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zNANO Forward Osmosis Membrane for Wastewater Treatment Processes

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Forward osmosis (FO) is used as a pretreatment to minimize reverse osmosis (RO) membrane fouling in short and long term spacecraft wastewater treatment processes. Commercially available FO membranes have low water flux rates resulting in large size and mass requirements in Forward Osmosis and Reverse Osmosis (FO/RO) systems. Large system size translates to higher launch cost. Therefore, FO membranes that have higher water flux rates improve the overall FO/RO system economics. This paper describes the ML-1 zNANO LLC lipid based FO membranes testing results. The zNANO membranes are based on a lipid-bilayer that can be used both in microfiltration and FO processes. zNANO membranes can be manufactured in a variety of layers configurations and electrical charges. This ability to manipulate membrane surface charges can be particularly useful as one can fabricate a membrane tailored for a specific process. This research characterized the unsupported zNANO ML-1 membranes in order to optimize their performance in terms of water flux rates and contaminant rejection. Initial testing results indicated that the ML-1 zNANO membranes have 12 times the water flux rates than that of commercially available membrane when deionized water was used as the feed and with 2 mol/l sodium chloride solution was used as the brine. When secondary wastewater was used as the feed solution, the ML-1 zNANO membrane has 4.4 times the water flux rates than that of the commercially available membrane. In addition, the zNANO ML-1 membrane reject 82±14ppm, 90±7ppm, 92±4ppm, 92±3ppm, 88±3ppm, and 86±17ppm of ammonium, potassium, magnesium, calcium, nitrate, sulfate, and total organic carbon respectively.

Nomenclature

ARC	Ames Research Center
DI	Deionized Water

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DOC	Direct Osmotic Concentration
EC	Electrical Conductivity
ERD	Energy Recovery Devices
FDFO	Fertilizer Draw Forward Osmosis
FO	Forward Osmosis
FO/RO	Forward Osmosis and Reverse Osmosis
FOB	Forward Osmosis Bag
FOST	Forward Osmosis Secondary Treatment
ISS	International Space Station
MABR	Membrane Aerator Biological Reactor
mS	milli Siemens
NASA	National Aeronautics and Space Administration
OMEGA	Offshore Membrane Enclosure for Growing Algae
RO	Reverse Osmosis
TOC	Total Organic Membrane
μS	micro Siemens

I. Introduction and Background

Forward Osmosis is a process where the osmotic potential between two fluids of differing solute/solvent concentration is equalized by the movement of solvent from the less concentrated solution to the more concentrated solution.[3] This is typically accomplished through the use of a semi-permeable membrane that separates the two solutions. Such FO processes and corresponding membranes have been researched and developed by NASA since 1995 for use in future water recycling systems aboard both short and long duration human space missions. The development process has been well documented in previous paper.[2] Recently, the Forward Osmosis Secondary Treatment (FOST) system was built and delivered to JSC in 2013. The FOST system was designed as a post treatment to the Membrane Aerated Biological Reactor (MABR). The MABR reduces the organic content of the wastewater while the FOST system will remove the dissolved solids.[6] In the FOST system, a FO module is used as a pretreatment step to minimize fouling in the reverse osmosis membrane. In this system, as in the DOC system,[3] clean water passes through the FO semi-permeable membrane into the osmotic agent (OA). Water is then removed from the OA through the RO system. Coupling the FO/RO systems together provides a RO concentrated salt solution. This solution drives water across the FO membrane. In addition, the FOST paradigm consists of a system of RO energy recovery pumps. Coupling the FO/RO systems and energy recovery devices (ERD) improves the wastewater treatment process in terms of power, size, mass, reliability and resupply. Since the water flux across the FO membrane is dependent on the membrane size and OA salt solution concentration, we hypothesize that improving the water flux rate across the FO membrane will lead to smaller, lighter, and lower power systems. The NASA ARC has an intensive membrane comparison project to develop and test a variety of FO membranes. Since 2010, NASA has collaborated with zNANO (San Jose, CA) to test its newly develop FO membranes. This paper describes the initial test results for the zNANO forward osmosis membrane. This testing is being done as a research effort to develop FO membrane with better performance characteristics and process-specific functionality. The results of these test can be used to re-evaluate the use of FO membranes in the Direct Osmosis Concentration (DOC) System,[3] the Forward Osmosis Cargo Transfer Bag (FOB),[5] the Habitat Water Wall,[4] the Sustainability Base,[7] the Forward Osmosis Secondary Treatment (FOST) system,[6] the Pressure Retarded Osmosis (PRO),[1] the Fertilizer Draw Forward Osmosis (FDFO),[9] and the Offshore Membrane Enclosure for Growing Algae (OMEGA).[8] The test results of the zNANO membrane are compared with commercially available FO membranes. In the testing of these membranes, DI water and secondary treated wastewater were used as a feed and 2 mol/l of salt water was used as a draw solution.

II. Materials and Procedure

Two setup methodologies were used for the comparative testing of the zNANO membranes. In both cases, flat sheet modules were designed and fabricated in-house at ARC. The module used in setup A has a larger active membrane area than that of setup B.

A. Materials and Test Setup

Experimental setup and flow diagrams of the test setup A are shown in Figures 1 left and 2, and setup B in Figures 1 right and 3. . Commercially available membranes and the zNANO membranes are used. The membranes used are single flat sheets with membrane areas of 0.037 m^2 for setup A and 4.25 × 10^{-4} m^2 for setup B. DI water produced by a system with an electrical conductivity of less than 10 μS/cm is used as a feed and 2 mol/l of brine is used as a draw solution. Stir plates are used to keep the solution well mixed.

For setup A, the membrane is installed in a stainless steel housing where it is sandwiched between plastic net spacers. O-rings and stainless steel plates are bolted together with fasteners. The solution flows into the module at the bottom and exits at the top outlet. This configuration allows air to exit from the top of the module. For the feed and brine side, flow rates and pressures are measured with analog gauges. A centrifugal magnetic drive pump (Cole Parmer 07003-04) is used to recirculate the brine solution at 7 GPH. Electrical conductivity and temperature of the feed are measured using a bench top conductivity meter (YSI 3200). A calibration curve is generated to correlate the conductivity measurement and sodium chloride concentration. A stir plate is used to stir the feed and brine solution. The volume of the feed solution is measure using a scale.

Setup B has an active membrane area of 4.25×10^{-4} m^2. The zNANO membrane is provided by the zNANO LLC. and cut to the same size as the commercially available membrane. For setup B, there is no pressure gauge and flow rate gauge for both the feed and brine lines. Masterflex double head peristaltic pump (Cole-Palmer 77120-62) are used to recirculate solution through both sides of the membrane at 14mL/hr. Conductivity, temperature, and mass of the feed solution are automatically recorded via hyper terminal for 24 hours.

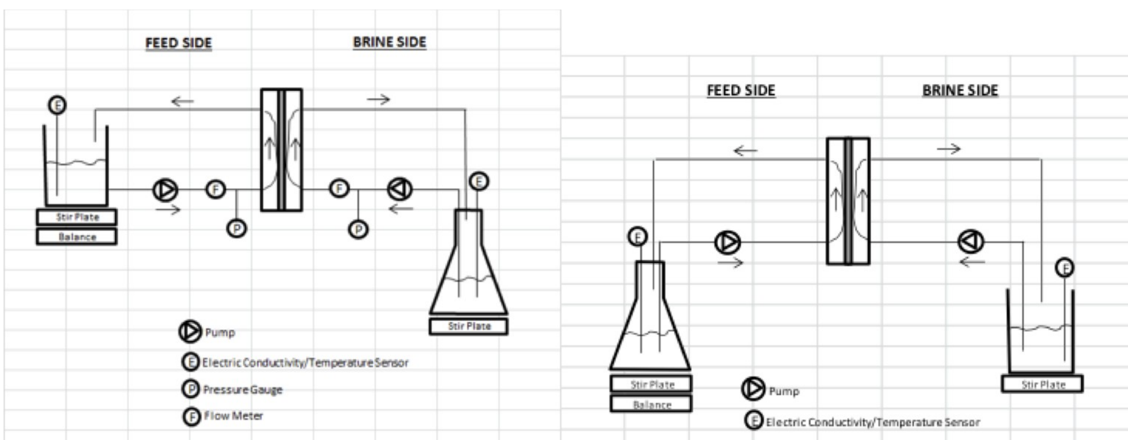

Figure 1. Left: Diagram of Test Setup A. Right: Diagram of Test Setup B, Small Housing.

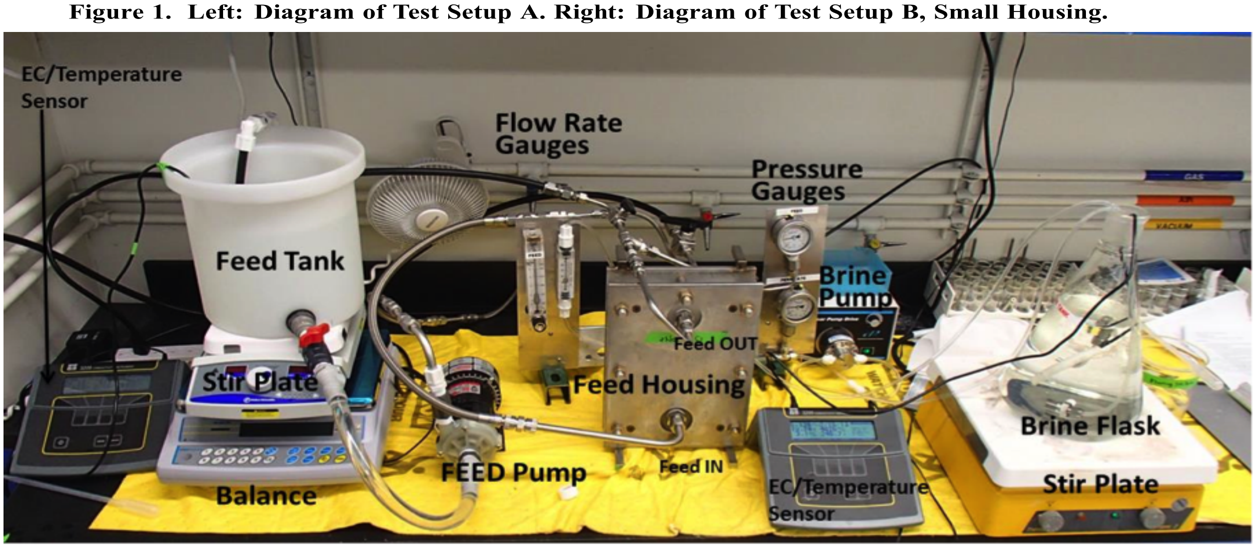

Figure 2. Test Setup A, 0.037m^2 active membrane area

B. Conditions and Parameters

Sample Analysis Methods are listed in Table 1. For DI water and salt tests, only electrical conductivity (EC) readings were recorded to determine the salt back flux and to determine the salt content in the brine tank. The relationship between salt in g/L and EC readings were determined via a calibration curve. For the wastewater tests, 40mL samples were collected and submitted to the analytical lab and analyzed the same day. When sample could not be analyzed in the same day, they are refrigerated at 4˚C.

Table 1. Sample Analysis Method

Analysis Items	Method/Equipment	Notes
Anion and Cation	ThermoFisher (Dionex) Ion Chromatograph with a conductivity detector	require at least 5 ml of sample
Total Organic Carbon (TOC)	Shimadzu Total Organic Carbon Analyzer, using UV-Persulfate Oxidation	require 40 ml

Test conditions and parameters are shown in the Table 2. For wastewater testing, the brine solution is 2 mol/l of the NaCl. NaCl is used due to its non-hazardous properties for spacecraft applications and as a source of comparison to previous FO data collected on the DOC. On the feed side, secondary wastewater from a local wastewater treatment plant was used. The initial feed pH measured 6.8 and the conductivity was approximately 2.2 mS/cm.

For setup A, a flow rate of 6.3×10−5m3/s (60GPH) is used for the feed, 7.4×10−6m3/s (≈ 7GPH) is used for the brine . The flow rate of the feed side is higher than the brine side to minimize concentration polarization. For setup B, measured flow rate is 2.48×10−7 m3/s for both feed and brine sides

Table 2. Test Conditions and Parameters

Test No.	Membrane	Feed Solution	Draw Solution	Flow Rate	Notes
1-1 to 1-3	Commercial Setup A	DI Water 8 Liters	Brine 2 mol/Liter 0.5 Liters	Feed:$6.3\times10^{-5}m^3/s$ (=60GPH) Brine:$7.4\times10^{-6}m^3/s$ (7GPH)	Used as a baseline case for comparison.
2-1 to 2-3	Commercial Setup A	Wastewater 8 Liters	Brine 2 mol/Liter 0.5 Liters	Feed:$6.3\times10^{-5}m^3/s$ (=60GPH) Brine:7.4×10-6m3/s (7GPH)	Used as a baseline case for comparison.
3-1	Commercial Setup B	DI Water 1 Liter	Brine 2 mol/Liter 0.5 Liters	Feed and Brine, 2.48×10^{-7} m^3/s	To compare with results of No.1-1 to 1-3 to know difference of the setup A and B under same condition.
4-1 to 4-3	zNANO Setup B	DI Water 2 Liters	Brine 2 mol/Liter 2 Liters	Feed and Brine, 2.48×10^{-7} m^3/s	
5-1 to 5-3	zNANO Setup B	Wastewater 1 Liter	Brine 2 mol/Liter 0.5 Liters	Feed and Brine, 2.48×10^{-7} m^3/s	

III. Results and Discussion

Test results of the commercial membranes and the zNANO membrane with setup A and B are summarized in the Table 3 and shown in Figures 4 through 7. Additional test results are shown in the Appendix.

Table 3. A table summarizing the test type, membrane and set-up used, and the associated results.

Test No.	Membrane	Feed	Set-up No.	Corrected Water Flux at 20 degree C [LMH]	Averaged Water Flux [LMH], Standard Deviation	Initial/Final EC on Feed [/cm]
1-1	Commercial	DI water	A	7.14	7.15 0.326, 4.55%	0.7/7.4μS
1-2				6.84		0.74/6.9μS
1-3				7.49		0.71/8.0μS
2-1	Commercial	Wastewater	A	5.64	5.2 0.384, 7.39%	1.9/2.1mS
2-2				5.05		2.2/2.4mS
2-3				4.92		2.3/2.5mS
3-1	Commercial	DI water	B	7.55		
4-1	zNano	DI water	B	84.8	86.1 4.09, 4.75%	2.4/30μS
4-2				90.7		1.8/28μS
4-3				82.9		1.0/23μS
5-1	zNano	Wastewater	B	20.1	23.1 4.8, 20.9%	2.4/3.2mS
5-2				20.4		2.3/3.6mS
5-3				28.6		2.9/3.9mS

Electrical conductivity of the feed side increases over time due to the volumetric concentration in the feed and a small amount of salt back-flux from the brine solution into the feed tank. The amount of salt back-flux can be calculated using Equation (1).

$$Loss_{NaCl} = C_1(V_0 - \Delta V) - C_0 V_0 \tag{1}$$

C_0: Initial NaCl Concentration in the feed solution [g/L]
C_1: Final NaCl Concentration in the feed [g/L]
V_0: Initial volume of the feed [L]
ΔV: Amount of transferred water from the feed to brine [L]

Water flux rates are parameters for modeling the performance of FO membranes. The water flux rates are dependent on temperature and can be adjusted using Equation (2).

$$Water\ Flux_{20°C} = Water\ Flux_{Average} * e^{522.9*\left[\left(\frac{1}{(T+125.64)}\right)-\left(\frac{1}{(20+125.64)}\right)\right]} \tag{2}$$

where T: average operating temperature of the feed in ˚C
T: average operating temperature of the feed in ˚C
J: Flux

Tests 2.1 to 2.3 show a back-flux of 0.8 g/L NaCl into the feed tank. This amount of salt back-flux significantly contributes to three different complications in the waste treatment process.

1. Salt loss on the OA side require a salt resupply cost to keep the OA tank replenished to prevent decreasing water flux rates decline.
2. As salt increase in the feed, the osmotic potential between the feed and the brine decrease thereby decreasing the water flux rates.
3. Salt back-flux into the feed side contributes to the added loading as the feed concentrated brine must be further processed downstream.

According to table 3, the standard deviation of the average water flux for the testing No.1-1 through 4-3 is less than 7.4%. On the other hand, standard deviations in testing No.5-1 through 5-3 are as high as 20.9%. The high standard deviations in tests 5-1 through 5-3 are due to the internal concentration polarization that existed in the smaller setup B. The modules used for the smaller test setup were not rated for high flow/pressure and a higher flow pump was not used. This setup was used because the vendor did not have a larger sheet of membrane available at the time of this testing. The low flow coupled with narrow flow channels lead to fouling at the membrane surface.

The water flux rates versus run time graphs are shown in Figure 4 through 7. Figures 4 through 5 show the graphs of water flux rates for experiments conducted using set-up A when DI water and secondary wastewater were used as the feed solution and 2 mol/L of NaCl was used in the brine solution. These graphs show that for the commercially available membrane, the water flux rates is 7.2LMH when deionized water was used as the feed solution and 5.2LMH when wastewater was used as the feed solution. Figure 5 shows that the water flux rate increases dramatically to 86.1 LMH (with DI water as the feed solution and 2 mol/L of NaCl was used as the brine) when the zNANO membrane were used.

Figure 7 shows the graph for the runs conducted with the zNANO membrane using wastewater as the feed solution and 2 mol/L NaCl as the brine. Here, water flux decrease from 86.1 LMH to 23.1 LMH due to the concentration polarization. These contaminants block out the permeation of water to the brine side. To avoid this kind of fouling on the membrane surface, more powerful pumps need to be used to provide more flow rate to the feed side to create turbulent shear flow on the membrane surface.

The notable result is that the water flux of the zNANO membrane is approximately 12x that of the commercial membrane for DI water and around 4.4x that of the wastewater feed solution.

To verify that data from both experimental set-ups are comparable, DI water test was conducted on both set-ups using the commercially available membrane. From Table 3, the result of the testing No.3-1 is within around 10% deviation of average of the testing No.1-1 through 1-3. The setup A and B are considered to be equivalent even though sizes of membranes installed and the flow rate of the feed and brine are different because the water flux rates are the approximately the same for the commercially available membrane.

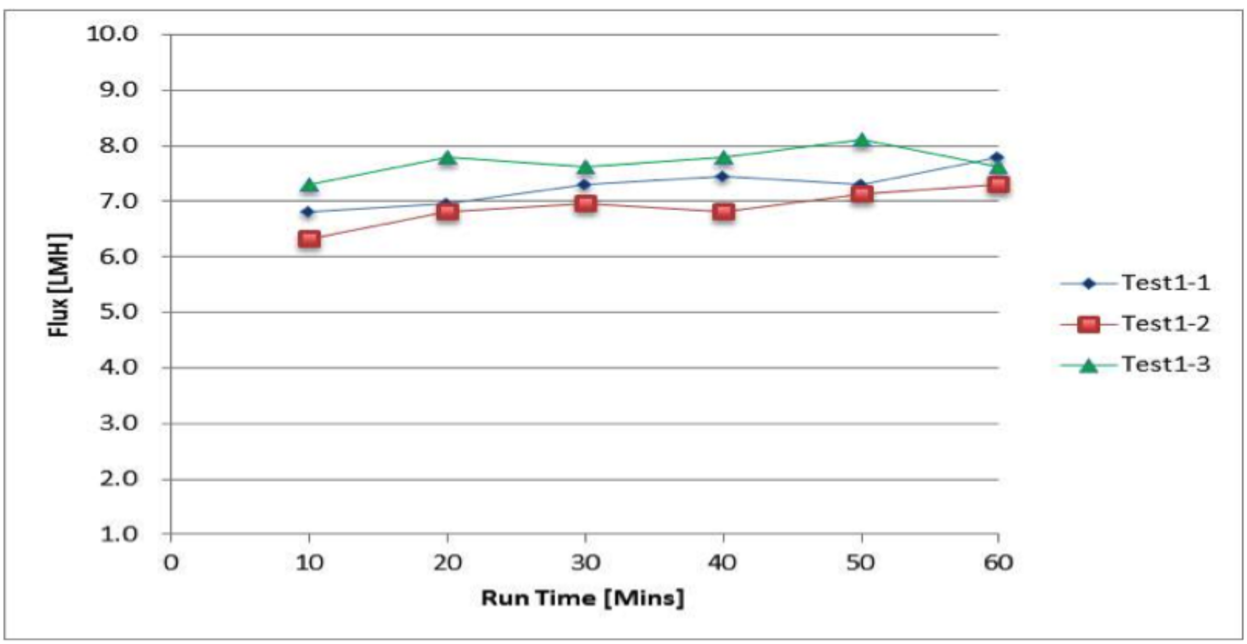

Figure 4. Water flux rate versus run time using the commercially available FO membrane, with DI water in the feed, and 2 mol/L NaCl solution in the brine, Test Setup A

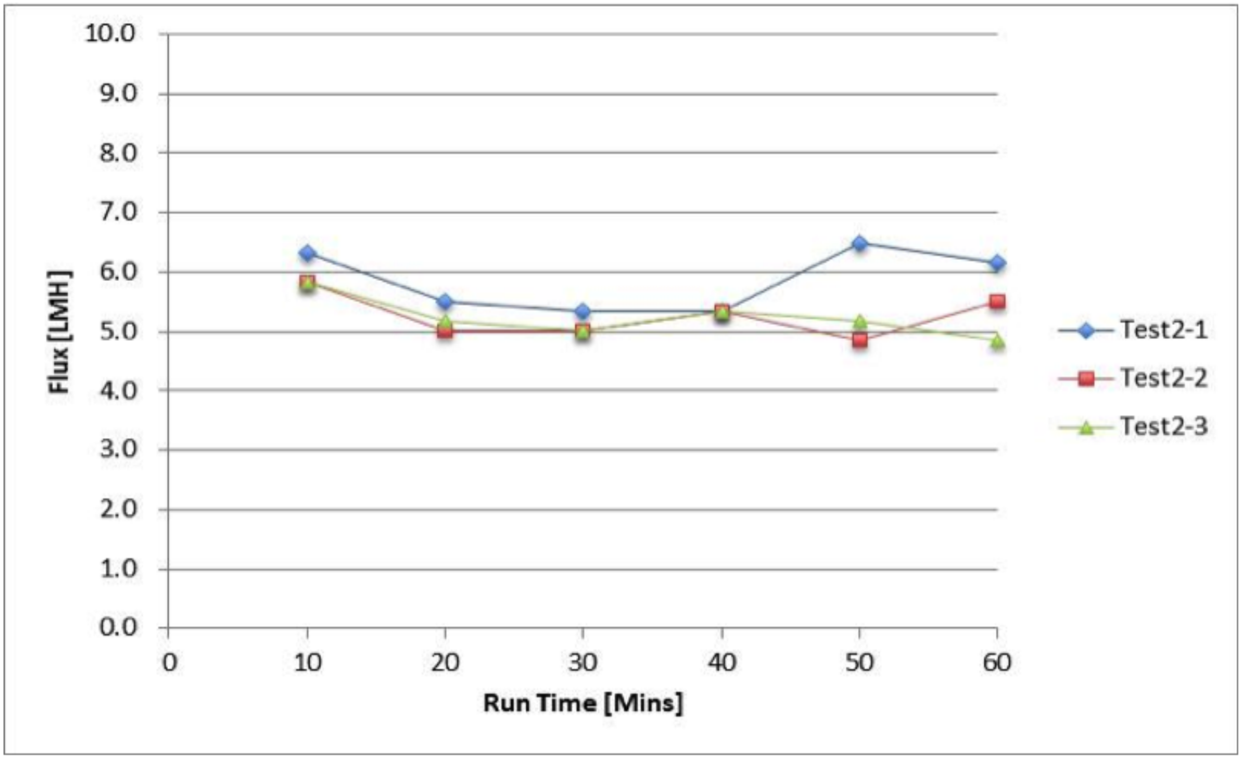

Figure 5. Water flux rate versus run time using the commercially available FO membrane, with wastewater in the feed, and 2 mol/L NaCl solution in the brine, Test Setup A.

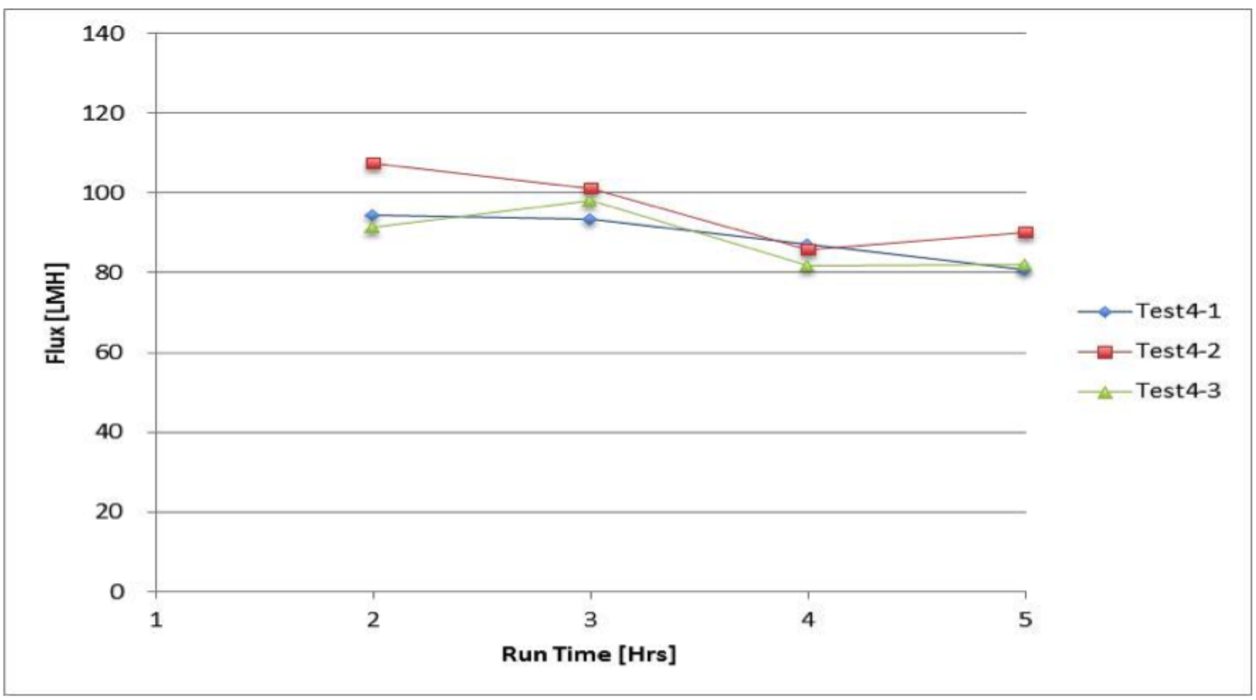

Figure 6. Water flux rate versus run time using the zNANO ML-1 FO membrane, with DI water in the feed, and 2 mol/L NaCl solution in the brine, Test Setup B.

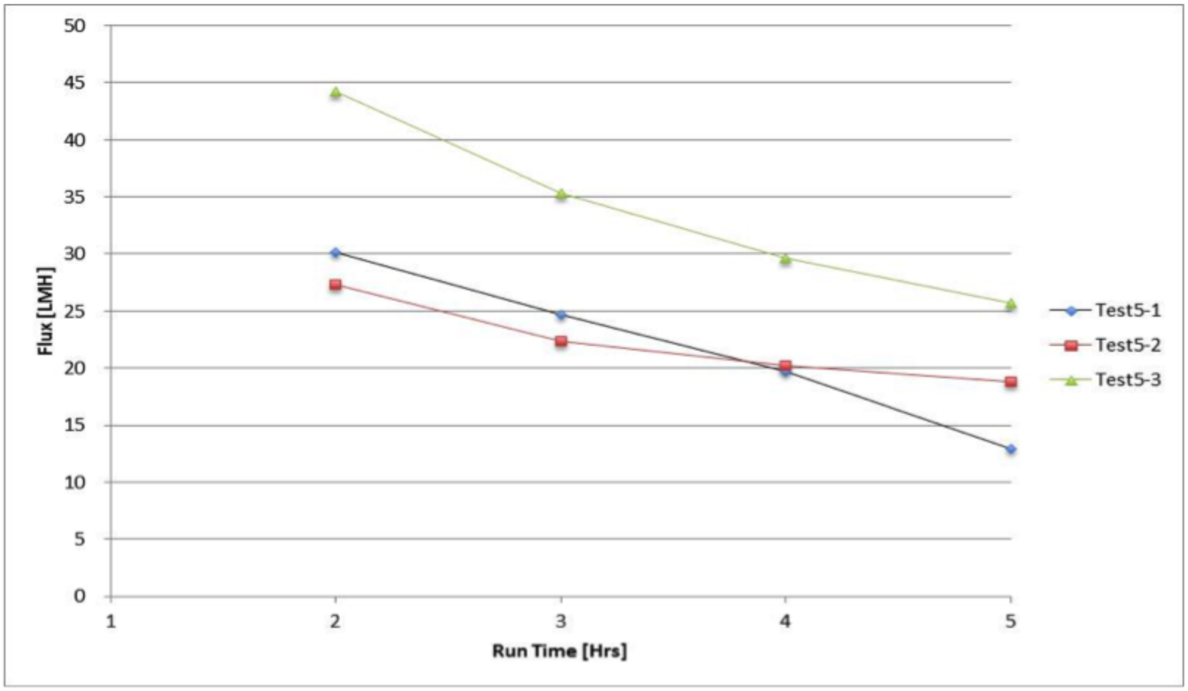

Figure 7. Water flux rate versus run time using the zNANO ML-1 FO membrane, with wastewater in the feed, and 2 mol/L NaCl solution in the brine, Test Setup B.

The ion rejections of both the commercially available and the zNANO ML-1 un-supported membranes are listed in Table . The zNANO membrane shows greater than 90% rejection of potassium, magnesium, and calcium. The zNANO membrane has 82±14%, 88±3%, and 86±17% rejection of ammonium, sulfate, and TOC respectively. However, both the ammonium and TOC data has high standard deviation due to the interference of the TOC and ammonium data in the analysis. The lowest rejection for the zNANO was for nitrates at 75±11%. Nitrite, bromide, and phosphate are present in amounts too low to detect. The rejection values for the commercial membranes are listed here as reference value. These contaminate rejection data show that the zNANO membrane can be used in FO wastewater treatment processes. Since the manufacturing of the ML-1 membrane, zNANO has fabricated a variety of other FO membrane types as well as improve on the ML-1.

Table 4: The table listing the ion rejections of both the commercial as well as the zNANO forward osmosis membrane using secondary wastewater as the feed and 2M sodium chloride as the brine. The result listed is based on the average ions rejection of all the triplicates runs.

Contaminant rejection		Commercial membrane	zNANO ML1
Run time		1 hour	24hours
NH_4^+	%	97 ± 2	82 ± 14
K+	%	98 ± 2	90 ± 7
Mg^{2+}	%	98 ± 1	92 ± 4
Ca^{2+}	%	98 ± 1	92 ± 3
NO^{3-}	%	100 ± 1	75 ± 11
SO_4^{2+}	%	96 ± 3	88 ± 3
TOC	%		86 ± 17
NO_2	low detection limit		
Br^{2-}	low detection limit		
PO_4^{2-}	low detection limit		

IV. Conclusions

Based on our testing, the zNANO membrane has a water flux rate 12x higher than the commercially available membranes when DI water was used in the feed and 2 mol/L NaCl was used as the brine solution. Although the water flux rates decreased when wastewater was used in the feed solution, in such a paradigm, the zNANO membrane still yields a water flux rate 4.4x higher as that of the commercially available membrane. In addition, zNANO membrane showed ion rejection of over 90% for potassium, magnesium, and calcium. These results indicate that the lipid based forward osmosis zNANO membrane has better performance than the commercially available membrane in terms of water flux rates and is competitive in terms of ion rejection. However, more tests are needed to confirm the zNANO membrane integrity over long periods of operation, specific contamination rejections (i.e. urea, ions), and incorporation of the membranes in a variety of different wastewater treatment configurations.

V. Future Works

There are several major areas of future works based on the high water flux results from the zNANO membrane. Due to the unavailability of zNANO's larger sheet of the ML1 FO membrane, two different test setups were used in this experiment. Therefore, to better confirm the data across the variable of membrane size, a larger sheet of ML1 membrane will be tested. Also, zNANO has developed a variety of FO membranes with specialized geometries such as spiral wound modules. These membranes will be tested at ARC to validate the durability of zNANO membrane in waste treatment system. Lastly, the zNANO membranes should be tested for a variety of contaminant rejections for operation in wastewater treatment processes.

Acknowledgments

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Appendix

Appendix-1 Detailed Test Results.

Table 4. Test Results of the commercial membrane for DI water and Wastewater, Setup A

	Test No. 1-1	No. 1-2	No. 1-3	No. 2-1	No. 2-2	No. 2-3
Test Duration[Hours]	1	1	1	1	1	1
Initial/Final EC on feed [S/cm]	7/74	37/345	71/800	19/21	11/12	23/25
Transferred Water Volume [Liters]	0.269	0.255	0.285	0.217	0.197	0.192
Averaged Water Flux [liter/m2/Hour]	7.27	6.89	7.7	5.86	5.32	5.19
Corrected Water Flux at 20 °C Average	7.14	6.84	7.49	5.64	5.05	4.92
Flux Standard Deviation	7.15 0.326, 4.55%			5.2 0.384, 7.39%		

Table 5. Test Results of the commercial membrane for DI water, Setup B

Commercial/DI water	Test No. 3-1
Test Duration [Hours]	16
Transferred Water Volume [Liters]	0.0533
Averaged Water Flux [liter/m2/Hour]	7.84
Corrected Water Flux At 20 degree C	7.55

Table 6. Test Results of the zNANO membrane for DI water and Waste water, Setup B

	No. 4-1	No.4-2	No.4-3	No. 5-1	No. 5-2	No. 5-3
Test Duration[Hours]	5	5	5	6	6	6
Initial/Final EC on feed [S/cm]	0.08	0.0643	0.0435	0.7500	0.6388	0.7435
Transferred Water Volume [Liters]	0.186	0.201	0.185	0.065	0.058	0.096
Averaged Water Flux [liter/m2/Hour]	87.6	94.8	87.1	20.9	20.8	31.4
Corrected Water Flux At 20 degree C	84.8	90.7	82.9	20.1	20.4	28.6
Average Flux	86.1			23.1		
Standard Deviation	4.09, 4.75%			4.8, 20.9%		

Table 7. A table listing the cations, anions, and TOC data for the wastewater run using the commercially available membranes.

Sample I.D.	Na^+	NH_4^+	K^+	Mg^{2+}+	Ca^{2+}	Cl^-	NO_3^-	SO_4^-	TOC
	ppm	Ppm	ppm	ppm	ppm	ppm	ppm	Ppm	ppm
Brine,Test2-1,after 0mins	42881	Nd	nd	nd	nd	76623	nd	Nd	cd
Brine,Test2-1,30mins	40995	Nd	nd	nd	nd	58809	nd	Nd	cd
Brine,Test2-1,60mins	37222	Nd	nd	nd	nd	69518	nd	Nd	cd
WW,Test2-1,0mins	220	27.3	22.4	34.6	37.6	396	65.7	62.1	3.2
WW,Test2-1,30mins	225	27.8	22.8	34.9	36.1	406	70.3	62	3.3
WW,Test2-1,60mins	230	28.3	23.3	35.5	38	416	72.2	66.3	3.8
Brine,Test2-2,0mins	40865	Nd	nd	nd	nd	74203	nd	Nd	cd
Brine,Test2-2,45mins	37606	Nd	nd	nd	nd	68098	nd	nd	cd
Brine,Test2-2,60mins	37245	Nd	nd	nd	nd	67466	nd	nd	cd
WW,Test2-2,0mins	270	45.4	24.9	38.1	39.3	488	56	66.1	3.5
WW,Test2-2,30mins	258	41.9	23.8	36.4	38.6	464	54.4	64.7	3.9
WW,Test2-2,60mins	267	43.6	24.4	37.2	39.6	476	55.5	65.3	3.7
Brine,Test2-3,0mins	41820	Nd	nd	nd	nd	73974	nd	nd	cd
Brine,Test2-3,30mins	37468	Nd	nd	nd	nd	68643	nd	nd	cd
Brine,Test2-3,60mins	37382	Nd	nd	nd	nd	68513	nd	nd	cd
WW,Test2-3,0mins	268	64.8	25.6	38.3	38.5	463	24.1	63.6	3.7
WW,Test2-3,30mins	264	63.8	25.4	38.2	40.6	465	20.6	64.6	4
WW,Test2-3,60mins	269	64.3	25.4	38.6	39.9	480	24.2	67.1	3.8

- nitrite, bromide, and phosphates are non-detected
- nd not detected, amount less than 0.5ppm
- cd cannot be detected due to interference of the chloride concentration

Table 8. A table listing the cations and anions data for the wastewater run using the zNANO FO membranes.

Sample I.D.	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	Cl^-	NO_3^-	SO_4^-
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Brine, Test 5-1, after 0Hours	39822	nd	nd	nd	nd	75544	nd	nd
Brine, Test5-1, 15.5 Hrs	38241	nd	nd	nd	nd	71294	nd	nd
WW, Test 5-1, 0 Hrs	245	34.8	21.8	25.7	29.7	472	82.5	75.9
WW, Test5-1, 15.5 Hrs	996	43.5	23.4	27.2	32.1	1669	103	90
Brine, Test 5-2, 0 Hrs	43687	nd	nd	nd	nd	83263	nd	nd
Brine, Test 5-2, 22 Hrs	41048	nd	nd	nd	nd	73573	nd	nd
WW, Test 5-2, 0 Hrs	112	8.3	9.8	11.1	12.9	212	47.1	36.6
WW, Test 5-2, 22 Hrs	459	8.5	9.4	10.6	12.2	923	76	40.9
Brine, Test 5-3, 0 Hrs	39831	nd	nd	nd	nd	75512	nd	nd
Brine, Test 5-3, 22.5 Hrs	36047	nd	nd	nd	nd	67775	nd	nd
WW, Test 5-3, 0 Hrs	367	5.9	21.4	26.4	30.3	725	136	81.9
WW, Test 5-3, 22.5 Hrs	1428	22.8	26	30.2	34.2	2342	112	73

- Nitrite, bromide, and phosphates are non-detected
- nd not detected, amount less than 0.5ppm
- cd cannot be detected due to interference of the chloride concentration

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