Biologically Pre-Treated Habitation Waste Water as a Sustainable Green Urine Pre-Treat Solution

W. Andrew Jackson[1], Bret Thompson[2], Ritesh Sevanthi[2], and Audra Morse[3]
Texas Tech University, Lubbock, TX, 79407

Caitlin Meyer[4] and Michael Callahan[5],
Johnson Space Center, Houston, TX, 77058

The ability to recover water from urine and flush water is a critical process to allow long term sustainable human habitation in space or bases on the moon or mars. Organic N present as urea or similar compounds can hydrolyze producing free ammonia. This reaction results in an increase in the pH converting ammonium to ammonia which is volatile and not removed by distillation. The increase in pH will also cause precipitation reactions to occur. In order to prevent this, urine on ISS is combined with a pretreat solution. While use of a pretreatment solution has been successful, there are numerous draw backs including: storage and use of highly hazardous solutions, limitations on water recovery (<85%), and production of brine with pore dewatering characteristics. We evaluated the use of biologically treated habitation wastewaters (ISS and early planetary base) to replace the current pretreat solution. We evaluated both amended and un-amended bioreactor effluent. For the amended effluent, we evaluated "green" pretreat chemicals including citric acid and citric acid amended with benzoic acid. We used a mock urine/air separator modeled after the urine collection assembly on ISS. The urine/air separator was challenged continually for >6 months. Depending on the test point, the separator was challenged daily with donated urine and flushed with amended or un-amended reactor effluent. We monitored the pH of the urine, flush solution and residual pH in the urine/air separator after each urine event. We also evaluated solids production and biological growth. Our results support the use of both un-amended and amended bioreactor effluent to maintain the operability of the urine /air separator. The ability to use bioreactor effluent could decrease consumable cost, reduce hazards associated with current pre-treat chemicals, allow other membrane based desalination processes to be utilized, and improve brine characteristics.

Nomenclature

COT	=	Wring Collector
EPB	=	Early Planetary Base
HC	=	Humidity Condensate
ISS	=	International Space Station
LEO	=	Low Earth Orbit
MABR	=	Membrane Aerated Bioreactor
TSS	=	Total Suspended Solids
UAS	=	Urine Air Separator
UPA	=	Urine Processor Assembly
VOC	=	Volatile Organic Carbon

[1] Professor, Civil and Environmental Engineering, MS 41023
[2] Graduate Student, Civil, Environmental and Construction Engineering, MS 41023
[3] Professor, Civil, Environmental and Construction Engineering, MS 41023

[4] Scientist and Project Manager Water Recovery Systems Group, EC3/Crew and Thermal Systems Division, NASA Johnson Space Center

I. Introduction

Habitation outside of low earth orbit (LEO) for extended periods will require closed loop life support systems particularly for water, which represents the greatest mass requirement. Even for extended habitation in LEO (e.g. ISS), wastewater recycling can decrease consumables reducing costs, reliability, and enable increased mission objectives. Currently, wastewater (urine +flush and humidity condensate (HC)) recycling is accomplished using combinations of physical and chemical treatment consisting of pretreatment and distillation (urine and flush only), followed by adsorptive/exchange beds as well as catalytic oxidation. Pretreatment of the urine is required to prevent biological activity and subsequent urea hydrolysis, which can comprise the urine-air separator and promote precipitate formation during distillation and ammonia volatilization. HC is not pretreated and biological growth in the storage tank is a common occurrence. For extended habitation outside of LEO, other wastewaters will likely be produced and available for recycling including hygiene, shower, and laundry effluents. These wastewaters would be biologically active and current systems inadequate for their treatment given the organic carbon load and presence of surfactants. Biological treatment as an initial processing stage could offer substantial benefits to current processing systems and resolve issues with the more diverse waste streams associated with habitation outside of LEO.

Biological treatment is ubiquitously used terrestrially and has been extensively evaluated for treatment of extraterrestrial wastewater as well (e.g. Sevanthi et al., 2016; Jackson et al., 2009; Christenson, D., 2013). Biological wastewater treatment can be used to oxidize organic matter to CO_2 and NH_4^+/NH_3 to NOx^-, which lowers the pH reducing volatilization of NH_3 and volatile organic carbon compounds (VOCs) and reducing precipitation potential. It is also possible to biologically convert NO_x^- to N_2 gas, a useful product. Advantages of biological treatment include recycling instead of storage of waste products, reduced downstream growth potential, and reduced VOC load to adsorptive beds with finite capacities. Biological treatment does require O_2 as a consumable (<3% of daily crew member consumption) but byproducts of biological treatment include metabolic water and CO_2 which can be used to produce O_2. Finally, biological waste water treatment is compatible with habitation scenarios that include plant growth as a means of processing hydroponic solutions and/or supplying water and nutrients for plant growth. One significant issue for the inclusion of biological treatment as a part of micro-gravity compatible habitation wastewater processing systems is the issue of biomass growth in fluid/air separators. For instance the urine-air separator on ISS can be comprised by biological growth.

The use of hazardous chemicals (H_3PO4 and CrO_3) can inhibit growth but are inherently hazardous and must be resupplied. Further their use produces a hazardous brine with poor dewatering characteristics and limits recovery by distillation to <85% (Jackson et al., 2014). Chemical pretreatment of other wastewaters (e.g. laundry, hygiene, shower) would require much larger chemical pretreat mass and concomitantly produce greater volumes of brines and hazardous waste. One potential solution to both enable the inclusion of biological wastewater treatment and reduce the need for chemical pretreatment would be the use of so called “green pre-treat” solutions. Green pretreat solutions are those that use chemicals that are less toxic and hazardous. Usually they substitute organic acids (e.g., citric, benzoic) for strong mineral acids (H_3PO_4 or H_2SO_4) and eliminate Cr^{+6} or replace it with other inhibitory compounds such as quaternary amines. The ability to control biological growth in the urine-air separator is a function of both the type and concentration of chemicals utilized as well as the operation of the urine-air separator.

The ISS urine-air separator system (UAS) includes a rotary type separator fan to produce negative air pressure, flush/pretreat pumps and numerous supporting mechanical equipment (Figure 1) (Carter, 2010). When activated, the separator starts to spin and a vacuum is created throughout the system by a fan located downstream of the Wring Collector (COT), a sponge filled vessel that is used to contain liquid carry over from the rotary separator (RS). Simultaneously, the dose pump starts injecting 2 pulses of ~ 1.75-1.85ml of pretreat chemical concentrate (10g/l and 1g/l) simultaneously with 2 pulses of 25-28ml flush water. Urine enters into a funnel and is pulled by vacuum into the RS. The separator contains a variable liquid volume ranging from a high of 450-500ml to a low of 150ml. When the separator volume reaches its high set point a valve opens and liquid is displaced to the urine tank, once the low set point is reached (~200ml) the valve closes. Urine void volumes can vary greatly (50 to >600 ml/event) and depending on the volume state of the RS when a urine event commences, the concentration of pre-treat chemical in the separator can vary greatly. However, assuming 6-8 urination events per crew/day, the UAS is frequently flushed except at night, reducing the potential for growth during any low concentration periods. Averaging of low and high event volumes insures the concentration of pretreat chemicals in the urine storage tank is adequate.

Two main issues could impact the ability to control growth: 1) the initiation of the flush solution at the beginning of the urination cycle rather than at its conclusion and 2) the small volume of flush solution (~54-60ml) relative to the hold-up volume of the RS (150-500ml). Increasing the flush volume would consume more potable water in the current configuration and require additional processing time by the UPA but should not lead to lower overall water recovery as the dilution of the urine would allow for higher recoveries in the distillation unit.

Inclusion of biological treatment would require a different pre-treat formulation, the low pH of the solution is itself not the main issue as dilution with other waste streams in the bioreactor and urea hydrolysis in the bioreactor would partially offset the low pH of the urine+flush wastewater. However, the use of Cr^{+6} is incompatible with biological treatment. Inclusion of biological treatment could also be beneficial to the overall system.

Excessive growth in the UAS is not desirable but the use of pre-treat is also required to prevent urea hydrolysis and subsequent pH increase with accompanying potential for precipitation and NH_3 volatilization. Inclusion of biological treatment prior to desalination eliminates or greatly reduces the need to lower pH by acid addition as the bioreactor eliminates urea and lowers the pH (5-7). We propose a new conceptual system design and pretreat regime to maintain the UAS integrity while allowing biological pretreatment. Our design also facilitates the inclusion of other wastewaters (e.g. shower, laundry, hygiene), supporting different long-term habitation architectures.

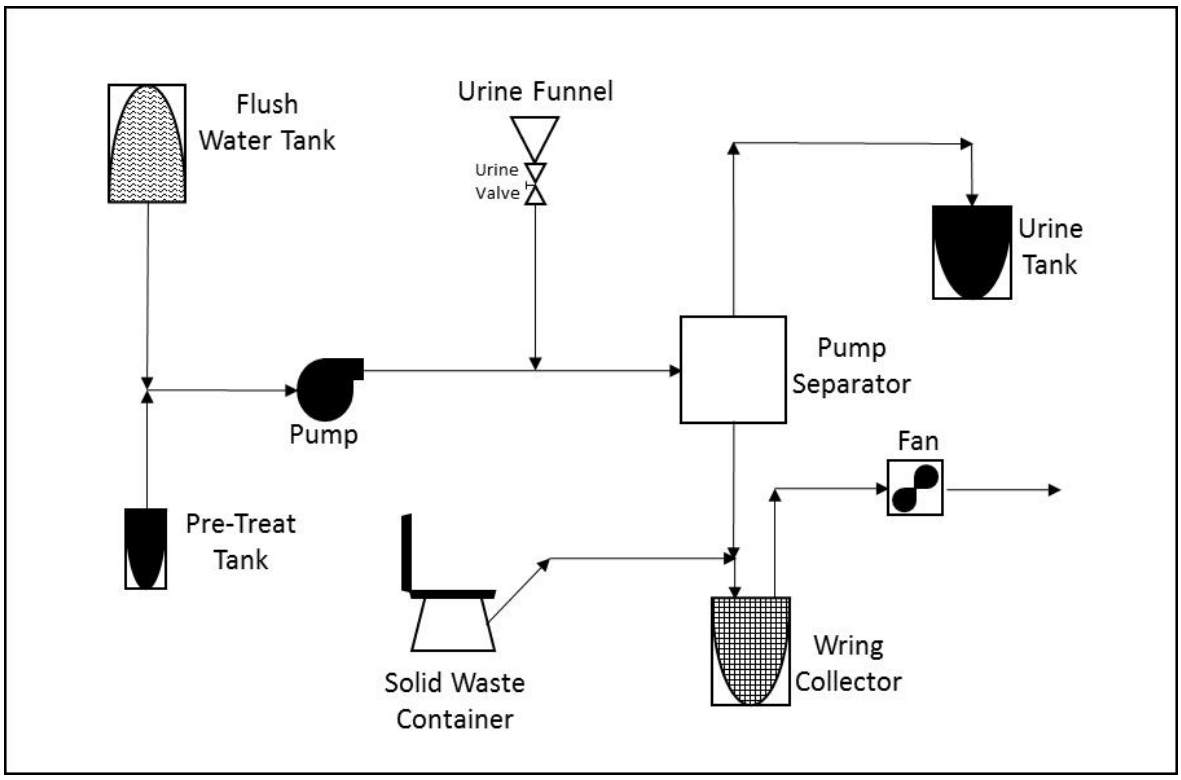


Figure 1. Conceptual diagram of the urine-air separator system on ISS.

II. Conceptual Design

Our conceptual design uses the bioreactor as an accumulator of all waste waters. While HC is the only other waste water currently produced on ISS, future missions could include shower, laundry, and/or hygiene. We propose to substitute treated bioreactor effluent for the potable water currently used for flush water, which would allow the use of larger flush volumes and even more frequent flush events (independent of any urine addition) (Figure 2). The use of greater volumes of flush water would have no negative impact on the desalination system as the flush water is returned to the biological reactor so only the volume of wastewaters entering the system (urine, condensate, shower, laundry, hygiene) are desalinated. Our configuration of using a bioreactor as an accumulator of all waste water would greatly reduce VOC load to the post processing system and would allow desalination of waste water containing surfactants by oxidizing the organic matter to CO_2. However, some form of biological pretreat is still required to prevent growth in the UAS system.

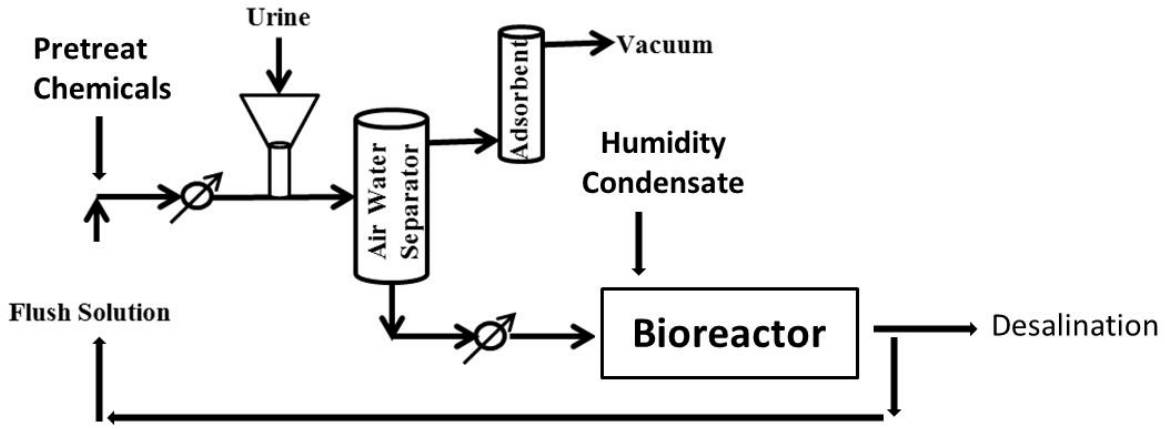


Figure 2. Conceptual diagram of the urine-air separator system with inclusion of biological treatment within the wastewater recycling system.

III. Objectives

The overall objective of this research effort was to evaluate alternative operational modes and chemical additions to control growth and maintain functionality of a UAS system in order to facilitate the inclusion of biological rectors into habitation water recycling architectures. We evaluated effluent of a bioreactor treating an ISS and EPB waste stream as well as evaluated the use of effluent from a bioreactor designed to produce low pH effluent (<3).We investigated using varying volumes of flush water and concentrations of organic and phosphoric acids. All experiments were conducted using a bench top UAS challenged with donated urine. We evaluated growth control using pH and suspended solids content of flush solutions. Solutions were tested for their ability to control growth between flush events, overnight and over a two-day dormancy period.

IV. Methods

A. Bench Scale Stability Testing

Bench scale static bottle tests were conducted to evaluate the biological stability of potential flush solutions produced from bioreactor effluent. The flush solutions evaluated were composed of either the effluent from a pilot scale bioreactor treating an ISS wastewater (urine+flush and humidity condensate) or a bench scale bioreactor designed to produce low pH effluent (<3) by additional treatment of the pilot scale bioreactor effluent. We evaluated both un-amended effluent and amended effluent, which contained citric acid, benzoic, or phosphoric acid (Table 1). Flush solutions composed of bioreactor effluent with or without chemical amendments were placed in 250ml Erlymyer flasks on a lab bench open to the atmosphere. Flasks were tested for pH and suspended solids after a one week period.

Table 1. Summary of bench top stability testing variables and final waste water quality results.

Bioreactor Type	Citric Acid (g/L)	Benzoic Acid (g/L)	Phosphoric Acid (g/L)	pH [Day 0]	TSS (mg/L) [Day 0]	pH [Day 7]	TSS (mg/L) [Day 7]
ISS	0	0	0	6.1	20.4	6.4	25.4
	0	0	2	2.6	25.2	2.4	43.25
	0	2	0	3.3	24.4	3.5	46.18
	10	0	0	2.3	26.7	2.4	154.95
	5	1	0	2.5	23.6	2.3	34.24
Low pH	0	0	0	2.3	48.6	2.4	54.35
	0	0	1	2.1	32.9	2.2	44.44
	0	1	0	2.2	30.1	2.2	32.61
	5	0	0	2.1	33.7	2.1	64.52

	2	1	0	2.1	49.8	2.1	55.56

B. Urine-Air Separator and Operational Regime

To simulate the operation of the ISS UAS, we used a stainless steel cylindrical container to mimic the rotary separator with a total volume ~750 ml which was placed on a stir plate (Figure 3). A magnetic stir bar inside the container was used to rotate the liquid during operation. The container was closed to the atmosphere using a plexiglass top in which two ports were placed. One port served as the influent for urine and flush solutions while the other was used to connect to a vacuum source. A third port was located on the side of the container, which was used to withdraw solution from the rotary separator during operation. The port height insured that at least 150ml of liquid was always maintained in the separator. A container filled with sponges was placed between the stainless steel rotary separator and vacuum source to simulate the wring collector. Pumps were used to withdraw fluid from the rotary separator and to add flush solution during operation. Urine was poured into a funnel while the system was under vacuum.

The system was operated daily throughout each work week over a period of 6 months. The system was operated from 1-5 times daily depending on urine donation availability. Only fresh urine was used to operate the system. The system was operated by turning on the vacuum and stir plate and pouring fresh donated urine into the funnel. The effluent pump was set at a rate of 80ml/min. Once the system had returned to a nominal 150ml volume, the flush solution pump was activated. Flush solution volume and composition varied (Table 1). During the flush cycle the volume of liquid in the RS was kept constant by withdrawing liquid at rate equal to the influent flow rate of the flushing solution. This volume was collected and is referred to as the "final flush". Once all the flushing solution had been pumped into the system, the influent and effluent valves were closed and the stir plate turned off until the next event.

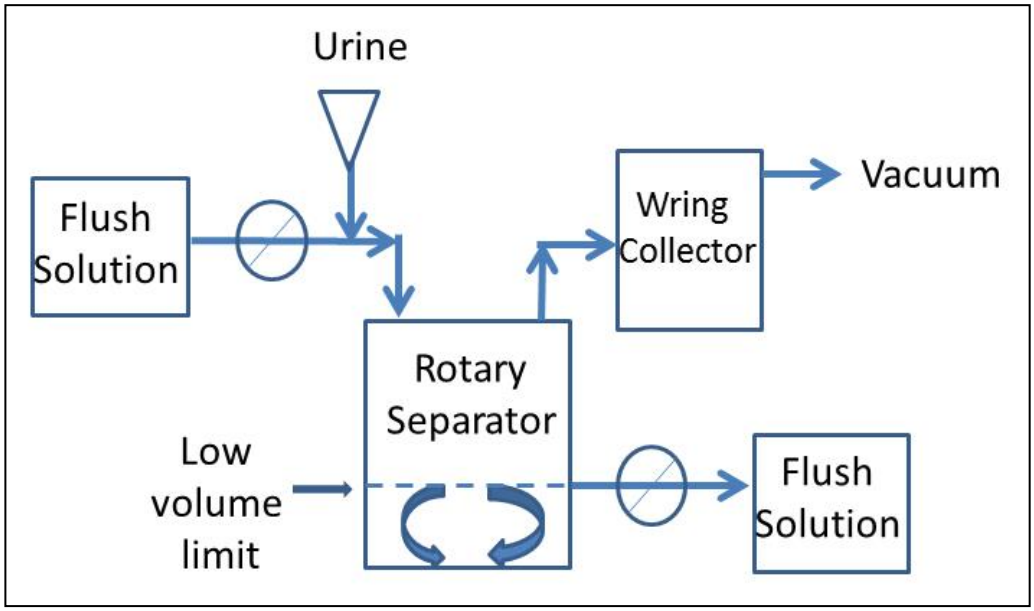


Figure 3. Schematic of the bench top UAS system for testing of flush solutions produced form bioreactor effluent.

The flush solution was composed of either the effluent from a pilot scale MABR treating an ISS wastewater (urine+flush and humidity condensate) or a bench scale bioreactor further treating the MABR effluent designed to produce low pH effluent (<3). These solutions were amended with organic acid at varying concentrations (Table 2)

Samples were taken of each urine donation, flush solution, as well as the final effluent composite of the flush solution (final flush). We also sampled the RS prior to the beginning of each cycle (pre-urination). All samples were tested for pH. We also evaluated the change in suspended solids over various dormancy periods including overnight and over weekends corresponding to approximately 17 hours and 65 hours, respectively. This was accomplished by measuring suspended solids in the final flush of the day and in the reactor after each dormant period.

Table 2. Summary of experimental parameters evaluated during operation of the urine-air separator.

Bioreactor Type	Flush Volume (mL)	Days	Number of Events	Citric Acid (g/L)	Benzoic Acid (g/L)	Phosphoric Acid (g/L)
EPB	450	6	6	5	0	0
ISS	200	16	23	5	0	0
	200	18	58	5	1	0
	100	15	33	5	1	0
	50	13	21	5	0	0
Low pH	450	8	15	0	0	0
	200	13	33	0	0	0
	200	29	78	5	1	0

V. Results

A. Bench Top Stability Testing of Flush Solutions

The effluent from a MABR treating an ISS type wastewater had an initial pH of 6.1, which was higher than acid amended ISS wastewater effluent (pH 2.6-3.3). Biological effluent with or without acid amendments maintained a constant pH over the 7 day evaluation period. Acid amendments only slightly lowered the pH (2.1-2.2) of the low pH bioreactor effluent (pH=2.3). TSS of the ISS wastewater effluent increased slightly (20.4 o 25.4 mg/l) over the 7 day incubation period with larger variations in acid amended ISS effluent but with the exception of the 10g/l citric acid amended test, TSS remained below 50 mg/l. Similar results (no substantial increase) were observed for the low pH effluent with and without acid amendments. Overall the results demonstrated that raw bioreactor effluent and low pH bioreactor effluent are stable over time and supported further testing using a simulated UAS.

B. Flush Solution Testing Using a Benchtop UAS

The bench top UAS has been operating since September 30th 2016. To date the UAS has processed 69L of urine over a 157 day period. While the number of events per day varied, with the exception of the December holidays (December 16th- January 4th) the system generally received >3 events per work day (Figure 4). Urine volume per event varied from 50 to 500 ml with an overall average of 248±86ml. The pH of the urine across all events varied from 4.7 to 7.2 with an overall average of 5.4±0.4 (Figure 5). While pH and TSS in the RS varied for each flush solution tested visual observations did not support biofilm or floc formation within the system. All tubing and connections in the system are standard 0.55cm diameter (similar to ISS) and no tubes or connections were replaced or cleaned over the 157 day operational period. It should be noted that unlike the ISS system, which is operated on a more frequent basis (more events per day (~32) and longer cycle each day (~16 hour), our system was operated only ~3 times per day and only over a 6-hour period. Further, the system was not operated over each weekend or for ~19day period over the December holiday. The lack of visible growth generally supports the ability to control excessive growth using the range of flush volumes, chemical additives, and biologically stabilized wastewater. While visible growth did not occur, the pH in the system and measurable growth (TSS) did vary between flush compositions.

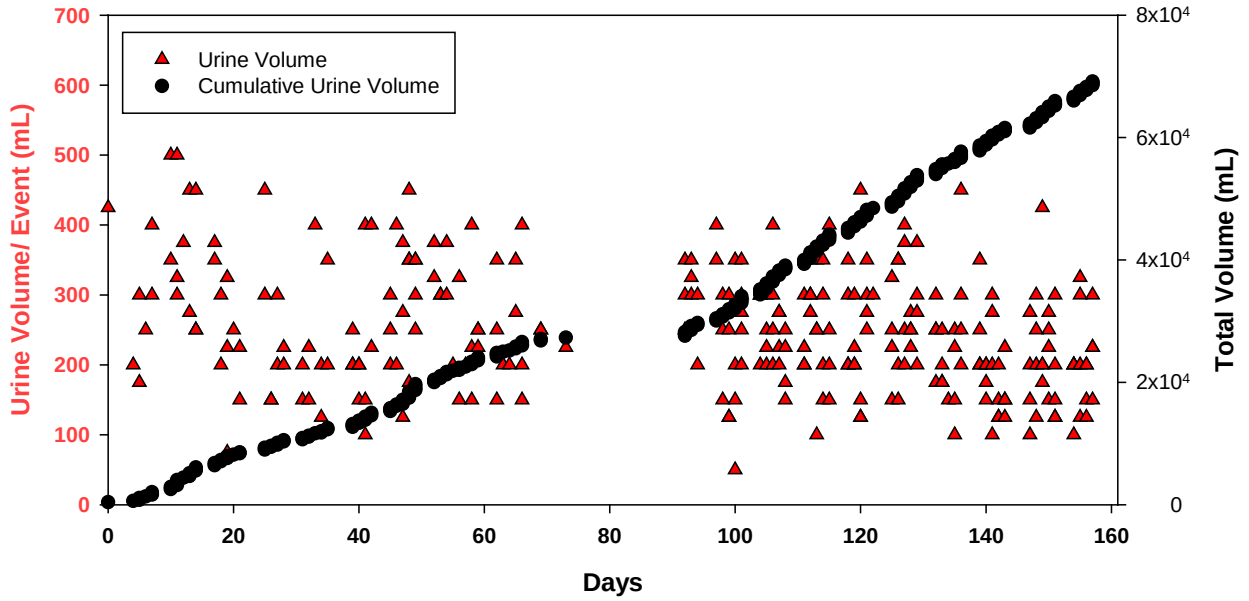


Figure 4. Cumulative urine volume processed and urine volume per event with respect to time.

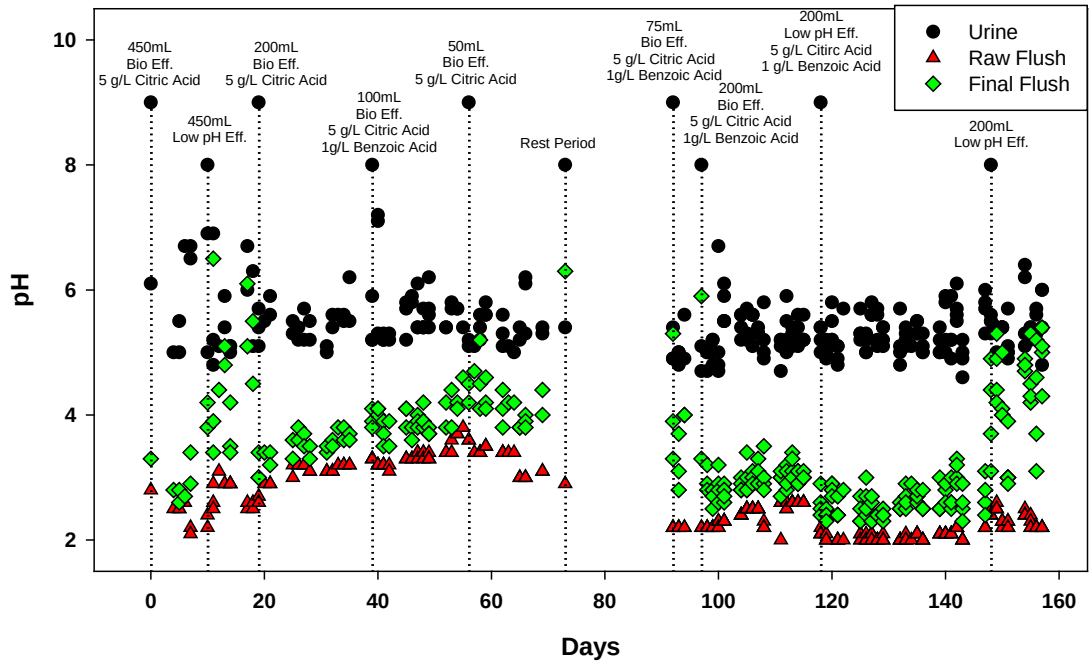


Figure 5. Variation in the pH of raw urine, flush solution, and final RS solution over the testing period. The time period over which each flush solution was tested is indicated by vertical dashed lines and text.

The pH of the flush solution using ISS treated bioreactor effluent (pH 5-7) amended with citric acid or citric acid and benzoic acid was <3.3±0.2 on average. When only citric acid (5g/l) was used to pretreat the bioreactor effluent, the pH of the final flush (volume equal to added flush volume) increased from 2.8 to 4.2 as flush volume decreased from 450ml to 50ml (Figure 6). The average pH of the solution in the RS prior to the next event (pre-urine sample), which includes both events the same day or the next work day (overnight or over weekend), had a similar range to the pH of the last flush (2.8 to 4.2) suggesting the pH in the RS was relatively stable. Addition of benzoic (1 g/l) to the citric acid amended bioreactor effluent did not consistently change the pH (3-4.2) of the flush solution pH, final flush,

or pre-urine samples compared to test points without benzoic (Figure 5). However, as previously observed for bioreactor effluent amended with citric acid only, larger flush volumes (200ml vs 100ml) produced lower final solution and pre-urine pH values.

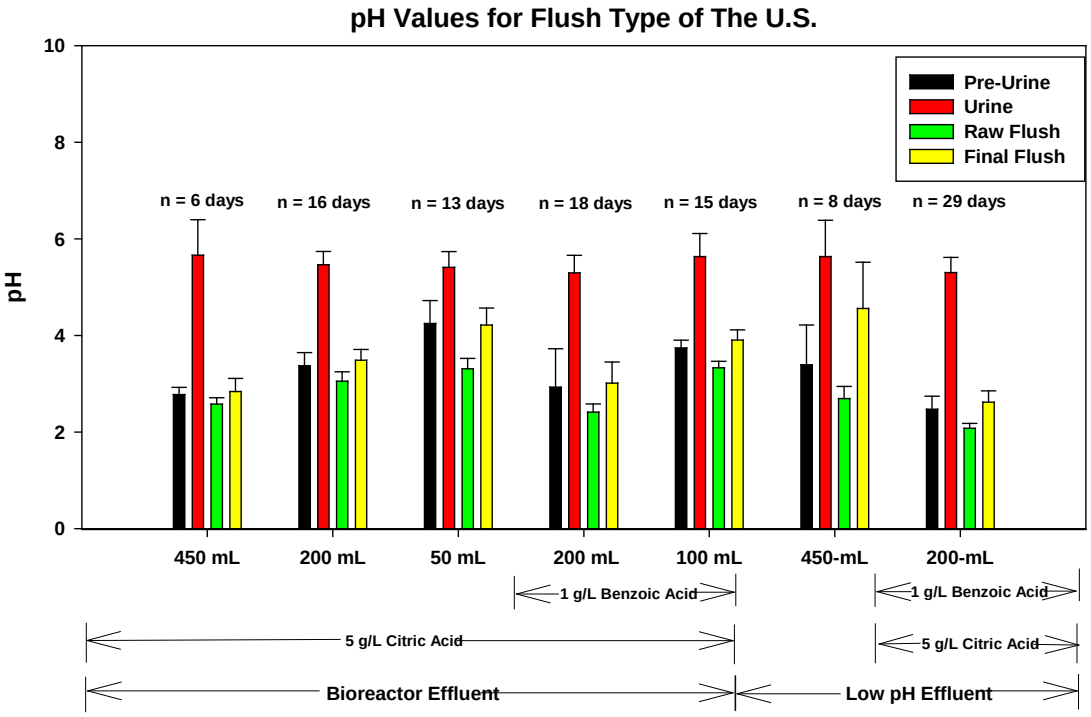
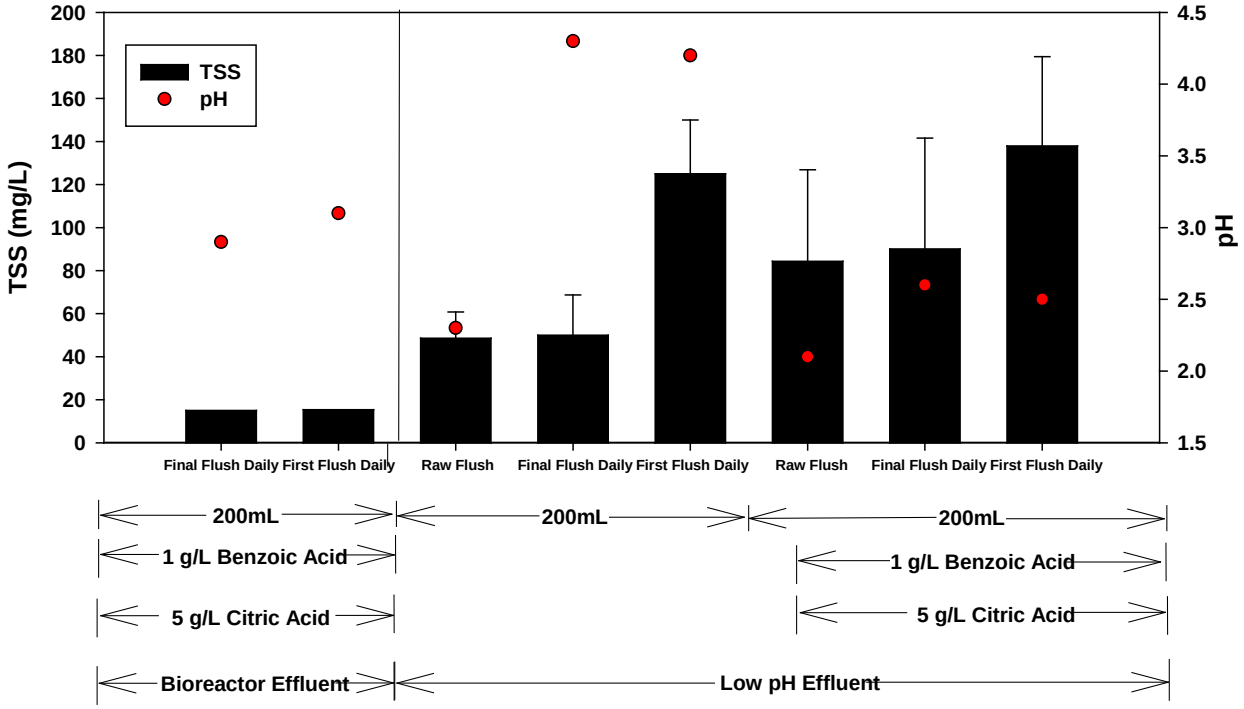


Figure 6. Variation in average pH of various flush solution compositions, urine, final RS solution and initial RS solution prior to the next flush event. Composition and flush volume is indicated below the X axis.

We also evaluated effluent from a bioreactor designed to produce a low pH effluent specifically for flushing. The effluent has a pH of 2.3±0.1. We evaluated the low pH bioreactor effluent with and without benzoic and citric acid. Addition of the benzoic and citric acid slightly lowered the pH (2.1 ±0.1) of the flush solution (Figure 6). The average pH of the final flush and pre-urine sample (4.3±0.8 and 4.1±1.3, respectively) using 450 ml of unamended (no organic acid) low pH bioreactor effluent was higher compared to bioreactor effluent with organic acid amendment. Low pH bioreactor effluent (200ml flush volume) amended with organic acid produced final flush and pre-urine samples with a pH (<3) similar to bioreactor effluent amended with organic acids but using 450 ml of flush solution.

For all the above testing, it should be noted that the pH of the total effluent from the RS per event, which includes urine and flush water would be higher. The pH of the volume removed from the RS during urine addition but prior to flushing ranged from 3-5 (data not presented). In our proposed architecture, this volume would be transferred to the bioreactor and thus would not be of concern and in any event the pH is low enough to prevent any significant NH_3volatization, as less than 0.01% of the total N would be in the NH_3 form at a pH of 5. However, while we maintained low pH values in the RS for all flushing solutions tested, low pH (2-4) alone does not necessarily equal a lack of growth. To evaluate growth directly, we monitored changes in TSS in the RS as well.

In order to further evaluate the ability of the bioreactor effluent flush solutions to control growth in the UAS, we occasionally measured the TSS in the RS after the last flush and prior to the first flush the following work day (14-20 hours later) for a subset of the flush solutions. The pH was generally stable overnight but generally TSS increased by a factor of 2, although the final concentration of TSS was still relatively low. This testing only evaluated flush volumes of 200ml which given the RS hold up volume (150ml) maybe insufficient to remove residual urine in the RS. The residual urine is volume is insufficient to increase the pH to values that would cause NH_3 volatilization or promote precipitation but does appear to facilitate some limited growth. We are in the process of testing larger flush volumes as well as the use of H_3PO_4 in place of organic acids.



VI. Conclusions

Our results support the use of bioreactor effluent to serve as a UAS system flushing solution, if amended with organic acids. The low pH bioreactor effluent may also be acceptable as a flushing solution without acid amendments during daily use although the complexity of an additional biological processor to produce the low pH solution would need to be considered compared to storage of organic acids. While we observed some increase in suspended solids most likely due to small amounts of residual suspended growth, we have not observed any biofilm growth or mold/fungal growth in the system. If future results continue to support the ability of amended bioreactor effluent to maintain UAS system functionality, the replacement of the current hazardous pretreat chemicals with the inclusion of a bioreactor could cause a cascade of advantages to the overall habitation system. Not only would the replacement of the current pre-treat chemicals reduce consumption of potable water and the hazards with the required use and storage of large volumes of pretreat solution required for extended missions out of LEO; but the inclusion of the bioreactor would also reduce VOC carry over to the post processing system, increase brine quality, and recovery by distillation, allow inclusion of membrane based delamination systems, and treatment of other waste streams such as shower, hygiene, and laundry.

Acknowledgments

We would like to acknowledge the Next Generation Life Support Program and AES program at NASA and the NASA Space Technology Research Fellowship program (NNX13AL52H) for funding this work. This work was also made possible by the efforts of the following student workers Kendall Chattman, Lakshmanan Somasundaram. We also appreciate the helpful knowledge and advice of Stephanie Walker in regards to operation of the ISS UAS.

References

Carter, D.L., "Status of the Regenerative ECLSS Water Recovery System", *Proceedings of the 40th International Conference on Environmental Systems*, AIAA-758248, 2010.

Christenson, D.; Morse, A.; Jackson, W. A.; Pickering, K. D.; Barta, D. J., "Optimization of a Membrane-Aerated Biological Reactor in Preparation for a Full Scale Integrated Water Recovery Test". *Proceedings of the 43rd International Conference on Environmental Systems*, AIAA-2013-3335, 2013.

Jackson, W.A., Barta, D.J., Anderson, M.S., Lange, K.E., Hanford, A., Shull, S.A., and Carter, L. (2014). Water Recovery form Brines to Further Close the Water Recovery Loop in Human Spaceflight. *Proceedings of the 44th International Conference on Environmental Systems.* ICES-2014-186

Jackson, W. A.; Morse, A.; McLamore, E.; Wiesner, T.; Xia, S., Nitrification-Denitrification Biological Treatment of a High-Nitrogen Waste Stream for Water-Reuse Applications. *Water Environment Research* **2009,** *81* (4), 423-431.

Sevanthi, R., Christenson, D., Morse, A., and Jackson, W.A. "Impact of waste stream composition and loading regime on the performance of a new flight compatible membrane-aerated biological reactor," Proceedings of the 46th International Conference on Environmental Systems, ICES-2016-413, 2016