Dataset of standard tests of Nafion 112 membrane and Membrane Electrode Assembly (MEA) activation tests of Proton Exchange Membrane (PEM) fuel cell

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Abstract:
Reported data in this paper are about Nafion 112 membrane standard tests and MEA activation tests of PEM fuel cell in various operation condition. Dataset include two general electrochemical analysis method, Polarization and Impedance curves. In this dataset, effect of different pressure of H\textsubscript{2}/O\textsubscript{2} gas, different voltages and various humidity conditions in several steps are considered. Details of experimental methods has been explained in this paper. Behavior of PEM fuel cell during distinct operation condition tests, activation procedure and different operation condition before and after activation analysis can be concluded from data. In Polarization curves, voltage and power density change as a function of flows of H\textsubscript{2}/O\textsubscript{2} and relative humidity. Resistance of the used equivalent circuit of fuel cell can be calculated from Impedance data. Thus, experimental response of the cell is obvious in the presented data, which is useful in depth analysis, simulation and material performance investigation in PEM fuel cell researches.

Highlights:
- Experimentally data useful for investigation and work on PEMFCs.
- Data obtained from various test conditions and changing variables that provides possibility for users to compare curves and resulted parameters.
- Observable graphical data in presented documents help better interpretation for research on PEMFC.

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1- Data Introduction

The experimentally resulted data shows the performance of a PEMFC at several percent of membrane compression, different applied voltages, different pressure of H₂/O₂ gas, and various humidity conditions of cathode and air, which can be used to study behavior of PEMFC that is necessary to research and development of fuel cells. In other words, the dataset help researchers and specialists who investigate and work on PEM fuel cells [1].

Polarization and Impedance curves have obtained from specific empirical operation condition. MEA structure defines as composition of anode, membrane and cathode. Temperature of anode, cathode and cell, pressure and flow rate of H₂/O₂ (ml.min⁻¹) have been considered as operation condition during the evaluation. In Polarization curves, cell voltage (V) per current density (mA.cm⁻²) and cell power density (mW.cm⁻²) per current density (mA.cm⁻²) has been obtained at various relative humidity, gas pressure and membrane compression. Impedance analysis were done at the end of the each activation set and procedure at different cell voltages, relative humidity and H₂/O₂ pressure. Also, in each activation procedure, analysis has been accomplished by repeat of activation sets [2].

Obtained data can be useful for simulation of PEMFC and simulation has important role in scientific and applied studies. The report provides necessary results and experimentally parameters such as temperature of anode, cathode and cell, pressure and flow rate of gasses, relative humidity, power density, current density, voltage and resistances of cells which are obligatory data for electrochemical, material, mechanical and electrical simulation of PEMFCs. Hence, obtained data used for simulation of PEM in OPEM [3] simulation software produced by Electrochemistry Simulation (ECSIM) organization research team and those are compatible with the most of used models, especially, Amphlett model [4] in OPEM software. The reported dataset is available on ECSIM organization GitHub account [5]. This work is licensed under a Creative Commons Attribution 4.0 International License. The images or other third party material in this article are included in the article’s Creative Commons license, unless indicated otherwise in the credit line; if the material is not included under the Creative Commons license, users will need to obtain permission from the license holder to reproduce the material [6].
2- Report Data
2-1- Standard test of Nafion 112 membrane
2-1-1- Experimental design, method and details

MEA structure for standard cell of Nafion 112 membrane includes anode, cathode and membrane composition. Composition of anode in standard test is Carbon paper Ballard, Platinum-Carbon 20%, Nafion solution (27% weight), 80 ml Isopropyl alcohol, and 20 ml double distillation water with 0.39 \( mg.cm^{-2} \) loading of Platinum. Also, composition of cathode including Carbon paper (CP) Ballard, Platinum-Carbon (Pt/C) 20%, Nafion solution (25% weight), 80 ml Isopropyl alcohol and 20 ml double distillation water with 0.39 \( mg.cm^{-2} \) loading of Platinum. Composition of second cathode is Carbon paper Ballard, Platinum-Carbon 20%, Nafion solution (20% weight), 80 ml Isopropyl alcohol, and 20 ml double distillation water with 0.39 \( mg.cm^{-2} \) loading of Platinum. Cell temperature is 75 degree centigrade (°C), anode temperature is 80°C and Cathode temperature was evaluated 48-59-70-75 °C. For activation, in first step a constant voltage at 0.6 V for 30 minutes applied, and then constant voltage at 0.2 V for 10 minutes and constant voltage at 0.7 V for 1 minute. In next step, constant voltage at 0.6 V for 60 minutes and then, constant voltage at 0.5 V for 40 minutes. At the end of activation operation, a constant current at 250 \( mA.cm^{-2} \) has been applied for 60 minutes.

Effect of percent of Nafion membrane N (%) and Relative Humidity of the Cathode RHC (%) on cell performance in various compression of membrane at various pressure was analyzed in Polarization curves. Changing in N (%) between 20% and 25%, RHC (%) between 30, 50, 80 and 100%, P between 5, 15 and 25 psig, and membrane compression percent (%) lead to change the value of voltage and power density per current density. From each polarization curve, in table (1-2) and figure (1-2), an example of data extracted from the curve and related polarization curve presented, respectively.
Figure (1-2): Polarization curve of standard test of Nafion 112 membrane at RHC 50%, membrane compression 5% and Nafion Percent 20%, 25%. (a) pressure 5 psig (b) 15 psig (c) 25 psig.

Table (1-2): Extracted data from figure (1-2), the values are: power density, current density and resistance.

| P(psig) | N(%) | MPD (mW.cm⁻²) | i(mA.cm⁻²) at 0.8V | i(mA.cm⁻²) at 0.5V | i(mA.cm⁻²) at 0.3V | R(mΩ) at 0.5V |
|---------|------|---------------|---------------------|--------------------|--------------------|---------------|
| 5       | 25%  | 291           | 60                  | 470                | 860                | 190           |
|         | 20%  | 304           | 50                  | 550                | 890                | 193           |
| 15      | 25%  | 316           | 80                  | 600                | 910                | 209           |
|         | 20%  | 362           | 50                  | 650                | 1100               | 195           |
| 25      | 25%  | 376           | 130                 | 750                | 1080               | 206           |
|         | 20%  | 490           | 28                  | 960                | 1350               | 147           |

2-2- Activation test of MEA

The start procedure for a new fuel cell membrane electrode assembly MEA may vary somewhat from application to application. What is important in any research or production environment is to be consistent with break in procedure that can be used. How the MEA is initially
broken-in can have long lasting effects on the ultimate performance of the MEA. Published procedures vary in specifics, but almost all follow a similar sequence:

1. Initial Start-Up
2. Load Cycling
3. Final Performance

The US Fuel Cell Council (USFCC) [7] published a standard for single cell testing that includes specific break-in procedures:

- Fuel: Hydrogen, 1.2 Stoich, 100% RH
- Oxidant: Air, 2.0 Stoich, 100% RH
- Temperature (C): 80°
- Pressures (psig): 25

Initial Startup: As required to reach 80°C

1. Cycle Step 1 (Perform Once): Hold 0.6 V for 60 mins
2. Cycling Step 2 (Perform 9 times): Hold 0.7 V for 20 mins, than hold 0.5 V for 20 mins
3. Constant Current Operation: Hold at 200 mA.cm⁻² for 720 mins (12 hrs)

Verify break-in status by repeating the polarization curve sequence three times, or as necessary, to ensure that the cell is broken-in. Remain at each sequence step for 20 minutes. The cell is considered broken-in when less than a 5 mV deviation from the previous polarization curve is recorded at 800 mA.cm⁻². A wait period of 10 minutes should be observed between polarization curves. During this period, return the gas flow rates to the equivalent of 10 stoich at 200 mA.cm⁻² and set the current to 800 mA.cm⁻².

Four analysis were done in activation tests of MEA; Cycling Potential between 0.2 and 0.7V, Constant Current 0.25Acm⁻², Constant Voltage 0.6V and standard test protocol. In all of them, specific MEA structure and activation procedure were applied and operation condition before and after activation procedure are similar. The membrane is Nafion 112, flow rate of H₂/O₂ measured 200/200 ml.min⁻¹, pressure at operation condition is 5 psig and temperature of anode, cathode and
cell is 60, 50 and 55°C controlled during all of the tests. After the activation procedure, operation condition in several relative humidity (30%, 50% and 100%), various pressure and flow rate (5 psig flow H₂/O₂ 200/200 ml.min⁻¹, 15 psig flow H₂/O₂ 300/300 and 25 psig flow H₂/O₂ 500/500) was determined. Also, temperature of anode, cathode and cell for RH 30% (T_a/T_c/T_cell: 80/49/75 °C), RH 50% (T_a/T_c/T_cell: 80/59/75 °C) and RH 100% (T_a/T_c/T_cell: 80/75/75 °C) were measured. On the other hand, each activation set contains repetition of the items of activation procedure. In other words, numbers of set means number of repetition.

2-2-1-Activation test MEA at cycling potential between 0.2 and 0.7 V
2-2-1-1-Experimental design, method and details

MEA structure in this analysis including anode components such as CP (TGP-0120), Pt/C 20%, 30% Nafion, 1.98 mg DL.cm⁻² (C28) with 0.38 mg.cm⁻² catalyst loading. Cathode composition is CP (TGP-0120), Pt/C 20%, 30% Nafion and 1.98 mg DL.cm⁻² (C28) with 0.38 mg.cm⁻² Catalyst loading. The membrane is Nafion 112. To start activation procedure, 10 minutes Open Circuit Voltage (OCV) time and then, one minute 0.7 V, 10 minutes 0.2 V (for five times) was applied. Finally, steps 1 and 2 repeated for three times.

In this analysis, the reported data are related to polarization curves after 1 hour during activation procedure, impedance curves at different voltage 0.3, 0.5, 0.7 and 0.8V for set 1, set 2 and set 3, at 60/50/55°C, 5 psig flow rate H₂/O₂: 200/200 ml.min⁻¹, polarization curves at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15 and 25 psig, impedance curves in various voltages at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15, and 25 psig in flow rate H₂/O₂: 200/200 ml.min⁻¹, polarization curves 0.7 V at the end of activation procedure in different cathode humidity condition 30, 50 and 100% at pressure at 5, 15, and 25 and impedance curves in various voltages at the end of activation procedure in different cathode humidity condition 30, 50 and 100% at pressure at 5, 15, and 25. Figures (2-2) and (3-2) represent polarization curves in three sets of activation procedure after 1 hour and impedance curves at different voltages for each sets. Table (2-2) shows the extracted data from related polarization curve.
Table (2-2) extracted data from polarization curve during activation procedure in three sets.

| Sets | OCV (mV) | MPD (mW.cm$^{-2}$) | R (mΩ) |
|------|----------|---------------------|--------|
| st 1 | 991      | 442                 | 70     |
| st 2 | 1003     | 453                 | 72     |
| st 3 | 1014     | 547                 | 70     |

Figure (2-2) Polarization curves during activation procedure (Polarization curves at end of each activation set in three sets).

Figure (3-2) impedance curves at different voltage (1) set 1, (2) set 2, (3) set 3, 60/50/55°C, 5 psig flow rate \( \text{H}_2/\text{O}_2: 200/200 \text{ ml.min}^{-1} \).
2-2-2 Activation test MEA at constant current 0.25 A.cm⁻²

2-2-2-1 Experimental design, method and details

MEA structure at constant current 0.25 A.cm⁻² containing CP (TGP-0120), Pt/C 20%, 30% Nafion, 1.98 mg DL.cm⁻² (C28) with 0.38 mg.cm⁻² of catalyst loading as anode composition with Nafion 112 membrane. Cathode components are containing CP (TGP-0120), Pt/C 20%, 30% Nafion and 1.98 mg DL.cm⁻² (C28) with 0.38 mg.cm⁻² of catalyst loading. 10 minutes OCV time as first step in activation procedure. Then, 30 minutes constant voltage 0.6 V and constant current 0.25A.cm⁻² (repeat for 18 time) was applied.

Obtained data from MEA activation test at constant current are summarized to polarization curves at end of each activation set in 17 sets, impedance curves at the end of each activation set at various voltages 0.3, 0.5, 0.7 and 0.8V at 60 /50/ 55°C, 5 psig flow rate H₂/O₂:200/200 ml.min⁻¹, polarization curves at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15 and 25 psig, impedance curves in various voltages at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15 and 25 psig and flow rate H₂/O₂: 200/200 ml.min⁻¹, polarization curves at the end of activation procedure in different cathode humidity condition 30, 50 and 100% at pressure at 5, 15 and 25 psig, impedance curves in different voltages at the end of activation procedure in different cathode humidity condition 30 a 50 and 100% at pressure at 5, 15 and 25 psig. In table (3-2) extracted data form polarization curves of 17 sets at the end of activation set provided. Figure (4-2) represents polarization curves of 17 sets at the end of activation set. In this figure, just power density was presented because the graph with two Y axis for top number of curves lead to a visualization problem called Chartjunk [8].

Table (3-2) extracted data form Polarization curves at end of each activation set in 17 sets.

| Sets | OCV(mV) | MPD(mW.cm⁻²) | R(mΩ) |
|------|--------|--------------|------|
| st 1 | 968    | 215          | 142  |
| st 2 | 979    | 345          | 122  |
| st 3 | 972    | 380          | 116  |
| st 4 | 990    | 460          | 102  |
| st 5 | 985    | 508          | 99   |
| st 6 | 977    | 529          | 94   |
| st 7 | 978    | 540          | 93   |
| st 8 | 982    | 594          | 92   |
| st 9 | 975    | 605          | 89   |
| st 10| 975    | 600          | 89   |
2-2-3 Activation test MEA at Constant Voltage 0.6 V
2-2-3-1 Experimental design, method and details

Composition of anode including CP (TGP-0120), Pt/C 20%, 30% Nafion, 1.98 mg DL cm$^{-2}$ (C28) with 0.38 mg.cm$^{-2}$ of catalyst loading. The Membrane is Nafion 112 and composition of Cathode is CP (TGP-0120), Pt/C 20%, 30% Nafion, and 1.98 mg DL.cm$^{-2}$ (C28) with 0.38 mg.cm$^{-2}$ of catalyst loading. In activation procedure of this analysis, a constant voltage 0.6 V has been applied.

Activation test of MEA at constant voltage 0.6 V was analyzed with repeat of activation sets. In this analysis, Polarization curves at end of each activation set in 9 sets, impedance curves at the end of each activation for 0.3, 0.5, 0.7 and 0.8 V at 60 /50/ 55°C, in 5 psig flow rate H2/O2:
200/200 \textit{ml.min}^{-1}, polarization curves at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15, and 25 \textit{psig}, impedance curves at various voltages at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15, and 25 \textit{psig}; flow rate H\textsubscript{2}/O\textsubscript{2}: 200/200 \textit{ml.min}^{-1}, polarization curves at the end of activation procedure in different air humidity condition 30, 50 and 100% at pressure at 5, 15, and 25 \textit{psig}, impedance curves at various voltages, at the end of activation procedure in different air humidity condition 30 and 100% at pressure at 5, 15, and 25 \textit{psig} have been done. Table (4-2) represents extracted data form polarization curve at the end of each activation set.

Table (4-2): extracted data from Polarization curves at end of each activation set in 9 sets.

| Sets | OCV (mV) | MPD (mW.cm\textsuperscript{-2}) | R (mΩ) |
|------|----------|-------------------------------|-------|
| st 1 | 965      | 769                           | 75    |
| st 2 | 980      | 925                           | 71    |
| st 3 | 980      | 959                           | 70    |
| st 4 | 980      | 970                           | 71    |
| st 5 | 984      | 967                           | 70    |
| st 6 | 980      | 977                           | 70    |
| st 7 | 983      | 971                           | 70    |
| st 8 | 982      | 1030                          | 66    |
| st 9 | 986      | 979                           | 65    |

Figure (5-2) Polarization curves at end of each activation set in 9 sets.
Activation of MEA at constant voltage 0.6 V repeated in exact MEA structure and operation condition but, after a treatment procedure. In first treatment method, electrodes were ultrasonicated in 10% Isopropyl solution for 60 min at 60°C. In Second treatment method, ultrasonication of electrodes was in water for 60 minutes at 60°C but, this treatment method has some differences in MEA structure, anode composition is A16: CP (TGP-0120), Pt/C 20%, 30% Nafion, and 1.98 mg DL.cm\(^{-2}\) with 0.396 mg .cm\(^{-2}\) catalyst loading. Also, cathode composition is A16: CP (TGP-0120), Pt/C 20%, 30% Nafion, and 1.98 mg DL.cm\(^{-2}\) with 0.4 DL.cm\(^{-2}\) catalyst loading.

Figures (6-2) and (7-2) show polarization curves at the end of activation set in 9 sets for ultra-sonication in Isopropyl solution and water, respectively. Tables (5-2) and (6-2) represents extracted data from polarization curves of figures (6-2) and (7-2), in order.

Table (5-2) extracted data Polarization curves at end of each activation set in 9 sets for ultra-sonication in Isopropyl solution

| Sets | OCV(mV) | MPD(mW.cm\(^{-2}\)) | R(mΩ) |
|------|---------|----------------------|-------|
| st 1 | 973     | 337                  | 122   |
| st 2 | 989     | 377                  | 128   |
| st 3 | 988     | 400                  | 124   |
| st 4 | 987     | 417                  | 121   |
| st 5 | 986     | 538                  | 98    |
| st 6 | 982     | 558                  | 98    |
| st 7 | 981     | 557                  | 95    |
| st 8 | 980     | 542                  | 99    |
| st 9 | 980     | 564                  | 99    |

Figure (6-2) Polarization curves at end of each activation set in 9 sets for ultra-sonication in Isopropyl solution
Table (6-2) extracted data from Polarization curves at end of each activation set in 9 sets for ultra-sonication in water

| Sets | OCV(mV) | MPD(mW.cm$^{-2}$) | R(mΩ) |
|------|---------|------------------|-------|
| st 1 | 960     | 330              | 98    |
| st 2 | 975     | 462              | 92    |
| st 3 | 978     | 513              | 91    |
| st 4 | 980     | 536              | 92    |
| st 5 | 980     | 618              | 87    |
| st 6 | 968     | 651              | 86    |
| st 7 | 977     | 665              | 85    |
| st 8 | 977     | 669              | 86    |
| st 9 | 980     | 672              | 87    |

Figure (7-2) Polarization curves at end of each activation set in 9 sets for ultra-sonication in water.

In final step of activation test at constant voltage 0.6 V, analysis was done at different MEA structure without treatment procedure. Anode components are C39: CP (TGP-0120), Pt/C 20%, 30% Nafion with 0.42 mg.cm$^2$ catalyst loading. Cathode composition is A16: CP (TGP-0120), Pt/C 20%, 30% Nafion with 0.396 mg.cm$^2$ catalyst loading. Table (7-2) and figure (8-2) are related to polarization curve at the end of activation set in 9 sets.
Table (7-2) extracted data from Polarization curves at end of each activation set in 9 sets.

| Sets | OCV(mV) | MPD(mW.cm⁻²) | R(mΩ) |
|------|---------|--------------|-------|
| st 1 | 942     | 462          | 82    |
| st 2 | 978     | 648          | 76    |
| st 3 | 975     | 714          | 77    |
| st 4 | 983     | 900          | 72    |
| st 5 | 980     | 929          | 72    |
| st 6 | 982     | 930          | 74    |
| st 7 | 982     | 924          | 74    |
| st 8 | 985     | 1014         | 72    |
| st 9 | 980     | 993          | 77    |

Figure (8-2) Polarization curves at end of each activation set in 9 sets.
2-2-4-Activation test MEA: standard test protocol
2-2-4-1-Experimental design, method and details

MEA structure include anode composition such as CP (TGP-0120), Pt/C 20%, 30% Nafion and 1.98 mg DL.cm$^{-2}$ (C29) with 0.377 mg.cm$^{-2}$ catalyst loading. Cathode components are CP (TGP-0120), Pt/C 20%, 30% Nafion and 1.98 mg DL.cm$^{-2}$ (C29) with 0.377 mg.cm$^{-2}$ catalyst loading. In activation procedure, 10 minutes OCV time and then, 60 minutes constant voltage 0.6 V was applied. In next steps, 14 minutes cycling potential between 0.7-0.5 V repeated for 10 times and a constant current 0.2 A.cm$^{-2}$ for 18 hours was applied. Table (8-2) and figure (9-2) presents polarization curves at end of activation set in 24 sets. In the mentioned figure, just power density was presented for Chartjunk problem.

In standard test protocol of MEA activation, reported data related to polarization curves at end of each activation set in 24 sets, impedance curves at the end of each activation set 0.3, 0.5, 0.7 and 0.8 V at 60 /50/ 55°C with 5psig flow rate H$_2$/O$_2$: 200/200 ml.min$^{-1}$, polarization curves at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15 and 25 psig, impedance curves in various voltages at the end of activation procedure in humidity condition 30, 50, 100% at 5, 15 and 25 psig flow rate H$_2$/O$_2$: 200/200 ml.min$^{-1}$, polarization curves at the end of activation procedure in different air humidity condition 30, 50 and 100% at pressure at 5, 15 and 25 psig, impedance curves in various voltages at the end of activation procedure in different cathode humidity condition 30 a 50 and 100% at pressure at 5, 15 and 25 psig.

Table (8-2) Extracted data from Polarization curves at end of each activation set in 24 sets.

| Sets | OCV(mV) | MPD(mW.cm$^{-2}$) | R(mΩ) |
|------|---------|------------------|-------|
| st 1 | 961     | 375              | 103   |
| st 2 | 963     | 460              | 98    |
| st 3 | 970     | 505              | 89    |
| st 4 | 977     | 617              | 85    |
| st 5 | 975     | 645              | 83    |
| st 6 | 974     | 650              | 82    |
| st 7 | 975     | 650              | 80    |
| st 8 | 982     | 702              | 81    |
| st 9 | 979     | 703              | 79    |
| st 10| 979     | 709              | 79    |
| st 11| 979     | 714              | 79    |
| st 12| 987     | 758              | 77    |
| st 13| 977     | 740              | 77    |
Figure (9-2) Polarization curves at end of each activation set in 24 sets.

Standard test protocol of MEA activation repeated with a difference in 0.38 mg.cm$^{-2}$ catalyst loading in anode and cathode composition as MEA structure (other components of anode and cathode are similar). In activation procedure, 10 minutes OCV time and then, 0.6 V constant voltage for 60 minutes applied. In next steps, 14 minutes cycling potential between 0.7-0.5 V repeated for 12 times and a constant current 0.2 A.cm$^{-2}$ for 12 hours was applied. Obtained data are the same as before protocol, exactly. Table (9-2) and figure (10-2) presents polarization curves at end of activation procedure in 19 sets. In that figure, there is only power density because, presentation of voltage lead to Chartjunk problem.
Table (9-2) Extracted data from Polarization curves at end of each activation set in 19 sets.

| Sets  | OCV(mV) | MPD(mW.cm⁻²) | R(mΩ) |
|-------|---------|--------------|-------|
| st 1  | 976     | 771          | 79    |
| st 2  | 983     | 887          | 76    |
| st 3  | 981     | 879          | 77    |
| st 4  | 991     | 542          | 68    |
| st 5  | 995     | 945          | 77    |
| st 6  | 995     | 952          | 78    |
| st 7  | 994     | 943          | 78    |
| st 8  | 997     | 930          | 78    |
| st 9  | 992     | 990          | 81    |
| st 10 | 996     | 963          | 77    |
| st 11 | 997     | 962          | 77    |
| st 12 | 994     | 954          | 77    |
| st 13 | 993     | 954          | 77    |
| st 14 | 992     | 904          | 93    |
| st 15 | 998     | 1022         | 76    |
| st 16 | 999     | 1025         | 75    |
| st 17 | 999     | 1019         | 74    |
| st 18 | 999     | 1010         | 75    |
| st 19 | 998     | 1011         | 75    |

Figure (10-2) Polarization curves at end of each activation set in 19 sets.
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