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Membrane contactor processes for wastewater reclamation in space Part I. Direct osmotic concentration as pretreatment for reverse osmosis

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Abstract

An innovative system that combines three different membrane processes for reclamation and reuse of wastewater in future space missions was evaluated. The direct osmotic concentration (DOC) system consists of an array of five reverse osmosis (RO) elements, a direct osmosis (DO) pretreatment process, and a combined direct osmosis/osmotic distillation (DO/OD) pretreatment process. Optimized operating conditions, including RO pressures, salt load in the brine loop, and flow velocities were determined for the three subsystems. Mass and heat transfer in the pretreatment processes were measured. Water flux in the DO process was found to be strongly dependent on the type of membrane used; it ranged from 10 to 25 l/(m^2 h) for a cellulose triacetate membrane specifically designed for this application and from 0.5 to 2 l/(m^2 h) for commercially available RO membrane. Water flux through the dual DO/OD process was also found to be highly dependent on temperature gradient across the membranes—increasing with increasing temperature gradient. The conditions for minimum energy consumption of the system were determined and used in estimating the specific energy cost of treating the wastewater generated in space. The weight of salt resupply for continuous operation was also estimated. When compared to alternative technologies, the DOC system provides high wastewater recovery (>95%), at low energy cost (<90×10^3 J/l (25 Wh/l)), with minimal resupply (<20 kg/year).

Keywords: Forward osmosis; Osmotic distillation; Reverse osmosis; Wastewater reclamation; Life support system

1. Introduction

1.1. Wastewater reclamation in long-term space missions

Long-term human missions in space require a continuous and self-sufficient supply of fresh water for consumption, hygiene, and maintenance. Unlike an orbital mission that can be resupplied, a long-range mission, like a human Mars exploration mission, depends on a water treatment system that recovers potable water from wastewater generated on board. The three main sources of wastewater that can be reclaimed and reused in long-term space missions are hygiene wastewater, urine, and humidity condensate. The system to treat these wastewaters must be reliable, durable, redundant, capable of recovering a high percentage of the wastewater, economical, and lightweight. Additionally, this system should operate autonomically with low maintenance and minimal consumables [1].

Different specialized systems have been evaluated by the U.S. National Aeronautics and Space Administration (NASA) over the years. These include the International Space Station (ISS) Baseline, which utilizes filtration beds and oxidation post-treatment; the ICB Bioreactor (BIO) system [2], which utilizes biodegradation followed by reverse osmosis

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Table 1
Mass equivalencies for a Mars reference mission [3]

Parameter	Equivalency
Volume (m^3/kg)	0.062
Power (W/kg)	
For transit	40
For surface	11
Heat rejection (W/kg)	25.4
Tankage (fluid) (m^3/kg)	0.005

(RO), oxidation, and ion exchange post-treatments; the vapor phase catalytic ammonia reduction (VPCAR) system [3,4], which uses distillation and oxidation; and the direct osmotic concentration (DOC) system, which uses direct osmosis (DO) and osmotic distillation (OD) as pretreatment for RO.

Wastewater treatment systems are central components of a spacecraft's life support system. They not only provide water treatment but are also a critical component in the oxygen reconstitution and humidity control systems. Because wastewater treatment systems on board a spacecraft account for approximately half of the total life support payload, significant cost savings are being sought after by optimizing these systems. A recent NASA trade study [3] compared the competing technologies on a mass equivalency basis for a Mars reference mission case study. The mass equivalencies of specific volume, specific power consumption, cooling capacity, and storage volume are given in Table 1. When comparing water treatment systems for this type of application, it is important to provide a common basis for comparison. The common basis here is a system that provides nearly 100% water recovery and is capable of meeting all of NASA's drinking water standards. A summary of the preliminary results (Table 2) reveals that the DOC system has the lowest mass equivalence. Furthermore, power consumption is a major consideration in space missions and preliminary results from the DOC system show that it has relatively low power requirements. Thus, it appears that the DOC system has the potential to become the leading wastewater treatment process for future long-term space missions. Yet, the results for the DOC system, especially the specific power consumption and mass of resupply, are preliminary only and require validation. Furthermore, long-term performance was not evaluated in the preliminary demonstration tests. For these reasons, additional investigation of the DOC system, including optimization of operating conditions with minimal resupply and power requirements, is required.

1.2. Membrane processes for wastewater reclamation

Membrane processes, and especially RO, are favorable separation processes for wastewater treatment because they have the advantages of high rejection, durability, small footprint, simple operation, and minimal resupply of consumable materials for continuous operation. However, RO membranes can be very sensitive to fouling by dissolved and undissolved molecules, particulate matter, salt precipitates, and microorganisms [5–7]. For this reason, RO systems, and especially those used for wastewater treatment, require pretreatment of the feed stream [7–9] to reduce membrane fouling and to ensure acceptable performance. Very often, other membrane processes such as microfiltration (MF) and ultrafiltration (UF) are used as pretreatment steps before RO. MF and UF are usually more cost effective than conventional (chemical and biological) pretreatment processes for wastewater conditioning before final treatment with RO [8]. Furthermore, the use of chemical or biological processes in closed systems, like life support systems in space, has substantial drawbacks. Consumables need to be carried to the mission and excess precipitates and biosolids require handling and disposal.

1.3. The NASA DOC test unit

The DOC system is a proprietary wastewater treatment system that was developed by Osmotek Inc. (Corvallis, Oregon) [10]. The NASA DOC test unit consists of a core RO cascade and two DO pretreatment stages, the first of which (DOC#1) utilizes a DO process only and the second (DOC#2) utilizes a unique combination of DO and OD to assist in rejecting small compounds, like urea, that easily diffuse through semi-permeable membranes. A schematic drawing of the DOC test unit is illustrated in Fig. 1.

1.4. Direct osmosis

In DO, a hypertonic solution (referred to as an osmotic agent (OA)) is recirculated on the permeate side of the membrane, while wastewater is recirculated on the feed side [11]. Contrary to RO, which utilizes total pressure difference, DO utilizes osmotic pressure difference as the driving force for mass transfer. As long as the chemical potential of water on the permeate side of the membrane is lower than that on the feed side, water will diffuse from the feed side through the semi-permeable membrane and dilute the OA. Therefore,

Table 2
Equivalent masses of different technologies for Mars reference mission scenario [3]

Technology	Weight (kg)	Specific power (J/kg (Wh/kg))	Volume (m^3)	Resupply (kg/year)	Total Mars reference mission equivalent mass (kg)
ISS baseline[a]	193	198×10^3 (55)	1.1	413	2463
BIO	396	1335.6×10^3 (371)	1.9	119	2416
VPCAR	68	1123.2×10^3 (312)	0.39	0	434
DOC	233	367.2×10^3 (102)[b]	0.78	0[b]	296

[a] Current state of the art.
[b] Preliminary results.

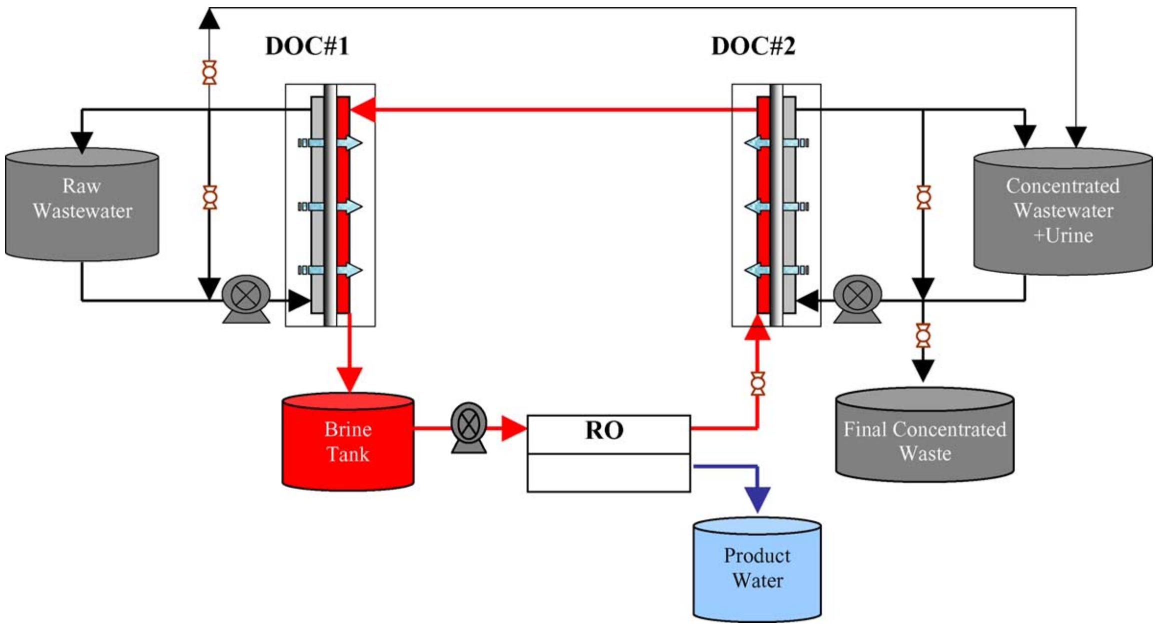

Fig. 1. Flow diagram of the NASA DOC test unit.

most of the energy invested in a DO process is devoted to reconcentration of the OA (which is carried out in the RO step).

Advantages of the DO process include its relatively low fouling potential, low energy consumption, simplicity, and reliability. Because the only pressure involved in the DO process is due to flow resistance in the membrane module (up to 138 kPa (20 psi)), the equipment used is very simple and membrane support is less of a problem. Pressure-associated membrane fouling is also greatly reduced.

Because the membranes used in DO are similar to those used in RO, most contaminants are rejected and only water and some small molecules permeate to the OA side [12,13]. However, it should be emphasized that at the same time that water is passing from the wastewater to the OA, some salt diffuses from the OA to the wastewater. This inevitable process is due to the high salt concentration of the OA and results in a gradual loss of salt from the OA that very slowly but continuously reduces the driving force for mass transfer [13]. One challenge in the current investigation is to balance the trade-off between a high OA concentration for better mass transfer and a low OA concentration for reduced salt loss.

Membrane selection and module design are also important steps in constructing a reliable and efficient DO system. The DO membrane should provide high rejection of both salt and organics but at the same time it should also provide high flux at low driving force. The DO module should provide a large surface area for mass transfer and should offer durable separation between the two streams. One of the main objectives of the current study is to investigate the performance (mass and heat transfer and solute rejection) of the two pretreatment plate-and-frame modules (DOC#1 and DOC#2) and to propose improvements to their design. In a subsequent study [14], these improvements will be further investigated.

1.5. *Osmotic distillation*

OD is combined with DO in DOC#2 primarily to assist with the rejection of urea. OD is essentially an isothermal membrane distillation process in which the driving force for evaporation is the partial vapor pressure gradient of water across a hydrophobic microporous membrane. The partial vapor pressure gradient is induced by a concentrated salt solution (i.e., an OA) on the permeate side of the membrane [15]. OD has traditionally been used in concentrating non-volatile solutes by means of isothermal evaporation. It has been used mostly in the food and pharmaceutical industries where elevated temperatures and pressures may damage the product [16,17].

OD performs better than DO in rejecting small, non-volatile molecules that easily diffuse through semi-permeable membranes. In wastewater, urea is of particular concern because of its prevalence and its poor rejection by RO membranes [18,19]. Urea's very low vapor pressure prevents it from evaporating through the microporous membrane in the OD process.

Because OD is an isothermal evaporative process, care must be taken to maintain an appropriate temperature profile across the OD membrane. Even under initially isothermal conditions, heat of evaporation is transported with the vapors to the OA side and may result in an OA solution that is warmer than the feed solution. This reversed temperature profile will stop vapor transport [17,20] and potentially result in reversed transport of water vapors. In the NASA DOC test unit specifically, heat generated in the system (e.g., by the RO pump or by friction in pipes) may induce a reverse temperature profile and cause flux reduction or reversed flux. Because the partial vapor pressure driving force generated by 100 g/l NaCl OA (with distilled water on the feed side) will be cancelled by a merely 1–2 °C warmer OA solution, it is apparent that reversed flux can easily occur if the temperature profile is not carefully maintained. For this reason, heat transfer issues (along with mass transport issues) are a central aspect of this investigation.

Membrane flooding by either the feed water or OA will stop the separation process and result in cross contamination of the two streams. Membrane flooding is controlled by membrane hydrophobicity, pore size, and pore geometry, and by the surface tensions of the liquids in contact with the microporous membrane. Membrane hydrophobicity, pore size, and pore geometry are controlled by membrane selection; liquid surface tension is controlled by operating conditions and solution chemistry. Therefore, special attention in the current investigation is given to the presence of surfactants in the feed and/or OA streams. If surfactants are present in high enough concentrations, the decreased surface tension of the liquid will cause the pores of the membrane to flood.

1.6. Combination of direct osmosis (DO) and osmotic distillation (OD)

An innovative combination of DO and OD (DO/OD) was introduced by Osmotek, Inc. for recovery of water from the mixture of concentrated wastewater and urine in DOC#2. The two membranes, one semi-permeable and one microporous, are laid on each other and mass transport is carried out in three steps: water diffuses from the wastewater stream through the semi-permeable membrane, evaporates through the hydrophobic microporous membrane, and then condenses in the OA (Fig. 2). The driving forces throughout the process are the osmotic pressure and partial vapor pressure gradients across the two membranes, which are induced by the concentration difference between the wastewater and the OA.

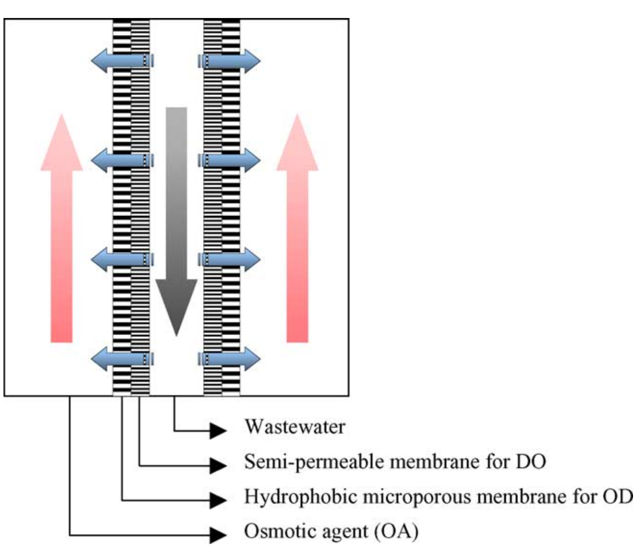

Fig. 2. Dual membrane configuration and flow pattern in DOC#2.

The two processes are combined so that the semi-permeable membrane protects the hydrophobic microporous membrane from fouling and pore flooding and the microporous membrane serves as an ultimate barrier to the transport of urea. For this reason, the integrities of the membranes in both DOC#1 and DOC#2 are very important in maintaining the proper functionality of DOC#2. In the simplest scenario, breach of membrane integrity in either of the modules will cause contamination of the OA with surfactants and flooding of the microporous membrane through its support side. This would lead to diffusion and flow of urea from the feed side of DOC#2 into the OA and from there to the product water.

One of the main drawbacks of OD and the dual DO/OD is that mass transfer in the OD process is slower than it is in DO by up to two orders of magnitude [16]. This results in difficulties with treating the daily volume of wastewater (of the reference mission) in the existing DOC#2 modules. This is further reason that the design of DOC#2 will be critically evaluated, improvements will be proposed, and in a subsequent investigation [14], flux enhancement will be achieved.

1.7. Solution chemistry and its effect on the performance of the DOC test unit

The concentration of the OA and the concentration of the wastewater are key parameters that dictate the performance of the entire DOC test unit. The amount of salt loaded in the OA loop is a variable in the system that can be arbitrarily chosen and markedly affect the concentration of the OA generated. The concentration of the OA is also controlled by the operating conditions (flowrates and pressures) in the RO subsystem. Additionally, the OA concentration along DOC#1 and DOC#2 is a function of both the concentration of OA generated and the concentration of wastewater. Because the NASA DOC test unit is operated in a semi-batch mode, wastewater concentration fluctuates during the course of operation. The concentration of wastewater in contact with the membrane varies with the wastewater volume in the tank and may also be controlled by the recycling ratio in the wastewater loop (Fig. 1). As an example of how these work together, the OA concentration along DOC#1 and DOC#2 declines faster if the initial OA concentration is high and the wastewater concentration is low (higher water flux and therefore faster dilution of the OA), and vice versa. Thus, determination of optimized operating conditions requires consideration of the complexity and dynamic nature of the system. The implications of changing operating parameters must be considered for the long-term stability of the combined system.

1.8. Objectives

The ultimate goal of the current investigation is to study the long-term performance, advantages, and limitations of the NASA DOC test unit and to validate the data for the mass metrics comparison between the DOC system and other potential wastewater treatment systems. The current paper presents the principles of the DOC system and results of performance testing of the refurbished NASA test unit.

For DOC#1, the mass transfer characteristics, salt and organics rejection, and power consumption were evaluated. After appropriate membranes were selected for DOC#2, the mass and heat transfer characteristics, urea rejection, and power consumption were studied. And finally, after restoration of the RO subsystem, salt and organics rejection and power consumption by the RO subsystem were evaluated. Results from this investigation will enable the optimal operating conditions and the system limits to be determined. Additionally, more extensive modifications to the NASA test unit are proposed in order to prepare the system for long-term evaluation.

2. Material and methods

The NASA DOC test unit (Fig. 3) designed and built by Osmotek Inc. was delivered to NASA-AMES (Moffett Field, CA) and tested for a short time. The system was later transferred to the University of Nevada, Reno for further investigation. Restoration activities were immediately performed upon receipt of the NASA test unit. Restoration processes for each of the three subsystems (the RO, DOC#1, and DOC#2 subsystems) are described in the following sections. Pressure indicators, flow meters, conductivity probes, and thermocouples were installed at various locations in the subsystems to monitor and control the operation of the system.

It should be noted that the NASA test unit was designed without internal cleaning mechanisms and frequent membrane cleaning was conducted manually. Cleaning of the membranes in the DOC pretreatment stages was found to be energy and water consuming. In future testing, a cleaning mechanism will be integrated into the system.

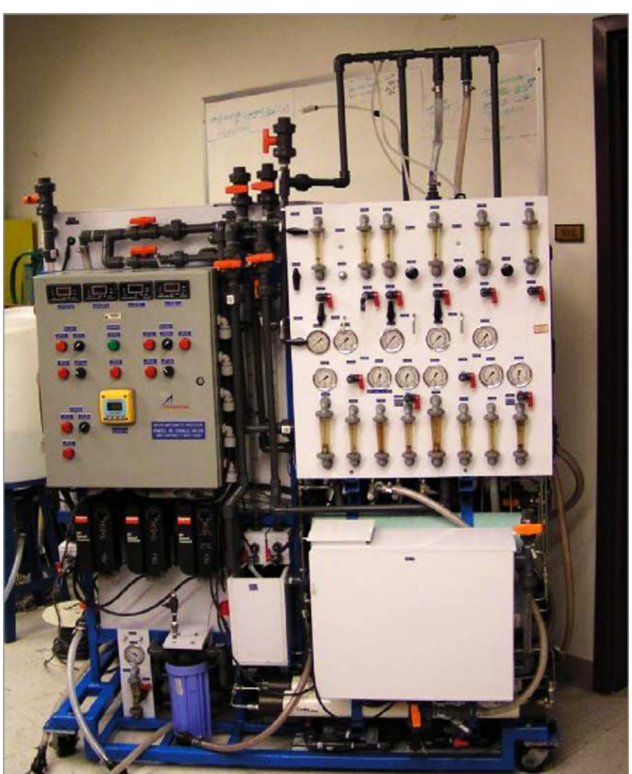

Fig. 3. NASA DOC test unit.

2.1. The RO subsystem

The RO subsystem (Fig. 4) is an array of five RO membrane elements arranged in four passes and driven by a positive displacement plunger pump (Model-2P411C, Dayton Electric MFG Co., Chicago, IL). Passes 1A and 1B were designed to concentrate the OA. The product of Pass 1A is sent to Passes 2, 3, and 4 to produce the final purified product water. Passes 2 through 4 were each designed to purify the product of the preceding pass, thereby assuring considerably high total rejection of contaminants.

Upon receiving the NASA DOC test unit, integrity tests were performed on the five RO elements. An external pump, pressure gauge, and backpressure valve were consecutively connected to each of the RO elements and salt rejection tests were performed on each element under the same pressure and feed salt concentration tested at the factory. In Pass 1, the original hollow fiber RO elements (DuPont B-10, Wilmington, DE) failed integrity tests and similar replacement was not available. These elements were therefore replaced with spiral wound membranes (SW30-4040 and SW30-4021, DOW-Filmtec, Minneapolis, MN). The RO elements in Passes 2, 3, and 4 passed integrity tests and were therefore used in subsequent experiments. Table 3 lists all of the RO elements used in further testing.

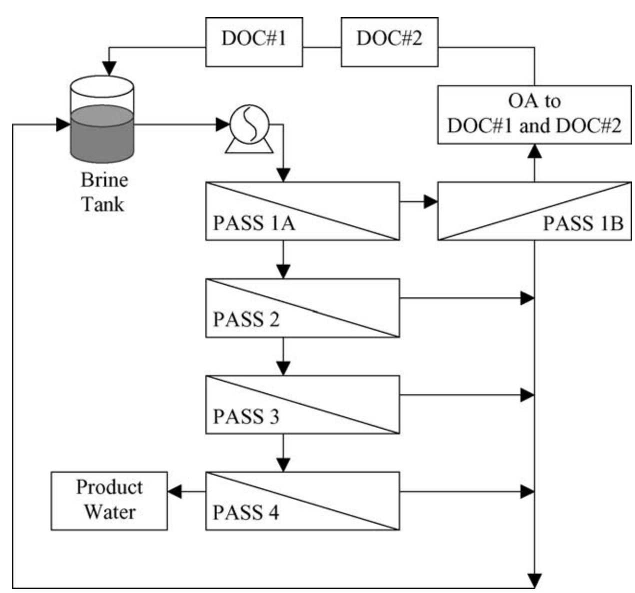

Fig. 4. Layout of the RO subsystem.

Table 3
RO elements used in the DOC system

Pass	Manufacturer	Model	Configuration	Maximum pressure (MPa (psi))
1A	DOW Filmtec	SW30-4040	Spiral wound	6.9 (1000)
1B	DOW Filmtec	SW30-4021	Spiral wound	6.9 (1000)
2	DuPont	B-9 Permasep	Hollow fiber	2.76 (400)
3, 4	Osmonics	DESAL-SG2525TH	Spiral wound	1.38 (200)

2.2. The DO subsystems (DOC#1 and DOC#2)

The DOC modules were designed and constructed in a plate-and-frame configuration. In this configuration, the feed wastewater and the OA flow on opposite sides of a flat sheet membrane with minimal total pressure applied on the membrane. The wastewater is recirculated on the active side of the membrane using a closed coupled centrifugal pump (Model OH75CP, Price Pumps, Sonoma, CA); the OA, driven by the RO pump, flows on the support side of the membrane in grooves carved into the plates. Each of the plates in the DOC module was machined from a 13 mm thick polycarbonate board. DOC#1 was assembled with eight plates and DOC#2 with four plates. Two membranes (two pairs of membranes in DOC#2) are placed between every two plates and the wastewater stream flows between the two membranes. A cross-section of the assemblage of plates and membranes is illustrated in Fig. 5 [21]. The membrane sheets initially installed in DOC#1 were a semi-permeable cellulose triacetate (CTA) membrane made by Osmotek, Inc. This membrane, in combination with a microporous hydrophobic membrane (Ultrex-Supplex, Burlington Industries, Greensboro, NC), was also initially installed in DOC#2.

Four additional T-type digital thermocouple thermometers (Model 600-1040, Barnant Comp., Barrington, IL) were installed at the wastewater and OA inlets and outlets of the DOC#2 subsystem to monitor the temperature profile development during operation of the system. The temperature profiles were correlated with the mass transfer observed in bench-scale tests described below.

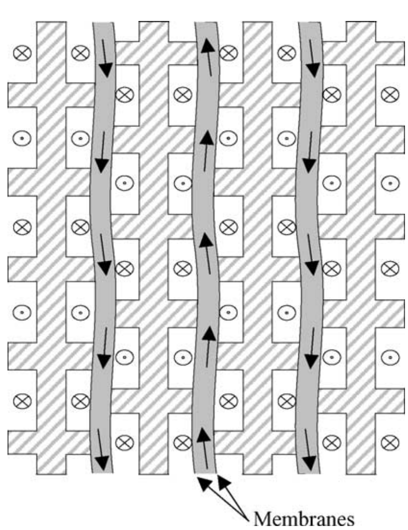

Fig. 5. Cross-section of the DOC plate-and-frame module. (⊙) Flow of OA in one direction; (⊗) flow of OA in the other direction; gray area—flow of wastewater; cross-hatched area—polycarbonate plates [21].

2.3. Bench-scale tests for DOC#1 and DOC#2 membrane selection

Bench-scale tests were performed to evaluate the existing and alternative semi-permeable and hydrophobic microporous membranes for DOC#1 and DOC#2. Bench-scale tests were carried out using a modified, vertically-oriented, acrylic SEPA-CF membrane cell (Osmonics, Minnetonka, MN) that utilizes a flat sheet membrane 139 cm^2 in surface area.

Four RO membranes were tested as potential DO replacements for the original CTA membrane provided by Osmotek. This included two cellulose diacetate/triacetate membranes (CE and CD, Osmonics) and two thin-film composite polyamide membranes (LFC1 and LFC3, Hydranautics, Oceanside, CA). These membranes were tested for flux and salt rejection. Additionally, the performance of the Osmotek CTA membrane was tested in a dual DO/OD configuration with the microporous membranes. Performances for a variety of OA concentrations were compared to results obtained with the original membranes.

Three microporous membranes were tested as potential replacements for the original Ultrex-Supplex hydrophobic membrane. These included two polytetrafluoroethylene (PTFE) (TefSep 0.22 μm pore size (TS22) and TefSep 1.0 μm pore size (TS1.0)) membranes and one polypropylene (PolySep 0.22 μm pore size (PP22)) membrane, all acquired from GE Osmonics (Minnetonka, MN). These membranes were tested for flux and salt and urea rejection.

2.4. Power consumption and optimization of operating conditions

The major energy consumers in the NASA DOC test unit are the two DO recirculation pumps and the high pressure RO pump. The electrical control system and the electronic sensors constantly consume 50 W; this is considered background energy consumption and cannot be altered or minimized. Otherwise, the DOC system was designed with wide operational flexibility that greatly affects the energy consumed by the overall process.

An electric power meter was installed between the power outlet and the DOC test unit to monitor power consumption. To evaluate how best to minimize power consumption, each of the three subsystems was operated independently under var-

Table 4
Sources of wastewater and their generation rate [4,22,23]

Source	Mass (kg/person day)
Hygiene water	25.3
Humidity condensate	1.8
Urine + flush	2.0
Total	29.1

ious operating conditions. For the RO subsystem, the pump speed was varied from 80 to 100% of maximum pump speed and the reject flowrate of Pass 1B was varied from 0.2 to 0.6 l/min. For DOC#1 and DOC#2, pump speeds were varied from 45 to 60% and from 55 to 65%, respectively, and the flow rates of the recirculated waste streams were varied from 9 to 15 l/min by controlling the recirculation ratio (the ratio between the flowrate of the recycled wastewater stream and the flowrate of wastewater entering the system).

2.5. Solution chemistries

Three wastewater simulants, corresponding to the main sources of wastewater on board a spacecraft, were used in this study. These included hygiene wastewater, humidity condensate, and urine. The average volume of these wastewaters generated per person per day in a space mission is given in Table 4 [22,23]. The NASA DOC test unit was constructed with tanks having a capacity to accommodate the daily wastewater generated by six crewmembers. The chemical compositions of the hygiene wastewater and humidity condensate streams (Table 5) were received from NASA-JSC (Houston, TX) [24]. In all bench-scale experiments involving feed wastewater, triply concentrated synthetic wastewater was used. This concentration represents the average wastewater concentration transferred from the DOC#1 subsystem to the dual membrane process in DOC#2. An on-going discussion occurring between different NASA research divisions is focused on whether urea prevails in a spacecraft's wastewater system or whether it rapidly degrades. Studies in the literature have also debated this issue [19,25]. However, because the NASA DOC test unit was specifically designed to treat wastewater that contains urea, an aqueous urea solution was used as the urine simulant in the current study. All the chemicals used for the humidity condensate and urine simulants are certified ACS grade and were obtained from Fisher Scientific (Pittsburgh, PA).

Table 5
Synthetic wastewater components [24]

Wastewater component	Quantity
Hygiene wastewater	
NASA whole body shower soap, (Ecolab, Inc., St. Paul, MN)	1.2 g/l wastewater
Humidity condensate	0.618 ml/l wastewater
Ethanol	117.98 g/l
2-Propanol	31.87 g/l
1-2, Propanediol	65.04 g/l
Caprolactam	23.74 g/l
2-(2-Butoxyethoxy) ethanol	3.43 g/l
4-Ethylmorpholine	3.85 g/l
Methanol	6.82 g/l
Formaldehyde	14.02 g/l
Formic acid	19.95 g/l
Propionic acid	6.53 g/l
Zinc acetate dihydrate	39.96 g/l
Ammonium bicarbonate	29.87 g/l
Ammonium carbonate	29.37 g/l
Urine	
Urea	5 g/l wastewater

Certified ACS grade NaCl (Fisher Scientific, Pittsburgh, PA) was used to prepare the OA for both the bench- and pilot-scale experiments. In all bench-scale experiments, the initial OA concentration was 100 g/l NaCl. In the NASA DOC test unit, 1000 g or 500 g NaCl were mixed with 20 l of deionized water to make initial OA solutions. The salt concentration in the OA was further controlled by the backpressure valve in Pass 1 of the RO subsystem.

3. Results and discussion

3.1. Membrane operation and performance in the RO subsystem

One of the main concerns with substituting a spiral wound element for a hollow fiber element in Pass 1A was the inevitable high pressure on the permeate side – pressure that drives the separation processes in Passes 2, 3, and 4. Membrane manufacturers strongly discourage any pressure inside the envelope of spiral wound elements. Also, the DOC test unit was not designed with a pressure relief system to protect Pass 1A against pressure surges during emergency shutdown. Having no technical alternative in the short run, controlled shutdown experiments were performed, in which pressure decline on both the feed and permeate sides of Pass 1A were closely monitored. Results confirmed that the pressures on both sides of the membrane declined at similar rates and that the feed pressure is always at least 690 kPa (100 psi) higher than the permeate pressure inside the envelope – providing sufficient protection to the integrity of RO element 1A.

Thereafter, the DOC test unit was loaded with fresh salt solution and only the RO subsystem was operated (DOC#1 and DOC#2 were bypassed) with 5.52 MPa (800 psi) on the feed side of Pass 1 and 2.07 MPa (300 psi) on the permeate side of Pass 1A. After allowing the RO subsystem to reach equilibrium, salt concentrations were measured in all the streams and salt rejection in each of the RO elements was calculated (Table 6). The low measured salt rejection of Pass 4 can be attributed to the high rejections provided by Passes 1, 2, and 3. The salt concentration of the feed into Pass 4 was 4.5 mg/l NaCl and the permeate concentration was 2.0 mg/l NaCl. In

Table 6
Salt rejection in the RO elements

Pass	RO element	Manufacturer	Nominal salt rejection (%)	Measured salt rejection	
				%	Feed conc. (ppm)
1A	SW30-4040	DOW-Filmtec	99.2	97.4	14,000
1B	SW30-4021	DOW-Filmtec	99.4	96.5	41,400
2	B-9	DuPont	94.0	73.2	367
3	Desal-SG2525TH	Osmonics	98.2	95.5	98
4	Desal-SG2525TH	Osmonics	98.2	55.9	4.5

rejection tests with higher feed salt concentrations, the salt rejection in Pass 4 was 95%.

3.2. Membrane integrity tests and performance in DOC#1

3.2.1. Membrane integrity tests

Due to the complex configuration of the DOC modules, determination of membrane integrity is not as simple for DOC#1 and DOC#2 as it is for the RO subsystem. Integrity testing was performed on DOC#1 in an RO mode. The OA was drained from the permeate side of DOC#1 and the channels were flushed with ultrapure water. Ultrapure water was recirculated on the feed (wastewater) side of the membrane at a feed pressure of 69 kPa g (10 psi g), and the flux of water and its conductivity were recorded until reaching a steady state condition. Upon addition of 1 g/l NaCl to the feed stream (bringing feed conductivity to 1800–2000 μS/cm), water flux declined by 49–63% and permeate conductivity rose from 30 μS/cm to 1100–1200 μS/cm, as shown in Fig. 6. Because the osmotic pressure of 1 g/l NaCl is approximately 69 kPa, the abrupt flux decline can be explained by elimination of the pressure driving force. The remaining permeate flowrate was therefore the result of leakage of feed solution through the CTA membrane inside the plate-and-frame module. This leakage was also confirmed by the substantial increase in permeate conductivity. Following these tests, a new set of CTA membranes was installed in DOC#1.

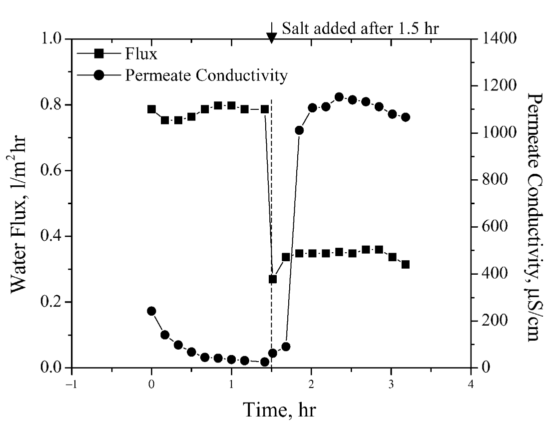

Fig. 6. Flux and permeate conductivity vs. time during integrity test of DOC#1.

3.2.2. Effect of feed chemistry and OA concentration on water flux

Bench-scale tests were performed on new samples of the CTA membrane to acquire performance benchmarks and data for energy consumption estimation. In order to evaluate the effect of contaminant concentration on mass transfer, three different feed solutions were tested on the bench-scale unit. The feed was either ultrapure water or doubly (2×) or triply (3×) concentrated NASA wastewater (hygiene plus humidity condensate only) at constant concentration. The effect of OA concentration on water flux through the CTA membrane for the three feed solutions is illustrated in Fig. 7. As water diffuses through the CTA membrane in the bench-scale apparatus, the OA is gradually diluted, thereby reducing the osmotic pressure driving force. As expected, flux decreases with decreasing OA concentration. It was also observed that salt diffuses from the OA to the wastewater side at an average rate of 300 mg NaCl for every liter of wastewater recovered in DOC#1.

The results also show that the presence of contaminants in the feed stream strongly affects the mass transport through the membrane. This is likely due to a surfactant (sodium methyl cocoyl taurate) that is present in the NASA shower soap. When the soap concentration was increased by a factor of three the flux decreased by almost 50%. It has been shown that, even at low concentrations, surfactants noticeably change the surface charge of RO membranes [26,27] and

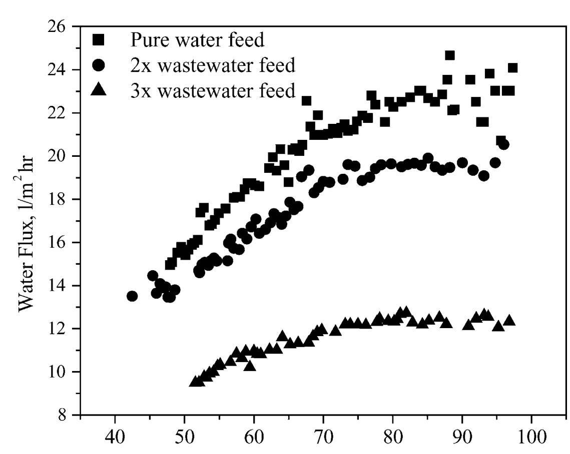

Fig. 7. Bench-scale results of flux vs. OA concentration in DO through the CTA membrane. Ultrapure water or concentrated wastewater was recirculated on the feed side.

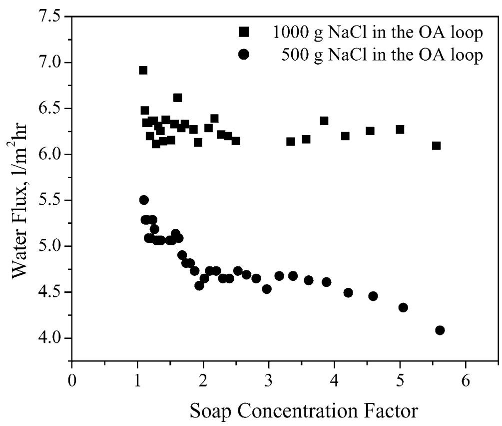

Fig. 8. Water flux in DOC#1 as a function of soap concentration for two salt loadings.

adversely affect the flux through these membranes [28]. In the NASA DOC test unit, the initial concentration of sodium methyl cocoyl taurate is high (>1.0 g/l), and it gets much higher (>15 g/l) as the wastewater becomes more concentrated. Additionally, the presence of trace amounts of surfactant was observed in the OA after several days of continuous experiments—probably due to diffusion of the surfactant across the CTA membrane. This could adversely affect the RO subsystem, and more critically, the microporous membrane in DOC#2.

The effect of feed soap concentration on water flux in DOC#1 is illustrated in Fig. 8 for two different salt loads in the OA stream. A soap concentration factor of 1 corresponds to NASA's hygiene and humidity condensate compositions (Table 5). The inlet OA concentration was approximately 32 g/l NaCl when the OA stream was loaded with 500 g NaCl and approximately 58 g/l NaCl when it was loaded with 1000 g NaCl. Feed wastewater and OA flowrates were 12.5 and 0.3 l/min, respectively. Results further confirm that system performance strongly depends on salt load and soap concentration—flux increases with an increased salt load to the OA loop and decreases as the soap concentration increases. More importantly, the fluxes presented in Fig. 8 (even at the level of 4 l/(m^2 h)) are high enough to treat the daily amount of wastewater generated—approximately 180 l/day for six crewmembers. With 500 g NaCl in the OA loop, 83 l of wastewater were treated in less than 6.5 h. However, as the wastewater becomes even further concentrated (6–15 times), the flux may drop to levels that are too low. For this reason, it is important to note from Fig. 8 that at a salt load of 1000 g NaCl, wastewater concentration has much less effect on water flux. This is a substantial advantage of using a higher salt load to the OA.

3.2.3. Performance of alternative DO membranes

Two cellulose diacetate/triacetate membranes (CE and CD, Osmonics) and two composite polyamide membranes (LFC1 and LFC3, Hydranautics) were also tested on the bench-scale test apparatus as an alternative to the CTA membrane provided by Osmotek. Feed solution was NASA wastewater (soap and humidity condensate only), the OA was 100 g/l NaCl, and flowrates were 1.5 l/min. Results (Table 7) show that the CTA membrane made by Osmotek provides significantly more water flux in DO mode than the other low pressure, low fouling RO membranes. The membranes were also tested for urea rejection to assess them for possible use in DOC#2 as well as DOC#1. A feed solution of 5 g/l urea was used. Urea rejection (data not shown) was below 50 percent for all the membrane tested (including Osmotek's CTA).

Table 7
Bench-scale results of the performance of selected membranes for DO

Membrane	Manufacturer	Water flux (l/(m^2 h))
CTA	Osmotek	17.4
CE	Osmonics	1.90
CD	Osmonics	1.97
LFC1	Hydranautics	0.54
LFC3	Hydranautics	0.66

Internal concentration polarization in RO membranes is believed to be the cause of low water flux in the DO mode [29]. The layered structure of dense RO membranes reduces the apparent osmotic pressure gradient across the membrane and thereby diminishes the driving force for mass transfer. Further research on the diffusion mechanism in DO mode will hopefully lead to improved membranes for the DO process.

3.3. Membrane replacement and performance in DOC#2

There were two major issues to be addressed in DOC#2. The first was prevention of flooding of the microporous membrane by either the wastewater or the OA and the second was determination of the effect of heat transport on mass transport in the module.

3.3.1. Inspection of membrane integrity

Membrane integrity tests were more complicated to perform on DOC#2 because mass transfer through its two membranes is more complex. The pores in the microporous membrane must be kept dry at all times in order to allow continuous evaporation of water through the membrane. This restriction requires that the membrane be very hydrophobic and have pores small enough to prevent pore flooding. Pore flooding in membrane evaporation processes is the result of exceeding the liquid entry pressure of water (LEPW), which is the minimum pressure at which water will overcome the hydrophobic forces of the membrane, penetrate the pores, and stop the evaporation process. LEPW is a function of the properties of the membrane, the liquid, and the interaction between them.

Upon first inspection of the membranes in DOC#2, it was apparent that the support layer of the Ultrex-Supplex membrane was completely soaked with water during the shutdown period. Contact angle measurements indicated that the contact angle of the Ultrex-Supplex membrane is lower than it is for most hydrophobic materials (95° versus 112° for polyte-

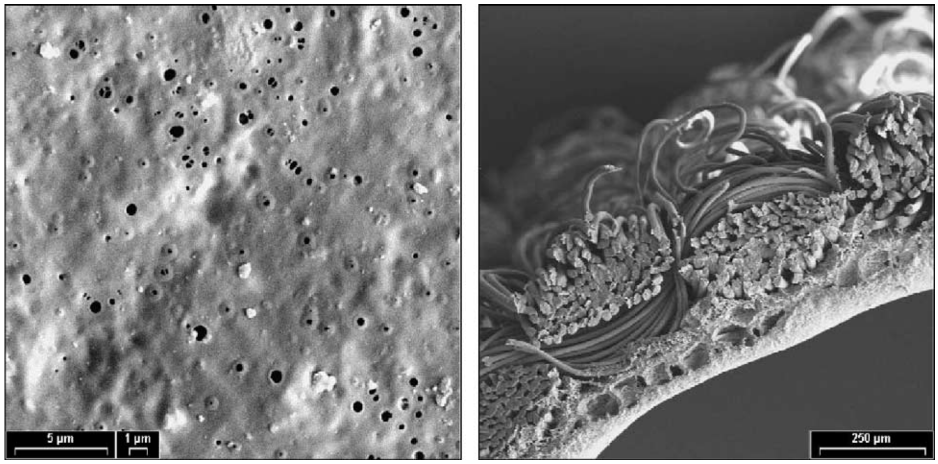

Fig. 9. SEM images of the active surface (left) and cross-section (right) of the Ultrex-Supplex membrane.

trafluoroethylene (PTFE) and 108° for polypropylene (PP)). SEM imaging (Fig. 9) revealed that the Ultrex-Supplex membrane has a wide pore size distribution. The large pores are most likely causing flooding of the membrane.

3.3.2. Membrane performance testing and selection

Water flux and salt and urea rejection for the original Ultrex-Supplex membrane was compared with the flux and rejection of several hydrophobic microporous membranes that were selected as possible replacements. Bench-scale testing was performed in two modes: OD and combined DO/OD (dual membrane configuration). In the OD mode, the water flux of the Ultrex-Supplex membrane was compared with the flux through one PP membrane (PP22) and two PTFE membranes (TS22 and TS1.0). Results (Table 8) indicate that the PP membrane has a similar flux to the Ultrex-Supplex membrane and the PTFE membranes have higher fluxes. Urea rejection in all cases was greater than 99%. When tested in dual DO/OD mode, more interesting data resulted. While the water fluxes through the dual CTA/TS22 and dual CTA/PP22 membranes were similar to the fluxes through the microporous membranes alone, the flux through the dual CTA/Ultrex was much higher and the urea rejection very low. This indicates that the Ultrex-Supplex membrane was flooded. Thus, the CTA membrane only protects suitable microporous membranes from surfactants. By comparing these results with Table 7, it can be seen that mass transport in the dual DO/OD configuration is limited by the evaporation rate through the microporous membrane. The flux through the CTA membrane alone in DO mode is approximately 17 l/(m^2 h) whereas fluxes through the dual membrane processes using the CTA membrane and the suitable OD membranes (the TS22 and PP22 membranes) are much less than 1 l/(m^2 h).

Table 8
Bench-scale results of the performance of selected membranes for DOC#2

Mode	Membrane used	Water flux (l/(m^2 h))	Urea rejection (%)
OD	Ultrex-Supplex	0.14	99.5
OD	PolySep 0.22 (PP22)	0.20	99.9
OD	TefSep 0.22 (TS22)	0.41	99.9
OD	TefSep 1.0 (TS1.0)	0.43	99.7
DO/OD	Dual CTA/Ultrex	4.7	51.3
DO/OD	Dual CTA/PP22	0.09	100
DO/OD	Dual CTA/TS22	0.42	99.8

Based on the bench-scale tests results, the TS22 membrane was chosen to replace the Ultrex-Supplex membrane. The TS1.0 membrane had a slightly higher flux in the bench-scale tests but was not chosen because its support layer was too coarse and prevented appropriate sealing between the DOC plates. The fluxes through the PP22 membrane were too low to treat the daily volume of wastewater to be processed by DOC#2.

3.3.3. Heat and mass transfer in DOC#2

The OD process is very sensitive to temperature differences across the membrane. The concentration gradient driving force in the OD process is much weaker than a temperature gradient driving force can be. In the NASA DOC test unit, heat is generated in the high-pressure RO pump. This may create a reverse temperature gradient between the wastewater stream and the OA (where the OA is warmer than the wastewater) that will reduce the driving force for evaporation and in the worst case, reverse the flux in DOC#2.

A set of performance experiments was conducted to monitor the temperature profile development in DOC#2. The inlet and outlet temperatures of both the wastewater stream and the OA stream were recorded until they reached steady state levels. The temperature profiles developed over time are shown in Fig. 10. Results show that as time progresses the temperatures at the outlets switch from the OA having a slightly higher temperature than the wastewater to the wastewater having a slightly higher temperature than the OA. Thus, inside DOC#2 there is a section of the module where the OA is warmer than the wastewater. At low temperature differences, the result is

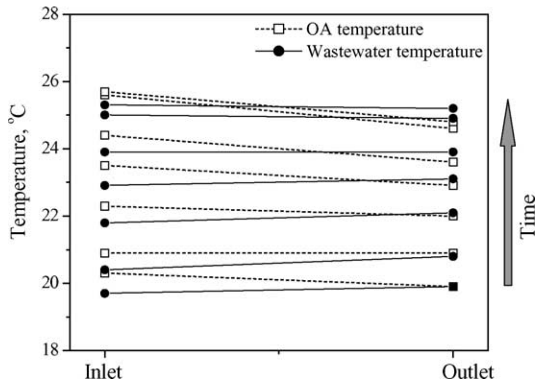

Fig. 10. Temperature profile developed in DOC#2.

diminished transport of water vapors. If the temperature difference is high enough, the thermal driving force will exceed the concentration driving force and flux will occur in the opposite direction. Because the internal flows of both streams in DOC#2 are rather complex, it is difficult to model and predict the size of the section in which the temperature gradient is reversed.

To solve the problem, a small heat exchanger that cools down the OA stream can be used to increase the thermal driving force in the desired direction. Evaluation of such a solution was studied and reported in a subsequent paper [14]. However, since the flow rate of the OA is rather slow (0.3 l/min) in the current configuration, the heat transfer efficiency within the module will slowly diminish. Thus, additional modification of the hydraulic system to increase the flow rate of the OA is expected to be necessary for long-term evaluation of the system.

In light of the internal concentration polarization suspected in the RO membranes for DO and the slow evaporation rates in OD, it is not surprising that the flux of water through DO/OD is so low. However, it is surprising that water diffuses through the CTA membrane although there is no OA between the two membranes. One hypothesis is that as water evaporates through the microporous membrane, low vacuum is induced between the membranes and the mass transport across the semi-permeable membrane is actually controlled by the resulting pressure gradient. Another hypothesis is that low pressures on the feed side will generate enough pressure gradient to enable water to diffuse across the membranes.

3.4. Optimization of energy consumption

To minimize the power consumed by the DOC test unit, each of the three subsystems was operated independently under various operating conditions and the power consumption was monitored and recorded. The major energy consumers are the three variable speed pumps installed in the DOC test unit and therefore, the main objective is to operate them as slowly as possible while remaining within necessary operating conditions.

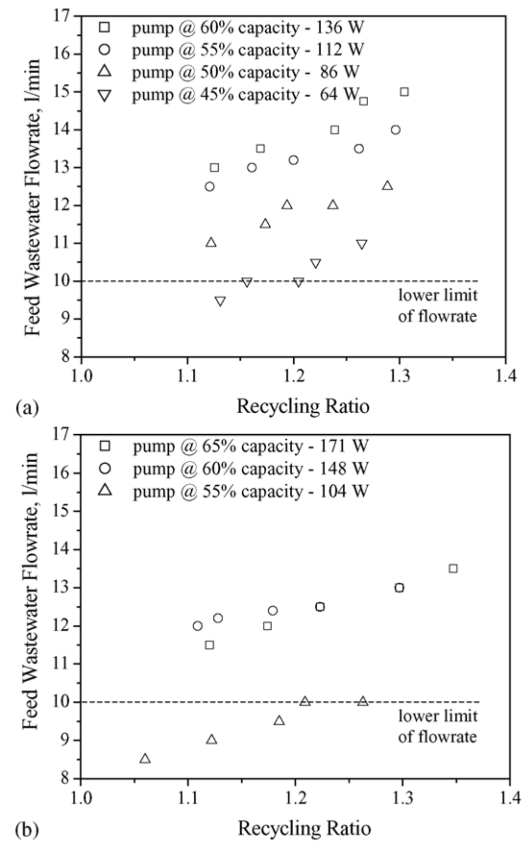

Fig. 11. Feed wastewater flowrate as a function of recycling ratio for a range of pump capacities for (a) DOC#1 and (b) DOC#2.

3.4.1. Optimization of power consumption in DOC#1 and DOC#2

To determine the operation conditions for minimum energy consumption in DOC#1 and DOC#2, the two most influential parameters, the hydraulic retention time of wastewater in the DOC module (denoted by recycling ratio) and the speed of the recirculation pump (denoted by percentage of maximum pump capacity) were varied. Two limiting operating conditions were maintained in the DOC modules: a minimum wastewater flowrate of 10 l/min (equivalent to 0.25 m/s tangential flow velocity) to reduce membrane fouling [21] and a maximum pressure of 103 kPa (15 psi g) at the wastewater inlet to prevent collapse and damage to the CTA membranes and flooding of the microporous membranes.

Feed wastewater flowrate for each of the DOC subsystems as a function of recycling ratio for several pump speeds is shown in Fig. 11. Power consumption is shown to be a function of pumping speed only—it was found that under constant speed of the recirculation pumps, increasing the recycling ratio did not affect the power consumption. Thus, increasing the recycling ratio only increased the wastewater flowrate in the DOC modules. This is beneficial because higher wastewater flowrates result in reduced potential for membrane fouling.

Based on these results, it was determined that DOC#1 will be operated at 50% pump capacity and 1.3 recycling ratio and

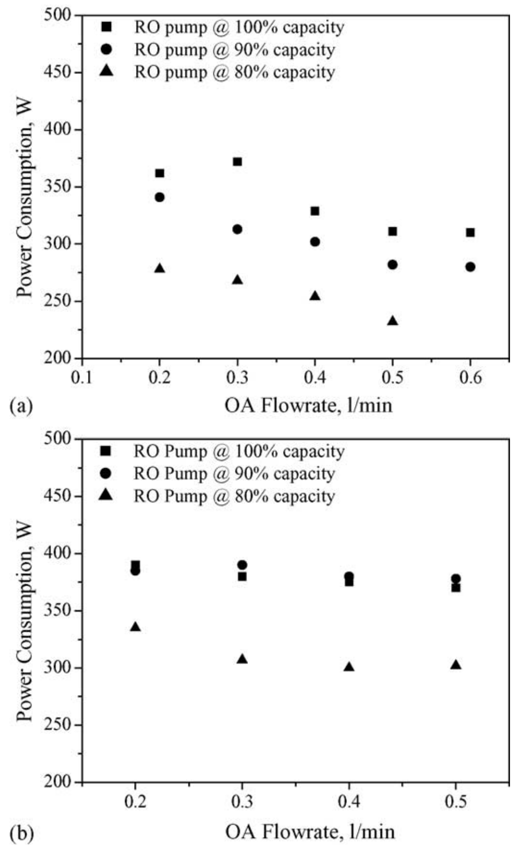

Fig. 12. Power consumption by the RO subsystem as a function of OA flowrate for a range of pump capacities. Results are for (a) 500 g NaCl and (b) 1000 g NaCl loaded in the system.

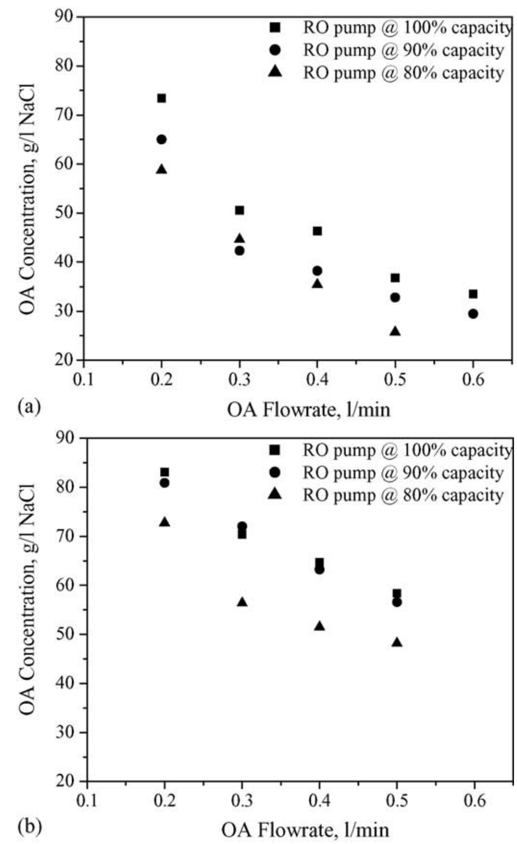

Fig. 13. OA concentration generated by the RO subsystem as a function of OA flowrate for a range of pump capacities. Results are for (a) 500 g NaCl and (b) 1000 g NaCl loaded in the system.

DOC#2 will be operated at 60% pump capacity and 1.3 recycling ratio. These operating conditions represent low energy consumption at high recycling ratio while maintaining the desired minimum wastewater flowrate of 10 l/min (0.25 m/s).

3.4.2. Optimization of power consumption in the RO subsystem

Power consumption in the RO subsystem is strongly dependent on the pump speed and the feed pressure in Pass 1 – consequently, power consumption increases with increasing concentration of the produced OA. In two sets of experiments, all subsystems of the DOC test unit were operated simultaneously and power consumption was measured. In the first set, 500 g of NaCl was loaded to the brine loop and in the second, 1000 g was loaded. The wastewater pumps were operated at the selected conditions (as described in the previous section), and the OA flowrate (at Pass 1B reject outlet) and the RO pump speed were varied. The power consumed by the RO subsystem is shown in Fig. 12. Three trends were observed. Power consumption generally increases with increasing pump speed, similar to DOC#1 and DOC#2. Power consumption generally decreases when OA flowrate increases. This is because the pressure in Pass 1 declines with increasing OA flowrate (opened backpressure valve). And lastly, power consumption increases with the higher salt loading in the OA loop because of the elevated osmotic pressure of the feed in Pass 1. When the power consumption of DOC#1 and DOC#2 are added in, the total power consumption is on average 300 W higher.

Because the salt load, OA flowrate, and OA concentration are interdependent, it is particularly difficult to determine the optimized operating conditions in the RO subsystem. It is desired that the OA flowrate at the outlet of Pass 1B be as high as possible to increase shear flow on the RO membrane and reduce concentration polarization and membrane fouling. On the other hand, because the rate of mass transfer in the DOC modules is dictated by the OA concentration, it is desirable to produce an OA having a high salt concentration (which results in a lower OA flowrate, as discussed below). Also, as shown in Fig. 8, a higher salt load results in a more constant water flux. For these reasons, all aspects need to be considered when trying to determine the optimal salt load, OA flowrate, and OA concentration.

The corresponding effect of the OA flowrate on the OA concentration generated at the reject port of RO element 1B is illustrated in Fig. 13. As anticipated, the OA generated is more concentrated when the OA flowrate decreases. This is because pressure increases with decreasing reject flowrate; thus, more water crosses the membrane and the reject is more

Table 9
Average water recoveries in the RO subsystem

RO element	Average recovery at optimized operation (%)
1A	50.9
1B	34.5
2	78
3	93.9
4	80.9

concentrated. Additionally, the concentration of the OA increases with increasing pump speed and with a higher salt loading in the OA loop.

Despite the minimal increase in power consumption, it was generally observed that operating with more salt loaded in the system is preferable. The RO subsystem operated with more stability (i.e., constant pressures, flowrates, and OA concentrations) when it was loaded with more salt.

Based on the overall performance of the DOC test unit, the optimized conditions for operating the RO subsystem were RO pump speed at 80%, OA flowrate at 0.4 l/min, and salt load of 1000 g. Feed pressure in Pass 2 was chosen at 2.07 MPa (300 psi) to maintain a proper driving force for water permeation in RO Passes 2, 3, and 4. These operating conditions were chosen because they represent the lowest energy consumption achievable at the maximal OA flowrate possible and at acceptable OA concentration. At OA flowrates higher than 0.4 l/min the stability of the RO subsystem deteriorated to an unacceptable level; permeate pressure in RO module 1A could not be met and therefore, the fresh water production rate declined below the rate of water extraction in DOC#1 and DOC#2.

It should be noted that the feed flowrates into RO elements 1A and 1B are very slow and that the recoveries are very high in the entire RO subsystem (Table 9). Experiments with the RO subsystem revealed that these operating conditions generate severe concentration polarization in RO elements 1A and 1B. More critically, these operating conditions generate eddies in the feed channels that accumulate a significant volume of gases at high pressure that cannot be swept out due to the slow feed flowrate. Both occurrences contribute to a sharp decline in the performance of the RO subsystem including sharp declines in pressures and permeation rates. Although initial performance could be restored by flushing the air out of the feed channels and flushing the elements with ultrapure water, longer term solutions are being pursued.

Using the results from the bench-scale tests (Fig. 7) and the results from the NASA DOC test unit (Figs. 12 and 13), the specific power consumption could be estimated. The calculations considered power consumption by the RO, DOC#1, and DOC#2 subsystems but ignored the contribution of DOC#2 to the total flux because very little of the raw wastewater is pretreated by DOC#2. Water flux as a function of OA concentration was used at the triply concentrated wastewater feed conditions (Fig. 7).

The effects of OA flowrate for two salt loadings and three RO pump speeds on the cost of reclaiming one liter of water are illustrated in Fig. 14. Three trends can be clearly observed. First, as the OA flowrate increases, water production is more energy intensive. This is because at higher OA flowrates, the OA is less concentrated and therefore a longer time is necessary to treat the wastewater. Second, higher salt loading results in less energy-intensive operation. This is because the OA is more concentrated, mass transfer in DOC#1 and DOC#2 is faster, and a shorter time of operation is necessary. And finally, as RO pump speed increases, the specific power consumption decreases because more water is produced.

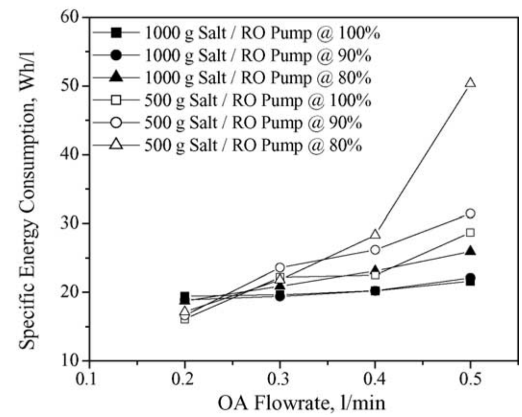

Fig. 14. Specific energy consumption as a function of OA flowrate for two salt loadings and a range of RO pump capacities.

4. Conclusions

The performances of the three membrane processes in the NASA DOC test unit were evaluated individually and in combination. DOC#1, the DO pretreatment module, can efficiently recover more than 90% of the total daily raw wastewater. The Osmotek CTA membrane was found to be highly effective in rejecting salt and organics as well as providing high water flux. However, trace amounts of surfactants were found to diffuse through the membrane; accumulation of these surfactants can threaten the OD process in DOC#2. To overcome this problem, tighter semi-permeable membranes will be evaluated for DO in the future and the effect of micelle formation on surfactant rejection in DO mode will be tested. The CTA membrane, like most RO membranes, performed very poorly in rejecting urea. DOC#2, the dual DO/OD pretreatment module, recovered only a fraction of the concentrated wastewater and urine it was designed to treat. Possible solutions include incorporation of membrane distillation (MD) in the DO/OD process or removal of urea in a separate process. Incorporation of MD involves establishment of a temperature gradient across the membranes. This is evaluated in a subsequent investigation [14]. Urea removal can be accomplished biologically or electronically through electrolysis and will be considered in the future.

The RO subsystem produces highly purified water. However, it requires redesign and reconstruction before long-term experiments start. Three major modifications must be considered. The first involves reducing the diameter of RO modules 1A and 1B to provide better mixing and reduced concentration polarization in the feed channels. This may also prevent the accumulation of gases in the feed channels. The second modification involves increasing the number of RO membrane elements. This would solve the high recovery problem that contributes to concentration polarization and gas accumulation in the RO elements. The last modification involves resizing the RO pump. The last modification is required because of the other two recommendations and it is anticipated that a larger pump will only marginally increase specific power consumption.

The specific power consumption of the DOC test unit is lower than that of the other competitive processes for wastewater reclamation in space. The DOC system essentially requires between 54–108 kJ/l (15–30 Wh/l) while the other systems require 198–1332 kJ/l (55–370 Wh/l) (Table 2). This makes further development of the DOC system very desirable and important for both space and terrestrial applications.

Results have shown that salt is lost from the OA to the wastewater stream at an average rate of 300 mg for every liter of wastewater recovered. Assuming that the lost salt cannot be recovered, the annual resupply of salt would be approximately 20 kg. This is a very low figure compared to the other technologies considered by NASA (up to 400 kg/year, from Table 2).

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