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Multifiltration Bed Replacement System for the International Space Station using Aquaporin Membranes and Humidity Condensate Ersatz Wastewater

Hali L. Shaw[1], and Kevin Howard[2]
KBRwyle, Moffett Field, CA, 94035

Michael T. Flynn[3]
NASA Ames Research Center, Moffett Field, CA, 94035

David Beeler[4]
Bionetics, Moffett Field, CA, 94035

Brian Kawashima [5]
NASA Ames Research Center, Moffett Field, CA, 94035

Thomas A. E. Andersen[6]
Aquaporin Space Alliance ApS, Odense, Denmark

Kim Kleinschmidt[7]
Danish Aerospace Company, Odense, Denmark

Jörg Vogel[8]
Aquaporin A/S, Copenhagen, Denmark

and

Jurek Parodi[9]
Science and Technology Corporation, Moffett Field, CA, 94035

The Multifiltration Bed system in the International Space Station (ISS) Water Processor Assembly (WPA) needs to be improved by reducing or eliminating the usage rate of expendable media, removing dimethylsilanediol, and reducing the overall system mass. The Multifiltration Bed Replacement technology is a pressure driven biomimetic membrane based system that is being evaluated to replace the function of the Multifiltration Beds in the ISS WPA. Tests were conducted to determine the performance of the Aquaporin Inside™ Hollow Fiber Module at a 98% water recovery ratio. A long-duration test of the membrane was also conducted to determine membrane life. The feed used for testing was the simulated ISS Marshall Space Flight Center humidity condensate ersatz. All tests operated at ambient temperature and a backpressure of 20 psi. Samples were analyzed for total organic carbon, dimethylsilanediol, acetate, ions, and volatiles including ethanol and acetone. The results

[1] Research Engineer, SCB Branch, Mail Stop 239-15, Moffett Field, CA 94035
[2] Engineer I, SCB Branch, Mail Stop 239-15, Moffett Field, CA 94035
[3] Physical Scientist, SCB Branch, Mail Stop 239-15, Moffett Field, CA 94035
[4] Scientist/Engineer, SCB Branch, Mail Stop 239-15, Moffett Field, CA 94035
[5] Student Volunteer, SCB Branch, Mail Stop 239-15, Moffett Field, CA 94035
[6] CEO M.Sc., E.E., Hvidkærvej 31A, st. 5250 Odense SV, Denmark.
[7] Mechanical Engineer, Hvidkærvej 31A, st. 5250 Odense SV, Denmark.
[8] Head of Technology Development, Ole Maaløes Vej 3, 2200 Copenhagen N, Denmark.
[9] Aerospace Engineer, SCB Branch, Mail Stop 239-15, Moffett Field, CA 94035

indicated that at a 97.6% ± 0.47% water recovery ratio, the membrane can reject approximately 50% of the total organic carbon and conductivity. For long-duration testing, the membrane has shown no degradation for 8711.8 hours and testing is ongoing.

AES = Advanced Exploration Systems
BQL = Below Quantitation Limit
DI = Deionized Water
DGBE = Diethylene Glycol Butyl Ether
DMSD = Dimethylsilanediol
ECLSS = Environmental Control and Life Support System
ESA = European Space Agency
nPA = 1-propanol
IPA = Isopropanol
ISS = International Space Station
JSC = Johnson Space Center
KSC = Kennedy Space Center
MFBR = Multifiltration Bed Replacement
MSFC = Marshall Space Flight Center
PFD = Process Flow Diagram
TOC = Total Organic Carbon
UPA = Urine Processor Assembly
VRA = Volatile Removal Assembly
WPA = Water Processor Assembly
WRR = Water Recovery Ratio

I. Introduction

The International Space Station (ISS) utilizes conventional mechanical technologies including the Water Processor Assembly (WPA).[2] The WPA uses adsorption and ion exchange multifiltration beds along with distillation and catalytic oxidation.[2-5] The two Multifiltration Beds are one of the main contributors to the resupply mass requirement and weigh 110 lbs each (50 kg).[8] These beds do not sufficiently remove trace contaminates such as dimethylsilanediol (DMSD).[2] DMSD is an ISS trace contaminant that was identified in 2010.[2] A replacement for the Multifiltration Beds has been developed, which uses biomimetic aquaporin protein membranes. The aquaporin membranes were commercialized by the company Aquaporin A/S. These membranes would reduce the overall mass and resupply mass of the Multifiltration Beds, and would address the buildup of DMSD.

The objective of the ISS Multifiltration Beds Replacement (MFBR) project is to evaluate the feasibility of developing a pressure driven membrane based system to replace the function of the Multifiltration Beds in the ISS WPA. The aquaporin membrane was tested under conditions that simulate the function of the proposed system at the bench scale (ground testing).[1] Membrane performance was based on the flux and the rejection characteristics of the aquaporin membrane using the Marshall Space Flight Center (MSFC) ISS humidity condensate ersatz as the feed. The objective of this study was to determine membrane performance at a 98% water recovery ratio (WRR) and membrane life.

II. Background

The results from a previous study, conducted in 2016, indicated that the Aquaporin Inside™ Hollow Fiber Modules perform better than Flat Sheet AIM™ 60 (at a 65% water recovery ratio) due to the rejection of contaminants.[1] Therefore, in this study, the 98% water recovery ratio tests and long-duration (membrane life) test were conducted using the Aquaporin Inside™ Hollow Fiber Module. The feed used for testing had a total organic carbon (TOC) concentration of approximately 57 ppm, and conductivity of approximately 170 µS/cm. In order to meet the criteria for success, the membrane needed to achieve a 50% reduction of the TOC and a 50% reduction of the conductivity. Based on the results from the 2016 study, the hollow fiber module and flat sheet membranes both met the TOC and conductivity criteria at a water recovery ratio of 65%.

Subsequently, in fiscal year 2017 the ISS Program Office identified the MFBR project as a method to address Multifiltration Beds logistics and trace contaminant issues. The project was then transferred from the Advanced Exploration Systems (AES) program to the ISS Program Office, and the technology is currently on an accelerated path to flight. Additionally, the European Space Agency (ESA) conducted two flight experiments, one in September 2015 and one in December 2016, to test the Aquaporin membranes on the ISS.[6,7]

III. Experimental Protocol

The water recovery ratio tests and long-duration (membrane life) testing operated with a backpressure of 20 psi and at ambient temperature. The Aquaporin Inside™ Hollow Fiber Module (MHF0032 and MHF0039) was used for all experiments. The hollow fiber module contained 107 fibers and had an active membrane area of 116 cm^2.

The feed used for testing consisted of the MSFC humidity condensate ersatz, which is a proprietary formulation containing 31 chemicals that simulate the wastewater from the WPA.[1] For the MSFC ersatz, the theoretical concentration of DMSD was 22.0 mg/L, the target TOC concentration was approximately 57 ppm, and the target conductivity was approximately 170 µS/cm. The feed was stored in the refrigerator at 4 °C and a new batch of feed was prepared every month.

The water recovery ratio tests were conducted in triplicate; the system was rinsed three times with DI water prior to starting a new test and the same membrane module was reused. The water recovery ratio was limited to 98% due to the 20-mL dead volume in the test system. Each test ended when the feed tank was empty or when air started to pass through the system. The feed volume used for testing was 1050 mL.

Figure 1 shows the process flow diagram (PFD) for conducting the 98% water recovery ratio tests. Figure 2 shows the actual system used for testing. The system operated in a batch mode and the feed recirculated back to the feed tank. When the feed tank was empty, a level switch turned off the pump to prevent the pump from burning out. Product and feed samples were taken at the end of each test.

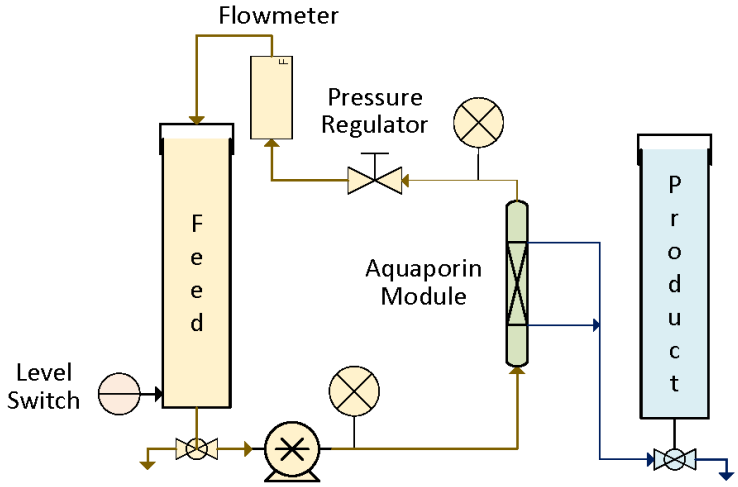

Figure 1. Process flow diagram for system used to conduct 98% water recovery ratio tests.

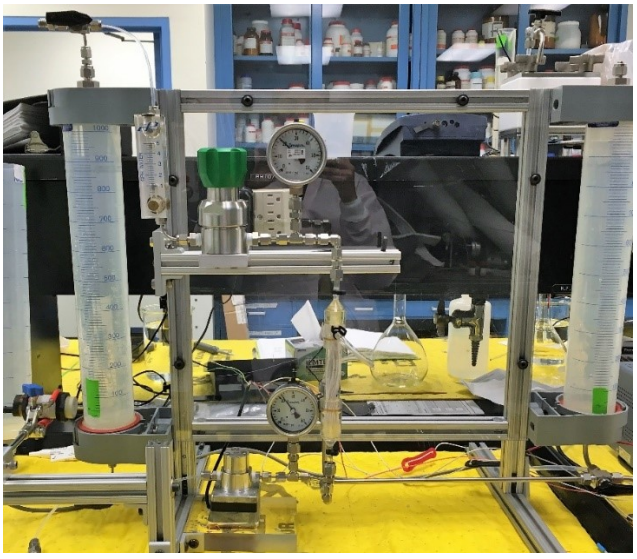

Figure 2. System used to conduct 98% water recovery ratio tests.

For the long-duration testing, the feed was replaced once a week to maintain the TOC concentration and conductivity. The 20-psi backpressure caused the flexible tubing to stretch over time. This tubing was replaced when leaks occurred, or when the pressures displayed on the pressure gauges were unstable. The water recovery ratio at the end of one week was approximately 14 percent due to the removal of samples. The long-duration test is complete when the membrane reaches a point of failure. Membrane failure is based upon the sample analysis results and the flux data.

Figure 3 shows the long-duration testing PFD and Figure 4 shows the actual system used for testing. The feed and product water recirculate back into the feed tank, and 40 mL product water samples were taken three times per week. After 5520 hours of testing, the three 40 mL samples collected per week were combined into one 120 mL sample. The three samples were collected on different days due to the low flux. The purpose of this change was to reduce costs for sample analysis, by reducing the number of samples being analyzed, and to increase the sample volume size for the analyses. Due to a low flux rate, a balance with a graduated cylinder inside was used to determine the flux over time. The feed recirculation flowrate was approximately 1.6 L/h at 20 psi. When a reduction in flowrate was observed on the feed inlet and outlet flowmeters, the backpressure regulator was opened briefly (about 1 minute) to purge any microbial growth that was restricting the flow through the feed stream. During this time, the recirculation flowrate was also increased from about 6 L/h to 30 L/h to assist in purging the microbial growth.

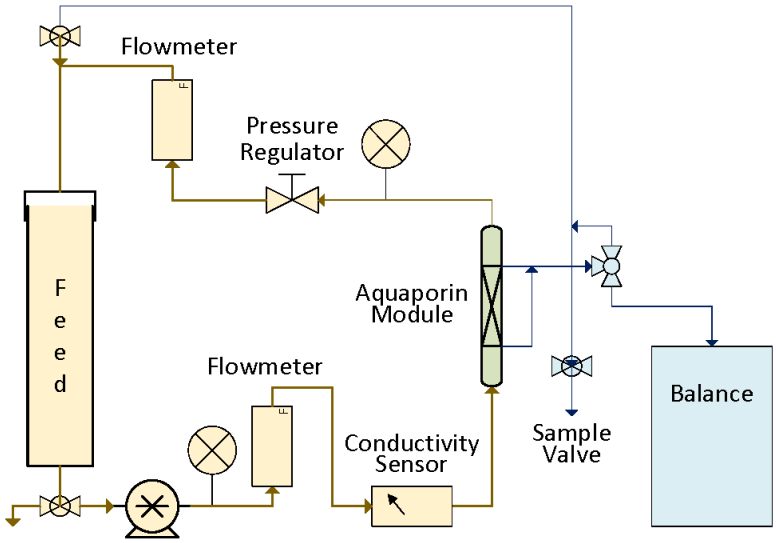

Figure 3. The PFD of the system used for long-duration testing.

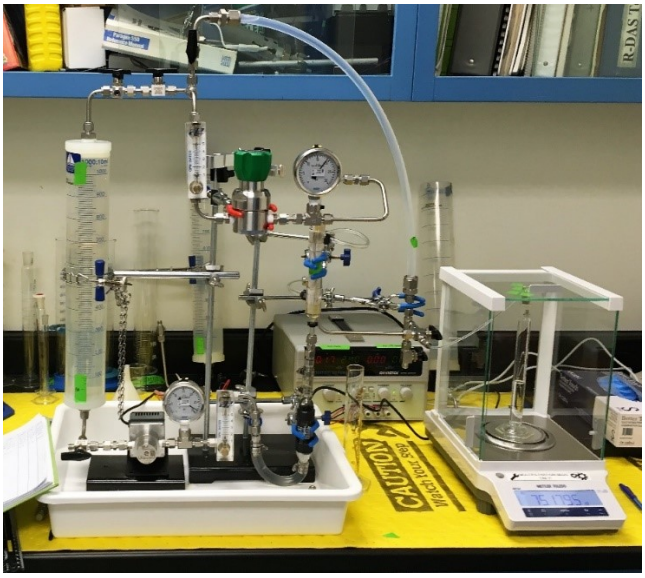

Figure 4. The system used for long-duration testing.

A. Sample Analysis

For the water recovery ratio tests, the samples were analyzed at MSFC's Environmental Control and Life Support System (ECLSS) Chemistry Laboratory for pH, conductance, TOC (persulfate-based analyzer), acetate, bromide, chloride, fluoride, nitrate, nitrite, phosphate, sulfate, ammonium, calcium, lithium, magnesium, potassium, sodium, 2-butanol, 2-methyl-2-propanol, methanol, ethanol, acetone, IPA (isopropanol), and nPA (1-propanol). The Toxicology and Environmental Chemistry Laboratories at Johnson Space Center (JSC) analyzed the samples for DMSD. Samples were shipped to MSFC and JSC overnight, in a cooler with ice packs, in order to prevent sample degradation and microbial growth.

For the long-duration tests, Kennedy Space Center (KSC)'s Chemical Analysis Lab analyzed the samples for DMSD, pH, conductivity, TOC, acetate, bromide, chloride, fluoride, nitrate, nitrite, sulfate, ammonium, calcium, potassium, sodium, 2-ethoxyethanol, diethylene glycol butyl ether (DGBE), methanol, ethanol, acetone, isopropanol (IPA), and 1-propanol (nPA). The first few batches of samples that were analyzed by KSC (prior to 1319 hours of testing) had low feed TOC values and some of the samples contained microbial growth. KSC filtered the samples to remove the microbial growth; however, it was determined that some sample was lost in the filter resulting in TOC values with a larger error. Subsequently, all future samples were no longer filtered and were shipped overnight in a cooler with ice packs, which prevented the microbial growth.

After 4727 hours, KSC stopped analyzing all samples, and MSFC and JSC conducted the remaining analyses in order to reduce costs. MSFC analyzed the samples for pH, conductivity, TOC, acetate, bromide, chloride, fluoride, nitrate, nitrite, phosphate, sulfate, ammonium, calcium, lithium, magnesium, potassium, sodium, 2-butanol, 2-methyl-2-propanol, methanol, ethanol, acetone, IPA, and nPA. JSC analyzed the samples for DMSD.

IV. Results

Figures 5 and 6 as well as Tables 1 and 2, show the results from the 98% water recovery testing using the Aquaporin Inside™ Hollow Fiber Module. The tables include both membrane performance as well as sample analysis results. The values with the less-than symbol indicate the detection limit or the highest reporting limit due to dilutions. Figures 11–27 show the analytical results for the long-duration testing; testing is in progress as the membrane has shown no degradation.

A. Water Recovery Ratio Results

The average run time was 219 hours, as shown in Table 1. The average percent water recovery ratio for the three tests was 97.6% ± 0.47%. Water recovery was calculated by dividing the final product volume by the initial feed volume. Table 2 includes the chemical analysis that was conducted by MSFC and JSC. Error values were calculated

based on the results from three tests using a 95% confidence interval. Figure 5 shows the flux over time and Figure 6 shows the water recovery ratio over time. Runs 1 and 2 were conducted using the same batch of feed; a leak occurred during Run 3 and the exact water recovery could not be determined. Subsequently, additional feed from a different batch was required to repeat Run 3; this experiment reached completion, although twice during the run the level sensor turned the pump off prematurely. The data shown in Figures 5 and 6 were based on the graduation marks on the feed and product tanks, which have an error of ±10 mL. The product volumes recorded were adjusted using a correction factor based on the total product water volume measured at the end of each test. The purpose of this correction was to account for the dead volume in the product stream.

Table 1. Operating conditions, volumes, and flux.

Run Time	h	219.32 ± 3.27
Water Recovery	%	97.62% ± 0.47%
Overall Flux	L/m^2*h	0.4029 ± 0.0042
Final Product Volume	L	1.0250 ± 0.0049
Initial Feed Voume	L	1.0500 ± 0.0003

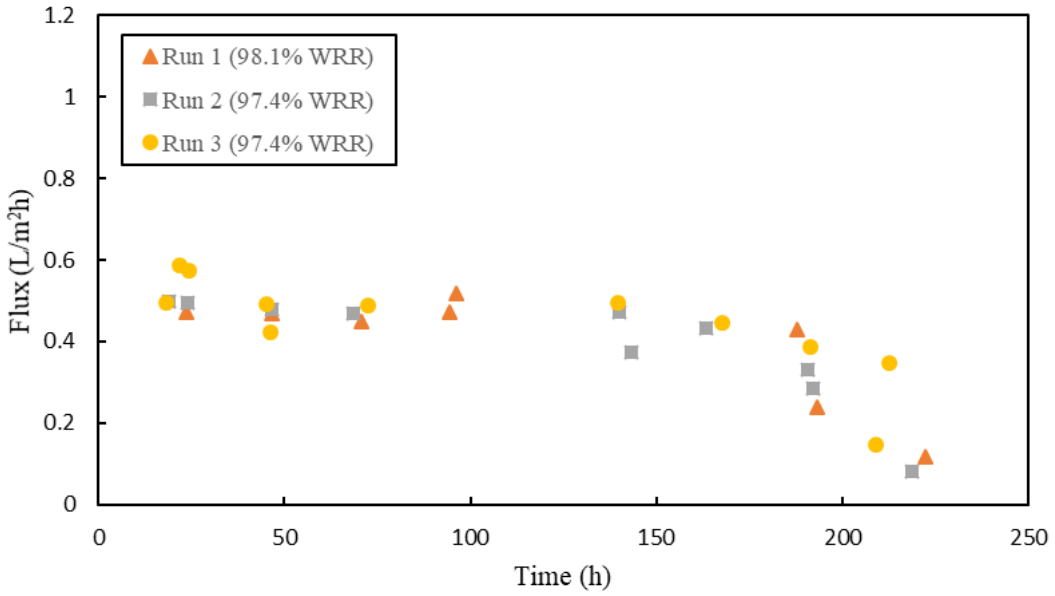

Figure 5. Flux versus time.

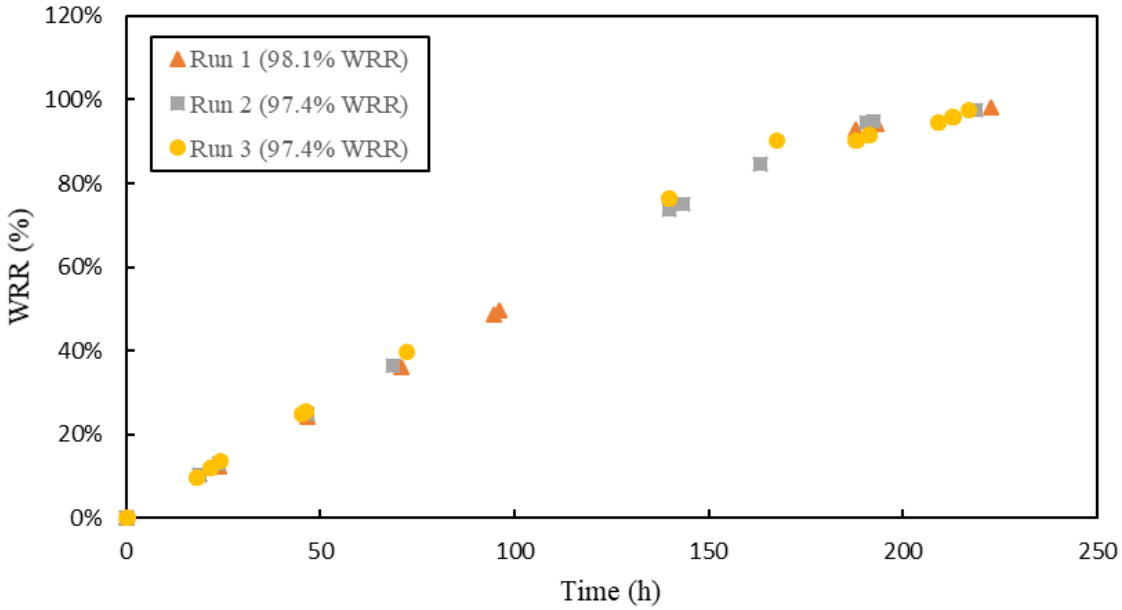

Figure 6. Percent water recovery ratio over time.

Table 2 shows the analytical results for the 98% water recovery ratio tests. The product water TOC concentration was 33.10 µg/mL, 32.10 µg/mL, and 17.10 µg/mL, for Runs 1, 2, and 3 respectively; this resulted in an average TOC concentration of 27.43 ± 10.14. Additionally, the product water conductivity was 73.57 ± 7.01 µS/cm. The theoretical concentration for DMSD in the feed was 22.0 µg/mL and the product contained 4.3 ± 0.85 µg/mL. (The feed was not analyzed for DMSD inorder to reduce cost.) For sodium, acetone, and alcohols, the results showed little to no rejection; the alcohols include methanol, ethanol, IPA, nPA, 2-Methyl-2-Propanol, and 2-Butanol. However, the results showed reductions for acetate, calcium, sulfate, chloride, and potassium.

Table 2. Analytical results for 98% water recovery ratio tests. The same batch of feed was used for Run 1 and 2; the feed for Run 3 was from a separate batch.

		Initial Feed Run 1, 2	Initial Feed Run 3	Final Feed	Final Product
pH		7.37	-	-	7.21 ± 0.20
Conductivity	µS/cm	133.00	162	-	73.57 ± 7.01
TOC	µg/mL	60.91	57.1	807.33 ± 55.21	27.43 ± 10.14
DMSD	µg/mL	-	-	-	4.30 ± 0.85
Acetate	µg/mL	40.46	17.2	254.00 ± 17.09	6.31 ± 1.02
Bromide	µg/mL	ND	<3.00	<60	<0.300
Chloride	µg/mL	23.92	22.2	440.67 ± 14.06	7.78 ± 1.55
Fluoride	µg/mL	0.08	<3.00	4.88	<0.300
Nitrate	µg/mL	BDL	<3.00	<60	<0.300
Nitrite	µg/mL	2.40	<3.00	11.91 ± 1.94	<0.300
Phosphate	µg/mL		<3.00	<60	<0.300
Sulfate	µg/mL	2.94	3.04	73.57 ± 5.12	0.38 ± 0.06
Ammonium	µg/mL	3.31	5.98	57.73 ± 3.84	3.70 ± 0.07
Calcium	µg/mL	1.12	4.05	25.17 ± 2.78	0.92 ± 0.26
Lithium	µg/mL	0.00	<2.00	<8.00	<0.200
Magnesium	µg/mL	0.00	<2.00	<8.00	<0.200
Potassium	µg/mL	12.24	23.7	378.00 ± 26.32	6.74 ± 5.11
Sodium	µg/mL	0.44	<2.00	9.00	0.70 ± 0.19
2-Butanol	µg/mL		<1.00	<1.00	<1.00
2-Methyl-2-Propanol	µg/mL		<1.00	<1.00	<1.00
Methanol	µg/mL	15.11	12.5	13.33 ± 1.83	12.87 ± 1.36
Ethanol	µg/mL	14.96	14.5	16.18 ± 8.70	10.32 ± 8.03
Acetone	µg/mL	2.88	2.74	5.18 ± 0.34	2.50 ± 0.19
IPA (Isopropanol)	µg/mL	1.44	<1.00	3.40 ± 0.06	<1.00
nPA (1-propanol)	µg/mL	BQL	<1.00	<1.00	<1.00

B. Long-duration Testing Results

The membrane life or long-duration test has been operating for approximately 8711.8 hours (testing began March 24, 2016) and testing is still in progress as membrane failure has not occurred. Figure 7 shows the flux data over time. The initial three data points were from a baseline test that was conducted prior to the long-duration testing. Figure 7 shows that there has not been a significant change in flux, or water flow across the membrane, since the test began. Based on this figure, membrane degradation has not occurred.

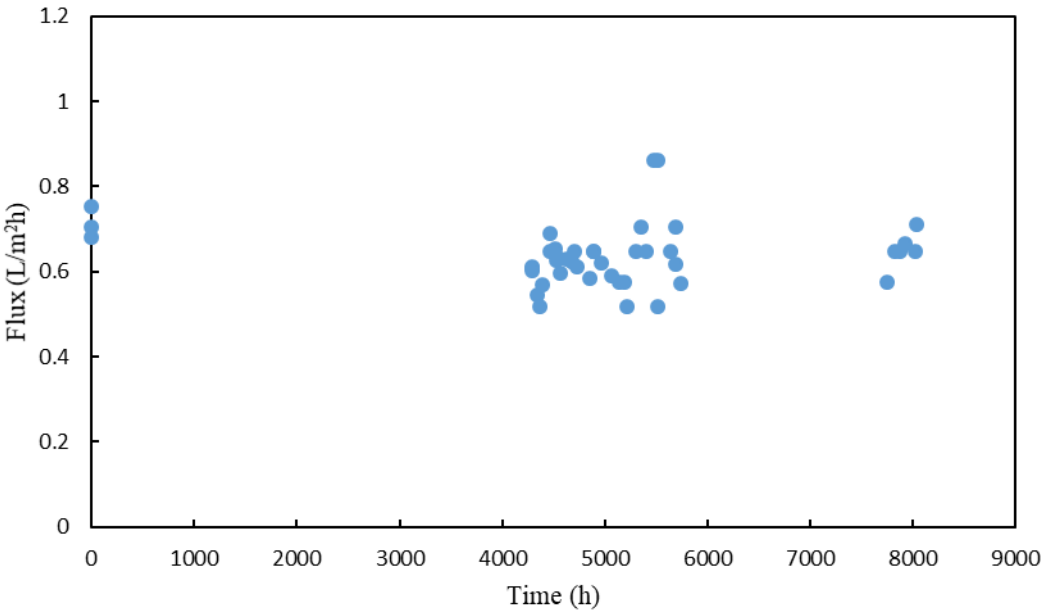

Figure 7. The product water flux versus time.

As long-duration testing has progressed, microbial growth in the feed has been observed, as shown in Figure 8. Microbial growth plugs the tubing and reduces the feed recirculation flowrate. To prevent the flowrate from dropping, the pressure regulator is briefly opened and the flow is increased to purge the tubing and remove the microbial growth. This method used to remove microbial growth from the system began at approximately 2281 hours of testing. Figures 9 and 10 show the difference between pressure adjustment (opening the regulator and purging any growth) and no pressure adjustment, between 8160 and 8711 hours. Figure 9, shows the feed flowrate of the flowmeter located before the membrane module. Figure 10 shows the feed flowrate of the flowmeter located after the backpressure regulator.

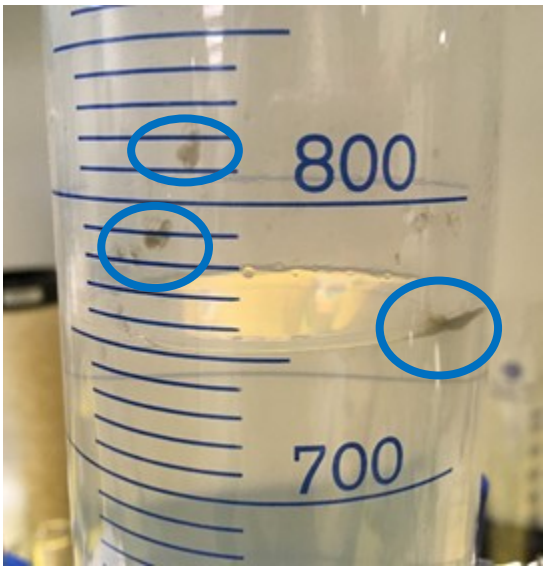

Figure 8. Microbial growth in the feed tank (circled).

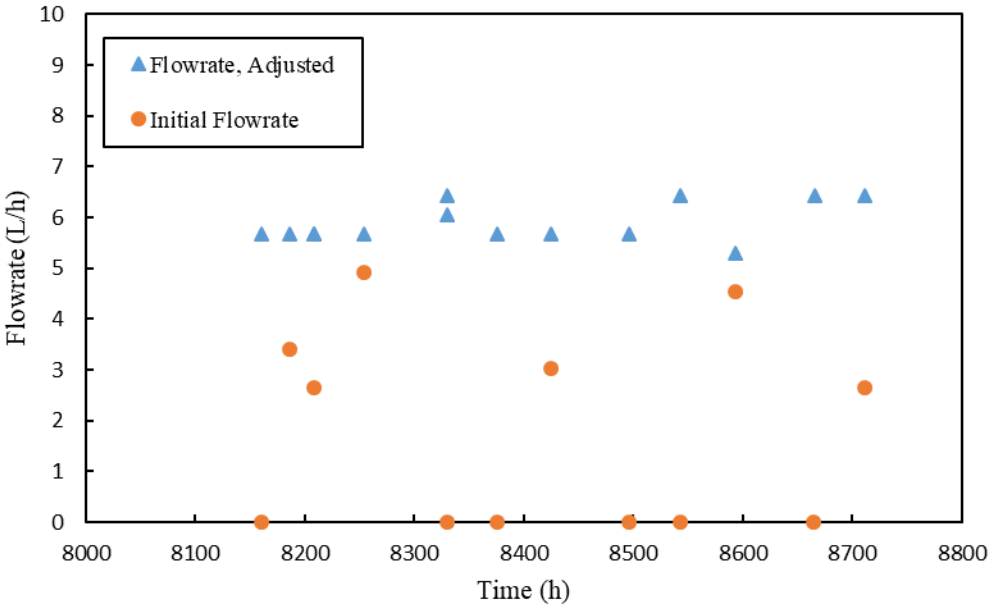

Figure 9. Feed flowrate of the flowmeter located before the membrane module. Initial flowrate versus flowrate after temporarily increasing the pressure and flowrate to purge the system. At any specific time, there are two data points, one showing the initial flowrate, and one showing the flowrate after the adjustment.

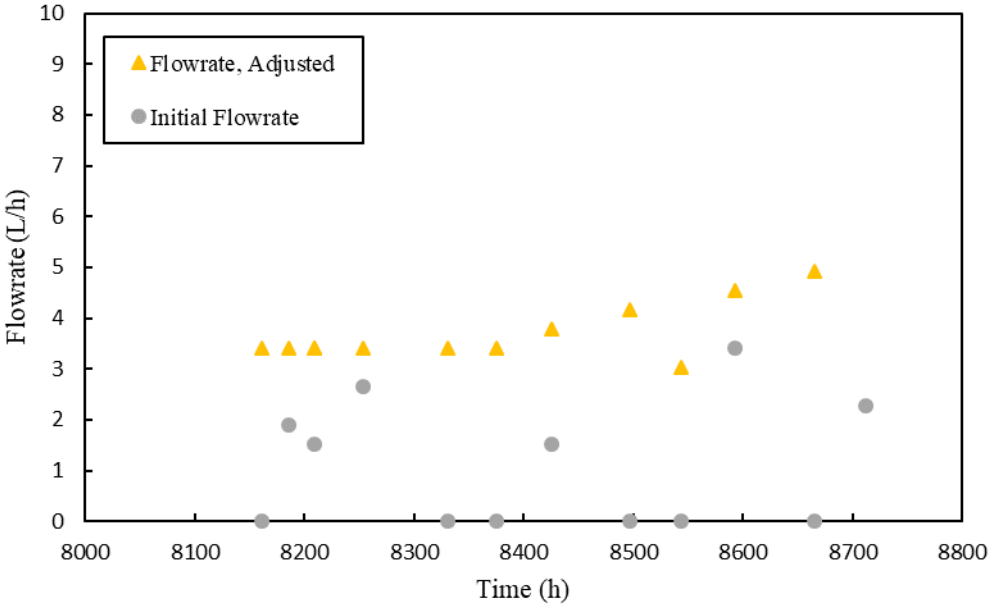

Figure 10. Feed flowrate of the flowmeter located after the membrane module (and after the back-pressure regulator). Initial flowrate versus flowrate after temporarily increasing the pressure and flowrate to purge the system. At any specific time, there are two data points, one showing the initial flowrate, and one showing the flowrate after the adjustment.

Figures 11–27 show the analytical results for the long-duration testing feed and product water samples. For calcium (Figure 15), sodium (Figure 18), ammonium (Figure 20), acetone (Figure 25), methanol (Figure 23), and ethanol (Figure 24), the results showed little to no rejection. The feed and product concentrations for IPA (Figure 26) and nPA (Figure 27) were mostly below <1 ppm. The results indicate significant reductions for TOC, DMSD, conductivity, acetate, and ions including chloride, potassium, and sulfate, as shown in Figures 11–14, 16, 17, 19 respectively. 2-ethoxyethanol and DGBE showed slight reductions although there was a bit of scatter in the data, as shown in Figures 21 and 22 respectively. The analyses for DMSD feed samples, 2-ethoxyethanol, and DGBE were conducted at KSC, and the analyses ended at approximately 4000 hours (Figures 12, 21, and 22). Other components in the product had very low concentrations including bromide (<0.3 ppm), fluoride (0-.3 ppm), nitrate (0-.3 ppm), nitrite (<0.3 ppm), phosphate (<0.3 ppm), lithium (<0.3 ppm), magnesium (<0.3 ppm), 2-butanol (<1.0 ppm), and 2-methyl-2-propanol (<1.0 ppm). MSFC analyzed the samples for phosphate, lithium, magnesium, 2-butanol, and 2-methyl-2-propanol starting at 4848 hours.

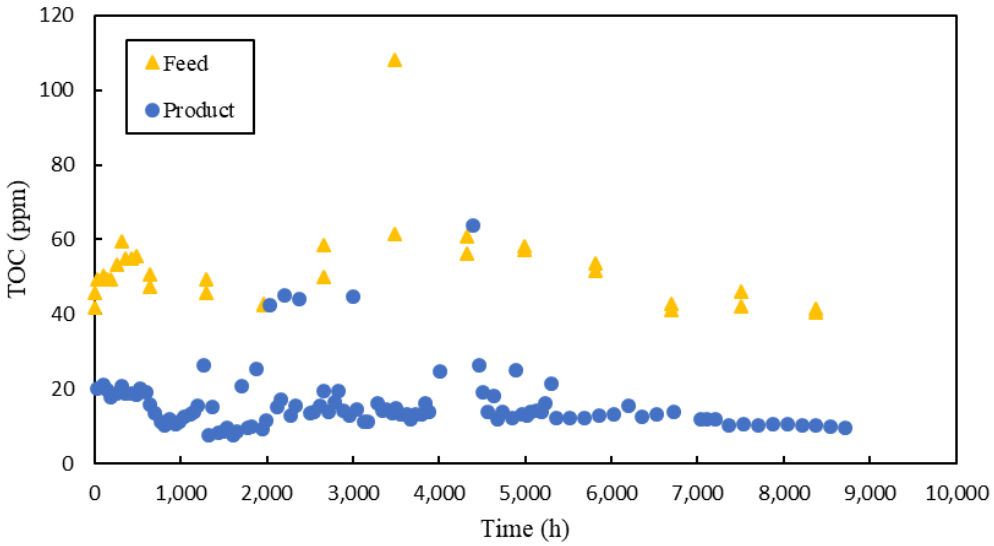

Figure 11. TOC concentration versus time for feed and product water samples.

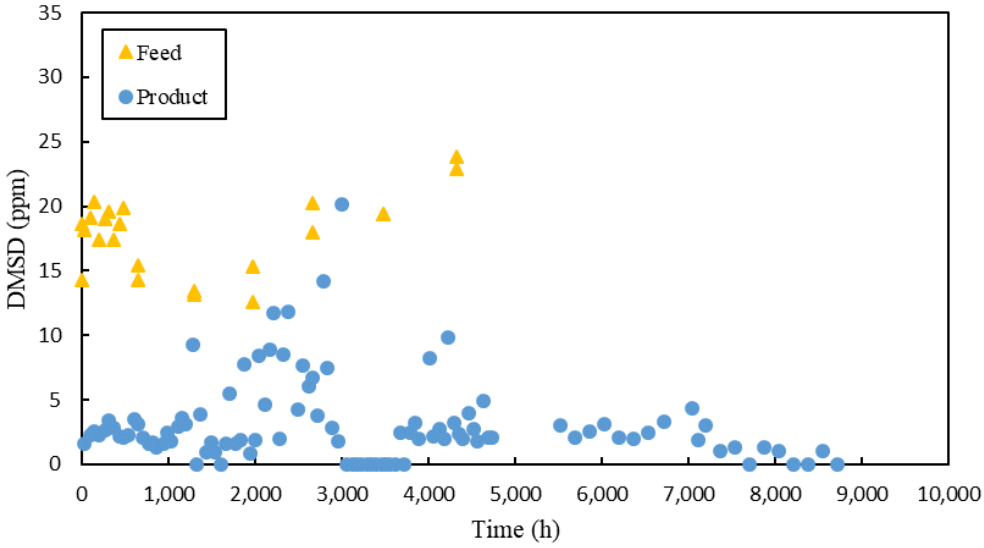

Figure 12. DMSD concentration versus time for feed and product water samples. After 4319 hours, DMSD was no longer analyzed to reduce cost.

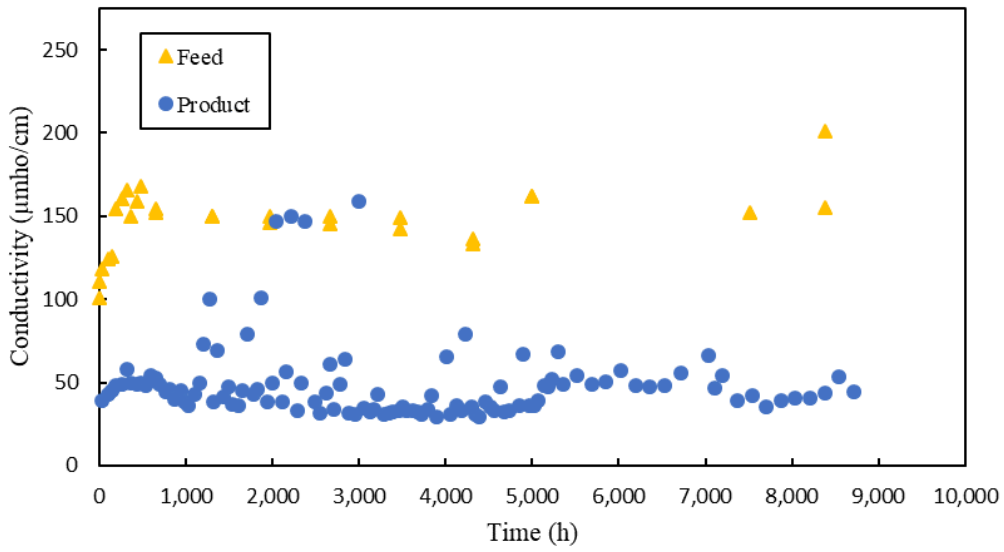

Figure 13. Conductivity versus time for feed and product water samples.

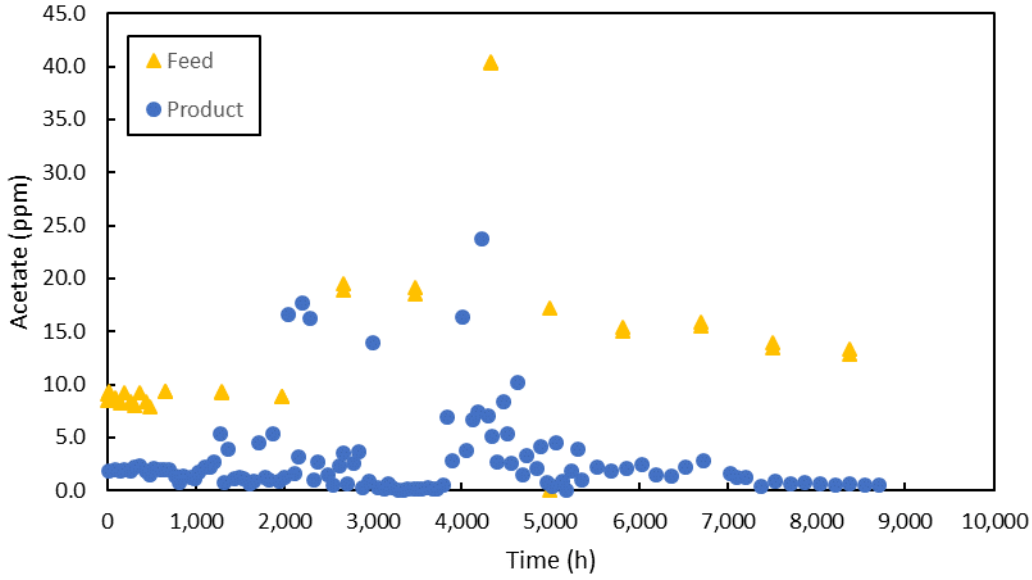

Figure 14. Acetate concentration versus time for feed and product water samples.

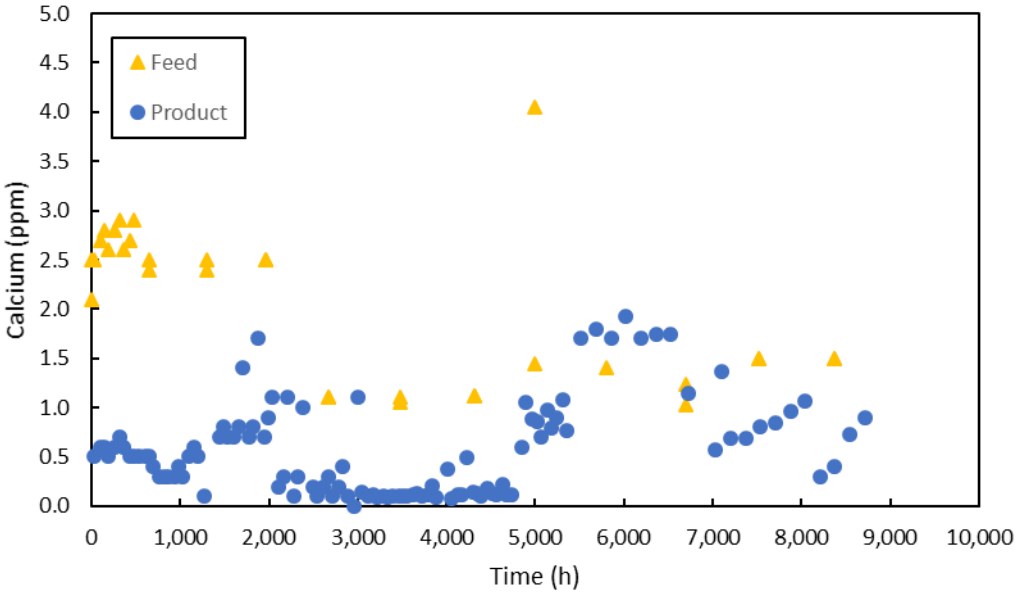

Figure 15. Calcium concentration versus time for feed and product water samples.

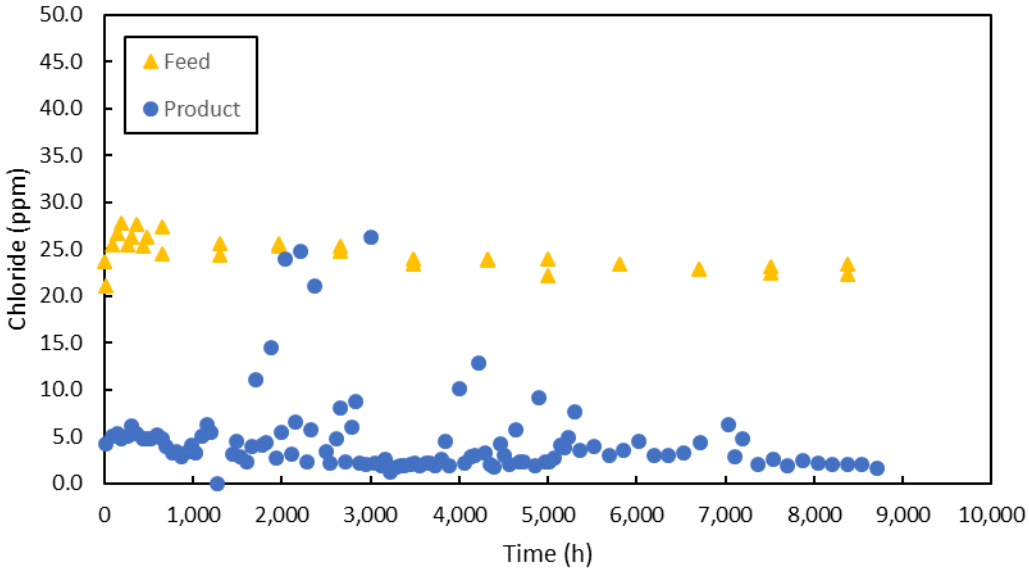

Figure 16. Chloride concentration versus time for feed and product water samples.

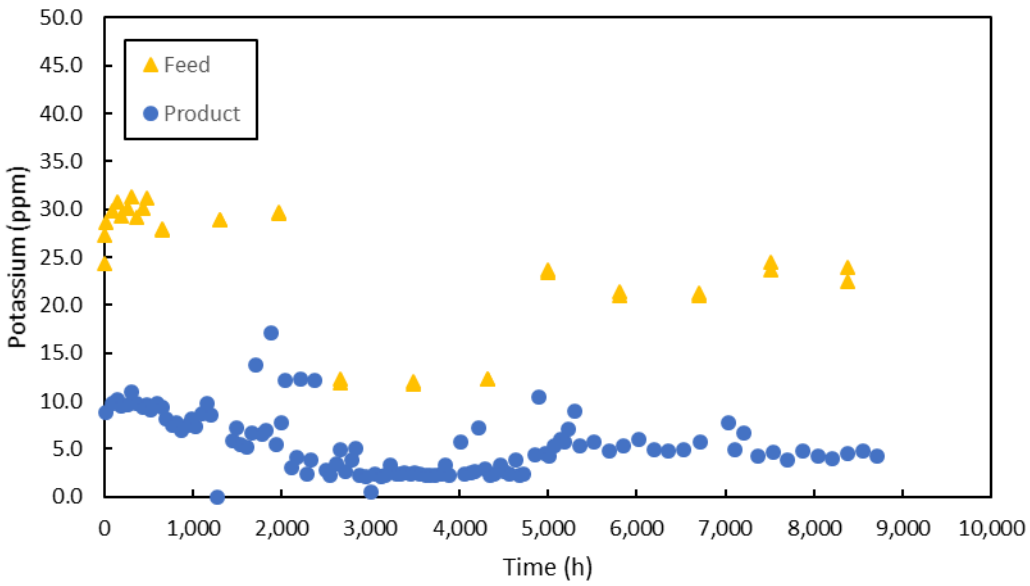

Figure 17. Potassium concentration versus time for feed and product water samples.

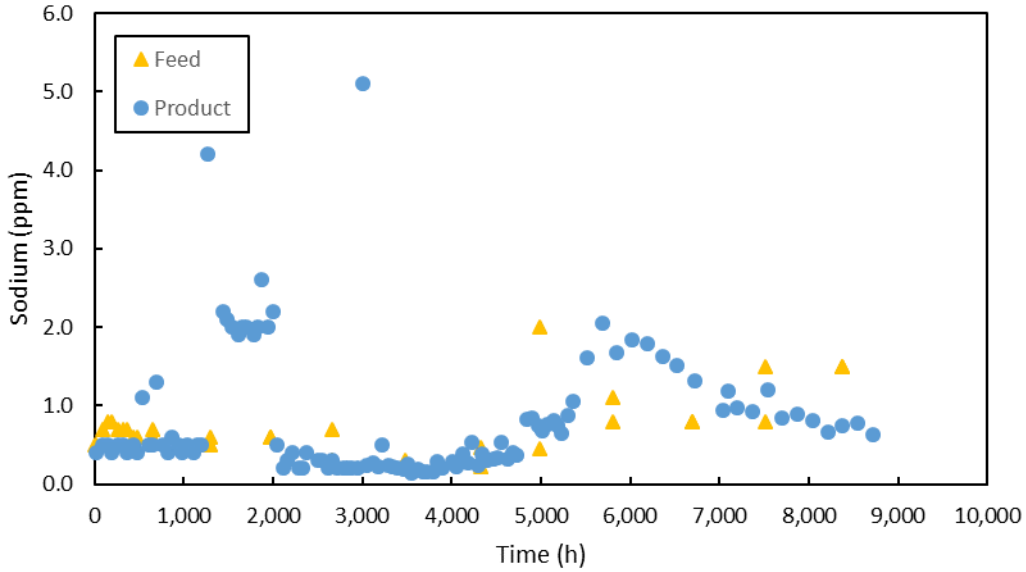

Figure 18. Sodium concentration versus time for feed and product water samples.

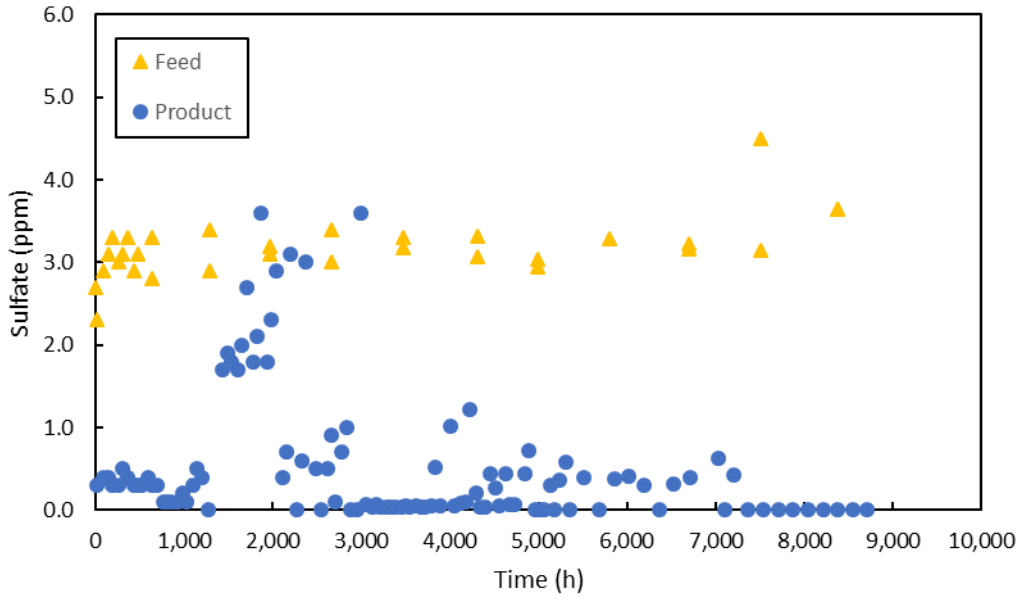

Figure 19. Sulfate concentration versus time for feed and product water samples.

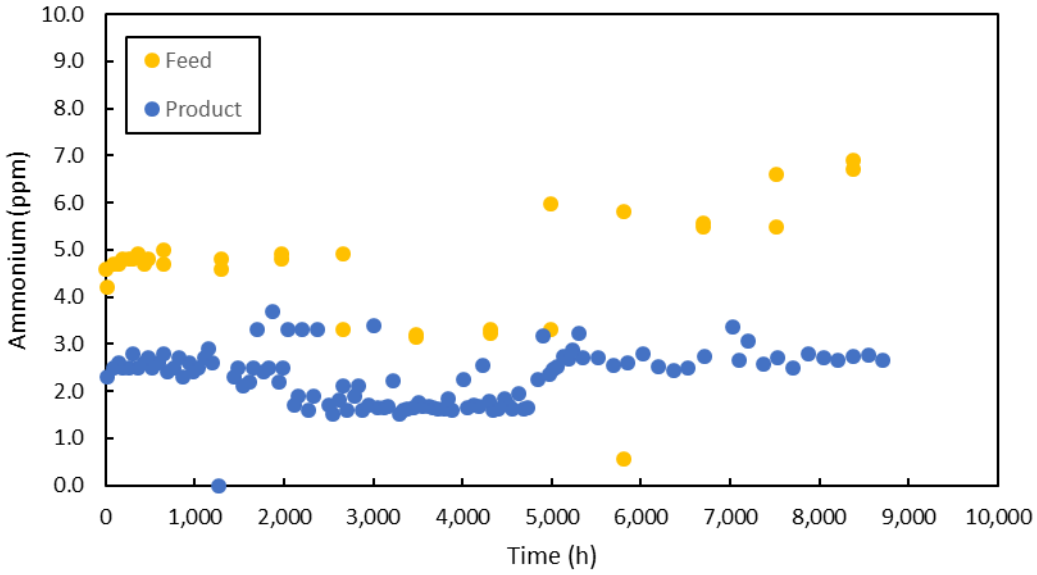

Figure 20. Ammonium concentration versus time for feed and product water samples.

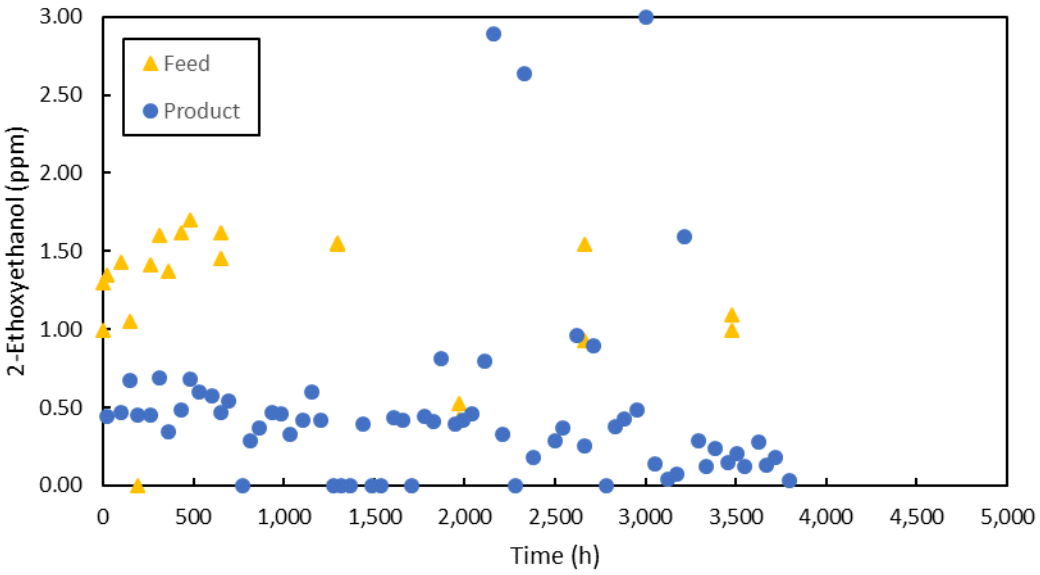

Figure 21. 2-Ethoxyethanol concentration versus time for feed and product water samples. Analysis ended at approximately 3793 hours and was conducted at KSC.

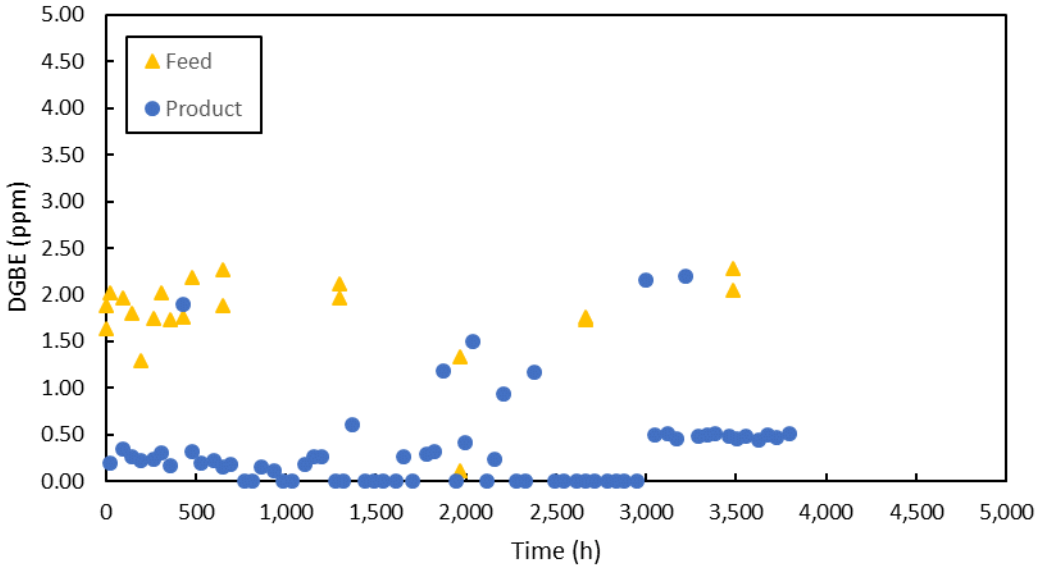

Figure 22. DGBE concentration versus time for feed and product water samples. Analysis ended at approximately 3793 hours and was conducted at KSC.

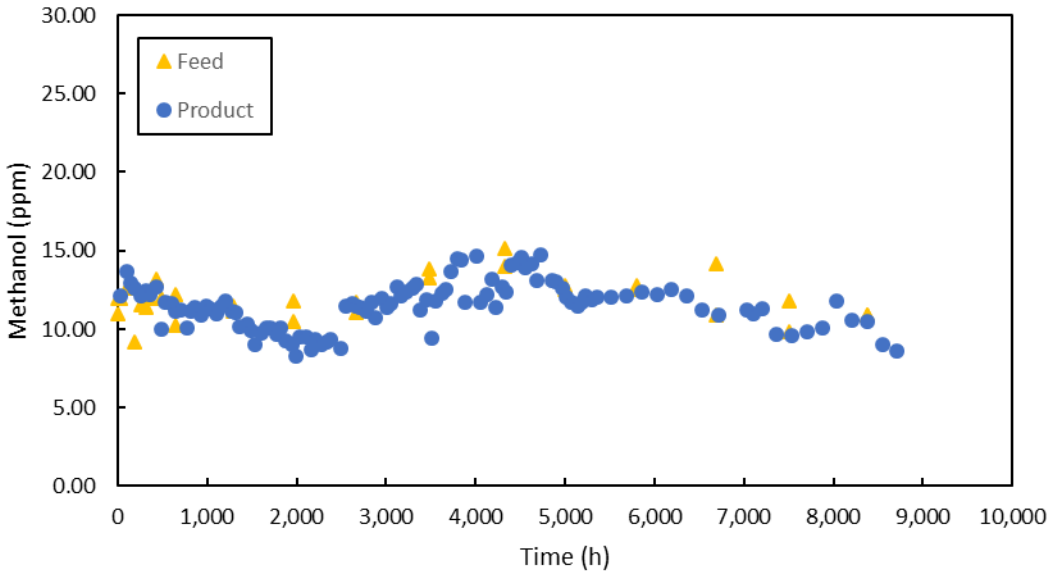

Figure 23. Methanol concentration versus time for feed and product water samples.

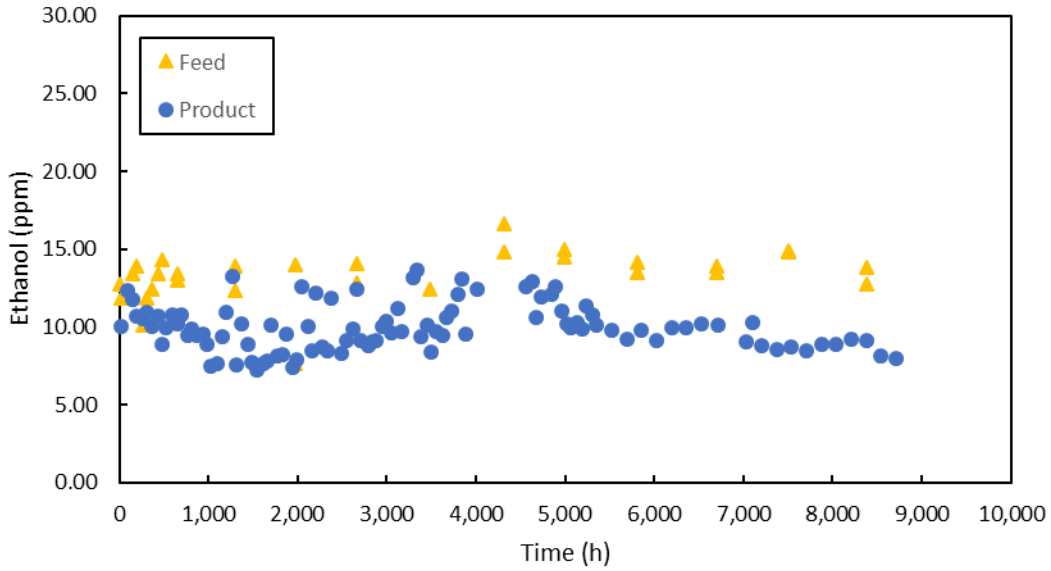

Figure 24. Ethanol concentration versus time for feed and product water samples.

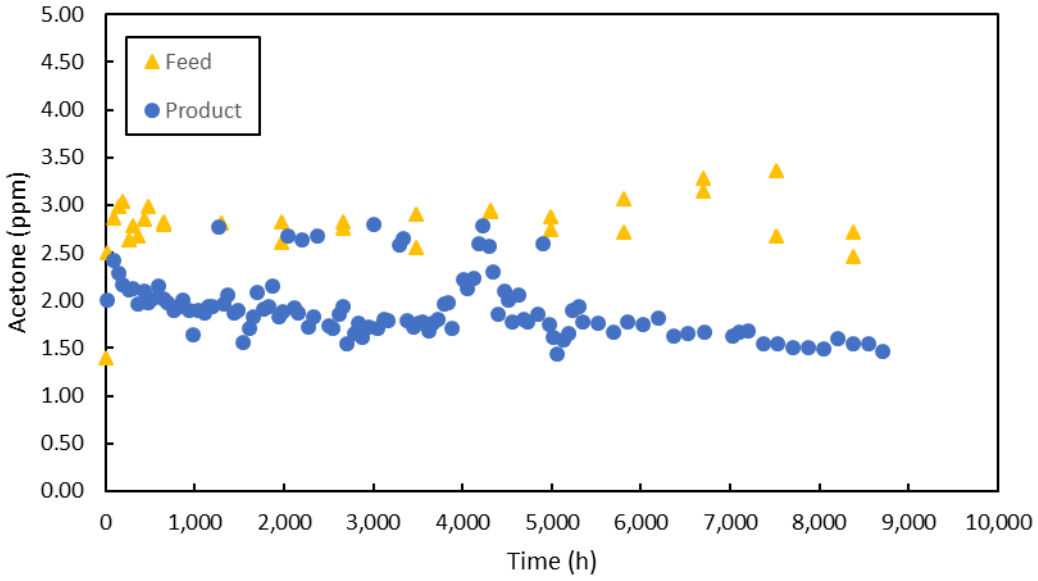

Figure 25. Acetone concentration versus time for feed and product water samples.

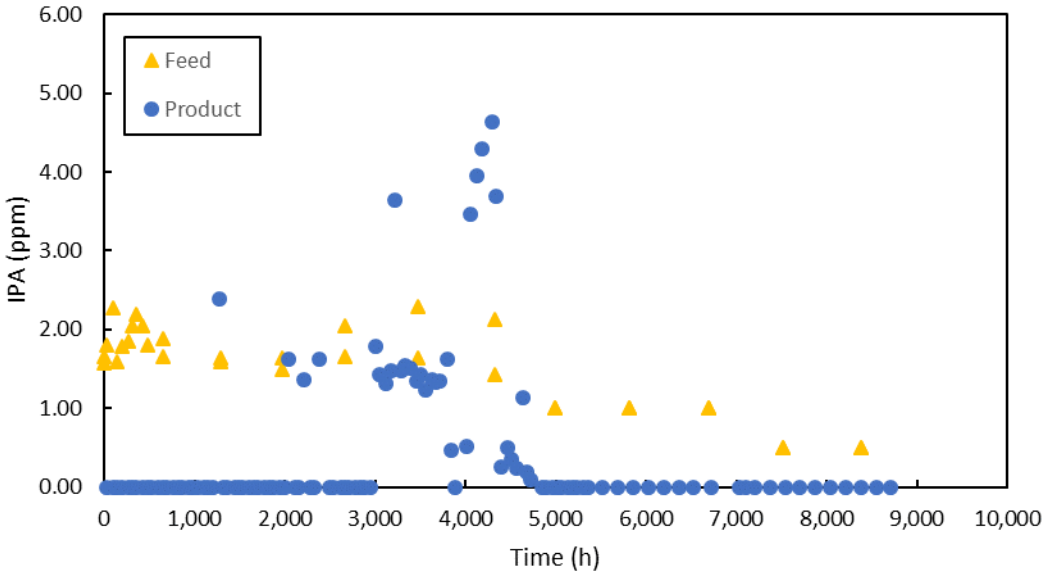

Figure 26. IPA concentration versus time for feed and product water samples.

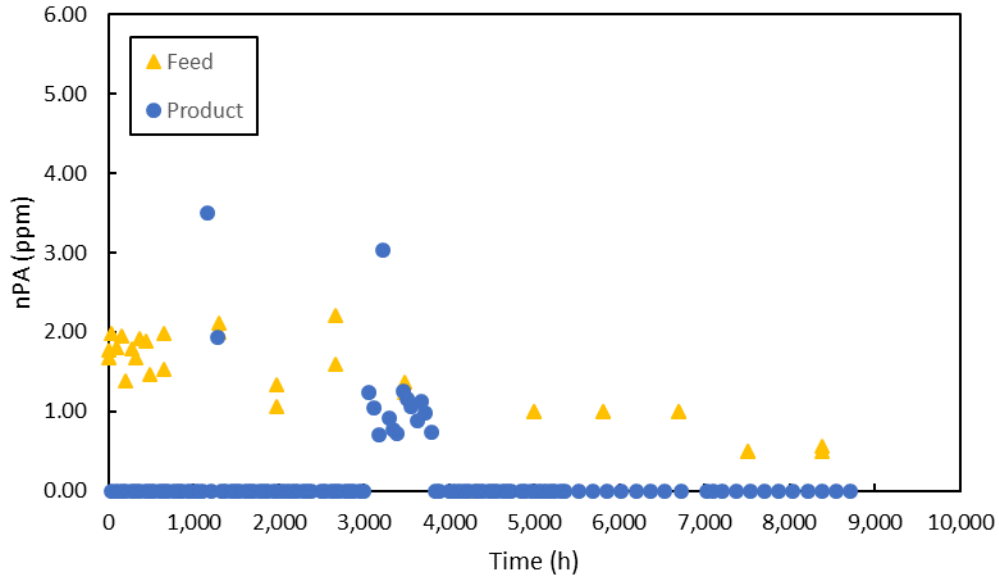

Figure 27. nPA concentration versus time for feed and product water samples.

V. Discussion

The results from this study show that the aquaporin membrane system meets the criteria at a 98% water recovery ratio, by achieving a 50% reduction in TOC concentration and conductivity. For long-duration testing results, TOC, conductivity, DMSD, ions, and alcohol concentrations have been relatively consistent except for calcium and sodium. Both calcium and sodium have shown inconsistent results; however, Figures 15 (calcium) and 18 (sodium) appear to have a similar trend. This correlation may be due to the relationship between aquaporins and calcium. A study by Martínez-Ballesta *et al.,* 2006, was conducted on the effects of calcium on aquaporins in salinity-stressed pepper plants.[9] Based on this study, aquaporin functionality may be influenced by the concentration of calcium. Calcium is an important factor in the maintenance of membrane integrity, ion-transport regulation, and potassium and sodium selectivity.[9] Scatter in all data may be due to changes in the shipping protocol (ice packs), filtration of samples prior to analysis, column changes for the analytical instruments, personnel changes, operational changes (pressure adjustment for microbial growth), and analytical laboratory changes.

In addition to the sample analysis results, concentration polarization appears to play an important role in membrane performance. Figure 28 indicates that if a system were to operate continuously at a 98% water recovery ratio and at 20 psi, the performance of the system would not be optimal. After a 90% water recovery ratio, the flux drops and this is most likely due to concentration polarization. This issue with concentration polarization may be addressed by redesigning the contactor specifically for a continuous flow or single pass system.

For future work, several tests would assist in a developing an optimum design. Long-duration testing has been conducted in a recirculation mode although development of a continuous flow system would simplify the integration with the ISS WPA to the point that the system would be a direct plug-in replacement for the Multifiltration Beds. No testing of a continuous flow system has been conducted, therefore, a long-duration test should be conducted using the single pass mode. In addition, a long-duration test should be conducted at the optimum water recovery ratio in order to determine membrane life; this test should also be conducted at the desired operating pressure (Volatile Removal Assembly (VRA) pressure is 65 psi). (The WWR for membrane life test conducted in this study was approximately 14%.) Other membranes should also be tested to determine the optimum membrane for this application such as a reverse osmosis membrane. A test also should be conducted with the system connected to the VRA, and pressure tests should be conducted to determine the effect of pressure on system performance.

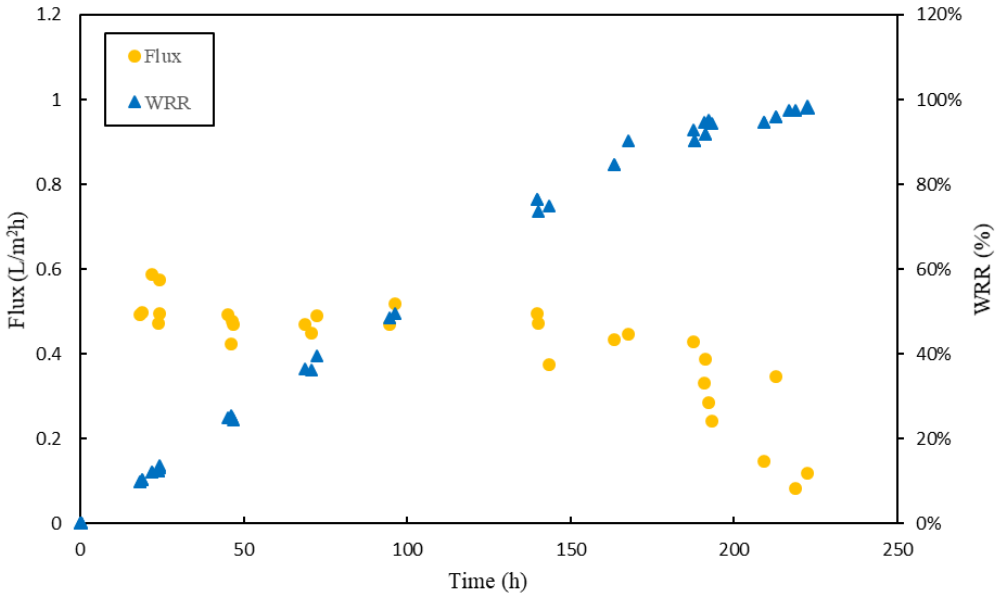

Figure 28. Flux and water recovery ratio versus time.

VI. Conclusions

The target TOC concentration for the ersatz or feed was approximately 57 ppm and the target conductivity was approximately 170 µS/cm. Based on the 98% water recovery testing, the membrane rejected approximately 50% TOC at a 97.6% ± 0.47% water recover ratio. The product TOC was 33.10 µg/mL, 32.10 µg/mL, and 17.10 µg/mL for Runs 1, 2, and 3 respectively. The average TOC was 27.43 ± 10.14 µg/mL, which is near the 50% rejection target. Additionally, the conductivity was reduced by approximately 50%; the product water specific conductance was 73.57 ± 7.01 µS/cm. The experiments would need to be repeated using the same batch of feed to reduce the error values. The theoretical feed concentration of DMSD was 22.0 µg/mL and the product contained 4.3 ± 0.85 µg/mL. For both the 98% water recovery ratio testing and long-duration (membrane life) testing, the results showed significant reductions for TOC, DMSD, conductivity, acetate, and ions including chloride, potassium, and sulfate. 2-ethoxyethanol and DGBE showed slight reductions although there was scatter in the data. Other components were below the detection limits including nitrate, nitrite, lithium, magnesium, 2-butanol, and 2-methyl-2-propanol. Acetone and alcohols showed little to no rejection including methanol, ethanol, IPA, and nPA. The feed and product concentrations for IPA and nPA were mostly below <1 ppm. Sodium and calcium showed inconsistent results. Furthermore, membrane life was determined to be a minimum of 8711.8 hours; testing began March 24, 2016 and testing will continue until the membrane fails. This test has been operating in a recirculation mode at 20 psi and approximately a 14% water recovery. The results indicate that purging the feed stream to remove microbial growth, by opening the pressure regulator and increasing the flowrate, could be an effective method for maintaining the feed recirculation flowrate.

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