Transport Properties and Performance of Polymer Electrolyte Membranes for the Hybrid Sulfur Electrolyzer

John A. Staser, a,b* Kirsten Norman, b Cy H. Fujimoto, b Michael A. Hickner, c,** and John W. Weidner, a,**z

a Center for Electrochemical Engineering, Department of Chemical Engineering, University of South Carolina, Columbia, South Carolina 29208, USA
b Sandia National Laboratories, Chemical and Biological Systems Department, Albuquerque, New Mexico 87123, USA
c Department of Materials Science and Engineering, The Pennsylvania State University, University Park, Pennsylvania 16802, USA

The water transport and SO2 crossover in the hybrid sulfur cycle electrolyzer were quantified for a poly(phenylene)-based proton exchange membrane and compared to the performance of industry-standard Nafion membranes. While Nafion exhibits good performance, there exists the possibility of a significant SO2 crossover, which can modify the electrode composition, consume current that should be used for hydrogen production, reduce SO2 to the hydrogen stream, and result in a loss of sulfur from the system. Recent research has focused on poly(phenylene)-based membranes that have exhibited high current density with good stability (both chemical and temperature) while limiting SO2 crossover. In this paper, we extend our previous water-transport-modeling work on Nafion membranes to this polymer electrolyte and directly compare the two in terms of electrolyzer performance and SO2 crossover. We show the ability of poly(phenylene) membranes to operate at elevated temperatures with improved performance over lower temperatures; the high temperature performance exceeds that of Nafion membranes.

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The hybrid sulfur thermochemical cycle has been investigated as a viable means to produce hydrogen efficiently on a large scale.1 This process has the advantage over traditional (i.e., coal gasification) methods for producing hydrogen in that when coupled with advanced nuclear reactors, it does not consume fossil fuels or produce CO2 while producing highly pure hydrogen.2,3 Gaseous SO2 fed to the anode compartment electrochemically reacts on a platinum catalyst with liquid water that crosses the membrane from the cathode due to a concentration gradient and pressure differential to form H2SO4 via the following reaction4

\[
\text{SO}_2 + 2\text{H}_2\text{O} \rightarrow \text{H}_2\text{SO}_4 + 2\text{H}^+ + 2e^- \quad E^0 = 0.158 \text{ V vs SHE} \tag{1}
\]

(standard hydrogen electrode).

The H+ ions produced in Reaction 1 migrate to the cathode, where they are reduced on platinum to H2

\[
2\text{H}^+ + 2e^- \rightarrow \text{H}_2 \quad E^0 = 0 \text{ V vs SHE} \tag{2}
\]

We have demonstrated a successful operation of this electrolyzer using Nafion membranes of different thicknesses (2, 5, and 7 mil) over a range of operating conditions (temperature, pressure differential, and current density).5-10 These investigations5-10 showed that SO2 crossover can result in the parasitic reduction to sulfur at the cathode. This crossover phenomenon can have detrimental effects on electrolyzer performance with the consumption of current, which reduces efficiency.

\[
\text{SO}_2 + 4e^- \rightarrow \text{S} + \text{O}_2 \tag{3}
\]

Increased water transport to the anode results in lower SO2 crossover to the cathode.4 However, increased water transport also results in more dilute sulfuric acid, which affects the overall process efficiency.1 Therefore, a membrane is needed to reduce SO2 crossover for a fixed water flux and cell voltage.

A series of alternative proton exchange membranes has been developed for fuel cell applications.11-12 These highly stable membranes, based on sulfonated Diels–Alder poly(phenylene) (SDAPP), were developed for use in direct methanol fuel cells to reduce methanol crossover in comparison to Nafion membranes.13 SDAPP membranes have an additional advantage over Nafion in that they are capable of operating at higher temperatures due to their relatively stiff polymer backbone and superior mechanical properties.10 The one major flaw of these materials, as in many other non-perfluorosulfonic acid proton exchange membranes (PEMs), is that their conductivity suffers greatly at low membrane water content. Because the SO2 electrolyzer in this work is fed with a liquid water stream at the cathode, there are no significant conductivity concerns. Moreover, the presence of liquid water at the cathode (under pressure when the reactor is run at temperatures above 100°C) presents a problem for Nafion in that the mechanical properties of the membrane are compromised to a greater extent when in contact with a liquid phase due to water plasticization.13

In this paper, we examine the SDAPP membrane with an ion-exchange capacity of 2.2 meq/g (SDAPP4)12 to determine its performance in the SO2 electrolyzer and to measure this membrane’s ability to reduce the SO2 crossover. We apply the previously developed water-transport model and compare the SO2 crossover in the SDAPP4 membrane to that in the N115 membrane both at 80°C and at elevated temperatures up to 120°C.

Experimental

The experimental setup was the same as that described previously.4 The cell was a standard 10 cm2 cell from Fuel Cell Technologies, Inc. The reactants and products were injected to the cell through Kynar manifolds instead of the aluminum end plates. The flow fields were the standard graphite variety used for fuel cells, which was made possible due to the low cell potentials used in this work. The Kynar/graphite assembly was sandwiched between aluminum end plates to provide compression, and the temperature was maintained by the use of heating rods inserted into the aluminum end plates.

Liquid water was fed to the cathode by a metering pump, and gaseous SO2 was fed to the anode. The cell was maintained at 80°C, and the water was heated to 88°C before being fed to the cathode. The N115 membrane electrode assembly (MEA) was purchased from Lynntech and had 1.5 mg/cm2 Pt black on the anode and cathode. The SDAPP MEA was prepared in acid form as described previously,12 and Nafion-based electrodes with a platinum loading of 1.5 mg/cm2 Pt black were hand-painted directly onto the membrane. The cells were run such that the conversion of SO2 was 20%. We have shown previously, however, that conversion and catalyst loading have little effect on the electrolyzer performance.2

* Electrochemical Society Student Member.
** Electrochemical Society Active Member.
*** E-mail: weidner@engr.sc.edu

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A pressure differential was maintained across the membrane by the use of a globe valve back-pressure regulator on the exit stream of the cathode. The gaseous feed stream to the anode was maintained at 101 kPa absolute. When the cell temperature was elevated to 120°C, both sides of the cell were pressurized to at least 300 kPa absolute to prevent water vaporization.

The electrochemical monitoring technique was used to determine the diffusion coefficient and the solubility of SO2 in Nafion. The electrolyzer was operated such that the water side served as the anode and the gas side was the cathode (i.e., polarity opposite to that of normal operation). The membrane pressure differential was ΔP = 0 kPa. N2 was initially sent to the gas side (cathode) and a voltage of 0.31 V was applied. Once the current had decayed to a steady-state value, the gas was switched to SO2. The voltage was high enough that the SO2 crossing the membrane to the water side (anode) was oxidized. The small current achieved was the SO2 crossover current. These values were then used to calculate the flux of SO2 as a function of the water flux by varying the current and membrane pressure differential.

The water uptake of the Nafion and SDAPP membranes was measured by immersing the membranes in varying concentrations of sulfuric acid. The membrane dry weight was compared to that measured after the membranes were equilibrated with the sulfuric acid solution. The weight change was taken to be due to water uptake by the membrane.

Model Development

The water-transport model was described in our previous papers. The governing equation for the water flux through the membrane is

$$N_w = \frac{P_m}{M_w \delta_m} \int_{\Delta f}^{\Delta f} D_w \delta m - \frac{\xi \delta_m i_{H_2SO_4}}{\lambda F} + \frac{P_m}{\delta_m} (P_c - P_a)$$  \[4\]

The parameters in Eq. 4 were determined in the same manner as our previous paper (the first term on the right is the water flux due to diffusion, the second term on the right is that due to the electro-osmotic drag, and the third term is that due to the pressure differential), and the equation is solved for the water flux $N_w$ once the integration limits for the diffusion term are known. The integration limits are the water content of the membrane at the anode and cathode, respectively. The cathode water content is fixed, but the anode water content is a function of the water activity at the anode. The functional form of $D_w$ was the same as for Nafion, except that the pre-exponential factors were slightly modified

$$D_w = 2.2 \times 10^{-3} \lambda (1 + e^{-0.28x}) \exp \left[ -\frac{2436}{T} \right] \quad \text{for } 0 < \lambda \leq 3$$  \[5a\]

$$D_w = 8.7 \times 10^{-4} \lambda (1 + 161 e^{-x}) \exp \left[ -\frac{2436}{T} \right] \quad \text{for } 3 < \lambda \leq 17$$  \[5b\]

The pre-exponential factors are a weak function of temperature, and the same values were used at each temperature in this paper. For lack of contrary evidence, the electro-osmotic drag coefficient, $\xi$, remained the same at 2.5. The membrane permeability was $P_m = 9.0 \times 10^{-11}$ mol/(cm² s kPa), and was determined by the slope of the water flux at zero current (open circuit) with respect to the membrane pressure differential, $\Delta P$. This method is identical to that outlined in our previous paper, but the permeability value for SDAPP is 8% lower than that of Nafion [1.1 × 10^{-10} mol /cm² s kPa]. The water uptake expression used in our previous paper was valid for operation at 30°C and was obtained by changing the humidity of gas in contact with the membrane. To refine the model, we performed a similar experiment at 80°C by equilibrating the membrane with varying concentrations of sulfuric acid at 80°C was the same as that of Nafion (less than 5% difference)

$$\lambda_a = 123.8y_a^2 - 224.01y_a + 134.14y_a - 16.35$$  \[6\]

The mole fraction of H2SO4 is determined from a material balance on the anode flow stream, assuming uniform conditions in the flow direction

$$y_{H_2SO_4} = \frac{i_{H_2SO_4}}{i_F} \left( N_w - \frac{i_{H_2SO_4}}{2F} \right)$$  \[7\]

where $y_{H_2SO_4}$ is the mole fraction of H2SO4. The water consumption rate is subtracted from the water flux (second term in the denominator) and reflects the water required for Reaction 1. The mole fraction is converted to moles of H2SO4 per liter of solution using tabulated data for sulfuric acid density (solution of H2SO4 and water).

In this paper, “sulfuric acid” will refer to the solution of H2SO4 and water. Therefore, the sulfuric acid production rate is the sum of the water balance at the anode and the H2SO4 flux determined by Faraday’s law

$$N_{H_2SO_4} = \left( N_w - \frac{i_{H_2SO_4}}{2F} \right) + \frac{i_{H_2SO_4}}{F}$$  \[8\]

The flux of SO2 through the membrane is given by

$$N_{SO_2} = \frac{C_{SO_2} M_M}{\lambda P_m} N_w - D_{SO_2} \frac{dC_{SO_2}}{dx}$$  \[9\]

Equation 9 can be solved with the boundary conditions $C_{SO_2} = C_{SO_2}^0$ at $x = 0$ (gas anode side) and $C_{SO_2} = 0$ at $x = 5$ (liquid cathode side). The flux of SO2 is a combination of convective transport with the water toward the anode (first term on the right) and diffusional flux to the cathode due to a concentration gradient (second term on the right).

To solve for the convective flux in Eq. 9, the water profile in the membrane must be known. The flux of water through the membrane, $N_{w}$, is solved from Eq. 4. Once the flux is known, Eq. 4 can be rearranged to solve for the λ profile through the membrane [i.e., $\lambda(x)$]. The λ value is the number of moles of water per mole of SO2 for Nafion membranes. The λ profile gives a direct correlation to the water content of the membrane and is needed to solve for the flux of SO2 (Eq. 9).

When the water flux obtained via Eq. 4 is zero (i.e., no pressure differential, open-circuit conditions) Eq. 9 reduces to the following form

$$N_{SO_2} = -D_{SO_2} \frac{dC_{SO_2}}{dx}$$  \[10\]

Equation 10 is Fick’s first law. Combining Eq. 10 with the transient material balance gives Fick’s second law

$$\frac{\partial C_{SO_2}(x,t)}{\partial t} = -D_{SO_2} \frac{\partial^2 C_{SO_2}(x,t)}{\partial x^2}$$  \[11\]

Equation 11 can be solved with the following initial and boundary conditions

$$C_{SO_2} = 0 \quad @ \quad t = 0$$  \[12a\]

$$C_{SO_2} = C_{SO_2}^0 \quad @ \quad x = 0$$  \[12b\]
to render the following equation for the SO₂ crossover current as a function of time

\[ i_{SO_2}(t) = \frac{2F \delta D_{SO_2}C_{SO_2}}{\delta M} \left( 2 \frac{n}{\sqrt{4\pi T}} \sum_{j=0}^{\infty} \exp \left[ -\frac{(2j+1)^2}{4\tau} \right] \right) \]  

[13]

The electrochemical monitoring technique is used to calculate the parameters \( D_{SO_2} \) and \( C_{SO_2} \) via a least-squares fit method at conditions under which Eq. 10 is valid (i.e., \( N_a = 0 \)). The current achieved during the electrochemical monitoring technique is small enough that water flux due to electro-osmotic drag is assumed to be negligible. The SO₂ flux is converted to a crossover current density by the following relationship

\[ i_{SO_2} = 2FN_{SO_2} \]  

[14]

Results and Discussion

The polarization curves for SDAPP4 and N115 at 80 and 120°C and \( \Delta P = 300 \) kPa are shown in Fig. 1. The points are data, and the lines are smooth curve fits to the data. The cell voltage is higher for the SDAPP4 membrane at 80°C, indicating a lower electrolyzer performance than N115 at these conditions. The SDAPP4 membrane exhibits better performance (i.e., a lower operating voltage at a given current density) than N115 when the temperature is raised to 120°C. At 80°C and 0.40 A/cm², the operating voltage for SDAPP4 is 1.01 V, while the operating voltage for N115 is 0.862 V. When the temperature is increased to 120°C, the operating voltage for SDAPP4 at 0.40 A/cm² is 0.75 vs 0.776 V for N115.

We have shown previously that water flux to the anode has a significant effect on electrolyzer performance. The water flux to the anode is shown in Fig. 2 for the same conditions as the polarization curves given in Fig. 1. The points are experimental data, and the lines are model predictions via Eq. 4. The results indicate that SDAPP4 exhibits a higher water flux to the anode at all current densities. For example, at 80°C and 0.20 A/cm², the water flux for SDAPP4 is 1.08 \( \times 10^{-5} \) mol/cm² s, while the water flux for N115 at the same temperature and current density is 0.89 \( \times 10^{-5} \) mol/cm² s. Thus, despite a slightly lower water permeability, as discussed above, SDAPP4 has a higher water flux. This is due to the SDAPP4 membranes being slightly thinner than N115 (4 vs 5 mil).

It is clear in Fig. 2 that the two membranes have similar water transport characteristics. The difference in water uptake as a function of sulfuric acid concentration for Nafion and the SDAPP membranes was less than 5%.

The sulfuric acid production rates of SDAPP4 and N115 at 80 and 120°C and at \( \Delta P = 300 \) kPa are shown in Fig. 3. The points are data, and the lines are model predictions via Eq. 7. As expected, the sulfuric acid production rate increases with current density due to the increased water flux to the anode at higher current density and the increased production rate of H₂SO₄. The sulfuric acid production rate of the SDAPP4 membrane is higher than that of the N115 membrane because SDAPP4 is thinner than N115, as discussed above, while having similar water-transport characteristics (the permeability of SDAPP4 is only slightly lower than Nafion), allowing for increased water transport to the anode at all current densities.

When the temperature is increased to 120°C, the sulfuric acid production rate increases at all current densities. The increase in acid production rate is due to increased water transport to the anode with temperature. For example, the sulfuric acid production rate of SDAPP4 at 80°C and 0.40 A/cm² is \( 1.29 \times 10^{-5} \) mol/cm² s. When the temperature is increased to 120°C, the sulfuric acid production rate increases to \( 1.67 \times 10^{-5} \) mol/cm² s. The increase in the sulfuric acid production rate is a weak function of temperature, as already reported. The membrane pressure differential and membrane thickness have a more pronounced effect.

The analysis of the sulfuric acid production rates leads to a discussion of the resulting acid concentration at the anode. The sulfuric

Figure 1. Polarization curves for SDAPP4 and N115. The points (N115 at 80°C, ○; N115 at 120°C, ●; SDAPP4 at 80°C, ○; SDAPP4 at 120°C, ●) are data, and the lines are smooth curve fits to the data. The membrane pressure differential was \( \Delta P = 300 \) kPa.

Figure 2. Water flux to the anode. The points (N115 at 80°C, ○; N115 at 120°C, ●; SDAPP4 at 80°C, ○; SDAPP4 at 120°C, ●) are data, and the lines are model predictions (Eq. 4). The membrane pressure differential was \( \Delta P = 300 \) kPa.

Figure 3. Sulfuric acid production rates. The points (N115 at 80°C, ○; N115 at 120°C, ●; SDAPP4 at 80°C, ○; SDAPP4 at 120°C, ●) are data, and the lines are model predictions (Eq. 8). The membrane pressure differential was \( \Delta P = 300 \) kPa.
acid concentrations at 80 and 120°C and at $\Delta P = 300$ kPa for SDAPP4 and N115 are shown in Fig. 4. The points are data, and the lines are model predictions. The concentration increases as the current density increases because of the increased production rate of $\text{H}_2\text{SO}_4$ at higher current density. The sulfuric acid concentration of SDAPP4 is lower than that of N115 due to the increased water transport to the anode for the thinner membrane. The sulfuric acid concentration decreases as the temperature increases due to the increased water flux to the anode at elevated temperatures, which serves to dilute the sulfuric acid product. For example, the sulfuric acid concentration of SDAPP4 at 80°C and 0.40 A/cm$^2$ is 6.62 M. When the temperature is raised to 120°C, the sulfuric acid concentration decreases to 5.48 M.

We have shown that the operating voltages can be compared as a function of sulfuric acid concentration. It was determined that for the same membrane operated with a different $\Delta P$, the cell voltage at each $\Delta P$ corresponded to the sulfuric acid concentration at each current density. This analysis showed that the operating voltage was a strong function of the sulfuric acid concentration, which is determined by the water flux to the anode. The water flux is controlled by the current density, temperature, membrane thickness, and pressure differential.

Examining the sulfuric acid concentration data in Fig. 4 once more, one concludes that the concentration for N115 at 120°C is similar to that of SDAPP4 at 80°C. If the membrane resistance for N115 and SDAPP4 were identical, then one would expect the polarization curves to be nearly identical in Fig. 1 for N115 at 120°C and for SDAPP4 at 80°C. This is clearly not the case, however, and the difference is due to the higher MEA resistance when SDAPP4 is used as the membrane. SDAPP has an intrinsically higher conductivity than Nafion in liquid water (200 mS/cm vs Nafion at 180 mS/cm at 80°C) but exhibits a higher MEA resistance due to the increased interfacial resistance of Nafion-based electrodes with a non-Nafion membrane. When the MEA resistance is accounted for and the increase in cell voltage is subtracted (internal resistance (iR) corrected), the curves are nearly identical at the two different temperatures (80 and 120°C), as shown in Fig. 5. The points in Fig. 5 are experimental data, and the line is a fit through all data sets. It is instructive to look at the iR corrected data as this representation gives a clearer picture of the intrinsic performance of the membrane. It is well known and has been documented in Ref. 18 that alternative membranes to Nafion have higher high frequency resistance than is anticipated in unoptimized MEAs. We believe that the variation in conductivity of the membrane with temperature plays a large role in determining the electrolyzer performance vs a large increase in the electrochemical conversion rate, as the temperature is raised.

In addition to better operation at higher temperature, SDAPP4 membranes have received attention for use in the hybrid sulfur electrolyzer due to the possibility of a lower SO$_2$ crossover than Nafion. With the water flux known via Eq. 4, we can use the data obtained from the electrochemical monitoring technique to determine the SO$_2$ crossover current density in the same manner as in our previous paper. The SO$_2$ crossover current density, i$_{SO_2}$, as a function of $\Delta P$ is shown in Fig. 6 at 80°C. As expected, the crossover current decreases as $\Delta P$ increases due to the increased water transport to the anode.

The solubility of SO$_2$ in each PEM is shown in Fig. 7 as a function of temperature, along with the diffusion coefficient values. These results quantify the extent to which the solubility of SO$_2$ decreases as the temperature is increased. The diffusion coefficient increases with temperature. The solubility of SO$_2$ is slightly lower in SDAPP4 than in N115; for example, at 80°C, the solubility of SO$_2$ in SDAPP4 is $1.40 \times 10^{-4}$ mol/cm$^3$, and it is $1.58 \times 10^{-4}$ mol/cm$^3$ for N115. The solubility in both membranes is very close to that of SO$_2$ in water, which is understandable given our knowledge of hydrated membrane electrolytes. However, the diffusion coefficients are different between the two membranes. For example, the diffusion coefficient for N115 at 80°C is $2.86 \times 10^{-6}$ cm$^2$/s, while the diffusion coefficient for SDAPP4 at 80°C is 2.55 cm$^2$/s.

![Figure 4](image-url) **Figure 4.** Sulfuric acid concentration for SDAPP4 and N115. The points (N115 at 80°C, ○; N115 at 120°C, ●; SDAPP4 at 80°C, ○; SDAPP4 at 120°C, ●) are data, and the lines are model predictions. The membrane pressure differential was $\Delta P = 300$ kPa.

![Figure 5](image-url) **Figure 5.** Cell Voltage - $iR$ for SDAPP4 and N115. The points (N115 at 80°C, ○; N115 at 120°C, ●; SDAPP4 at 80°C, ○; SDAPP4 at 120°C, ●) are data, and the line is a fit through the data points. The membrane pressure differential $\Delta P = 300$ kPa.

![Figure 6](image-url) **Figure 6.** SO$_2$ crossover current density as a function of $\Delta P$. The points (N115, ○; SDAPP4, ●) are data, and the lines are the model predictions based on the calculated water flux values. The cell temperature was 80°C.
The model predictions show that water flux can be satisfactorily related to water flux when the electrolyzer is under load. We have, however, shown that SO2 crossover can be satisfactorily related to water flux when the electrolyzer is under load. We have, however, shown that SO2 crossover can be satisfactorily related to water flux when the electrolyzer is under load. The points in Fig. 8 correspond to crossover currents corresponding to the steady-state current measured when SO2 crossing the membrane to the water side is oxidized to H2SO4.

Due to the lower solubility at elevated temperatures and the increased water flux to the anode, the SO2 crossover current density, \( i_{SO2} \), decreases with increasing temperature, as shown in Fig. 8. The crossover current is lower for SDAPP4 at all temperatures reported due to the increased water flux to the anode for the thinner membrane. The points in Fig. 8 correspond to crossover currents corresponding to the steady-state current measured when SO2 crossing the membrane to the water side is oxidized to H2SO4.

In addition to increasing the water flux, and hence the convective exchange capacity of 2.2 meq/g, for use in a hybrid sulfur electrolyzer and compared it to a N115 membrane. The SDAPP4 membrane was investigated as a potential alternative membrane to Nafion because of its improved operation at elevated temperature and its reduced SO2 crossover. We have shown that SDAPP4 does indeed show an improvement in performance (i.e., lower cell voltage) over N115 when operated above 100°C, and exhibits a lower SO2 crossover under the same operating conditions. The lower SO2 crossover could result in decreased sulfur poisoning at the cathode, potentially increasing the lifetime of the electrolyzer.

We investigated an alternative PEM, SDAPP4 with an ion-exchange capacity of 2.2 meq/g, for use in a hybrid sulfur electrolyzer and compared it to a N115 membrane. The SDAPP4 membrane was investigated as a potential alternative membrane to Nafion because of its improved operation at elevated temperature and its reduced SO2 crossover. We have shown that SDAPP4 does indeed show an improvement in performance (i.e., lower cell voltage) over N115 when operated above 100°C, and exhibits a lower SO2 crossover under the same operating conditions. The lower SO2 crossover could result in decreased sulfur poisoning at the cathode, potentially increasing the lifetime of the electrolyzer.

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**List of Symbols**

- \( C_i \): concentration of species \( i \), mol/cm³
- \( D_i \): diffusion coefficient of species \( i \), cm²/s
- \( E^0 \): standard reduction potential
- \( i \): current density, A/cm²
- \( M_i \): molecular weight of species \( i \), g/mol
- \( N_i \): flux of species \( i \), mol/cm² s
- \( P_i \): pressure in region \( i \), kPa
- \( P_m \): membrane permeability, mol/(cm s kPa)
- \( \Delta P \): pressure differential across the membrane (\( P_c - P_m \)), kPa

\[ 10^{-6} \text{ cm}^2/\text{s} \]
Greek

- \( \delta \) thickness of the catalyst coated membrane, cm
- \( \Delta P \) pressure gradient across the membrane (\( P_c - P_a \)), kPa
- \( \lambda \) water content of the membrane, mol H\(_2\)O/mol \( \text{SO}_3^{-}\)
- \( \xi \) electro-osmotic drag coefficient, H\(^+\)/H\(_2\)O
- \( \rho \) density of Nafion, g/cm\(^3\)

Subscripts

- \( a \) anode
- \( c \) cathode
- \( w \) water

References


\[ x \] distance into the membrane, cm
\[ y_i \] mole fraction of species \( i \)