure 5 on the right, illustrates resistance vs. pressure, showing three trends. The ohmic resistance from EIS at 2.0 V, ohmic from polarization curve, and mass transport from EIS at 2.0 V. The Ohmic from EIS begins high at 0.33 $\Omega\cdot\text{cm}^2$ from 0-1 MPa, sharply drops to 0.26 at 2 MPa, and then stays roughly flat 0.23–0.24 up to 6 MPa. This indicates an initial improvement in electrical conductivity with pressure followed by stabilization. Ohmic from polarization, initially begins at 0.35 and decreases gradually, somewhat unevenly, to 0.34 at 3 MPa, lastly, increases to around 0.34 $\Omega\cdot\text{cm}^2$ up to 6 MPa. Mass transport is at a very low resistance of 0.07 between 0-1 MPa and steadily increases to 0.12 at 3 to 4.2 MPa. This indicates that mass

transport losses are decreased with pressure at the low pressure but then increase as pressure continues to increase, possibly due to mass transport limitations such as diffusion limitations [31].

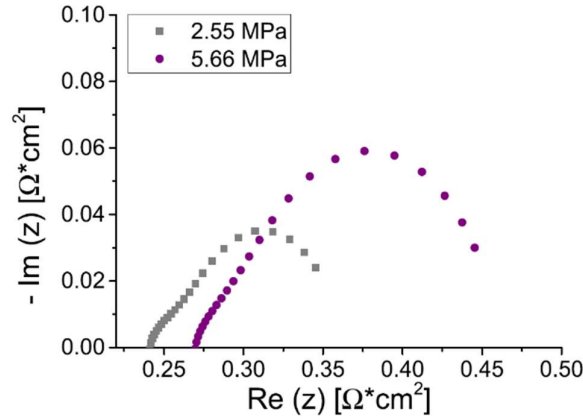


Figure 6 Example for measured impedance spectra, expanded mesh design [31].

This graph illustrates the relationship between real impedance and negative imaginary impedance in units of $\Omega \cdot \text{cm}^2$, for two different pressures of 2.55 MPa and 5.66 MPa. At 2.55 MPa, the impedance data starts close to 0.25 and then extends to form a large semi-circle-like arc, peaking at around imaginary impedance 0.035 at real impedance 0.32, before rounding downwards to $\text{Re}(Z) \approx 0.5$ with $-\text{Im}(Z) \approx 0.00$. This shows a higher overall impedance, with a larger degree of spread as compared to the other pressure. At 5.66 MPa, the arc is smaller, and has shifted to the left, and peaking, higher than the 2.55 MPa, at $-\text{Im}(Z) \approx 0.09$ at $\text{Re}(Z) \approx 0.3$, and then rapidly descending at $\text{Re}(Z) \approx 0.35$ to similar $-\text{Im}(Z) \approx 0.00$. This relationship reflects lower impedance and better conductivity at the higher pressure, with the system responding more efficiently [31].

This article by Rost *et al* (2022) [30], describes a new hydraulic compression PEMWE test cell with uniform pressure distribution of up to 25 cm^2 active area, which can be operated at conditions of temperatures up to 80°C and current densities of 6.0 A/cm^2 . The hydraulic clamping system guarantees an even current density and temperature distribution. Polarization curves and EIS indicate repeatable performance with low ohmic losses. Key performance indicator suggests suitability for component testing, best at moderate compression pressures. The design improves the reliability of testing for PEMWE materials. As shown in the following figure polarization curves of cell, pressure by 8 bar and temperature at 80°C water flow rate of 50 ml/min , test received based on two types of membrane *Nafion N115* and *Nafion N117*.

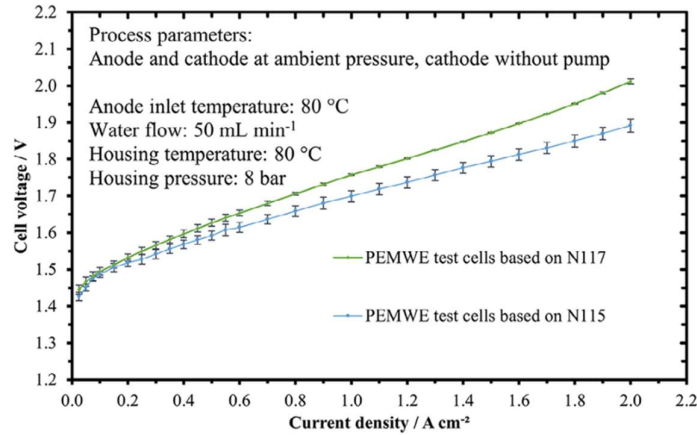


Figure 7 Polarization curves of polymer electrolyte membrane water electrolyzer (PEMWE) test cells equipped with different commercially available catalyst-coated membranes (CCMs) determined at an operating temperature of 80°C and a hydraulic medium pressure level[30].

In basic terms, this illustration presents the comparison of the performance of two proton exchange membrane water electrolyzer test cells having a Nafion 117 membrane and a Nafion 115 water splitting sample cells, respectively, indicating how much voltage is needed to split water into hydrogen and oxygen with increasing production rate current density under 80°C temperature, with the water flow rate set at 50 mL/min, the electrodes maintained at ambient pressure and the cells maintained at 8 bar housing pressure. The N115 configuration containing a 125 μm membrane generates more current with less voltage going from 1.45 V to 2.5 V as current density increases from 0 to 2.2 A/cm² as a function of the overpotentials (and reduced ionic resistance inside the cell, leading to more energy efficient with hydrogen produced per energy unit. However, the N117, which had a 175 μm membrane thickness, increased voltage requirements voltage going from 1.5 V to 1.9 V, which suggested higher ohmic losses and less energy efficiency, especially as the load increased, which could contribute to higher energy costs. These trends are linked to membrane thickness, but are also partial functions of temperature, pressure and flow rates, leading to different costs, life span, etc, of hydrogen production. In agreement with the results displayed in the plot, the N115 configuration would demonstrate a more energy efficient set of operating conditions [30].

This report by Frensch et al (2018) [37], investigates the effects of PEMWE cell compression based on polarization curves, EIS, and modelling as a function of current (0.01 – 2.0 A/cm²), temperature (60 – 80°C) and pressure (0.77 – 3.45 MPa). Compression lowers contact resistance thereby improving performance, but it is clear this process also requires optimization; no significant magnitude of severe mass transport associated losses was observed. The plot indicates that there was more than one slope that relate to either electrochemical reactions or ohmic effects associated with non-dedicated electrodes. KPIs confirm improvements in the performance attributed to compression and the EIS confirm reduction of ohmic losses associated without losses from mass transport at the high currents. The plot represents the impedance data to their model fits shown in the Nyquist form at different currents level and both temperatures at 2.30 MPa pressure.

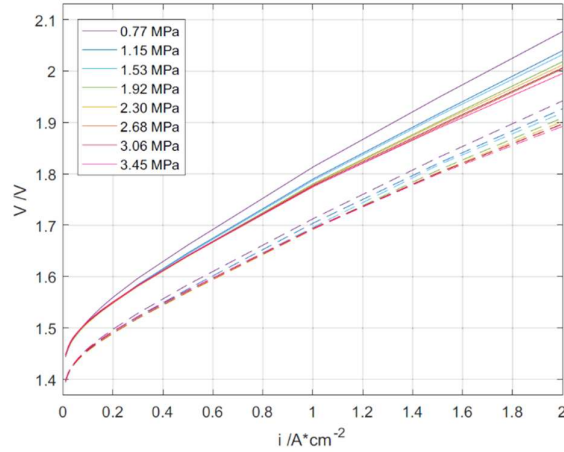


Figure 8 Polarization curves for all compressions at 60°C (solid) and 80°C (dashed) [37].

In simple terms, this graph illustrates the voltage required to drive an electrochemical process, like splitting water into hydrogen and oxygen, as the production rate of current density increases, with individual tests run at various pressures ranging from 0.77 MPa to 3.45 MPa, where each coloured line shows the effect of pressure on system efficiency and demonstrates that higher pressures tend to require more voltage to achieve the same output. This implies some trade-off in performance. The voltage 1.4 to 2.1 V steadily increases with current density 0 to 2 A/cm², starting from around 1.4–1.5 V and approaching 1.9–2.1 V, which appears to be linear with a steep slope for the higher pressure tests; notably, the 0.77 MPa test climbs gradually to about 2.08 V while the 3.45 MPa condition rises to about 2.1 V, indicating that higher efficiencies are achieved at lower pressures and that higher pressures may incur decreased efficiencies but with more voltage supplied to reach a plateau due to the overpotential and related resistances represented by mass transport or ohmic losses, which then raise energy costs and heat levels. More voltage at higher pressures thus also yields less efficiency, whereas lower pressures would improve efficiency. However, gas removal and/or stability with higher loads may be a compromise at lower pressures as well, greatly affecting the design approach of an electrolyzer that strives to work closely to performance and cost objectives [37].

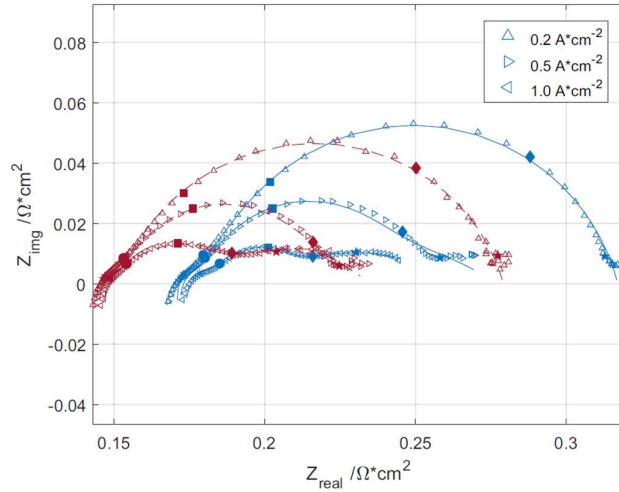


Figure 9 Impedance data at 2.30 MPa and different currents as a Nyquist plots. The marks represent the frequencies 10000Hz, 1000 Hz ● (C), 100 Hz ■, 10Hz ♦, and 1 Hz ▲. The respective model fits are shown for both 60 C (solid) and 80 C (dashed) [37].

Essentially, this Nyquist plot generated with electrochemical impedance spectroscopy (EIS) displays the electrical resistance and reactance of an electrochemical system, likely a proton exchange membrane PEM electrolyzer, while operating at different applied loads with current densities of 0.2, 0.5 and 1.0 A/cm². This highlights how impedance, the combination of resistance and capacitance behaves as a function of defining losses in the slope including slow reactions, ohmic resistance and others. The Nyquist graph displays data reflected as negative imaginary impedance ($-Z_{imag}$, -0.04 to 0.08 $\Omega \cdot \text{cm}^2$) and real impedance (Z_{real} , 0.15 to 0.3 $\Omega \cdot \text{cm}^2$). Each data curve circles in the Nyquist reflects a semicircle depressed shape that is observed to shrink as current density increased. The 0.2 A.cm² exhibits the largest arc with a peak at $-Z_{imag} \approx 0.07$ and has a relatively high charge transfer resistance, indicating low efficiency with an expensive voltage with 0.5 A.cm² and 1.0 A.cm² representing relatively improved conductivity with a peak at 0.05 and 0.04 compared to the 0.2 A.cm² test. The distortion noted in the 1.0 A.cm² test indicates potential mass transport limitations, inefficiencies or durability issues of the 1.0 A.cm² load related to the charge transfer mechanism for the material system performance. It is important to understand these trends, because they are influenced by a relatively similar trajectory on the kinetic properties of the electrode materials, the intrinsic properties of the membrane; principles of current density-based volumetric gas bubble formation reduction in effective volumetric surface area with each of these operating conditions effect the overall device's performance including overall efficiencies as well as power consumption for specific hydrogen production rates. Note, as current density increases the performance subsequently increases followed with reductions in potential durability of the membrane for long-term operation of the system high volumetric currents [37].

The influence of porous transport layer compression on hydrogen permeation through the PEMWE is investigated at PTL compressions ranging between 0.1 and 2 MPa. It is found that, as PTL compression increases, permeation increases linear or nonlinear correlation as a function of current density, and cell voltage decreases. The investigations were carried out at operating conditions of 60°C. In addition to the hydrogen permeation rate, EIS was used to further separate ohmic and activation losses. Several key performance indicators were measured up to 4 mA/cm² at high PTL compression, and it was found that when the PTL compression was high 10 – 20 mV lower than at lower PTL compression is measured. These results demonstrate the potential of PTL compression to play an important role in operating a safer and more efficient PEMWE system. MEAs were assembled by hot pressing one anode coated PTFE and one cathode coated PTFE together using a *Nafion* 212 membrane thickness of 50 mm at 130°C and 0.5 kN/cm² for 3 minutes. These outcomes suggest that the compression of the C-PTL in a PEM water electrolyzer cell not only affects the hydrogen permeation and cell voltage but is also related to current densities of up to 2 A/cm² at maximum current density values reported in the literature when measuring the hydrogen permeation. Additionally, the magnitude of compression of the C-PTL influences the correlation is linear or otherwise depending on the amount of compression of the C-PTL, as can clearly be seen in the current density range of up to 4 A/cm² in the below figure [34].

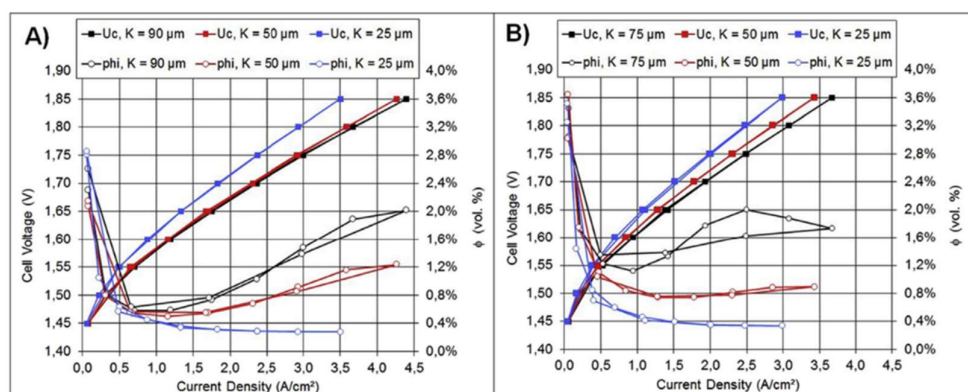


Figure 10 Cell voltages and hydrogen volume fraction, data with different compression values K characterized once; B) data characterized three times, each time with an increased compression value K [34].

This image shows the performance of an electrochemical cell, most probably a proton exchange membrane water electrolyzer, shown in comparison in panels A and B to investigate the relationship of cell voltage (U_c) and a volume percent (ϕ - gas crossover or void fraction) as the production rate (current density) goes aloud for different K , which it likely thickness in μm , panel A tested 90, 50, and 25 μm and panel B tested 75, 50, and 25 μm . Consider the impact of thicker K providing a more favourable regime as voltage is required to be lower, but ϕ is higher indicating greater potential gas related issues. In practical terms, for both panel A and B the cell voltage is around 1.45 V and increases with current density up to 4.5 A/cm². The thicker K (90 μm or 75 μm) has a relatively slow increase in cell voltage to ~1.65 V, while thinner K (25 μm) has a steeper increase to ~1.85 V. The result is better cell performance and

lower energy requirements. It's worth noting that thicker K likely reducing ways to multiplex the cells in this case do improve energy in the sense of reducing cell voltage and therefore energy cost per production maybe at the expense of restricting a compact device. Meanwhile phi is a higher value for thicker K $\sim 3.5\%$ for $90\ \mu\text{m}$, $\sim 3.8\%$ for $75\ \mu\text{m}$ and at high current density reduce to $0 - 0.5\%$ however may relate to potential safety and durability issues as phi may indicate potential gas crossover or issues related to flooding at low currents, reduced at high current due to handling more gas more efficiently. These tendencies, all of which are also determined by K thickness which decreases ohmic losses but leads to diffusion problems, current density, and things like membrane properties or operating conditions, result in the electrolyser's efficiency, gas purity, the lifetime of system components, and design trade-offs in this hydrogen production [34].

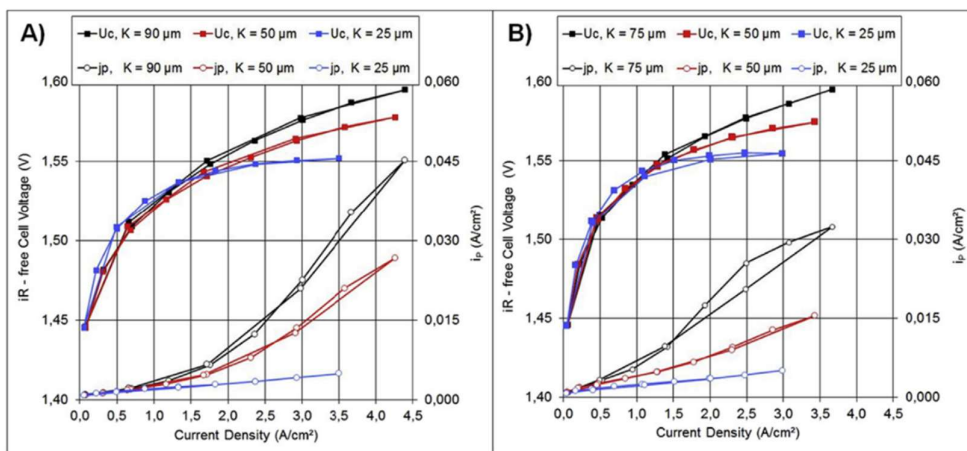


Figure 11 cell voltages and permeation current densities as a function of current density A) Data with different compression values K characterized once; B) data characterized three times, each time with an increased compression value K [34].

To simply compare the performance of an electrochemical cell, likely some form of a proton exchange membrane water electrolyzer, across the two panels (A and B) an examine has been done by Stähler et al (2022) [34], in which how the cell voltage (U_c) and the IR-free cell voltage (j_p possibly corrected for ohmic losses) vary as the production rate of current density increases based on the specified K values likely thickness in μm . Panel A is testing K values of 90, 50, and 25 μm , while panel B is testing K values of 75, 50, and 25 μm . The starting voltages for both panels at $0\ \text{A/cm}^2$ for both the U_c and j_p was around $1.40 - 1.45\ \text{V}$ and generally increased with increasing current density to a max of $4.5\ \text{A/cm}^2$. In panel A, at the end of the study the thicker K $90\ \mu\text{m}$, rose the least steeply to $1.60\ \text{V}$ and $j_p\ 0.06\ \text{A/cm}^2$; thin K $25\ \mu\text{m}$, was around the same point to $1.55\ \text{V}$ with $j_p\ 0.015\ \text{A/cm}^2$. In panel B, $75\ \mu\text{m}$ at the end is $1.60\ \text{V}$ with $j_p\ 0.06\ \text{A/cm}^2$ versus $25\ \mu\text{m}$ blue at 1.55 with $j_p\ 0.015\ \text{A/cm}^2$. This shows that the thicker K must rise less which can improve operating efficiency with the gas generating process because the thicker K is providing a better co-catalyst for the energy input cell voltage needed in a water split reaction. These j_p trends could indicate that the thicker layers have higher ohmic losses or other variables like gas evolution, affecting purity or stability of the hydrogen; thus, all

trends shown for the K thickness and current density throughout the study influence the energy efficiency, operational costs, and quality of hydrogen production at the anode of the electrolyzer [34].

Another study by Ortiz et al (2024) [38], examines MEA compression of 30 – 60%, no absolute pressure in PEMWE, which shows the optimal 30 – 40 % compression balances ohmic and mass transport overpotentials, while further compression only increases the mass losses. The breakdown of overpotential is assessed in a similar manner to the EIS in assessing the losses within the cell voltage and overpotentials constitute key performance indicators. In this study electrolyser cells were analysed at various degrees of compression to determine the optimum cell compression level in terms of performance and membrane deformation. As the membrane is pressed into the pores of the anodic Ti-PTL, this resulted in increased membrane deformation, as well as decreased effective membrane thickness as the cell compression increased. Furthermore, the performance coefficients of the cell compression were studied to determine if the deformation observed had any effect. For the very low cell compression of 20 %, these results showed that increased resistance was then sourced from the contact resistance of the CL and the PTL. While the other extreme at 60 % compression resulted in increased cell voltage due to mass transport losses, although, a lower ohmic overpotential was seen. The estimated ohmic and mass transport overpotentials confirmed 40 % as the optimal compression level as a balance between membrane deformation, without incurring further penalties due to high mass transport resistance at high current densities of 3.0 A/cm^2 . In summary, both trends require to select the optimal compression based on the target operating current density. As future electrolyzers are aimed to have a higher current density, higher compression may be desired. However, too much compression can lower life span because of the delamination due to high deformation [38].

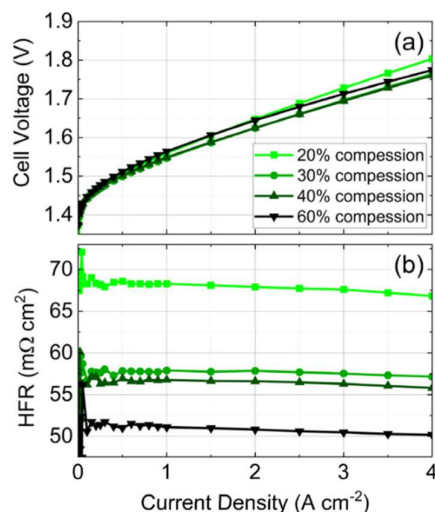


Figure 12 Polarization curve of N212 PEMWE cells with an average anodic loading under different compression levels present comparable performances at compressions of 30 and 40 % (a) The cells' HFR shows a decreasing trend for higher compression levels, going from

approximately $67.5 \text{ m}\Omega \text{ cm}^2$ to $50.5 \text{ m}\Omega \text{ cm}^2$ at 3 A/cm^2 for a compression of 20 % and 60 % respectively (b) [38].

The graph depicted describes the performance at various mechanical compression levels by plotting cell voltage along the vertical axis against current density. The curve arise, with the lowest compression at 20%, being around 1.5 volts at the start and increasing to plateau at a volts range of approximately 1.8v, 30% is slightly lower; 40% is even lower; and 60% is the lowest at around 1.4 volts at the start of the test with the longest plateau, increasing to approximately 1.75 volts at its maximum; indicating increased mechanical compression is detrimental to voltage efficiency due to decreased mobility of the ions, increased internal resistance or structural failure of the cell [38].

This research investigates the contact resistance created between the anode catalyst layer and titanium-based porous transport layer, in a polymer electrolyte membrane water electrolysis (PEMWE) setup, testing metal coatings *Pt, Au, Ir* at thicknesses from 2 – 400 *nanometers* under clamping pressure ranging from 0.5 – 2 *MPa* of compression pressure. The cell is operated at 70°C absolute, the cell pressure of 1 *bar* absolute and no differential cathode or anode pressures. The membrane is *Nafion 117*, and a water flow rate of 200 *ml/min* is supplied at the anode. There are no differences in gas diffusion layers, GDLs where sintered titanium PTL is used at anode with coatings, and carbon paper substrate is used in the cathode. Key performance indicators, KPIs are the polarization curves (IV curves show best performance at beginning of life, with the Pt, and very good durability with the 10 *nm Ir* during 510 *h* for both tests cycling 1.5 – 2.2 *V* or 0 – 2.2 *V*, and almost a 50 *mV* increase in voltage after cycling for the thin *Ir*). From EIS analysis, the ohmic resistance decrease with stress for example 20 – 30% decrease from 0.5 to 2 *MPa*. The durability testing shows that the thin *Ir* 10 *nm* is the best, low-cost way to reduce contact resistance, and produce stability [39].

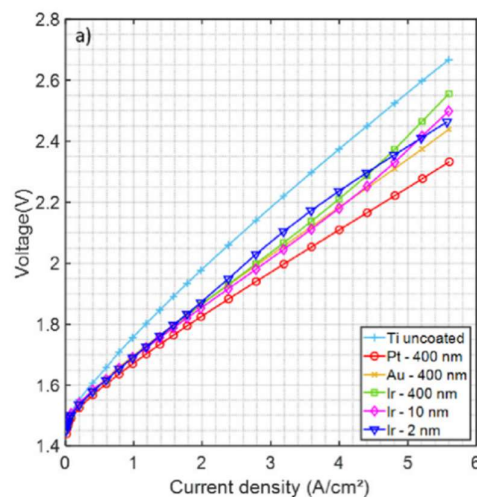


Figure 13 a) Polarization curves at the beginning of life for the different coated and uncoated Clamping pressure = 5.1 *MPa* [39].

This graph displays the electrochemical performance of numerous metal coatings on titanium substrates, probably for applications that involve water electrolysis or oxygen evolution reactions, placing cell voltage on the y-axis and current density on the x-axis. Simply put, it indicates how much voltage goes into achieving specific current densities for the different catalysts with less voltage indicating higher efficiency/catalytic activity for the same current. The trends show upward-sloping curves for each catalyst, starting at around 1.4V at zero current density and increase with increasing current density. For example, titanium uncoated requires the highest voltages representing the worst performance because it had a higher overpotential; platinum 400 nm and gold 400 nm performed somewhat better, but still higher voltage than both iridium variants; also, iridium 400 nm improved performance; iridium 10 nm improved performance to lower voltages applied at the same current; and especially iridium 2 nm and least voltages applied at the same current. Overall, thinner iridium layers on the titanium substrate 10 nm and 2 nm are indicative that as the iridium goes down to 2nm coating thickness, it clearly shows improved activity as a catalyst and lesser voltage applied which is beneficial from an energy efficiency and cost prospective in electrochemical devices. It is possible that using thinner iridium layer allows for better utilization of the iridium surface area, or evenless material resistance due to less material in layering process [39].

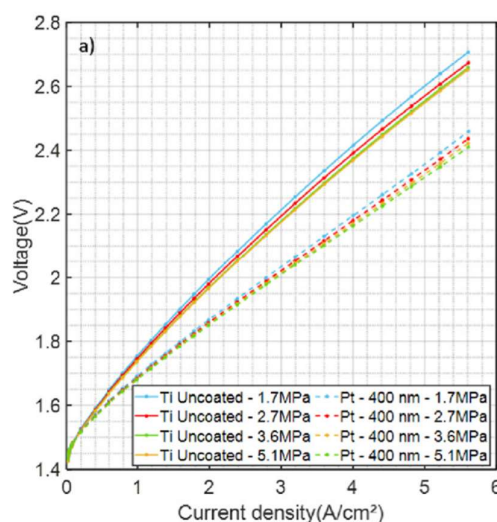


Figure 14 a) Polarization curves at the beginning of life for the Pt-coated and uncoated PTLs for different clamping pressures [39].

This graph compares the electrochemical performance of uncoated titanium (Ti) anodes to titanium (Ti) anodes coated in platinum (Pt) 400 nm thick, measured under various contact pressures, presumably in an electrolyzer configuration, with cell voltage plotted as a function of current density. More simply, it describes how much electrical push voltage is required to achieve increasing current through the device at different pressures, with lower voltage meaning improved efficiency at the same current. The trends show upward-sloping curves for all conditions starting near 1.4V at 0 current and curving up to approximately 2.6-2.7V at 6 A/cm²; and at for each pressure condition 1.7 MPa, 2.7 MPa, 3.6 MPa, 5.1 MPa, that the dotted lines Pt coated require a lower voltage than solid line uncoated Ti; thus showing that the Pt

coating increases catalytic activity thereby reducing overall overpotential; and that for all pressures the voltage required increased for both coated and uncoated conditions; both curves shift slightly upward at higher pressures so greater voltages needed, indicating that increased contact pressure increases resistance or mass transport efficiencies, which consequently negatively effects total energy efficiency, durability, and operational costs for high-pressure electrochemical systems [39].

The purpose of this investigation is to evaluate the influence of bolt torque ranging from 0 to 20 Nm, correlating to clamping pressures reaching $\sim 3\text{MPa}$ on the active area and contact resistance on the performance of a 50cm^2 single cell PEMWE. The compression pressure range is inferred from the torque 10 – 15 Nm is deemed optimal with a reported 1 – 2 MPa produced on the MEA. The graph provided indicate a non-uniform pressure distribution, higher pressure under the lands. Operating temperature cell is maintained at 60°C . Cell pressure is maintained at atmospheric 1 bar. The membrane is Nafion 117. Water flow rate is at 200 ml/min at the anode. There was no mention of GDLs specifically and did not appear to be varied; carbon paper was used at the cathode and Ti felt was used at the anode. The KPIs were polarization curves noting optimal efficiency at 15 Nm using PTFE/EPDM gaskets and optimum efficiency using silicon was noted from 10 – 15 Nm. The maximum reported efficiency was 65% at a current density of 1 A/cm^2 and contact resistance noting that contact resistance decreases with increased torque, $50\text{ m}\Omega\text{ cm}^2$ at low torque to $< 10\text{ m}\Omega\text{ cm}^2$ at a torque of 15 Nm [36].

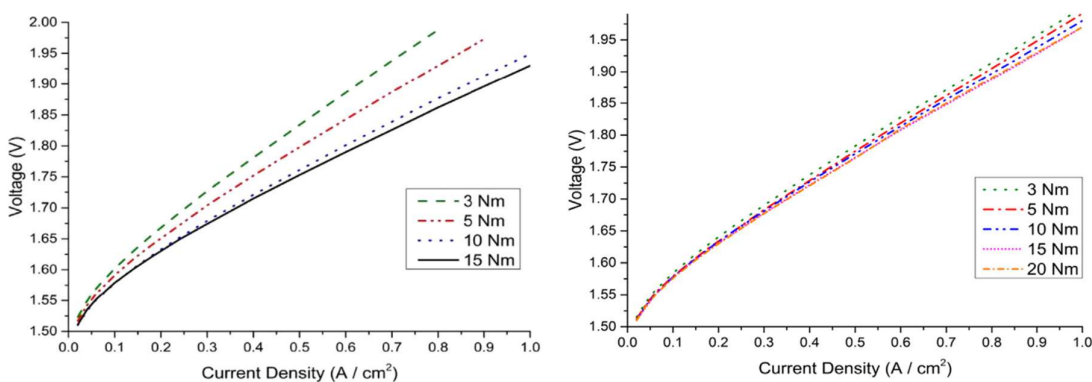


Figure 15 left: The polarization curve with PTFE gasket material at 3, 5, 10, 15, and 20 Nm clamping bolt torques & right: The polarization curves of the cell with silicon gasket material at 3, 5, 10, 15, and 20 Nm clamping bolt torques [36].

This graph shows the electrochemical performances of electrodes with different thicknesses of nanomaterials likely catalyst coatings like iridium, reporting voltage vs current, probably used for electrolysis of water for example. Essentially, it is indicating how much voltage it takes to drive increasing current; the lower the voltage required, the more efficient it is. The trends indicate an upward sloping curve which starts around 1.50 to 1.55V at zero current and continues to climb until approximately 1.90 to 2.00 V at 1.0 A/cm^2 . The results indicate the thinnest layer 3 Nm, required the least amount of voltage followed by the 5 Nm layer, 10 Nm layer, and the 15 Nm layer needing the highest voltage. This indicates a trend of decreasing

overpotential which demonstrates too, that thinner coating of nanomaterials assists catalytic activity by reducing overpotential effects which ultimately improve energy efficiency during electrochemical processes. It is also important to consider optimizing materials for electrochemical processes in relation to the performance outcome from the tests [36].

The graph displays the electrochemical polarization behavior of various catalysts or electrodes that have different types of nanomaterial thicknesses possibly coatings like iridium, and plots volts against current density, likely for use in something like oxygen evolution in water electrolysis. To put it simply, this graph shows how much voltage is required to drive increasing electrical current through this system for each layer thickness for these thin layers. The voltage is less to create increased current, where the lower the slope of the line, the better the performance. The trends shown are almost linear upwards for each layer's thickness as they all begin to reach around 1.50-1.55V at zero current and reach around 1.90-1.95V at 1.0 A/cm². Overall, it is reasonable to see the trend where the thinnest layer (green dotted line 3 Nm) has the lowest voltages at higher amperage (>2.50), followed by additional lower layer thickness 5Nm, 10Nm, 15Nm, and finally 20Nm, which is the thickest layer with the highest required voltage. Thin coatings improve catalytic efficiency through reducing overpotential and optimizing reaction kinetics which will all affect minimizing energy consumption, cost, and scale in reliable electrocatalytic devices such as fuel cells or electrolyzers [36].

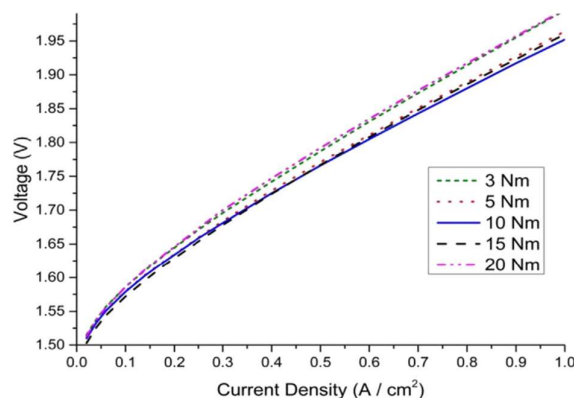


Figure 16 The polarization curves with EPDM gasket material at 3, 5, 10,15 and 20 Nm clamping bolt torques [36].

This electrode performance data presents the electrochemical activity of electrodes that are composed of nanomaterials of varied size, where voltages are plotted against currents. The observed electrochemical performance is likely applicable to electrochemical energy applications potentially hydrogen production in electrolysis. Accordingly, the figure provides a plot of the voltage required to drive a given current through the electrode. Less voltage indicates more efficient electrochemical activity. The trends in the figure exhibit upward sloping curves that begin at approximately 1.50-1.55V for 0 A/cm² and rise to approximately 1.90V -1.95V for 1.0 A/cm². A nanomaterial with a thickness of 3 Nm is the least voltage dependent, then 5 Nm, 10 Nm, 15 Nm, and 20 Nm, having the highest voltage with greater current. This trend indicates that thinner nanomaterial layers lead to increased catalytic activity by decreasing overpotential for a given current density, while also reducing energy efficiency

in electrochemical processes, as well as reducing the amount of material in the electrode itself [36].

This study explores and assesses a new thin titanium liquid gas diffusion layer (LGDL) for PEM electrolyzer cells. The thin LGDL 25 μm thick with straight, adjustable pores, is benchmarked against thick titanium felt LGDLs 350 μm thick with random pores reference to corrosion, ohmic, interfacial and transport losses, to increase the efficiency of hydrogen production. Testing in a bench scale PEMEC demonstrated that the thin LGDL yielded lower voltages at higher efficiencies that suggested their suitability for use in lightweight, low-cost PEMECs. Operating Cell Temperature Standard temperature of 80°C is set for all testing which falls within the 60 – 80°C temperature range. EIS (Electrochemical Impedance Spectroscopy) Measurement of cell impedance were conducted from 15 kHz to 10 mHz with 15 points per decade, using Nyquist plots for analysis. The purpose of the EIS is to quantify the differences in ohmic, and interfacial resistances realized by the thin LGDL. Polarization curve tested with current densities varying from 0 to 2 A/cm² each step held for 5 minutes. The thin LGDL reaches 1.69 V at 2 A/cm², with efficiency around 88% which is the highest efficiency recorded. This indicates significant reductions in ohmic, interfacial and mass transport losses. Efficiency up to 88% at 2 A/cm² due to the features of the thin LGDL. Multifunctional performance increased in electrical/thermal conductivity, liquid/gas permeability, and corrosion resistance. The thin LGDL can now reduce the overall weight/volume of the PEMEC by 90%. Testing at 80°C in PEM electrolysis with an active area of 5 cm². Combined water (deionized) flow rate of 20 ml/min at the anode. The membrane is Nafion 115 (125 μm thick) [40].

Gas Diffusion Layers were extensively researched and made of titanium felt 350 μm thick, 20 μm in diameter of fiber, 100 μm average pore size, 78% open porosity features; and arranged in a random arrangement. A slim titanium LGDL 25 micrometers thick, 100 micrometers pore size, 30% porosity, with straight and flat pores is helpful because the thin LGDL reduces conductive paths, improves interfacial contacts, and allows for model validation [40].

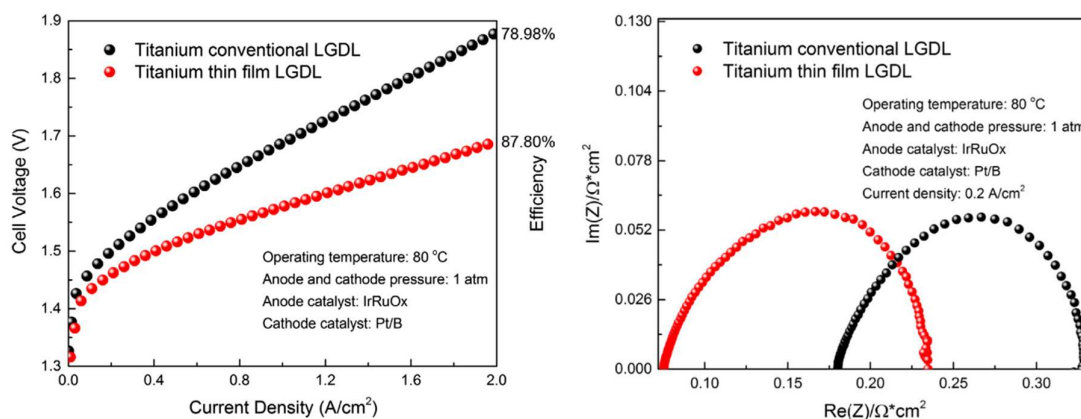


Figure 17 Performance comparisons of PEMEC with titanium thin LGDL and conventional LGDL red colour & EIS comparisons of PEMEC with titanium thin etched LGDL and conventional LGDL (red colour: titanium thin LGDL with thickness of 25 μm , pore size of 100 μm and porosity of 0.30; black colour: titanium felt LGDL with thickness of 350 μm , average pore size of 100 μm and porosity of 0.78) [40].

This graph illustrates an electrolyzer's performance with two different titanium-based gas diffusion layers (GDLs), conventional and thin film, by the plotted cell voltage against current density at 80 degrees Celsius and 1 atm with an IrRuOx catalyst and a Pt/B catalyst. In a general sense, this indicates how much voltage is required to drive a certain current, with lower voltage representing better performance and efficiency. The trends show an upward-sloping curve that starts near 1.3 volts, and while both GDL chemistries trend upwards, the thin film GDL maintains lower voltages, while the conventional GDL increased to about 1.9 volts at 2 A/cm² and both catalysts reach 2 A/cm². The thin film GDL reached only 1.7 volts and exhibited a performance efficiency of 87.80% at 2 A/cm², versus the conventional GDL at 78.98% efficiency. This data indicates that the energy loss is lower with the thin film GDL as compared to the conventional GDL. The thin film GDL design is indicative of either better gas diffusion or reduced resistance, which impacts the energy cost of operating the system, as well as the performance [40].

This graph presents the electrochemical impedance of the electrolyzer using conventional and thin film titanium gas diffusion layers (GDLs) plotting imaginary impedance and real impedance ($\text{Re}(Z)/\Omega\text{cm}^2$) at 80°C, 1 atm pressure. Put simply, it summarizes some of the electrical resistance characteristics of the system, whereby a smaller semicircular shaped arc shown on the graph indicates a lower resistance and subsequently better performance. The trends yield semicircular shaped arcs where the thin film GDL semicircle shape trend is smaller when compared to the conventional GDL suggesting a lower impedance and higher ionic conductivity, likely resulting in an enhanced efficiency or power output by reducing energy losses in the electrochemical reaction process [40].

The experiments were run on a PEMWE stack homemade in-house producing 1 kW of power. These operational conditions like pressure varied from 1 to 5 bar (testing) up to nominal 20 bar; anode pressure at 1 atm. Working Cell temperatures were varied from 40 to 60 °C. Higher temperatures generally result in lower charge transfer resistances. The Nyquist plots reveal 5 processes in total; however, a low-frequency resistance, HFR $\sim 0.1 - 0.2 \Omega \text{ cm}^2$, decreasing as temperatures increase. The polarization curve was tested which implied by the current measurements 37.5 to 50 A, 1.5 to 2 A/cm² for 25 cm² cells in which total charge transfer has been observed to decrease as current, temperature, and pressure all increase [42].

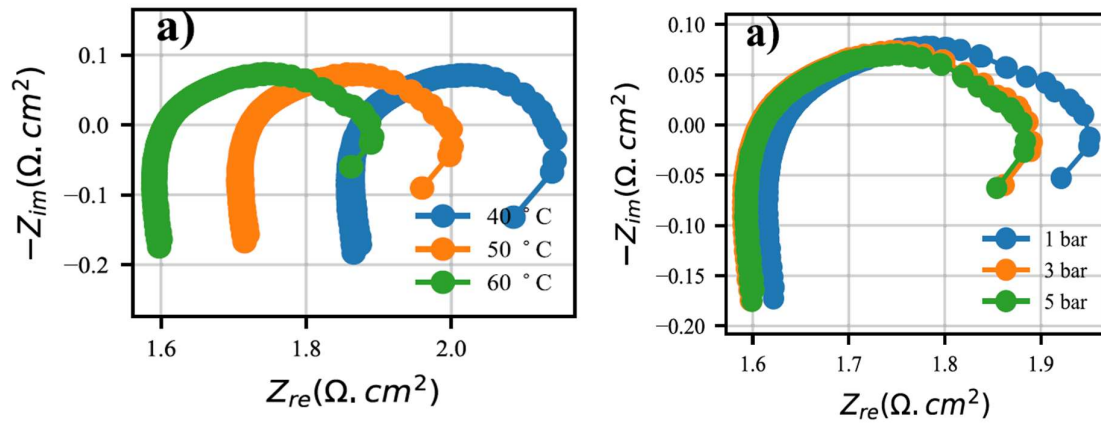


Figure 18 The measured impedance spectra, the estimated polarization and high-frequency resistances under different temperatures (a) Spectra measured at 50 A under 40 ° C, 50 ° C, and 60 ° C & The measured impedance spectra and the estimated polarization and high-frequency resistances under different cathode pressures (a) Spectra measured at 50 A under 1, 3, and 5 bar [42].

The graph 18 on the left, illustrates the electrochemical impedance of a system at varying temperatures 40°C, 50°C, and 60°C, with the imaginary impedance plotted against the real impedance. In simple terms, this can be interpreted as resistance in relation to temperature, where a smaller semicircle indicates lower resistance and improved performance. The trends indicate semicircular shapes; a trend towards 60°C indicates the lowest semicircle for electrical resistance, and there is a similar trend with 50°C, and 40°C has the largest semicircle, suggesting the higher temperature equated to a reduction in impedance because of a possibly increased rate of ions traveling or kinetics of reaction, ultimately affecting the overall efficiency and power output of the system [42].

This graph 18 on the right, shows the electrochemical impedance of an electrolyzer, all shown at varying pressures of 1 bar, 3 bar, and 5 bar. The plot shows imaginary impedance versus real impedance, which generally depicts changes in electrical resistance with varying pressures, where a smaller semicircle reflects a lower resistance and a better performing component. The curves are indicative of a semicircle trend, with the highest pressure being represented at 1 bar being the largest semicircle, then followed by 3 bar, and 5 bar being progressively smaller, implies that increasing pressure to the system effectively reduces impedance likely by improving gas diffusion or contact, thus improving efficiency and result in power density effecting performance overall [42].

This research is focusing on optimizing carbon-based gas diffusion layers at the cathode in PEM water electrolysis for cost reduction compared to titanium PTLs. Key topics include GDL compression analysis, impact of micro-porous layers at the cathode, and their role in improving catalyst utilization for ultra-low Pt loadings 0.05 mgPt·cm⁻². Compression Pressure Range varied from 0% to 80% clamping pressure of 1-2.5 MPa. Gas Diffusion Layers like MGL370 (370 μm thick) at cathode; Pt-coated Ti Fiber PTL (250 μm) at anode. MPLs tested on GDLs for low-loading CCMs [29].

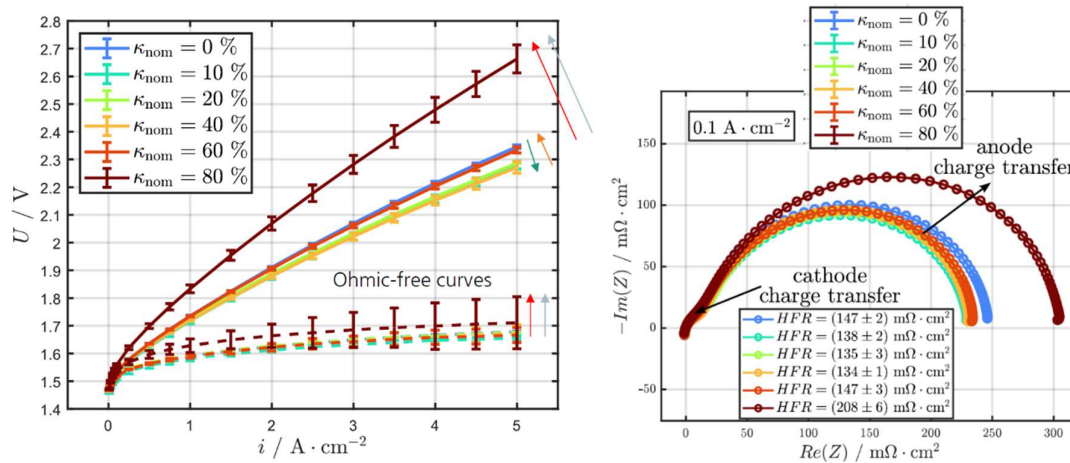


Figure 19 Increasing performance between 0% and 40% compression polarization curve & Increasing Ohmic and kinetic overpotential between 60% and 80% compression [29].

The graph 19 on the left, indicate that lower voltages are preferable for higher efficiencies. The voltage difference between curves can be considered a measure of the work done to overcome system resistance. The curves begin trending upwards around 1.4 - 1.6V while increasing the current density. As can be seen, the curves given the right levels of compression they began at, especially for the 0% compared to 80% compression levels. Overall, the dashed lines (the ohmic-free) curves demonstrate each levels ideal voltage levels with no resistance which adds a fit measure of what could be accomplished if no resistance were present. Thus, compression in the initial state increases performance considerably until get into an increasing current area of the curves however resistance will still between levels at high currents even as the compression increased [29].

EIS and KPI Analysis for example polarization curves show increasing performance from 0-40% compression due to better contact, worsening at 40-60%, and kinetic or residual increases at 60-80%. Ohmic-free curves highlight contact issues. EIS implied in overpotential breakdown (ohmic, kinetic, residual), with MPLs reducing cathode charge transfer and low-frequency impedance mass transport for low Pt loadings [29].

The graph 19 on the right, provides a summary of the electrochemical impedance of an electrolyzer, as a function of nominal compression 0% to 80% and high-frequency resistance 147 to 208 $m\Omega \cdot cm^2$ with a current density of $0.1 A \cdot cm^{-2}$, which plots the imaginary impedance, against the real impedance. Basically, the electrical resistance is shown as a function of compression and resistance levels with the smaller the semicircle the better based on level of resistance. The trends demonstrate a semicircular shape, higher compression such as 80% combined with higher HFR 208 $m\Omega \cdot cm^2$ will show larger semicircles which yields higher impedance as compared to lower HFR 147 $m\Omega \cdot cm^2$ or lower compression which generated smaller semicircles that improved charge transfer at both the cathode and anode, facilitating improved overall efficiency by lowering energy losses through the system [29].

This article investigates the performance of a PEM water electrolyzer with ultralow loading of IrO₂ anode 0.1 mg/cm² electro-deposited loading and a Pt/C cathode 0.4 mg/cm². This research focuses on the effect caused by variation of operating temperature, pressure, water flow rate, and the thickness of gas diffusion layer, all at elevated conditions. Important findings are that at a temperature of 120°C and a pressure of 2.5 bar, the current density reaches a value of 1.79 A/cm² at 1.6 V, which is comparable to using a powder anode loading of 2.0 mg/cm². The condensation of water vapor during pressurization leads to a decrease in concentration losses and, also, there is an optimal thickness to balance between mechanical support and diffusion. The operating cell temperature was 120°C in primary, as the temperature was increased to maximize performance. Regarding EIS and KPI Analysis, the EIS measurements followed Nyquist plots that showed the charge-transfer resistance decreases with in response time to increase pressure, with the appearance of mass-transport arcs from low to high frequency conditions. Cell Pressure is 1-2.5 bar, but close to the 1 - 5 bar, pressurization is used to improve performance because it increases vapour density. The membrane is a Nafion 212 (N212, 50 µm thick). The Flow Rate is set between 0.8-3.2 ml cm⁻² min⁻¹ (for example, in experimental runs at 6.25 cm² an effective flow rate could be 15 ml/min). Pressure at Cathode and Anode are the same pressure on both sides (1-2.5 bar). Gas Diffusion Layers used are Toray carbon papers, the thicker DLs had the highest R_{ohm} , minimal impact at high pressure. Polarization curves indicate that current increased under changing pressure and flow. The EIS was used to analyze overpotentials for example, can see smaller arcs with pressure and diffusion branches at lower flow [41].

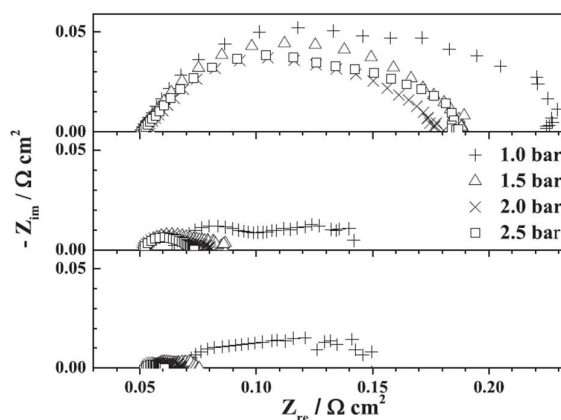


Figure 20 Nyquist plots of electrochemical impedance spectroscopy for the PEMWE single cell with the IrO₂/CP anode at various pressures [42].

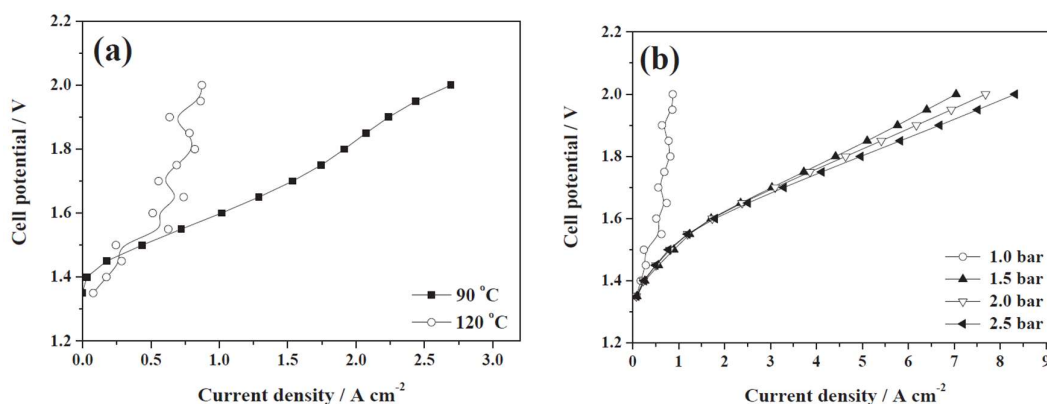


Figure 21 Polarization curves of the PEMWE single cell with IrO₂/CP (a) at 90 and 120 °C (atmospheric pressure) and (b) at various pressures from 1.0 to 2.5 bar (120 °C) [42].

2.2.2 Effects of Compression on Carbon-Based GDLs in PEM Electrolyzers

Carbon-based gas diffusion layers (GDLs), such as carbon papers and carbon cloth, because of their mechanical compliance and responsive pore structure under pressure of the assembly, are widely used on the cathode side of the PEM electrolyzers. Carbon felts under 0.5 – 3 MPa steady-state conditions, showing that moderate compression (1 – 1.5 MPa) mitigates the contact resistance by (> 2.5 MPa) while increasing conductivity; on the other hand, high compression decreases permeability by a factor of 5, resulting in mass transport losses of 0.2 V at 2 A/cm². Similarly, the tests in high-pressure conditions (10 – 30 bar) at 2 MPa demonstrated stable conductivity. Furthermore, at 3 MPa, the losses increased by water flooding. A graph shows that permeability drops from $8 \times 10^{-12} \text{ m}^2$ at 0.5 MPa to $3 \times 10^{-13} \text{ m}^2$ at 3 MPa, with an increase of pressure drop by 15% and an increase of overpotentials by 0.15 V at high compression [37].

The experimental findings show the balance of the compression effects. Between the GDL, catalyst layer, and bipolar plate, moderate clamping pressure mitigates interfacial contact resistance between these three layers and improves ohmic performance [31], [32]. Borgardt et al., (2019) [31], demonstrated the high frequency resistance and, in commercial cells, the increased bolt torque. However, microstructural densification, reducing porosity and effective pore, which blocks gas escape routes, happens in excessive compression [33], [42]. this leading to a higher mass transport resistance and larger impedance arcs with low frequency [33]. Cyclic compression tests (1 – 3 MPa) under dynamic conditions further define fiber breakage after 1000 cycles and reduce porosity by 10% and efficiency by 15% [37].

Also, compression effects on hydrophobic treatments and durability of the materials. Carbon-based GDLs with different PTFE loadings illustrate different tolerances. Higher PTFE maintains water management under moderate compression but intensifies pore blockage at excessive levels [24], [26]. Furthermore, the cyclic compression precipitates degradation of PTFE binders and adhesion of the MPL, enhances contact resistance, and causes performance decay after several hundred cycles [35]. These studies, put under study the essential need for

careful assembly torque control [33], [42]. For applications of the cathode with low corrosion, optimization of PTFE and MPL thickness can change the ideal compression to balance electrical contact and the capacity of the mass transport.

2.2.3 Effects of Compression on Titanium-Based GDLs in PEM Electrolyzers

Titanium GDLs are examined for compression in PEM electrolyzers. It confirmed durability with titanium felt, mesh, and sintered PTLs usually used on the anode side of the PEM electrolyzers to prevent oxidation and corrosion. The structure of the titanium is mechanically harder and has a different response to the compression in comparison with carbon substrates [31]. in a steady-state condition tested titanium under 1 – 4 MPa, finding 1.5 – 2 MPa mitigated contact resistance by $40 \text{ m}\Omega \cdot \text{cm}^2$, lowering voltage by 0.05 V at 1 A/cm^2 , but > 3 MPa increased overpotential by 0.18 V. This study demonstrated micro-cracks after 2000 cycles and reduced durability by 18%. A high pressure (20 bar) at 2 MPa was tested and maintained performance but increased losses by 10% [34], [43].

The experimental findings show that the properties of titanium PTLs are unique. Titanium PTLs demonstrate a higher mechanical strength but contact variations. In comparison with carbon materials, Ti PTLs can support higher compressive loads. Nevertheless, the behaviour of the electrical contact strongly depends on coating characteristics and production procedures [21], [22]. Another study examined *TiH* (titanium hydride) treatments that maintained performance under compression [22]. Coatings enhanced durability and mitigated contact resistance, with $\text{IrO}_2/\text{TiO}_2$ or $\text{Au} - \text{TiO}_2$ coatings on Ti PTLs decreasing charge transfer resistance and increasing reliability under compression cycles [27], [28].

The EIS improved after coated PTLs were compressed and used during the time. The effects of the hydrogen permeation and pressure are also significant. Another study showed the permeation of the gas under the high pressure and current density and illustrated the coupling between compression, pressure, and crossover [44], [45]. Under the compression they can maintain better than meshes, but the costs and the complexity of the manufacturing will increase [21], [22], [27], [34].

The experimental studies on titanium hydride PTLs; and another one on coated titanium PTLs, the important outcomes are PTLs for reducing contact resistance allow more excessive compression while handling the anode in corrosive conditions. However, to avoid the negative effect of the transport and to decrease crossover at high compression, coatings and the structure of the pore must be optimized [22], [27], [34], [44].

2.2.4 Hybrid and Novel Materials

Hybrid carbon-titanium gas diffusion layers (GDLs) and porous transport layers (PTLs) are the samples of hybrid and novel materials. By combining durability, conductivity, corrosion resistance, and improved mass transport, hybrid and novel materials have the potential to

increase the performance of the proton exchange membrane (PEM) electrolyzers. Experimental studies tested hybrid carbon-titanium GDL under steady state compression conditions, and the compression range was from 1 to 2.5 MPa. At 1.5 MPa these tests showed that the contact resistance was reduced by $30 \text{ m}\Omega \cdot \text{cm}^2$. It leads to an improvement of efficiency by 10% at a current density of 1.5 A/cm^2 . Higher pressure tests at around 15 bar and 2 MPa showed that transport properties will increase compared to titanium alone [43], [46].

Hybrid materials consist of carbon metal composites, coated PTLs, pore-graded substrates, and novel manufacturing like powder rolling for ultra-thin titanium PTLs, targeted to optimize these important properties. For example, Liu et al. (2022) [28], showed that hybrid coatings like $\text{Au} - \text{TiO}_2/\text{Ti}$ Under compression conditions improve both ohmic and mass transport losses by increasing conductivity and protection from corrosion. Also demonstrated that these coatings improve the behaviour of the bubble separation and mitigate high-frequency impedance. Identically showed under different compression levels, enhancing two-phase transport with Lettenmeier et al. (2017) [24], which tested a better gas removal and reduced the losses of the mass transport at high current densities for graded structures [24], [29]. According to the manufacturer, the novel methods, like ultra-thin powder-rolled titanium PTLs, reduce ohmic losses and maintain mechanical stability. In the experimental tests, they confirmed their ability to withstand assembly compression [21].

Representative experiments showed that in this field, Lettenmeier et al., (2017) [24], concentrates on pore-graded GDLs; Liu et al., (2022) [28], tested $\text{Au} - \text{TiO}_2/\text{Ti}$ hybrid coatings; Zhang et al., (2025) [21], highlighted the fabrication and testing of ultra-thin Ti PTLs; and Stiber et al., (2022) [13], investigated experimental compression with steel-based low-cost designs. The practical concept of these materials (hybrid and graded structures) is the ability to work under compression conditions, maintaining mass transport and enhancing contact resistance. Furthermore, these improvements come with an increased cost and complexity of the manufacturing to ensure sustainable performance.

2.2.5 Moderate and High Compression Comparison

Experimental tests showed that moderate compression (1 – 2 MPa), by decreasing contact resistance ($40 - 50 \text{ m}\Omega \cdot \text{cm}^2$), improved the performance. However, Frensch et al. (2018) [37], said that by maintaining porosity for transport, efficiency is enhanced by 8 – 10% at 1 A/cm^2 . On the other hand, high compression ($> 3 \text{ MPa}$) increases losses by $0.15 - 0.2 \text{ V}$ [43]. Moderate compression balances benefits for carbon GDLs, while hybrids increased the range to 2.5 MPa [46]. According to the moderate compression, the benefits are lower contact resistance, better mechanical contact, and more uniform distribution of the current [30], [31]. On the other hand, there is a risk of minor densification if material is very compliant, but usually with sustainable bounds [24].

Table 1 shows the tests of compression on GDLs in a PEM water electrolyzer, demonstrating conditions and material effects.

Table 2: Compression tests in PEM Electrolysis.

Material	Compression (MPa)	Key Findings	Impact on KPIs	Source
Carbon GDLs	0.5-3 (steady/cyclic)	1.5 MPa reduces resistance by $50 \text{ m}\Omega\cdot\text{cm}^2$; $>3 \text{ MPa}$ increases losses by 0.15 V	Efficiency +8%, durability -15% at high	[31]
Titanium GDLs	1-4 (steady/cyclic)	2 MPa lowers voltage by 0.05 V ; $>3 \text{ MPa}$ increases overpotential by 0.18 V	Efficiency +5%, transport losses at high	[34]
Hybrid GDLs	1-2.5 (steady/cyclic)	1.5 MPa reduces resistance by $30 \text{ m}\Omega\cdot\text{cm}^2$; minimal porosity loss	Efficiency +10%, stable durability	[30]

The tests on GDL compression highlighted the gaps in dynamic conditions. But moderate compression pressure ($1 - 2 \text{ MPa}$) decreases resistance [37], [46].

2.3 The Role of KPIs in Compression Effects

The key performance indicators (KPIs), namely cell voltage, energy efficiency, hydrogen production rate, and durability metrics, provide quantitative performance metrics adopting components and provide guidance to optimize components for target high current density operational assessment [40]. This section explores how the KPIs, observed by several methods, including electrochemical impedance spectroscopy (EIS), may be impacted by compression, as separated amongst efficiency, productivity, and durability metrics [47].

2.3.1 Criteria of the Efficiency

Efficiency metrics, including energy efficiency and cell voltage, measure how efficiently electrical input is converted to the chemical energy of hydrogen, which is very sensitive to the effects of compression. Energy efficiency is usually calculated using the higher or lower heating value of hydrogen (HHV , 141.8 MJ/kg , LHV , 120 MJ/kg):

$$\eta = (HHV \cdot \dot{m}_{H_2}) / (\text{Electrical energy input}) \\ = (39.4 \text{ kWh/kg} \cdot \dot{m}_{H_2}) / (V_{\text{cell}} \cdot I)$$

Where ($\dot{m}_{H_2}$) is the hydrogen production rate (kg/s), (V_{cell}) is cell voltage (V), and (I) is current (A) [48]. Cell voltage provides a direct measure of efficiency and includes the contributions of the standard potential (1.23 V), activation overpotential (η_{act}), ohmic overpotential (η_{ohm}), and mass transport overpotential (η_{mt}):

$$V_{\text{cell}} = E^0 + \eta_{\text{act}} + \eta_{\text{ohm}} + \eta_{\text{mt}}$$

Compression affects these overpotentials by changing contact resistance and transport properties [32].

Mo et al., (2016) [40], experiments indicated that moderate compression (1.5 MPa) on carbon GDLs decreased by 50 mV at 1 A/cm², and the energy efficiency improved from 65% to 72% (higher heating value) because of lower contact resistance, with high level of compression (3 MPa) had increased by 0.2 V which decreased the overall efficiency to 60% due to loss of porosity and accumulation of gas. Similarly, Salehmin et al. (2022) [47], showed titanium PTLs at 2 MPa resulted in a cell voltage of 1.85 V at 1 A/cm² for 70% efficiency and increasing the compression to 4 MPa produced a cell voltage of 2.05 V for an efficiency of 64%, resulting from limited water flow.

High-pressure operation further considered in Bin et al., (2024) [45], demonstrated that the effects of compression are even further increased at higher pressures 30 bar, so that for an initial compression of 2 MPa maintained an efficiency of 68%, but an increase to 3.5 MPa decreased efficiency to 62%. Each of the studies described above highlight that moderate compression optimize efficiency by balancing ohmic and transport losses. This will be an important consideration in the optimization experiments presented in this thesis.

2.3.2 Productivity of The Hydrogen

Gas productivity, expressed as an instantaneous amount of hydrogen being produced (e.g. in mol/s or kg/h), is a very important KPI for measuring PEM electrolyzers, linearly related to compression through altering mass transport. Production rates are detailed by Faraday's law:

$$\dot{m}_{H_2} = (I \cdot M_{H_2}) / (n \cdot F)$$

where ($\dot{m}_{H_2}$) is the molar mass of hydrogen (2g/mol), (n) is the number of electrons (2), and (F) is Faraday's constant (96,485 C/mol). Since compression has an effect on the current density (I) of the PEM electrolysis through mass transport issue, we can see that gas productivity would be significantly affected [44].

Bernt et al. (2020) [44], looked at experimenting at 1 A/cm² on titanium PTLs under 2 MPa compression comparing productivity/value for a hydrogen production rate of 0.05g/s for each 100 cm² electrode based on electrolysis site area; when increasing compression to 3 MPa, the hydrogen production rate dropped by 10%, due to gas bubbles remaining trapped in the catalyst layers - restricting access for water to the catalyst. Tjarks et al. (2018) [48], investigated high-pressure electrolysis (10 – 30 bar) with 1.5 MPa compression on carbon GDLs, and showed stable hydrogen production rates of 0.06 g/s; however, at a compression of 3 MPa, hydrogen production was reduced to 0.04 g/s, due to the 15% reduction in porosity and 20% increase on pressure drop. These findings are in line with CFD models presented in Weber et al., (2023) [49], which predicted a 12% decrease in production rate at a similar high compression, corresponding closely to experimental data but was within 3% when compared to tentative predictions of the models.

Suermann et al., (2017) [50], also evaluated the use of electrochemical hydrogen compression. The authors indicated that moderate compression (1.8 MPa) maximized production due to less gas crossover. However, the compression increased gas crossover by about 5%, and ultimately production was reduced.

2.3.3 Durability Criteria

Durability measures can include voltage drift, and lifetime, to quantify the stability of PEM electrolyzers, under compression. This is noteworthy for real-world applications where product lifetimes of 50,000 – 80,000 hours are desirable [47]. Voltage drift is seen as the percentage increase in the cell voltage as a function of time (e.g., < 1% per 1000 hours) and to assess degradation within GDLs, PTLs, and/or catalysts. Compression can factor in the durability component due to the mechanical stress and pore volume, which are both altered based on the physical characteristics of the component or layer.

In reports provided from Martin et al., (2022) [32], they indicated that carbon GDLs when oriented under 1.5 MPa of compression exhibited a voltage drift of 0.5% per 1000 hours, and product lifetime remained around 40,000 hours, however as compression was increased to 3 MPa, voltage drift increased to 2% with degradation of the cell lifetime reducing to 25,000 hours. In Stähler et al., (2020) [34], titanium PTLs demonstrated better durability under compression with the associated degradation in component performance being a value of 0.3% (2 MPa) with a lifetime of 45,000 hours. These findings complement structural mechanics models in Srour et al., (2024) [39], which predicted a 15% loss in porosity at high compression, equal to a 10% loss in durability.

Salehmin et al. (2022) [47], reported that compacting to high pressure (30 bar) caused greater durability loss when the data were taken under compression. Also highlighted how 3 MPa of compression resulted in an 8% increase in hydrogen crossover, speeding up the damage to the membrane. This study shows how a range of compression even over 1 and up to about 2 MPa conserves durability.

2.3.4 Integration with EIS Studies

EIS is a key diagnostic tool for assessing the effects of compression on the cell performance, as it can resolve ohmic, charge-transfer and mass transport resistances which are directly related to KPIs. Table 2 summarizes KPIs and their relationships to each EIS study and shows what compression can influence in a measurable way.

Table 3: KPIs and EIS Studies in Compression Effects.

KPI	Description	Compression Effect	EIS Insight	Source
Energy Efficiency	% HHV (39.4 kWh/kg)	1.5-2 MPa: +10-12%; >3 MPa: -5-10% due to transport losses	Ohmic resistance drops at moderate compression	[47]
Cell Voltage	V at 1-2 A/cm ²	2 MPa: 1.83-1.85 V; >3 MPa: +0.2 V (transport losses)	Mass transport arc grows at high compression	[32]
Hydrogen Production	g/s or mol/s	2 MPa: 0.05-0.06 g/s; >3 MPa: -10-15% (gas entrapment)	Transport resistance increases at high compression	[34]
Voltage Drift	% increase per 1000 hours	1.5 MPa: 0.3-0.5%; 3 MPa: 1.5-2% (degradation)	Ohmic/charge-transfer resistance rises with time	[44]

Several earlier EIS studies, such as Martin et al., (2022) [32], report that moderate compression can reduce the ohmic resistance by $40 \text{ m}\Omega \cdot \text{cm}^2$, which ultimately relates to a percentage increase in efficiency. This study and others exhibit the consequences of high compression on the mass transport arc, which the Nyquist plots showed was associated with the formation of gas bubbles. The EIS data from Stähler et al., (2020) [34], illustrates the clarity to quantify what it meant to have a 15% increase in transport resistance under 3 MPa of compression and the relationship to distorted productivity losses presented by Bernt et al., (2020) [44]. Together these studies inform the way EIS data were used to assess the performance of the cell compression in this thesis.

2.4 EIS as Diagnostic Tool in Compression Studies

Electrochemical impedance spectroscopy (EIS) is a powerful diagnostic instrument to facilitate understanding of the impact of compression on proton exchange membrane (PEM) electrolyzer performance, due to its operation can distinguish between ohmic, charge-transfer, and mass transport resistance contributions. EIS is established through analysis of Nyquist plots and Bode plots, measuring resistance increments which respond to varying compression's, allowing researcher to discover the contributing factors to the cell operational performance [51].

2.4.1 EIS for Resolving Resistances

EIS investigates the impedance of a PEM electrolyzer through a small alternating current (AC) signal over a frequency range (typically 0.1 Hz to 100kHz), resulting in three resistance components: ohmic resistance (R_{ohm}), charge-transfer resistance (R_{ct}), and mass transport resistance (R_{mt}). Equivalent circuit models are applied, such as a Randles circuit with a parallel resistor and capacitor for charge transfer and a Warburg type element to represent mass transport:

$$Z(\omega) = R_{ohm} + (R_{ct}) / (1 + j\omega C_{dl} R_{ct}) + Z_W(\omega)$$

where ($Z(\omega)$) is the impedance, (ω) is angular frequency, (C_{dl}) is double-layer capacitance, and (Z_W) is the Warburg impedance for diffusion [52].

EIS was applied in combination with reference electrode measurements to quantify the contribution of resistances in a PEM electrolyzer under 1 – 3 MPa compression [52]. At 1.5 MPa, (R_{ohm}) decreased by $40\text{ m}\Omega \cdot \text{cm}^2$. In contrast, mass transport resistance increased by $30\text{ m}\Omega \cdot \text{cm}^2$ at 3 MPa, coinciding with an increase of 0.15 V cell voltage, in accordance with KPI losses in Martin et al., (2022) [32]. Maoulida et al., (2025) [53], have also applied EIS and studied dynamic operation which indicated that after 1000 hours of 1 – 3 MPa compression cycles (which reflects gas bubbles), the mass transport resistance had increased by 20%.

These studies illustrate that EIS has the sensitivity to investigating compression effects, with the identification of suitable compression levels (1 – 2 MPa) that provided max resistances and minimized transport losses are crucial aspects of the experimental design pursued in this thesis.

2.4.2 Interpretation of Nyquist and Bode Plots

Nyquist and Bode plots are valuable for the interpretation of EIS data when studying compression values, and they offer interpretations of resistance changes in a visual way without an accompanying fundamental explanation. The Nyquist plots have a semicircular high to mid-frequency section that represents ohmic resistance plus charge transport resistance on the x-axis real impedance and the y-axis (negative imaginary impedance) and a low-frequency tail that represents mass transport resistance. Compression typically results in decreased semicircle diameter (R_{ohm}) decreases; however, if significant, compression extends the low-frequency tail, the mass transport resistance increases [54].

In Kimmel et al., (2025) [55], Nyquist plots for a PEM electrolyzer stack evaluated under 2 MPa conditions exhibited an obviously compact ohmic and charge transfer resistance of $140\text{ m}\Omega \cdot \text{cm}^2$. However, the tail was longer for a 4 MPa test condition, indicating a greater mass transport resistance, due to gas bubble entrapment $35\text{ m}\Omega \cdot \text{cm}^2$ increased mass transport resistance. Bode plots which are the plots of impedance magnitude and phase angle versus frequency, characterize the underlying resistance behaviour of the components with specific frequency-related effects. For example, Maoulida et al., (2025) [53], when the device was evaluated at a pressure of 1.5 MPa, the high-frequency impedance (10 kHz) dropped by $30\text{ m}\Omega \cdot \text{cm}^2$, lower mass transport resistance of course, while the measured low-frequency impedance (0.1 Hz) was a $20\text{ m}\Omega \cdot \text{cm}^2$ increase assessed at 3 MPa, which further implies limitations related to transport. These plots offer quantitative equivalent values to help optimize cell performance, as they indicate isolated resistance contributions of all components gathered by each compression cycle.

2.5 Literature Gaps and Relevance to Present Work

Most literature adopts an isolated approach, examining compression, KPIs, or EIS independently, with few studies combining these to holistically address cell performance [47],

[53]. This section synthesizes these gaps, highlights the fragmented nature of current research, and articulates the novelty of this thesis. Moreover, these sections can integrate experimental studies of compression, KPIs, and EIS analysis to enhance cell performance for enhanced efficiency, productivity, and durability.

2.5.1 Gaps in Compression-Related Research on PEM Electrolyzers

The literature reviewed a tendency to study compression effects in isolation, limiting the understanding of their interaction with other performance factors. Studies like Lettenmeier et al., (2017) [24] and Bautkinova et al., (2023) [22], focus primarily on material properties such as cell performance, GDLs, titanium hydride PTLs without linking these to long-term performance metrics under dynamic compression. Other experimental studies on carbon GDLs, titanium PTLs, and hybrid materials emphasize short-term compression effects (1-4 MPa) on contact resistance and transport but rarely explore cyclic compression typical of renewable-driven operation [43]. For example, Frensch et al., (2018) [43], reported a 15% durability loss in carbon GDLs under cyclic compression but did not integrate EIS to recognize underlying mechanisms.

Weber et al., (2023) [49], accurately predicted a 10% efficiency loss at 3 MPa, yet differences of 5-10% in durability predictions highlight limitations in simulating transient compression cycles [39]. In other section, KPIs, efficiency, hydrogen productivity, and durability show compression's impact for example 10-12% efficiency gains at 1.5-2 MPa but are typically evaluated in steady-state conditions. Besides, few studies like Martin et al., (2022) [32], addressing high-pressure or dynamic operation. As can be seen, EIS has ability to resolve ohmic, charge-transfer, and mass transport resistances, but most studies focus on static compression, along with limited exploration of EIS under cyclic loading [51].

This fragmented approach focusing on either compression, KPIs, or EIS creates a gap in understanding how these factors interact under real-world conditions, also in this fragmentation, electrolyzers operate with fluctuating power inputs. For example, Bernt et al., (2020) [44] and Stähler et al., (2020) [34], link compression to productivity and durability losses. However, they do not use EIS to quantify resistance changes over time. In the same way, Zuo et al., (2025) [56], models EIS spectra accurately for steady-state compression but underestimates dynamic degradation by 10-15%, as mentioned before in [57]. This absence of integration limits the development of GDL and PTL designs optimized for dynamic, high-current-density operation, a critical need for scalable hydrogen [47].

2.5.2 Insufficient Integration of Compression, KPIs, and EIS in PEM Electrolyzer Research

Few studies integrate compression, KPIs, and EIS to provide a comprehensive view of cell performance. Maoulida et al., (2025) [53], is an obvious exception, using EIS to study dynamic compression cycles (1-3 MPa) and adding a 20% increase in terms of mass transport resistance

to a 10% productivity drop, consistent with Bernt et al., (2020) [44]. However, it focuses more on short-term cycles (1000 hours) and does not explore long-term durability (>50,000 hours) [47]. In a similar case, Böhre et al., (2024) [52], combines EIS with reference electrode measurements to quantify ohmic and transport resistances under 1.5-3 MPa. This achieves to a 5% efficiency improvement at 2 MPa but it does not address hybrid materials or dynamic operation beyond 2000 hours. Other integrated efforts, such as Kimmel et al., (2025) [55], examines stack degradation using EIS. Moreover, the author said a 15% increase in charge-transfer resistance at 4 MPa, but there is not a connection to compression's impact on KPIs like hydrogen production rate. These studies demonstrate the potential of integrated approaches but are limited in scope, typically focusing on specific materials or short-term performance, leaving a gap in comprehensive, long-term studies under dynamic conditions [53].

2.5.3 Novelty of the Present Work

This thesis addresses these gaps by integrating experimental studies of the effects of the compression on the cell performance, KPIs, and EIS to optimize cell performance in PEM electrolyzers. Unlike most literature, which separates compression effects, KPIs, or EIS diagnostics, this work combines these aspects in order to enhance cell performance [52], [53]. The novelty lies in the integrated experimental framework by combining the effect of the compression on the cell performance tests, KPI measurements (e.g., cell voltage, polarization curve, durability (extrapolation to 1000 hours), and EIS diagnostics to assess cell performance, unlike the other approaches in [34], [49]. This thesis tried to resolve the contributions of the resistances for example ohmic resistance, charge transfer resistance and mass transport resistance and correlating these with KPIs, this thesis provides a comprehensive dataset. For example, experiments will build on by applying EIS across a range of compression levels and materials to quantifying improvements in cell performance.

Chapter 3. Experimental

3.1. Electrochemical test bench

The experimental tests were performed on a PEM electrolyzer. The test station is located in HYSYLAB which is a laboratory for the research on hydrogen production technologies with different test benches at the Environment Park in Turin.



Figure 22 Environment park- HYSYLAB.

3.1.1. Explanation of the test bench

The experimental test bench is designed by engineering company TECNODELTA S.r.l to characterize the performance of electrochemical systems used for hydrogen production. Its purpose is to test both single electrolytic cells and small multi-cell assemblies (stacks). Hydrogen is generated by supplying the devices with demineralized water and electrical power. The characterization process includes measuring voltage–current (polarization) curves and performing electrochemical impedance spectroscopy (EIS) to analyze the cells. These analyses make it possible to evaluate cell performance by examining the irreversible phenomena that occur during operation. Furthermore, the devices will undergo long-duration tests to identify possible degradation mechanisms. The test bench must support experiments on low-temperature electrolytic devices up to 150 °C and maximum operating pressure of 25bar. Figure 3.2 shows the test bench used to conduct these experiments.



Figure 23 Test Bench.

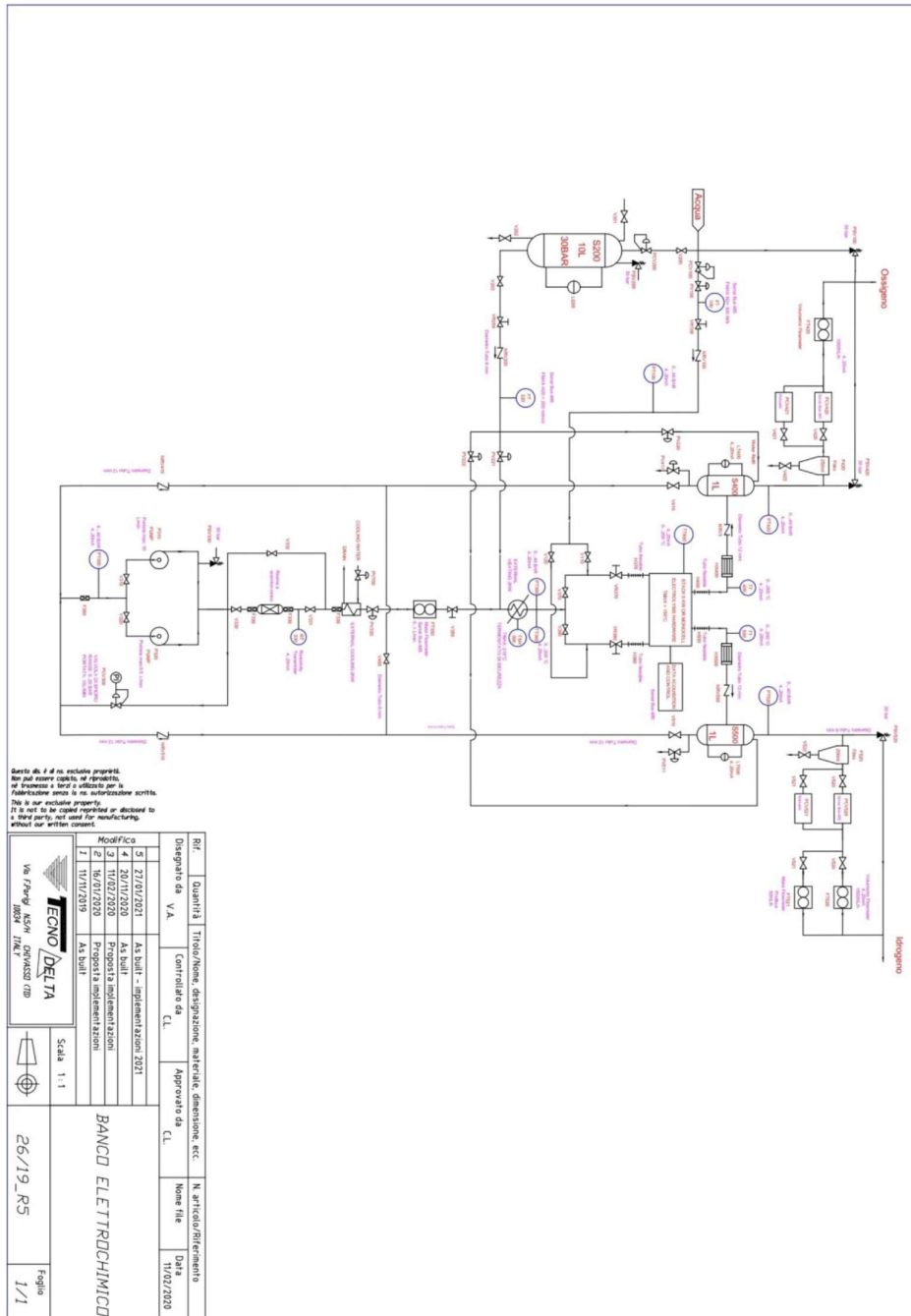


Figure 24 P&I of test bench.

The external lines are the main components of the test bench, one delivering nitrogen and the other providing compressed air. Nitrogen is used for purging and maintaining system pressure, whereas compressed air ensures proper operation of the pneumatic valves. Because the electrolysis process requires a large volume of demineralized water, the setup includes a 10-liter storage tank. This water tank feeds the system through two internal loops, one to the cathode side and the other to the anode side.

After leaving the tank, the water undergoes additional purification via ion-exchange resins located downstream of the pumping section, ensuring conductivity remains below 1 $\mu\text{S}/\text{cm}$. The tank supplies the electrochemical cell through two separate circulation loops: one feeding the anode and one feeding the cathode. Water directed to the cathode loop is used solely for thermal management rather than for the electrolysis reaction itself.

At the outlets of both the anode and cathode, the resulting two-phase mixtures must be separated. This occurs in two stages. First, the streams are cooled using condensers positioned immediately after each outlet. These condensers have a tube-in-tube configuration, with the hot process stream flowing through the inner tube and mains-supplied cooling water circulating in the surrounding annulus. The second stage is the actual phase separation, carried out in dedicated gas-liquid separator vessels. Each separator is a one-liter tank equipped with two outlets: an upper outlet for the gas-rich phase and a lower outlet for the liquid-rich phase. The separators are installed at the same height to ensure stable control of the demineralized water inventory. To further enhance separation efficiency, each separator is connected to an additional 250 mL tank positioned above it.

Water circulation is provided by a volumetric pump able to work with a maximum flowrate of 10 L/min. A second volumetric pump with a higher maximum flow rate is included for redundancy and to support stack-level operation. Before entering the cell, the pumped water is heated to the required operating temperature using an external electric heater. Heat transfer to the fluid is achieved through a plate heat exchanger, and temperature stability is maintained by a PID controller. To maintain and regulate the temperature and pressure values in the system there are two pressure controllers, anodic and cathodic one on each side, conductivity prob, pressure transducers, mass flow meter for demineralized water and finally valves and temperature thermocouples.

Overall control of the test bench is managed through a LabVIEW-based system (National Instruments, LabVIEW 2009). This interface communicates with the power supply, measurement instruments, and safety hood, it allows to control voltage and current. It also records and stores data, which can be displayed in a user-friendly format. The LabVIEW environment includes both a front panel and a block diagram, as illustrated in Figure 25.

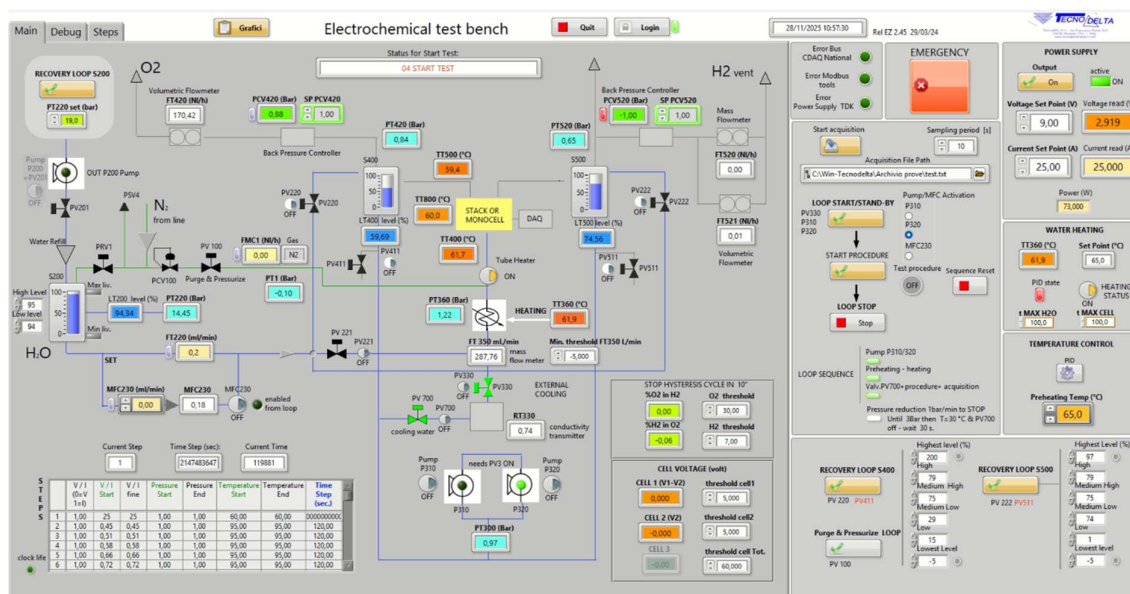


Figure 25 Software interface.

The front panel shows real-time values of all operating variables along with the data acquired from the system. Water can be delivered to either the anode or the cathode, although most PEM electrolyzers use an anode-side water feed since water is consumed at that electrode. In the test bench described here, water is supplied to both the anode and cathode circuits in order to replicate and study the operating conditions typically observed in industrial PEM electrolyzers.

3.1.2. Electrochemical cell (Test 1)

The PEM cell under study is a single squared cell geometry and active area of 25 cm² cell. The MEA was composed by Nafion® 115 membrane for electrolyse applications. The surface of the Nafion is coated with two catalytic layers, a thin layer of carbon supported platinum with 3 mg/cm² Pt (Pt on advanced carbon) at the cathode for HER and a thicker layer of unsupported iridium with 3 mg Ir /cm² at the anode for OER; it is reported in Figure 26.

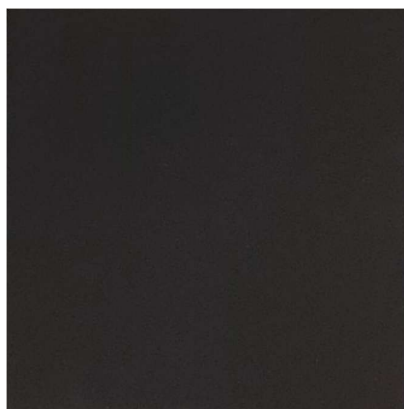


Figure 26 MEA composed by Nafion 115.

It was sandwiched between Ti mesh GDL of 180 μm thickness on the anode side and Freudenberg H23C6 carbon paper GDL with MPL of 250 μm on the cathode side. These are reported respectively in Figure 27.

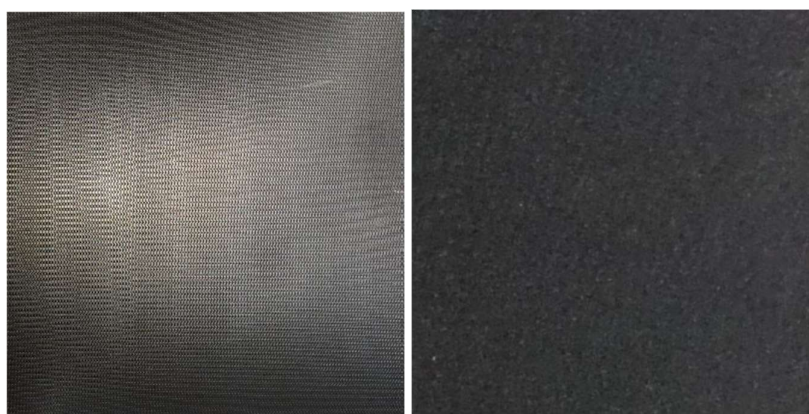


Figure 27 left: Titanium mesh GDL – right: H23C6 Carbon paper GDL.

3.1.3. Electrochemical cell (Test 2)

The MEA for the second test was composed by Nafion® 115 membrane for electrolyse applications. The surface of the Nafion is coated with two catalytic layers, a thin layer of carbon supported platinum with 1 mg/cm² Pt (Pt on advanced carbon) at the cathode for HER and a thicker layer of unsupported iridium with 2 mg Ir /cm² at the anode for OER; it is reported in Figure 28.

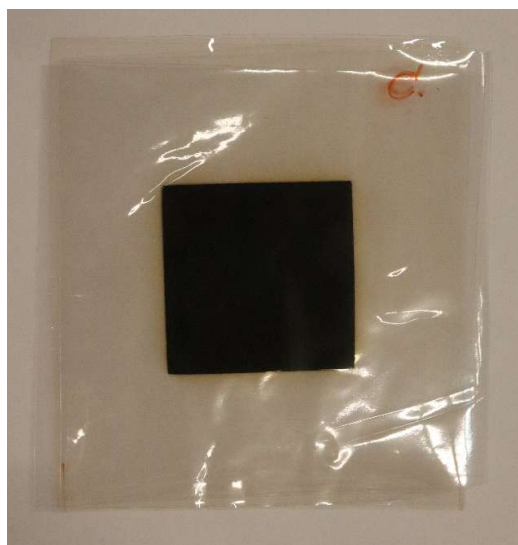


Figure 28 MEA composed by Nafion 115.

The MEA for the second test, was sandwiched between another kind of GDL which was Titanium Fiber Felt GDL or Sintered Titanium Fiber GDL of 0.25mm thickness with 60-70% porosity on the anode side and Freudenberg H23C6 carbon paper GDL like the first test with MPL of 250 μm on the cathode side. These are reported respectively in Figure 29.



Figure 29 left: Titanium Fiber Felt GDL – right: H23C6 Carbon paper GDL.

The electrolysis cell fixture used in this work is the quickCONNECTfixture FC25/125 LC 8 bar supplied by balticFuelCells GmbH. The device consists of a pneumatic-actuated housing that maintains the required contact pressure on the active area and accommodates interchangeable cell blocks without reconnecting media or electrical lines. The active components are mounted between graphite and titanium flow-field plates, while high-current electrical connections are provided through Multi Contact terminals with separate sense lines for ohmic-loss-free voltage measurement. All conductive parts are electrically insulated from the external frame, and the end-plate structure machined from a robust metal alloy ensures uniform compression and sealing during operation. The fixture integrates dedicated ports for media supply, heating/cooling circuits, and temperature sensing, and it is rated for operation up to 8 bar and 90 °C. The complete assembly of the quickCONNECTfixture FC25/125 LC 8 bar used for PEM/AEM electrolysis testing is shown in Figure 30.

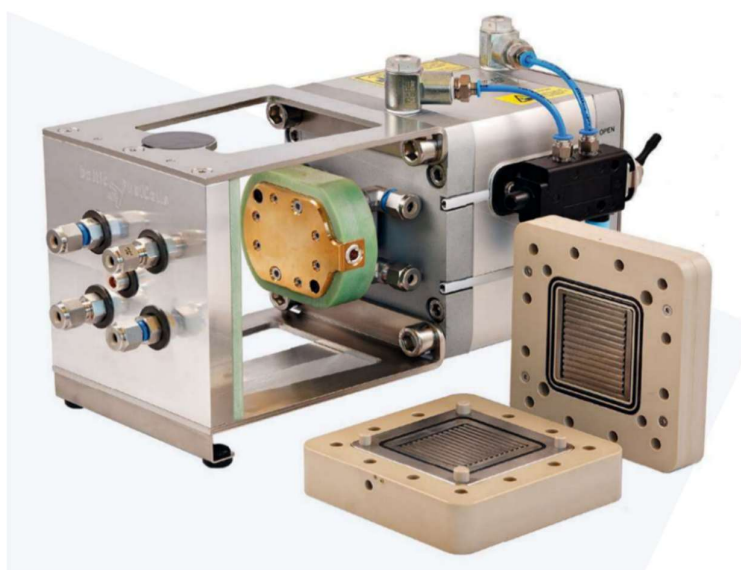


Figure 30 qCf FC25/125 LC 8bar.

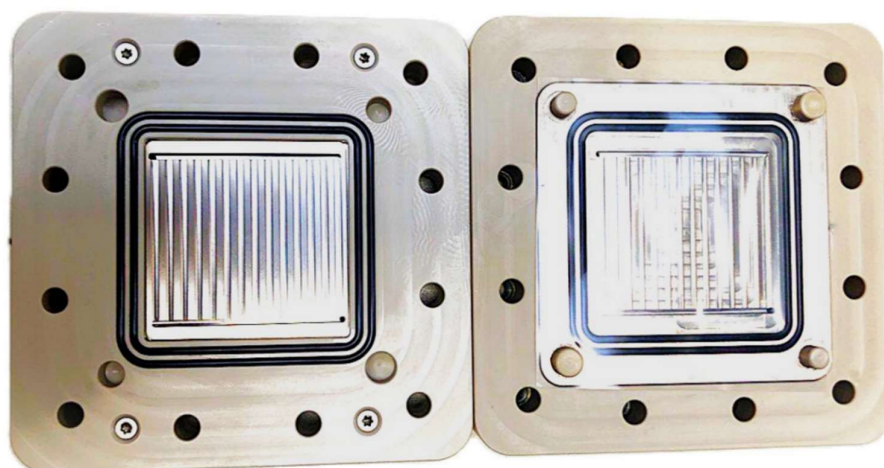


Figure 31 Cell housing.

3.1.4 The quickCONNECTfixture preparation of the Setup

Before operating the quickCONNECTfixture FC25/125 LC 8 bar, the entire setup must be prepared to ensure safe and reproducible electrolysis testing. The cell block is first assembled with the selected internal components membrane, GDL/PTL, and flow-field plates and positioned inside the fixture frame, which allows insertion without reconnecting media or electrical lines. All media supply tubes for water, gases, and cooling must be connected to the dedicated 6 mm quick-coupling ports, ensuring electrical insulation of all metallic lines. The heating/cooling circuit is filled with non-conductive fluid, and the temperature sensor is inserted into the dedicated 1.5 mm port. Electrical connections are established through the high-current Multi Contact terminals, while the sense leads are attached to the gold-plated measurement plugs to enable ohmic-loss-free voltage monitoring. Finally, the pneumatic regulator is connected to the compressed-air line, and the desired contact pressure on the active area is set using the pressure-control system. Once all connections are verified and the media are preheated to the operating temperature, the fixture is ready for electrolysis testing.

3.1.5 Compression Pressure Definition in the quick Connect Fixture

To ensure controlled and reproducible mechanical compression of the PEMWE, a quick Connect fixture (Baltic) was used. In this system, the applied compression force is generated by a pneumatic cylinder, meaning that the pressure set on the air regulator (pneumatic pressure) is not identical to the pressure experienced by the cell. In fact, the pneumatic pressure is responsible for the pressure applied on the cell, in which the pneumatic pressure acts on the large cylinder area, and the resulting force is transmitted through the piston to the much smaller active cell area. Therefore, the actual cell compression pressure is calculated using the ratio of the cylinder area to the cell area.

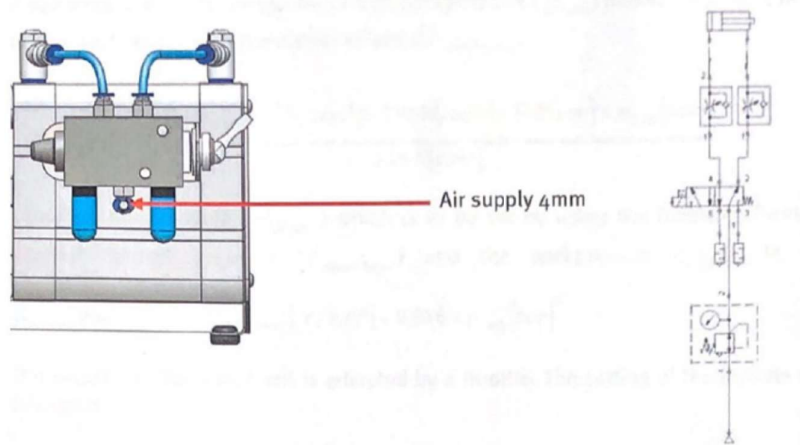


Figure 32 Pneumatics on the compressed air cylinder and circuit diagram.

The following diagram (Fig. 33) shows the relation of the contact pressure on the active cell area (surface pressure) and the adjust pressure in the air supply unit. The best working range for the contact pressure is at approx. 1-2 N/mm² as mentioned in manual of the Baltic cell.

$$F/A[\text{N/mm}^2] = \frac{\pi d^2}{40 \times A} [\text{mm}^2] \times p[\text{bar}] = \frac{\pi \times 125^2}{40 \times 2500} \times p[\text{bar}] = 0.49 \times p[\text{bar}]$$

F/A =Force per active area, A = Active area, d =cylinder diameter, p =inlet pressure on cylinder (compressed air).

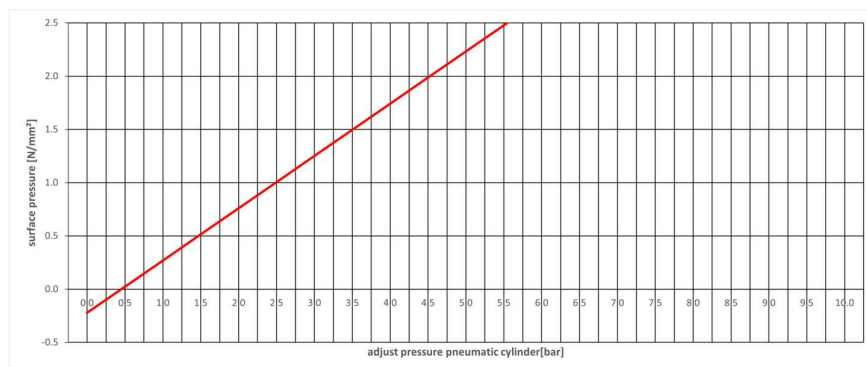


Figure 33 contact pressure depending on air pressure supply.

For operating at the cell block with backpressure (P_{cell}) it has been considered invoice for the contact pressure on the active cell area (F_{active}).

$$P_{cell}[bar] = \frac{4}{\pi} \times (F_{active}[N/mm^2] \times 25000[mm^2]) + (70^2)[mm] \times P_{cell}[bar]) / 15500[mm^2]$$

Calculating the pressure (P_{cell}) Which must be set by using the following formula. The desired contact pressure (F_{active}) and the backpressure (P_{cell}) has been entered in the formula.

$$P_{cell}[bar] = 2.05 \times F_{active}[N/mm^2] + 0.316 \times P_{cell}[bar]$$

The velocity of the piston rod is adjusted by a throttle. The setting of the throttle should not be changed as it was mentioned in manual.

This conversion ensures accurate and repeatable compression conditions during electrochemical testing. The calculated cell pressures corresponding to the pneumatic pressures used in this work are summarized in Table 4.

Table 4: Pneumatic Pressure vs. Cell Compression Pressure.

Pneumatic Pressure (bar)	Cell Pressure (bar)	Cell Pressure (kPa)	Cell Pressure (N/mm²)
2.4	9.55	955	0.955
3.3	14.46	1446	1.446
4.4	19.37	1937	1.937
4.9	21.82	2182	1.2182
5.0	22.31	2231	2.231

3.2. Cell assembly

3.2.1 cleaning

Before assembling the cell, it was necessary to thoroughly clean the main components that form the internal structure of the electrolyzer. In particular, the O-rings, the gas diffusion layers (GDLs), and the bipolar plates (BPs) must be cleaned carefully. Although several cleaning methods are available, cleaning with sprayed water was selected for this work.

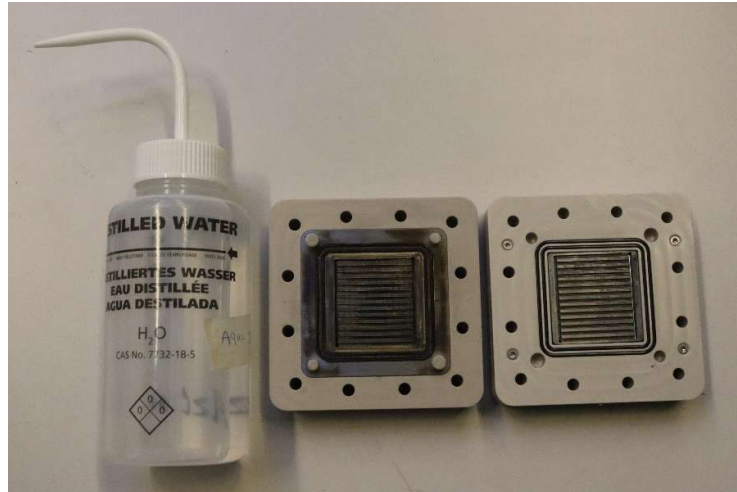


Figure 34 Cleaning procedure.

3.2.2 Assembly procedure

The procedure consists into cut the gas diffusion layers with the same dimensions of housing. The cut procedure is reported in Figure 35.

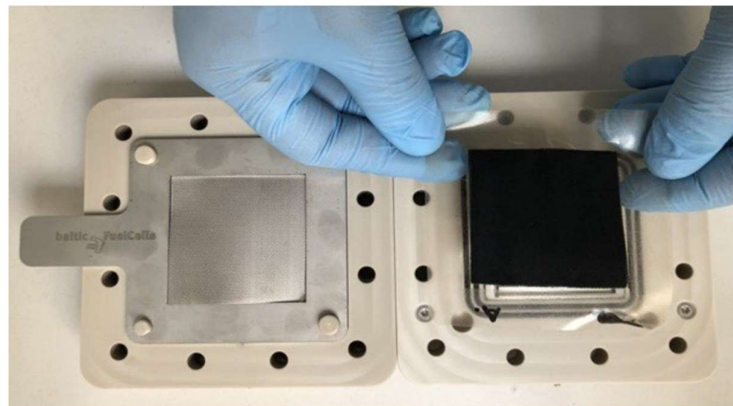


Figure 35 Cutting procedure.

At the end of the cleaning phase and checking that the force applied to the bolts is correct, the installation of the membrane (MEA) with the relative GDLs was foreseen both for the anode and the cathode side. However, the chosen method to make the installation was the "layered" method. It consists, into insert the different elements one by one on the chosen flow field plate.

In this case, the chosen flow field plate was the anode side, due to the fact that Titanium GDL was used and so to avoid its movement, it was better to place the other elements on its. Moreover, the bolts can be inserted from cathode end plates, and they are a guide to avoid the movement of all elements constituting the cell. Later, the GDL of anode was inserted. The MEA was placed above it.

For the installation of the MEA, an appropriate humidification, spraying demineralized water over the surface, is important to ensure that it adheres well to the structure. The GDL for cathode side was after installed over the MEA.

Placing these elements, the flow field plate, also humidified, was placed above them and the structure was closed by means of End Plates and with the help of 12 bolts, as said. The last procedure is reported in Figure 36.

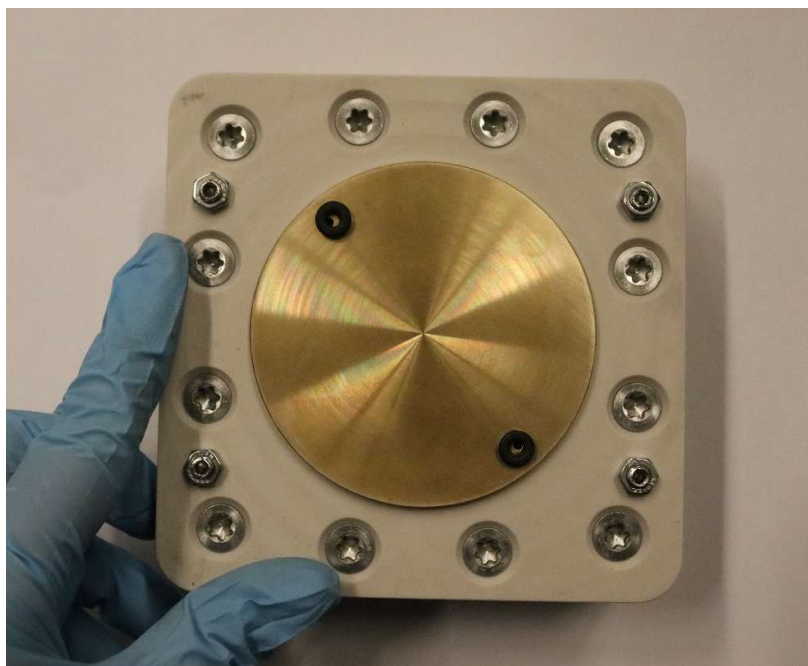


Figure 36 Housing closed and correctly tightened.

The bolts must be properly tightened, because, as said, incorrect compression of the cell leads to degradative effects on the cell. After closing, the cell was placed on the experimental bench and connected with the latter. In this sense, there are 2 tubes per anode side and 2 tubes per cathode side, two for inlet and two for outlet. The cables necessary to supply the electrical power were also attached to the cell. Finally, the cell heating systems were also installed, in order to reach the desired temperature and maintaining it in steady-state condition for carrying out the tests, as it is possible to see in Figure 37.

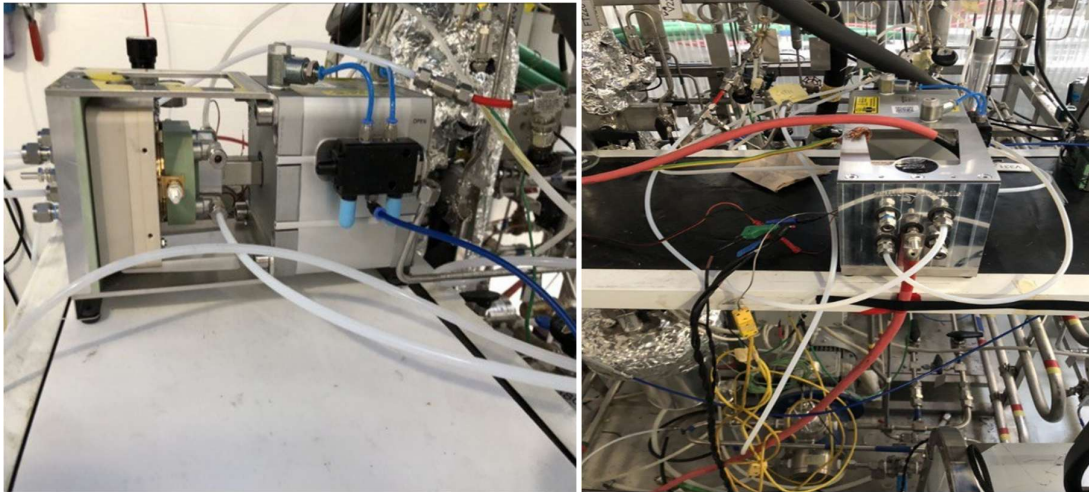


Figure 37 left: Housing placed on Test Bench - right: Housing placed on Test Bench with power supply connected.

3.2.3 Pressure regulator setup

After the first step of cell assembling, the pressure regulator is set in order to have a right control of cell pressure. The setting is made on the pressure regulator that is reported in Figure 38.



Figure 38 left: Air Pressure Regulator – right: assembled Baltic Fuel Cells to the test bench.

Chapter 4. ECM & EIS

4.1 Introduction of ECM & EIS

One of the most powerful diagnostic tools for resolving the individual physicochemical processes controlling the performance and degradation of proton exchange membrane (PEM) electrolyzer is Electrochemical impedance spectroscopy (EIS). By perturbing the system with a small-amplitude AC signal and analyzing the frequency-dependent response. EIS enables the separation of ohmic, charge-transfer, double-layer, and mass-transport contributions to the overall cell impedance [58]. This capability of EIS makes it particularly valuable for evaluating operating-condition effects, such as pressure, voltage, and humidity on the dynamic behavior of fuel cell components.

In PEM electrolyzer variations in compression and applied voltage can significantly affect reaction kinetics, water management, and gas diffusion. Each of them has a different effect on the impedance spectrum. For example, increasing pressure typically enhances reactant availability and reduces mass-transport limitations. On the other hand, changes in cell voltage influence the balance between activation and ohmic losses. These effects show as shifts in the high-frequency intercept, changes in the diameter of the mid-frequency semicircle, and modifications in the low-frequency tail associated with diffusion processes.

Equivalent circuit modeling (ECM) is widely suggested to quantitatively interpret these spectral changes. Circuits incorporating resistive elements, constant phase elements (CPEs), and Warburg-type diffusion components provide a physically meaningful representation of the electrochemical and transport phenomena occurring within the cell. Instead of ideal capacitors the use of CPEs accounts for surface heterogeneity and non-ideal double-layer behavior. Warburg elements capture finite-length or semi-infinite diffusion effects in the gas diffusion layer and catalyst layer.

In this study, EIS measurements were performed under multiple operating conditions, including pressures of 2.4 to 5 bar and voltages ranging from 0 to 1.5 V. The corresponding impedance spectra were fitted using equivalent circuits that show the main processes at each condition. By comparing the extracted parameters like ohmic resistance R_s , charge-transfer resistance R_1 , double-layer behavior (CPE1), and diffusion-related elements (CPE2, Warburg) the influence of operating conditions on the electrochemical performance of the PEM is systematically evaluated. This section provides an integrated explanation of these results, connecting the observed impedance features to illustrating the physical mechanisms and highlighting the operational sensitivities of the system [58].

4.2 EIS Test Equipment and Set-up

In order to perform EIS measurements, it is necessary to have, Potentiostatic or Galvanostatic or a load bank. Also, Frequency response analyser, FRA and finally DC power supply.

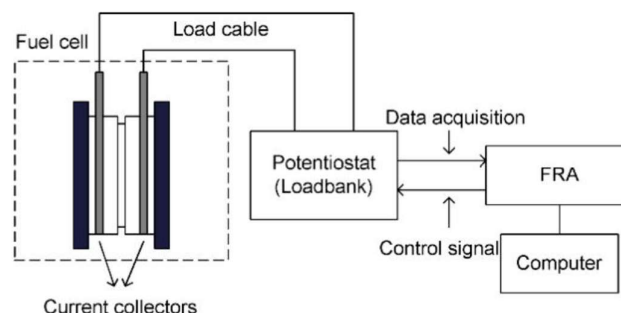


Figure 39 Schematic EIS measurement configuration.

Electrochemical impedance spectroscopy (EIS) is typically performed by using a four-electrode configuration consisting of a working electrode (WE), counter electrode (CE), and two reference/sense electrodes (RE1 and RE2). The WE (anode) measure the current through the cell. On the other hand, the CE (cathode) forces the cell with voltage or current. The two reference electrodes measure the potential difference. This procedure effectively forms a two-wire measurement system.

To minimize stochastic and systematic errors and obtain reliable EIS data, several setups are required:

- avoid parasitic currents in the load,
- keep the setup free from electromagnetic interference,
- use short, shielded, low-inductance twisted cables,
- ensure large cross-sections for WE and CE connections.

In an EIS experiment, the system is perturbed with a small AC signal and its response is recorded. The perturbation can be controlled by current (Galvanostatic mode) or by voltage (Potentiostatic mode). In galvanostatic mode, a constant DC current is applied, and the frequency response analyzer (FRA) superimposes an AC current perturbation through the potentiostatic. The resulting voltage response is analyzed to obtain impedance. In potentiostatic mode, the roles of voltage and current are reversed, yielding admittance.

Impedance or admittance at each frequency is calculated as the ratio of the output response to the input perturbation. By sweeping the perturbation frequency across the FRA's range, the full impedance spectrum is obtained.

The amplitude of the perturbation must remain small enough to ensure linearity, yet large enough to maintain an acceptable signal-to-noise ratio; excessively low amplitudes make it difficult for the FRA to distinguish true system response from noise.

	Test input	Symbol	Unit	Recommended value / range
	Number of perturbation frequency data per decade of frequency range	PPD	-	minimum 3
	Number of perturbation cycles during τ_{ACT} (τ_{ACT} precedes τ_{ACS})	n_{ACT}	-	minimum 2 ([†])
	Number of perturbation cycles during τ_{ACS} used to record the EIS spectra (τ_{ACS} follows τ_{ACT})	n_{ACS}	-	minimum 3
	Range of perturbation frequencies	f	Hz	10^{-2} – 10^{+6}
Potentiostatic method	load (voltage) perturbation peak-to-peak amplitude	U_{AC}	V	typically 5-10 mV _{rms} to be less than the thermal voltage, U_T (e.g. <30 mV at 80°C cell temperature) ([†])
Galvanostatic method	load (current) perturbation peak-to-peak amplitude	I_{AC}	A	0.5% – 5% of I_{DC} ([†])

Figure 40 Input parameters and settings for the EIS instrument [60].

The Nyquist plot is graphically represented by the AC impedance spectrum. Another type of plot is the so called Bode plot. These graphs show the tests output parameters gained from the EIS measurement.

Output	Parameter type	Measurement uncertainty	Sampling rate	
$U_{AC,k}(f)$	primary	$\pm 1\%$	$\geq 20 (\tau_{ACS}(f)/\tau_{ACT}(f))$	Galvanostatic method
$I_{AC,k}(f)$	primary	$\pm 1\%$	$\geq 20 (\tau_{ACS}(f)/\tau_{ACT}(f))$	Potentiostatic method
$Z_{IM,k}(f)$	secondary	– ([†])	–	Nyquist plot ([†])
$Z_{RE,k}(f)$	secondary	– ([†])	–	Nyquist plot ([†])
$ Z_k (f)$	secondary	– ([†])	–	Bode plot ([†])
$\theta_k(f)$	secondary	– ([†])	–	Bode plot ([†])

Figure 41 Output parameters for the EIS measurement [60].

4.2.1 Measurement Modes in AC Impedance Spectroscopy

AC impedance spectroscopy can be performed in two modes, one is Potentiostatic and the second is Galvanostatic. Each of them with specific advantages, limitations, and typical operating conditions. Although the choice of mode can influence practical aspects of the measurement. The resulting impedance spectra are generally comparable.

4.2.1.1 Potentiostatic Mode

An AC voltage perturbation is applied to the working electrode, and the current response is measured. It uses the full frequency range of the FRA/potentiostat but requires careful control of voltage amplitude that should remain small (typically 5–15 mV) to ensure linearity and avoid exceeding the current limits of potentiostat. Also, low cell impedance ($\approx 1 \Omega$) can cause large currents even with small voltage perturbations, affecting signal-to-noise ratio.

Typical the frequency range is between 100 kHz to 10 mHz, with ~ 10 points per decade. Often performed at different DC voltage levels to study operational behavior of PEM electrolyzers. Several studies [59], [60], [61], have used potentiostatic EIS across similar voltage ranges (1.4–1.8 V) and frequency range.

4.2.1.2 Galvanostatic Mode

An AC current perturbation is applied, and the voltage response is measured. Typical frequency range is between 200 kHz to 2 mHz. Allows larger current perturbations without significantly disturbing cell voltage, improving the control of current, signal-to-noise ratio and measurement accuracy.

Perturbation amplitude must still remain within the linear regime. In fact, the recommended values are 0.5–5% of the DC current. Voltage instability can reduce accuracy at high current densities, especially at low frequencies. Some studies used frequency ranges from 100 kHz to 0.01 Hz and current amplitudes around 24.5 mA/cm² [61].

4.3 Test Procedure

The EIS measurements were performed using the Energy-Lab XM System which is Solartron Analytical, AMETEK SI and it is operated through XM-studio ECS software. The setup includes a PGSTAT potentiostatic or galvanostatic, an internal 2 A power booster, and a frequency response analyzer (FRA).

The potentiostatic or galvanostatic tests enable standard DC electrochemical tests, and when coupled with the FRA, it can also perform impedance measurements. The instrument records data at up to 1 million samples per second, operates within a ± 8 V voltage range, supports currents up to ± 300 mA, and offers a bandwidth of 1 MHz.

The internal booster extends the system's capability for higher-power electrochemical cells, providing a maximum output of ± 8 V / ± 2 A. The FRA generates and analyzes AC perturbations with high accuracy across its full operational frequency range of 10 μ Hz to 1 MHz, enabling high-quality impedance spectroscopy over a wide spectrum.



Figure 42 Energy lab-XM System.

The test procedure consists of three main steps:

1. connecting the electrochemical cell to the ModuLab XM system,
2. creating a project in XM-studio and setting the test parameters,
3. running the experiment.

Correct wiring is essential. The potentiostat is linked to the internal booster and then connects to the cell. On the booster, the counter electrode (CE) is attached to the positive terminal and the working electrode (WE) to the negative terminal. The ModuLab XM's working and counter leads are connected to the cell's anode and cathode, respectively, while the reference lead is connected to both electrodes. This configuration ensures proper signal control and accurate impedance measurement.

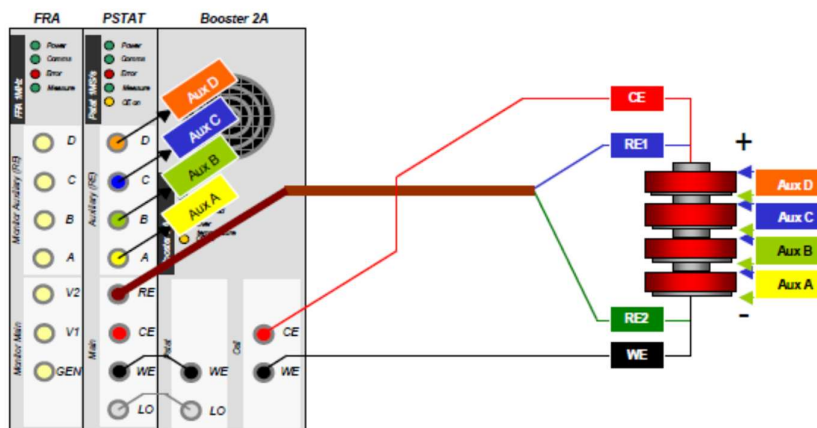


Figure 43 Schematic configuration of the connection between the cell and the device.

Before running the experiment, it is necessary to create a project in the XM-studio ECS software. This enables the running of the experiments named steps that are DC voltage control, DC current control, impedance current control and impedance voltage control. The main reason of this step is the characterization of the electrolytic cell with technique of the impedance spectroscopy. The chosen steps are the last two which are impedance current control and impedance voltage control.

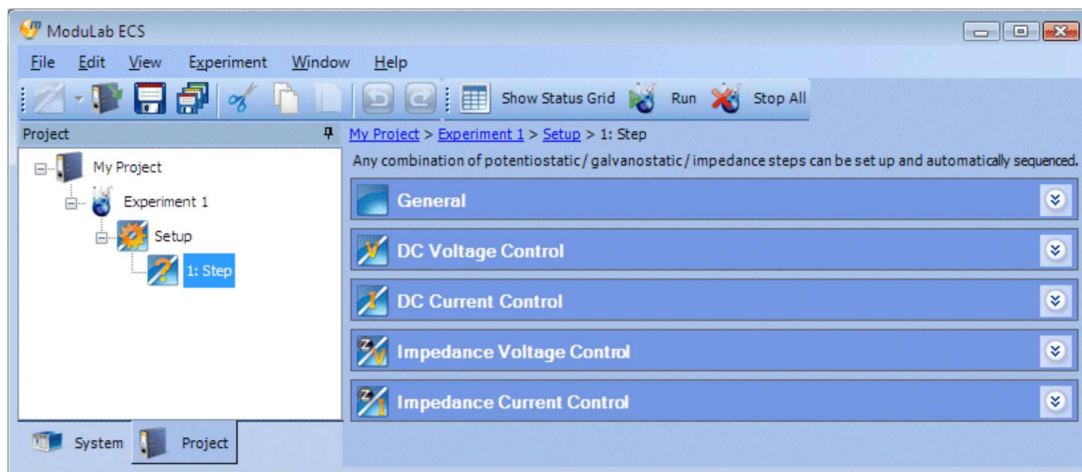


Figure 44 Types of experiments in the XM-studio ECS software.

For the impedance voltage control Potentiostatic impedance is selected and for the current control the galvanostatic impedance. These illustrates the two EIS measurements modes for this work.

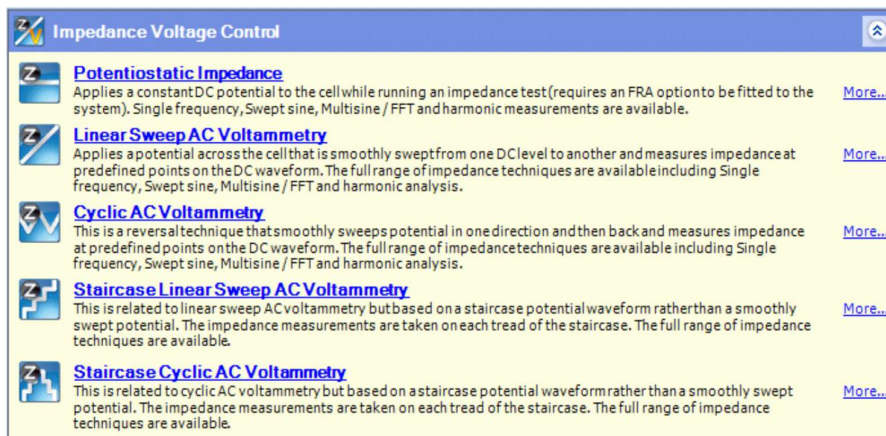


Figure 45 Possible step types for Impedance Voltage Control.

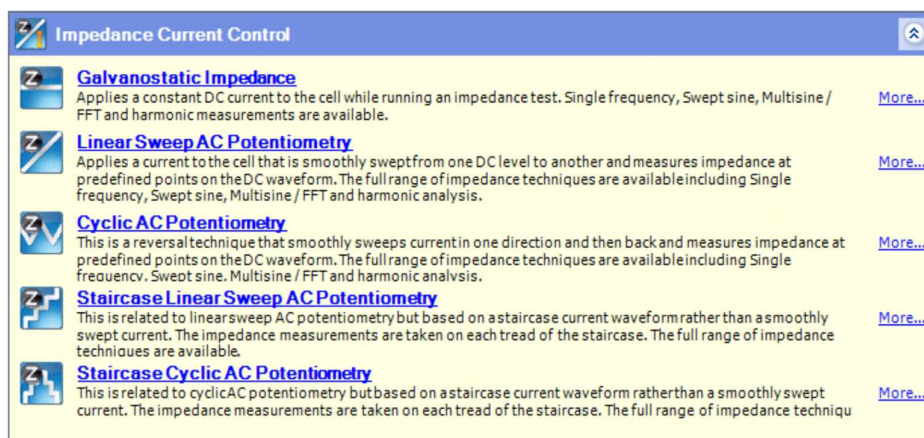


Figure 46 Possible step types for Impedance Current Control.

The first step for each EIS measurement mode is the definition of the experiment setup item including different fields, in particular Cell setup and the requirements of the hardware.

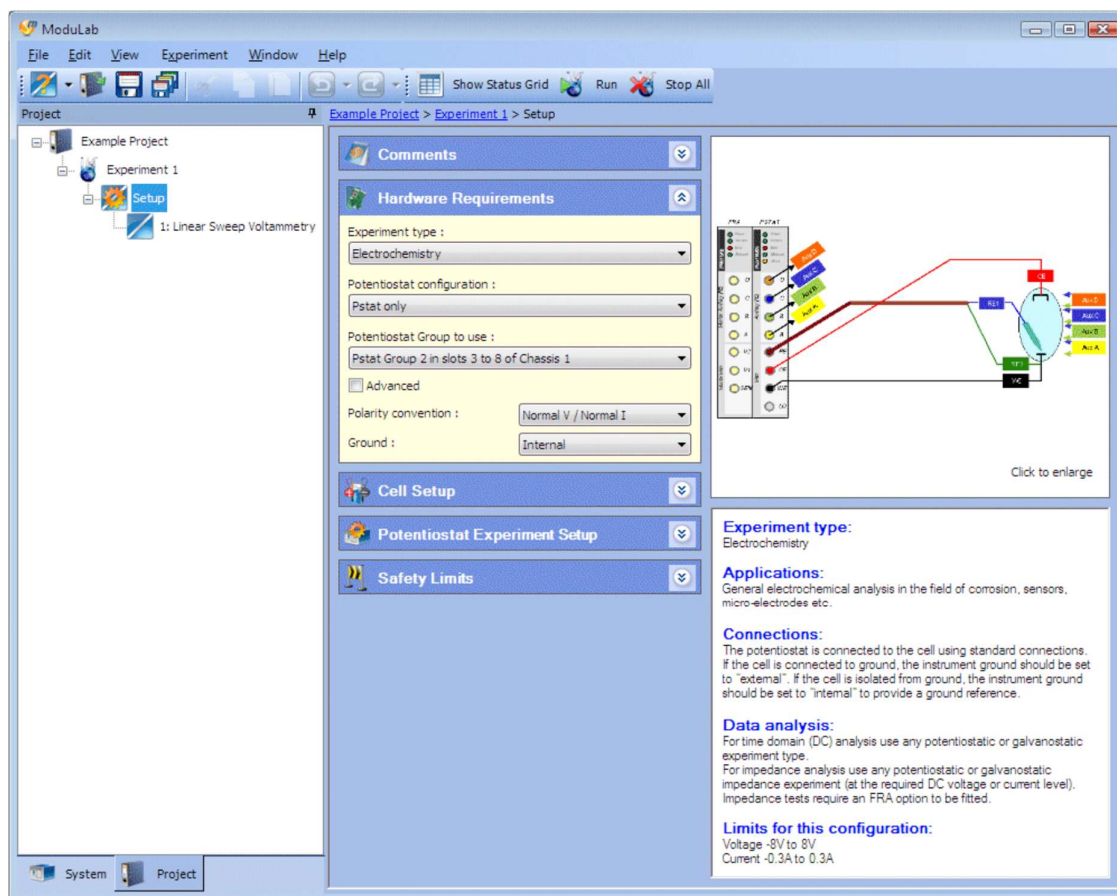


Figure 47 Cell setup on the software.

The hardware requirement shows the configuration of the connection for the experiment. The cell setup specifies some explanation about electrochemical cell such as the electrode active area, the reference electrode type, density and equivalent weight.

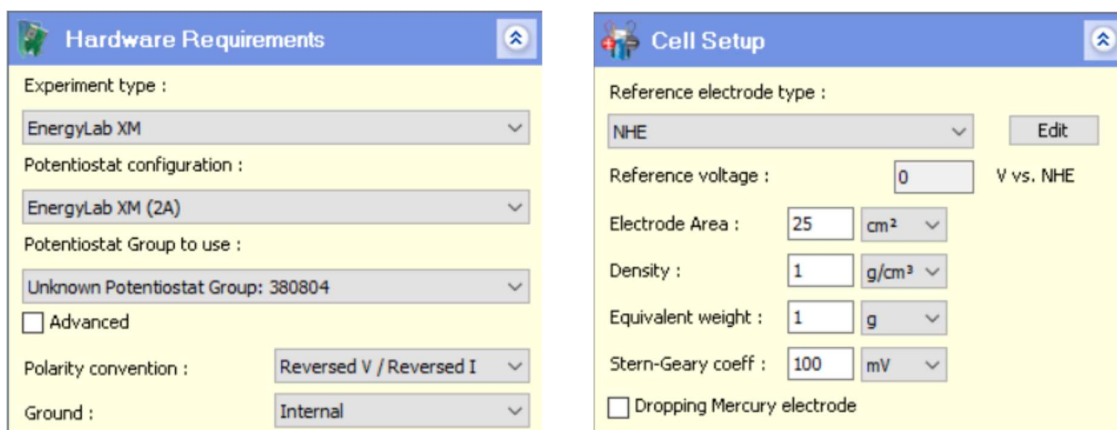


Figure 48 Hardware requirements and cell setup.

There is a definition of the tests input for both impedance experiments which is sub screens. One of them is scan setup and the other is impedance setup. The scan setup shows the values related to the excitation signals that is applied to the cell. The impedance setup illustrates the frequency range to be tested. Since the system has a frequency range on top of the DC scan that has been specified from the scan setup.

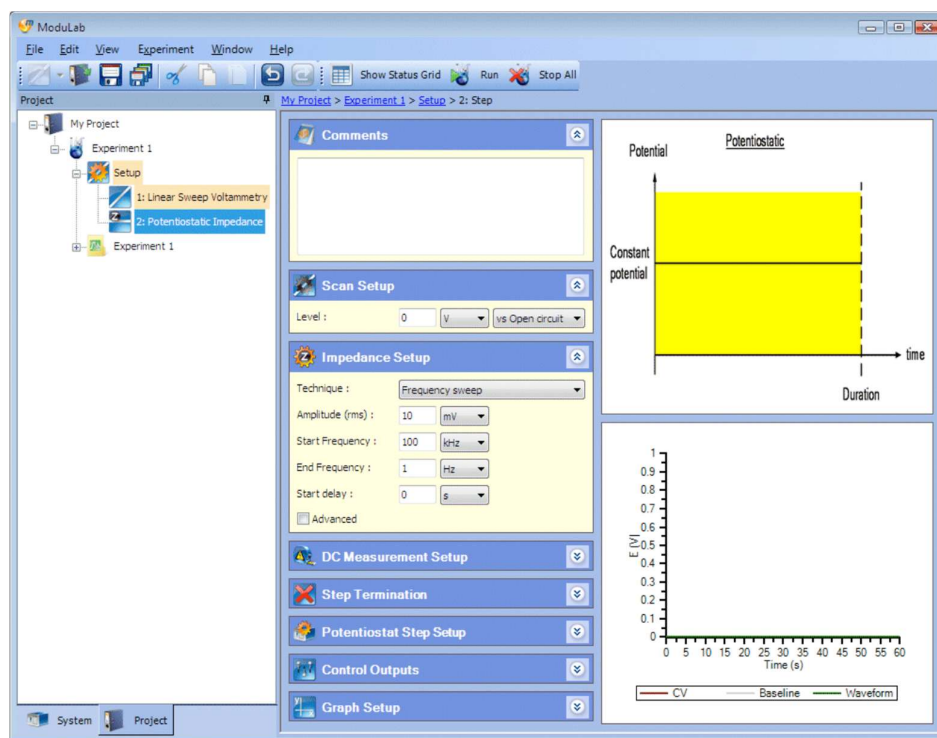


Figure 49 scan setup and the impedance setup.

Several parameters must be defined in the impedance Setup. First, the technique field specifies the type of AC stimulus used for the measurement. The most common choice is a frequency sweep, where a single sinewave is swept continuously from the start to the end frequency. Alternative options include single-frequency mode that applies one sinewave at a fixed

frequency. Also, Multi-sine mode that applies multiple sinewaves simultaneously, based on a Fast Fourier Transform distribution, and sweeps them across the selected frequency range.

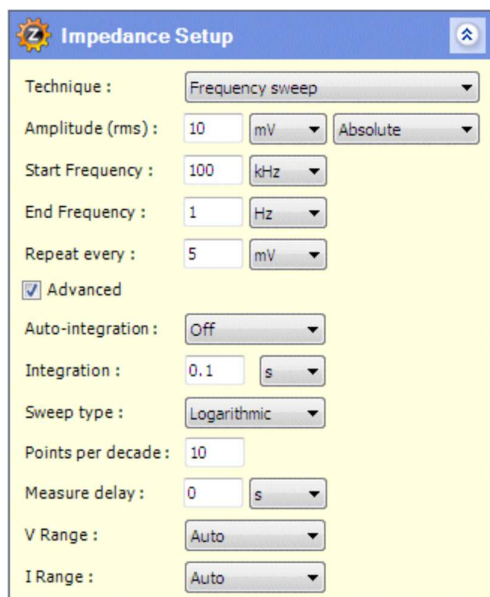


Figure 50 Impedance setup.

The amplitude field sets the RMS value of the AC perturbation, kept constant during the measurement step. It is defined in absolute terms for both potentiostatic and galvanostatic modes. The amplitude must be chosen carefully to maintain system linearity while ensuring an adequate signal-to-noise ratio. When using a frequency sweep, the user specifies the Start Frequency and End Frequency.

Noise reduction can be improved through signal integration, which narrows the measurement bandwidth and increases the signal-to-noise ratio at the cost of longer measurement time. Auto-integration adjusts integration time automatically to achieve a target statistical accuracy, while manual integration allows the user to set a fixed time in seconds or cycles. The sweep type determines how frequency varies during the sweep; a logarithmic sweep is preferred, with at least 10 points per decade.

Finally, the Scan Setup defines the DC value of the applied stimulus. In potentiostatic mode, the potential is specified relative to the reference electrode as this provides the most appropriate reference point for the measurement.



The scan setup appears as bellow.



Finally, the experiment will run after the wires are connected and the project has been created and set.

4.4. Data Quality Kramers Keronig (KK) Analysis

In the modeling and fitting procedures, the structure of the error in the measurement data plays an important role. There are two kinds of errors, one is stochastic errors and two is the systematic (bias) error. By selecting appropriate experimental parameters, the stochastic errors can be minimized. On the other hand, the bias errors are typically associated with violations of the Kramers–Kronig relations. To reduce random noises and nonlinear responses, in particular the amplitude of the perturbation must remain small.

In this work, the Lin-KK tool used to determine the Kramers–Kronig stability of impedance spectra. The tool provides a graph that shows the X axis of frequency in Hz, and the Y axis shows the relative deviation between the experimental data and an ideal KK based fit which is the residual in percent. Low residuals show high quality of the impedance data with minimal noise and no time-variance. All spectra of the test one in potentiostatic and of the test two in potentiostatic and galvanostatic were evaluated using the complex-fit and RC-auto modes of Lin-KK. Example spectra shows both valid and invalid cases. Invalid spectra typically exhibit biased residuals, signalling time-dependent behaviour.

A key step in improving the identification of the model and parameter estimation is data pre-processing, which includes monitoring and filtering the recorded data. Through the Nyquist and Bode plots, monitoring is performed, which help identify artefacts especially at high frequencies that caused by wiring and the essential inductance of electrical leads.

Corrupted data points are removed during the filtering stage. These include outliers, wild points, and measurements taken at the power-grid frequency and its harmonics which are known to produce unreliable results. Wild points are defined as data that deviate from the general trend of the spectrum.

Finally, the EIS spectra used in this study were not corrected for parasitic inductive effects originating from the measurement cables.

The graphs below show the impedance spectra of the test one in potentiostatic from 0-volt, 0.5-volt, 1 volt, 1.3 volt and 1.5 volt and the impedance spectra of the test two in potentiostatic 1.3 volt and 1.5 volt and galvanostatic 1.3 amps and 1.5 amps with good and low quality are reported. The graphs with low voltage in test one show a noisy behavior due to the exhibition of biased residuals, which indicates time-variance but in the potentiostatic mode 1.3 and 1.5 volt and also in galvanostatic mode 1.3 amps and 1.5 amps indicate valid impedance spectra.

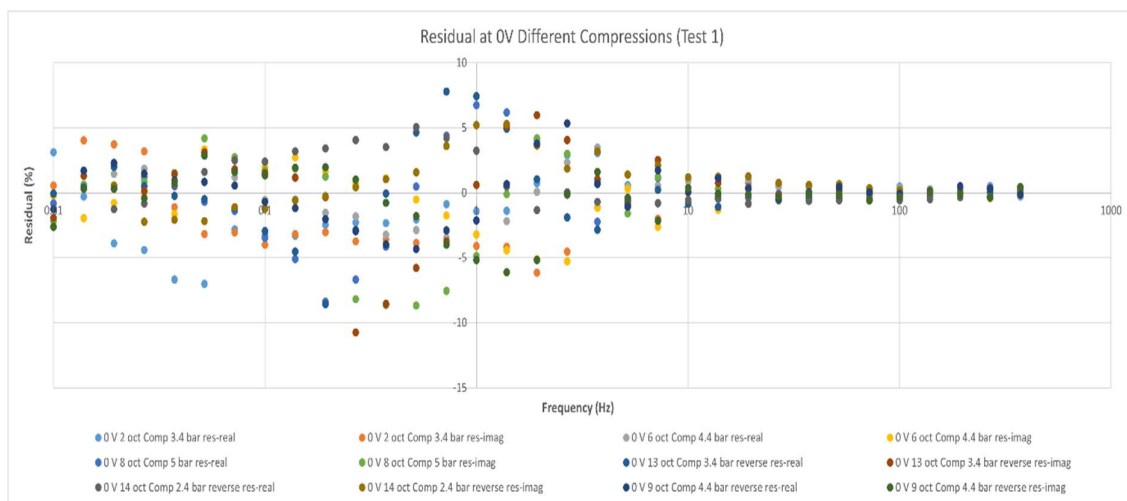


Figure 51 Impedance spectrum with noisy residuals at potentiostatic 0V (Test 1).

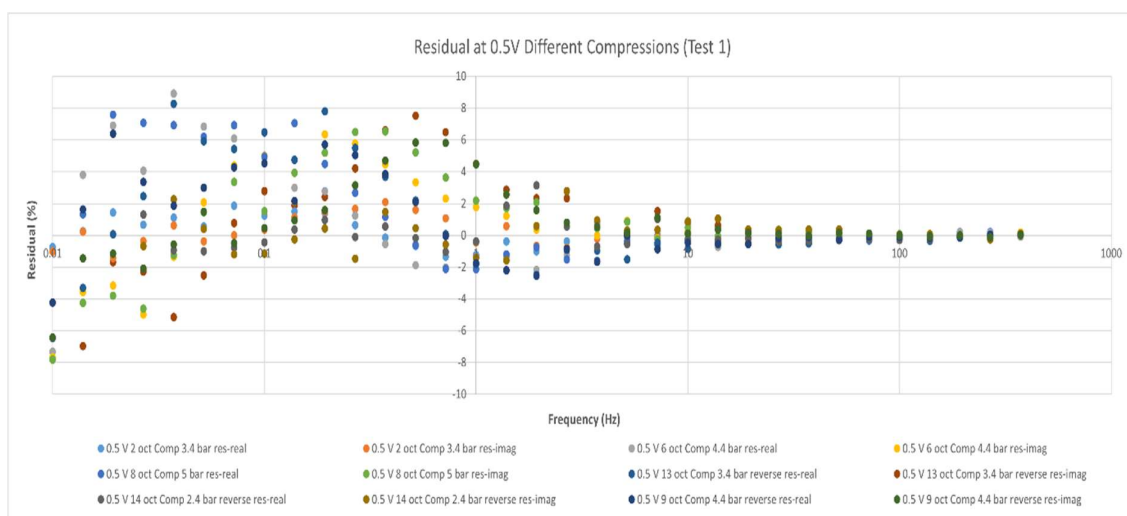


Figure 52 Impedance spectrum with noisy residuals at potentiostatic 0.5V (Test 1).

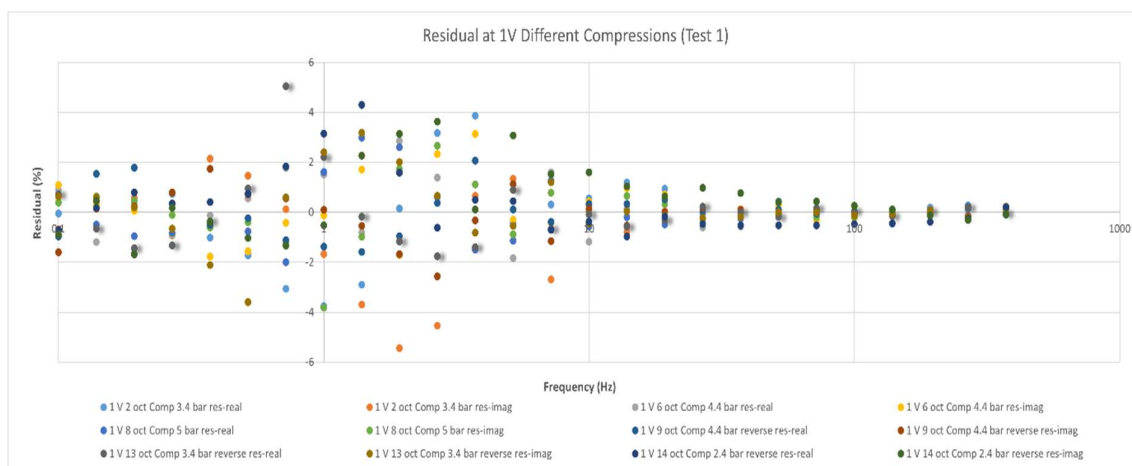


Figure 53 Impedance spectrum with moderate noisy residuals at potentiostatic 1V (Test 1).

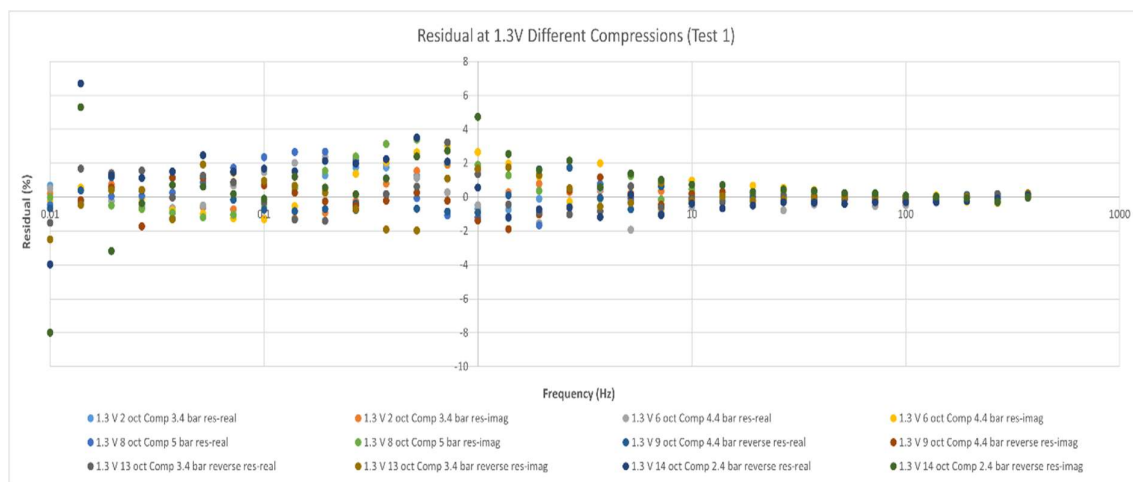


Figure 54 Impedance spectrum with moderate noisy residuals at potentiostatic 1.3V (Test 1).

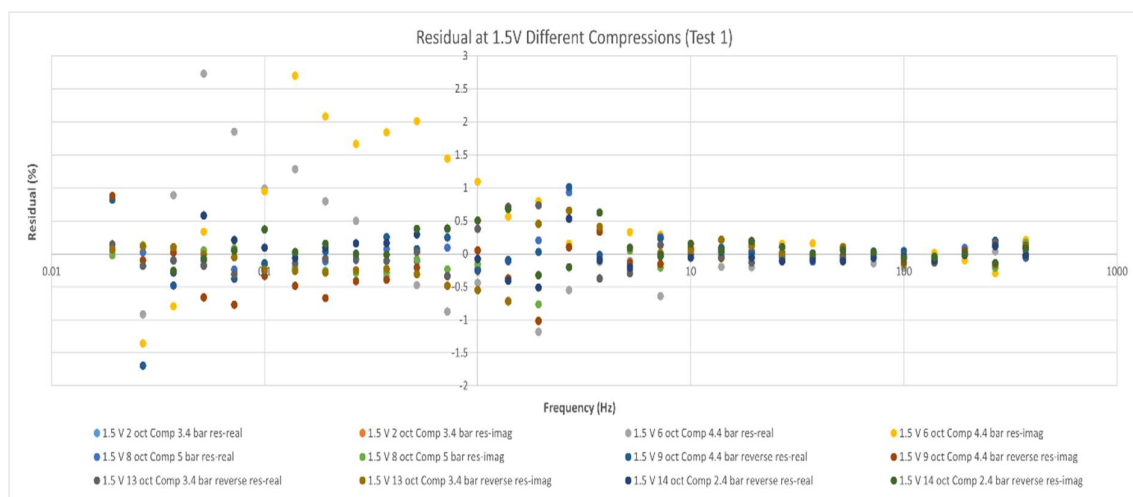


Figure 55 Impedance spectrum with acceptable residuals at potentiostatic 1.5V (Test 1).

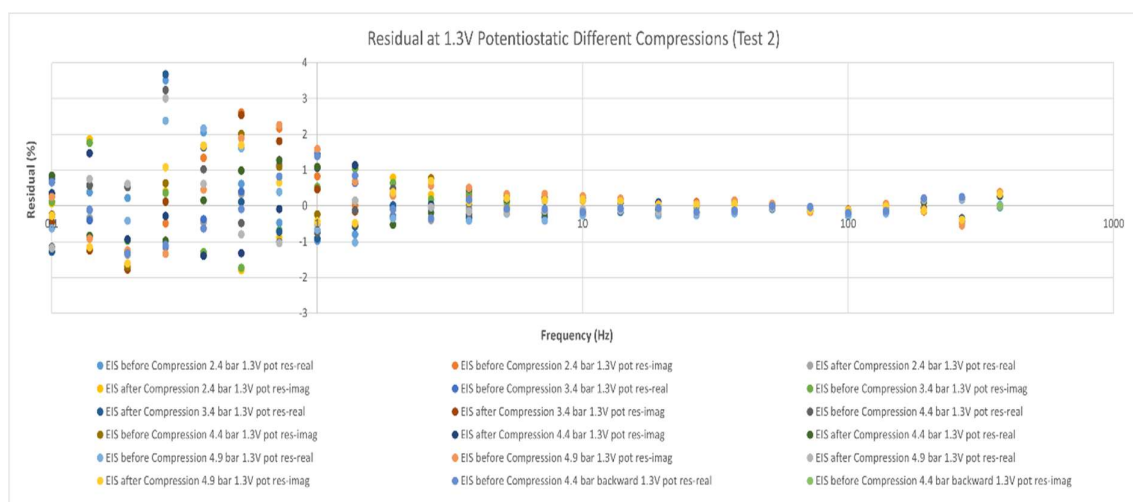


Figure 56 Impedance spectrum with acceptable residuals at potentiostatic 1.3V (Test 2).

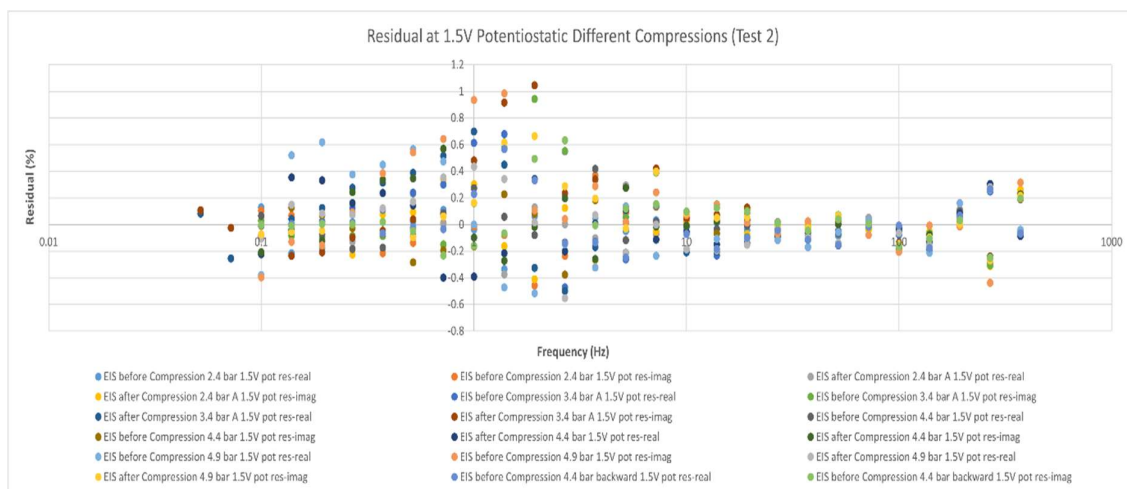


Figure 57 Impedance spectrum with acceptable residuals at potentiostatic 1.5V (Test 2).

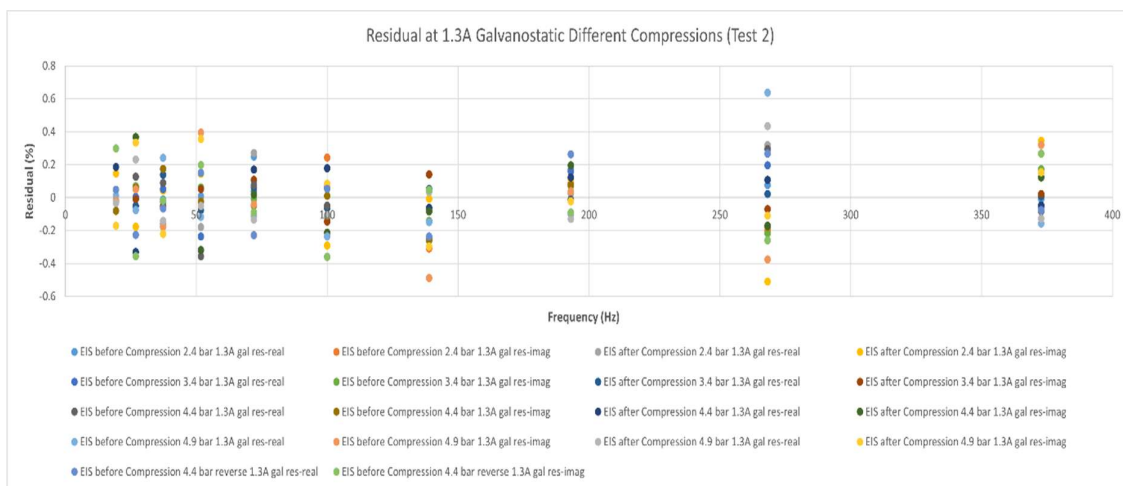


Figure 58 Impedance spectrum with acceptable residuals at Galvanostatic 1.3A (Test 2).

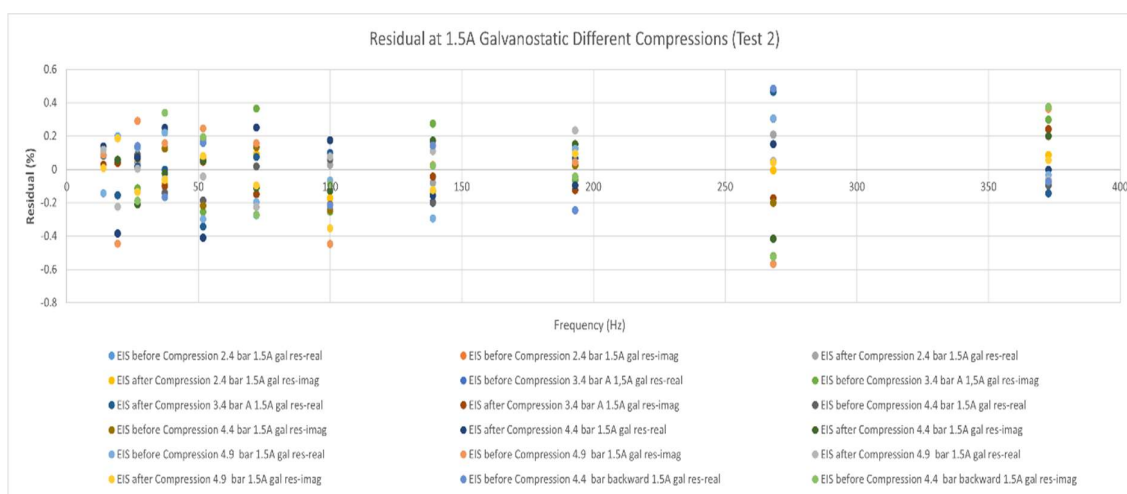


Figure 59 Impedance spectrum with acceptable residuals at Galvanostatic 1.5A (Test 2).

4.5 Selection of the Equivalent Circuit Model

The selection of an appropriate equivalent circuit model (ECM) is a critical step in interpreting electrochemical impedance spectroscopy (EIS) data, particularly for PEM fuel cells and PEM water electrolyzers. Because multiple circuit configurations can often reproduce similar impedance responses, the model must be chosen based on both the characteristic features of the measured spectra and the underlying physicochemical processes of the system [58].

In this study, the ECM was selected through a systematic evaluation of the impedance spectra obtained under different pressures and operating voltages. Across all operating conditions, the Nyquist plots consistently exhibited three dominant regions:

1. a high-frequency intercept associated with ohmic resistance,
2. a mid-frequency depressed semicircle corresponding to charge-transfer and double-layer effects, and
3. a low-frequency tail or inductive loop linked to mass-transport limitations or dynamic surface processes.

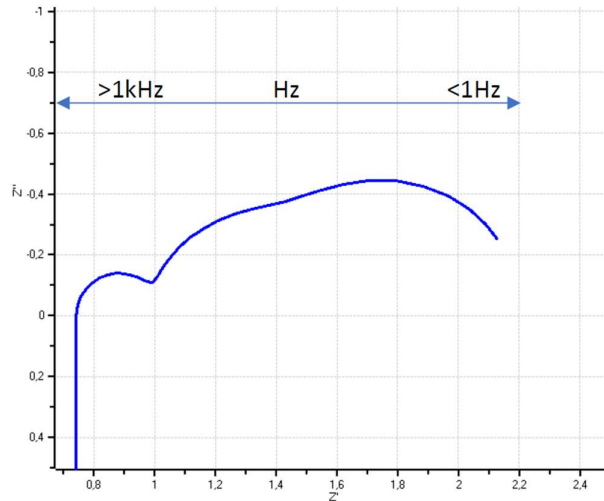


Figure 60 frequency range.

These spectral features are well-documented in PEM systems and typically require combinations of resistive, capacitive, and constant phase elements (CPEs) to accurately reproduce the non-ideal behavior of porous electrodes [62], [63].

Given the non-ideal capacitive response observed in all datasets—evidenced by depressed semicircles rather than ideal arcs CPEs were preferred over ideal capacitors. This choice is consistent with the literature, where CPEs are used to represent distributed time constants arising from surface roughness, heterogeneous catalyst layers, and non-uniform current distribution. Additionally, at elevated voltages and pressures, several spectra exhibited low-frequency inductive behavior, which has been attributed to gas evolution dynamics,

adsorption/desorption processes, or measurement-related inductances (Franzetti et al., 2023). To account for this, an inductive element or a negative-resistance ZARC component was incorporated depending on the specific spectral shape.

After comparing multiple candidate circuits including classical Randles-type models, Warburg-based diffusion models, and multi-ZARC configurations the LR(QR)(QR)-type model was selected as the most appropriate for this dataset. This model provided:

- stable convergence during fitting,
- physically meaningful parameter trends across pressure and voltage levels, and
- the lowest fitting residuals without over-parameterization.

The final ECM therefore consists of a series ohmic resistance (R_s), followed by two parallel R-CPE branches representing high- and mid-frequency charge-transfer processes, and, when required, an additional inductive or negative-ZARC element to capture low-frequency behavior. This configuration aligns with models previously validated for PEM fuel cells and electrolyzers operating under varying pressure and gas-evolution conditions [62], [64].

4.5.1 High-frequency (HF) region

The high-frequency (HF) region of an EIS spectrum is independent of cell potential and is mainly associated with distributed resistance within the catalyst layer and structural characteristics of the MEA. HF features may appear as a full or partial semicircle, or as a 45° line typically linked to proton-conductivity limitations in the catalyst layer.

These effects often arise from current constriction, which is caused by poor contact between the membrane and catalyst layer. Such poor contact driven by MEA properties like catalyst loading, ionomer content, porosity, and fabrication method reduces the effectiveness of the three-phase boundary and hinders proton transport.

The HF intercept on the real axis provides the ohmic resistance R_Ω , which reflects contact resistances and proton/electron transport through the membrane and conductive components. Variations in R_Ω are linked to temperature, membrane hydration, and membrane degradation. Inductive artifacts from wiring and cell geometry may also appear in this region.

To model the HF behavior, the equivalent circuit includes a series inductance (inductive effects), a series resistance (ohmic losses), and when current constriction is present a parallel combination of a resistance and a constant phase element (CPE) representing the HF semicircle.

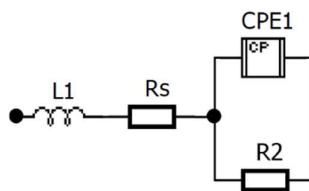


Figure 61 CPE and resistance.

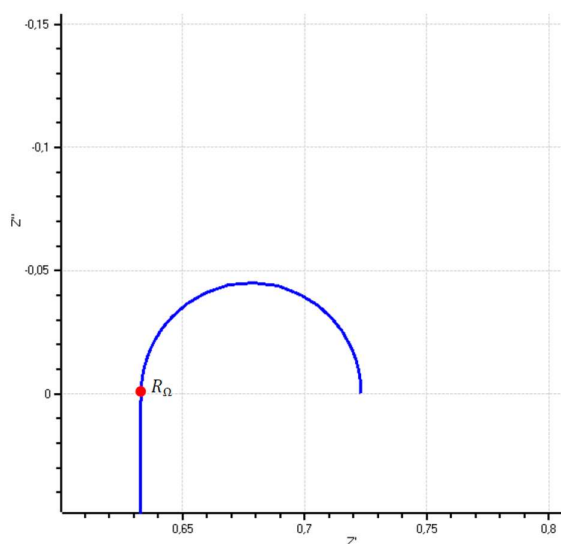


Figure 62 CPE & ohmic resistance.

4.5.2 Mid-frequency (MF) region

The mid-frequency (MF) region of an EIS spectrum depends on the applied potential. As the overpotential increases, this MF feature typically decreases because reaction kinetics increase. This region is generally associated with the oxygen evolution reaction (OER) at the anode, though some studies also associate it with the hydrogen evolution reaction (HER) at the cathode.

In the Nyquist plot, the MF region appears as one or sometimes two partially overlapping semicircles, each representing a distinct charge-transfer process. These processes are modeled using a parallel combination of a charge-transfer resistance and a constant phase element (CPE), illustrating the kinetic behavior of the electrochemical reactions occurring in this frequency range.



Figure 63 charge transfer resistance and CPE.

In the Nyquist plot, when only one arc is shown in the mid frequency region, it is corresponding to a single peak in the same frequency range in the bode plot. In fact, this is modelled with a single charge transfer resistance in parallel with a CPE, on the other hand, two merged semicircles with two peaks in the bode plot are simulated with two charge transfer resistance in parallel with two CPE in series.

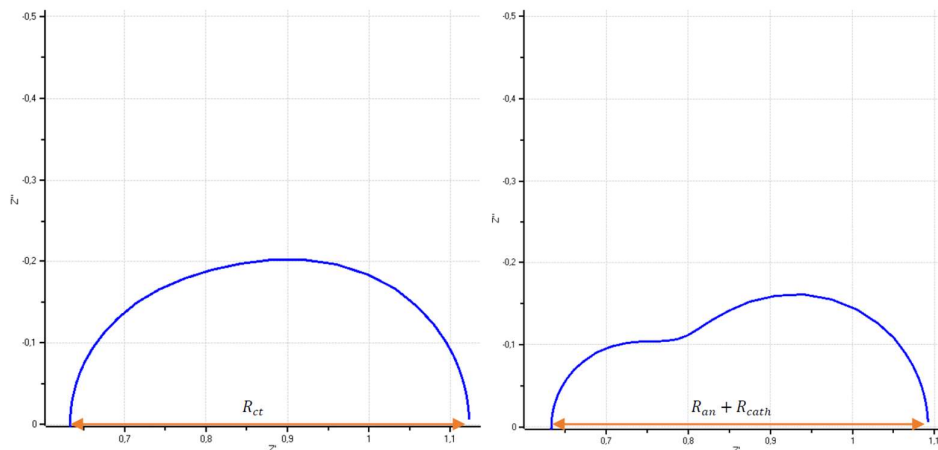


Figure 64 R_{ct} , R_{an} and R_{cath} .

4.5.3 Low-frequency (LF) region

The low-frequency (LF) region of an EIS spectrum is dependent on the applied potential and typically increases with increasing overpotential. This LF feature illustrates mass-transport limitations in the cell. In most PEM electrolyzer systems, it shows as an additional semicircle in the Nyquist plot. It is indicating finite diffusion, rather than a 45° tail associated with semi-infinite diffusion.

Mass-transport effects in this region are modeled using Warburg elements. This may be placed either in series with the entire ($R_{ct} \parallel CPE$) block representing the mid-frequency kinetics, or only in series with the charge-transfer resistance.

Overall, the number of arcs observed in the impedance spectrum depends on the processes occurring in the cell. A single arc suggests negligible mass-transport limitations. On the other hand, two or three merged arcs indicate the presence of faradaic and mass-transport phenomena.



Figure 65 Warburg elements in series with other elements.

4.6 ECM Fitting Results

The equivalent circuit model was fitted to impedance spectra collected at six compression levels. The extracted parameters include the ohmic resistance (R_s), the charge-transfer resistance (R_{ct}), and the CPE parameters associated with double-layer and mass-transport processes. The following subsections summarize the observed trends and their physical interpretation.

4.6.1 Initial Estimation of the Parameters

An initial estimation has been done through the software in the section fitting with the circle fitting option. This option allows to have an initial estimation of the principal parameters such as ohmic resistances and capacitance at high and low frequencies. This initial estimation is useful to fit the model function obtained from the ECM. The circle fitting method predicts two options which make a choice. The prefit option is selected to fit the circles through three points selected in the impedance.

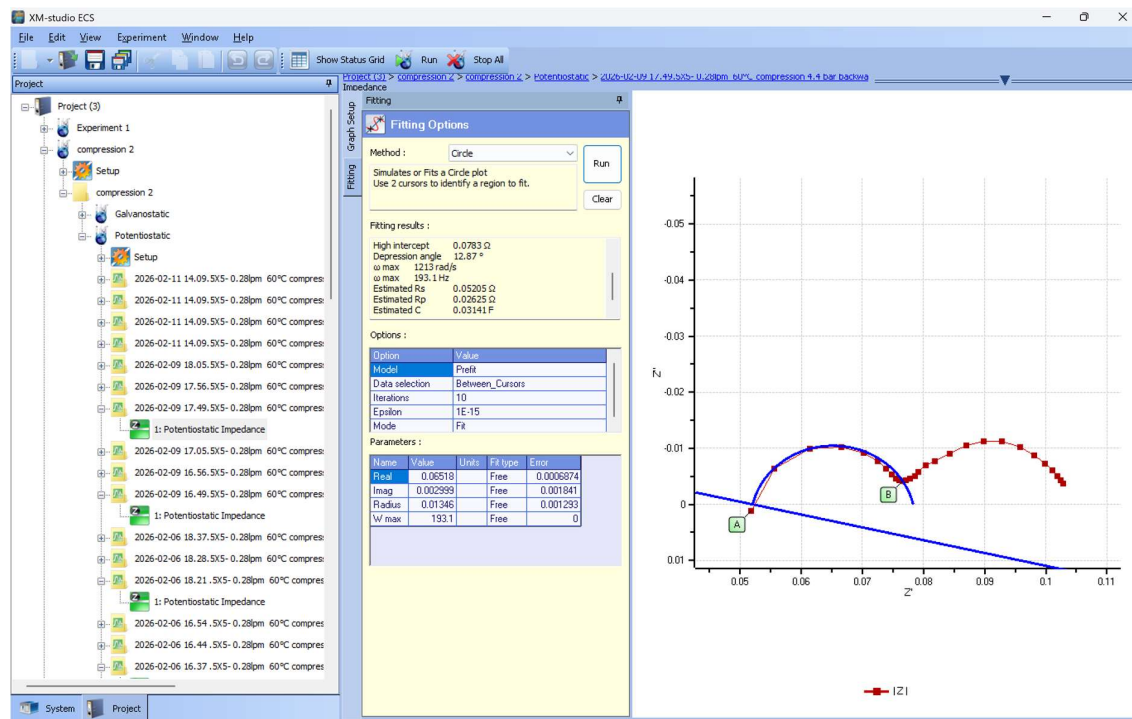


Figure 66 fitting of the EIS graphs.

4.6.2 Model Development

The construction of the model and approximation to the equivalent circuit model are both performed by the software. To obtain a good fit of the impedance data a procedure which is multi step is carried out.

4.6.2.1 Construction of the ECM

The ECM is built by using the tool available in the software based on the analysis of the impedance diagram. The equivalent circuit model usually consists of a series connection of the ohmic resistance, inductance and two resistance and CPE elements in parallel.

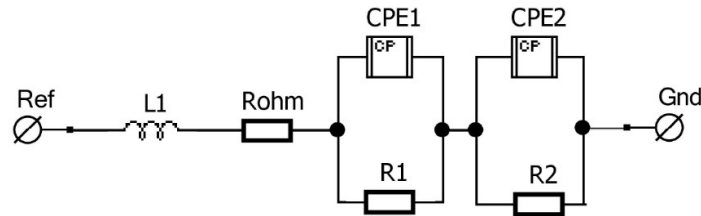


Figure 67 Example of the equivalent circuit.

4.6.2.2 Initial Approximation

Equivalent circuit model is used to obtain an initial approximation. In this part only the parameter of ohmic resistance is fixed and the rest of the parameters are unfixed and obtained by using the values from the prefit circle. Constant phase element has two variables as shown in the below picture. TDE shows the parameter Q in the formulas and PHIDE shows the exponent n .

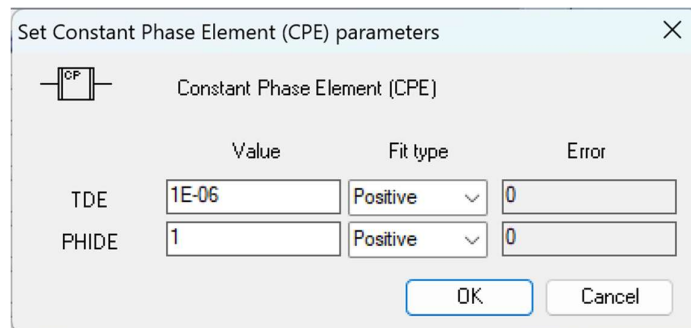


Figure 68 CPE set.

One important thing is that the PHIDE which is n must be equal to one ($n=1$). On the other hand, TDE which is Q must leave with its default value. It is noteworthy to mention that before running the fitting it is important to set the weighting as data-modulus weight. Also, it is necessary to choose point A and B on the graph.

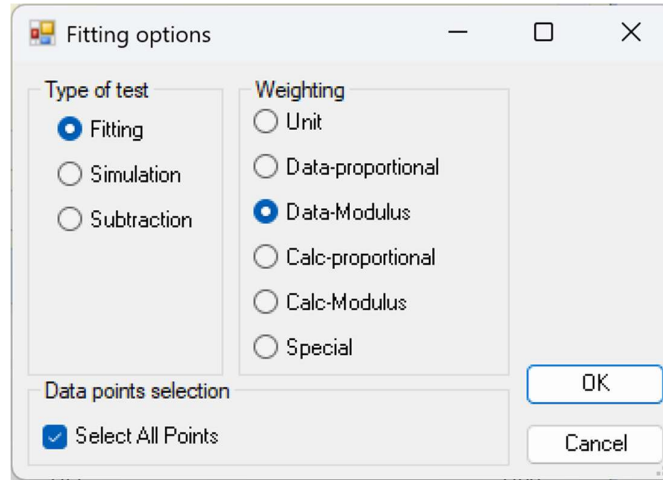


Figure 69 Fitting options.

After all, it is possible to run the software and finally get the first approximation of the data.

4.6.2.3 Warburg Element and a new Circuit

If a diffusion is present, it should be considered. It is necessary to add a Warburg element in parallel to the CPE because the mass transport processes occur in parallel to the charge transfer reaction. The Warburg element is Open Warburg (W_o). Open Warburg has two variables such as R that represents R_0 , T shows Q .

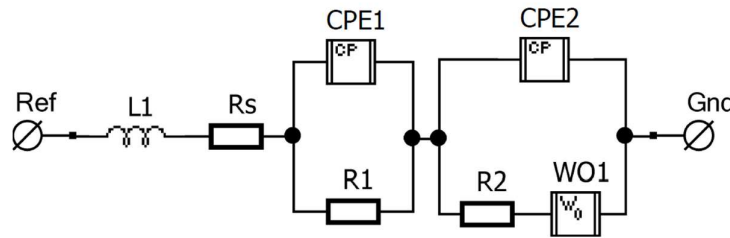


Figure 70 alternative ECM.

4.7 Evaluation of Fitting Scenarios and Selection of the Final ECM Configuration

To ensure the physical relevance and robustness of the extracted parameters, multiple fitting scenarios were evaluated using both the XM-Studio software and a Python-based fitting routine. Each scenario was designed by modifying the constraints applied to key ECM parameters, particularly the ohmic resistance R_s , the CPE exponent n , and the charge-transfer and mass-transport resistances.

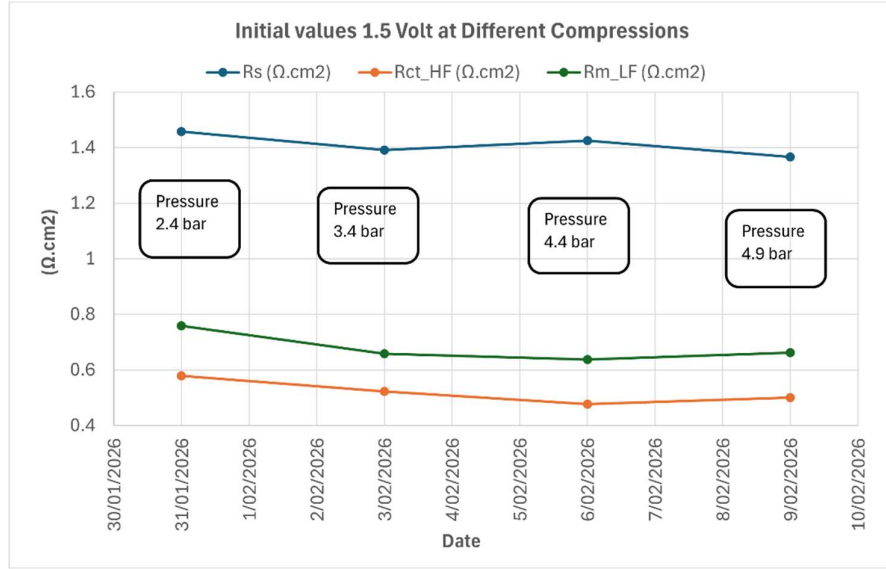


Figure 71 Initial values with Pre fitting.

4.7.1 Fixing the Ohmic Resistance Based on Analytical Estimation (Scenario 1)

In the first scenario, the ohmic resistance R_s was calculated analytically from the high-frequency intercept of the Nyquist plot using the standard relation:

$$R_s = \frac{Z'(\omega \rightarrow \infty)}{A}$$

Where:

- $Z'(\omega \rightarrow \infty)$ is the real part of the impedance at the highest measured frequency,
- A is the active electrode area.

The obtained value of R_s was fixed in XM-Studio, while all other parameters including charge-transfer resistance, CPE parameters, and mass-transport resistance were left unfixed. This particularly regarding the magnitude of R_{ct} , the CPE exponent n , and the overall semicircle diameter. The fitted curve showed excellent agreement with the experimental Nyquist data.

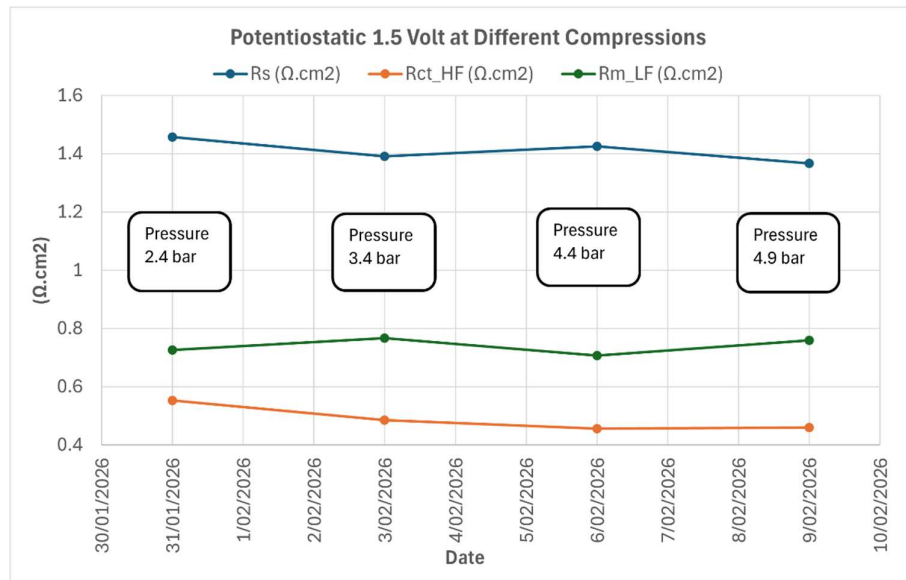


Figure 72 Results of the Resistances with First Scenario.

4.7.2 Fixing the CPE Exponent n (Scenario 2)

In the second scenario, the CPE exponent n was fixed to a literature-based value (typically between 0.8–1.0 for PEM systems), while all other parameters were left free. The graph below shows the behavior of the resistances with this scenario.

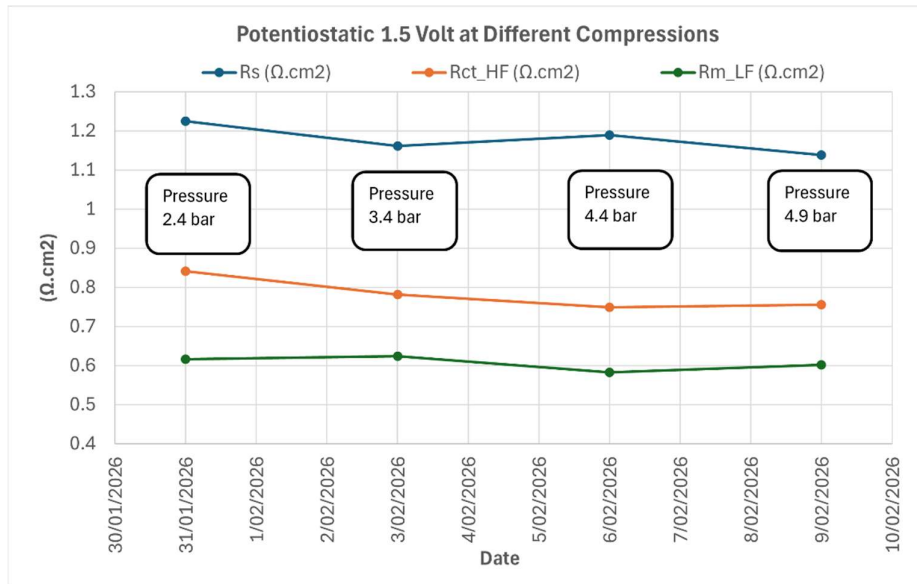


Figure 73 Results of the Resistances with Second Scenario.

4.7.3 All Parameters Unfixed (Scenario 3)

In this scenario, no constraints were applied to any ECM parameter. All parameters including R_s , CPE parameters, R_{ct} , and mass-transport resistance were left completely free during the fitting process. Although this approach provides maximum flexibility to the solver, it typically leads to non-physical parameter values for example unrealistically low or high R_s , or CPE

exponents outside the expected 0.7–1.0 range, overfitting where the algorithm fits noise rather than the true electrochemical behavior also poor reproducibility because small changes in initial guesses lead to large variations in results and finally deviation from literature trends especially in R_{ct} and mass-transport resistance.

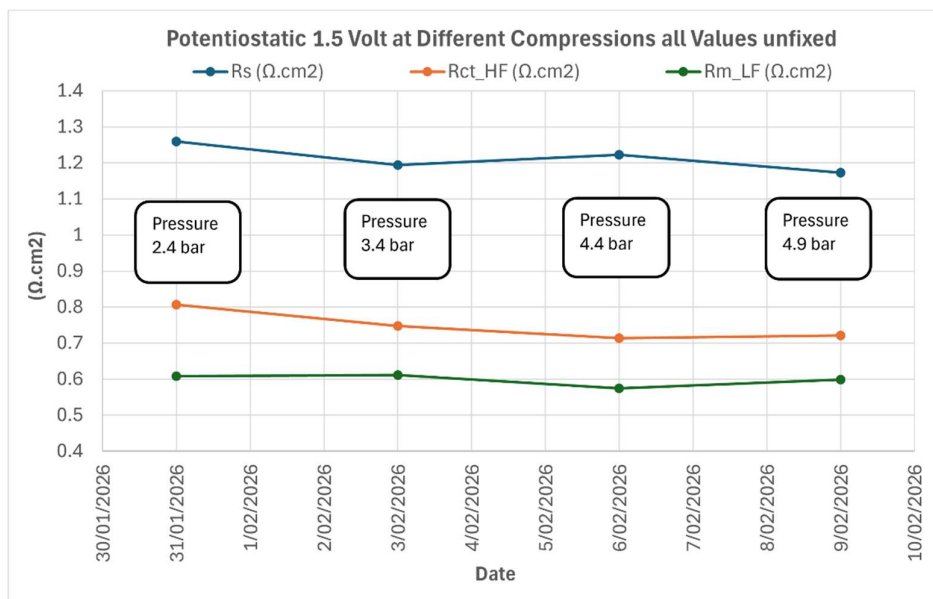


Figure 74 Results of the Resistances with Third Scenario.

4.7.4 Python-Based Fitting Routine (Scenario 4)

A fourth scenario was implemented using a custom Python fitting script in Jupiter Notebook, following the same parameter constraints as Scenario 1 and Scenario 2. The results confirmed the XM-Studio findings with fixing R_s (Scenario 1) produced physically meaningful and literature-consistent parameters, whereas fixing n (Scenario 2) led to unrealistic resistance values. Therefore, Scenario 1 was selected as the final ECM fitting configuration for subsequent analysis. The python code & fitting results are available in the appendix.

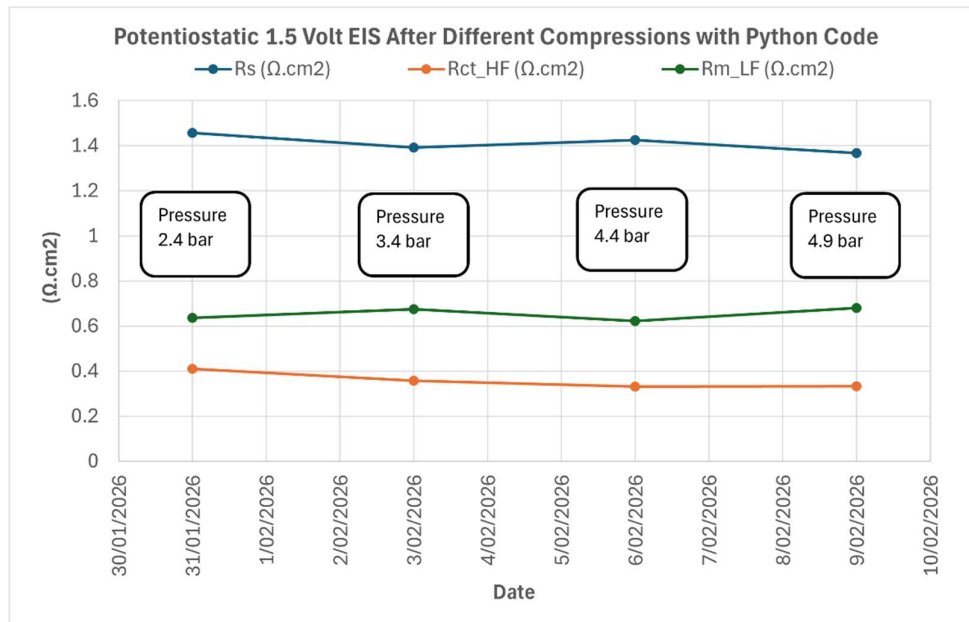


Figure 75 Results of the Resistances with Python Code Scenario.

4.7.5 Final Results

First scenario is selected as the results was closer to the literature, in which only the ohmic resistance was kept fixed and all the other parameters unfixed and run to get the results.

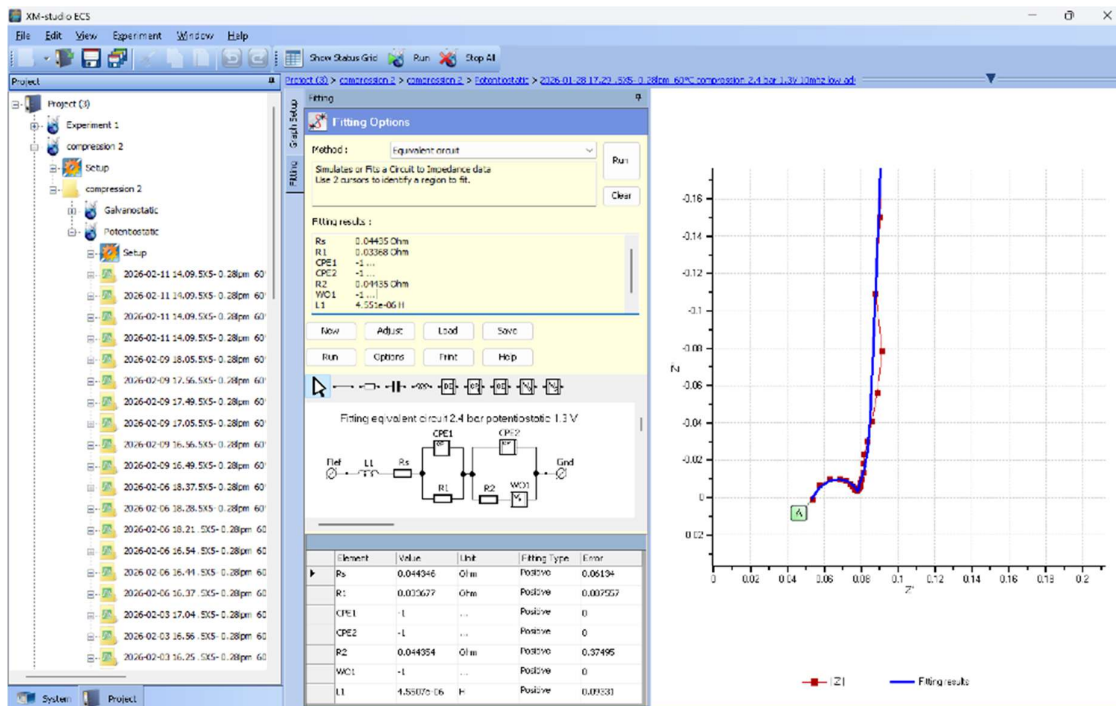


Figure 76 Fitting result.

Chapter 5. Results

5.1 Results of the Test One

The aim of the test one to perform electrochemical characterization of the new cell by carrying out EIS measurements. The test one is performed at 60°C, and different pressures (3.4 bar, 4.4 bar, 5 bar, 4.4 bar descent, 3.4 bar descent, 2.4 bar descent) mass flow rate of approximately 200-300 mL/min.

Potentiostatic test, performed for the test one at different DC value of voltage (0 V, 0.5 V, 1 V, 1.3 V, 1.5 V) vs Reference by varying the frequency from 100 kHz to 10 mHz in single sine mode and using a sinusoidal excitation signal of 10 mV root mean square (rms).

5.1.1 Compression Effect of the Test One

The experiment was carried out at operating temperature of 60 °C and varying the imposed cell compression. The purpose of the test was to evaluate the effect of the compression on the cell performance and the cell impedance through EIS analysis. First, the cell was operated at a temperature of 60°C, while the cell compression was increased from 3.4 bar to 5 bar and then the cell compression descended from 5 bar to 2.4 bar. EIS measurements were performed only after the cell had reached steady state conditions at each compression.

5.1.2 Basics of Degradation

The initial voltage drop is likely associated with membrane re-hydration and thermal stabilization reducing ohmic resistance; however, direct confirmation for this specific cell requires targeted measurements in-situ membrane water content or EIS during the start-up transient. The membrane's proton conductivity drops when it dries out, increasing the cell's ohmic resistance. The membrane hydration strongly modulates proton conductivity, making water management an important design parameter [65].

5.1.3 Analysis of Voltage Behavior Under Different Conditions (Test 1)

Degradation test performed at 60°C and at different compression levels and constant current density of 1 A/cm²:

The degradation test is carried out at a constant current density of 1 A/cm² and the cell voltage increase over time is monitored. It consists of two tests one test carried out in October and another one in January. The first test is performed at 60°C and compression levels of 3.4 bar, 4.4 bar, 5 bar and the descent compression of 4.4 bar to 3.4 bar and finally 2.4 bar. The second one has been explained in the following part of the second test. During the first test in October, EIS tests have been performed after stabilization of the voltage at each compression level. Only one types of EIS tests have been used:

- Potentiostatic test, performed at 0 DC V by varying the frequency from 100 kHz to 10 mHz in single sine mode and using a sinusoidal excitation signal of 10mV root mean square (rms)

As can be seen in the figure 77 the voltage increased up to more than 3 volts due to a startup transient then the voltage rapidly drops and stabilizes around 2.6701–2.6756 V. An EIS test performed at pressure 3.4 bar then the test continued again at the same pressure. In comparison with the part after the first EIS at pressure 3.4 bar the voltage again rises rapidly up to 2.73 V then decreases and stabilises in a bit higher level with respect to the previous voltage at around 2.70-2.71 V. The voltage was increasing and entering the degradation phase when the second EIS test performed. In comparison with the descent pressure at 3.4 bar, the voltage increases rapidly up to around more than 3 volts then decreases rapidly and stabilizes to 2.77 V. It is expected to have an increase of the cell potential therefore a decrease of the cell performance.

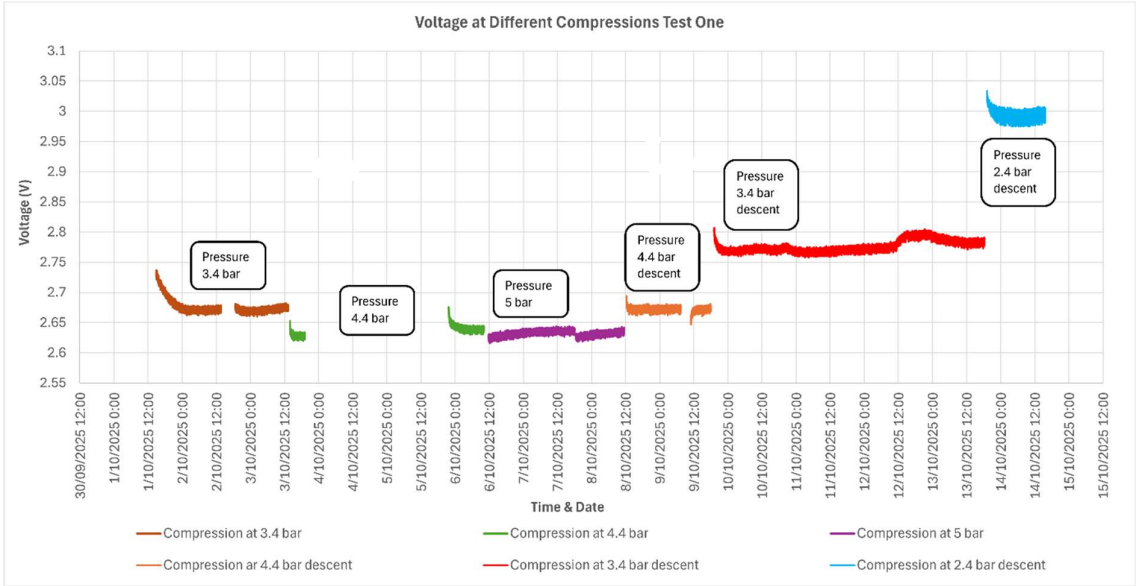


Figure 77 Voltage behavior during the time (Test 1).

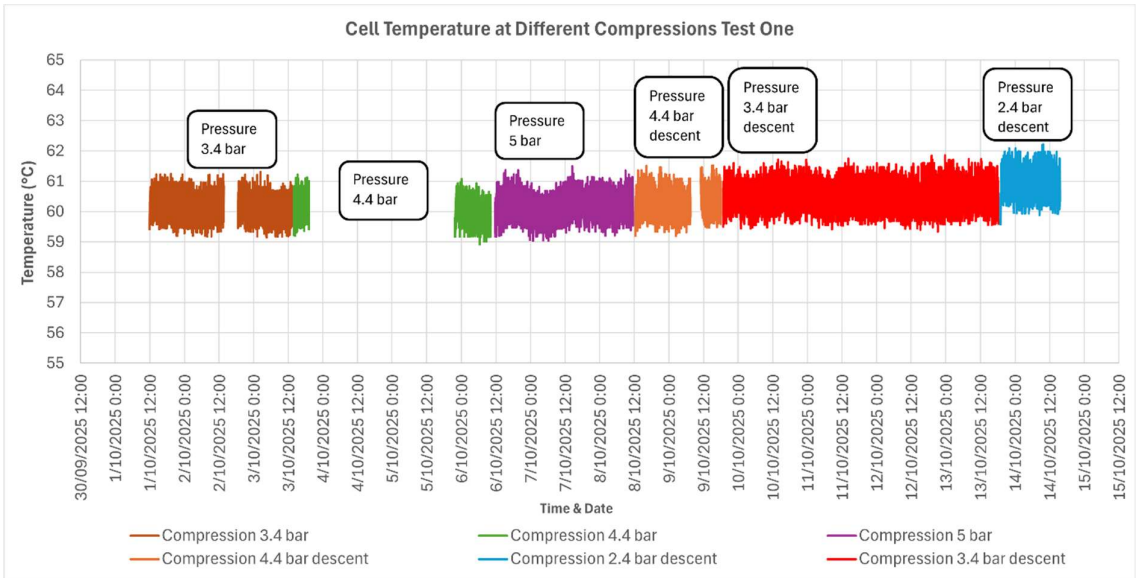


Figure 78 Cell Temperature behavior during the time (Test 1).

5.1.3.1 Effect of the Degradation at Compression 3.4 bar (Test 1)

In test one at compression 3.4 bar, the system operated for around 44.49 h but the duration of stabilization which was around 15.83 h reduced from the total hours and resulting in an effective duration of 28.66 h after removing the unstable region. The voltage increased from 2.6701 V at the beginning of the interval to 2.6756 V at the end. Based on this change, the degradation rate was calculated as $191 \mu\text{V}/\text{h}$, indicating a steady voltage rise under this pressure condition.

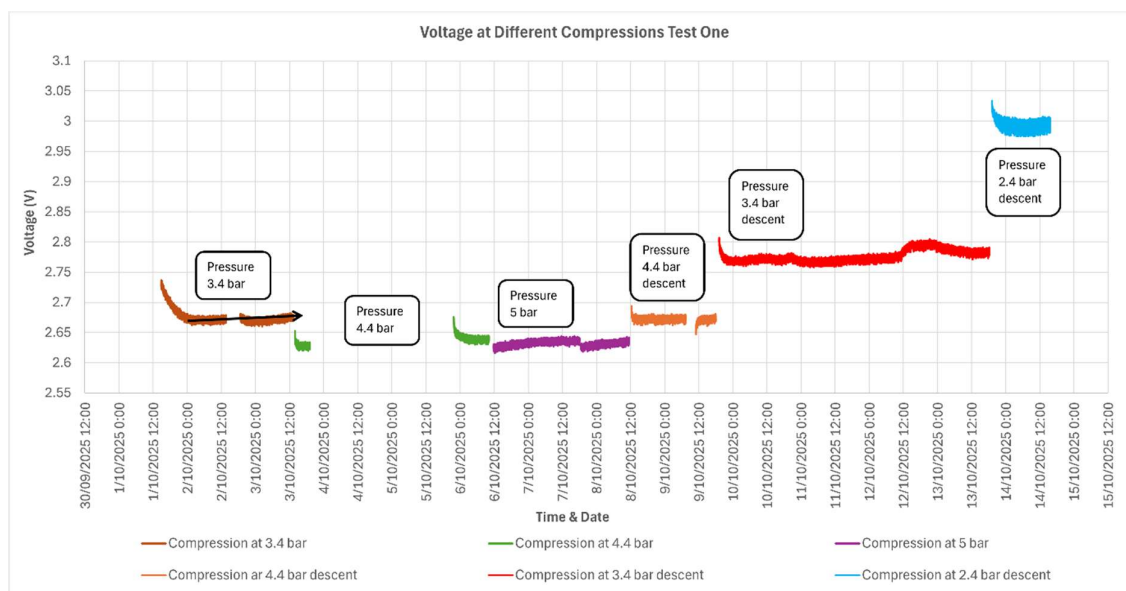


Figure 79 Degradation behavior at Compression 3.4 bar during the time (Test 1).

5.1.3.2 Effect of the Degradation at Compression 4.4 bar (Test 1)

As the figure shows at the initial part, a rapid increase of voltage has been seen up to around 2.67 V due to the start up the system and a rapid decreased and stabilize to around 2.64 V. Then a system shutdown happened on 3 October at night after a restart of the system on 5 October at night has been done. As can be seen an increase of the voltage at the initial part and during the stabilization of the voltage at around 2.65 V then an EIS test has been done. After EIS the graph starts from downward because of a mistake that the system has been started working from a high cell temperature of 85°C so the voltage behavior is lower with respect to the previous part at pressure 4.4 bar descent. There has been an unexpected shut down of the system due to the power outage in the lab on 9 October in the early morning, the system restarts again at pressure 4.4 bar descent. The figure shows that the voltage doesn't change that much and stabilized until at evening then another EIS has been done. Finally, as can be observed a small improvement in voltage but more transient behavior. The initial voltage was 2.627 V, and it end at 2.628 V with duration of 3.7 h that the duration of stabilization and shutdown has been eliminated so the degradation rate of this compression level is $270 \mu\text{V}/\text{h}$.

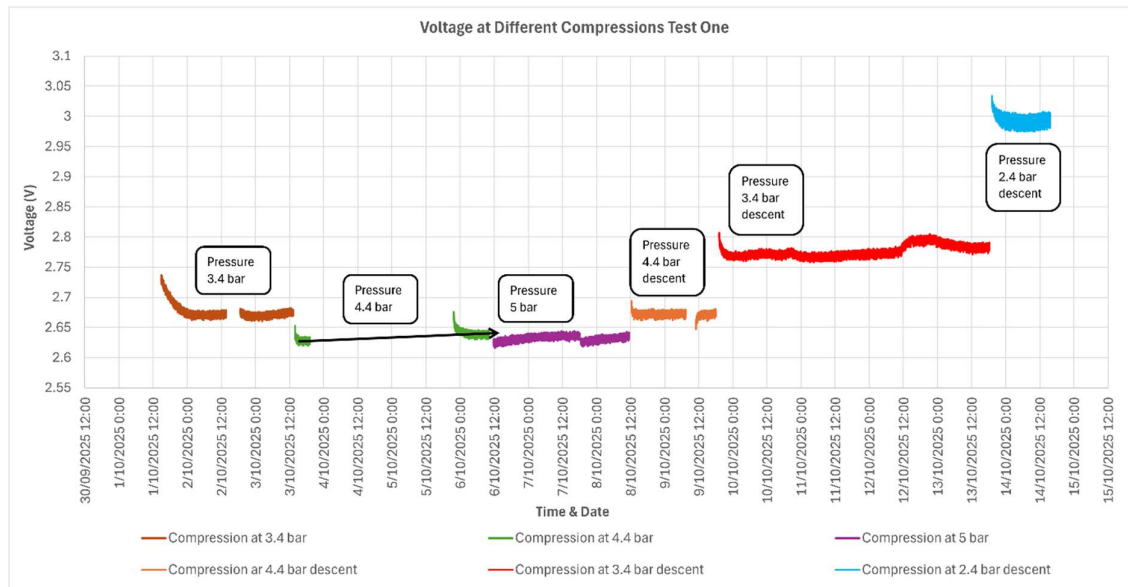


Figure 80 Degradation behavior at Compression 4.4 bar during the time (Test 1).

5.1.3.3 Effect of the Degradation at Compression 5 bar (Test 1)

Initially voltage decreases slightly around 2.63 V. Then an EIS test has been done, but only noises obtained. The system restarts again at pressure 5 bar and the graph show a reduction of the voltage at around 2.62 V and starts increasing the voltage up to around 2.64 V. Finally, another EIS test has been performed but without noises. In results, initially good performance, but faster long-term degradation. Higher pressure shows a better short-term performance, faster long-term degradation. The degradation rate of this compression level has been calculated for both parts separately. The initial voltage of the first part was 2.626 and the voltage ends at around 2.637 V for duration of 28.6 h, so the degradation rate of this compression level is $380 \mu V/h$. For the second part which is the part after the EIS test, the initial voltage was at around 2.627 V and the end voltage was 2.635 V for duration of 16.9 h, so the degradation rate of the second part at this compression level has been calculated as $470 \mu V/h$. The total degradation rate of this compression level has been done by the value of $850 \mu V/h$.

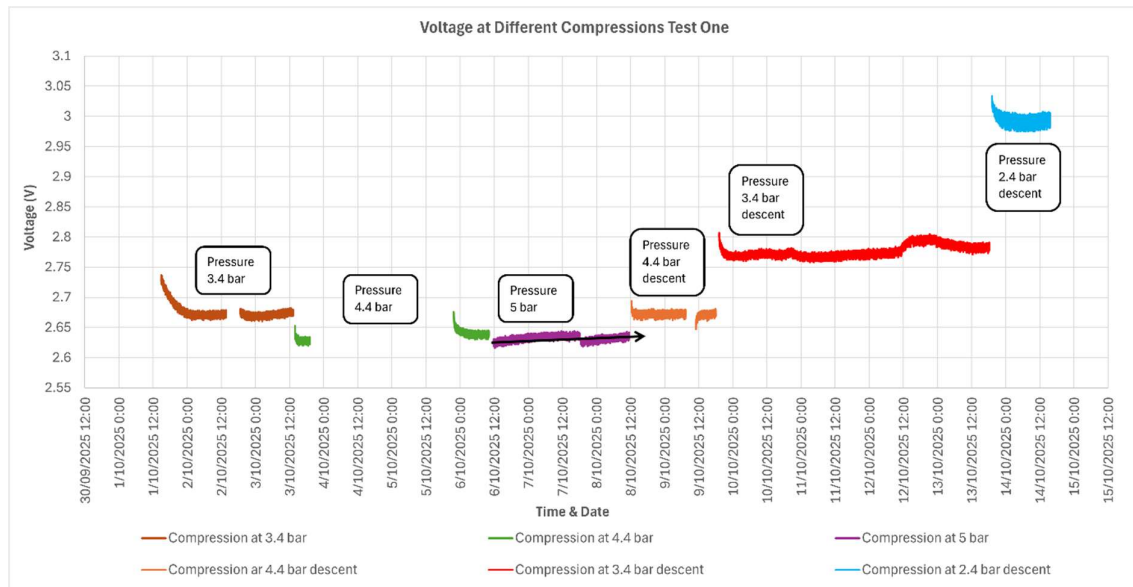


Figure 81 Degradation behavior at Compression 5 bar during the time (Test 1).

5.1.3.4 Effect of the Degradation at Compression 4.4 bar descent (Test 1)

At this compression level of 4.4 bar descent, the degradation rate has been calculated separately which means the operation of the system before and after power outage. According to the first part, the system operated for around 17 h, and the duration of stabilization reduced. The voltage increased from 2.671 V at the beginning of the interval to 2.6739 V at the end. Based on this change, the degradation rate was calculated as $170 \mu V/h$. For the second part after EIS test, the initial voltage was around 2.671 V and at the end was 2.673 V for duration of 5.22 h, so finally the degradation rate has been calculated as $72 \mu V/h$. The total degradation rate of this compression level has been done by the value of $242 \mu V/h$.

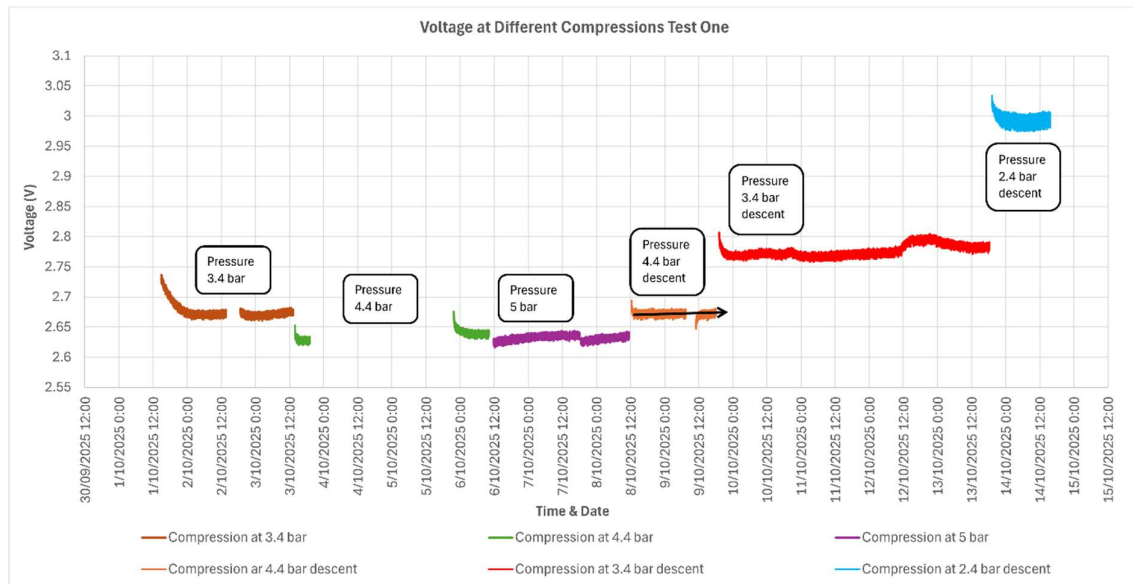


Figure 82 Degradation behavior at Compression 4.4 bar descent during the time (Test 1).

5.1.3.5 Effect of the Degradation at Compression 3.4 bar descent (Test 1)

At compression 3.4 bar descent, the system operated for around 90 h, but the duration of stabilization and the unstable region have been removed from the total hours. The voltage increased from 2.7674 V at the beginning of the interval to 2.7895 V at the end. Based on this change, the degradation rate was calculated as $160 \mu V/h$.

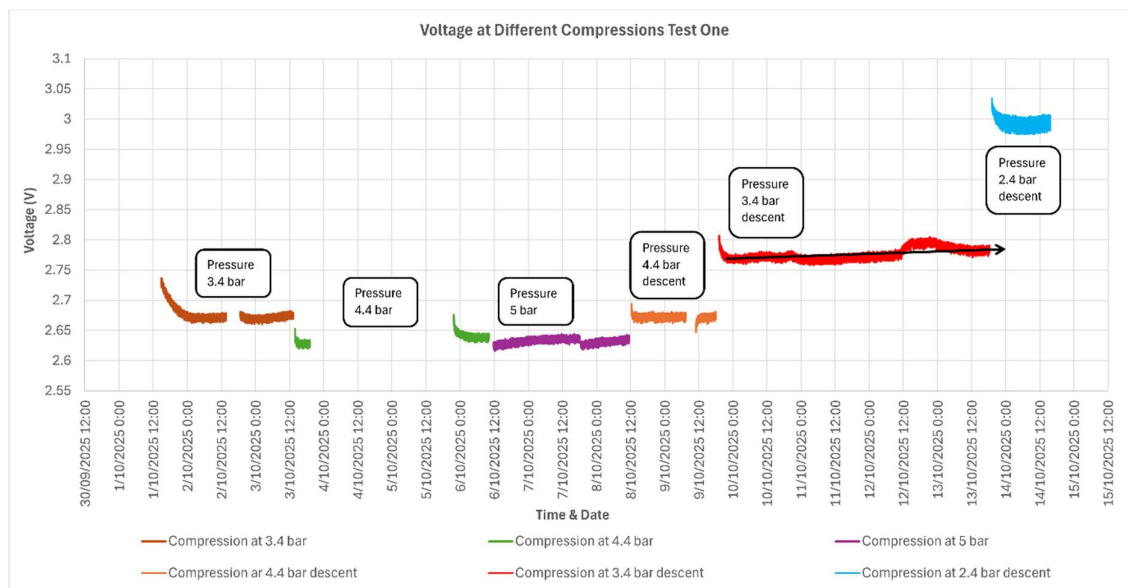


Figure 83 Degradation behavior at Compression 3.4 bar descent during the time (Test 1).

5.1.3.6 Effect of the Degradation at Compression 2.4 bar descent (Test 1)

After the descent pressure of the 3.4 bar, the compression level of 2.4 bar descent has been decided to see the behaviour of the voltage. As can be seen in the figure, the voltage rises rapidly due to the start of the system and increased up to around more than 3 volts and decreases fast and stabilizes at the beginning 2.993 V and at the end the voltage was around 2.999 V for duration of 7 hours, so the degradation rate of this compression level has been calculated as $760 \mu V/h$. Shortly after the EIS test the compression level of 1.4 bar descent has been tried, but it was impossible due to the high mass flow rate passing through the tubes.

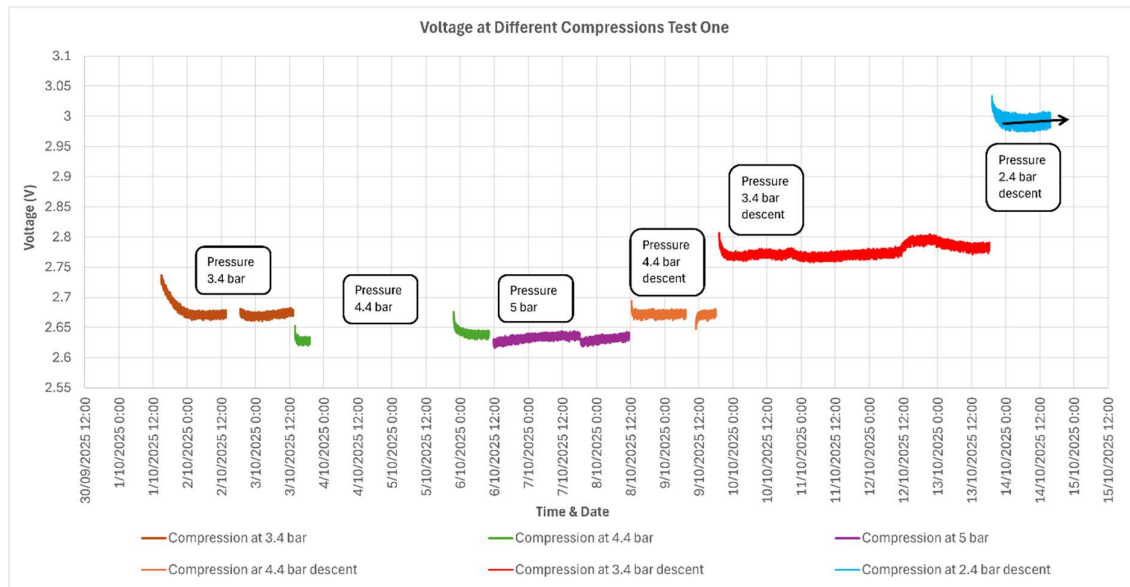


Figure 84 Degradation behavior at Compression 2.4 bar descent during the time (Test 1).

5.1.3.7 Analysis of the Degradation Effect (Test 1)

During the long-term operation test at 60 °C, the cell was operated under various pressures ranging from 3.4 to 5 bar. The initial voltage was approximately 2.6701 V, which gradually increased to about 2.999 V by the end of the experiment. Excluding the shutdown and normal phases, the cell was actively operating for around 253 h, during which the total voltage rise of 328.9 mV corresponds to an average degradation rate of about 328 $\mu\text{V}/\text{h}$. Higher pressures (up to 5 bar) improve performance momentarily but accelerate long-term degradation, likely due to increased compression and mass transport limitations. When the degradation is evaluated for each pressure level, a clear dependence is observed. At 3.4 bar and the descent compression of 3.4 bar, the degradation rate was relatively low around 191 and 160 $\mu\text{V}/\text{h}$, respectively and it reflects the optimum compression level, also stable operation and sufficient water supply. The compression level of 4.4 bar in both forward and descent resulted in a moderate rate of 270 $\mu\text{V}/\text{h}$ and 242 $\mu\text{V}/\text{h}$ respectively, while at 5 bar the degradation accelerated significantly to 850 $\mu\text{V}/\text{h}$. This behaviour is consistent with findings in literature Bernt & Gasteiger (2019) [66] and Wang et al. (2025) [67], showing that higher pressure improves gas removal and instantaneous performance but enhances mass-transport limitations that promote irreversible degradation.

Overall, Among the tested compression levels, 3.4 bar descent showed the lowest degradation rate of 160 $\mu\text{V}/\text{h}$. Pressures above 4.4 bar or below 3 bar led to significant instability and sharp increases in degradation up to 850 $\mu\text{V}/\text{h}$, indicating that the optimal operating pressure range is around 3.4 bar. higher operating pressures enhance instantaneous performance but accelerate long-term degradation through increased mechanical stress and reduced mass-transport uniform.

5.1.4 EIS Characterization at Different Compression level (Test 1)

The Nyquist plots in Figure 1 show the impedance response of the PEM electrolyzer cell under varying operating pressures. Each curve represents the complex impedance as a function of frequency, where the real part (Z') is plotted on the x-axis and the negative imaginary part ($-Z''$) on the y-axis. The overall shape of the plots provides insight into the electrochemical and transport processes occurring inside the cell, including ohmic resistance, charge transfer, and mass transport phenomena.

At the high-frequency region, the intercept of each curve with the x-axis corresponds to the ohmic resistance (R_s) of the cell. This component mainly represents the ionic resistance of the membrane, the electronic resistance of electrodes, and contact resistances. As the pressure increases from 3.4 to 5 bar, the R_s slightly shifts, which can be attributed to the mechanical compression of the cell components.

The mid-frequency semicircle represents the charge transfer resistance (R_{ct}) and the double-layer capacitance behavior at the electrode electrolyte interface. The diameter of this semicircle decreases as the cell pressure increases, indicating that the electrochemical reactions proceed more efficiently at higher pressures. This improvement is mainly due to enhanced gas removal from the catalyst surface higher pressure facilitates bubble detachment, keeps more active sites available for reaction, and therefore reduces R_{ct} . Consequently, the smaller semicircles observed at 4.4 and 5 bar suggest better electrochemical kinetics and improved overall cell performance.

In the low-frequency region, the curves show an elongated tail, which corresponds to mass transport limitations often modeled by a Warburg element. This region is influenced by the diffusion of gases and the removal of generated bubbles through the porous gas diffusion layers (GDLs).

5.1.4.1 Impedance Response Under Potentiostatic Operation (0 V)

5.1.4.1.1 Data quality assessment

The KK validation shows that impedance spectra obtained under voltage control are characterized by high level of residuals. In Nyquist plots it is observed that the shape is made of one semicircle in the HF and a tail in the mid frequency region. For this impedance spectra only ohmic resistance has been analyzed due to the high level of residual in KK analysis which shows a high noisy result.

5.1.4.1.2 Modelling

Figure 86 show impedance spectra obtained 60°C at different Compression level with potentiostatic tests. At 60 °C and a potentiostatic operating voltage of 0 V, the Nyquist plot compares the impedance response of the cell before and after applying different compression levels. For each pressure step, the high frequency intercept on the real axis represents the ohmic resistance, while the diameter of the main arc is related to the polarization (charge transfer and mass transport) contributions.

Figure 85 shows impedance spectra obtained at different compression level with potentiostatic tests. The high frequency arc seems affected by the pressure whereas the second incomplete arc bends down to the real axis with increasing pressures. The imaginary part of all HF arcs at 60°C reaches a maximum at ω_{max} equal to 193 Hz.

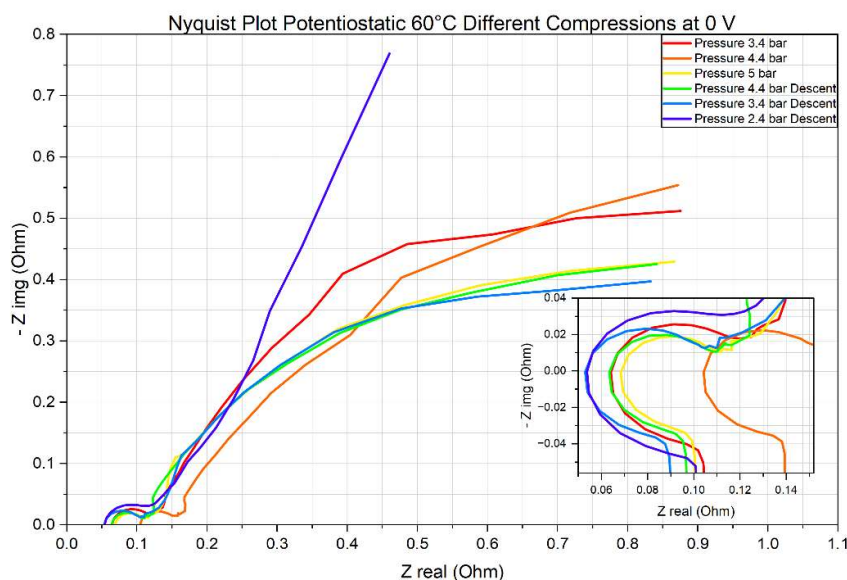


Figure 85 Nyquist plot Potentiostatic 60°C Different Pressures at 0 V (Test 1).

5.1.4.1.3 Discussion

Electrochemical impedance measurements performed at 0 V shows that the pressure from 3.4 bar to 5 bar the ohmic resistance does not follow a steady trend. The values of ohmic resistance from compression 3.4 bar to 4.4 bar increased gradually from $1.6 \Omega \cdot \text{cm}^2$ to $2.6 \Omega \cdot \text{cm}^2$, thereafter from pressure 3.4 bar to 5 bar ohmic resistance decreased within a range of $1.60\text{--}1.71 \Omega \cdot \text{cm}^2$. This gradual increase in ohmic resistance at compression 4.4 bar was due to the shutdown that happened during operation of this compression level. During the descent pressure sequence from 4.4 bar to 3.4 bar and 2.4 bar the ohmic resistance reduced from 1.5 to $1.3 \Omega \cdot \text{cm}^2$.

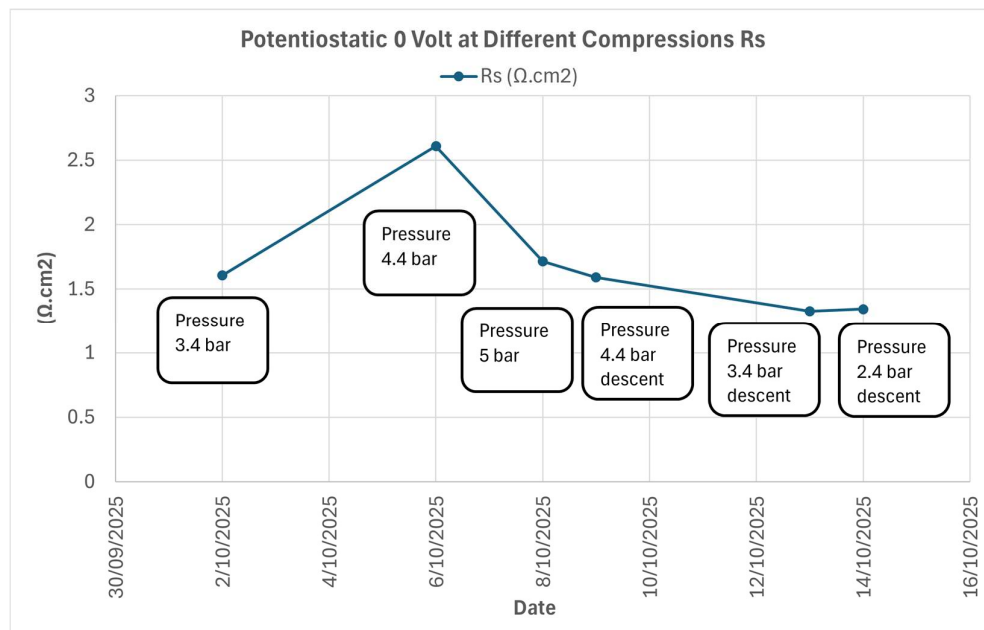


Figure 86 Trend of the ohmic resistance in the HF region at 60°C & 0 Volt (Test 1)

5.1.4.2 Impedance Response Under Potentiostatic Operation (0.5 V)

5.1.4.2.1 Data quality assessment

The KK validation shows that impedance spectra obtained under voltage control are characterized by high level of residuals. In Nyquist plots it is observed that the shape is made of one semicircle in the HF and a tail in the mid frequency region. Also, for this impedance spectra only ohmic resistance has been analyzed due to the high level of residual in KK analysis which shows a high noisy result.

5.1.4.2.2 Modelling

Figure 87 show impedance spectra obtained 60°C at different Compression level with potentiostatic tests. At 60 °C and a potentiostatic operating voltage of 0.5 V, the Nyquist plot compares the impedance response of the cell before and after applying different compression levels. The high frequency arc is affected by the pressure; value of pressure increases this tends to reduce its diameter. A tail appears at lower frequencies, maybe it is an insight of a second semicircle.

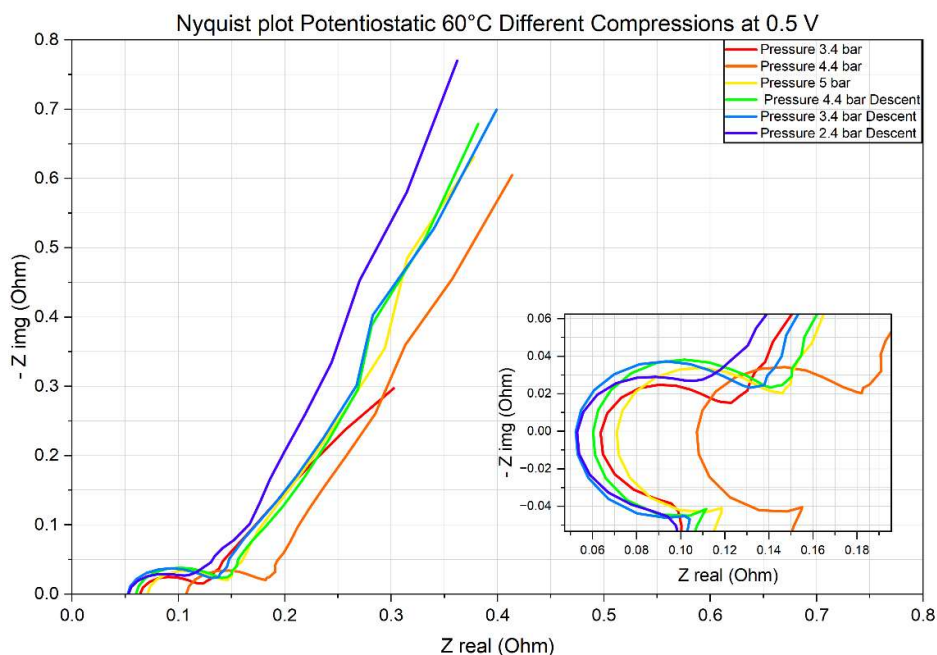


Figure 87 Nyquist plot Potentiostatic 60°C Different Pressures at 0.5 V (Test 1).

5.1.4.2.3 Discussion

As the pressure increases from 3.4 bar to 4.4 bar, R_s increased from $1.59 \Omega \cdot \text{cm}^2$ to $2.68 \Omega \cdot \text{cm}^2$ and then decreased to $1.77 \Omega \cdot \text{cm}^2$ at compression 5 bar. During the descent pressure from 4.4 bar to 3.4 bar and 2.4 bar, ohmic resistance decreases from $1.50 \Omega \cdot \text{cm}^2$ to $1.32 \Omega \cdot \text{cm}^2$. The shutdown that occurs during the compression level of 4.4 bar effects on a gradual increase of ohmic resistance.

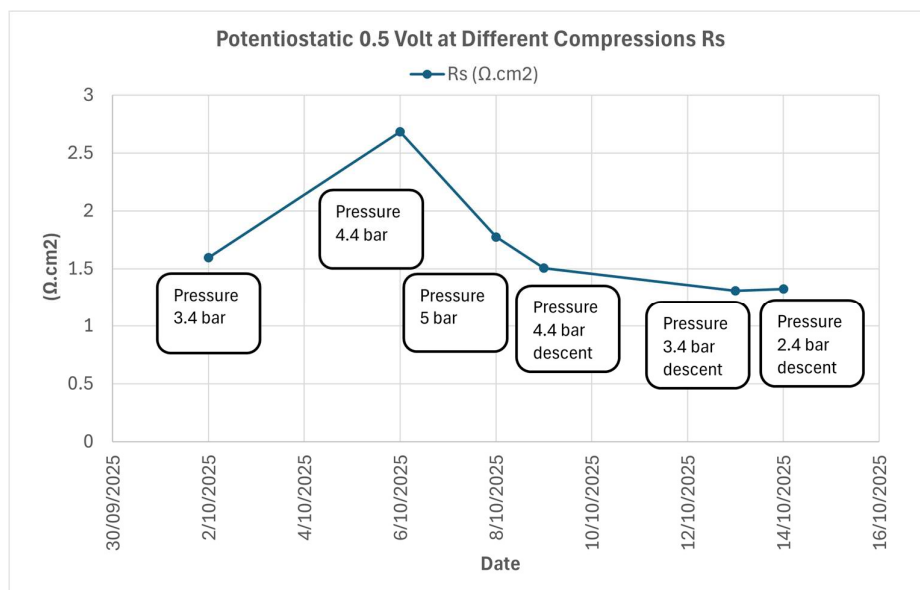


Figure 88 Trend of the ohmic resistance in the HF region at 60°C & 0.5 Volt (Test 1).

5.1.4.3 Impedance Response Under Potentiostatic Operation (1V)

5.1.4.3.1 Data quality assessment

The KK validation shows that impedance spectra obtained under voltage control are characterized by moderate level of residuals. In Nyquist plots it is observed that the shape is made of one semicircle in the HF and a tail in the mid frequency region. Also, for this impedance spectra only ohmic resistance has been analyzed due to the high level of residual in KK analysis which shows a noisy result.

5.1.4.3.2 Modelling

Figure 89, show impedance spectra obtained 60°C at different Compression level with potentiostatic tests. At 60°C and a potentiostatic operating voltage of 1 V. At 60°C and a potentiostatic operating voltage of 1 V, the Nyquist plot compares the impedance response of the cell before and after applying different compression levels. The high frequency arc is affected by the pressure; value of pressure increases this tends to reduce its diameter and shows a decreasing behavior in the descent compression from 4.4 bar to 2.4 bar. A tail appears at lower frequencies, maybe it is an insight of a second semicircle.

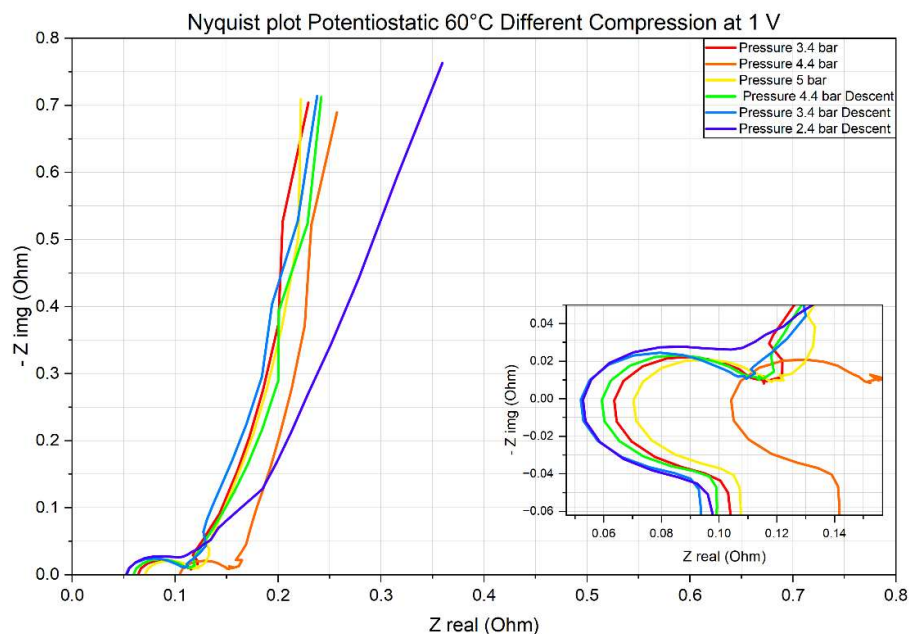


Figure 89 Nyquist plot Potentiostatic 60°C Different Pressures at 1 V (Test 1).

5.1.4.3.3 Discussion

As can be seen, the pressure increases from 3.4 bar to 4.4 bar, ohmic resistance increased gradually from $1.59 \Omega \cdot \text{cm}^2$ to $2.60 \Omega \cdot \text{cm}^2$ and then decreased to $1.76 \Omega \cdot \text{cm}^2$ at pressure 5 bar. In the descent pressure region, ohmic resistance decreases from $1.48 \Omega \cdot \text{cm}^2$ to $1.32 \Omega \cdot \text{cm}^2$ at compression 4.4 bar to 2.4 bar descent.

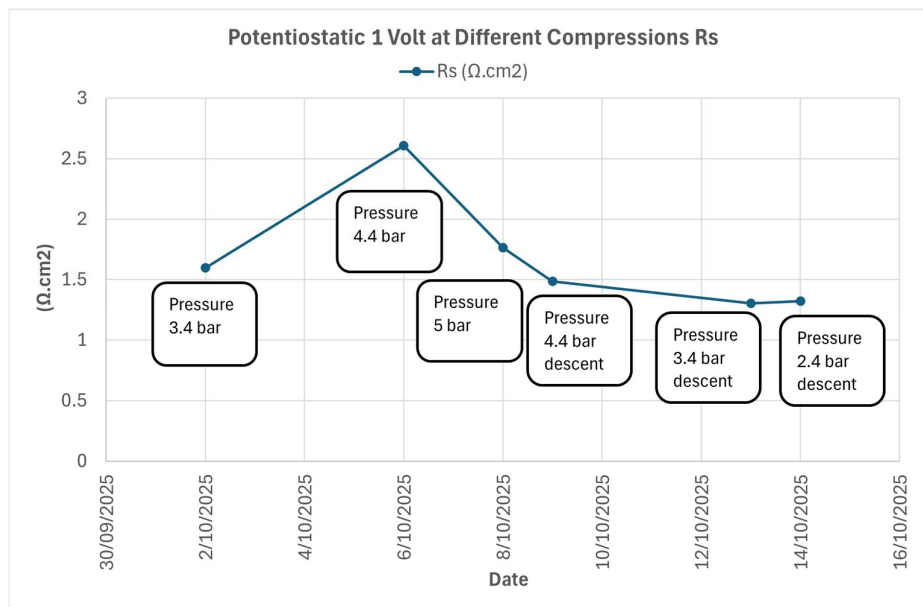


Figure 90 Trend of the ohmic resistance in the HF region at 60°C & 1 Volt (Test 1).

5.1.4.4 Impedance Response Under Potentiostatic Operation (1.3V)

5.1.4.4.1 Data quality assessment

The KK validation shows that potentiostatic mode have some tests with low quality impedance spectra in low frequency region. The ECM used to fit experimental data is LR(QR)W due to the presence of noise at low frequency which makes difficult to get good results.

5.1.4.4.2 Modelling

The figure below shows a comparison between impedance spectra obtained with potentiostatic mode at 1.3 V with different compression levels and EIS tests performed. The shape of the impedance shows a high frequency semicircle and a tail for low frequency region mostly affected by noise. The high frequency intercept with the real axis is not much affected by the variation of the compression only on 4.4 bar, instead height and diameter of the HF arc show a substantial increase with increasing compression level.

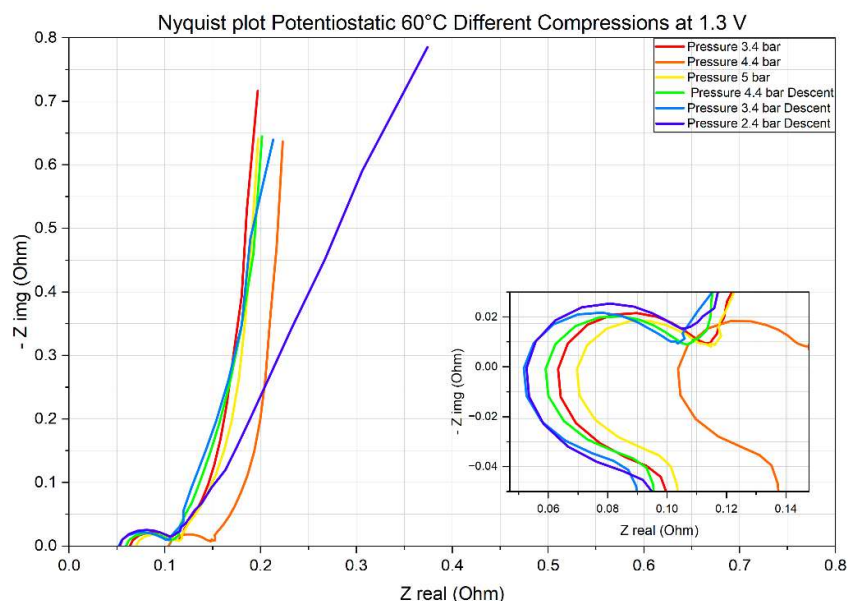


Figure 91 Nyquist plot Potentiostatic 60°C Different Compressions at 1.3 V (Test 1).

5.1.4.4.3 Discussion

Figure 92, shows different resistances such as ohmic resistance and charge transfer resistance. EIS measurements were performed after each compression step at 3.4, 4.4 and 5 bar also for the descent compression of 4.4 bar to 3.4 bar to 2.4 bar.

The ohmic resistance R_s at compression 3.4 bar to 4.4 bar increased gradually from $1.58 \Omega \cdot \text{cm}^2$ to $2.59 \Omega \cdot \text{cm}^2$ due to a shutdown occurred and thereafter from 4.4 bar to 5 bar decreased to $1.74 \Omega \cdot \text{cm}^2$. For the descent compression the ohmic resistance decreases linearly from $1.48 \Omega \cdot \text{cm}^2$ to $1.31 \Omega \cdot \text{cm}^2$ from compression level of 4.4 bar descent to 2.4 bar descent. The charge-transfer resistance shows a moderate increase at compression level of 3.4 bar forward to 3.4 bar descent from about 0.89 to $1.61 \Omega \cdot \text{cm}^2$ then decreases back to $0.80 \Omega \cdot \text{cm}^2$ at compression level of 2.4 bar descent.

The capacitance of the high frequency region shows a linear decreasing behavior from compression 3.4 bar forward to compression 4.4 bar descent from 0.008 to 0.006 F, at compression 4.4 bar descent to 3.4 bar descent the capacitance increased gradually to 0.01 F and decreased to the value of 0.004 F at compression level of 2.4 bar descent. This indicating high compression resulting in reduction of the capacitance.

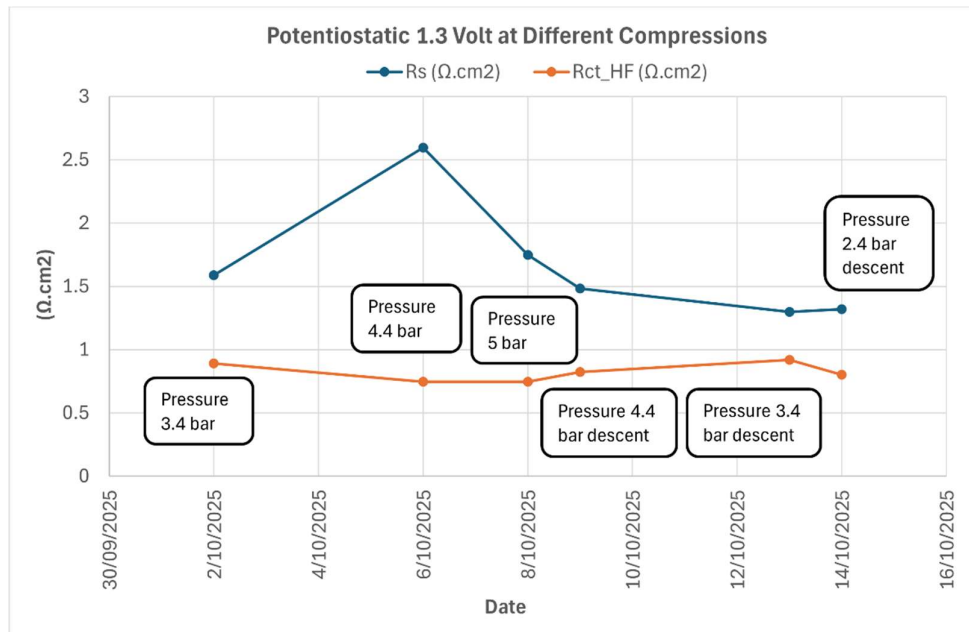


Figure 92 Trend of the ohmic resistance & the charge transfer resistance in the HF region at 60°C & 1.3 Volt (Test 1).

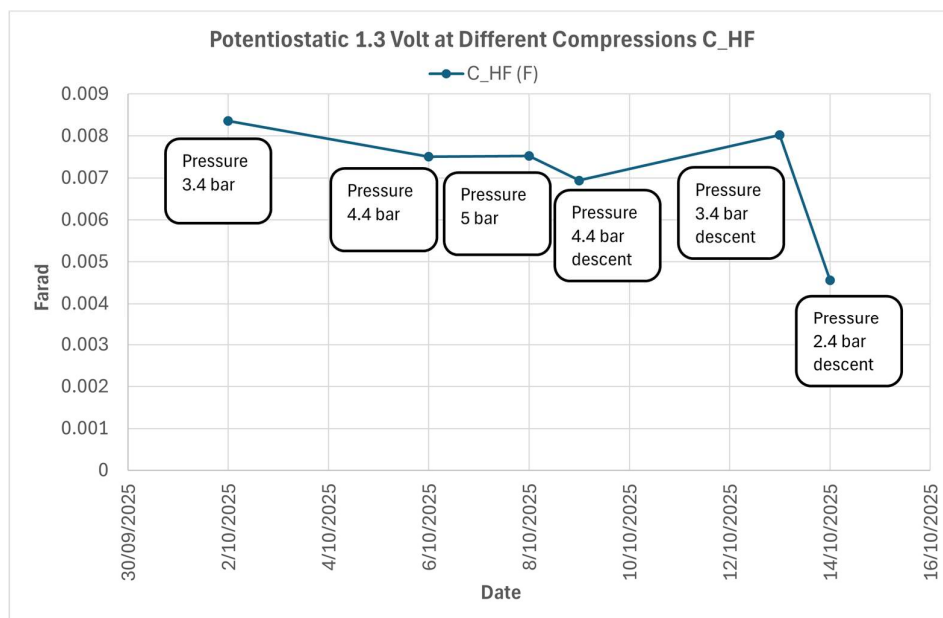


Figure 93 Trend of the capacitance in the HF region at 60°C & 1.3 Volt (Test 1).

5.1.4.5 Impedance Response Under Potentiostatic Operation (1.5V)

5.1.4.5.1 Data quality assessment

The KK validation shows that impedance spectra obtained under voltage control are characterized by very low residuals. In Nyquist plots it is observed that the shape is made of two semicircles, one in the HF and a second semicircle in the mid frequency region. In this case, the equivalent circuit transitions LR(QR)(QR) used to fit experimental data.

5.1.4.5.2 Modelling

Figure 94, show impedance spectra obtained 60°C at different Compression level with potentiostatic tests. At 60 °C and a potentiostatic operating voltage of 1.5 V, the Nyquist plot compares the impedance response at different compression levels. Impedance spectra characterized by an arc at high frequency and a second arc at mid-low frequency. The low frequency features show a dependence on compression the descent compression level from 3.4 bar to 5 bar tends to increase its diameter. Same consideration for tests performed, the LF arc shows a dependence on the compression, and it tends to increase as the compression level increased. In general, the compressed states tend to show a reduced real-axis intercept and a smaller arc diameter, indicating a decrease in both ohmic and polarization resistances.

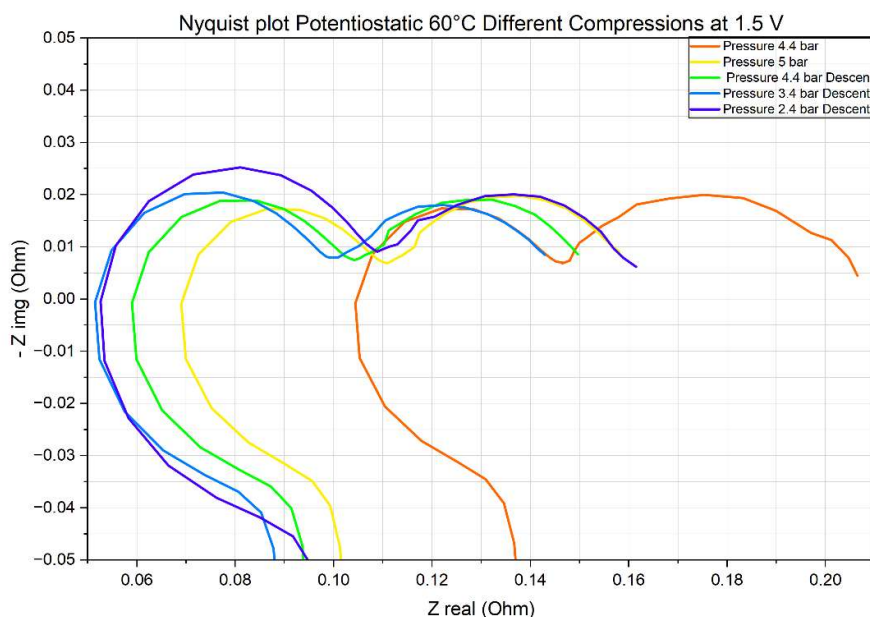


Figure 94 Nyquist plot Potentiostatic 60°C Different Pressures at 1.5 V (Test 1).

5.1.4.5.3 Discussion

EIS measurements were performed at each compression step at 3.4, 4.4 and 5 bar also at descent compression of 4.4 bar, 3.4 bar and 2.4 bar. The EIS data of 3.4 bar in forward has been lost due to an unexpected shutdown. The ohmic resistance R_s from compression 4.4 bar forward to 2.4 bar descent decreased from $2.61 \Omega \cdot \text{cm}^2$ to $1.31 \Omega \cdot \text{cm}^2$. On the other hand, The high-frequency charge-transfer resistance R_{ct} shows an increase from about 0.97 to 1.27

$\Omega \cdot \text{cm}^2$. Also, the low-frequency mass-transport resistance R_m remains nearly constant with a slight increase from roughly 1.47 to $1.54 \Omega \cdot \text{cm}^2$.

The capacitance of the high frequency region decreased from 0.017 to 0.012 F, indicating a compression reduction of the capacitance. This behavior is consistent with improved electrode membrane contact at higher compression. The low-frequency capacitance shows an overall fluctuation trend over the period, decreasing from about 7.69 F to approximately 6.35 F. This behavior suggests an enhancement of the capacitive contribution associated with slow mass-transport processes after the different compression steps.

Overall, the total polarization resistance R_p shows a clear decreasing trend, dropping from roughly $5.06 \Omega \cdot \text{cm}^2$ at the beginning of the measurements to about $4.13 \Omega \cdot \text{cm}^2$, where it then stabilizes.

The total resistance decreased from $5 \Omega \cdot \text{cm}^2$ to $4 \Omega \cdot \text{cm}^2$. The compression of 4.4 bar forward shows the highest total resistance on the other hand the lowest value of total resistance was obtained by the compression 3.4 bar descent. The combined behavior of R_s , R_{ct} and R_m confirms that cell compression mainly benefits the polarization processes (charge transfer and mass transport), while preserving a nearly constant ohmic contribution under potentiostatic operation at 1.5 V.

The total polarization resistance R_p varies with each compression step but shows an overall decreasing tendency. This reduction indicates improved charge-transfer and mass-transport performance in descent compression levels.

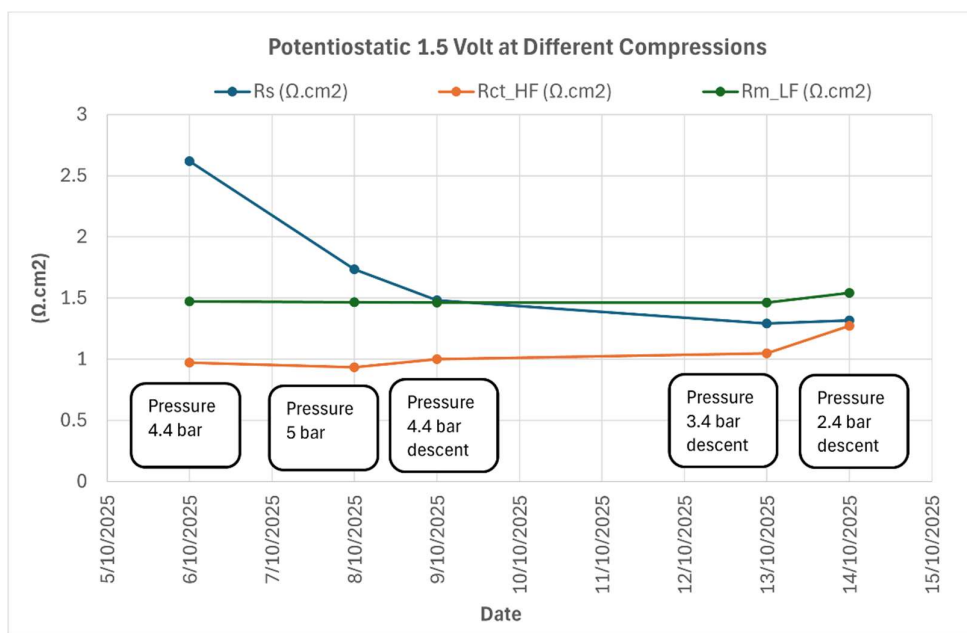


Figure 95 Trend of the ohmic resistance, the charge transfer resistance & mass transport resistance at 60°C & 1.5 Volt (Test 1).

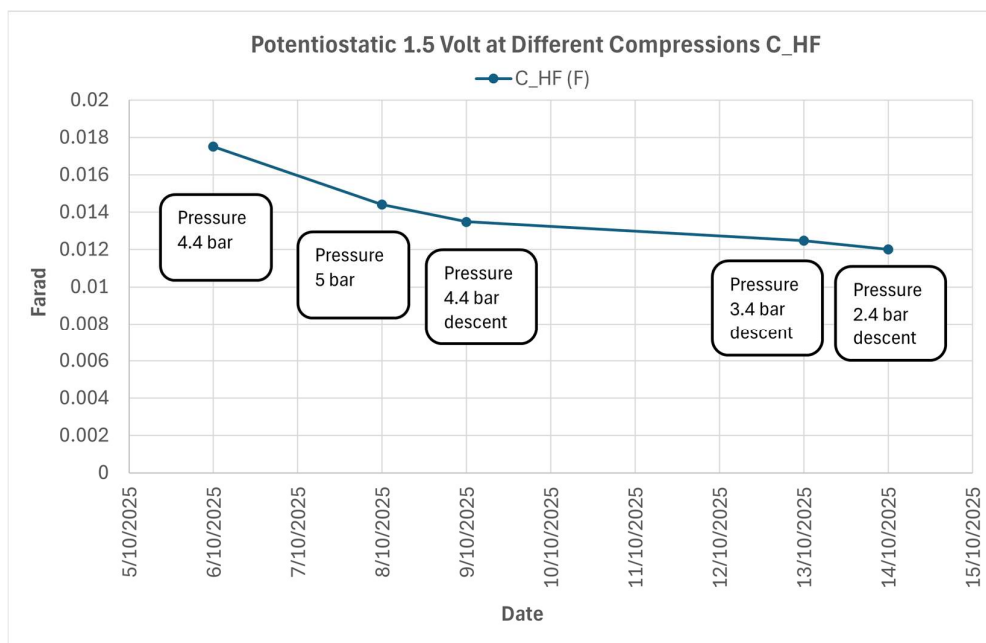


Figure 96 Trend of the capacitance in the HF region at 60°C & 1.5 Volt (Test 1).

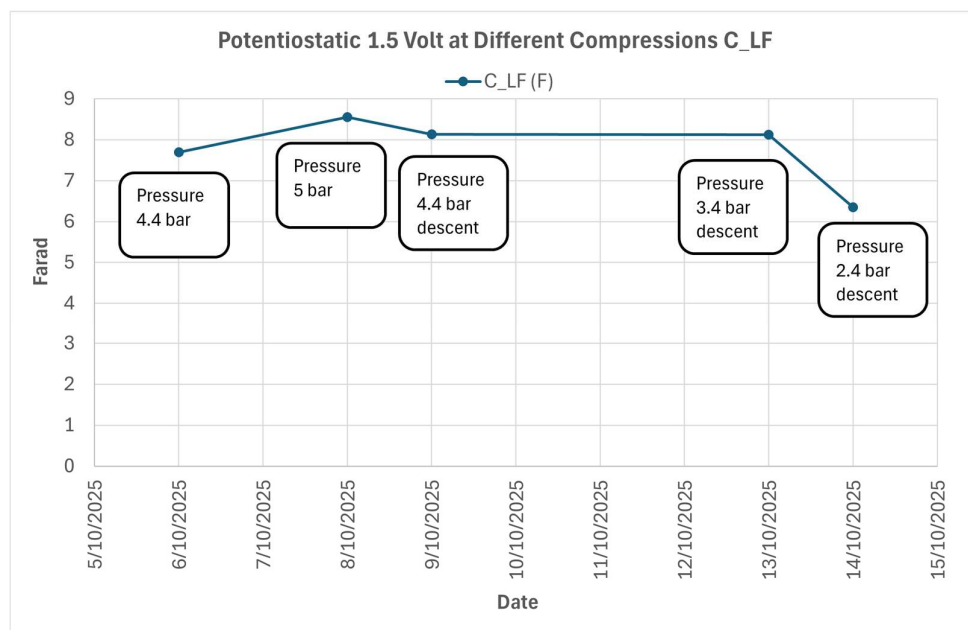


Figure 97 Trend of the capacitance in the LF region at 60°C & 1.5 Volt (Test 1).

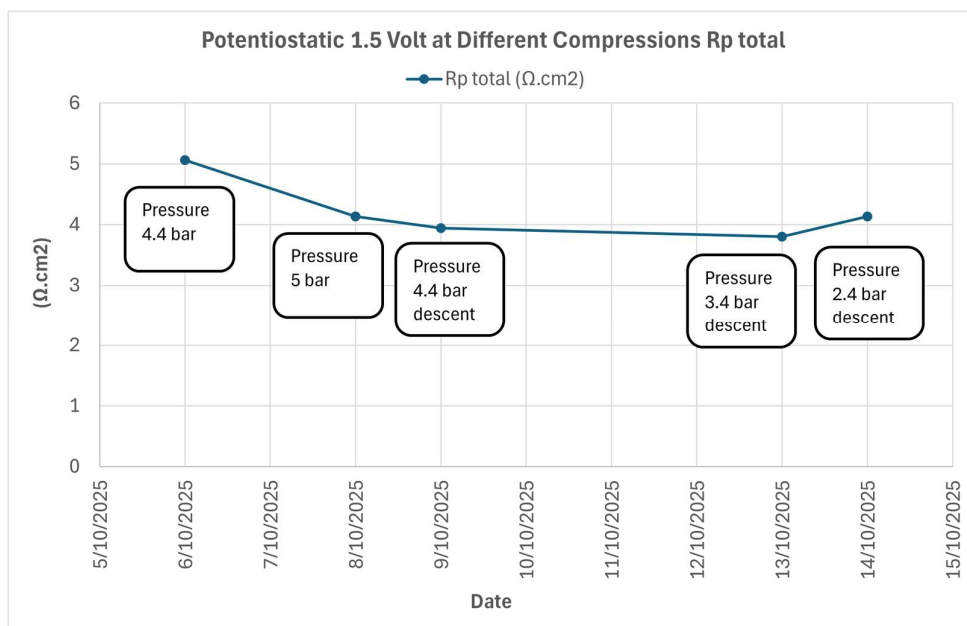


Figure 98 Trend of the total resistance at 60°C & 1.5 Volt (Test 1).

5.1.4.5.4 Discussion

This plot summarizes the evolution of the main resistance components extracted from all EIS measurements performed at each compression step at 0V to 1.5 V. Considering the range 0-1.5 V, it is possible to notice that the ohmic resistance is varying with voltage (from 1.60 to 1.31 Ωcm2). The highest ohmic resistance refers to the compression 4.4 bar which the value is 2.6 Ωcm2 from 0-1.5 V and then decreased. On the other hand, about the HF arc, as the results was obtained only for 1.3 V and 1.5 V, the charge transfer resistance decreased from 0.89 to 1.61 Ωcm2 at compression 3.4 bar to 3.4 bar descent then decreased to 1.27 Ωcm2 at compression 2.4 bar descent. The capacitance at high frequency region decreased from 0.017 F to 0.012 F.

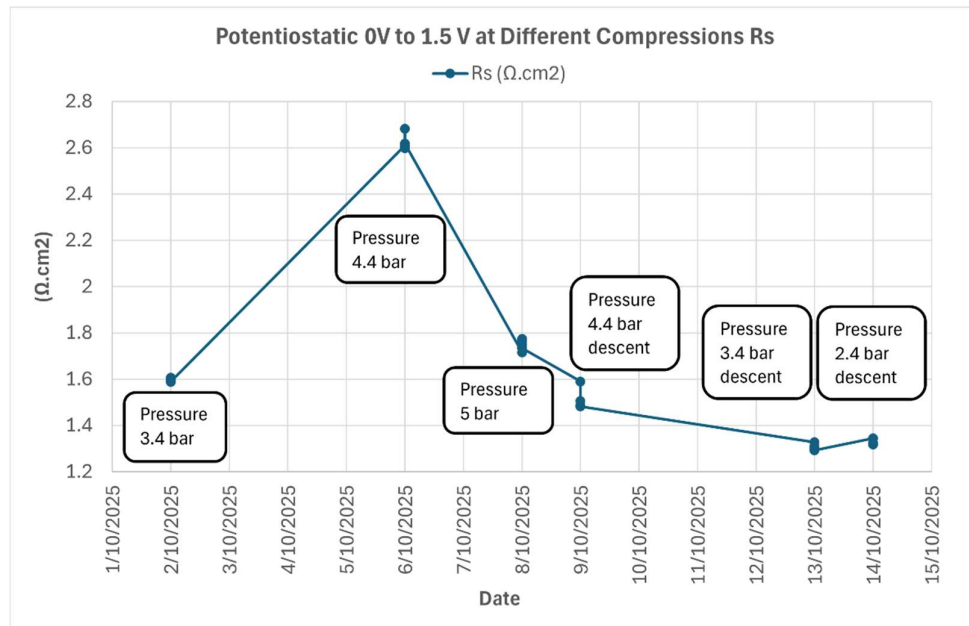


Figure 99 Trend of the ohmic resistance at different compressions & 60°C from 0 Volt to 1.5 Volt (Test 1).

5.2 Results of the Test Two

The aim of the test two is to perform electrochemical characterization of the new cell by carrying out the Polarization Curve (I-V Curve), EIS measurements, degradation and extrapolation. The test two is performed at 60°C, and different compressions (2.4 bar, 3.4 bar, 4.4 bar, 4.9 bar, 4.4 bar descent, 3.4 bar descent, 2.4 bar descent) mass flow rate of approximately 200-300 mL/min.

Potentiostatic test, performed for the test two at different DC value of voltage (1.3 V, 1.5 V) vs reference by varying the frequency from 100 kHz to 10 mHz in single sine mode and using a sinusoidal excitation signal of 10 mV root mean square (rms).

Galvanostatic test, performed at 1000 mA (RMS) AC current of (1.3 A, 1.5 A) is superimposed on DC currents between 0–2000 mA, sweeping frequencies from 10 kHz to 1 mHz.

5.2.1 EIS Analysis & Protocols of the Test Two

The provided graph shows the cell voltage of a Proton Exchange Membrane Water Electrolyzer (PEMWE) as a function of time and date, starting from 28 January 2026 to approximately January 11, 2026 (about 15 days). The cell operates at a constant current density of 1 A/cm², a temperature of 60°C and the cell area is 25 cm². These conditions are typical for evaluating long-term performance in PEMWE systems, where voltage is a key indicator of efficiency. The interventions like pressure adjustments, shutdowns, or restarts, often restoring lower voltage levels.

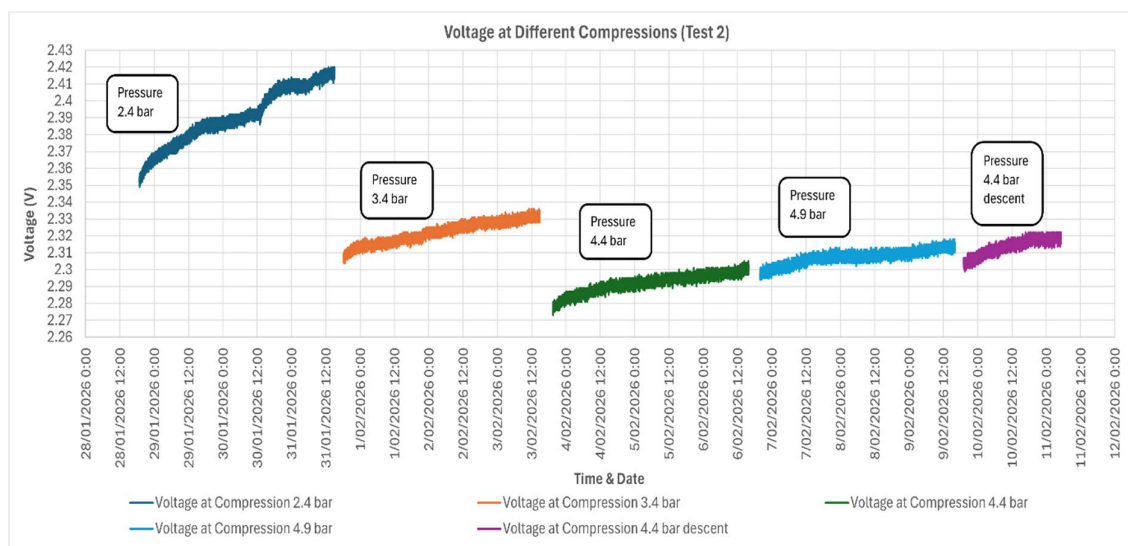


Figure 100 Trend of the voltage at different compressions during time (Test 2).

For calculation of the degradation for each compression level, a calculation has been made in which the degradation of the first compression at 2.4 bar has been calculated as before but for calculation of the other compression levels, the cell already degraded. So for the rest of the compression levels, the degradation occurred in previous compression levels has been eliminated for better comparison of the compression impacts on the performance. Figure 102 shows this behavior.

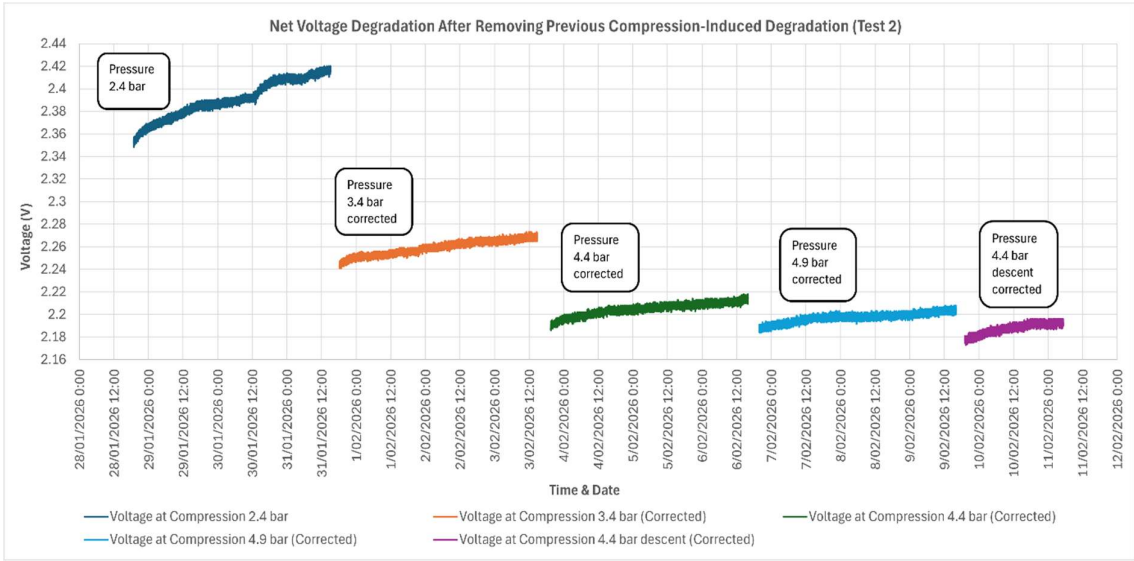


Figure 101 Net voltage degradation after removing previous compression- induced degradation.

As can be seen, the voltage decreased when the degradation occurred in previous compression levels has been eliminated. Figure 103, shows both the voltage curve and the graph of eliminated degradation of the previous compression levels.

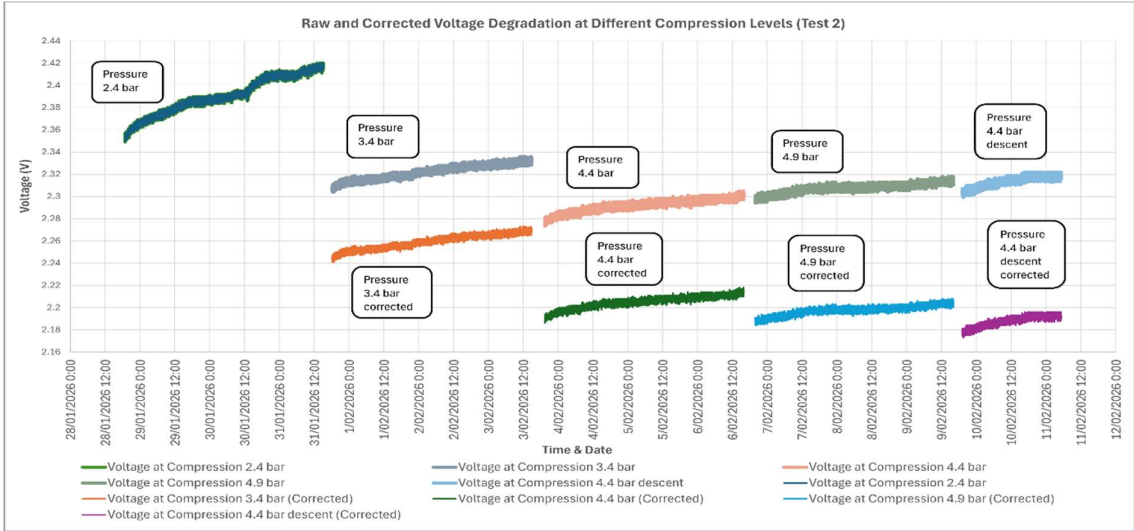


Figure 102 Raw & corrected voltage degradation at different compression levels.

5.3 Validation of the experimental results

The MEA experienced a conditioning phase to guarantee reliable and reproducible cell performance, before starting the polarization measurements. The process started with a hydration phase. In hydration phase, the demineralized water was circulated through the electrolyzer under open-circuit conditions for several hours. This procedure enabling the membrane to achieve a sufficient level of proton conductivity. After hydration was finished, the activation phase began. During this phase, the applied current was gradually modified to enable the cell voltage to achieve a stable operating level. This preliminary sequence ensured that the electrolyzer functioned under stable and clearly defined conditions before operating the experimental tests. According to the recommendations provided by the membrane manufacturer, after these two steps, an additional stabilization check was performed to verify the stability of the cell response over time. For this purpose, the electrolyzer was operated at a constant current density of 1 A/cm² for a specific period, and the corresponding voltage evolution was monitored. The outcome of this stabilization step is presented in Figure 103.

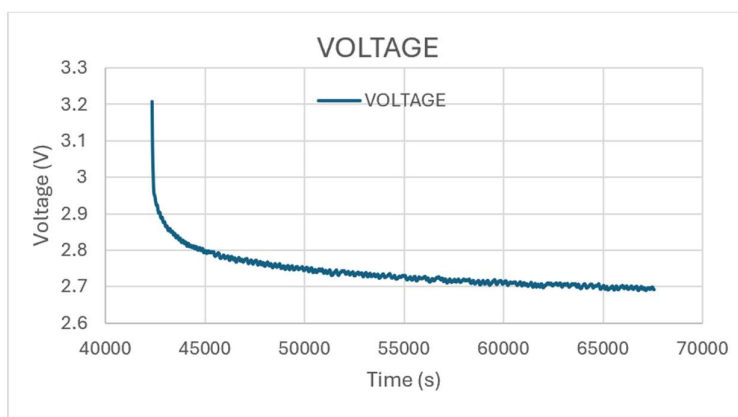


Figure 103 Stabilization procedure on the cell.

5.3.1 Polarization curves

The polarization curve was recorded to evaluate the electrochemical performance of the MEA, after completing the hydration and activation stages. The test included of monitoring the cell voltage under steady-state conditions while changing the applied current, on the other hand, keeping the operating temperature and outlet pressure constant. All other parameters remained unchanged, while each measurement step corresponded to a fixed time interval during which the voltage was acquired. A timestep of 30 s was selected, as preliminary checks confirmed that the cell voltage stabilized within this period. All experiments were carried out at constant pressure, temperature, and timestep, while the current was increased from 0 to 2 A and then decreased back to 0 A. An example of the applied steps is shown in Table 5.

Table 5: Step set on LabView.

Step	Current [A]	Pressure [bar]	Temperature [°C]	Timestep [s]
1	0.25	1	60	30
2	0.25	1	60	30

3	0.25	1	60	30
4	0.61	1	60	30
...	...	...	...	30
49	20	1	60	30
50	20	1	60	30
51	22.53	1	60	30
...	...	...	...	...
65	50	1	60	30
66	50	1	60	30

The first configuration results are shown in Figure 104 and Figure 105 Polarization curves are recorded at different compression levels and show a clear dependence of the cell voltage on the applied cell compression. At a given current, the voltage varies with compression, indicating a trade-off between improved electrical contact and increased mass transport limitations. Among the different compression levels, the curve of compression 4.4 bar consistently shows the lowest voltage at a given current. This indicates that this compression provides the most suitable balance between electrical contact and mass-transport behavior within the MEA. For example, 2.4 and 3.4 bar, which are the lower compression levels, show the results in higher ohmic resistance due to insufficient mechanical contact, while excessive compression 4.9 bar may limit gas and water pathways, increasing mass-transport losses. Therefore, 4.4 bar can be considered the optimal compression condition for the tested configuration. At 1 A/cm², the cell compression at 4.4 bar showed the lowest voltage, indicating the highest performance compared to 3.4, 2.4 and 4.9 bar.

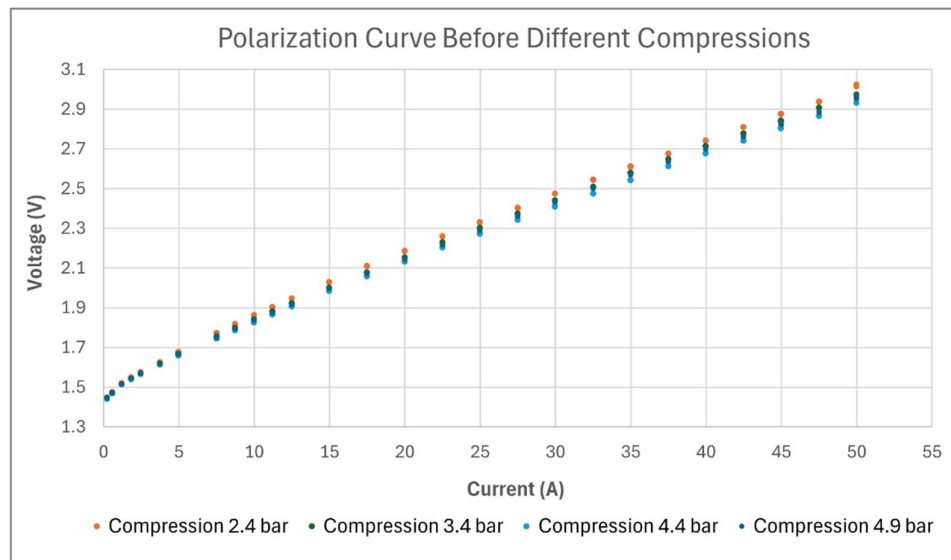


Figure 104 Polarization curves in constant cell temperature 60°C & before different compression levels.

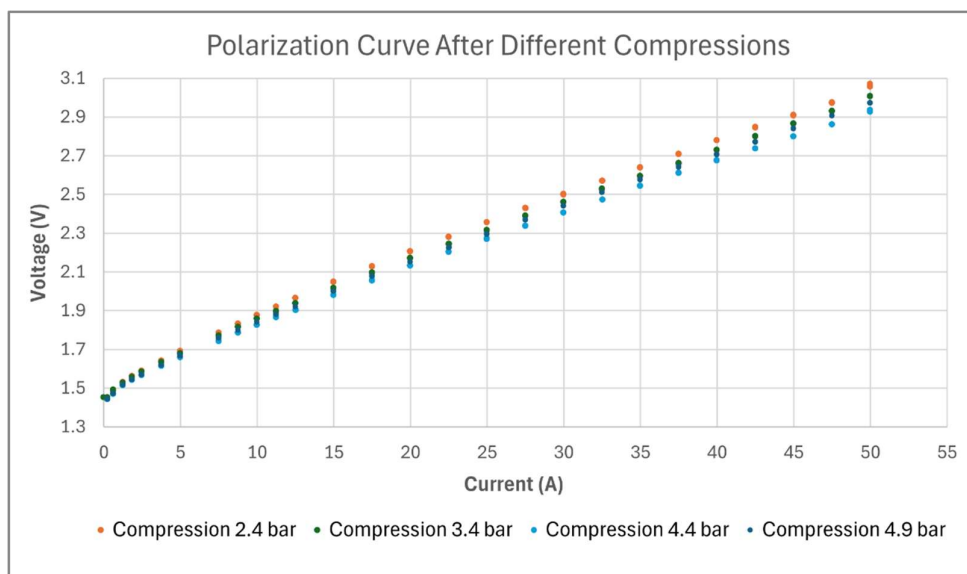


Figure 105 Polarization curves in constant cell temperature 60°C & after different compression levels.

5.4 Analysis of Voltage Behavior Under Different Conditions (Test 2)

Degradation test performed at 60°C and at different compression levels and constant current density of 1 A/cm²:

The degradation test is carried out at a constant current density of 1 A/cm² and the cell voltage increase over time is monitored. It consists of two tests one test carried out in October and another one in January. Test one already discussed. The second one was performed at temperature of 60°C and different compression levels from 2.4 bar to 3.4 bar followed by 4.4 bar and 4.9 bar and after the descent compression of 4.4 bar. During the first one, EIS tests have been performed after stabilization of the voltage at each compression level.

5.4.1 Effect of the Degradation at Compression 2.4 bar (Test 2)

During Test 2, the cell was started to operate at a compression pressure of 2.4 bar following an initial EIS measurement before and after each compression level. The system remained under continuous operation from 28 January 2026 at 16:50:59 until 31 January 2026 at 15:07:55, resulting in an effective operating duration of 68.27 h after removing the initial unstable region. The beginning of interval voltage was 2.354 V, while the final voltage at the end of the operating period reached 2.417 V.

The degradation rate was calculated directly from the voltage increase over the measured duration, yielding a value of 0.00092281 V/h, corresponding to approximately 922 $\mu\text{V}/\text{h}$. This result shows a clear and voltage rise during the 2.4 bar operation. This result indicating a measurable degradation trend within the observed timeframe. At the end of the test in compression 2.4 another EIS measured following by another EIS before starting the next compression level. The extracted degradation rate will be used for comparison with the other pressure levels investigated in Test 2.

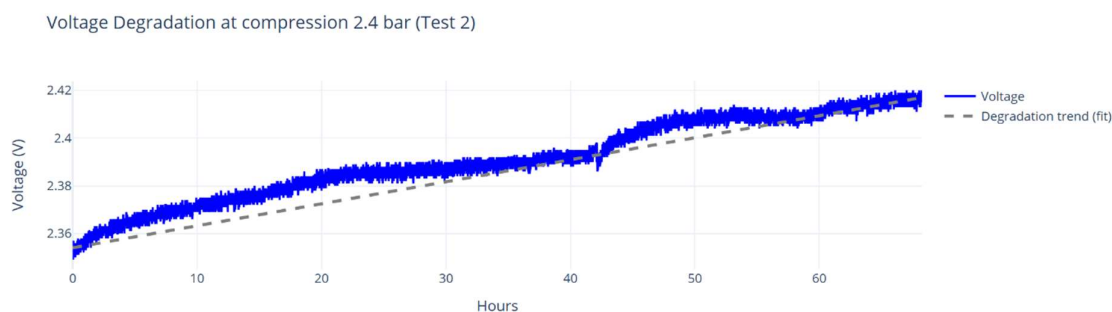


Figure 106 Degradation behavior at Compression 2.4 bar during the time (Test 2).

5.4.2 Effect of the Degradation at Compression 3.4 bar (Test 2)

After compression 2.4 bar, the cell continued to operate at a compression pressure of 3.4 bar and an initial EIS measurement. The system remained under continuous operation from 31 January 2026 at 17:44:26 until 3 February 2026 at 14:51:23. This shows an effective operating duration of 68.67 h after removing the initial unstable region. The beginning-of-interval voltage

for compression level of 3.4 bar was 2.308 V, while the final voltage at the end of the operating period reached 2.332 V.

The degradation rate was calculated directly from the voltage increase over the measured duration, and showed a value of 0.0003495 V/h, corresponding to approximately 349 $\mu\text{V}/\text{h}$.

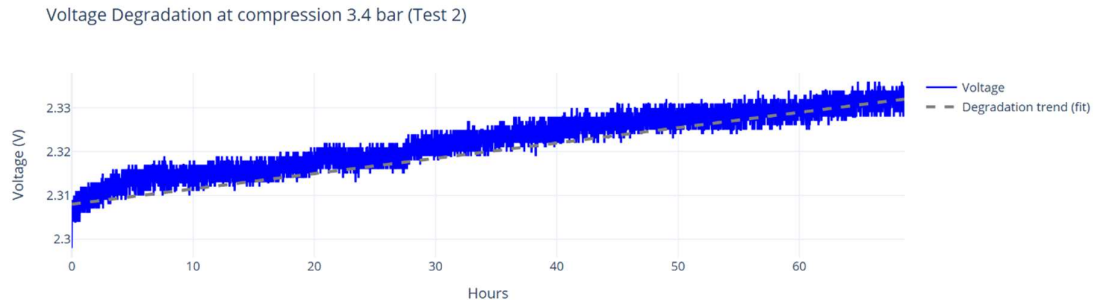


Figure 107 Degradation behavior at Compression 3.4 bar during the time (Test 2).

5.4.3 Effect of the Degradation at Compression 4.4 bar (Test 2)

At compression 4.4 bar, the degradation was performed from 3 February 2026 until 6 February 2026 a total number of 68.43 h of stable operation. The voltage rise from 2.278 V to 2.301 V over this interval corresponds to a degradation rate of 0.00033611 V/h and 336 $\mu\text{V}/\text{h}$. The observed increase shows a measurable and continuous degradation pattern characteristic of the operating conditions applied in this pressure level.

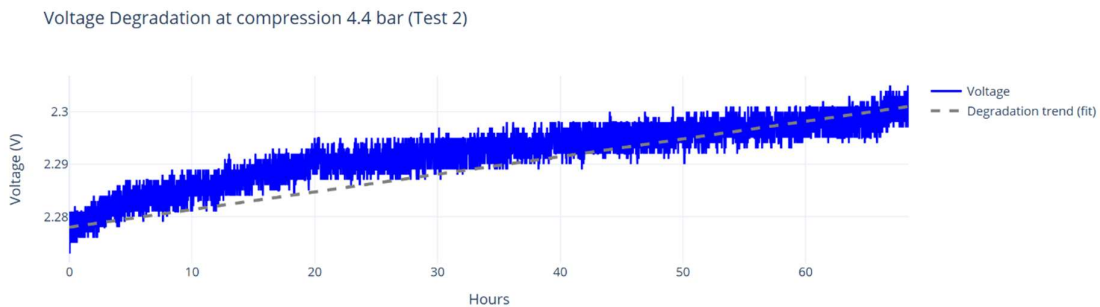


Figure 108 Degradation behavior at Compression 4.4 bar during the time (Test 2).

5.4.4 Effect of the Degradation at Compression 4.9 bar (Test 2)

Under the operating pressure of 4.9 bar, the system ran from 6 February at 19:55:17 until 9 February 2026 at 16:06:40, for 68.18 h. During this period the voltage rose from 2.298 V to 2.314 V. The calculated degradation rate of 0.00023467 V/h and 234 $\mu\text{V}/\text{h}$.

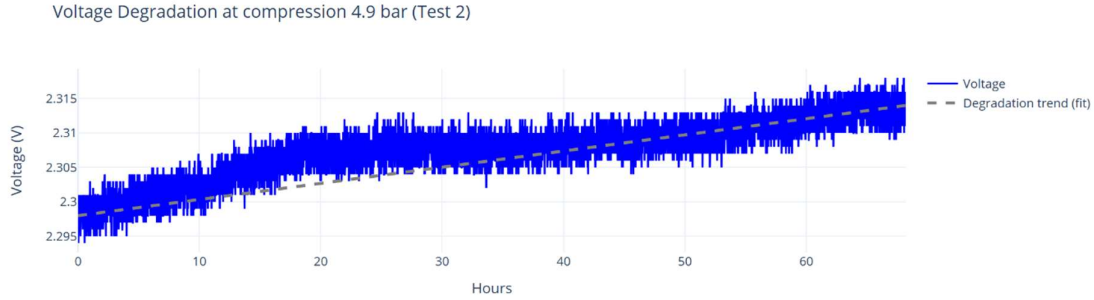


Figure 109 Degradation behavior at Compression 4.9 bar during the time (Test 2).

5.4.5 Effect of the Degradation at Compression 4.4 bar Descent (Test 2)

At descent compression of 4.4 bar, the degradation was performed from 9 February 2026 at 19:08:10 until 11 February 2026 at 5:14:10 a duration of 34.1 h of stable operation. The voltage rise from 2.303 V to 2.319 V over this interval corresponds to a degradation rate of 0.0004692 V/h and 469 μ V/h. The duration of this compression level is less than the others because the system shut down after the duration of 34.1 hours and stoped working due to the leakage of the test station.

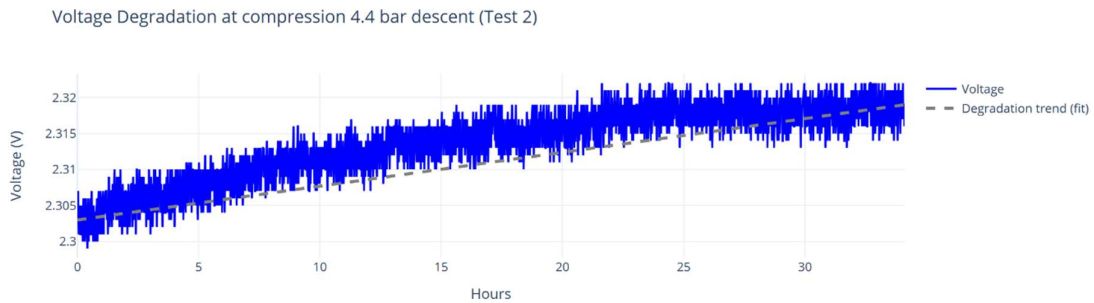


Figure 110 Degradation behavior at Compression 4.4 bar descent during the time (Test 2).

5.5 EIS Characterization Before and After Compression Variation

The second test performed at different compression levels, each three days and around 72 hours including the time measured for EIS tests before and after each compression levels (at evening) of the degradation test. Two types of EIS tests have been used:

- Potentiostatic test, performed at 0 DC V by varying the frequency from 100 kHz to 10 mHz in single sine mode and using a sinusoidal excitation signal of 10mV root mean square (rms).
- Galvanostatic test, performed at 0 DC mA by varying the frequency from 200 kHz to 0,002 Hz in single sine mode and using a sinusoidal excitation signal of 1000 mA root mean square (rms).

5.5.1 Impedance Response Under Potentiostatic Operation (1.5 V)

5.5.1.1 Data quality assessment

The KK validation shows that impedance spectra obtained under voltage control are characterized by very low residuals. In Nyquist plots it is observed that the shape is made of two semicircles, one in the HF and a second semicircle in the mid frequency region. In this case, the equivalent circuit transitions LR(QR)(QR) used to fit experimental data.

5.5.1.2 Modelling

Figure 111, show impedance spectra obtained 60°C at different Compression level with potentiostatic tests. At 60 °C and a potentiostatic operating voltage of 1.5 V, the Nyquist plot compares the impedance response of the cell before and after applying different compression levels. For each pressure step, the high-frequency intercept on the real axis represents the ohmic resistance, while the diameter of the main arc is related to the polarization (charge-transfer and mass-transport) contributions. EIS measurements were performed after each compression step at 2.4, 3.4, 4.4 and 4.9 bar. The high-frequency region, represented by the first semicircle, allows the extraction of the ohmic resistance (HF intercept with the real axis) and the charge-transfer resistance (diameter of the HF arc). The mass-transport resistance was obtained from the diameter of the low-frequency arc.

A clear evolution of the spectra is observed when moving from the EIS before compression to the EIS after compression curves at each compression level. In general, the compressed states tend to show a reduced real-axis intercept and a smaller arc diameter, indicating a decrease in both ohmic and polarization resistances.

Impedance spectra are characterized by an arc at high frequency and a second arc at mid-low frequency. The low frequency features show a dependence on compression value, at compression 4.4 bar, this tends to reduce its diameter. The LF arc shows a dependence on the compression, and it tends to reduce as the compression is increased.

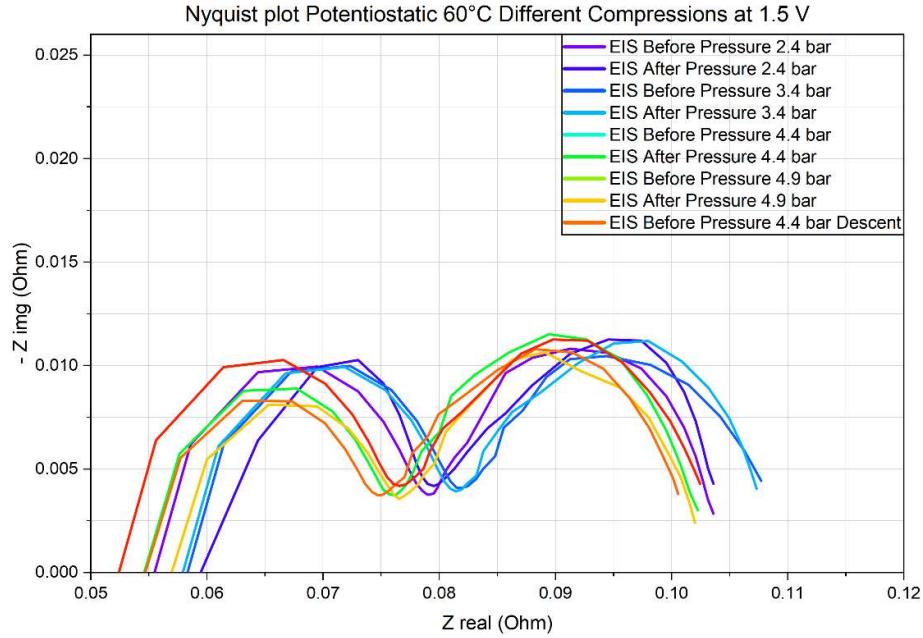


Figure 111 Nyquist plot Potentiostatic 60°C Different Pressures at 1.5 V (Test 2).

5.5.1.3 Discussion

The ohmic resistance R_s decreased over the period from approximately 1.45 to $1.36 \Omega \cdot \text{cm}^2$ from the compression 2.4 bar to 4.9 bar respectively. On the other hand, The high-frequency charge-transfer resistance R_{ct} shows a moderate reduction from about 0.55 to $0.45 \Omega \cdot \text{cm}^2$. Also, the low-frequency mass-transport resistance R_m remains nearly constant with a slight increase from roughly 0.72 to $0.75 \Omega \cdot \text{cm}^2$. The ohmic and charge transfer resistance decreasing by increasing the compression.

The capacitance of the high frequency region decreased from 0.0104 to 0.0095 F, indicating a compression reduction of the capacitance. The low-frequency capacitance shows an overall increasing trend over the period, rising from about 7.02 F to approximately 8.19 F. This behavior suggests an enhancement of the capacitive contribution associated with slow mass-transport processes after the different compression steps .

The total polarization resistance R_p shows a clear decreasing trend, dropping from roughly $2.73 \Omega \cdot \text{cm}^2$ at the beginning of the measurements to about $2.58 \Omega \cdot \text{cm}^2$. This reduction in total resistance reflects an overall improvement in the electrochemical kinetics under potentiostatic operation at 1.5 V.

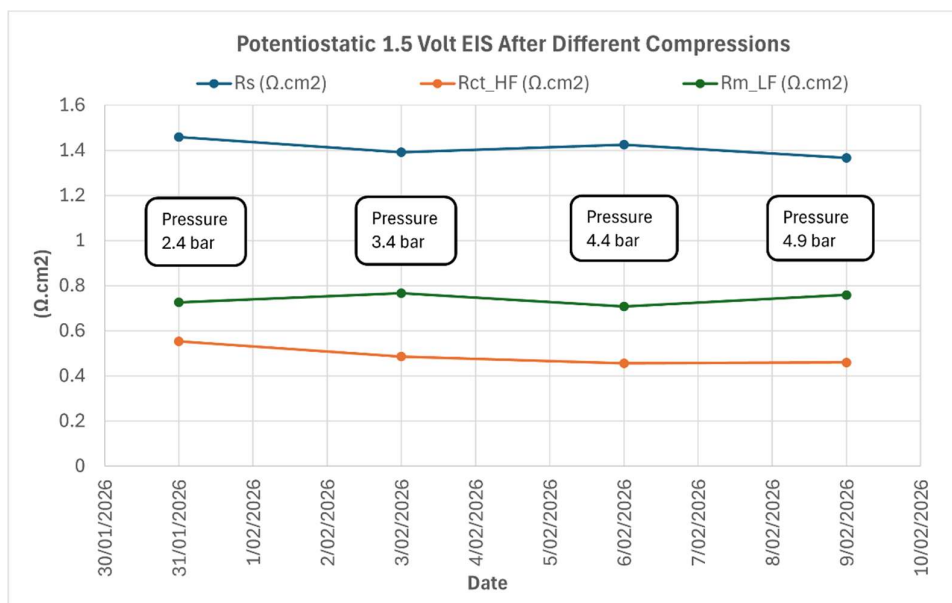


Figure 112 Trend of the ohmic resistance, the charge transfer resistance & mass transport resistance at 60°C & 1.5 Volt (Test 2).

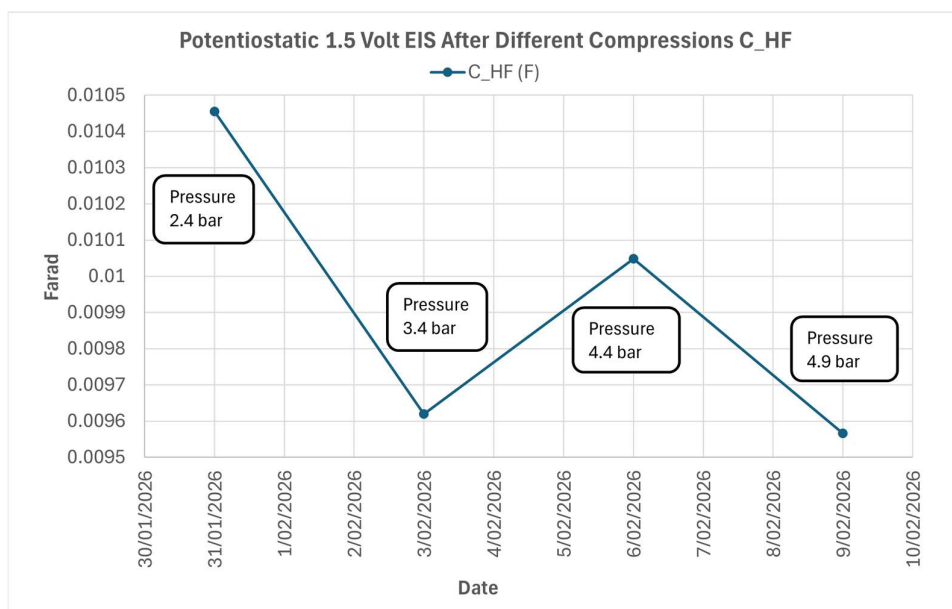


Figure 113 Trend of the capacitance in the HF region at 60°C & 1.5 Volt (Test 2).

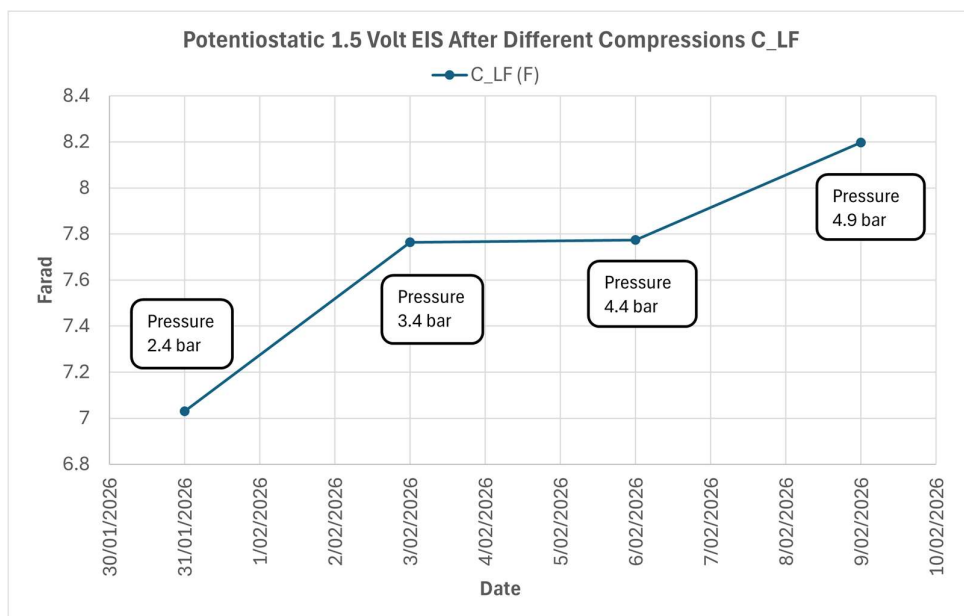


Figure 114 Trend of the capacitance in the LF region at 60°C & 1.5 Volt (Test 2).

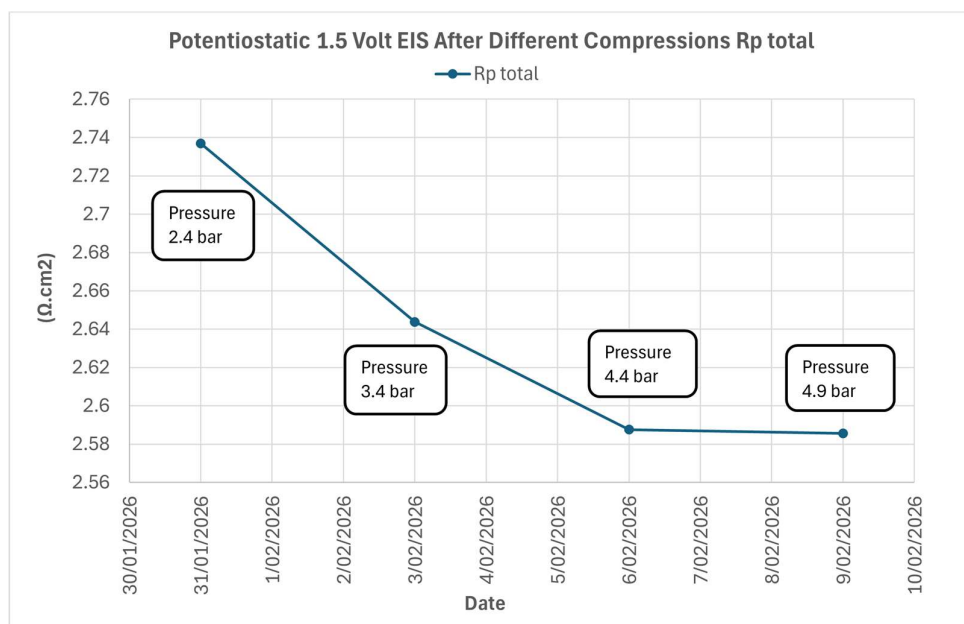


Figure 115 Trend of the total resistance at 60°C & 1.5 Volt (Test 2).

5.5.1.4 Discussion

The ohmic resistance R_s decreased throughout the entire period from $1.38 \Omega \cdot \text{cm}^2$ to $1.31 \Omega \cdot \text{cm}^2$, at compression 2.4 bar to compression 4.9 bar respectively. The ohmic resistance decreased by increasing the compression from 2.4 bar to 4.9 bar.

The high-frequency charge-transfer resistance R_{ct} is increased slightly from $0.53 \Omega \cdot \text{cm}^2$ to $0.54 \Omega \cdot \text{cm}^2$ and has a fluctuation behaviour at compression 2.4 bar to 4.9 bar respectively. The low-frequency mass-transport resistance R_m illustrates a slight increase from $0.72 \Omega \cdot \text{cm}^2$ to $0.78 \Omega \cdot \text{cm}^2$ over time.

The high-frequency capacitance shows moderate fluctuations over the period, with a slight overall increase from 0.0092 F to 0.0093 F. The low-frequency capacitance C exhibits a clear fluctuation trend from the beginning to the end from the value of 8.17 F to 8.13 F, from the compression at 2.4 bar to 4.9 bar respectively.

The total polarization resistance R_p varies with each compression step but shows an overall decreasing tendency from $2.65 \Omega \cdot \text{cm}^2$ to $2.64 \Omega \cdot \text{cm}^2$. This reduction indicates improved charge-transfer and mass-transport performance as compression increases, while short-term fluctuations reflect the transitions between “before” and “after” states at each pressure level.

The pairs of points showing a sudden change between two successive measurements correspond to the before and after states of each compression step. The vertical gap between these points reflects the immediate impact of the applied compression on the cell, after increasing the pressure, ohmic resistance and charge transfer resistance slightly decrease due to improved contact and charge-transfer conditions.

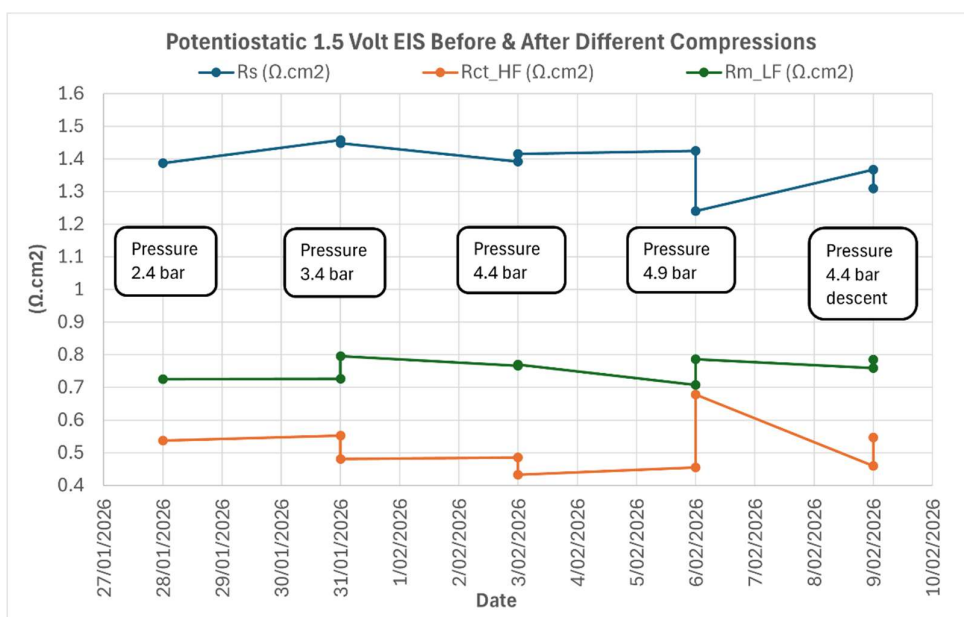


Figure 116 Trend of the ohmic resistance, the charge transfer resistance & mass transport resistance EIS before & after different compressions at 60°C & 1.5 Volt (Test 2).

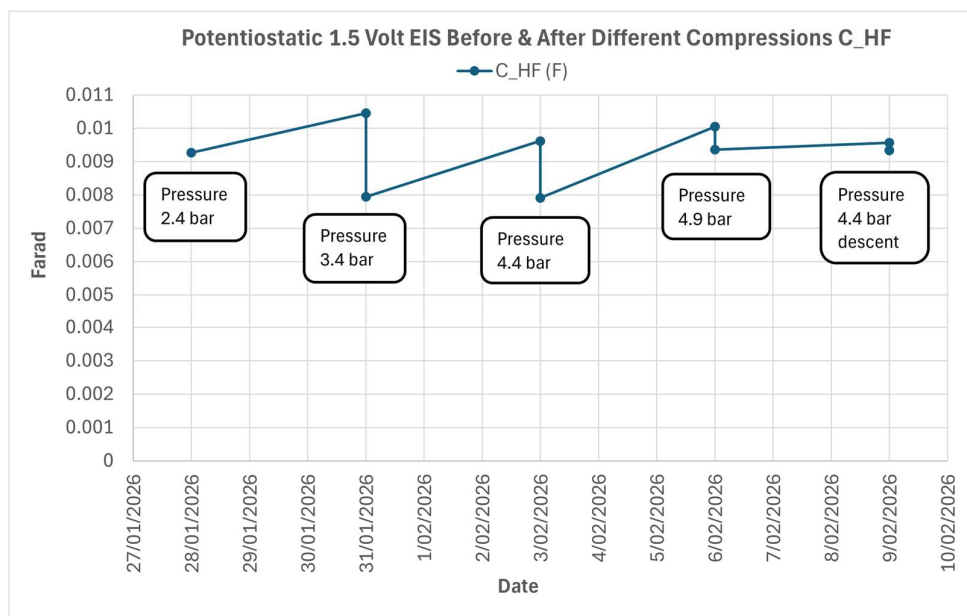


Figure 117 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.5 Volt (Test 2).

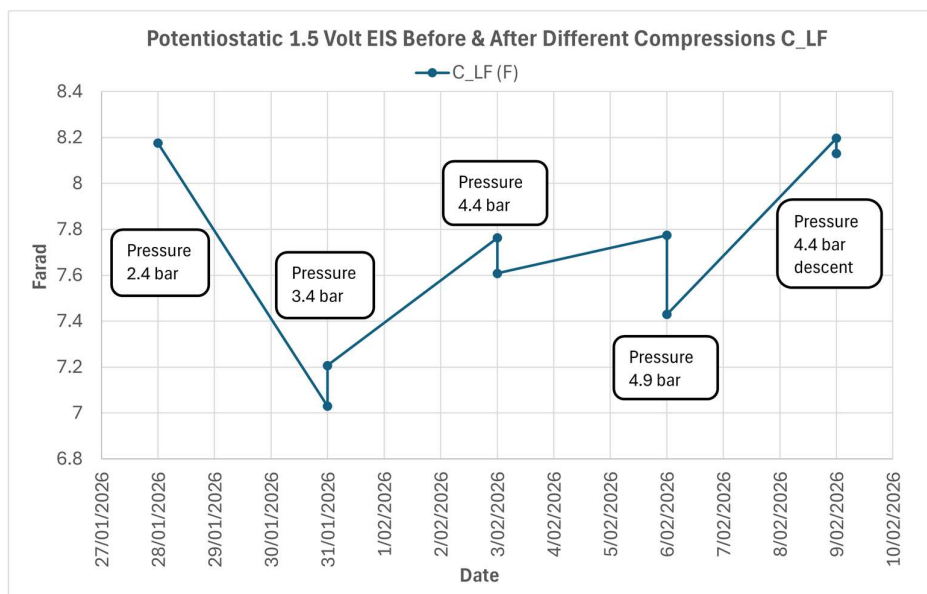


Figure 118 Trend of the capacitance in the LF region EIS before & after different compressions at 60°C & 1.5 Volt (Test 2).

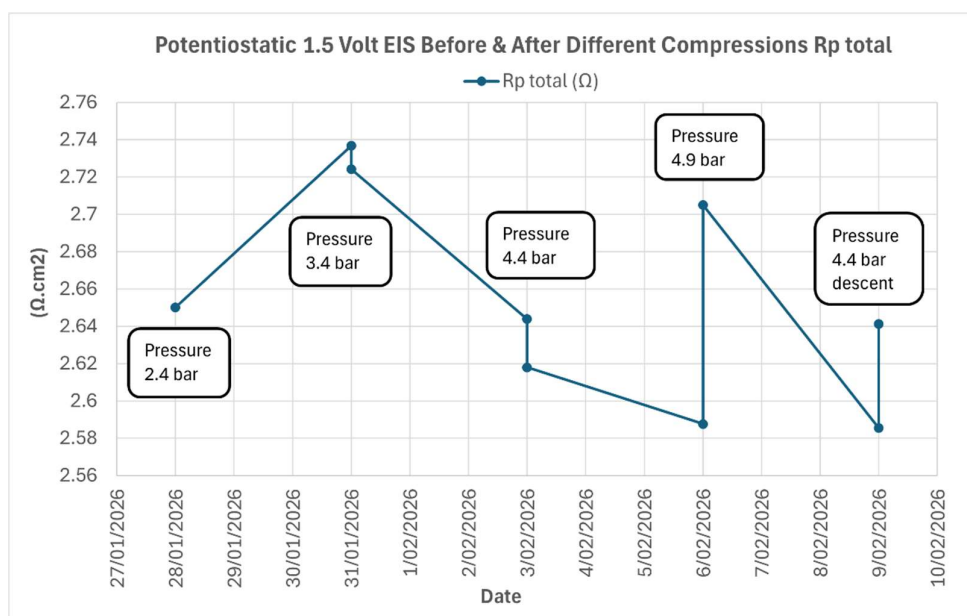


Figure 119 Trend of the total resistance before & after different compressions at 60°C & 1.5 Volt (Test 2).

5.5.2 Impedance Response Under Potentiostatic Operation (1.3 V)

5.5.2.1 Data quality assessment

The KK validation shows that potentiostatic mode have some tests with low quality impedance spectra in low frequency region, which are invalid so not used in the analysis. The Nyquist plot shows the compression effect before and after each compression level. The ECM used to fit experimental data is LR(QR)W due to the presence of noise at low frequency which makes difficult to get good results.

5.5.2.2 Modelling

The figure below shows a comparison between impedance spectra obtained with potentiostatic mode at 1.3 V with different compression levels and EIS performed before and after each compression. The shape of the impedance shows a high frequency semicircle and a tail for low frequency region. The high frequency region represented by the semicircle can be analysed because we can clearly see the ohmic resistance, intercept of the high frequency arc with the real axes, and the charge transfer resistance, represented by the intercept at mid frequency.

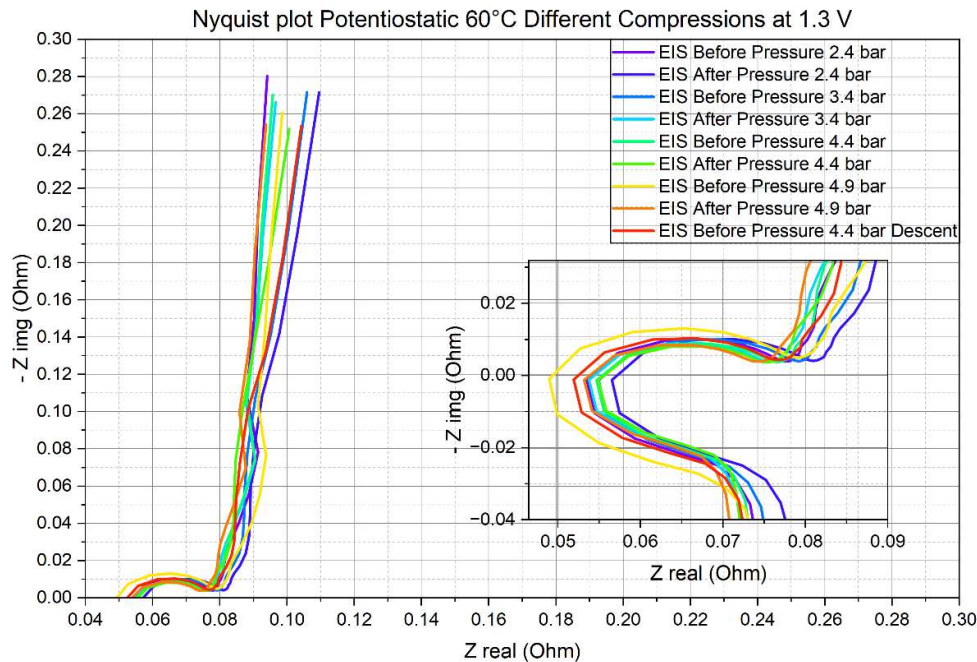


Figure 120 Nyquist plot Potentiostatic 60°C Different Pressures at 1.3 V (Test 2).

5.5.2.3 Discussion

Figure bellow shows different resistances such as ohmic resistance and charge transfer resistance and also the mass transport resistance. EIS measurements were performed after each compression step at 2.4, 3.4, 4.4 and 4.9 bar.

The ohmic resistance R_s decreased over the period from approximately 1.42 to $1.34 \Omega \cdot \text{cm}^2$. The charge-transfer resistance shows a moderate reduction from about 0.45 to $0.38 \Omega \cdot \text{cm}^2$.

The capacitance of the high frequency region decreased gradually from 0.0057 to 0.0054 F, at compression 2.4 bar to 4.4 bar respectively and finally the resistance increased to 0.0057 F at compression 4.9 bar.

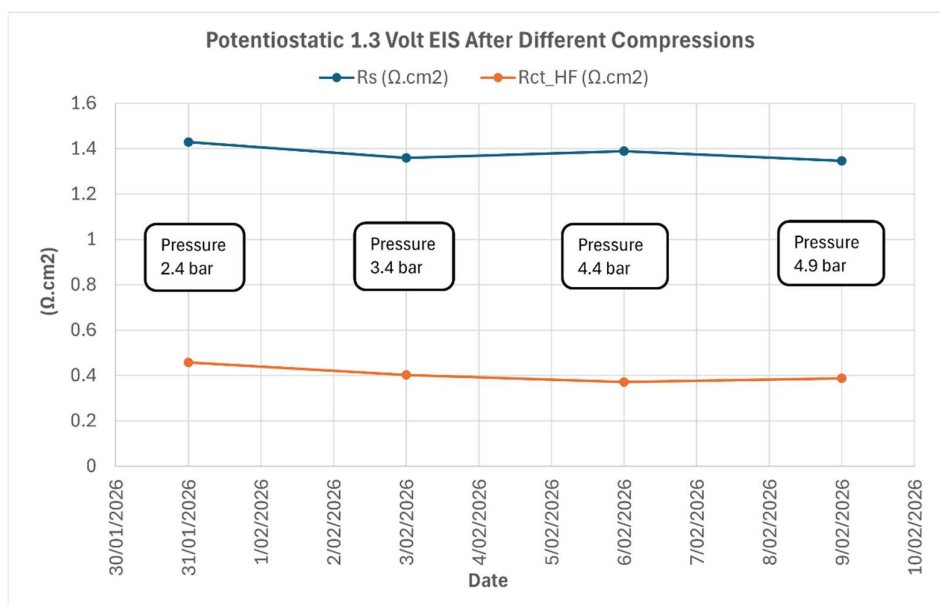


Figure 121 Trend of the ohmic resistance & the charge transfer resistance at 60°C & 1.3 Volt (Test 2).

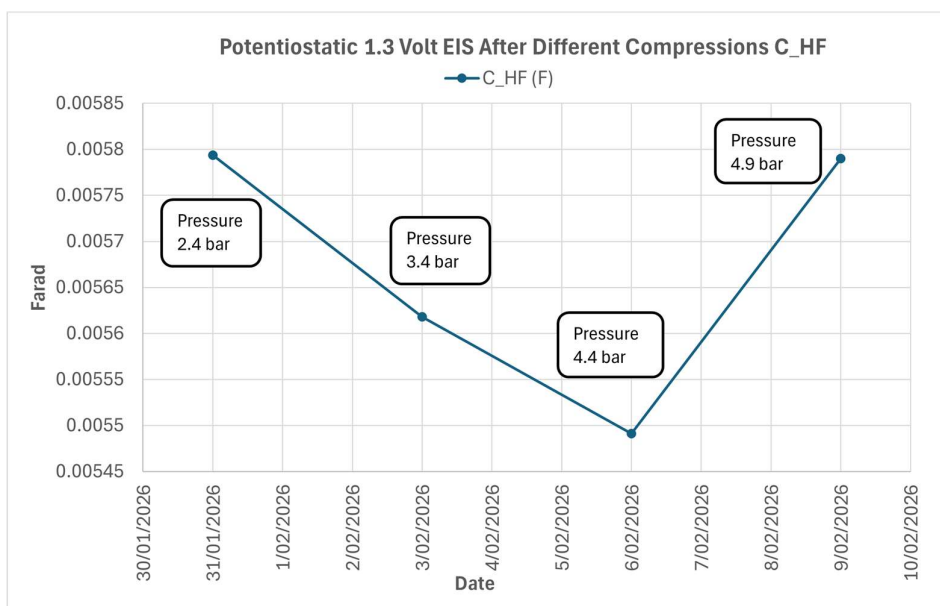


Figure 122 Trend of the capacitance in the HF region at 60°C & 1.3 Volt (Test 2).

5.5.2.4 Discussion

The figure below illustrates the ohmic resistance decreased between approximately $1.33 \Omega \cdot \text{cm}^2$ to $1.31 \Omega \cdot \text{cm}^2$. On the other hand, the high-frequency charge-transfer resistance shows a high decreasing trend in compression 2.4 bar to 4.9 bar, moving from values near $0.47 \Omega \cdot \text{cm}^2$ toward approximately $0.38 \Omega \cdot \text{cm}^2$, and at the final point the charge transfer resistance came back to

near $0.46 \Omega \cdot \text{cm}^2$ with a noticeable spike around 06/02/2026 corresponding to a compression transition. The high-frequency capacitance increased from about 0.005 F to roughly 0.006 F over the time and has fluctuation behavior.

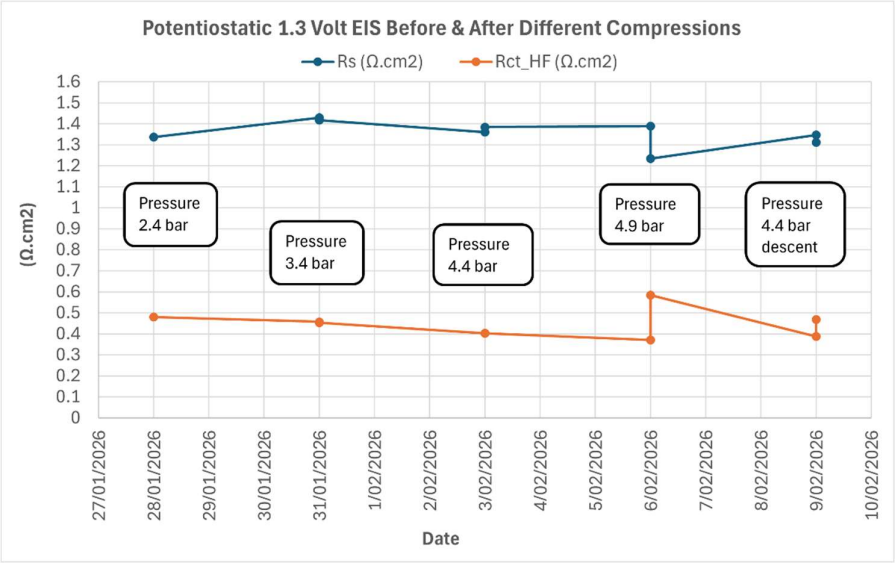


Figure 123 Trend of the ohmic resistance & the charge transfer resistance for EIS before & after different compressions at 60°C & 1.3 Volt (Test 2).

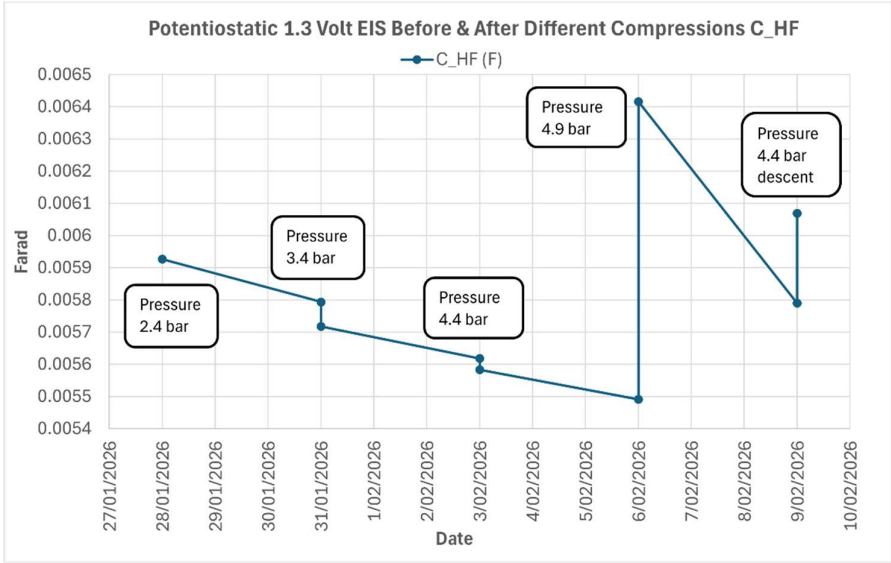


Figure 124 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.3 Volt (Test 2).

5.5.3 Impedance Response Under Galvanostatic Operation (1.5 A)

5.5.3.1 Data quality assessment

The Kramers- Kronig (KK) validation for galvanostatic operation at 1.5 A obtained, affected by time variance residuals are observed. The EIS test obtained before and after each compression level. The ECM used to model experimental data consists of three elements: LR(QR)W.

5.5.3.2 Modelling

Impedance spectra under current control are characterized by only an arc at high frequency and all the noises has been eliminated. The Nyquist plot shows the high frequency parameters at different compressions from 2.4 bar, 3.4 bar, 4.4 bar and 4.9 bar and the descent compression of 4.4 bar. The impedance spectra show a dependence on compression variations. As the compression increased this tends to reduce its diameter.

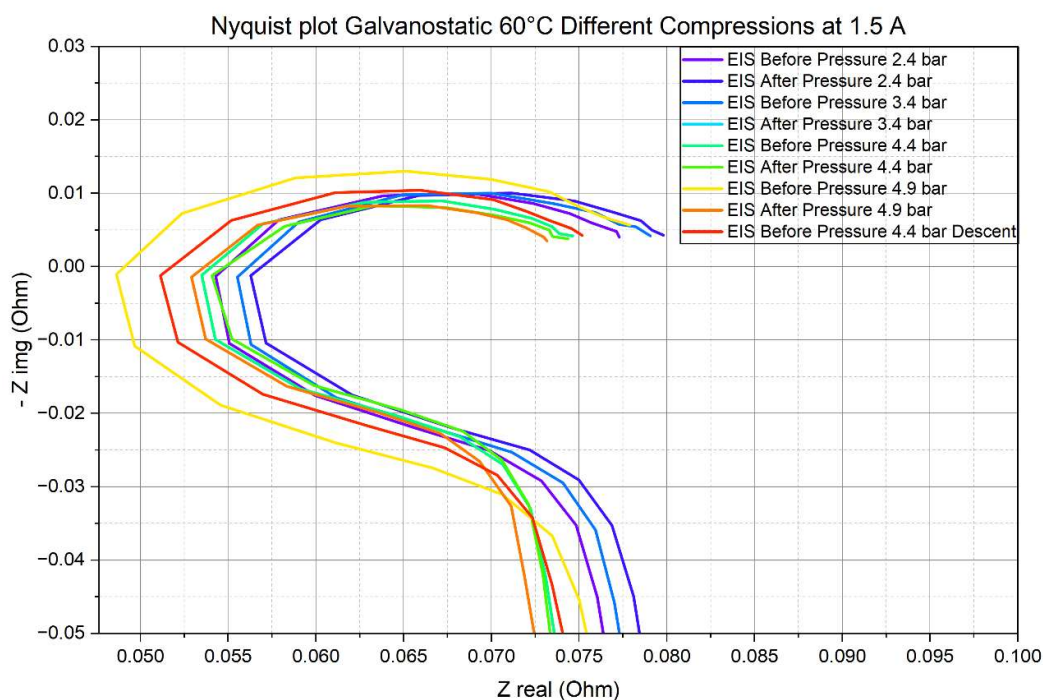


Figure 125 Nyquist plot Galvanostatic 60°C Different Pressures at 1.5 A (Test 2).

5.5.2.4 Discussion

The figure below shows the ohmic resistance decreased with increasing the compression from 1.42 and 1.34 $\Omega \cdot \text{cm}^2$. On the other hand, the high-frequency charge-transfer resistance shows a high decreasing trend in compression 2.4 bar to 4.9 bar, moving from values near 0.56 $\Omega \cdot \text{cm}^2$ toward approximately 0.48 $\Omega \cdot \text{cm}^2$. The high-frequency capacitance increases from about 0.012 F to roughly 0.013 F over the time.

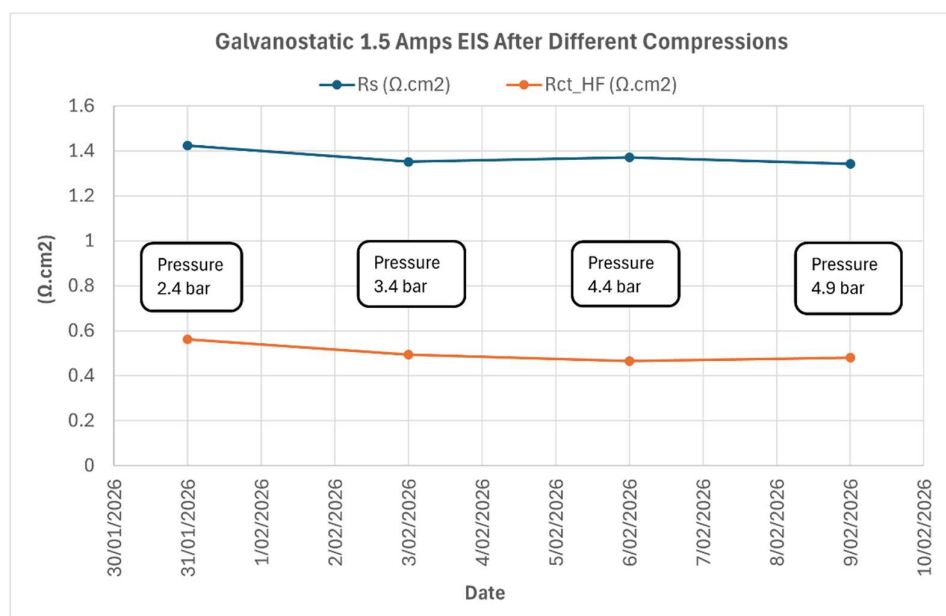


Figure 126 Trend of the ohmic resistance & the charge transfer resistance at 60°C & 1.5 A (Test 2).

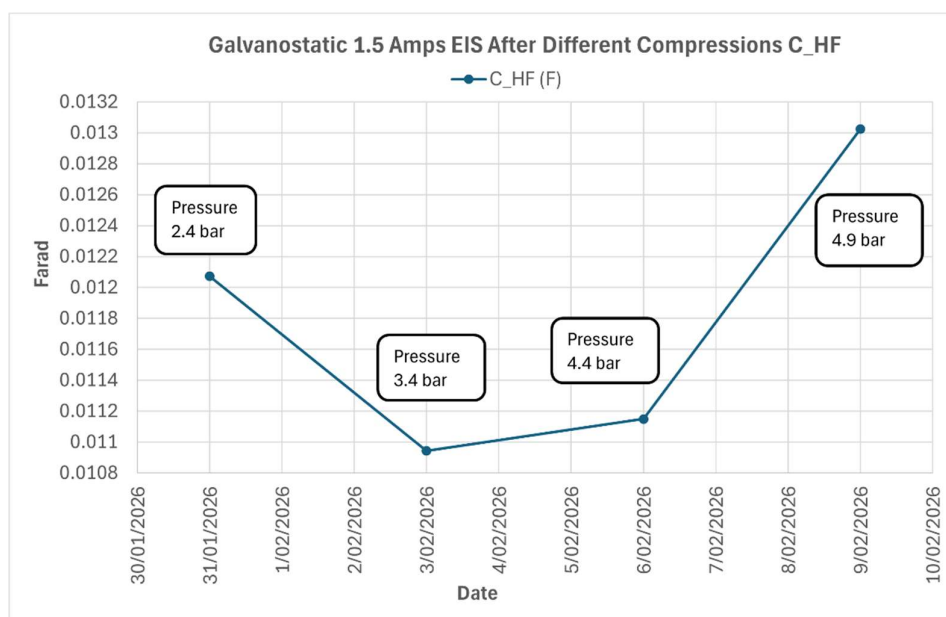


Figure 127 Figure 123 Trend of the capacitance in the HF region at 60°C & 1.5 A (Test 2).

The figure below shows the ohmic resistance decreasing from the compression 2.4 bar forward to compression 4.4 bar descent from $1.37 \Omega \cdot \text{cm}^2$ to $1.29 \Omega \cdot \text{cm}^2$. On the other hand, the high-frequency charge-transfer resistance shows an increase as the compression increased from 2.4 bar to 4.4 bar descent, moving from values near $0.53 \Omega \cdot \text{cm}^2$ toward approximately $0.58 \Omega \cdot \text{cm}^2$. The high-frequency capacitance increases from about 0.010 F to roughly 0.013 F over the time.

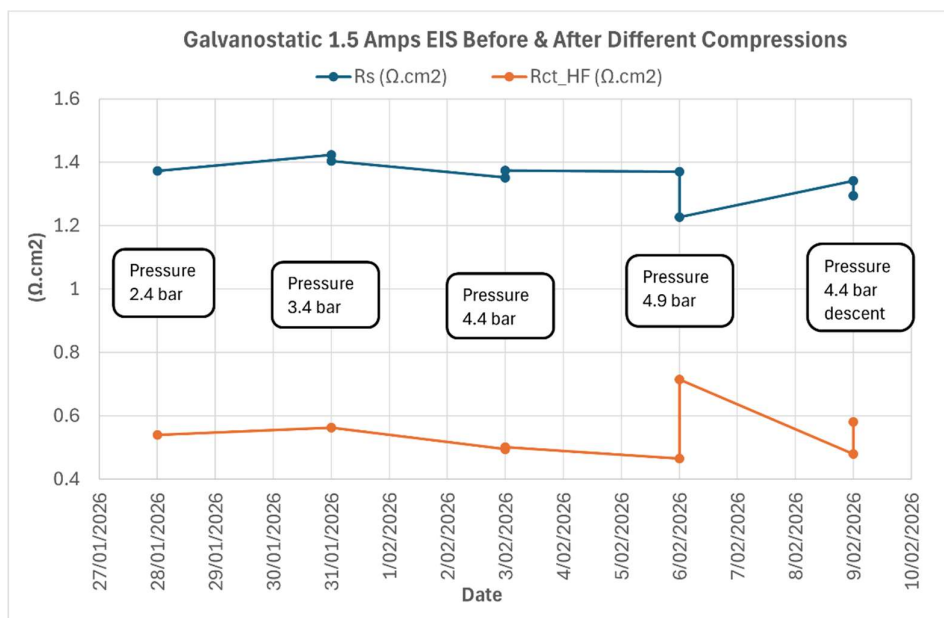


Figure 128 Trend of the ohmic resistance & the charge transfer resistance for EIS before & after different compressions at 60°C & 1.5 A (Test 2).

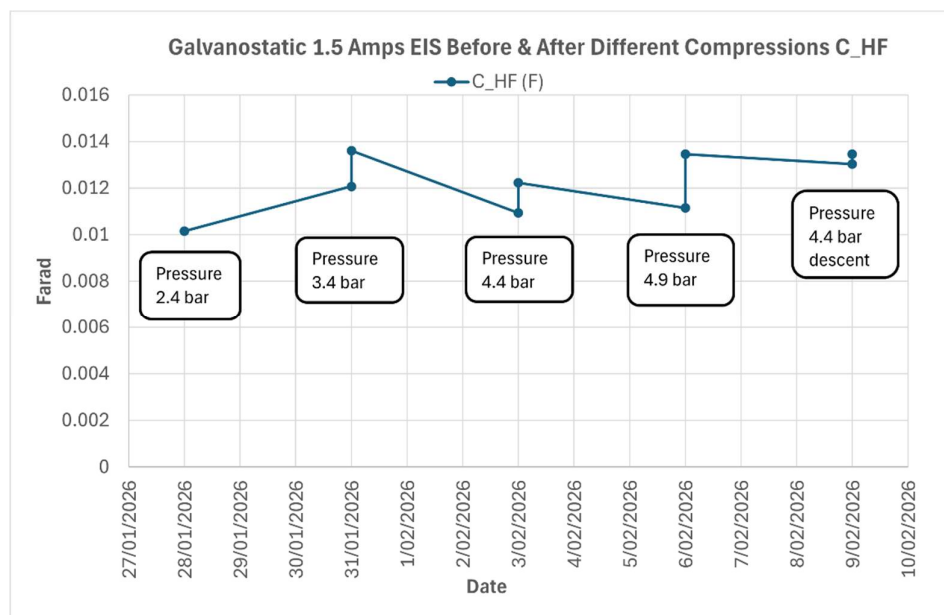


Figure 129 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.5 A (Test 2).

5.5.4 Impedance Response Under Galvanostatic Operation (1.3 A)

5.5.2.1 Data quality assessment

The KK validation shows that potentiostatic mode have some tests with low quality impedance spectra in low frequency region, which are invalid so not used in the analysis. The Nyquist plot shows the compression effect before and after each compression level. The ECM used to fit experimental data is LR(QR)W due to the presence of noise at low frequency which makes difficult to get good results.

5.5.2.2 Modelling

The figure below shows a comparison between impedance spectra obtained with potentiostatic mode at 1.3 V with different compression levels and EIS performed before and after each compression. The high frequency region represented by the semicircle can be analysed because we can clearly see the ohmic resistance, intercept of the high frequency arc with the real axes. The high frequency features show a dependence on compression levels and shows that as the compression increased it tends to reduce its diameter.

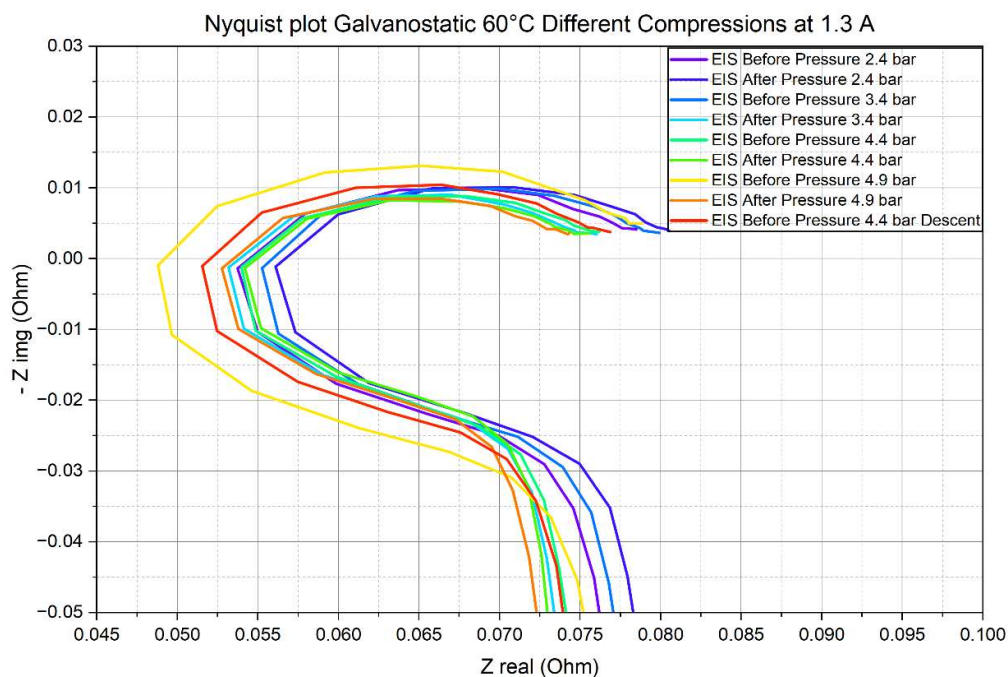


Figure 130 Nyquist plot Galvanostatic 60°C Different Pressures at 1.3 A (Test 2).

5.5.2.3 Discussion

Figure below shows the ohmic resistance R_s decreasing over the time from approximately 1.41 to 1.33 $\Omega \cdot \text{cm}^2$. The charge-transfer resistance shows a moderate reduction from about 0.56 to 0.47 $\Omega \cdot \text{cm}^2$. The capacitance of the high frequency region decreased from 0.012 to 0.010 F, at compression 2.4 bar to 4.9 bar respectively.

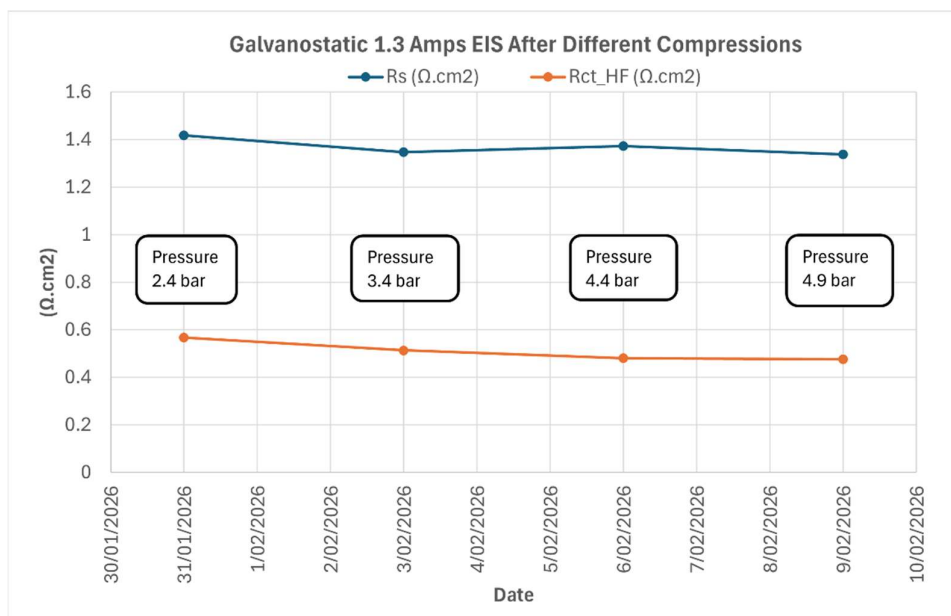


Figure 131 Trend of the ohmic resistance & the charge transfer resistance at 60°C & 1.3 A (Test 2).

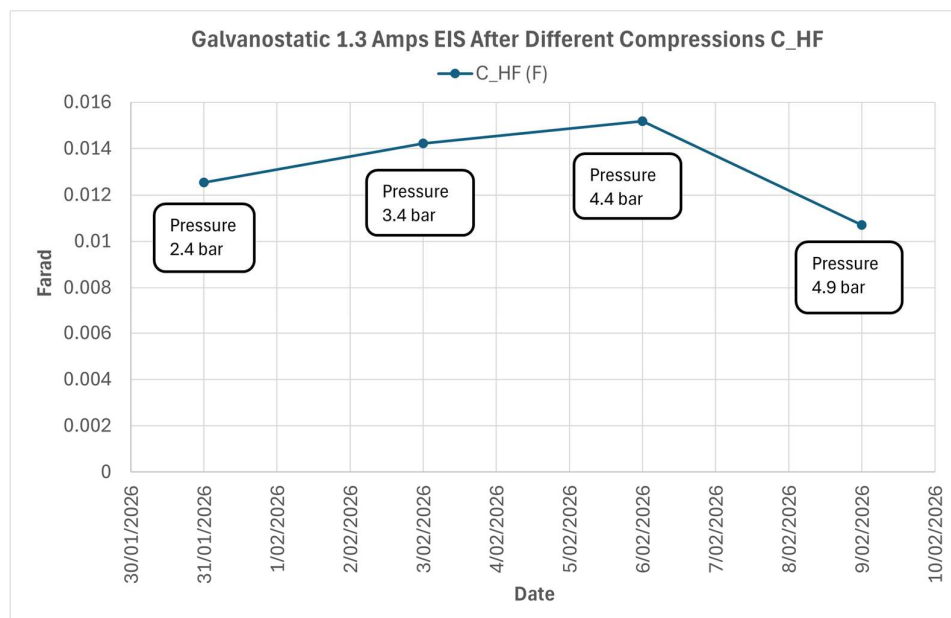


Figure 132 Figure 123 Trend of the capacitance in the HF region at 60°C & 1.3 A (Test 2).

5.5.2.4 Discussion

The figure below shows the ohmic resistance decreased from $1.37 \Omega \cdot \text{cm}^2$ and $1.30 \Omega \cdot \text{cm}^2$. On the other hand, the high-frequency charge-transfer resistance shows a slight increase, moving from values near $0.55 \Omega \cdot \text{cm}^2$ toward approximately $0.57 \Omega \cdot \text{cm}^2$. The high-frequency capacitance increases steadily from about 0.011 F to roughly 0.012 F over the time.

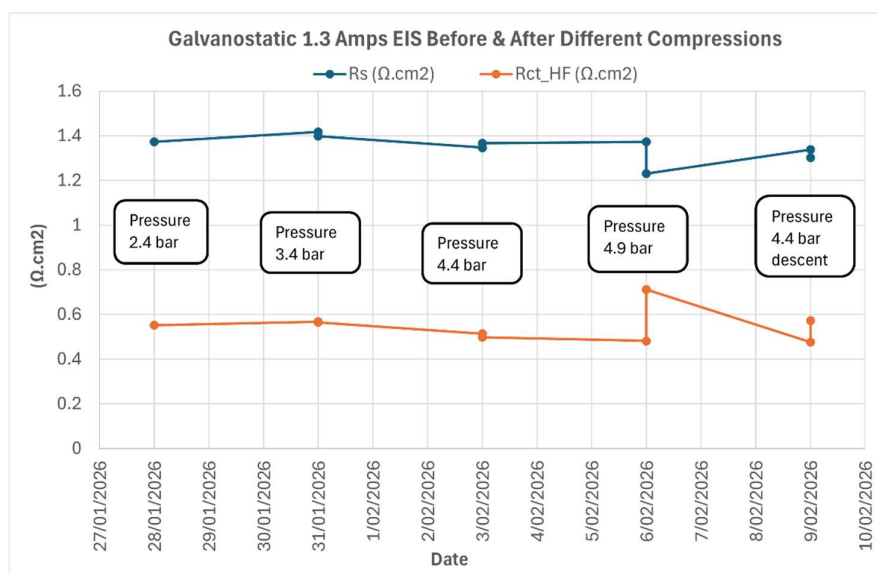


Figure 133 Trend of the ohmic resistance & the charge transfer resistance for EIS before & after different compressions at 60°C & 1.3 A (Test 2).

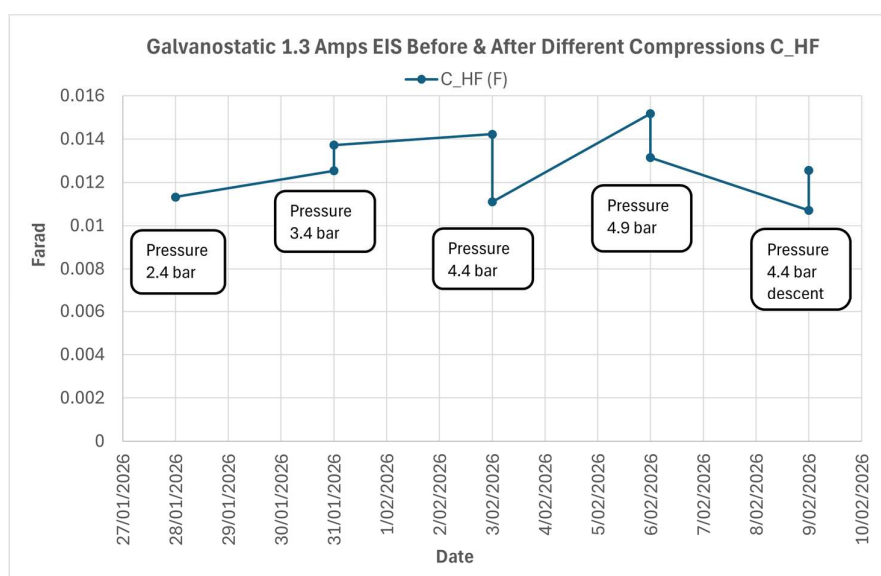


Figure 134 Trend of the capacitance in the HF region EIS before & after different compressions at 60°C & 1.3 A (Test 2).

5.6. Analysis of Voltage Evolution via Linear Extrapolation (Test 2)

To evaluate the long-term durability of the cell under the selected operating conditions, the evolution of the cell voltage was analyzed through a linear extrapolation approach. The experimental voltage values were collected during the initial phase of the durability test and subsequently fitted using a linear regression model of the form:

$$V(t) = a t + b$$

This formulation assumes that the degradation rate remains constant during the time, allowing the short-term trend to be projected toward longer operating periods. The fitting procedure was applied to the range of the measured data, ensuring that the regression coefficients show the real behavior of the system during the initial stages of operation.

The resulting linear fit illustrated a high degree of consistency with the experimental points, confirming that the voltage increase follows an approximately linear trend within the monitored interval. Based on the obtained regression parameters, the voltage evolution was extrapolated up to 1000 h of continuous operation. This represents the reference duration adopted in standardized durability protocols for electrochemical devices.

The extrapolated curve provides an estimate of the expected voltage at 1000 h ($V_{1000,fit}$), enabling the quantification of the long-term degradation rate. The increase in voltage with respect to the beginning-of-interval value (V_0) was calculated according to:

$$\Delta V_{1000} = \frac{V_{1000,fit} - V_0}{V_0} \times 100$$

This metric allows for a direct comparison of degradation severity across different operating conditions or test configurations.

The graphical representation of the measured data, the fitted linear trend, and the extrapolated region clearly highlights the progressive voltage rise associated with performance degradation. The limited dispersion of the experimental points around the fitted line indicates stable operation during the monitored period, while the extrapolated segment provides a first-order estimation of the long-term behavior. Although linear extrapolation does not capture potential nonlinear degradation mechanisms that may emerge at extended operating times, it offers a practical and widely adopted method for preliminary durability assessment when long-duration tests are not fully available.

5.1.4.1 Linear Extrapolation at 2.4 bar (Test 2)

After cleaning the dataset and removing the initial unstable region, the steady state voltage values corresponding to the operation at compression 2.4 bar were analyzed. Based on the processed data, the degradation rate was determined to be 0.000922 V/h, and the beginning-of-interval voltage was 2.354 V. Using these parameters, the voltage trend was extended to 1000 h, resulting in a predicted voltage of $V_{1000} = 3.276$ V. The relative increase with respect to the initial voltage corresponds to 39 %/1000 h. Overall, the extrapolated trend indicates a gradual and consistent voltage rise under this pressure level.

$$\text{Linear Fit Equation: } V = 0.000922 * t + 2.354$$

Voltage Degradation with Linear Extrapolation to 1000 Hours at compression 2.4 bar (Test 2)

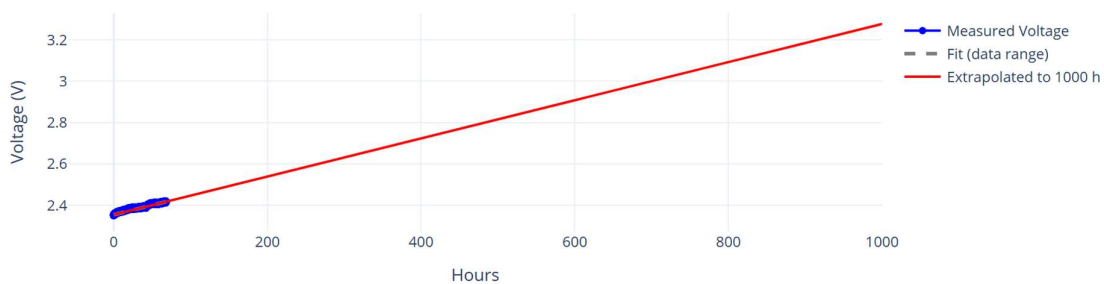


Figure 135 Linear extrapolation to 1000h at compression 2.4 bar (Test 2).

5.1.4.1 Linear Extrapolation at 3.4 bar (Test 2)

The voltage values corresponding to the operation at compression 3.4 bar were analyzed and the degradation rate was calculated to be 0.000349 V/h, and the initial voltage was 2.308 V. The voltage trend was extended to 1000 h, resulting in a predicted voltage of $V_{1000} = 2.657$ V. The increase value of voltage with respect to the initial one corresponds to 15 %/1000 h.

$$\text{Linear Fit Equation: } V = 0.000349 * t + 2.308$$

Voltage Degradation with Linear Extrapolation to 1000 Hours at compression 3.4 bar (Test 2)

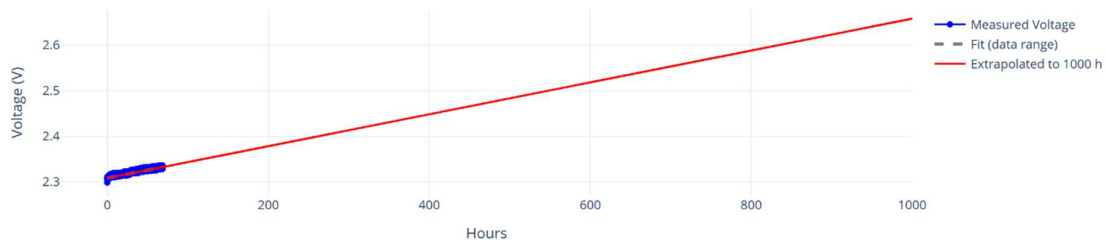


Figure 136 Linear extrapolation to 1000h at compression 3.4 bar (Test 2).

5.1.4.1 Linear Extrapolation at 4.4 bar (Test 2)

The stable voltage values corresponding to the operation at compression 4.4 bar were analyzed. Based on the data, the degradation rate was obtained to be 0.000336 V/h, and the beginning-of-interval voltage was 2.301 V. Using these parameters, the voltage trend was extended to 1000 h, resulting in a predicted voltage of $V_{1000} = 2.614$ V. The relative increase with respect to the initial voltage corresponds to 13 %/1000 h. Overall, the extrapolated trend indicates a gradual and consistent voltage rise under this pressure level.

$$\text{Linear Fit Equation: } V = 0.000336 * t + 2.301$$



Figure 137 Linear extrapolation to 1000h at compression 4.4 bar (Test 2).

5.1.4.1 Linear Extrapolation at 4.9 bar (Test 2)

The voltage values corresponding to the operation at compression 4.9 bar based on the processed data were analyzed, the degradation rate was determined to be 0.000234 V/h, and the beginning-of-interval voltage was 2.298 V. Using these parameters, the voltage trend was extended to 1000 h, resulting in a predicted voltage of $V_{1000} = 2.532$ V. The relative increase with respect to the initial voltage corresponds to 10 %/1000 h. Overall, the extrapolated trend indicates a gradual and consistent voltage rise under this pressure level.

$$\text{Linear Fit Equation: } V = 0.000234 * t + 2.298$$



Figure 138 Linear extrapolation to 1000h at compression 4.9 bar (Test 2).

5.1.4.1 Linear Extrapolation at 4.4 bar descent (Test 2)

The steady state voltage values corresponding to the operation at compression 4.4 bar descent were analyzed. The degradation rate was determined to be 0.000469 V/h, and the beginning-of-interval voltage was 2.303 V. Using these parameters, the voltage trend was extended to 1000 h, resulting in a predicted voltage of $V_{1000} = 2.772$ V. The relative increase with respect to the initial voltage corresponds to 20 %/1000 h. Overall, the extrapolated trend indicates a gradual and consistent voltage rise under this pressure level.

$$\text{Linear Fit Equation: } V = 0.000469 * t + 2.303$$

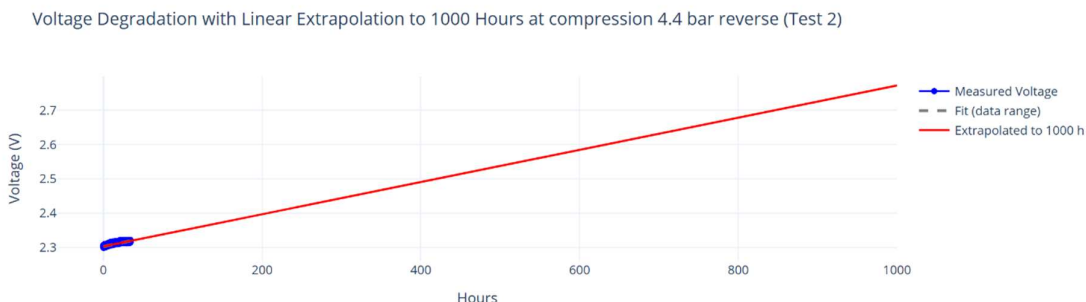


Figure 139 Linear extrapolation to 1000h at compression 4.4 bar descent (Test 2).

The effect of different compression levels on both the performance and durability of the cell was evaluated. At compression 2.4 bar, the cell exhibited the highest ohmic resistance and the lowest performance. The compression 4.4 bar reduced mass transport resistances, also in polarization curve resulting in the lowest cell voltage at 1 A/cm² and thus the best performance. However, further increasing the compression to 4.9 bar it provides a significant improvement in degradation performance.

Chapter 6: Conclusions and Future Work

6.1 Conclusions

6.1.1 Conclusion of the Test One

The first experimental tests focused on identifying the optimal operating pressure for stable and efficient PEM electrolyzer cell performance. Across all evaluated parameters, including polarization behavior, degradation, ohmic resistance, and impedance characteristics, the contact pressure at 3.4 bar descent consistently provided the most balanced and favourable operating point. At this pressure, the cell exhibited lower overall voltage, decreased on the order of a few tens of millivolts compared to higher pressure conditions, and reduced ohmic resistance, showing a value of roughly 0.051Ω . The degradation rate in this level of compression was calculated as $160 \mu V/h$. No significant increase in mass transport limitations, which typically emerge at higher pressures. Therefore, 3.4 bar is identified as the optimal pressure for Test 1, offering a stable compromise between cell performance, electrochemical performance, and manageable transport phenomena.

6.1.2 Conclusion of the Test Two

For the second test, that has been done with a different set of CCM and GDLs the degradation rate, ohmic resistance and charge transfer resistance decreased with increasing the compression. The mass transport resistance increased slightly with some fluctuations, but the lowest value is at the contact pressure of 4.4 bar. In fact, the second test illustrates multi objective optimum between 4.4 and 4.9 bar investigated long term degradation, detailed EIS behavior, and high-pressure performance. Unlike Test 1, no single pressure emerged as an optimal across all metrics. Instead, the results revealed a multi objective trade-off such as minimum degradation over time at contact pressure of 4.9 bar that was calculated as $234 \mu V/h$ and also the lowest ohmic resistance with the value of 0.049Ω . Best polarization curve performance which is the lowest cell voltage at a given current density was observed at contact pressure of 4.4 bar, making it the most efficient pressure for instantaneous performance. In resistance behavior, no single pressure minimized all resistance components. Instead, the results indicate that for durability and long-term stability compression 4.9 bar is preferred. Thus, the optimal operating region for test 2 can be defined as a contact pressure between 4.4 and 4.9 bar.

The effect of compression levels on the cell performance was primarily evaluated using the second test under potentiostatic & galvanostatic control. At the reference current density of $1 A/cm^2$, increasing compression levels exhibited the lowest cell voltage and the lowest ohmic and charge transfer resistances, indicating the best electrochemical performance. In fact, increasing the compression increased the performance by decreasing the degradation rate and the ohmic and charge transfer resistance as the contact pressure of 4.9 bar exhibited the lowest

value while the mass transport resistance was optimum at compression 4.4 bar. Also, polarization curve showed an optimum behavior at compression 4.4 bar.

Therefore, the contact pressure level between 4.4 bar and 4.9 bar can be considered as an optimum compromise between performance and long-term durability.

6.2 General Discussion (both tests)

This thesis illustrated a comprehensive experimental investigation into the effect of compression on the performance and electrochemical behaviour of various Proton Exchange Membrane (PEM) electrolyser cell configurations. By changing the applied compression and analysing its impact on key performance indicators (KPIs) and Electrochemical Impedance Spectroscopy (EIS) responses. The study provided valuable insights into the electrochemical interactions controlling PEM electrolyser operation.

The experimental results showed that compression plays a critical role in determining cell performance, the quality of the contact resistances, and mass-transport behaviour. High compression was shown to significantly reduce contact resistance, leading to lower high-frequency resistance (HFR) and increased voltage performance across a wide range of current densities. These improvements were observed across different membrane–electrode assemblies (MEAs) and gas diffusion layers (GDLs).

EIS analysis showed that optimal compression reduces both ohmic and charge-transfer resistances. Also, EIS analysis contributing to improved overall cell performance. However, high compression resulted in important effects, including GDL deformation, restricted water transport, and increased mass-transport limitations. These phenomena showed in elevated low-frequency impedance and performance degradation at high current densities.

Overall, the findings illustrate the importance of cell compression conditions in PEM electrolyser systems. The results provide a deeper understanding of the interaction between effects of compression on the cell performance and electrochemical processes. It is enhancing performance and supporting the development of next-generation PEM electrolyzers. This work is aligned with Borgardt et al. (2019) and Frensch et al. (2018) [31], [37].

6.3 Future Work

Although this study provides a detailed assessment of compression effects on PEM electrolyser performance, several opportunities remain for further research:

1. Long-term durability studies

Future work should research how optimal and high compression levels influence long-term degradation mechanisms, including catalyst separation, membrane thinning, and GDL structural. Accelerated stress tests (ASTs) combined with periodic EIS measurements could provide deeper findings into ageing behaviour.

2. Water distribution and imaging

Advanced diagnostic tools such as neutron imaging, X-ray tomography, or optical visualisation could be used to study water transport and GDL deformation under compression. These techniques would help validate the mass-transport limitations observed in this study.

3. Modelling development

Developing multi-physics models that couple mechanical compression, electrochemical kinetics, and two-phase flow would enable predictive optimisation of compression levels for different cell characterization.

4. Investigation of alternative GDL and MEA materials

Future studies could explore how novel GDL materials (e.g., titanium felts with tailored porosity, hybrid structures, or coated meshes) respond to compression. Similarly, MEAs with different membrane thicknesses may show different compression performance relationships.

5. Integration with dynamic operating conditions

Since PEM electrolyzers are increasingly used in renewable-powered systems, future work should examine how compression interacts with dynamic load cycles, start-up/shut-down sequences, and fluctuating water supply conditions.

Appendix: List of Tables

Table 1 Results of the ohmic resistance different compressions potentiostatic at 60°C & 0 volt (Test1)

Date	Compression (bar)	Voltage (V)	Rs (Ω)	Rs (Ω .cm2)
2/10/2025	3.4	0	0.064285	1.607125
6/10/2025	4.4	0	0.10437	2.60925
8/10/2025	5	0	0.0686	1.715
9/10/2025	4.4 descent	0	0.063616	1.5904
13/10/2025	3.4 descent	0	0.05314	1.3285
14/10/2025	2.4 descent	0	0.0538	1.345

Table 2 Results of the ohmic resistance different compressions potentiostatic at 60°C & 0.5 volt (Test1)

Date	Compression (bar)	Voltage (V)	Rs (Ω)	Rs (Ω .cm2)
2/10/2025	3.4	0.5	0.06386	1.5965
6/10/2025	4.4	0.5	0.10732	2.683
8/10/2025	5	0.5	0.071	1.775
9/10/2025	4.4 descent	0.5	0.0602174	1.505435
13/10/2025	3.4 descent	0.5	0.052356	1.3089
14/10/2025	2.4 descent	0.5	0.053	1.325

Table 3 Results of the ohmic resistance different compressions potentiostatic at 60°C & 1 volt (Test1)

Date	Compression (bar)	Voltage (V)	Rs (Ω)	Rs (Ω .cm2)
2/10/2025	3.4	1	0.0638538	1.596345
6/10/2025	4.4	1	0.10432	2.608
8/10/2025	5	1	0.0705375	1.7634375
9/10/2025	4.4 descent	1	0.059469	1.486725
13/10/2025	3.4 descent	1	0.0521156	1.30289
14/10/2025	2.4 descent	1	0.05284	1.321

Table 4 Results of the ohmic resistance different compressions potentiostatic at 60°C & 1.3 volt (Test1)

Date	Compression (bar)	Voltage (V)	Rs ($\Omega \cdot \text{cm}^2$)	Rct_HF ($\Omega \cdot \text{cm}^2$)	C_HF (F)
2/10/2025	3.4	1.3	1.58875	0.89265	0.008362
6/10/2025	4.4	1.3	2.5976	0.74695	0.0075027
8/10/2025	5	1.3	1.7483225	0.746425	0.0075276
9/10/2025	4.4 descent	1.3	1.4836575	0.82305	0.0069392
13/10/2025	3.4 descent	1.3	1.2981	1.6153	0.016987
14/10/2025	2.4 descent	1.3	1.3193375	0.801325	0.004564

Table 5 Results of the ohmic resistance different compressions potentiostatic at 60°C & 1.5 volt (Test1)

			High Frequency			Low Frequency		
Date	Compression	Voltage	Rs		Rct	C	Rm	C
Unit	(bar)	(V)	($\Omega \cdot \text{cm}^2$)	($\Omega \cdot \text{cm}^2$)	Farad	($\Omega \cdot \text{cm}^2$)	Farad	Rp total
2/10/2025	3.4	1.5	0	0	0	0	0	($\Omega \cdot \text{cm}^2$)
6/10/2025	4.4	1.5	2.617875	0.9713	0.017516	1.4716	7.6958	0
8/10/2025	5	1.5	1.734125	0.934	0.014415	1.466775	8.5568	5.060775
9/10/2025	4.4 descent	1.5	1.4819125	1.0002	0.013488	1.46395	8.1364	4.1349
13/10/2025	3.4 descent	1.5	1.29255	1.04735	0.012476	1.464525	8.1219	3.9460625
14/10/2025	2.4 descent	1.5	1.318545	1.27365	0.012015	1.542225	6.3528	3.804425

Table 6 Results of the resistances & capacitance different compressions potentiostatic at 60°C & 1.5 volt (Test2)

Date	Compression (bar)	Voltage (V)	High Frequency			Low Frequency		Rp total (Ω.cm2)
			Rs (Ω.cm2)	Rct (Ω.cm2)	C Farad	Rm (Ω.cm2)	C Farad	
28/01/2026	Before 2.4 bar	1.5	1.387	0.537725	0.009272	0.72535	8.1753	2.650075
31/01/2026	After 2.4 bar	1.5	1.45775	0.5527	0.010455	0.726425	7.0296	2.736875
31/01/2026	Before 3.4 bar	1.5	1.44775	0.480625	0.0079497	0.79585	7.207	2.724225
3/02/2026	After 3.4 bar	1.5	1.3915	0.485675	0.0096196	0.766675	7.7634	2.64385
3/02/2026	Before 4.4 bar	1.5	1.415	0.43255	0.0079103	0.770425	7.6083	2.617975
6/02/2026	After 4.4 bar	1.5	1.425	0.45535	0.010048	0.7073	7.7739	2.58765
6/02/2026	Before 4.9 bar	1.5	1.23975	0.6783	0.0093614	0.786925	7.4289	2.704975
9/02/2026	After 4.9 bar	1.5	1.367	0.459725	0.0095659	0.75885	8.1973	2.585575
9/02/2026	Before 4.4 bar descent	1.5	1.31	0.546125	0.0093422	0.7852	8.1303	2.641325

Table 7 Results of the resistances & capacitance different compressions potentiostatic at 60°C & 1.3 volt (Test2)

Date	Compression (bar)	Voltage (V)	High Frequency		
			Rs (Ω.cm2)	Rct (Ω.cm2)	C Farad
28/01/2026	Before 2.4 bar	1.3	1.33725	0.479425	0.0086142
31/01/2026	After 2.4 bar	1.3	1.429	0.457325	0.0057936
31/01/2026	Before 3.4 bar	1.3	1.41705	0.453675	0.0057176
3/02/2026	After 3.4 bar	1.3	1.35975	0.4024	0.005618
3/02/2026	Before 4.4 bar	1.3	1.3845	0.402775	0.0055826
6/02/2026	After 4.4 bar	1.3	1.389	0.37165	0.0054912
6/02/2026	Before 4.9 bar	1.3	1.234	0.585	0.0064158
9/02/2026	After 4.9 bar	1.3	1.34625	0.38725	0.0057901
9/02/2026	Before 4.4 bar descent	1.3	1.31125	0.468475	0.0060694

Table 8 Results of the resistances & capacitance different compressions galvanostatic at 60°C & 1.5 amps (Test2)

Date	Compression (bar)	Current (A)	High Frequency		
			Rs (Ω .cm ²)	Rct (Ω .cm ²)	C Farad
28/01/2026	Before 2.4 bar	1.5	1.3726625	0.5398	0.010145
31/01/2026	After 2.4 bar	1.5	1.423295	0.56265	0.012074
31/01/2026	Before 3.4 bar	1.5	1.404575	0.5634	0.013599
3/02/2026	After 3.4 bar	1.5	1.3517575	0.494825	0.010943
3/02/2026	Before 4.4 bar	1.5	1.374	0.50115	0.012235
6/02/2026	After 4.4 bar	1.5	1.37082	0.46575	0.01115
6/02/2026	Before 4.9 bar	1.5	1.2275	0.714525	0.013458
9/02/2026	After 4.9 bar	1.5	1.3417975	0.480275	0.013025
9/02/2026	Before 4.4 bar descent	1.5	1.295	0.5812	0.013458

Table 9 Results of the resistances & capacitance at different compressions galvanostatic at 60°C & 1.3 amps (Test2)

Date	Compression (bar)	Current (A)	High Frequency		
			Rs (Ω .cm ²)	Rct (Ω .cm ²)	C Farad
28/01/2026	Before 2.4 bar	1.3	1.37335	0.551875	0.011327
31/01/2026	After 2.4 bar	1.3	1.4175	0.56755	0.012543
31/01/2026	Before 3.4 bar	1.3	1.3978125	0.565075	0.013719
3/02/2026	After 3.4 bar	1.3	1.3469125	0.5135	0.014225
3/02/2026	Before 4.4 bar	1.3	1.36595	0.497375	0.011106
6/02/2026	After 4.4 bar	1.3	1.373215	0.481425	0.015176
6/02/2026	Before 4.9 bar	1.3	1.230565	0.710975	0.013153
9/02/2026	After 4.9 bar	1.3	1.337635	0.47675	0.010711
9/02/2026	Before 4.4 bar descent	1.3	1.3015	0.573125	0.012547

Table 10 Fitting results of potentiostatic at 1.3-volt 60°C -before 2.4 bar (Test2)

EIS Before Compression 2.4 bar 28/01/26 Potentiostatic at 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05349 Ohm	Fixed	0.05933
Rct	0.019177 Ohm	Positive	0.032254
CPE1	-1 ...	Positive	0
TDE	0.0059262	Positive	0.26747
PHIDE	1.2406	Positive	0.029036
WO1	-1 ...	Positive	0
R	0.035652	Positive	0.080445
T	0.27776	Positive	0.081641
L1	2.97E-06 H	Positive	0.10389

Table 11 Fitting results of potentiostatic at 1.3-volt 60°C -after 2.4 bar (Test2)

EIS After Compression 2.4 bar 31/01/26 Potentiostatic at 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05716 Ohm	Fixed	0.05933
Rct	0.018293 Ohm	Positive	0.032642
CPE1	-1 ...	Positive	0
TDE	0.0057936	Positive	0.26409
PHIDE	1.2519	Positive	0.028572
WO1	-1 ...	Positive	0
R	0.03642	Positive	0.078628
T	0.027912	Positive	0.081425
L1	2.81E-06 H	Positive	0.10829

Table 12 Fitting results of potentiostatic at 1.3-volt 60°C -before 3.4 bar (Test2)

EIS Before Compression 3.4 bar 31/01/26 Potentiostatic 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.056682 Ohm	Fixed	0.05933
Rct	0.018147 Ohm	Positive	0.032047
CPE1	-1 ...	Positive	0
TDE	0.0057176	Positive	0.25769
PHIDE	1.2543	Positive	0.027817
WO1	-1 ...	Positive	0
R	0.035954	Positive	0.07762
T	0.27582	Positive	0.080369
L1	2.8416e-06 H	Positive	0.10399

Table 13 Fitting results of potentiostatic at 1.3-volt 60°C -after 3.4 bar (Test2)

EIS After Compression 3.4 bar 03/02/26 Potentiostatic 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05439 Ohm	Fixed	0.05933
Rct	0.016096 Ohm	Positive	0.035765
CPE1	-1 ...	Positive	0
TDE	0.005618	Positive	0.2935
PHIDE	1.267	Positive	0.03126
WO1	-1 ...	Positive	0
R	0.037329	Positive	0.075235
T	0.30017	Positive	0.077657
L1	2.6271e-06 H	Positive	0.11657

Table 14 Fitting results of potentiostatic at 1.3-volt 60°C -before 4.4 bar (Test2)

EIS Before Compression 4.4 bar 03/02/26 Potentiostatic 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05538 Ohm	Fixed	0.05933
Rct	0.016111 Ohm	Positive	0.034611
CPE1	-1 ...	Positive	0
TDE	0.0055826	Positive	0.28135
PHIDE	1.2683	Positive	0.029974
WO1	-1 ...	Positive	0
R	0.035829	Positive	0.074911
T	0.28338	Positive	0.077082
L1	2.6614e-06 H	Positive	0.11074

Table 15 Fitting results of potentiostatic at 1.3-volt 60°C -after 4.4 bar (Test2)

EIS After Compression 4.4 bar 06/02/26 Potentiostatic 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05556 Ohm	Fixed	0.05933
Rct	0.014866 Ohm	Positive	0.038724
CPE1	-1 ...	Positive	0
TDE	0.0054912	Positive	0.32088
PHIDE	1.2769	Positive	0.033866
WO1	-1 ...	Positive	0
R	0.036529	Positive	0.077259
T	0.30625	Positive	0.079692
L1	2.5437e-06 H	Positive	0.12413

Table 16 Fitting results of potentiostatic at 1.3-volt 60°C -before 4.9 bar (Test2)

EIS Before Compression 4.9 bar 06/02/26 Potentiostatic 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.04936 Ohm	Fixed	0.05933
Rct	0.0234 Ohm	Positive	0.026214
CPE1	-1 ...	Positive	0
TDE	0.0064158	Positive	0.20959
PHIDE	1.218	Positive	0.02331
WO1	-1 ...	Positive	0
R	0.043063	Positive	0.070463
T	0.34926	Positive	0.07318
L1	3.0811e-06 H	Positive	0.093144

Table 17 Fitting results of potentiostatic at 1.3-volt 60°C -after 4.9 bar (Test2)

EIS After Compression 4.9 bar 09/02/26 Potentiostatic 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05385 Ohm	Fixed	0.05933
Rct	0.01549 Ohm	Positive	0.033583
CPE1	-1 ...	Positive	0
TDE	0.0057901	Positive	0.27799
PHIDE	1.267	Positive	0.029587
WO1	-1 ...	Positive	0
R	0.036364	Positive	0.069848
T	0.30575	Positive	0.0721
L1	2.5718e-06 H	Positive	0.10861

Table 18 Fitting results of potentiostatic at 1.3-volt 60°C -before 4.4 bar descent (Test2)

EIS Before Compression 4.4 bar Descent 09/02/26 Potentiostatic 1.3-volt T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05245 Ohm	Fixed	0.05933
Rct	0.018739 Ohm	Positive	0.033549
CPE1	-1 ...	Positive	0
TDE	0.0060694	Positive	0.27554
PHIDE	1.2439	Positive	0.029927
WO1	-1 ...	Positive	0
R	0.039214	Positive	0.078794
T	0.32792	Positive	0.081517
L1	2.7926e-06 H	Positive	0.11456

Table 19 Fitting results of potentiostatic at 1.5-volt 60°C -before 2.4 bar (Test2)

EIS Before Compression 2.4 bar 28/01/26 Potentiostatic at 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.05548 Ohm	Fixed	0.037322
Rct	0.021509 Ohm	Positive	0.016626
CPE1	-1 ...	Positive	0
TDE	0.009272	Positive	0.1179
PHIDE	1.1733	Positive	0.013734
Rmt	0.029014 Ohm	Positive	0.032155
CPE2	-1 ...	Positive	0
TDE	8.1753	Positive	0.053406
PHIDE	0.7829	Positive	0.033241
L1	2.8331e-06 H	Positive	0.046539

Table 20 Fitting results of potentiostatic at 1.5-volt 60°C -after 2.4 bar (Test2)

EIS After Compression 2.4 bar 31/01/26 Potentiostatic at 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.05831 Ohm	Fixed	0.037322
Rct	0.022108 Ohm	Positive	0.028268
CPE1	1 ...	Positive	0
TDE	0.010455	Positive	0.21162
PHIDE	1.156	Positive	0.024957
Rmt	0.029057	Positive	0.058275
CPE2	1 ...	Positive	0
TDE	7.0296	Positive	0.097862
PHIDE	0.82441	Positive	0.056665
L1	2.8301e-06 H	Positive	0.084448

Table 21 Fitting results of potentiostatic at 1.5-volt 60°C -before 3.4 bar (Test2)

EIS Before Compression 3.4 bar 31/01/26 Potentiostatic 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.05791 Ohm	Fixed	0.037322
Rct	0.019225 Ohm	Positive	0.024146
CPE1	1 ...	Positive	0
TDE	0.0079497	Positive	0.1613
PHIDE	1.222	Positive	0.018334
Rmt	0.031834 Ohm	Positive	0.035525
CPE2	1 ...	Positive	0

TDE	7.207	Positive	0.064185
PHIDE	0.7942	Positive	0.038572
L1	2.3532e-06 H	Positive	0.076003

Table 22 Fitting results of potentiostatic at 1.5-volt 60°C -after 3.4 bar (Test2)

EIS After Compression 3.4 bar 03/02/26 Potentiostatic 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.05566 Ohm	Fixed	0.037322
Rct	0.019427 Ohm	Positive	0.018849
CPE1	1 ...	Positive	0
TDE	0.0096196	Positive	0.13489
PHIDE	1.1773	Positive	0.015649
Rmt	0.030667 Ohm	Positive	0.031629
CPE2	1 ...	Positive	0
TDE	7.7634	Positive	0.052623
PHIDE	0.77676	Positive	0.032676
L1	2.6636e-06 H	Positive	0.051498

Table 23 Fitting results of potentiostatic at 1.5-volt 60°C -before 4.4 bar (Test2)

EIS Before Compression 4.4 bar 03/02/26 Potentiostatic 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.0566 Ohm	Fixed	0.037322
Rct	0.017302 Ohm	Positive	0.026911
CPE1	1 ...	Positive	0
TDE	0.0079103	Positive	0.17385
PHIDE	1.2313	Positive	0.019638
Rmt	0.030817 Ohm	Positive	0.045103
CPE2	1 ...	Positive	0
TDE	7.6083	Positive	0.072665
PHIDE	0.78028	Positive	0.044018
L1	2.2379e-06 H	Positive	0.078123

Table 24 Fitting results of potentiostatic at 1.5-volt 60°C -after 4.4 bar (Test2)

EIS After Compression 4.4 bar 06/02/26 Potentiostatic 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.057 Ohm	Fixed	0.037322
Rct	0.018214 Ohm	Positive	0.020144
CPE1	1 ...	Positive	0
TDE	0.010048	Positive	0.15059
PHIDE	1.178	Positive	0.017415
Rmt	0.028292 Ohm	Positive	0.03311
CPE2	1 ...	Positive	0
TDE	7.7739	Positive	0.057432
PHIDE	0.79533	Positive	0.03438
L1	2.5586e-06 H	Positive	0.056628

Table 25 Fitting results of potentiostatic at 1.5-volt 60°C -before 4.9 bar (Test2)

EIS Before Compression 4.9 bar 06/02/26 Potentiostatic 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.04959 Ohm	Fixed	0.037322
Rct	0.027132 Ohm	Positive	0.01873
CPE1	1 ...	Positive	0
TDE	0.0093614	Positive	0.11568
PHIDE	1.1528	Positive	0.013841
Rmt	0.031477 Ohm	Positive	0.037172
CPE2	1 ...	Positive	0
TDE	7.4289	Positive	0.06278
PHIDE	0.75782	Positive	0.040622
L1	3.1435e-06 H	Positive	0.048376

Table 26 Fitting results of potentiostatic at 1.5-volt 60°C -after 4.9 bar (Test2)

EIS After Compression 4.9 bar 09/02/26 Potentiostatic 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.05468 Ohm	Fixed	0.037322
Rct	0.018389 Ohm	Positive	0.019605
CPE1	1 ...	Positive	0
TDE	0.0095659	Positive	0.13713
PHIDE	1.1833	Positive	0.015856
Rmt	0.030354 Ohm	Positive	0.0321

CPE2	1 ...	Positive	0
TDE	8.1973	Positive	0.052082
PHIDE	0.76068	Positive	0.033166
L1	2.6012e-06 H	Positive	0.05099

Table 27 Fitting results of potentiostatic at 1.5-volt 60°C -before 4.4 bar descent (Test2)

EIS Before Compression 4.4 bar Descent 09/02/26 Potentiostatic 1.5-volt T 60°C			
HF & LF SEMI CIRCLES			Error
Rohm	0.0524 Ohm	Fixed	0.037322
Rct	0.021845 Ohm	Positive	0.017731
CPE1	1 ...	Positive	0
TDE	0.0093422	Positive	0.11675
PHIDE	1.1712	Positive	0.013696
Rmt	0.031408 Ohm	Positive	0.030182
CPE2	1 ...	Positive	0
TDE	8.1303	Positive	0.049899
PHIDE	0.7479	Positive	0.032864
L1	2.8254e-06 H	Positive	0.045976

Table 28 Fitting results of galvanostatic at 1.3-amps 60°C -before 2.4 bar (Test2)

EIS Before Compression 2.4 bar 28/01/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.054934 Ohm	Fixed	0.028401
Rct	0.022075 Ohm	Positive	0.030729
CPE1	1 ...	Positive	0
TDE	0.011327	Positive	0.23466
PHIDE	1.148	Positive	0.028685
L1	2.5986e-06 H	Positive	0.11158

Table 29 Fitting results of galvanostatic at 1.3-amps 60°C -after 2.4 bar (Test2)

EIS After Compression 2.4 bar 31/01/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0567 Ohm	Fixed	0.028401
Rct	0.022702 Ohm	Positive	0.028802
CPE1	1 ...	Positive	0
TDE	0.012543	Positive	0.22166
PHIDE	1.1311	Positive	0.027476
L1	2.5832e-06 H	Positive	0.10577

Table 30 Fitting results of galvanostatic at 1.3-amps 60°C -before 3.4 bar (Test2)

EIS Before Compression 3.4 bar 31/01/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0559125 Ohm	Fixed	0.028401
Rct	0.022603 Ohm	Positive	0.026966
CPE1	1 ...	Positive	0
TDE	0.013719	Positive	0.22659
PHIDE	1.1215	Positive	0.028259
L1	2.5844e-06 H	Positive	0.10978

Table 31 Fitting results of galvanostatic at 1.3-amps 60°C -after 3.4 bar (Test2)

EIS After Compression 3.4 bar 03/02/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0538765 Ohm	Fixed	0.028401
Rct	0.02054 Ohm	Positive	0.028963
CPE1	1 ...	Positive	0
TDE	0.014225	Positive	0.24814
PHIDE	1.1244	Positive	0.03075
L1	2.3806e-06 H	Positive	0.11845

Table 32 Fitting results of galvanostatic at 1.3-amps 60°C -before 4.4 bar (Test2)

EIS Before Compression 4.4 bar 03/02/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.054638 Ohm	Fixed	0.028401
Rct	0.019895 Ohm	Positive	0.033601
CPE1	1 ...	Positive	0
TDE	0.011106	Positive	0.23664
PHIDE	1.158	Positive	0.028661
L1	2.4677e-06 H	Positive	0.10373

Table 33 Fitting results of galvanostatic at 1.3-amps 60°C -after 4.4 bar (Test2)

EIS After Compression 4.4 bar 06/02/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0549286 Ohm	Fixed	0.028401

Rct	0.019257 Ohm	Positive	0.031941
CPE1	1 ...	Positive	0
TDE	0.015176	Positive	0.27601
PHIDE	1.1226	Positive	0.034211
L1	2.2781e-06 H	Positive	0.1284

Table34 Fitting results of galvanostatic at 1.3-amps 60°C -before 4.9 bar (Test2)

EIS Before Compression 4.9 bar 06/02/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0492226 Ohm	Fixed	0.028401
Rct	0.028439 Ohm	Positive	0.02387
CPE1	1 ...	Positive	0
TDE	0.013153	Positive	0.19366
PHIDE	1.1066	Positive	0.024665
L1	2.8323e-06 H	Positive	0.10632

Table 35 Fitting results of galvanostatic at 1.3-amps 60°C -after 4.9 bar (Test2)

EIS After Compression 4.9 bar 09/02/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0535054 Ohm	Fixed	0.028401
Rct	0.01907 Ohm	Positive	0.035311
CPE1	1 ...	Positive	0
TDE	0.010711	Positive	0.25016
PHIDE	1.166	Positive	0.030055
L1	2.4279e-06 H	Positive	0.10833

Table 36 Fitting results of galvanostatic at 1.3-amps 60°C -before 4.4 bar descent (Test2)

EIS Before Compression 4.4 bar Descent 09/02/26 Galvanostatic at 1.3Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05206 Ohm	Fixed	0.028401
Rct	0.022925 Ohm	Positive	0.028246
CPE1	1 ...	Positive	0
TDE	0.012547	Positive	0.21641
PHIDE	1.1308	Positive	0.026855
L1	2.5449e-06 H	Positive	0.1059

Table 37 Fitting results of galvanostatic at 1.5-amps 60°C -before 2.4 bar (Test2)

EIS Before Compression 2.4 bar 28/01/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0549065 Ohm	Fixed	0.028401
Rct	0.021592 Ohm	Positive	0.032237
CPE1	1 ...	Positive	0
TDE	0.010145	Positive	0.22364
PHIDE	1.1619	Positive	0.027069
L1	2.6095e-06 H	Positive	0.10164

Table 38 Fitting results of galvanostatic at 1.5-amps 60°C -after 2.4 bar (Test2)

EIS After Compression 2.4 bar 31/01/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0569318 Ohm	Fixed	0.028401
Rct	0.022506 Ohm	Positive	0.027902
CPE1	1 ...	Positive	0
TDE	0.012074	Positive	0.21486
PHIDE	1.1365	Positive	0.026499
L1	2.6048e-06 H	Positive	0.10171

Table 39 Fitting results of galvanostatic at 1.5-amps 60°C -before 3.4 bar (Test2)

EIS Before Compression 3.4 bar 31/01/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.056183 Ohm	Fixed	0.028401
Rct	0.022536 Ohm	Positive	0.027215
CPE1	1 ...	Positive	0
TDE	0.013599	Positive	0.22773
PHIDE	1.1238	Positive	0.028363
L1	2.5785e-06 H	Positive	0.11078

Table 40 Fitting results of galvanostatic at 1.5-amps 60°C -after 3.4 bar (Test2)

EIS After Compression 3.4 bar 03/02/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0540703 Ohm	Fixed	0.028401
Rct	0.019793 Ohm	Positive	0.033008

CPE1	1 ...	Positive	0
TDE	0.010943	Positive	0.23184
PHIDE	1.1608	Positive	0.028023
L1	2.4225e-06 H	Positive	0.1036

Table 41 Fitting results of galvanostatic at 1.5-amps 60°C -before 4.4 bar (Test2)

EIS Before Compression 4.4 bar 03/02/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.05496 Ohm	Fixed	0.028401
Rct	0.020046 Ohm	Positive	0.029485
CPE1	1 ...	Positive	0
TDE	0.012235	Positive	0.22985
PHIDE	1.146	Positive	0.028038
L1	2.4617e-06 H	Positive	0.10389

Table 42 Fitting results of galvanostatic at 1.5-amps 60°C -after 4.4 bar (Test2)

EIS After Compression 4.4 bar 06/02/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0548328 Ohm	Fixed	0.028401
Rct	0.01863 Ohm	Positive	0.034064
CPE1	1 ...	Positive	0
TDE	0.01115	Positive	0.24356
PHIDE	1.1623	Positive	0.029309
L1	2.3849e-06 H	Positive	0.10398

Table 43 Fitting results of galvanostatic at 1.5-amps 60°C -before 4.9 bar (Test2)

EIS Before Compression 4.9 bar 06/02/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0491 Ohm	Fixed	0.028401
Rct	0.028581 Ohm	Positive	0.023592
CPE1	1 ...	Positive	0
TDE	0.013458	Positive	0.19122
PHIDE	1.1034	Positive	0.024431
L1	2.8866e-06 H	Positive	0.10303

Table 44 Fitting results of galvanostatic at 1.5-amps 60°C -after 4.9 bar (Test2)

EIS After Compression 4.9 bar 09/02/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0536719 Ohm	Fixed	0.028401
Rct	0.019211 Ohm	Positive	0.030751
CPE1	1 ...	Positive	0
TDE	0.013025	Positive	0.23976
PHIDE	1.1437	Positive	0.029309
L1	2.3478e-06 H	Positive	0.10846

Table 45 Fitting results of galvanostatic at 1.5-amps 60°C -before 4.4 bar descent (Test2)

EIS Before Compression 4.4 bar Descent 09/02/26 Galvanostatic at 1.5Amps T 60°C			
HF SEMI CIRCLE			Error
Rohm	0.0518 Ohm	Fixed	0.028401
Rct	0.023248 Ohm	Positive	0.02637
CPE1	1 ...	Positive	0
TDE	0.013458	Positive	0.22218
PHIDE	1.1199	Positive	0.027738
L1	2.6014e-06 H	Positive	0.10963

A.1 — Python Code for Definition of Circuit Elements (R, L, CPE, Warburg)

```
def Z_R(R):
    return R

def Z_L(L, freq):
    w = 2*np.pi*freq
    return 1j*w*L

def Z_CPE(Q, n, freq):
    w = 2*np.pi*freq
    return 1 / (Q * (1j*w)**n)

def Z_parallel(Z1, Z2):
    return 1 / (1/Z1 + 1/Z2)

def Z_Ws(R, T, PHI, freq):
    w = 2*np.pi*freq
    x = (1j*w*T)**PHI
    return R * np.tanh(x) / x
```

A.2 — Full Equivalent Circuit Model (Path B)

```
def Z_model(params, freq):

    L0, R0, R1, Q1, n1, R2, Q2, n2, WR, WT, WPHI = params
    ZL = Z_L(L0, freq)

    ZR0 = Z_R(R0)
    Z1 = Z_parallel(Z_R(R1), Z_CPE(Q1, n1, freq))

    Z2 = Z_parallel(Z_R(R2), Z_CPE(Q2, n2, freq))

    ZW = Z_Ws(WR, WT, WPHI, freq)
    return ZL + ZR0 + Z1 + Z2 + ZW
```

A.3 — Data Loading and Preprocessing

```
df = pd.read_csv("file.csv")

freq = df.iloc[:,0].values

Zr = df.iloc[:,1].values
Zi = df.iloc[:,2].values
Z = Zr + 1j*Zi
idx = np.argsort(freq)
freq = freq[idx]
Z = Z[idx]
```

A.4 — Parameter Initialization and Bounds

```
initial = np.array([
    1e-6,   # L0
    0.0556, # R0
    0.02092, # R1
    0.039418595, # Q1
    1,      # n1
    0.02548, # R2
    8.797562526, # Q2
    1,      # n2
    0.0098,  # WR
    0.1,     # WT
    0.5      # WPHI
])
status = [
    'positive', # L0
    'positive', # R0
    'positive', # R1
    'positive', # Q1
    'fixed',   # n1
    'positive', # R2
    'positive', # Q2
    'fixed',   # n2
    'fixed',   # WR
    'free',    # WT
    'fixed',   # WPHI
]
```

A.5 — Optimization Procedure (Run)

```
init, lb, ub = build_bounds(initial, status)

def residuals(p):
    Zm = Z_model(p, freq)

    return np.concatenate([(Zm.real - Z.real), (Zm.imag - Z.imag)])

res = least_squares(residuals, init, bounds=(lb, ub))

params = res.x
print("\n=== SINGLE RUN RESULTS ===")

names = ["L0", "R0", "R1", "Q1", "n1", "R2", "Q2", "n2", "WR", "WT", "WPHI"]

for n, v in zip(names, params):
    print(f"{n:5s} = {v:.6g}")
```

A.6 —Nyquist Plot (Matplotlib)

```
freq_pred = np.logspace(np.log10(freq.min()), np.log10(freq.max()), 400)
Zfit = Z_model(params, freq_pred)

plt.figure(figsize=(7,7))

plt.plot(Z.real, -Z.imag, 'o', mfc='white', mec='blue', label='Data')

plt.plot(Zfit.real, -Zfit.imag, '-', lw=2, c='red', label='Fit')
plt.xlabel("Z' (Ohm)")

plt.ylabel("-Z'' (Ohm)")

plt.legend()
plt.grid(alpha=0.3)
plt.gca().set_aspect('equal', 'box')
plt.show()
```

Bibliography

- [1] I. Staffell *et al.*, “The role of hydrogen and fuel cells in the global energy system,” *Energy Environ. Sci.*, vol. 12, no. 2, pp. 463–491, 2019, doi: 10.1039/C8EE01157E.
- [2] S. A. Grigoriev, V. N. Fateev, D. G. Bessarabov, and P. Millet, “Current status, research trends, and challenges in water electrolysis science and technology,” *International Journal of Hydrogen Energy*, vol. 45, no. 49, pp. 26036–26058, Oct. 2020, doi: 10.1016/j.ijhydene.2020.03.109.
- [3] T. Wang, X. Cao, and L. Jiao, “PEM water electrolysis for hydrogen production: fundamentals, advances, and prospects,” *Carb Neutrality*, vol. 1, no. 1, p. 21, Dec. 2022, doi: 10.1007/s43979-022-00022-8.
- [4] S. Shiva Kumar and V. Himabindu, “Hydrogen production by PEM water electrolysis – A review,” *Materials Science for Energy Technologies*, vol. 2, no. 3, pp. 442–454, Dec. 2019, doi: 10.1016/j.mset.2019.03.002.
- [5] M. Carmo, D. L. Fritz, J. Mergel, and D. Stolten, “A comprehensive review on PEM water electrolysis,” *International Journal of Hydrogen Energy*, vol. 38, no. 12, pp. 4901–4934, Apr. 2013, doi: 10.1016/j.ijhydene.2013.01.151.
- [6] M. Kisti *et al.*, “Recent Advances in Polymer Electrolyte Membrane Water Electrolyzer Stack Development Studies: A Review,” *ACS Omega*, vol. 10, no. 10, pp. 9824–9853, Mar. 2025, doi: 10.1021/acsomega.4c10147.
- [7] K. Ayers, “High efficiency PEM water electrolysis: enabled by advanced catalysts, membranes, and processes,” *Current Opinion in Chemical Engineering*, vol. 33, p. 100719, Sep. 2021, doi: 10.1016/j.coche.2021.100719.
- [8] J. Wang *et al.*, “Comparative experimental study of alkaline and proton exchange membrane water electrolysis for green hydrogen production,” *Applied Energy*, vol. 379, p. 124936, Feb. 2025, doi: 10.1016/j.apenergy.2024.124936.
- [9] B. Endrődi, C. A. Trapp, I. Szén, I. Bakos, M. Lukovics, and C. Janáky, “Challenges and Opportunities of the Dynamic Operation of PEM Water Electrolyzers,” *Energies*, vol. 18, no. 9, p. 2154, Apr. 2025, doi: 10.3390/en18092154.
- [10] M. F. Ahmad Kamaroddin *et al.*, “Membrane-Based Electrolysis for Hydrogen Production: A Review,” *Membranes*, vol. 11, no. 11, p. 810, Oct. 2021, doi: 10.3390/membranes11110810.
- [11] K. Zhang *et al.*, “Status and perspectives of key materials for PEM electrolyzer,” *Nano Research Energy*, vol. 1, p. e9120032, Dec. 2022, doi: 10.26599/NRE.2022.9120032.
- [12] L. Messing *et al.*, “Carbon Bipolar Plates in PEM Water Electrolysis: Bust or Must?,” *Advanced Energy Materials*, vol. 14, no. 40, p. 2402308, Oct. 2024, doi: 10.1002/aenm.202402308.
- [13] S. Stiber *et al.*, “A high-performance, durable and low-cost proton exchange membrane electrolyser with stainless steel components,” *Energy Environ. Sci.*, vol. 15, no. 1, pp. 109–122, 2022, doi: 10.1039/D1EE02112E.
- [14] J. O. Majasan, J. I. S. Cho, I. Dedigama, D. Tsaoulidis, P. Shearing, and D. J. L. Brett, “Two-phase flow behaviour and performance of polymer electrolyte membrane electrolyzers: Electrochemical and optical characterisation,” *International Journal of*

- Hydrogen Energy*, vol. 43, no. 33, pp. 15659–15672, Aug. 2018, doi: 10.1016/j.ijhydene.2018.07.003.
- [15] A. Bayat, P. K. Das, G. Saha, and S. C. Saha, “Proton Exchange Membrane Electrolysis Revisited: Advancements, Challenges, and Two-Phase Transport Insights in Materials and Modelling,” *Eng*, vol. 6, no. 4, p. 72, Apr. 2025, doi: 10.3390/eng6040072.
 - [16] E. Wallnöfer-Ogris *et al.*, “A review on understanding and identifying degradation mechanisms in PEM water electrolysis cells: Insights for stack application, development, and research,” *International Journal of Hydrogen Energy*, vol. 65, pp. 381–397, May 2024, doi: 10.1016/j.ijhydene.2024.04.017.
 - [17] T. L. Doan *et al.*, “A review of the porous transport layer in polymer electrolyte membrane water electrolysis,” *Intl J of Energy Research*, vol. 45, no. 10, pp. 14207–14220, Aug. 2021, doi: 10.1002/er.6739.
 - [18] D. L. Canelas Montaña, P. Ruiz García, A. Jerez Navarro, M. Aguirre Gomez, and J. J. López Cascales, “Effect of the Cathodic Gas Diffusion Layer on the Performance of a Proton Exchange Membrane Electrolyzer,” *Energies*, vol. 17, no. 21, p. 5298, Oct. 2024, doi: 10.3390/en17215298.
 - [19] Y. Huang *et al.*, “Understanding the impact of the gas diffusion layer structure on catalyst utilization in the PEM water electrolyzer,” *Next Energy*, vol. 8, p. 100319, Jul. 2025, doi: 10.1016/j.nxener.2025.100319.
 - [20] K. W. Ahmed, M. J. Jang, M. G. Park, Z. Chen, and M. Fowler, “Effect of Components and Operating Conditions on the Performance of PEM Electrolyzers: A Review,” *Electrochem*, vol. 3, no. 4, pp. 581–612, Sep. 2022, doi: 10.3390/electrochem3040040.
 - [21] H. Zhang *et al.*, “Investigation on the Influence of Structural Parameters in the Porous Transport Layer on the Mass Transfer Process within Pem Water Electrolyzer,” 2025, SSRN. doi: 10.2139/ssrn.5100798.
 - [22] T. Bautkinova *et al.*, “Introducing titanium hydride on porous transport layer for more energy efficient water electrolysis with proton exchange membrane,” *Journal of Power Sources*, vol. 565, p. 232913, May 2023, doi: 10.1016/j.jpowsour.2023.232913.
 - [23] Z. Kang, S. M. Alia, J. L. Young, and G. Bender, “Effects of various parameters of different porous transport layers in proton exchange membrane water electrolysis,” *Electrochimica Acta*, vol. 354, p. 136641, Sep. 2020, doi: 10.1016/j.electacta.2020.136641.
 - [24] P. Lettenmeier *et al.*, “Comprehensive investigation of novel pore-graded gas diffusion layers for high-performance and cost-effective proton exchange membrane electrolyzers,” *Energy Environ. Sci.*, vol. 10, no. 12, pp. 2521–2533, 2017, doi: 10.1039/C7EE01240C.
 - [25] A. S. Pushkarev *et al.*, “On the influence of porous transport layers parameters on the performances of polymer electrolyte membrane water electrolysis cells,” *Electrochimica Acta*, vol. 399, p. 139436, Dec. 2021, doi: 10.1016/j.electacta.2021.139436.
 - [26] J. C. Cruz *et al.*, “Electrochemical and Microstructural Analysis of a Modified Gas Diffusion Layer for a PEM Water Electrolyzer,” *International Journal of Electrochemical Science*, vol. 15, no. 6, pp. 5571–5584, Jun. 2020, doi: 10.20964/2020.06.12.
 - [27] T. L. Doan, H. E. Lee, M. Kim, W. C. Cho, H. S. Cho, and T. Kim, “Influence of IrO₂/TiO₂ coated titanium porous transport layer on the performance of PEM water electrolysis,” *Journal of Power Sources*, vol. 533, p. 231370, Jun. 2022, doi: 10.1016/j.jpowsour.2022.231370.

- [28] G. Liu, S. Peng, F. Hou, B. Fang, and X. Wang, "Au-TiO₂/Ti Hybrid Coating as a Liquid and Gas Diffusion Layer with Improved Performance and Stability in Proton Exchange Membrane Water Electrolyzer," *Molecules*, vol. 27, no. 19, p. 6644, Oct. 2022, doi: 10.3390/molecules27196644.
- [29] N. Hensle, J. Hoffmann, Z. Najafianashrafi, T. Smolinka, P.-Y. A. Chuang, and A. Weber, "Understanding the Use of Carbon-Based Porous Transport Layers at the Cathode in Pem Water Electrolysis," 2025, *SSRN*. doi: 10.2139/ssrn.5117944.
- [30] U. Rost *et al.*, "A novel advanced test system for polymer electrolyte membrane water electrolysis based on hydraulic cell compression," *Fuel Cells*, vol. 22, no. 6, pp. 284–289, Dec. 2022, doi: 10.1002/fuce.202200068.
- [31] E. Borgardt *et al.*, "Impact of clamping pressure and stress relaxation on the performance of different polymer electrolyte membrane water electrolysis cell designs," *International Journal of Hydrogen Energy*, vol. 44, no. 42, pp. 23556–23567, Sep. 2019, doi: 10.1016/j.ijhydene.2019.07.075.
- [32] A. Martin *et al.*, "The Effect of Cell Compression and Cathode Pressure on Hydrogen Crossover in PEM Water Electrolysis," *J. Electrochem. Soc.*, vol. 169, no. 1, p. 014502, Jan. 2022, doi: 10.1149/1945-7111/ac4459.
- [33] I. V. Zenyuk, D. Y. Parkinson, L. G. Connolly, and A. Z. Weber, "Gas-diffusion-layer structural properties under compression via X-ray tomography," *Journal of Power Sources*, vol. 328, pp. 364–376, Oct. 2016, doi: 10.1016/j.jpowsour.2016.08.020.
- [34] M. Stähler, A. Stähler, F. Scheepers, M. Carmo, W. Lehnert, and D. Stolten, "Impact of porous transport layer compression on hydrogen permeation in PEM water electrolysis," *International Journal of Hydrogen Energy*, vol. 45, no. 7, pp. 4008–4014, Feb. 2020, doi: 10.1016/j.ijhydene.2019.12.016.
- [35] U. Babic, M. Tarik, T. J. Schmidt, and L. Gubler, "Understanding the effects of material properties and operating conditions on component aging in polymer electrolyte water electrolyzers," *Journal of Power Sources*, vol. 451, p. 227778, Mar. 2020, doi: 10.1016/j.jpowsour.2020.227778.
- [36] O. F. Selamat and M. S. Ergoktas, "Effects of bolt torque and contact resistance on the performance of the polymer electrolyte membrane electrolyzers," *Journal of Power Sources*, vol. 281, pp. 103–113, May 2015, doi: 10.1016/j.jpowsour.2015.01.162.
- [37] S. H. Frensch, A. C. Olesen, S. S. Araya, and S. K. Kær, "Model-supported characterization of a PEM water electrolysis cell for the effect of compression," *Electrochimica Acta*, vol. 263, pp. 228–236, Feb. 2018, doi: 10.1016/j.electacta.2018.01.040.
- [38] E. Cruz Ortiz, N. Van Treel, S. Koch, S. Vierrath, and M. Bühler, "The effect of compression on PEM Electrolyzer membrane electrode Assemblies," *Journal of Power Sources*, vol. 614, p. 235018, Sep. 2024, doi: 10.1016/j.jpowsour.2024.235018.
- [39] T. Srouf *et al.*, "On the contact resistance between the anode and the porous transport layer in a proton exchange membrane water electrolyzer," *International Journal of Hydrogen Energy*, vol. 58, pp. 351–361, Mar. 2024, doi: 10.1016/j.ijhydene.2024.01.134.
- [40] J. Mo *et al.*, "Thin liquid/gas diffusion layers for high-efficiency hydrogen production from water splitting," *Applied Energy*, vol. 177, pp. 817–822, Sep. 2016, doi: 10.1016/j.apenergy.2016.05.154.

- [41] B.-S. Lee *et al.*, “Polarization characteristics of a low catalyst loading PEM water electrolyzer operating at elevated temperature,” *Journal of Power Sources*, vol. 309, pp. 127–134, Mar. 2016, doi: 10.1016/j.jpowsour.2015.12.139.
- [42] J. Ge, A. Higier, and H. Liu, “Effect of gas diffusion layer compression on PEM fuel cell performance,” *Journal of Power Sources*, vol. 159, no. 2, pp. 922–927, Sep. 2006, doi: 10.1016/j.jpowsour.2005.11.069.
- [43] J. Liu, Y. Yang, F. Kerner, and D. Schröder, “Unraveling the impact of compression on the performance of porous transport layers in water Electrolyzers,” *Applied Energy*, vol. 381, p. 124982, Mar. 2025, doi: 10.1016/j.apenergy.2024.124982.
- [44] M. Bernt, J. Schröter, M. Möckl, and H. A. Gasteiger, “Analysis of Gas Permeation Phenomena in a PEM Water Electrolyzer Operated at High Pressure and High Current Density,” *J. Electrochem. Soc.*, vol. 167, no. 12, p. 124502, Jan. 2020, doi: 10.1149/1945-7111/abaa68.
- [45] S. Bin *et al.*, “High-pressure proton exchange membrane water electrolysis: Current status and challenges in hydrogen production,” *International Journal of Hydrogen Energy*, vol. 67, pp. 390–405, May 2024, doi: 10.1016/j.ijhydene.2024.04.188.
- [46] L. Zhu, A. Tugirumubano, and A. Bazylak, “Revealing localized compression induced degradation mechanisms in polymer electrolyte membrane water electrolyzers,” *Energy Conversion and Management*, vol. 340, p. 119947, Sep. 2025, doi: 10.1016/j.enconman.2025.119947.
- [47] M. N. I. Salehmin, T. Husaini, J. Goh, and A. B. Sulong, “High-pressure PEM water electrolyser: A review on challenges and mitigation strategies towards green and low-cost hydrogen production,” *Energy Conversion and Management*, vol. 268, p. 115985, Sep. 2022, doi: 10.1016/j.enconman.2022.115985.
- [48] G. Tjarks, A. Gibelhaus, F. Lanzerath, M. Müller, A. Bardow, and D. Stolten, “Energetically-optimal PEM electrolyzer pressure in power-to-gas plants,” *Applied Energy*, vol. 218, pp. 192–198, May 2018, doi: 10.1016/j.apenergy.2018.02.155.
- [49] C. C. Weber, J. A. Wrubel, L. Gubler, G. Bender, S. De Angelis, and F. N. Büchi, “How the Porous Transport Layer Interface Affects Catalyst Utilization and Performance in Polymer Electrolyte Water Electrolysis,” *ACS Appl. Mater. Interfaces*, vol. 15, no. 29, pp. 34750–34763, Jul. 2023, doi: 10.1021/acsami.3c04151.
- [50] M. Suermann, T. Kiupel, T. J. Schmidt, and F. N. Büchi, “Electrochemical Hydrogen Compression: Efficient Pressurization Concept Derived from an Energetic Evaluation,” *J. Electrochem. Soc.*, vol. 164, no. 12, pp. F1187–F1195, 2017, doi: 10.1149/2.1361712jes.
- [51] F. Maoulida, D. Guilbert, M.-B. Camara, and B. Dakyo, “Dynamic electrical degradation of PEM electrolyzers under renewable energy Intermittency: Mechanisms, diagnostics, and mitigation strategies – A comprehensive review,” *Renewable and Sustainable Energy Reviews*, vol. 225, p. 116170, Jan. 2026, doi: 10.1016/j.rser.2025.116170.
- [52] L. V. Bühre *et al.*, “Analysis of Kinetic and Ohmic Resistances in PEM Water Electrolysis through Reference Electrode Measurements,” *J. Electrochem. Soc.*, vol. 171, no. 5, p. 054518, May 2024, doi: 10.1149/1945-7111/ad4b5d.
- [53] F. Maoulida, D. Guilbert, M.-B. Camara, and B. Dakyo, “Dynamic electrical degradation of PEM electrolyzers under renewable energy Intermittency: Mechanisms,

- diagnostics, and mitigation strategies – A comprehensive review,” *Renewable and Sustainable Energy Reviews*, vol. 225, p. 116170, Jan. 2026, doi: 10.1016/j.rser.2025.116170.
- [54] L. V. Bühre *et al.*, “Analysis of Kinetic and Ohmic Resistances in PEM Water Electrolysis through Reference Electrode Measurements,” *J. Electrochem. Soc.*, vol. 171, no. 5, p. 054518, May 2024, doi: 10.1149/1945-7111/ad4b5d.
- [55] B. Kimmel *et al.*, “Investigation of the Degradation Phenomena of a Proton Exchange Membrane Electrolyzer Stack by Successive Replacement of Aged Components in Single Cells,” *ACS Sustainable Chem. Eng.*, vol. 13, no. 11, pp. 4330–4340, Mar. 2025, doi: 10.1021/acssuschemeng.4c07358.
- [56] J. Zuo, N. Y. Steiner, Z. Li, and D. Hissel, “Interpreting Full-Frequency Impedance Spectrum for Pem Electrolyzers: Distribution of Relaxation Times-Based Modeling,” 2024, *SSRN*. doi: 10.2139/ssrn.5039136.
- [57] J. Woelke, A. Rex, C. Eckert, B. Bensmann, and R. Hanke-Rauschenbach, “Predicting future polarization curves from operating data: Machine learning-based investigation of degradation modeling concepts for PEM water electrolysis,” *Energy and AI*, vol. 21, p. 100547, Sep. 2025, doi: 10.1016/j.egyai.2025.100547.
- [58] D. D. Macdonald, “Reflections on the history of electrochemical impedance spectroscopy,” *Electrochimica Acta*, vol. 51, no. 8–9, pp. 1376–1388, Jan. 2006, doi: 10.1016/j.electacta.2005.02.107.
- [59] S. Siracusano, S. Trocino, N. Briguglio, V. Baglio, and A. S. Aricò, “Electrochemical Impedance Spectroscopy as a Diagnostic Tool in Polymer Electrolyte Membrane Electrolysis,” *Materials*, vol. 11, no. 8, p. 1368, Aug. 2018, doi: 10.3390/ma11081368.
- [60] C. Rozain and P. Millet, “Electrochemical characterization of Polymer Electrolyte Membrane Water Electrolysis Cells,” *Electrochimica Acta*, vol. 131, pp. 160–167, Jun. 2014, doi: 10.1016/j.electacta.2014.01.099.
- [61] S. Siracusano, N. Van Dijk, R. Backhouse, L. Merlo, V. Baglio, and A. S. Aricò, “Degradation issues of PEM electrolysis MEAs,” *Renewable Energy*, vol. 123, pp. 52–57, Aug. 2018, doi: 10.1016/j.renene.2018.02.024.
- [62] L. Zhao, H. Dai, F. Pei, P. Ming, X. Wei, and J. Zhou, “A Comparative Study of Equivalent Circuit Models for Electro-Chemical Impedance Spectroscopy Analysis of Proton Exchange Membrane Fuel Cells,” *Energies*, vol. 15, no. 1, p. 386, Jan. 2022, doi: 10.3390/en15010386.
- [63] I. V. Pushkareva, M. A. Solovyev, S. I. Butrim, M. V. Kozlova, D. A. Simkin, and A. S. Pushkarev, “On the Operational Conditions’ Effect on the Performance of an Anion Exchange Membrane Water Electrolyzer: Electrochemical Impedance Spectroscopy Study,” *Membranes*, vol. 13, no. 2, p. 192, Feb. 2023, doi: 10.3390/membranes13020192.
- [64] J. Zuo, N. Y. Steiner, Z. Li, and D. Hissel, “Quantitative investigation of internal polarization in a proton exchange membrane water electrolyzer stack using distribution of relaxation times,” *Applied Energy*, vol. 386, p. 125543, May 2025, doi: 10.1016/j.apenergy.2025.125543.
- [65] K. Khoiruddin, C. S. A. Nandar, S. Kawi, T. M. Lim, and I. G. Wenten, “Advanced proton exchange membranes for high-efficiency fuel cells: material innovations and

- durability optimization,” *Energy Mater.*, vol. 5, no. 11, Aug. 2025, doi: 10.20517/energymater.2025.62.
- [66] A. Weiß, A. Siebel, M. Bernt, T.-H. Shen, V. Tileli, and H. A. Gasteiger, “Impact of Intermittent Operation on Lifetime and Performance of a PEM Water Electrolyzer,” *J. Electrochem. Soc.*, vol. 166, no. 8, pp. F487–F497, 2019, doi: 10.1149/2.0421908jes.
- [67] C. R. Wang *et al.*, “Proton Exchange Membrane (PEM) Water Electrolysis: Cell-Level Considerations for Gigawatt-Scale Deployment,” *Chem. Rev.*, vol. 125, no. 3, pp. 1257–1302, Feb. 2025, doi: 10.1021/acs.chemrev.3c00904.