# **Results of the Alternative Water Processor Test, A Novel Technology for Exploration Wastewater Remediation**

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Biologically-based water recovery systems are a regenerative, low energy alternative to physiochemical processes to reclaim water from wastewater. This report summarizes the results of the Alternative Water Processor (AWP) Integrated Test, conducted from June 2013 until April 2014. The system was comprised of four (4) membrane aerated bioreactors (MABRs) to remove carbon and nitrogen from an exploration mission wastewater and a coupled forward and reverse osmosis system to remove large organic and inorganic salts from the biological system effluent. The system exceeded the overall objectives of the test by recovering 90% of the influent wastewater processed into a near potable state and a 64% reduction of consumables from the current state of the art water recovery system on the International Space Station (ISS). However, the biological system fell short of its test goals, failing to remove 75% and 90% of the influent ammonium and organic carbon, respectively. Despite not meeting its test goals, the BWP demonstrated the feasibility of an attached-growth biological system for simultaneous nitrification and denitrification, an innovative, volume- and consumable-saving design that does not require toxic pretreatment.

#### Nomenclature

AWP	=	Alternative Water Processor
ARC	=	Ames Research Center
AWRS	=	Advanced Water Recovery Systems
BWP	=	Biological Water Processor
DI	=	Deionized
DOC	=	Direct Osmotic Concentrator
ECLSS	=	Environmental Control and Life Support Systems
FO	=	Forward Osmosis
FOST	=	Forward Osmosis Secondary Treatment

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GC-TCD	=	Gas Chromatography-Thermal Conductivity Detector
GLS	=	Gas Liquid Sperator
IAWRS	=	Integrated AWRS
IC	=	Ion Chromatography
ISS	=	International Space Station
JSC	=	Johnson Space Center
KSC	=	Kennedy Space Center
LEO	=	Low Earth Orbit
MABR	=	Membrane Aerated Bioreactor
NGLS	=	Next Generation Life Support
OA	=	Osmotic Agent
P/C	=	Physiochemical
RO	=	Reverse Osmosis
SOA	=	State of the Art
SND	=	Simultaneous Nitrification/Denitrification
TIC	=	Total Inorganic Carbon
TN	=	Total Nitrogen
TOC	=	Total Organic Carbon
TTU	=	Texas Tech University
VCD	=	Vapor Compression Distillation

# I. Introduction

Water is the most precious resource to enable human exploration of space beyond low earth orbit (LEO). It is not only used for consumption, but it is used for hygiene activities, as a thermal coolant fluid, and can be catalyzed to provide oxygen to a crew. Water represents the majority of up-mass for all ECLSS components, comprising up to 90% of the mass for a given mission (Liskowsky, 2010). Because of its importance to life support systems, the energy requirements to transport water into space, and the impracticality of launching the entire volume needed for a long duration mission, the ability to reuse water is paramount. Per the Space Technology Human Health, Life Support and Habitations Systems Roadmap, an integrated water recovery system needs to be capable of >95% water recovery by 2022[1].

There are two general mechanisms that are used to reclaim water from wastewater. The first are strictly physiochemical (P/C) processes. The current state of the art (SOA) P/C process is vapor compression distillation (VCD), which is currently practiced on the International Space Station. Physiochemical processes, such as distillation, typically require elevated temperatures and operate at non-atmospheric pressures and require a significant amount of consumables to keep the hardware operational. The most toxic consumable for VCD operations is a sulfuric/phosphoric acid/hexavalent chromium solution which stabilizes the urine to prevent urea hydrolysis and control biological growth[2]. Once the wastewater is stabilized, the waste constituents are concentrated via distillation. That toxic residual mixture (i.e. brine), containing water, must be discarded, leading to lower overall water recovery rates[3].

An alternative to a strictly P/C system is a biologically-based process which uses bacteria to transform the wastewater constituents into products which can be reused for other life support activities. Biological systems require less energy than P/C systems because bacteria use their inherent metabolic processes to transform the wastewater. Additionally, biological systems operate at ambient temperatures and require only slightly pressures (10-20psig) for microgravity compatibility. Because the constituents are transformed rather than stabilized, the toxic chemicals that are used to prevent urea hydrolysis are not needed and the metabolic by-products ( $CO_2$  and  $N_2$ ) can be used for other life support activities.

In 2011, the Next Generation Life Support (NGLS) Project initiated the Alternative Water Processor (AWP) Element, the objective of which was to develop a next-generation water recovery system using a biological water processor (BWP) as the primary water treatment subsystem and a forward and reverse osmosis process as a secondary subsystem to reclaim water from wastewater. The Alternative Water Processor Element was a joint effort among scientists and engineers at Texas Tech University (TTU), NASA's Johnson Space Center (JSC), NASA's Kennedy Space Center (KSC) and NASA's Ames Research Center (ARC). The primary objectives of the AWP Integrated Test were as follows:

1. Demonstrate minimum of 85% water recovered during steady state conditions from a wastewater stream

2. Demonstrate a 20% reduction in consumables from the current, SOA physiochemical system

This was the first biologically-based integrated water recovery test to be performed at JSC since the Integrated AWRS (IAWRS) Test 2001 and was the culmination of several years of research, development, and testing at ARC and TTU to develop the design for the primary and secondary systems, respectively.

# **II.** System Description

The AWP was made up of two subsystem, the Biological Water Processor and the Forward Osmosis Secondary Treatment System, or the FOST.

#### A. Biological Water Processor (BWP)

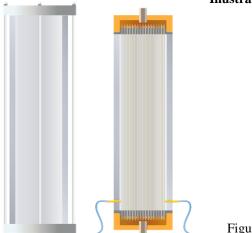
The BWP was responsible for removing the organic carbon and nitrogen from the waste stream via nitrification and denitrification/carbon oxidation. The BWP was comprised of four membrane aerated bioreactors (MABR).

The objective for BWP testing was to demonstrate the use of a membrane-based biological subsystem for simultaneous organic carbon removal and nitrification of ammonia as the initial process for potable water recovery. Specifically:

- 1. Achieve, at minimum, 75% ammonium and 90% organic carbon removal as compared to the influent waste stream
- 2. Quantify the production of metabolic gases, such as carbon dioxide and nitrogen, as biological subsystem byproducts

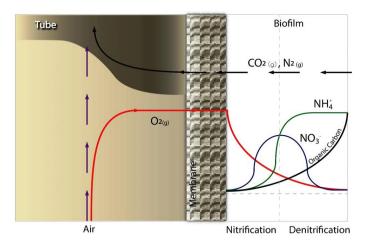
MABR design concept as applied to spacecraft water recovery was developed in the Jackson Lab at TTU[4]. The original design at TTU was a single cylinder containing gas permeable membranes submerged in a wastewater. At the top and bottom of the reactor were gas (oxygen) plenums which facilitated gas transfer through the membranes, and subsequently the reactors, for carbon oxidation and nitrogen removal to take place. The influent entered at two ports at the bottom of reactor and the effluent left at two ports at the top of the reactor.

Testing at TTU identified several areas for improvement which were included in the JSC design. The first change was the addition of a second shell to provide additional structural support to minimize stress on the membranes. During testing at TTU, the single-shell reactor developed a torque; the membranes and the reactor developed a slight rotation due to the stress the membranes applied to the inlet and outlet gas plenums. In response to this malfunction, JSC designed a means of structural support by encasing the membranes before submerging into the wastewater. The design was coined the "clamshell". A second modification to TTU's original design was to induce additional mixing within the reactors. The TTU team observed "dead spots" in the reactor; places of little to zero flow where inert biomass would settle. This effectively reduced the active volume within the reactor. In order to counteract that occurring in the JSC system, the outlet ports were offset 90° from the inlet ports to aid in mixing through the tube bundle.



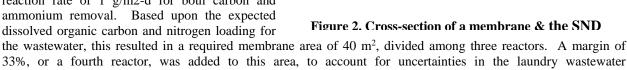
Illustrations of the reactor design and clamshell are illustrated in

Figure 1.



#### Figure 1. MABR and Clamshell design

The oxygen header provided air or oxygen to 506 platinum-cured silicone, hollow tube membranes (Dow Corning, P/N 508-010). Each of the four MABRs has a free liquid volume of 63.9 L, which is reduced to 55 L working volume when the tubes are incorporated. The tubes provide 11 m<sup>2</sup> of surface area for aeration and biofilm support. The resulting surface area to volume ratio, a critical sizing parameter for attached growth  $200 \text{ m}^2/\text{m}^3$ . Each air plenum MABRs was contained a Viton O-ring and stainless steel rods to seal the pressurized liquid chamber through compression. The MABR was sized based upon a reaction rate of 1 g/m2-d for both carbon and ammonium removal. Based upon the expected dissolved organic carbon and nitrogen loading for



33%, or a fourth reactor, was added to this area, to account for uncertainties in the laundry wastewater characteristics at the time. This membrane area was divided among four reactors for ease of fabrication and assembly.

The MABR utilized a process known as simultaneous nitrification and denitrification, or SND, to remove

organic carbon and nitrogen from the wastewater. Terrestrial processes for suspended-growth biological nitrification and denitrification are typically decoupled and carried out in separate tanks, where the conditions for nitrification and denitrification can be independently controlled. MABR, the complementary processes of nitrification and denitrification/carbon oxidation be coupled into a stratified biofilm attached to a membrane. Figure 2 is an illustration of a crosssection of the membrane at the membrane-biofilm interface and the complementary chemical processes that are occurring.

Breathing air was supplied at flow rates ranging from 50 to 1000 sccm and 10-20 psi on



the

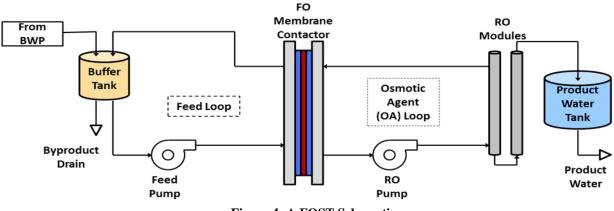
inside of the hollow, tubular membrane. Ambient oxygen diffused through the membrane into the bulk fluid. Nitrifying bacteria, located on the membrane surface and at the interior of the stratified biofilm, utilized the oxygen to oxidize ammonium to either nitrite or nitrate). The heterotrophic, denitrifying bacteria on the exterior of the

biofilm and in the bulk fluid reduced the oxidized ammonium to nitrogen and carbon dioxide gas[. The gas diffused back into the membrane due to the slightly elevated liquid pressure, and vented to the laboratory environment. In a spacecraft with a closed loop life support system, the product gases can theoretically be returned to the cabin environment for atmosphere recovery and reuse. It should be noted that the AWP system was designed to be able to utilize air or pure O2 as reactor gas. Shortly after the reactor entered nominal flow-through operations, the test team decided to transition from pure oxygen to breathing air. The rationale for this change will discussed later.

#### **B. FOST**

The FOST was the secondary subsystem of the AWP and used forward and reverse osmosis technologies to remove inorganic contaminants from the BWP effluent. It was developed and built by scientists at NASA's Ames Research Center (ARC) in California. FOST is the next generation of staged forward and reverse osmosis technology first used in the Direct Osmotic Concentration (DOC) System, tested at JSC in 2010-2011. The objective of FOST testing was to demonstrate the use of a forward osmosis/reverse osmosis membrane configuration as a method of selective filtration for concentrating and removing inorganic constituents from a processed water stream. A simplified schematic of the FOST is given in Figure 3.

The FOST was comprised of two pumps; a feed pump that transferred feed through a Forward Osmosis (FO) membrane, and a high pressure (500-1000psi) Reverse Osmosis (RO) pump which drove the FO product through the RO membrane. Two FO membranes were used during FOST testing; a spiral wound membrane from Hydration Technologies Inc (HTI model number 4040FO-MS) and a flat sheet membrane from Porifera. The RO membrane was a commercial RO membrane system from Katadyn (80E)[5].



**Figure 4: A FOST Schematic** 

The function of the FOST was to remove dissolved solids, ammonia, and suspended solids from the BWP effluent. During the IAWRS test, the secondary system consisted of a single RO unit. One notable issue observed during testing was fouling in the RO system, leading to lower rejection rates, lower permeate recovery and subsequently, lower permeate quality[6]. In order to mitigate this issue, the FOST incorporated a forward osmosis component upstream of the RO in order to counteract the previously-observed detrimental effects of fouling. Forward osmosis (FO) uses the osmotic potential between two fluids of differing solute/solvent concretions to drive a solvent from a less concentrated solution to a more concentrated solution via a semi-permeable membrane. In this

application, the FO rejects large organics and ammonia, such as those that are typically present in a biological effluent, allowing the water and ionic species to pass through. Because the colloids and other high molecular weight organics are left behind, the highly sensitive RO membrane is preserved, increasing the time between fouling events, and thereby prolonging the life of the RO membrane and reducing consumables[5]. The RO then rejects the smaller-sized ionic species to produce near-potable water.

Once the BWP effluent entered the common collection tank between the two sub-systems (aka buffer tank), the FO pump drove BWP effluent through the FO membrane and into a concentrated osmotic agent (10g/L NaCl). Once the osmotic concentration

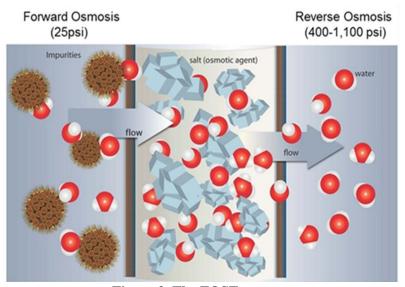


Figure 3. The FOST process.

between BWP effluent and the (now) diluted osmotic agent equalized, the reverse osmosis (RO) pump used high pressure to drive permeate from the OA product the RO membranes, removing salt and inorganic ions and producing high-quality product water. During the process, the osmotic agent was re-concentrated and was subsequently able to be reused for multiple test runs.

# **III.** Test Decription

#### A. Wastewater Description

Only pretreated urine, flush water, and humidity condensate are reclaimed for consumption on the ISS. The wastewater composition processed by the AWP was an exploration waste stream, comprised of urine (minus the pretreatment), flush water, and humidity condensate as above, but also hygiene and laundry wastewaters. Table 2 provides an overview of the composition and volume of each type of wastewater based on a 4-person crew (3 male,

1 female), the estimated size of a crewed exploration mission beyond LEO. The values below are based upon historically observed output for ISS and were previously used in other advanced life support testing[7, 8].

Wastewater (WW) Type		WW Per Event (kg/event) in liters	Events Per Day Per Crewmember (event/day-CM)	Total WW vol for Four Crew (kg/day-crew)	Personal Care Products
U	rine	1.2 (per day)	N/A	$4.8^{8}$	
	Oral Hygiene	0.1	2	0.8	1.0 g of Arm & Hammer Toothpaste
ne	Hand Wash	0.125	8	4.0	No-Rinse Shampoo, NASA
Hvgiene	Shower	6.0	1	24.0	Formulation. 1.5 g for hand wash, 25.0 g for shower
H	Shave	0.15	1	0.15	0.8 g Neutrogena Men Shave Cream
	Urinal Flush	0.3 (per day)	N/A	1.2	
	umidity ondensate	1.95 (per day)	N/A	$7.8^{9}$	
L	aundry	30	N/A	15 kg per day on a 2-day cycle	15 g of Seventh Generation Natural 2X Concentrated Laundry Liquid (Free and Clear)
			TOTAL	57.75	

Table 1. Baseline wastewater constituents and volumes based on a 4-person crew

The total urine production per crew per day for the AWP test was base lined at 4.8 liters per day. However due to the high concentration of nitrogen needed to adequately mimic on-orbit urine, the donor urine was augmented to achieve 10 g/L total nitrogen (TN). Therefore, the total contribution of urine to the BWP influent was 9.1 L. The additional 4.3 L, referred to as "augmentation", was required to take in account the differences in concentration between the urine generated by a crew in microgravity and the urine generated by donor pool of well-hydrated individuals in a terrestrial office environment. It has been well documented that urine contributed in microgravity contains higher concentrations of organic and inorganic compounds, including a concentration of 10 g/L TN [9]. In order to reach the TN target without adversely affecting the total volume processed per day, an additional 4.3 liters of urine displaced 4.3 L of DI water as part of the humidity condensate contribution. The daily humidity condensate contribution is 7.8 L, which is created using 78 mL of a concentrate solution in 7.722 L of DI water. Laundry was performed every other day and the volume of laundry water was split between the two days in order to prevent unnecessary fluctuations in wastewater composition. The volume of laundry water was 30 L per use; 15 L of laundry wastewater were contributed to the BWP daily.

#### **B.** Sample Collection, Analysis and Monitoring

### 1. Sample Collection

The activity of the system was monitored in three ways; in-line sensors, liquid and gas analysis. Inline pH and dissolved oxygen (DO) concentrations were monitored using a bioluminescent dissolved oxygen probe (Hach 9020000) and pH sensor (Hach DPD1P1). The probes were installed in the recycle loop of the BWP to provide real-time data on system operations. Data was collected each second and transferred via a LabVIEW data acquisition (DAQ) and control system. In addition to recording data, the controls system allowed for manual control of the reactor; control of the feed transfer and influent pumps, and manual control of the gas mass flow controllers and pressure controllers to supply facility air/oxygen to the reactors and allowed for the automatic switchover to backup gas supplies in the event of facility gas loss. Liquid samples from the BWP were collected each morning from the feed tank and the gas liquid separator (GLS) for pH, total organic and inorganic carbon (TOC/TIC), total nitrogen (TN), and inorganic ions via ion chromatography (IC). The effluent gas from each MABR and the GLS were

<sup>&</sup>lt;sup>8</sup> Increased urine volume to 9.1 liters to take in account the 10g/L nitrogen present in crew urine. The volume of the humidity condensate make-up water was reduced to accommodate the increased urine volume to maintain the overall daily wastewater volume

<sup>&</sup>lt;sup>9</sup> Refer to footnote 1

analyzed using a 3000A Micro Gas Chromatograph with a Thermal Conductivity Detector (GC-TCD) from Inficon (Agilent) with 4 channels to measure the concentration of N2, O2, CO2 and N2O. FOST feed pH was monitored using a Hach DPD1P1. The probe was placed within the tank to titrate the feed pH as needed. Liquid samples from the FOST were collected from each run. Feed, brine and product samples were analyzed for pH and inorganic ions via ion chromatography (IC).

# **IV.** Test Summary

The AWP test began with the inoculation of the BWP on April 19th, 2013. The composition of the BWP volume at inoculation was as follows: 47 liters of inoculum, 6 liters of urine in 167 liters of deionized (DI) water, and 30 grams of ammonium bicarbonate as an additional source of ammonium and inorganic carbon for a total volume of 220 liters. The inoculum source was a culture from Texas Tech University collected from an inoculum tank acclimated to urine and cultivated at JSC. The system was placed on recycle in order to establish a nitrifying biofilm on the surface of the silastic membranes. Pure oxygen was supplied to the reactors at a pressure between 6-10 psi(g) and a flow rate of 50 sccm.

Seventy-one (71) days after inoculation, the BWP transitioned to processing a full exploration wastewater solution (Table 1). The period between inoculation and the processing of a full wastewater was significantly longer than expected. The inoculation of the IAWRS bioreactor took approximately 30 days and the test team anticipated that the duration to inoculate the BWP would be on the same scale. For the first 32 days after inoculation, the system was in recycle and was fed only if there was a loss of fluid in the reactor due to gas accumulation or to introduce additional inoculum. The steps from inoculation to processing a full wastewater are summarized in Table 2. Transition from inoculation to full wastewater.

Day After Inoculation	Date	Step	Percentage of daily urine volume added	Influent composition
34	5/23/1013	First	11%	1 liter of urine in DI
36	5/25/2013	Second	50%	4.6 liters of urine in DI
40	5/29/2013	Third	22%	2 liters of urine in DI
43	6/1/2013	Fourth	66%	6 liters of urine in DI
45	6/3/2013	Fifth	100%	9.1 liters of urine in DI
71	6/28/13	Sixth	100%	9.1 liters of urine in wastewater

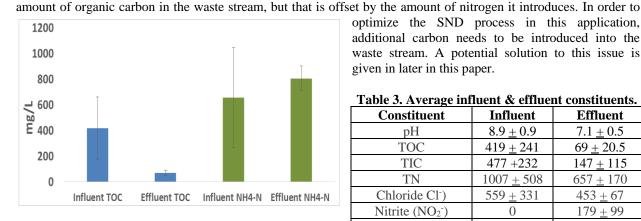
Table 2. Transition from inoculation to full wastewater.

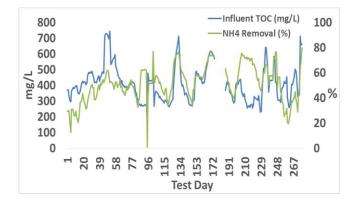
The BWP was in operation as a four-reactor system for 283 consecutive days, processing approximately 12,000 liters of wastewater. The FOST was operated, non-consecutively, for 30 days as compared to 283 days of BWP operation. The limited number of FOST test days in comparison to the BWP was three-fold. First, the FOST was only intended to operate with BWP effluent that had been processed from a full wastewater strea. As a result, the initial operation was delayed by 71 days. Secondly, since the FOST was built at ARC and delivered to JSC for testing, operators at JSC needed time for system check-out and troubleshooting. Third, due to the high pressures required of the reverse osmosis process, was operated during traditional work hours. As a result, no testing occurred during flex Fridays, weekends, holidays, and the government furlough in October 2013.

### **IV.** Results

#### A. BWP

A summary of the influent and effluent is given in Table 3. Average influent & effluent constituents. The average influent concentration of organic carbon during testingwas 419 mg/L, while the effluent concentration was 69 mg/L. Concentrations of influent and effluent ammonium were 806 and 358 mg/L, respectively. This corresponds to a TOC removal rate of 83.6 % and 55.5% of influent NH4. The removal efficiencies were below the success criteria for this test, which were 90% and 75% removal of influent organic carbon and ammonium nitrogen, respectively. Ammonium removal generally trended with the concentration of TOC present in the influent waste; higher organic carbon concentrations leads to higher ammonium removal rates. Carbon is required for both nitrification and denitrification. Inorganic carbon is needed as an alkalinity source for nitrification and for the generation of bacterial cells, since nitrification is an autotrophic, rather than a heterotrophic reaction. Denitrifying bacteria break down organic carbon in the presence of nitrite and/or nitrate. Influent TOC trends with Influent TN, logical, since urine comprises 16% of the influent volume. This leads to a bit of a conundrum, urine introduces a significant





#### Figure 6. Seven day running average of influent TOC and ammonium removal.

and use of the proprietary software with the system.

Table 4. Effluent gas concentrations provides a summary of the average concentration of effluent gas components, while a snapshot of reactor activity in a given week is presented in Figure 7.

Figure 7 shows the effluent CO2 and N2O concentrations from each each reactor. CO2 and N2O are product gasses from the SND processes; CO2 is the product from carbon oxidation, while N2O is the intermediate gas

optimize the SND process in this application, additional carbon needs to be introduced into the waste stream. A potential solution to this issue is given in later in this paper.

Table 3. Average	influent &	effluent	constituents.

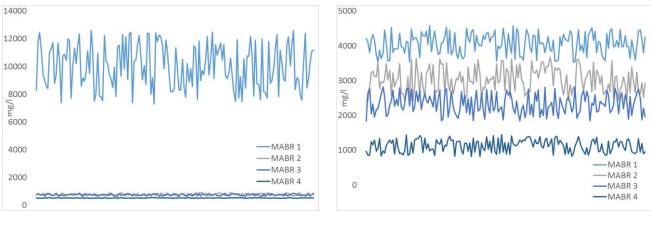
Table 5. Average influent & efficient constituent									
Constituent	Influent	Effluent							
pH	8.9 <u>+</u> 0.9	7.1 <u>+</u> 0.5							
TOC	419 <u>+</u> 241	69 <u>+</u> 20.5							
TIC	477 +232	147 <u>+</u> 115							
TN	1007 <u>+</u> 508	657 <u>+</u> 170							
Chloride Cl <sup>-</sup> )	559 <u>+</u> 331	453 <u>+</u> 67							
Nitrite (NO <sub>2</sub> <sup>-</sup> )	0	179 <u>+</u> 99							
Nitrate (NO <sub>3</sub> <sup>-</sup> )	0	81 <u>+</u> 68							
Phosphate (PO <sub>4</sub> <sup>3-</sup> )	153 <u>+</u> 86	126 <u>+</u> 20							
Sulfate (SO <sub>4</sub> <sup>2-</sup> )	148 <u>+</u> 104	142 <u>+</u> 21							
Sodium (Na <sup>+</sup> )	367 <u>+</u> 198	294 <u>+</u> 59							
Ammonium (NH <sub>4</sub> <sup>+</sup> )	806 <u>+</u> 390	358 <u>+</u> 96							
Potassium (K <sup>+</sup> )	323 <u>+</u> 156	247 <u>+</u> 37							
Calcium (Ca <sup>2+</sup> )	98 <u>+</u> 109	36 <u>+</u> 35							
Magnesium (Mg <sup>2+</sup> )	47 <u>+</u> 38	23 <u>+</u> 7							

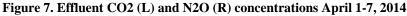
Effluent gas samples were collected every hour from each MABR and from the system GLS starting on January 24th. The delay was due to primarily to installation issues. Agilent was the original vendor for the micro GC when it was first purchased. Between the date the system was purchased to the date it was to be used as a part of the AWP test, the technology was acquired by Inficon. The vendor had to become aquaited with the specifics of that model, which led to problems with installation of the device

MABR	able 4 Eff	O <sub>2</sub> luent <u>cas</u> co	CO2 ncentratio	N2O ons(ppm)
1	663344	1709424	5802	2557
2	708773	1916107	952	2563
3	702139	1902777	474	1625
4	701540	1793121	574	924

produced during denitrification. Two things stand out. One, the production of CO2 and N2O is greatest in the first reactor, particularly CO2 production. The operation of the system was to maximize mixing between all four reactors and essentially having a continuously stirred tank reactor system (CSTR) broken up between four reactors. That was not the case. Second, the gas production continuously fluctuates. In order to determine whether the fluctuation was a result of the 16 hour feeding cycle, we plotted gas production with the inline dissolved oxygen (DO) measurements during a 24-hour period in the middle of that week. DO is a good representation of how the reactor operated on a daily basis. When the system was

processing wastewater, DO dropped due to consumption by the microorganisms in the reactor. Once feeding was complete, the DO would rise. In this instance, there does not seem to be a correlation between gas production and feeding cycles. As stated above, due to technical difficulties with installation of the GC the number of gas samples collected during the 4 reactor system is limited. Additional analysis of the data generated during the loading and rapid start evaluation will be presented in a subsequent test report.





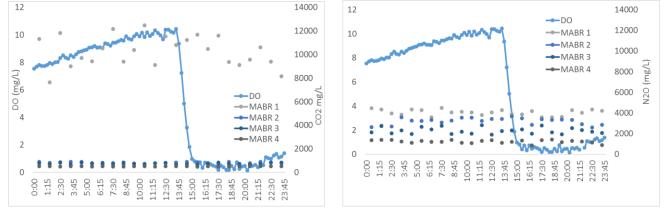


Figure 8. DO and effluent CO2 (L) and N2O (R) concentration over a 24-hr period (April 5, 2014)

# **B. FOST**

Two FO membranes were used during testing; a Hydration Technology Innovations (HTI) membrane and a FO membrane from Porifera. Since testing with the Porifera membrane occurred after the completion of integrated testing in a 4 reactor system, this report will focus on data generated from the HTI membranes. Tables 4 and 5 are a summary of the performance of the FOST using the HTI membrane, which accounted for 13 of the 30 test runs. Initial test runs were performed without the addition of acid (Table 7), followed by test runs to lower the pH of the BWP effluent (Table 8). The addition of an acid aids in the rejection of ions through the FO module, particularly ammonium.

	Table 5. FOST performance summary with un-aciditied BWP effluent.													
	pН	K	Cl-	$NO_2^-$	NO <sub>3</sub> -	PO4 <sup>3-</sup>	$SO_4^{2-}$	Na <sup>+</sup>	$NH_4^+$	$K^+$	Ca <sup>2+</sup>			
Feed	7.64	5.55	448	107	89	147	187	301	412	278	94			
Permeate	8.47	< 0.01	97	1.4	1.5	1	2	61	42	4	< 0.5			
% Removed			78.4	98.7	98. <i>3</i>	99.6	99.2	79.7	89.9	<i>98.5</i>	<99.5			

 Table 5. FOST performance summary with un-acidified BWP effluent.

	Table 6. FOST performance summary with acidified BWP effluent.													
	pН	Κ	Cl	$NO_2^-$	NO <sub>3</sub> -	PO4 <sup>3-</sup>	$SO_4^{2-}$	Na <sup>+</sup>	$\mathrm{NH_{4}^{+}}$	$\mathbf{K}^+$	Ca <sup>2+</sup>			
Feed	5.71	5.23	987	89	68	204	181	322	179	487	157			
Permeate	5.50	< 0.01	22.1	< 0.5	< 0.5	< 0.5	< 0.5	108	< 0.5	< 0.5	< 0.5			

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% Removed		978	<994	< 99 3	<99.8	< 99 7	66.4	< 99 7	< 99 9	<99.7
/o Kemoveu		27.0	>>>.+	< 77.J	>>>.0	>>>./	00.4	>>>./	>>>.>	>>>./

Table 7. Comparison of cit	of actum	icu anu un	-actuilleu	Divi cillu		JIK Stanut	11 U.S.	
	pН	Cl-	$NO_2^-$	$NO_3^-$	$SO_4^{2-}$	Na <sup>+</sup>	$\mathrm{NH_4^+}$	$\mathbf{K}^+$
HSIR std	4.5-9.0	250	1	10	250	322	1	340
Permeate	8.47	97	1.4	1.5	2	61	42	4
Acidified Permeate	5.50	22.1	< 0.5	< 0.5	< 0.5	108	< 0.5	< 0.5

There is a significant reduction of the ionic species in the permeate in both the un-acidified and acidified feed; however nearly all the ions are below the detection limit in the pH adjusted feed. How this impacts consumable usage is described in the following section.

# V. Calculation of System Metrics

Two system metrics were defined for the Alternative Water Processor (AWP) based on the tested exploration wastewater load: (1) percent wastewater recycling (or percent water recovery), and (2) percent consumable reduction from state-of-the-art (SOA) ISS water processor technologies. This section describes the calculation and results of these metrics

Table 8. A summary of consumables of the SOA water recovery technology and the Awr			
	Consumable	Consumables Estimate (upmass / L water recovered)	Assumptions and Rationale
SOA	Multifiltration and ion exchange beds	20 g	Bed lifetimes were based on an early space station projection for a feed that included hygiene wastewater [10].
BWP & FOST consumables	Oxygen (21% in air)	$2.9\pm0.2$ g	BWP oxygen consumption was estimated from the nominal air feed rate (assuming 21 vol% $O_2$ in air) and measurements of the effluent gas $O_2$ concentration.
	Salt (NaCl)	$0.8\pm0.8~{ m g}$	FOST OA NaCl losses were estimated from mass balances using sample analysis data from 6 FOST runs with the HTI FO membrane.
	Acid	10.6 ± 6.1 g for 1 M H <sub>3</sub> PO <sub>4</sub>	Acid volume required to adjust the feed (BWP effluent) pH depended strongly on BWP effluent conditions, target pH, and type and strength of acid solution employed. Consumables were calculated from test volumes and then extrapolated based upon BWP effluent averages.
		3.4 ± 2.0 g for 5 M HCl	Acid volume required to adjust the feed (BWP effluent) pH depended strongly on BWP effluent conditions, target pH, and type and strength of acid solution employed. Consumables were calculated from test volumes and then extrapolated based upon BWP effluent averages.

Table 8. A summary of consumables of the SOA water recovery technology and the AWP

Water loss from the BWP is negligible, so the system water recovery is equal to the FOST water recovery. As previously discussed, two forward osmosis membranes were tested during FOST operations with BWP effluent, each producing different average water recovery. With a Hydration Technology Innovations (HTI) FO membrane,

the FOST achieved an average of  $90.1 \pm 7.6\%$  water recovery over 17 runs. With a Porifera FO membrane, the FOST achieved an average of  $79.1 \pm 14.2\%$  water recovery over 17 runs. Overall, the FOST achieved  $84.6 \pm 12.5\%$  water recovery. Results with the HTI FO membrane are assumed to best represent the FOST capability. The observed differences with FO membrane type could be due to a number of factors, including membrane sizing and upstream BWP performance.

The consumable reduction calculation involves estimating both SOA consumables for an exploration-like wastewater load and AWP consumables based on test data. For the metric calculation, only multifiltration and ion exchange beds were included as SOA consumables. Bed lifetimes were based on an early space station projection for a feed that included hygiene wastewater [10]. The resulting estimate was 20 grams per liter (g/L) of water produced.

In comparison, AWP consumables were assumed to include BWP oxygen consumption, salt (NaCl) losses from the FOST osmotic agent (OA), and acid required for FOST feed pH adjustment. Any consumables required for polishing FOST product water to potable status were not included. These contributions, listed in Table 9, illustrate the individual AWP consumables estimates. A worst case estimate is  $14.3 \pm 6.2$  g/L of product water, a 28% reduction from SOA, which includes phosphoric acid for the FOST pH adjustment. A best case estimate is  $7.1 \pm 2.1$  g/L of product water, a 64% reduction from SOA. This best case includes 5 M HCl used for FOST pH adjustment.

BWP oxygen consumption was estimated from the nominal air feed rate (using an average O2% from effluent gas samples) and measurements of the effluent gas O2 concentration. The resulting consumable estimate was  $2.9 \pm 0.2$  g/L of AWP product water at 90% water recovery.

Best estimates of the AWP system metrics are summarized in Table 1and compared to project threshold values and research and technology development (R&TD) goals.

#### VI. Discussion

Although the AWP did succeed in its overall objectives to reclaim at least 90% of the influent waste stream and to lower consumable mass by more than 20 %, optimization of the individual subsystems is still necessary to achieve >95% water recovery as laid out in the Space Technology Human Health, Life Support and Habitations Systems Roadmap [1]. The BWP removed only 55.6 % of the influent NH4 and 83.6% organic carbon, which is below the success criteria for the BWP of 75% and 90% respectively. It is likely that incomplete nitrogen and carbon removal is due to carbon scarcity. Carbon is needed for both cell growth, respiration and nitrification [11]. The carbon to nitrogen (C:N) ratio of influent organic carbon to ammonia during the AWP test was 0.55:1, which is weighted heavily towards nitrogen. Most literature suggests a C:N ratio of 3-5:1 for optimal nitrification; however Terada et al. observed significant removal of TOC and nitrogen at a ratio as low as 1.5:1 [12].

Water recovery testing at JSC has demonstrated high carbon and nitrogen removal at lower carbon to nitrogen ratios as well. During the IAWRS test in 2000, the C:N ratio was 0.8:1 and the associated ammonium and TOC removal was 75% and 90% in a two reactor system separating nitrification and denitrification [6]. The predominance of nitrogen in the influent is due to the increase in urine as a fraction of the wastewater to adequately mimic the composition of concentrated urine on orbit and the limited amount of carbon introduced during hygiene activities. In addition, the introduction of the laundry wastewater, which introduced some carbon in the form of laundry detergent, diluted the overall wastewater by providing more water volume with limited soap.

Since the efficiency of the reactor is dependent on the availability of carbon substrate, the AWP system should consider incorporating additional carbon. While increasing the volume of carbon as soap in the laundry and hygiene fraction of the exploration wastewater stream is ill-advised, as it would merely increase mission consumables, an exploration BWP may incorporate other carbon-rich wastewaters. Solid waste leachate is the liquid extracted from solids processing and is a subsystem in an advanced ECLSS architecture. Lechate is high in organic carbon, with concentrations typically in the 1000-3000 mg/l range (Damiano, Jambeck, & Ringelberg, 2014). A small volume of leachate added to the influent can provide the needed carbon to optimize the biochemical process and further close the life support loop.

#### A. Lessons Learned

Testing did demonstrate that biologically-based water recovery subsystem combined with a secondary osmotic membrane system is a potential candidate in an ECLSS architecture portfolio. The AWP integrated test demonstrated that the fundamental processes are capable of high water recovery, low consumable, near-closed loop systems when compared to the SOA. However, there are multiple areas requiring improvement.

Incomplete nitrification, that is nitrite  $(NO_2)$  as the final product rather than nitrate (NO3) is sufficient for carbon and nitrogen removal. Both nitrite and nitrate can be used as terminal electron acceptors for dentrification/carbon

oxidation. Incomplete nitrification uses less oxygen to convert ammonium and the system and operate at a wider range of free ammonia (FA) concentrations. Free ammonia concentrations at < 1 mg-N/L inhibits nitrite oxidation, < 10 mg/L inhibits ammonia oxidation [13]. During testing, FA concentration mostly remained below concentration that is inhibitory to ammonia oxidation but above concentration typically associated with inhibition of nitrite oxidation. By using nitrite as a terminal electron acceptor, there is a more robust consortium and there are mass savings in O2.

The duration from inoculation to processing a full wastewater solution observed during testing is unacceptable for a long duration exploration mission and is one of the primary reasons there is hesitation with using a BWP as a primary water processor for exploration applications. The causes of extended inoculation period during AWP testing are two-fold:

First, the test team's assumptions of the inoculation protocol were significantly off. The team assumed that the nitrification inoculation protocol from the IAWRS test would be sufficient for this test. In a SND system, it is necessary to initiate nitrification and subsequently establish a robust nitrifying biofilm on the membranes so once the surfactant components are introduced heterotrophic denitrifying bacteria will not outcompete the slow metabolizing autotrophic bacteria, so it is important to have a sufficient quantity of bacteria to seed the system. The surface area to volume ratio for the nitrifying reactor during the IAWRS test was an order of magnitude lower than the SA:V for the AWP test;  $13 \text{ m}^2/\text{m}^3$  for the IAWRS test as compared to 200 m2/m3 for the AWP test. Larger surface areas requires a high concentration of inoculum to populate the surface of the membrane and we did not account for that.

During the summer of 2013, team members at TTU began to evaluate different inoculation methods to reduce the time from inoculation to nominal processing. They observed that filling a reactor with filtered inoculum and allowing individual cells to colonize a surface in a hospitable liquid environment reduced the time from inoculation to steady-state operations. Details regarding applying the TTU method at JSC are summarized in "Rapid Start-up and Loading of an Attached Growth, Simultaneous Nitrification/Denitrification Membrane Aerated Bioreactor [14]and details will be presented in a subsequent test report.

The second cause for delay was using pure O2 under pressure during inoculation. Pure O2 under pressure, which was to ensure gasses dissolved into solution, caused the DO to constantly exceeded 20 mg/L, levels which are toxic to microorganisms [15]. In order to reduce the concentration of DO in the system but provide sufficient oxygen, the test team transitioned the BWP to breathing air starting on May 2nd, 13 days after inoculation. Since the system was designed for oxygen use only, gas transfer lines needed to be built from the facility air source in the lab to the reactors, so the system was fed air from K-bottles at first. The gas flow rate remained at 50 sccm until May 29th after discussions with colleagues at TTU. After increasing the flowrate, the system was able to handle increased volumes of urine until it was able to process nominal feed, including hygiene. The lessons learned from inoculating the BWP were applied to a subsequent evaluation to identify a method or methods that would reduce inoculation time. Those results are also presented in "Rapid Start-up and Loading of an Attached Growth, Simultaneous Nitrification/Denitrification Membrane Aerated Bioreactor" [14].

This second observation allowed the JSC team to conclude that pressurized air is more suitable for an SND reactor than pressurized pure O2. Materials compatibility for system and operator safety is easier if pure oxygen under pressure can be avoided. In addition, operators can have finer control of oxygen concentration in the reactors without concern for oxygen toxicity and can maximize SND process and minimize use of breathing air as consumable.

In addition to the scientific lessons learned related to the establishment of nitrification, there were some mechanical difficulties that impacted day-to-day operations. These mechanical lessons learned are (1) condensation in the gas lines due to the pressure differential of a gas/liquid system and (2) biomass accumulation affecting fluid flow.

Condensation in the gas lines was a chronic issue throughout testing. Due to the positive pressure differential between the liquid and gas phases, water vapor passed through the membranes along with the other product gases. The accumulation of condensate damaged the mass flow controllers on the gas outlet of each reactor, and intermittently blocked gas flow out of the reactor. The MFC's were relocated so that the condensate would not pool at their inlet, but the high humidity continued to pose problems for the electronic components of the MFC. Condensation also affected GC analysis due to water vapor buildup in the system. The periodic "bake-out" periods to dry out the columns were insufficient, which led to the GC being taken offline for maintenance. During the rapid start evaluation, the test team added desiccant beds at the outlet of the reactors upstream of the mass flow controllers to prevent humidity exposure. The impact of the desiccant beds will be discussed in a subsequent test report on the rapid start evaluation.

#### **B.** Forward Work

Although the test team has demonstrated that a membrane based biological system coupled with a secondary membrane system can successfully reclaim water from and exploration waste stream, additional work still needed to optimize the systems.

Alternative system operations and bioreactor geometries also need to be further evaluated to determine the optimal configuration of a bioreactor. In previous studies [6] nitrification and denitrification were decoupled and operated as separate systems. Although it is an elegant solution to operate both biological processes in a single reactor and to maximize substrate utilization in a single attached growth system, NASA must investigate the advantages and disadvantages of decoupled (traditional) vs. attached-growth, coupled (novel) systems. In addition, a cylindrical reactor may not be the best configuration in an exploration habitat, where space limitations will be a concern. A test is currently underway evaluating a rectangular cross-flow reactor, which may maximize mixing, aeration and surface area. The Jackson lab is exploring both system operations and reactor geometries as part of an AES contract in CY 2016 and 2017.

How does the inclusion of additional carbon sources affect the SND process? Most research on SND systems have been done at C:N ratio of 3-5:1, but previous testing at JSC demonstrated that high ammonium (75%) and TOC (90%) removal can occur at ratios slightly below 1:1. The test team is working with solid waste investigators at other NASA centers to develop a test to challenge a BWP with leachate from various solid waste processors.

Does the activity of a wastewater consortium change as a result of spaceflight? Results from several flight experiments have demonstrated that bacteria experience physiological changes in microgravity, including up regulation of biofilm formation. To this point there have not been any experiments conducted evaluating the effect of spaceflight on a multispecies environmental biofilm, nor has a wastewater bioreactor been tested on-orbit. Any flight experiment evaluating multispecies biofilm development in microgravity would provide very useful data.

All there additional alternative FO and/or RO membrane technologies that can mitigate fouling for extended periods of time? Porifera, the company that supplied one of the FO membranes for testing, was founded as a result of developing a unique FO membrane technology. Academic institutions and e numerous companies (e.g. Dow) are also investigating membrane improvements.

# VII. Conclusion

The ability to develop a mature ECLSS system capable of processing numerous types of wastewaters in a long duration human exploration mission is paramount to a mission's success. The Alternative Water Processor test successfully demonstrated that a membrane based biological system coupled with a secondary membrane system can reclaim greater than 90% of the an influent wastewater containing urine, condensate, hygiene and laundry wastewaters, while simultaneously reducing consumables by 64% from the SOA water recovery system which reclaims water from urine and humidity condensate and no surfactant containing wastewaters. Additional research and evaluations focusing on optimizing the processes; such as the introduction of additional waste streams to provide additional carbon sources and de-coupling of the nitrification and denitrification/carbon oxidations processes for the BWP, and the evaluation of alternative processing processes are in work, with the goal of developing a biologically based water recovery system to be evaluated on orbit.

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#### References

- 1. Chandler, F. 2015 NASA Technology Roadmaps. 2015 September 8, 2015; Available from: http://www.nasa.gov/offices/oct/home/roadmaps/index.html.
- 2. Winkler, E.H., Verostko, C. E., Dehner, G. F. Urine Pretreatment for Waste Water Processing Systems. in Thirteenth Intersociety Conference on Environmental Systems 1983. San Francisco.
- 3. Carter, L., Brown, C., Orozco, N. Status of ISS Water Management and Recovery. in 43rd International Conference on Environmental Systems 2013. Vail: AIAA.
- 4. Jackson, A., Peterson, Kristin, Morse, Audra, Landas, Nicholas, and Garland, Jay L. . Development and Testing of a TRL 5 Bioreactor for Pretreatment of a Lunar Surface Wastestream. in 40th International Conference on Environmental Systems 2010. Barcelona, Spain AIAA.

International Conference on Environmental Systems

- Richardson, T.M., Flynn, M., Sampson, J., Palmer, G., Berliner, A., Trieu, S., Yendler, A., Beeler, D., Garza, S. Design, Construction, and Testing of the Forward Osmosis Secondary Treatment System to Treat Bioreactor Effluent. in 43rd International Conference on Environmental Systems. 2013. Vail, CO.
- 6. Campbell, M.L., Finger, B.W., Verostko, C.E., Wines, K.R., Pariani, G.M., Pickering, K.D., . Integrated Water Recovery System Test. in 32nd International Conference on Environmental Systems (ICES). 2003. Vancouver, BC: SAE.
- 7. Crenwelge, L., Pickering, K., Carter, L., Callahan, M., Flynn, M., McQuillan, J., *Exploration Life Support Water Recovery System Distillation Comparison Test Plan.*, CTSD, Editor. 2009: Houston, TX.
- 8. Hanford, A.J., Advanced Life Support Baseline Values and Assumptions Document., A.L. Support, Editor. 2004.
- 9. Smith, S.M., McCoy, T., Gazda, D., Morgan, J. L. L., Heer, M., Zwart, S. R. Space flight calcium: Implications for astronaut health, spacecraft operations, and Earth. Nutrients, 2012.
- 10. Parker, D.S., O'Conner, E. W., Bagdigian, R. SS Water Reclamation System Design. in 29th International Conference on Environmental Systems. 1999. Denver, CO: SAE.
- 11. Tchobanoglous, G., et al., Wastewater engineering : treatment and reuse. 2003, Boston: McGraw-Hill.
- 12. Terada, A., Hibiya, K., Nagai, J., Tsuneda, S., Hirata, A., *Nitrogen removal characteristics and biofilm analysis of a membrane-aerated biofilm reactor applicable to high-strength nitrogenous wastewater treatment.* Journal of Bioscience and Bioengineering, 2003. **95**(2): p. 170–178.
- 13. Anthonisen, A.C., Loehr, R.C., Prakasam T.B.S., Srinath, E.G., *Inhibition of Nitrification by Ammonia and Nitrous Acid*. Water Pollution Control Federation, 1976. **48**(5): p. 835-852.
- 14. Meyer, C.E., Pensinger, S., Pickering, K.D., Barta, D., Shull, S., Vega, L.M., Christensen, D., Jackson, W.A. Rapid Start-up and Loading of an Attached Growth, Simultaneous Nitrification/Denitrification Membrane Aerated Bioreactor. in 45th International Conference on Environmental Systems 2015. Bellevue, WA.
- 15. Baez, A. and J. Shiloach, *Effect of elevated oxygen concentration on bacteria, yeasts, and cells propagated for production of biological compounds.* Microbial Cell Factories, 2014. **13**(1): p. 1-7.