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Treatment of Spacecraft Wastewater Using a Hollow Fiber Membrane Biofilm Redox Control Reactor

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Abstract The purpose of this project was to develop and evaluate design concepts for biological treatment reactors for the purification of spacecraft wastewater prior to reverse osmosis treatment. The motivating factor is that wastewater recovery represents the greatest single potential reduction in the resupply requirements for crewed space missions. Spacecraft wastewater composition was estimated from the characteristics of the three major component streams: urine/flush water, hygiene water, and atmospheric condensate. The key characteristics of composite spacecraft wastewater are a theoretical oxygen demand of 4519 mg/L, of which 65% is nitrogenous oxygen demand, in a volume of 11.5 liters/crew-day. The organic carbon to nitrogen ratio of composite wastewater is 0.86. Urine represents 93% of nitrogen and 49% of the organic carbon in the composite wastestream. Various bioreaction scenarios were evaluated to project stoichiometric oxygen demands and the ability of wastewater carbon to support denitrification. Ammonia nitrification to the nitrite oxidation state reduced the oxygen requirement and enabled wastewater carbon to provide nearly complete denitrification. A conceptual bioreactor design was established using hollow fiber membranes for bubbleless oxygen transfer in a gravity- free environment, in close spatial juxtaposition to a second interspaced hollow fiber array for supplying molecular hydrogen. Highly versatile redox control and an enhanced ability to engineer syntrophic associations are stated advantages. A prototype reactor was constructed using a microporous hollow fiber membrane module for aeration. Maintaining inlet gas pressure within 0.25 psi of the external water pressure resulted in bubble free operation with no water ingress into hollow fiber lumens. Recommendations include the design and operational testing of hollow fiber bioreactors using 1. partial nitrification/nitrite predenitrification, 2. limited aeration for simultaneous nitrification/denitrification or for nitrite reduction/ammonia oxidation, and 3. hydrogenotrophic denitrification.

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1. INTRODUCTION

For long term crewed missions into space, the greatest potential for reducing the resupply requirements for any life support resource is the recovery and reuse of wastewater produced by humans (1). Spacecraft wastewater (i.e., urine, atmospheric humidity condensate, and hygiene water) represents the largest projected wastestream in crewed space systems, with per capita production estimates ranging from 10 to 27 L/day (2,3). Biological treatment can be used to remove organics and nitrogen in the water recovery train, prior to reverse osmosis treatment. Targets of spacecraft biotreatment are physiological organics and nitrogen, hygiene water organics (surfactants), volatile organic chemicals in wastewater or in the supplied spacecraft atmosphere gas, and specific organics such as antibiotics and personal care products. The technical challenge of employing biological treatment as one component within spacecraft water recovery systems is to design bioreactors that are small, reliable, resistant to perturbation, easily maintainable, and safe, while producing an effluent quality suitable for post treatment with reverse osmosis membranes. Supporting feedstocks should be readily available. The Membrane Biofilm Redox Control Reactor is ideally suited to these requirements. Hollow fiber membranes provide a bubbleless gas supply that is suitable for gravity free operation in space, and have a high surface area per reactor volume for gas transfer and biofilm attachment. The corresponding volumetric reaction rates are high, resulting in a small volume and mass requirement. The membrane biofilm reactor is also well suited to space flight application because it is a semi-enclosed system that will minimize operator attention and the exposure of the spacecraft atmosphere to microbial populations within the bioreactor. The reactor can also be designed and operated to provide for varied redox environments to achieve total nitrogen removal. Terrestrial applications are limited but are being actively researched for both oxygenation reactions (4-12), simultaneous nitrification/denitrification (13-14), and hydrogenotrophic reactions (15-18).

2. WASTEWATER COMPOSITION

Spacecraft wastewater production and composition was estimated from wastewater formulations in the Advanced Life Support Baseline Values and Assumptions Document (2), recent stated estimates from Johnson Space Center (3), and estimates of specialty chemicals usage for personal hygiene and the chemical composition hygiene products (19,20). A summary of wastestream volume and composition is presented in Table 2. The three major components are urine with flush water, condensate, and hygiene water. The hygiene water carbon and nitrogen content were estimated based on the chemical composition of a commercial surfactant and a final surfactant concentration of 461 mg/L in the composite wastewater (20). Urine with flush water has the highest nitrogen and organic carbon concentrations, and accounts for 93% of the nitrogen

Table 1 Spacecraft Wastestream Composition

Source	Daily volume per crew	Total nitrogen (mg/L)	Total organic carbon[1] (mg/L)	NOD[2] (mg/L)	COD[3] (mg/L)	ThOD (mg/L)
Urine with flush water	2.0	3423	1556	15643	4512	20156
Condensate	2.3	38	358	174	1038	1212
Hygiene water	7.2	60	330	274	957	1231
Composite: Urine/ condensate/hygiene	11.5	640	549	2927	1592	4519

[1] Total organic carbon, not including urea organic carbon in urine
[2] Nitrogenous oxygen demand, computed using $Y_{O/N} = 4.14$
[3] Chemical oxygen demand, computed using COD/TOC = 2.9
[4] Theoretical oxygen demand exerted, computed as the sum of COD and NOD

and 49% of TOC in the composite wastewater. The estimated nitrogenous oxygen demand of the composite wastestream is 2927 mg/L, versus an estimated chemical oxygen demand of 1592 mg/L. Hygiene water and condensate comprise 37% and 13% of organic carbon, respectively. The major fraction of urine nitrogen (81%) is as urea or its hydrolysis product, ammonia nitrogen. Creatinine and hippuric acid comprise 77% of the urine organic carbon. The ratio of organic carbon to nitrogen in the composite wastestream is 0.86, indicating that the composite wastestream is nitrogen rich and organic poor from a classical nitrogen removal perspective.

3. BIOCHEMICAL TRANSFORMATIONS

Aerobic heterotrophic oxidation, aerobic nitrification, and heterotrophic anoxic denitrification are well-characterized processes applied extensively in environmental biotechnology, and experience is increasing in autotrophic denitrification. Recently, microorganisms have been elucidated that catalyze denitrification of nitrite using ammonia as electron donor under anaerobic conditions (21,22). Several microorganisms with anaerobic ammonia oxidation capability are known. Similar to nitrifiers, these microorganisms are autotrophs with low yield coefficients and low growth rates. In general, the ubiquity of metabolic capability for anaerobic ammonia oxidation in the environment is unknown. The bioreactor conditions needed to manifest anaerobic nitrite reduction/ammonia oxidation are long solids retention times, a limited oxygen supply, and appropriate pH. These conditions can be established in a hollow fiber membrane bioreactor, and may be similar to conditions that would promote total nitrogen removal in a single hollow fiber membrane biofilm reactor by nitrification/ denitrification.

To provide an evaluation framework for reactor design, idealized bioreaction scenarios were developed for spacecraft wastewater using stoichiometric relationships between carbon, oxygen, and nitrogen derived from the electron equivalents involved in oxidation and reduction reactions

Table 2. Spacecraft wastewater bioreaction scenarios.

	Bioreactions	O_2 demand, mg/L	Alkalinity, mg/L as $CaCO_3$	Residual NO_3 or NO_2, mg/L
1	Complete organics oxidation and complete nitrification to NO_3	-4133	-4515	640
2	Complete nitrification to NO_3; denitrification with available TOC	-2652	-3209	275
3	Complete nitrification to NO_2; denitrification with available TOC	-1985	-3064	31
4	Complete organics oxidation and balanced nitrification/ anaerobic ammonia oxidation	-2577	-2427	0

and accounting for some degree of net biomass synthesis. Four scenarios are listed in Table 2. In principal, any of these idealized stoichiometries can be established in an appropriate reactor configuration. The first, completely aerobic oxidation of organics and nitrification of ammonia to nitrate, exerts the greatest exerted oxygen demand, consumes the most alkalinity, and has the highest nitrogen residual as nitrate. In scenario 2, ammonia is completely nitrified and nitrate is denitrified with wastewater organics. Denitrification results in a reduction in oxygen demand and in alkalinity consumption. A residual nitrate nitrogen of 275 mg/L is present because there are insufficient electron equivalents of organic carbon in spacecraft wastewater to completely denitrify nitrate. Reaction scenario 3 is similar to scenario 2, except that ammonia is nitrified only to the +III oxidation state (nitrite). The oxygen demand and alkalinity consumption are lowered. Since nitrite denitrification requires less organic carbon than nitrate, the residual nitrogen is reduced to only 31 mg/L (as nitrite), and the requirement for supplemental carbon (or hydrogen) for denitrification is reduced. Reaction scenario 4 combines aerobic organic oxidation, nitrification of 50% of kjeldahl nitrogen to nitrite, and nitrite reduction/ammonia oxidation under anaerobic conditions. This bioreaction scenario is calculated for complete nitrogen removal, and results in a low alkalinity consumption.

Although the reaction scenarios presented in Table 2 will never be totally segregated in an operating bioreactor, the analysis does allow some important conclusions. First, an aerobic first stage treatment would require the highest oxygen transfer capacity and a high dose of electron donor for nitrate denitrification. Predenitrification, simultaneous nitrification/denitrification, or a segregated wastestream feeding approach could be used to exploit wastewater organics for denitrification. The fact that 49% of composite wastewater organics are contained in the urine stream that contains the bulk of wastewater nitrogen suggests that predenitrification or simultaneous nitrification/denitrification should be pursued. Nitrifying ammonia to nitrite, rather than allowing it to proceed to nitrate, can produce lower. Hydrogenotrophic denitrification and nitrite reduction/ammonia oxidation are processes that can be integrated within hollow fiber reactors with limited oxygen supply, or applied as follow up processes in downstream reactor compartments.

4. DESIGN CONCEPTS

Oxygen supply is critical to organic and hydraulic loading. Organic removal rates as high as 27 kg/m^3-day of total chemical oxygen demand have been achieved in hollow fiber membrane bioreactors operated on pure oxygen (11). These high volumetric rates suggest that spacecraft wastewaters in an aerobic only treatment mode (Table 2, scenario 1) require retention times on the order of 4 hours for hollow fiber specific surface areas of 322 m^{-1}. Denitrification using wastewater organic carbon would reduce oxygen demand and proportionally reduce required retention times, although ammonia oxidation (nitrification) would still comprise the major fraction of oxygen demand. Experimental testing using analog spacecraft wastewater is needed to gain confidence in projecting required reactor loadings and retention times.

Hollow fiber membranes are manufactured down to diameters on the order of 250 um, which provides a high surface area per reactor volume for transfer of gases and for biofilm attachment. Packing densities of 2 to 5% can achieve specific surface areas of 150 to 500 m^2/m^3 reactor volume while maintaining centerline separation distances of 0.5 to 2 mm. These factors can result in very high volumetric reaction rates. Hollow fiber membranes can be manufactured in dense, microporous, or composite configurations (Table 3).

Table 3 Candidate Hollow Fiber Membrane Materials

Material	Advantage	Limitation	Characteristic
Microporous hydrophobic polypropylene	Very high oxygen permeabilities	Must maintain lumen pressure below bubble point or pressurize reactor liquid	Liquid film diffusional resistance to mass transfer
Nonporous silicone rubber	Can be operated at high lumen gas pressures without bubble formation	Not available in small diameters	High membrane resistance to mass transfer
Nonporous composite	Very high oxygen permeabilities Can be operated at high lumen gas pressures without bubble formation Resistant to abrasion Mechanical strength	Limited availability	Thin nonporous membrane layer Liquid film diffusional resistance to mass transfer

For microporous fibers, lumen gas pressure must be maintained below the bubble point for bubbleless operation, and a high lumen pressure can be maintained if the liquid is pressurized so the transmembrane pressure does not exceed 2 to 3 psi or less. Accumulation of water vapor in the fiber lumen has been reported with high shell side pressures (15). Hollow fiber membranes can be operated in a flow through or a dead end mode. Dead end mode can potentially achieve 100% gas transfer efficiency, but may result in the accumulation of water vapor and nitrogen in the lumen; either could reduce oxygen transfer or cause operational problems.

For spacecraft application, nonporous hollow fibers avoid the need to balance lumen and external water pressure. Nonporous silicone membranes are ready available and have the highest intrinsic permeabilities of commercially available dense materials, and are recommended for evaluation. Their oxygen transfer capability can be enhanced with pure oxygen if needed. If silicone membranes prove inadequate, they can be substituted with a composite membrane with higher areal oxygen delivery rates or higher specific surface area.

Liquid recycle serves two functions: diluting the feedstream and maintaining superficial velocity. Recycle results in more uniform solute concentrations across the bioreactor and avoids potential toxicity. A recycle ratio of 10 to 20 can provide substantial reduction of the actual influent concentration entering the reactor. For a wastewater ammonia-N of 640 mg/L and 95% removal, recycle ratios of 10 and 20 reduce the actual reactor influent ammonia-N levels to 87 mg/L and 61mg/L, respectively. Another potentially important benefit of recycle is to even out pH gradients that could occur, for example, from nitrification. In a reactor with no recycle, destruction of alkalinity by nitrification could make it difficult to adjust the feed pH at the influent end in order to achieve a pH all across the length of the reactor within the acceptable range for nitrifying organisms. Recycle would reduce the pH gradient and result in a more uniform concentration profile. The second important effect of recycle flowrate is on the superficial liquid velocity. Superficial velocity produces bulk liquid transport and mixing, mass transfer from bulk liquid to and from attached biofilms, and shearing or sloughing of attached microbial biomass. It may also help to avoid channeling. At long reactor residence times, the need for bulk liquid movement (as assessed by superficial velocity of Reynolds number) may predominate over the need for wastewater dilution, and high effective recycle ratios would produce a reactor liquid phase approaching completely mixed. A wide range of recycle rates have been used in experimental studies of hollow fiber bioreactors, and optimal recycle ratios must be experimentally determined.

Limited oxygen supply operation can be used to achieve simultaneous nitrification and denitrification of terrestrial wastewaters in a single reactor, and deserved evaluation for spacecraft wastewaters. Limited oxygen supply operation can be established by gradually reducing the lumen gas pressures in a nitrifying reactor. Continuous operation with limited oxygen supply could lead to a high oxygen transfer efficiency and reduced oxygen concentrations and denitrification in outer biofilm layers. Denitrification under this scenario requires an adequate supply of electron donor. In limited oxygen supply operation, heterotrophic utilization of organics could exert a strong competition with nitrifiers for oxygen at the membrane surfaces and limit the availability of electron donor for denitrification.

The Membrane Biofilm Redox Control Reactor is a new alternative reactor configuration that contains two separate hollow fiber gas supply systems: one for oxygen and the other for hydrogen gas. Hollow fiber membranes from the two systems are interspaced in close spatial juxtaposition, allowing solute transport to and from biofilms attached to either the oxygen or hydrogen membranes. The Membrane Biofilm Redox Control Reactor is a new treatment concept that has not been attempted for any wastestream, but could provide substantial benefits to spacecraft wastewater recycling. Both O_2 and H_2 will be produced on future spacecraft from the electrolysis of water, so H_2 gas will be an available feedstock. A possible strategy for complete nitrogen removal is an alternating sequence of 1. oxygen on/hydrogen off followed by 2. oxygen off/hydrogen on. Nitrification and organics oxidation would occur during oxygenation period, while denitrification would occur in the hydrogenation period. Based on alkalinity consumption by nitrification and its restoration by denitrification, monitoring of pH and feedback to gas supplies could be developed as a process control strategy. The alternating use of oxygen and hydrogen, as well as the ability to alter the timing and intensity of the supply of these oxidizing and reducing gases, will create a highly flexible and versatile reactor system. The ability to precise engineer desired microbial syntrophic associations will be substantially enhanced over current technology.

Nitrite reduction to N_2 using ammonia as electron donor is a reaction sequence that could become established in bioreactors operated in a limited oxygen regime. Oxidation of about 50% of ammonia to nitrite, followed by nitrite reduction/ammonia oxidation, would result in complete nitrogen removal. To accumulate nitrite in the first step, an oxygenation strategy in needed to decouple nitrite oxidation from ammonia oxidation to nitrite. One potential strategy is pulsed on/off operation of oxygen supply. This could be achieved by relatively rapid step up/step down alternations in lumen gas pressure. Each pressure upstep would result in a nonsteady diffusion pulse of molecular oxygen to biofilm reaction sites. During the pressure-off downstep, oxygen diffusion to catalytic sites will cease, and nitrite from partial nitrification would counterdiffuse with ammonia from the bulk liquid to reach ammonia oxidizing/nitrite reducing bacteria in the anoxic biofilm. Nonsteady oxygenation during a limited duration pressure pulse has a similarity to the surface renewal theory of oxygen mass transfer across open gas liquid interfaces, which hypothesizes non-steady diffusion of oxygen from liquid packets that have temporally limited surface contact intervals.

An approach to optimize the spacecraft wastewater organic carbon for nitrogen removal is predenitrification (Figure 1) with an aerobic, limited oxygenation regime reactor that nitrifies to nitrite. An important feature of this design is the recycle of nitrite in the aerobic reactor effluent to the denitrification reactor, allowing efficient use of influent organic carbon to fuel denitrification. In Figure 1, a hydrogenotrophic denitrifying reactor is shown for follow up removal of the nitrite that is not recycled to the anoxic reactor. Ultrafiltration retentate is partially recycled to the hydrogenotrophic reactor influent.

5. PROTOTYPE

A laboratory scale hollow fiber membrane oxygenation bioreactor (Figure 2) was constructed to evaluate its ability to nitrify and achieve total nitrogen removal from an analog spacecraft wastestream. The reactor consisted of a vertically oriented column with an overall length of 42 cm and an internal diameter of 5.7 cm. The reactor was subdivided by a horizontal ported flow distribution plate into an upper reaction zone (30.5 cm), and a lower mixing zone. Wastewater flow is mixed with recycle flow before entering the reaction zone. A CellGas hollow fiber oxygenation module (Model CG2M-020-01N, Spectrum Labs, Inc., Laguna Hills, CA) was

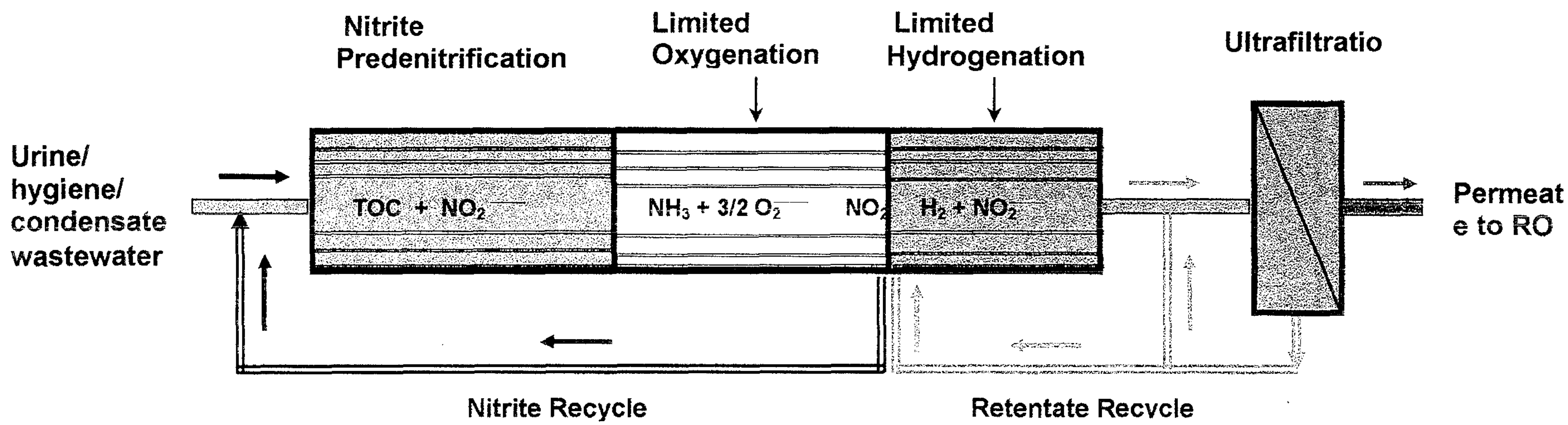

Figure 1. Hollow Fiber Bioreactor Zonation

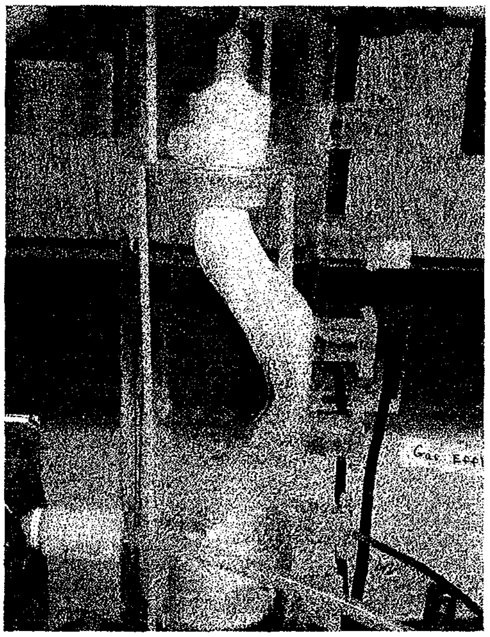

Figure 2. Prototype Hollow Fiber Biofilm Reactor

installed for oxygen transfer. The module consisted of a bundle of approximately 1540 polypropylene hollow microporous membrane fibers potted into a PVC endcap and flow header. The hollow fibers had an active length of 20.7 cm, 0.20 mm inner diameter, 0.25 mm outer diameter, a 250 um wall thickness, and a pore size of 0.05 um. The module had 0.25 m^2 of surface area based on the outer diameter of the hollow fiber, which provided a reactor specific surface area of 322 m^{-1}. The CellGas module was connected to horizontal pipes that were mounted through horizontal ports. The gas was supplied by an air cylinder, flowed through a metering bubble flowmeter (Shorate), into and through the CellGas module, and through an effluent metering valve. Pressure gages were used to measure inlet and outlet gas pressure; both gages were placed on the CellGas module side of the flow meters.

A series of tests were performed to determine the range of pressures and pressure differentials that would result in no bubble formation or water ingress. The pressure drop between module inlet and module outlet remained at approximately 1.5 psi throughout the tests. Across a range of water pressures of 3 to 10 psi, maintaining an inlet gas pressure in the range of 0.25 psi above or below the reactor water pressure resulted in neither bubble formation nor water ingress into the lumen. When inlet gas pressure was 0.75 psi or more greater than water pressure, bubbles were formed at all water pressures. An inlet gas pressure 1 psi or more below water pressure resulted in water ingress in most cases. Operation with an inlet gas pressure within 0.25 psi of water pressure precluded bubble formation and water ingress.

Gas liquid mass transfer capabilities were evaluated by applying the abiotic dynamic method to determine a first order oxygen transfer rat constant K_La. Forward flow was discontinued and the reactor was operated as a closed water system using the recycle pump. Deionized water was deoxygenated with sodium sulfite and spiked with 2.0 mg/L cobalt chloride as catalyst. The water was pumped into the reactor, the recycle pump was turned on, and the airflow was started.

The water temperature was approximately 24C during oxygenation tests. The oxygen transfer rate constant was evaluated at two different liquid flowrates that produced superficial velocities of 0.068 cm/sec and 1.5 cm/sec. The experimental K_La were 0.0013 min^{-1} at the low flowrate and 0.0061 min^{-1}, or 4.7 times higher, at the high flowrate. This indicated that oxygen transfer from the hollow fiber membranes was strongly influenced by liquid film mass transfer resistance.

The design of the bioreactor prototype will be used to evaluate bioreactor features and their effects on biological treatment of spacecraft wastewater, including the use of air versus pure oxygen, operation in gas flow through or dead end mode, low oxygen operation, and temporal variation of gas pressure (on/off operation; pressure fluctuations) to evaluate performance.

6. CONCLUSIONS AND RECOMMENDATIONS

The development of hollow fiber membrane bioreactor systems for spacecraft water recovery is in its infancy, and the Hollow Fiber Membrane Biofilm Redox Control Reactor, with its dual oxygen and hydrogen feed, appears to be highly promising. Experimental evaluations should be conducted to provide performance data upon which to develop process designs for flight testing and to advance to Technology Readiness Level 3. Application of hollow fiber redox control bioreactors requires extensive experimental evaluations on the unique composition of spacecraft wastewaters to develop a confident basis for system design. Ground based testing should be conducted first on simulated spacecraft wastewater containing all three spacecraft wastewater sources, followed by the use of real urine with simulated hygiene and condensate water.

Several system configurations of hollow fiber bioreactor should be evaluated in parallel to delineate the effects of multiple issues of reactor design and operation on the transformations of nitrogen and organic carbon, including: hollow fiber array design, superficial liquid velocity, nitrite recycle from partial nitrification, and oxygenation and hydrogenation regimes. For each reactor configuration, influent, intermediate, and effluent monitoring should be conducted to delineate process performance and allow changes in operation to be monitored for their effect on treatment efficacy. Mass balance analysis and stoichiometric relationships should be applied, and net biomass and process residuals should be quantified. In addition, pH control strategies and fouling and flux decline of hollow fiber membranes should be evaluated.

The hollow fiber membrane biofilm redox control reactor configurations that are recommended for parallel evaluation on the composite spacecraft wastestream are:

1. aeration, phasing into limited oxygen operation for simultaneous nitrification/denitrification, nitrite production, or nitrite reduction/ammonia oxidation
2. predenitrification/aeration phasing into predenitrification/limited oxygen operation,
3. oxygen/hydrogen with varied gas application regimes
4. pulsed oxygenation for 50% ammonia nitrification to nitrite, followed by ammonia oxidation/nitrite reduction

Follow-up experiments could consist of coupling a hydrogenation reactor to the end of the predenitrification/limited oxygen reactor to achieve complete nitrogen removal. Cell retention using microfiltration or ultrafiltration should then be incorporated into the experimental evaluations. Finally, effluent water quality must be evaluated for its suitability as a feedwater to reverse osmosis.

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