



Testing and Selection of Water Deionization Resin for PEM Regenerative Fuel Cell

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Summary

This test report identifies an ion-exchange resin for water deionization (DI) that meets purification requirements for proton-exchange membrane (PEM) regenerative fuel cell (RFC) operation. Testing performed under NASA's RFC project evaluated high-temperature DI products from three vendors (A, B, and C) and DI cartridges from two additional vendors to determine which products best meet RFC requirements for H₂O purity, high-temperature operation, and operational capacity. DI products were tested in open-loop and closed-loop tests at ambient and high temperatures and data were analyzed. Results found vendor A resin superior in terms of both high DI capacity and H₂O ionic impurity suppression.

Acronyms

DI	deionization
EZ	electrolyzer
FC	fuel cell
ISS	International Space Station
LDPE	low-density polyethylene
OGA	oxygen generation assembly
PEM	proton-exchange membrane
RFC	regenerative fuel cell
RO	reverse osmosis
SS	stainless steel
TDS	total dissolved solids
UFS	United Filtration Systems

Introduction

Water deionization (DI), a critical purification process that removes ionic impurities in water, is closely tied to the development of ion-exchange technology. The synthetic polymer ion-exchange resins widely used in industry, health care, and food and beverage preparation do not meet the requirements for water DI in proton-exchange membrane (PEM) regenerative fuel cells (RFCs). Achieving the pure H₂O requirements for a lunar PEM RFC is a unique challenge due to limited energy availability, a desire for system simplicity, mass limitations, and a general lack of DI polymer resins designed for operations above 60 °C. This report describes testing performed under NASA's RFC project to evaluate high-temperature DI products from three vendors and DI cartridges from two additional vendors to determine which products best meet RFC requirements for H₂O purity, high-temperature operation, and operational capacity.

RFC DI: Process and Challenges

In PEM RFCs, H₂O is the sole fuel cell (FC) reaction product as well as the only reactant for the electrolyzer (EZ) supply (Ref. 1). Contamination of this H₂O can cause damage to the electrochemical cells of both the FC and the EZ. In general, ionic impurities interact with anionic chain sites on the PEM. They partially block the conductive pathway of protons through the membrane and can also cause side reactions on FC and EZ electrodes. Removal of these impurities via H₂O DI is crucial to RFC system health.

In PEM RFCs, the DI operation involves both FC product H₂O and EZ inlet H₂O. The FC product H₂O DI removes acidic chemicals generated during normal FC operation to minimize corrosion of wetted metallic materials. The EZ inlet H₂O DI removes impurities in the feed H₂O that could damage the EZ. The lunar RFC developed by NASA's RFC project operates in a wide temperature range of 4 to 85 °C. To maintain high energy and power efficiency, the DI operation for the RFC should occur at a similar temperature range.

Beyond the cell level, FC product H₂O is collected from the FC, EZ inlet H₂O is fed to the EZ, and coolant H₂O is circulated to the FC and EZ cooling manifolds. There are cumulative voltage gradients across the cells and cooling manifolds of the H₂O flow paths. Ionic impurities increase H₂O conductivity and can induce H₂O electrolysis in the coolant manifolds and between the cells of the FC and the EZ (Ref. 2). If the ionic impurities are not mitigated, shunt currents will lead to parasitic power losses and corrosion of RFC metal surfaces. The FC stack, EZ stack, and cooling loop all require low-conductivity H₂O to avoid shunt currents. Removal of ionic impurities is crucial for proper operation of the RFC.

Temperature Considerations in H₂O DI

H₂O purification is an essential and widely used process because pure H₂O is vital to industrial production, health care, and human daily life. Semiconductor and pharmaceutical industries use H₂O with purity requirements similar to those of the RFC. The industrial H₂O purification process starts with reverse osmosis (RO) to remove the bulk of impurities and follows with multiple ion-exchange processes or DI processes to remove ionic impurities. The industrial H₂O DI process is mostly carried out below 40 °C. At high temperatures, the DI process becomes complicated due to the poor thermal stability of anion-exchange resin. Major DI resin manufacturers focus on offering versatile products for ambient-temperature H₂O DI, primarily due to limited demand for high-temperature DI products (Refs. 3 and 4).

H₂O DI is also used on the International Space Station (ISS) for H₂O electrolysis within the Oxygen Generation Assembly (OGA) to supply O₂ for crew respiration. The OGA operates at the average temperature aboard the ISS, between approximately 18 and 27 °C. Therefore, the ISS OGA also makes use of ambient-temperature H₂O DI technology (Refs. 5 and 6).

H₂O Impurities in RFCs

Impurities in H₂O include nonionic soluble organics, solids, bacteria, and ionic species. In RFCs, the amounts of nonionic soluble organics, solids, and bacteria are very low. Because normal RFC operation does not generate them, typically no mitigation for these impurities is required. However, the presence of ionic species in H₂O can pose a danger to RFC systems.

Because RFC systems operate at elevated temperatures and under pressure, the polymeric materials commonly used in semiconductor applications do not provide sufficient mechanical strength for RFC service. Instead, metallic materials that offer high strength and excellent corrosion resistance are a more appropriate choice for long-term RFC applications. Stainless steel (SS) plumbing is widely used in the RFC assembly. SS is suitable for processing and storage of tap H₂O, but its use with high-purity H₂O is

limited due to the dissolution of ions into H₂O. The interaction between SS and H₂O produces metal oxides on the SS surface and leaches ions into the H₂O, even when the SS surface is passivated. High temperature exacerbates the issue (Refs. 7 to 9).

The measurable iron release rate from SS is well reported for pharmaceutical applications, but SS can still be used in pharmaceutical processing. In semiconductor processing, the purity requirement is more stringent, because even minor leaching from SS equipment ruins semiconductor H₂O. In the RFC assembly, the dissolution of SS, especially at high temperatures, has to be considered and carefully mitigated.

H₂O Purity for PEM EZ

The PEM EZ used in the RFC system has stringent H₂O purity requirements. Ionic impurities from dissolved solids, corrosion byproducts of wetted metallic surfaces (mostly SS), and acids produced from normal FC and EZ operations (Ref. 1) can adversely affect the performance of the EZ. As such, DI of the supply H₂O is required. Acidic chemicals in H₂O corrode wet metal surfaces and leach ions. Pure H₂O can also leach ions from wetted metal surfaces (again, mostly SS). The large and wet surface areas within an RFC system make it prone to ionic contamination, causing various problems for long-term operation. Therefore, H₂O DI must be carried out continuously to control ionic impurities.

Conductivity as Indicator of Purity

An important indicator for ionic impurities in H₂O is electrical conductivity, which is proportional to the summation of individual ionic mobilities multiplied by their respective charges and concentrations. Since ionic mobility increases with temperature, electrical conductivity does as well. At high concentrations, ionic interaction impedes ion motion and reduces ionic mobility. Electrical conductivity fails to keep up linearly with ionic concentrations. For H₂O free from ionic impurities, H⁺ and OH⁻ are the only ionic species. Their ion mobilities and concentrations determine the electrical conductivity of pure H₂O. An equilibrium exists between H⁺ and OH⁻ in H₂O, and the equilibrium constant increases with temperature. High temperature boosts the concentrations and mobilities of H⁺ and OH⁻ to raise electrical conductivity. Due to the temperature dependence of H₂O electrical conductivity, it is standard practice to measure and report the conductivity at 25 °C (Ref. 10). For measurements carried out at other temperatures, present-day H₂O conductivity measurement technology can automatically calibrate, convert, and report results at 25 °C. In this work, H₂O conductivity results are always reported for conductivity at 25 °C.

Any addition of ionic impurity to H₂O increases the respective ionic conductance and the overall electrical conductivity. Pure H₂O possesses a minimum electrical conductivity of 0.055 μS/cm, or the highest resistivity of 18.2 MΩ·cm. Elevation of electrical conductivity correlates to increased ionic conductance and contamination by ionic impurities. Conductivity is widely used as a marker for H₂O purity, and this report follows the protocol. EZ vendors set H₂O purity requirements in terms of conductivity or resistivity. H₂O with resistivity above 7 MΩ·cm is considered sufficiently pure for the EZ.

Standard DI Ion-Exchange Process

The standard technology to remove dilute ionic impurities is to use an ion-exchange process via resin cartridge (or bed). When H₂O is passed through an ion-exchange resin cartridge, the cationic impurities are exchanged for H⁺ and the anionic impurities are exchanged for OH⁻. Excess H⁺ and OH⁻ from this ion exchange neutralize to the equilibrium concentrations. Resins are generally classified as cationic and anionic resins, and specifically as resins for heavy metal, halogenic, silicate, and so forth. Multiple cartridges can be deployed for an H₂O ion-exchange process, based on the H₂O source and the types of

impurity. The selection of an ion-exchange resin cartridge takes into account the minimization of mass, volume, and pressure drop to maximize RFC specific energy and efficiency. A mixed-resin approach, mixing cationic and anionic resins in a single cartridge, is the choice for a RFC because the cartridge removes the ions (cations) leached from the metal surface and the acids (protons and anions) generated from normal FC and EZ operations at the same time. The removal of both cationic and anionic impurities in H₂O via mixed ion-exchange resin is known as H₂O demineralization or, as used in this report, H₂O DI.

DI Products for High-Temperature Applications

The available commercial DI products are resins and cartridges. Resins are active ingredients; cartridges are packages with active ingredients. A cartridge can be fitted into a housing and operates readily, whereas DI resin must be packed into a column for effective ion-exchange operation.

For a liquid-anode-fed EZ in a RFC, the nominal operating temperature is 60 to 80 °C. This becomes the temperature range for H₂O DI operation. It is crucial to ensure that the DI cartridge can purify H₂O to the targeted conductivity within this expected temperature range. Typical commercial DI products fall short. Ordinary commercial DI products are not designed to operate at the nominal RFC working temperature between 60 to 80 °C; consequently, they may not meet RFC H₂O DI requirements.

DuPont has been a trailblazer in ion-exchange polymer technology and remains a major supplier of ion-exchange resin. Whereas most common H₂O DI cartridges are designed for ambient-temperature use at the temperature range of approximately 4 to 38 °C, Dupont's widely used AmberLite™ (DuPont de Nemours, Inc.) MB20 H/OH ion-exchange resin has a better temperature range of approximately 5 to 60 °C. (Ref. 4). However, usage above 60 °C impacts H₂O purity and resin life.

Vendors of elevated-temperature DI products publish limited data regarding operational capacity and achievable H₂O purity. This investigation evaluated DI resins and cartridges from a variety of vendors to determine which DI products best meet RFC H₂O purity requirements. The investigation also measured the operational capacity of DI cartridges. As part of the RFC system, the mass and volume of the DI cartridge are included in those of the RFC system, therefore effective utilization of the DI cartridge capacity will directly improve the specific energy and efficiency of the RFC.

For this effort, three vendors provided products capable of operating above 60 °C (Refs. 11 to 17):

- Vendor A offers a wide range of DI cartridges and specific ion-exchange cartridges. Typically, its products are rated for up to 40 °C, but its RT3 cartridge is designed for high-temperature applications. The RT3 product is rated for continuous 80 °C operation.
- Vendor B has designed a high-temperature resin for sustained operation at 80 °C and intermittent (~1 h) operation up to 93 °C.
- Vendor C offers Thermax DI resin, which has a maximum temperature stability of 80 °C.

Experimental Methods

This section describes the test setup and materials used in testing.

Test Setup

Figure 1 presents diagrams of the H₂O DI testing setup for ambient-temperature testing (Figure 1(a)), high-temperature testing (Figure 1(b)), and closed-loop testing (Figure 1(c)). H₂O is typically pumped with a gear pump from the H₂O source through a heater, the DI housing, and a flowmeter toward the drain, where it can be disposed, collected, or circulated back to the H₂O source.

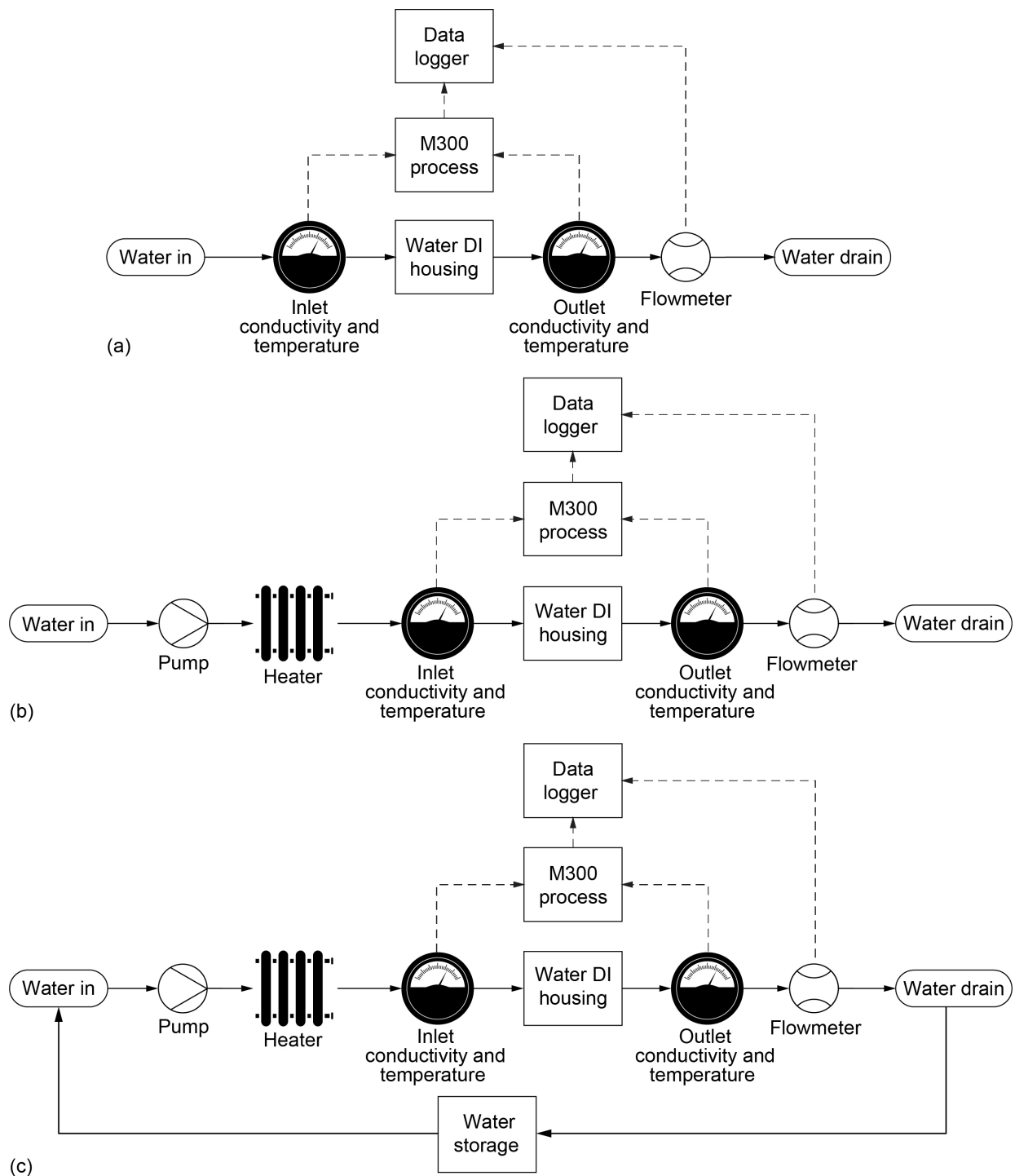


Figure 1. Diagrams for H₂O DI testing. (a) Ambient temperature. (b) High temperature. (c) Closed loop.

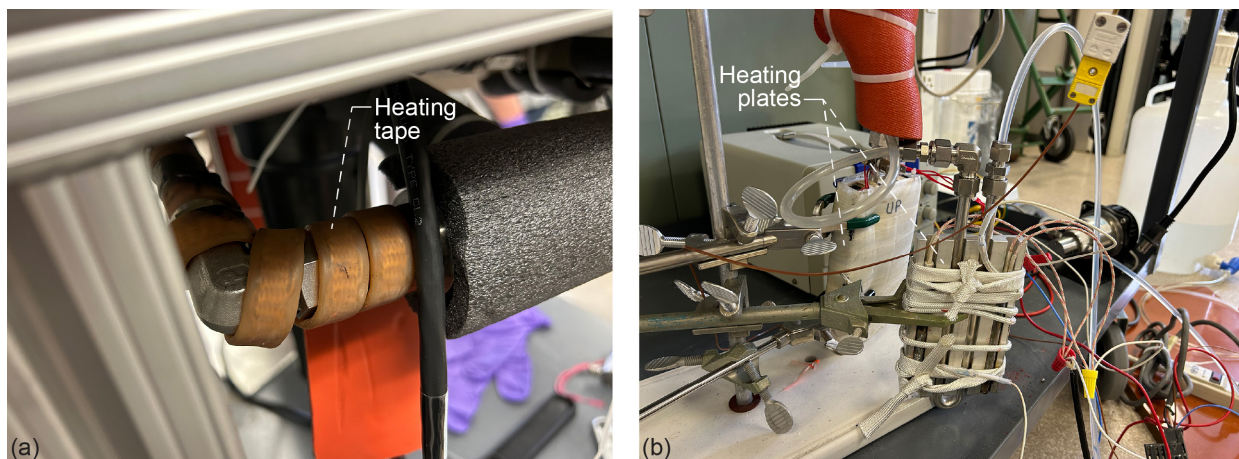


Figure 2. Temperature-controlled testing setup. (a) Heating tapes. (b) Heating plates (red arrows).

For ambient-temperature testing (Figure 1(a)), no pump or heater is needed if tap H_2O is the H_2O source. The initial flowmeter in this research effort was an Omega FLR1006 flow sensor (Ref. 18) with a temperature rating of 0 to 50 °C. The Omega flowmeter failed when the H_2O temperature reached 55 °C and was replaced with a more versatile FTO flowmeter.

For high-temperature testing (Figure 1(b)), a Flow Technology FTO-5NIXW-LHC-1 Omniflo[®] turbine flowmeter was used. Two Mettler Toledo[®] (Mettler-Toledo GMBH) InPro 7005-VP Series 2-electrode conductivity meters were used to measure conductivity and temperature, one on either side of the DI filter. The meters were powered by a Mettler Toledo[®] multi-parameter transmitter M300 process (Ref. 19). The transmitter also converts conductivity and temperature data to analog outputs of 4 to 20 mA. All data were recorded by a Graphtec[®] (Graphtec Corp.) midi logger GL220.

The ambient-temperature testing did not use any type of temperature control. For the higher temperature testing, silicone rubber heating tapes (72 and 144 W) were wrapped around SS tubing, and two lab-made heating plates (300 and 425 W) for H_2O passing through were implemented. Figure 2 shows heating tapes and heating plates placed upstream of the first conductivity meter. The typical heating efficiency, between approximately 50% and 60%, was deemed acceptable for a short-duration test stand.

Materials

For water DI via ion exchange, the DI resin is packed in a filter cartridge that is then fitted into a DI housing. Upon entering the filter housing, H_2O flows through the resin bed for ion exchange.

Resin

Resin can be purchased in prepacked cartridges or in bulk, depending on the application. This series of tests used a combination of prepacked cartridges and bulk resin packed into empty cartridges prior to testing. As noted in the Introduction, three vendors supplied DI resins capable of operating above 60 °C:

- Vendor A: RT3 cartridge rated for continuous 80 °C operation
- Vendor B: High-temperature resin for sustained operation at 80 °C and intermittent (~1 h) operation up to 93 °C
- Vendor C: Thermax DI resin, which has a maximum temperature stability of 80 °C

Filter Housing

The DI filter housing in this investigation was a SpectraPure® (SpectraPure Inc.) FA-HSG-HT-10L-DF high-temperature filter housing made of talc-filled polypropylene, which can be used for both ambient-temperature and high-temperature testing.

Filter Cartridges

The filter cartridges had to be 10 in. long for both ends of the SpectraPure® filter housing to seal properly. From among several commercially available DI cartridge options, three were selected for testing. The following vendors supplied 10-in.-long cartridges:

1. Aries FilterWorks® (ResinTech, Inc.) (Ref. 3), part number AF-10-4010 (prepacked, easily emptied for repacking)
2. SpectraPure®, part number DI-SB-CI-10HC (prepacked, not reusable)
3. United Filtration Systems (UFS) (empty)

The Aries FilterWorks® cartridge and the SpectraPure® cartridge had the same diameter, 3 in. The outer diameter of the UFS cartridge was slightly less at 2.84 in. The UFS cartridge was a hollow cylinder shape with an annular ring cross section, and the other cartridges had a solid cylinder. The UFS cartridge had a removable cap for resin installation and removal. For high-temperature DI resins supplied by vendors B or C, 0.38 kg was packed in the UFS cartridge and 0.62 kg was packed in the AF-10-4010 cartridge.

The prepacked cartridges were fitted into the DI housing and tested directly. The DI resins from vendors B or C were first packed into the UFS cartridge or the empty AF-10-4010 cartridge. The two resins were tested in both types of cartridges. All DI products tested in this report are listed in Table 1.

Ambient-Temperature and High-Temperature Testing

DI resin evaluation can be subclassified into ambient-temperature testing and high-temperature testing. For ambient-temperature testing (Figure 1(a)), there is no heater. Inlet and outlet temperatures are measured along with the conductivities. For high-temperature testing (Figure 1(b)), the heater is on. The heater power and the H₂O flow rate are adjusted so that inlet and outlet temperatures are within the intended range.

DI testing can be further subclassified into single-pass testing and closed-loop testing. H₂O exited from the DI housing is disposed of or collected for the single-pass testing. For fixed-volume closed-loop testing, H₂O exited from the DI housing at the H₂O drain is collected in a storage area (Figure 1(c), bottom) and then circulated back for a repetitive DI process while conductivities data are recorded.

Table 1. DI resins and cartridges evaluated by this investigation

Vendor	Product type	Part number	Cartridge size, in.	Rated temperature, °C
Aries FilterWorks®	Cartridge	AF-10-4010	ø3 × 10	4 to 38 (approx.)
SpectraPure®	Cartridge	DI-SB-CI-10HC	ø3 × 10	Up to 40
Vendor A	Cartridge	DI-MBHT-RT3-10L-20	ø3 × 10	Up to 80
Vendor B	Resin	IC093	ø2.8 × 10 (UFS); ø3 × 10	Sustained 80; intermittent 93
Vendor C	Resin	MB-115 BG	ø2.8 × 10 (UFS); ø3 × 10	Up to 80

Data Processing

Microsoft® Excel® (Microsoft Corp.) spreadsheet software was used to aid in postprocessing of the data collected and plotting of results. Microsoft® Excel® imports the raw data and vectorizes the relevant test parameters, including time stamp, H₂O flow rate, inlet and outlet temperature, and inlet and outlet conductivity, using conversion coefficients to convert the raw signal into the test parameter.

Multiple methods, ranging drastically in degrees of complexity, exist to calculate the quantity of ions in H₂O. One of the more complex methods involves accounting for each species of ionic impurity and the corresponding contribution to overall conductivity measured. To implement this method, either detailed chemical analysis of the source H₂O would be required or a machine-learning algorithm could be implemented to iterate the concentrations of suspected ionic impurities until the resulting predicted conductivity agrees with the measured conductivity.

A simpler approach is to linearly convert the conductivity measurement into an equivalent total dissolved solids (TDS) measurement. TDS is a measure of the total concentration of dissolved matter in water (Ref. 20). TDS can be determined experimentally by evaporating a sample volume and gravimetrically weighing the remaining solid residue. This can be time-consuming and expensive. Also, heating evaporates dissolved carbon dioxide and hydrohalic acids and decomposes bicarbonate, which introduces error beyond measurement error. Various sources have developed conversion factors from TDS to electrical conductivity of the form

$$TDS = EC * f \quad (1)$$

where TDS is total dissolved solids in mg/L, EC is electrical conductivity in $\mu\text{S}/\text{cm}$, and f is the conversion factor. The value of f is impacted by several factors, including temperature, the soluble ionic composition, and concentration, where pH and bicarbonate concentration have significant impacts (Ref. 20, table in appendix).

The lowest reported value of f found in a literature survey was 0.5. This is also the most conservative value, that is, using a higher value for f will result in a higher prediction of TDS removed by the DI filter. For this reason, a conversion factor of 0.5 was used with the expectation to overpredict the mass of DI resin required, as the “true” value of f could lie closer to 0.55.

The inlet (pre-filter) conductivity is used to estimate the TDS entering the filter, whereas the outlet conductivity (post-filter) reading is used to estimate the remaining TDS in the water. The TDS removed per time stamp in the data collected is calculated using Equation (2),

$$TDS_{removed}^i = (TDS_{in}^i - TDS_{out}^i) V^i \quad (2)$$

where $TDS_{removed}$ is in milligrams of TDS, V is volume of water (L) processed through the filter between timesteps, superscript i indicates the i^{th} time-step, subscript in indicates the measurement was taken at the inlet conductivity sensor (pre-filter), and subscript out indicates the measurement was taken at the outlet conductivity sensor (post-filter). $TDS_{removed}$ determines the mass removed at each time step. The flow rate from the mass flowmeter is integrated using the trapezoidal rule numerical method. The mass of TDS removed in each step is summed to determine the cumulative TDS, converted from milligrams to grains (a unit of mass measurement equal to 0.065 grams of TDS) removed during the test.

$$TDS_{removed, total} = \sum TDS_{removed}^i \quad (3)$$

Multiple test days, each covered by an individual data file, are required to deplete resin ion-exchange capacity. The spreadsheet is run for each test, and the calculated TDS removed outputs are summed to determine the total capacity for each test sample (ion-exchange resin cartridge).

Testing and Results

Open-Loop Tests

Tap H₂O was used for open-loop testing due to the unavailability of FC product H₂O or EZ inlet H₂O. Tap H₂O is easily accessible and can be used for DI capacity testing. However, tap H₂O has a much higher concentration of TDS than would be expected from FC product H₂O. To reach the desired purity (7 MΩ·cm) of product H₂O in a single pass through an Aries FilterWorks® AF-10-4010 cartridge, the TDS for inlet water was limited below 200 ppm (Ref. 3). Typical tap H₂O in the United States contains approximately 350 ppm TDS (Ref. 21; see p. 11 for the miscellaneous category of TDS). To reduce the impurities in tap H₂O to reach 7 MΩ·cm resistivity, single-pass DI filtration is insufficient. A pretreatment stage (also known as primary treatment) comprising RO or primary DI to remove some impurities is the first step. Next, another polishing stage comprising a recirculatory loop and using a special-grade resin for high-purity H₂O is required. Because the FC product H₂O and EZ inlet H₂O are quite pure, cartridges with special-grade resin (designed for final polish of ultrapure H₂O) can be used directly. A suitable resin for a RFC would have a high DI capacity, final polishing capability, and high-temperature performance.

Ambient-Temperature Open-Loop Test

For this test, no temperature control is implemented, and H₂O is not recirculated. Ambient temperature is normally within the RFC operation temperature range of approximately 4 to 85 °C, and the measured DI capacity at ambient temperature is a valuable parameter.

The Aries FilterWorks® AF-10-4010 cartridge was tested first. Its DI capacity was determined to be equivalent to 451±17 grains of CaCO₃ among triplicate cartridges, matching the 450-grain capacity reported in the Aries FilterWorks® technical data sheet (Ref. 3). The good calibrations of the flow sensor and conductivity meters make the DI capacity measurement accurate.

A SpectraPure® cartridge rated up to 40 °C and a vendor A cartridge rated to 80 °C were tested next. Their performances were comparable to that of the AF-10-4010 cartridge. Differences in DI running times for the three cartridges were within 5%. DI capacity information was not provided by SpectraPure® or vendor A.

Despite efforts to make test conditions consistent, there were some noticeable variations. Tap H₂O conductivity changed over time per inlet conductivity measurement, which reflected variable amounts of TDS. The inlet H₂O conductivity was in the range of 290 to 320 μS/cm over the test duration of 4 months. The H₂O temperature also fluctuated between 17 and 28 °C. The H₂O flow rate was maintained at 1 L/min ±4%. The variations generated variable running times for the cartridges. For DI capacity measurements that include the variation of flow rate and conductivity (see Eq. (3)), the results are more consistent than those for running time alone.

The vendor B resin was packed into a UFS cartridge initially, but outlet conductivity was rather high (~60 μS/cm, whereas outlet conductivities for the Aries FilterWorks® and SpectraPure® cartridges were below 1 μS/cm). Because the UFS cartridge resin volume is ~60% that of the other cartridges, the linear flow rate of H₂O in the resin bed was higher, reducing contact time and thereby impeding ion-exchange performance. The Aries FilterWorks® cartridge can be easily opened and refilled, so the vendor B resin was subsequently packed into an empty Aries FilterWorks® cartridge.

When using the same filter housing as the previous resin cartridge (Aries FilterWorks® AF-10-4010), the cartridge filled with vendor B resin produced H₂O with a conductivity of 30 µS/cm. When the flow rate was reduced from 1.00 to 0.25 L/min, the conductivity decreased to about 17 µS/cm. The conductivity quickly rose back when the 1-L/min flow rate was resumed. However, the overall DI capacity was comparable to that of the same-sized Aries FilterWorks® cartridge. It was also consistent with the listed capacity in the vendor's technical data sheet (262 mEq for 0.65 lb, Ref. 14).

Testing of the other high-temperature resin (vendor C) revealed comparable performance (within ±10%) to the vendor B resin. The outlet H₂O conductivity was slightly lower, and the capacity was similar.

The test results of the two high-temperature resins at ambient temperature were presented to the respective vendors, who considered the results reasonable. Both suppliers suggest that inlet tap H₂O must be treated so that the outlet H₂O can reach a purity of 7 MΩ·cm resistivity. Figure 3 compares the ambient-temperature single-pass performance of five resins using similar-sized cartridges and under the nominal flow rate of 1 L/min.

The advantage of high TDS in tap H₂O is that DI testing can be completed in a relatively short time, making test conditions easier to manage. Despite the difference in temperature ratings among the three cartridges, the outlet conductivity for all three reached well below 1 µS/cm. This is close to the target of 0.14 µS/cm (or 7 MΩ·cm) for EZ H₂O.

High-Temperature Open-Loop Test

The EZ H₂O flow rate is 1 L/min, and the same flow rate was selected for ambient-temperature DI testing. For high-temperature tests between 60 and 80 °C, heating water at the same 1-L/min flow rate would require 4-kW heating power, assuming 100% efficiency. Because the overall heating power available for this test was about 1 kW and the efficiency was far less than 100%, the flow rate had to be reduced for high-temperature testing between 60 and 80 °C.

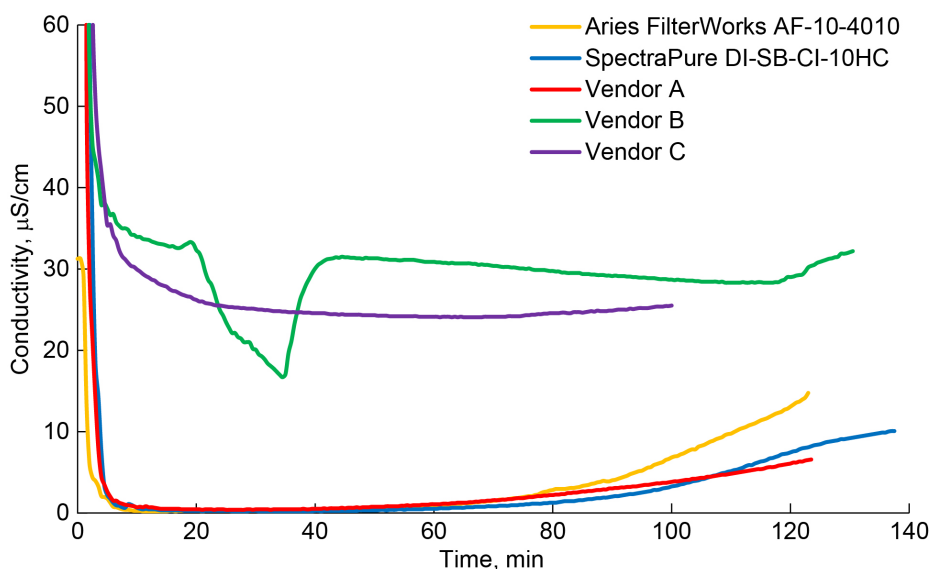


Figure 3. Ambient-temperature single-pass DI testing at nominal flow rate of 1 L/min. For vendor B resin, flow rate was reduced to 0.25 L/min at 19 min and restored at 40 min, which corresponds to a reduction in conductivity.

High-temperature tests were carried out at 60, 70, and 80 °C with the vendor A cartridge, the vendor B resin, and the vendor C resin. The vendor B and C resins were packed in an AF-10-4010 cartridge for testing. Whereas the ambient-temperature test took only ~2 h at a flow rate of 1 L/min, the reduced flow rate and an unexpectedly high DI capacity at high temperature resulted in a much longer testing time. For example, the testing of vendor C resin at 70 °C at a flow rate of 0.25 L/min took a total of 18 h across four separate days. Each test session had a warmup and cooldown period at the startup and shutdown, respectively. These warmups and cooldowns are reflected in the data, which combine each of the test sessions into a single timeline.

In an ion-exchange column, only part of the zone participates in ion exchange; this is known as the mass transfer zone. With a higher ion-exchange rate, the mass transfer zone becomes sharper, and DI capacity becomes higher. A low flow rate also helps to decrease the mass transfer zone and increases the DI capacity.

The test results for vendor B resin at high temperature are comparable to those of vendor C resin. Both show a higher DI capacity between approximately 60 and 80 °C than at ambient temperature. Apparently, high DI capacity at high temperature benefits from a high ion-exchange rate and a narrower mass transfer zone due to reduction in flow rate.

For DI capacities tested at 70 °C, the vendor A cartridge has the highest capacity. More important, the outlet conductivity is lower because the vendor A resin is able to remove ions faster than the vendor B and C resins. The outlet conductivity is below 0.14 $\mu\text{S}/\text{cm}$ for 8 h (meeting the EZ H₂O purity requirement) and remains below 1 $\mu\text{S}/\text{cm}$ for another hour. Figure 4 shows the results of 70 °C testing of three high-temperature DI products under the approximate flow rate of 0.25 L/min. The conductivity spikes in Figure 4 for outlet H₂O from vendor B and C resins are attributable to the low temperature of the cartridge between testing days. After warming up of the cartridge, the conductivity decreases. This is consistent with high DI capacity at high temperature. The measured high-temperature DI capacities are listed in Table 2.

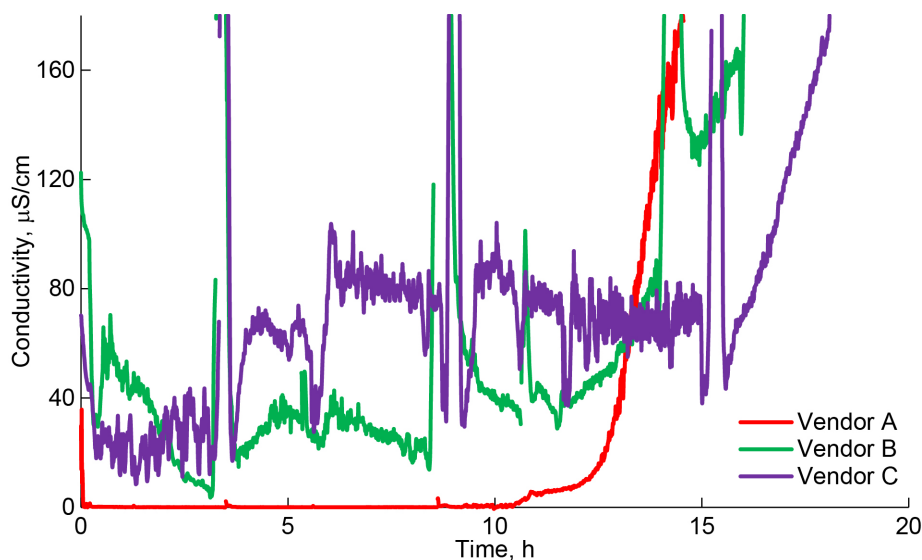


Figure 4. Single-pass DI testing at 70 °C at flow rate of ~0.25 L/min.

Table 2. High-temperature DI capacity
(in grains of CaCO₃) of Aries FilterWorks®
AF-10-4010 size cartridge

Temperature, °C	Vendor A	Vendor B	Vendor C
60	510	382	489
70	528	478	505
80	503	459	451

Closed-Loop Tests

Further testing was performed to determine the lowest conductivity vendor B and C resins can generate for EZ H₂O. A closed-loop test was required due to the high TDS concentration of tap H₂O. Closed-loop testing was not necessary for the vendor A cartridge because it can generate H₂O with conductivity low enough to meet EZ H₂O purity requirements from a single pass of tap H₂O at both ambient temperature and high temperature.

Ambient-Temperature Closed-Loop Test

Ambient-temperature closed-loop testing was carried out with vendor B resin at a flow rate of 1 L/min. For the closed-loop test, 30 L of H₂O was collected into a reservoir after one pass through a DI cartridge. Initially, the conductivity of the tap H₂O was ~300 µS/cm. After one pass through the cartridge containing vendor B resin, the conductivity was reduced to 30 µS/cm. The outlet H₂O conductivity was further reduced to 3 µS/cm with 30-µS/cm H₂O as the inlet source. After 30 min of a continuous DI operation run, outlet H₂O conductivity was reduced to 1 µS/cm. The outlet conductivity reached a minimum of 0.4 µS/cm with 60 min of continuous DI circulation. The ultimate conductivity for the resin was ~1 µS/cm, depending on number of passes of H₂O through the DI bed, the temperature, and the rated normal capacity according to the vendor B resin manufacturer. Pure H₂O with conductivity below 0.5 µS/cm at 80 °C can be maintained with the resin (Ref. 15).

High-Temperature Closed-Loop Test

For high-temperature closed-loop testing, vendor B resin was selected first. To facilitate heating of H₂O storage for high-temperature testing, a SS vessel was used initially. The flow rate was kept at 1 L/min. Due to excessive ion leaching from the large surface area of the brand-new SS vessel at high temperatures (>60 °C), the lowest outlet conductivity in the continuous DI test of 4 h was ~74 µS/cm, much higher than that of the ambient-temperature test. The SS vessel was replaced with a 20-L Nalgene® (Thermo Fisher Scientific, Inc.) low-density polyethylene (LDPE) round carboy. Tap H₂O was used for high-temperature closed-loop DI testing. For high-temperature (>60 °C) testing, the outlet conductivity reached 0.223 µS/cm after 96 min and stabilized. When the closed-loop testing for vendor C resin was carried out at ambient temperature, the conductivity reached 0.132 µS/cm at 1 h and further decreased to 0.12 µS/cm at 75 min. Even with plastic H₂O storage, the SS plumbing in the DI testing assembly still leached ions at high temperature and affected H₂O DI testing.

A series of 10 tests was carried out with both vendor B and vendor C resins in a UFS cartridge. For each test, 7 L of tap H₂O were used. Figure 5 shows that with a new vendor C resin, outlet conductivity reached 0.14 µS/cm sooner at ambient temperature than at high temperature (70 °C) despite high DI capacity for the resin at high temperature. The discrepancy is attributable to ions leaching from the SS plumbing in the DI testing assembly, which is exacerbated at high temperatures. SS tubing was used between the inlet conductivity meter and the DI filter housing as well as between the DI filter housing and

the outlet conductivity meter (see Figure 1). The H₂O reaches very high purity exiting from the DI filter housing, but high-temperature ion leaching from the tubing between the filter housing and outlet conductivity meter may reduce its purity. Additionally, the SS heating plates and the section of SS tubing wrapped with heating tape (see Figure 2) are heated during high-temperature DI testing. Direct heating on SS accelerates ion leaching into H₂O, because the SS with high surface temperatures is in contact with the H₂O. The benefit of high-temperature DI operation is better ion-exchange kinetics; the disadvantage is more ion leaching from SS plumbing. Figure 5 shows the advantage of ambient-temperature DI operation over high-temperature DI operation for vendor C resin. The inlet conductivity profile is similar; however, the ambient-temperature outlet conductivity decreases more rapidly. Similar testing of vendor B resin shows a different trend, as seen in Figure 6. The conductivity of outlet H₂O decreases faster at high temperature than at ambient temperature. Vendor B resin is designed and marketed for high-temperature applications (Ref. 15).

In the ambient-temperature single-pass testing, the outlet conductivity for vendor C resin was lower than that of the vendor B resin (see Figure 3). The difference was rather small. Again, the ambient-temperature closed-loop test results show that outlet conductivities for vendor C resin decreased more rapidly, and to a lower level, than vendor B conductivities. It took less than 1 h for vendor C resin to suppress the tap water conductivity to 0.14 $\mu\text{S}/\text{cm}$, the benchmark for EZ H₂O conductivity. After 2 h, the water conductivity of vendor B resin was still above 0.25 $\mu\text{S}/\text{cm}$ and stabilized (see Figure 7). This demonstrates that vendor C resin can purify H₂O to meet EZ operational requirements, whereas the vendor B resin falls short. The testing outcome for vendor B resin seems consistent with the vendor's report (Ref. 15).

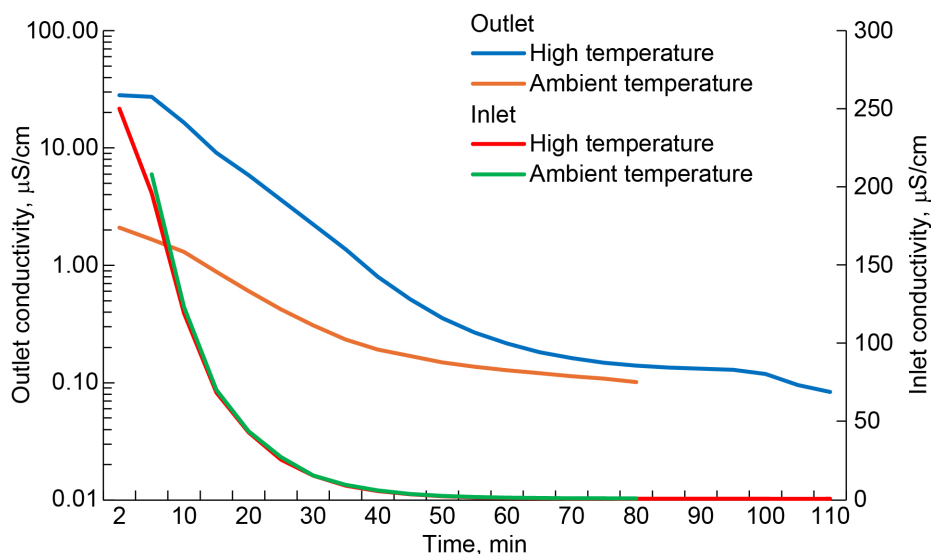


Figure 5. Testing with new vendor C resin in UFS cartridges. For easy comparison, outlet conductivity is in logarithmic scale and inlet conductivity is in linear scale.

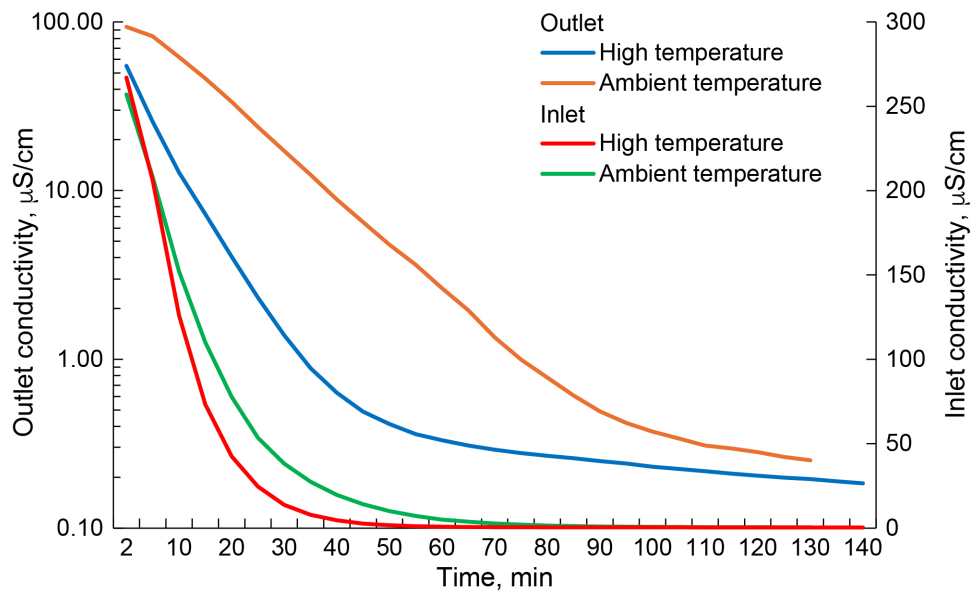


Figure 6. Testing with new vendor B resin in UFS cartridges. Outlet conductivity is in logarithmic scale and inlet conductivity is in linear scale.

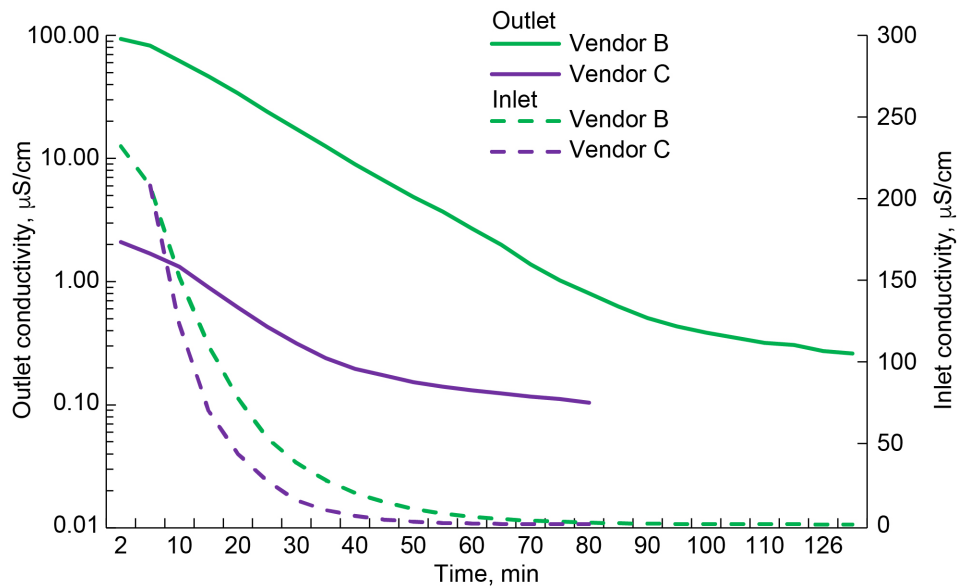


Figure 7. Ambient-temperature testing with new vendor B and new vendor C resins. H_2O conductivities for vendor C resin decrease faster, to a lower level. Outlet conductivity is in logarithmic scale and inlet conductivity is in linear scale.

Concluding Remarks

Summary of Investigation

Existing commercial deionization (DI) products were tested and ranked to select a high-temperature H₂O DI ion-exchange resin that meets the requirements for proton-exchange membrane (PEM) regenerative fuel cell (RFC) operation, specifically for H₂O conductivity below 0.14 $\mu\text{S}/\text{cm}$ for the electrolyzer (EZ) H₂O purity requirement. Testing performed under NASA's RFC project evaluated DI resin products from three vendors, A, B, and C, and cartridges from two additional vendors. Results indicated that the vendor A product performed best in terms of both high DI capacity and suppression of H₂O ionic impurities to the targeted level. The vendor A product meets the PEM RFC operational need, whereas other high-temperature H₂O DI products actually fall short.

Future Work

Future work includes testing the vendor A cartridge in the closed-loop configuration, as well as testing for H₂O with an ionic impurity level and flow rate close to that of PEM RFC H₂O and fuel cell (FC) H₂O, instead of testing tap H₂O at a reduced flow rate. The FC H₂O can be artificial or synthetic instead of being generated by FC operation, which can take significantly longer and demands a better temperature-control system. By using synthetic FC H₂O instead of tap H₂O, a more realistic DI capacity can be measured, and it will be directly applicable to the service time for FC operation.

Remaining Issue and Potential Solution

The rapid laboratory testing of H₂O DI resin and the straightforward conclusions drawn from it are well suited for short-term RFC laboratory evaluations. However, for long-term RFC deployment, relying solely on H₂O DI resin to mitigate ionic impurities may be inadequate. Ionic leaching from stainless steel (SS) components into RFC H₂O remains a challenge, especially at high temperatures. At $\sim 70^\circ\text{C}$, a new SS surface can leach large quantities of ions and disrupt the H₂O DI ion-exchange process. For passivated surfaces, the leaching rate is lower and can be controlled by using ion-exchange resins for PEM RFC operation, but the process does introduce some error or uncertainty in DI capacity measurement. In addition, the consumption of H₂O DI resin in the RFC system makes a large DI cartridge necessary. Application of protective polymeric coatings, such as those used in semiconductor processing, might be beneficial to prevent or reduce leaching in PEM RFC systems and H₂O DI testing systems, but that is beyond the scope of this investigation.

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