

Silver Foam: A Novel Approach for Long-Term Passive Dosing of Biocide in Spacecraft Potable Water Systems – Update 2025

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A spacecraft water disinfection system, suitable for extended length space exploration, should prevent or control the growth of microbes, prevent or limit biofilm formation, and prevent microbiologically influenced corrosion. In addition, the system should have minimal maintenance requirements, be chemically compatible with all materials in contact with the water, be safe for human consumption, and be suitable to be shared across international spacecraft platforms and mission architectures. Silver ions are a proven broad-spectrum potable water biocide under investigation for future exploration missions. Control-release technology is an attractive option for developing a high-reliability passive silver dosing device. This paper describes the continued development of a nanoparticle/polyurethane (NP/PU) composite foam for the controlled release of silver ions and is intended to build upon the 2024 International Conference on Environmental Systems (ICES) paper number 33. This paper provides the updated performance results for the product variability testing, microbial check valve (MCV) testing, and advancements made in the synthesis of the silver chloride (AgCl) NP/PU composite foams, referred to as AgFoams. The ultimate goal of the project is to develop a stable and reliable passive dosing silver ion release device for use in future spacecraft potable water systems.

Acronyms and Nomenclature

Ag^+	=	silver ion
$AgCl$	=	silver chloride
ARC	=	Ames Research Center
ICP	=	inductively coupled plasma
ICES	=	International Conference on Environmental Systems
ISE	=	ion selective electrode
ISS	=	International Space Station
JSC	=	Johnson Space Center
NASA	=	National Aeronautics and Space Administration
NP	=	nanoparticle

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<i>ppb</i>	=	parts per billion
<i>ppm</i>	=	parts per million
<i>PU</i>	=	polyurethane
<i>PWD</i>	=	Potable Water Dispenser
<i>MCV</i>	=	Microbial Check Valve
<i>SEM</i>	=	scanning electron microscope
<i>SWEG</i>	=	Spacecraft Water Exposure Guidelines
<i>TOC</i>	=	total organic carbon
<i>WHO</i>	=	World Health Organization
<i>WPA</i>	=	Water Processor Assembly

I. Introduction

SILVER biocide is being investigated by NASA for future mission architectures, as the replacement for the current iodine water disinfectant that has been used within the International Space Station (ISS) Water Processor Assembly (WPA). Several key benefits make silver ion (Ag^+), the biocidal component in silver biocide, an attractive choice for future spacecraft water recovery systems. Ag^+ is an effective disinfectant at levels that are safe for human consumption (below 400 ppb); unlike iodine, there is no need to remove silver ion biocide at the potable water dispenser (PWD). Ag^+ can also lower the life support risk and improve long term mission flexibility since it has the potential to be used across multiple spacecraft platforms.

Both active and passive silver ion dosing systems have been explored. The active dosing approaches include a study at Johnson Space Center (JSC) on the active release of Ag^+ using electrolysis, as well as a study at Ames Research Center (ARC) which utilized direct injection from a concentrate solution. As a compatible alternative to active dosing, the passive dosing approach proposed here relies on the concept of a silver compound nanocomposite polyurethane foam (referred to as Silver Foam or AgFoam). This paper follows six previous ICES papers, which further detail the concept creation, material synthesis decisions, Ag^+ release properties results, mathematical modeling on dosing behavior, risk mitigation plan, reduction of total organic carbon (TOC) release from AgFoam (one of the higher identified risks), and the 1-Year-Flow-Through test results.^{1,2,3,4,5,6}

The focus areas for the past year for the AgFoam as a doser have been in reducing product variability, mainly via updates in our in-house AgCl synthesis method and changes in how the particles are incorporated into the PU foam. A triplicate product variability test was completed using these new synthesis methods, the results will be detailed in this paper. In parallel, the AgFoam is also being investigated for use as a microbial barrier or microbial check valve (MCV) with a successful 3-month test completed in triplicate so far.

II. Background

Silver biocide and Ag^+ doser development has been an ongoing area of research for NASA in an effort to replace or reduce the need for iodinated water future spacecraft water processing systems, which are primarily modeled after the ISS-WPA. Most of the research thus far has been on developing an Ag^+ doser to replace the need for an iodinated resin and downstream removal of iodine, shown in Figure 1. There is also a second location in the WPA where iodinated resin is utilized as a sterile barrier, namely the MCV that connects the dirty side of the WPA from the clean side while allowing for recirculation of reject water.

As a passive dosing device, AgFoam is similar to the resin in that there are no power requirements and the material itself should provide a sterile zone. It is for this reason that an MCV test plan was made for AgFoam to determine whether the same device/material that has been designed as an Ag^+ Doser could serve as a replacement for the current MCV. This paper will discuss the AgFoam test results for both applications, doser and MCV, which each have unique requirements.

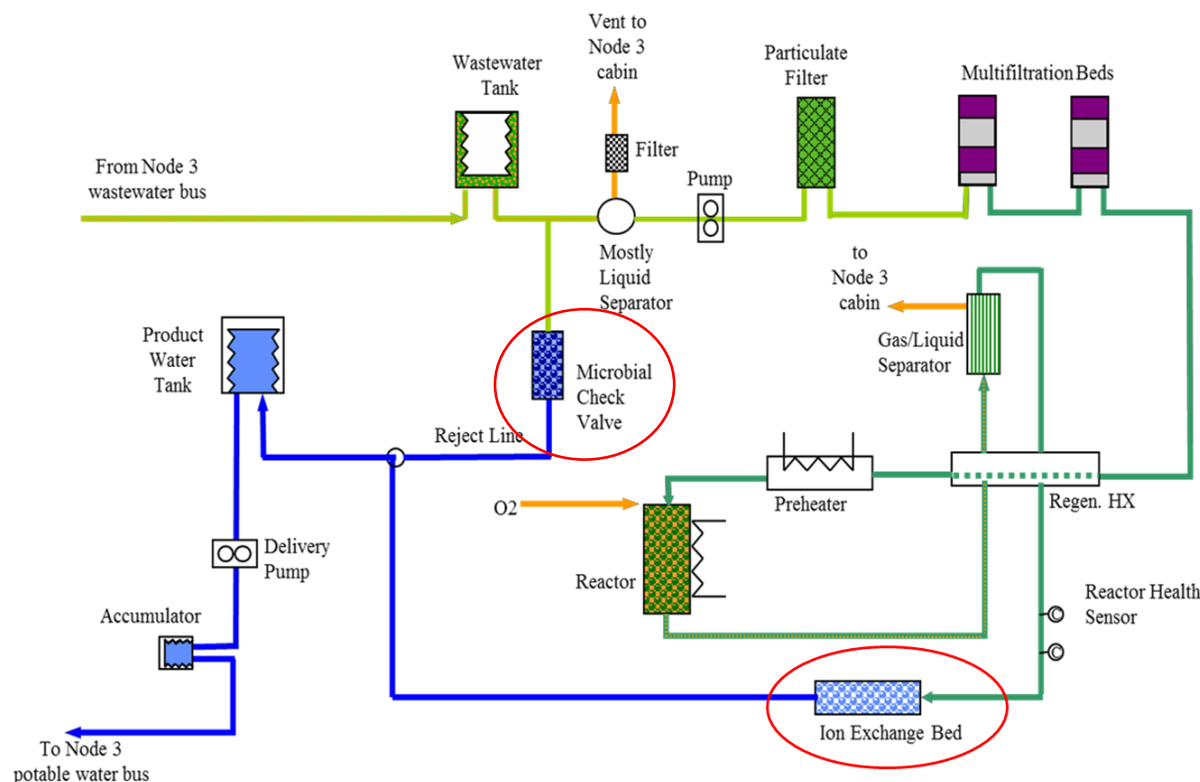


Figure 1. Illustration of ISS WPA Process. Currently, a biocide doser in the form of an iodinated resin bed is used in two locations of the WPA: the Microbial Check Valve and the Ion Exchange Bed, circled above in red. The Ion Exchange Bed also acts as a final scrubber for WPA that removes the remaining byproducts from the catalytic reactor in addition to dosing iodine.⁷

A. Ag^+ Doser Design and Requirements:

For the Ag^+ dosing application, passive-dosing Ag^+ technology is of particular interest for use in spacecraft potable water systems. Major advantages of passive-dosing technologies are their inherent high reliability and their relative ease of adaptation into the current water recovery systems. To develop such technology, several requirements, standards, and guidelines are being considered. Primarily, for any Ag^+ dosing technology, the system must dose Ag^+ at a steady concentration that is both effective against microbial growth and safe for human consumption. The upper limit of the concentration range was chosen according to the Spacecraft Water Exposure Guidelines (SWEG) for 1000-day missions.⁸ For these reasons, combined with reviewing studies on the effective biocidal Ag^+ concentration, a target range of 200 to 400 parts per billion (ppb) of Ag^+ has been selected.^{9,10,11,12}

Contact time requirements, determined by system flowrates, must also be considered, because of their direct effect on the Ag^+ release rate. The current WPA system on the ISS has an average flowrate of 0.10 to 0.15 L/min, which is the range that will be targeted. The target life span for a dosing device is a minimum of one year without replacement; during that period, the system should maintain dosing capabilities within the accepted range. In addition, the device must retain enough structural integrity to avoid any problems caused by mechanical breakdown, in the dosing system or water system components downstream, such as changes in Ag^+ release rates, clogging or pressure drops, or contaminants that could affect the potability of the product water.

AgFoam was developed to meet the above requirements of an Ag^+ passive-dosing device. The AgFoam is comprised of two key components. The first component is a silver compound in a nanoparticle (NP) form; it is the source of the silver ions with a very large surface area. The second component is the foam material. It is a microporous structure that provides a stable, high surface area and keeps the silver compound nanoparticles embedded and immobilized. As water flows through the foam structure, Ag^+ is released to the bulk solution via dissolution of the silver compound nanoparticles. To ensure a near constant release of Ag^+ over the useful life of the device, several design parameters are considered. The first is governed by the Noyes-Whitney Equation (Eq.1), which relates the

compound particle size and solubility to its dissolution rate. The second is the mass loading and distribution of silver particles within the foam structure. These constraints determine the total available surface area, consumable mass, sizing, and ultimate lifetime of the biocide delivery system. By controlling these parameters as a function of flowrate, it is possible to control the dosing rate for the system.¹³

Scanning Electron Microscope (SEM) imaging of the AgFoam composite structure is depicted in Figure 2. Figure 3 shows the same image with a zoomed in graphical representation depicting particles embedded in the polyurethane matrix and an illustration of the Noyes-Whitney Equation as it relates to Ag⁺ dissolution from the AgFoam.

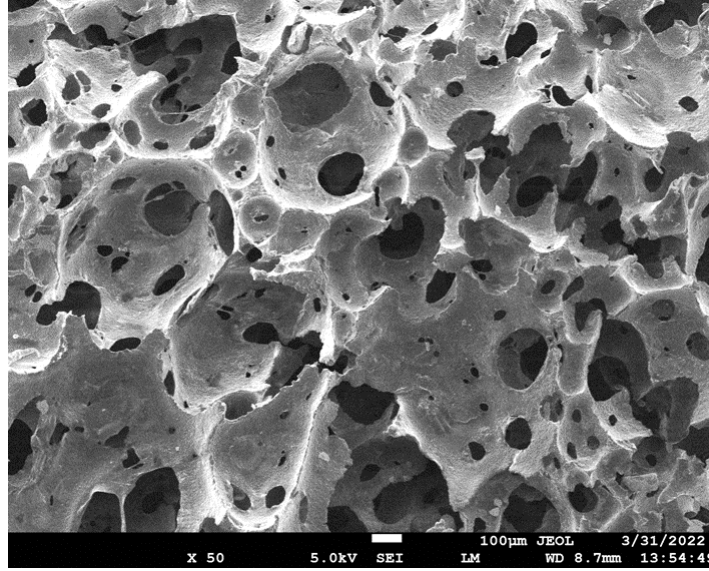


Figure 2. SEM Image of 20 wt% AgFoam Sample from the Accelerated 1-Year-Flow-Through Test.

Noyes-Whitney Equation¹³

$$\frac{dm}{dt} = A \frac{D}{d} (C_s - C_b) \quad (1)$$

Where

dm/dt = solute dissolution rate ($\text{kg} \cdot \text{s}^{-1}$)

m = mass of dissolved material (kg)

t = time (s)

A = surface area of the solute particles (m^2)

D = diffusion coefficient ($\text{m}^2 \cdot \text{s}^{-1}$) of the solute in the solvent

d = thickness of the concentration gradient (m)

C_s = particle surface (saturation) concentration (kg/L)

C_b = concentration in the bulk solvent/solution (kg/L)

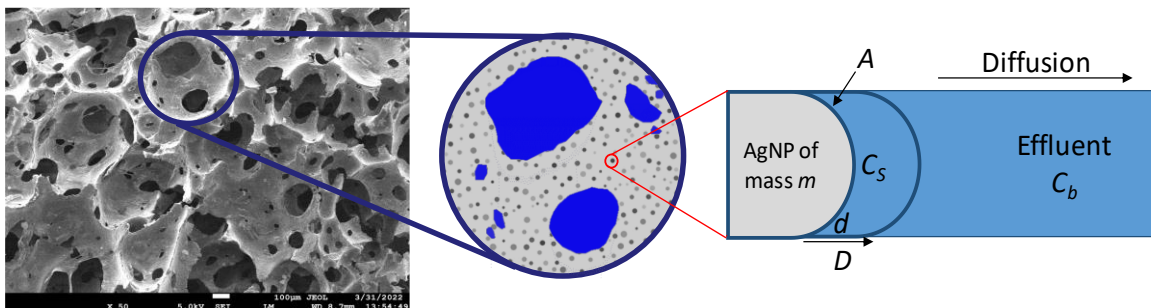


Figure 3. Illustration of the Noyes-Whitney equation in relation to the dosing function of the AgFoam. A cross section of the AgNP/PU composite foam is represented to the left, which shows the PU foam (light grey), the open foam pores (blue), and the AgNPs (black). To the right is an enlarged depiction of a AgNP (grey).

AgCl was selected for the silver compound based on its saturation limit, which is 2000 ppb or roughly 1500 ppb of Ag^+ . This limit is relatively close to the target dosing range of 200 – 400 ppb when compared to the solubility of other potential silver compounds.¹ The low solubility of AgCl helps to set the upper limit of the dosing concentration. It also ensures a more consistent Ag^+ release rate for a longer period since the AgCl cannot rapidly dissolve away during periods of system dormancy. The foam matrix selected was polyurethane because of its porosity, high surface area, and well-documented biocompatibility.^{14,15}

B. MCV Design and Requirements:

A Microbial Check Valve (MCV) for use in a spacecraft water system has separate and unique requirements when compared to Ag^+ doser requirements. It is a standalone application that must be compatible with the Ag^+ doser but does not necessarily have to utilize the same disinfection technology. The main purpose of an MCV is to prevent microbial cross-over from the dirty side to the clean side of a water processor, both during recirculation events and long periods of dormancy. The MCV has no dosing requirements and can utilize any silver-compatible disinfection technology to prevent cross-over. It must be compatible with the same system pressure, flowrate, temperature, and pH requirements as the Ag^+ doser while also not being negatively impacted by the higher impurity levels in the waste-water side of the system.

Previous MCV testing has been performed at JSC using UV as a sterilization method but was unable to prevent microbial cross-over. Proof-of-concept testing that mimicked the UV study's test conditions was initiated last year and published in ICES-2024-033.⁶ This paper will summarize the completed triplicate MCV test, with plans for longer and higher fidelity testing in the future. In all the MCV tests discussed in this paper the same AgFoam that was designed as a doser is used without any modifications.

III. Product Variability Testing

The overall goal of the Product Variability task is to prove that previous successes and results can be repeated and to identify and limit factors that result in variations in performance between batches of AgFoams. This has been a continued effort, with details of earlier work shared in our previous ICES papers. This paper will discuss breakthroughs made in in-house AgCINP synthesis and AgFoam synthesis techniques and will report on the results of the Product Variability Triplicate Test performed in early 2025.

A. AgCl Nanoparticle Synthesis Improvements

After limited success in working with our previous AgCl manufacturer to improve product quality and consistency, the team decided to return to in-house production for the time being. A brief literature search uncovered several methods of silver halide nanoparticle production, traditionally used in the photography industry, whose methods seemed promising for our own research goals.^{16,17} Several variations in technique were performed, ultimately landing on a new method for in-house AgCINP synthesis that has been the standard method of production for the Product Variability Test AgFoams discussed in this paper.

1. AgCl Nanoparticle Synthesis Method

Materials used for this in-house synthesis method include: AgNO_3 (99.9999% purity, CAS-No. 7761-88-8, Sigma Aldrich), KCl (99.99% purity, Fischer Scientific), powdered gelatin (Knox), and bromelain enzyme capsules (food grade, Doctor's Best). A 2.5 wt.% gelatin solution is made by dissolving 15 grams of powdered gelatin to 600 mL of milli-Q DI water and heating while stirring on a hot plate to 40°C. Once fully dissolved, half of the volume is transferred to a secondary beaker to be used for the KCl solution. A 1M solution of KCl is prepared, using sonication if necessary to ensure that the KCl is fully dissolved. In a separate beaker, a 300 mL 1M solution of AgNO_3 is prepared using milli-Q DI with no dissolved gelatin.

All three beakers are then transferred to a dark box and arranged as depicted in Figure 4. The gelatin solution is stirred to create a vortex (200 RPM, ramped up to 600 RPM as the reaction proceeds) and heated to 35°C. Both the AgNO_3 and KCl solutions are pumped at equivalent flowrates into the bulk solution, taking extra care to ensure that the streams do not come into contact before mixing in with the bulk solution. The reaction will result in an opaque white solution that resembles milk.

While the reaction is taking place, an enzyme solution containing 4 grams of bromelain enzyme dissolved in 80 mL of milli-Q DI water can be prepared, using sonication to fully disperse the enzyme. This solution is then spun down in a centrifuge to separate out any solids that remain, leaving a light brown liquid. Once all the reactants have been added to the bulk solution, the enzyme solution is added to the reaction beaker. The solution is stirred at 35°C for at least an hour to give the enzyme enough time to break down the gelatin matrix.

Next, the solution is divided evenly into 500 mL centrifuge bottles and centrifuged at 4000 rpm for 40 minutes. The mixture should separate, leaving a white cake of AgCl nanoparticles at the bottom with a slightly yellow liquid on top. The liquid is discarded, and the cake is washed once using milli-Q DI heated to 40°C before being centrifuged and decanted a final time. The finished cake is then dried in a freeze dryer.



Figure 4. Typical AgCl synthesis set-up. 1M solutions of AgNO_3 and KCl (left beakers) are pumped at equivalent flowrates into a bulk solution of 2.5 wt.% gelatin (right beaker).

2. Dissolution Test Results

As a method of checking particle quality, dissolution testing was performed on all in-house AgCl batches with the new gelatin method outlined above. In brief, this testing involves weighing out a small amount of particles, pulverizing them for consistency, and measuring the Ag^+ concentration of a beaker of water over time once the particles are introduced. This has been the most reliable method so far of determining the rate at which any given batch of AgCl will release Ag^+ into the water once it has been incorporated into an AgFoam. More details of this test method can be found in ICES paper 033, 2024.

Figure 5 shows the dissolution test results of AgCl using the new gelatin method. The trends in green show earlier batches where bromelain was not yet being used to digest the gelatin. This resulted in more batch variability and substantially longer washing processes, up to 7 washes versus the single wash when the enzyme is used. The trends in blue show various batches of AgCl produced using the same method but washed with enzyme as well. It's hypothesized that these enzyme-washed batches are more consistent due to the simplification of the washing process, which removes some potential for human error. The enzyme likely does a better job of removing the gelatin completely, leading to a higher yield of the smallest size-fraction of particles that were most likely lost in the first few washes in previous batches due to their light weight and inability to be easily separated from solution.

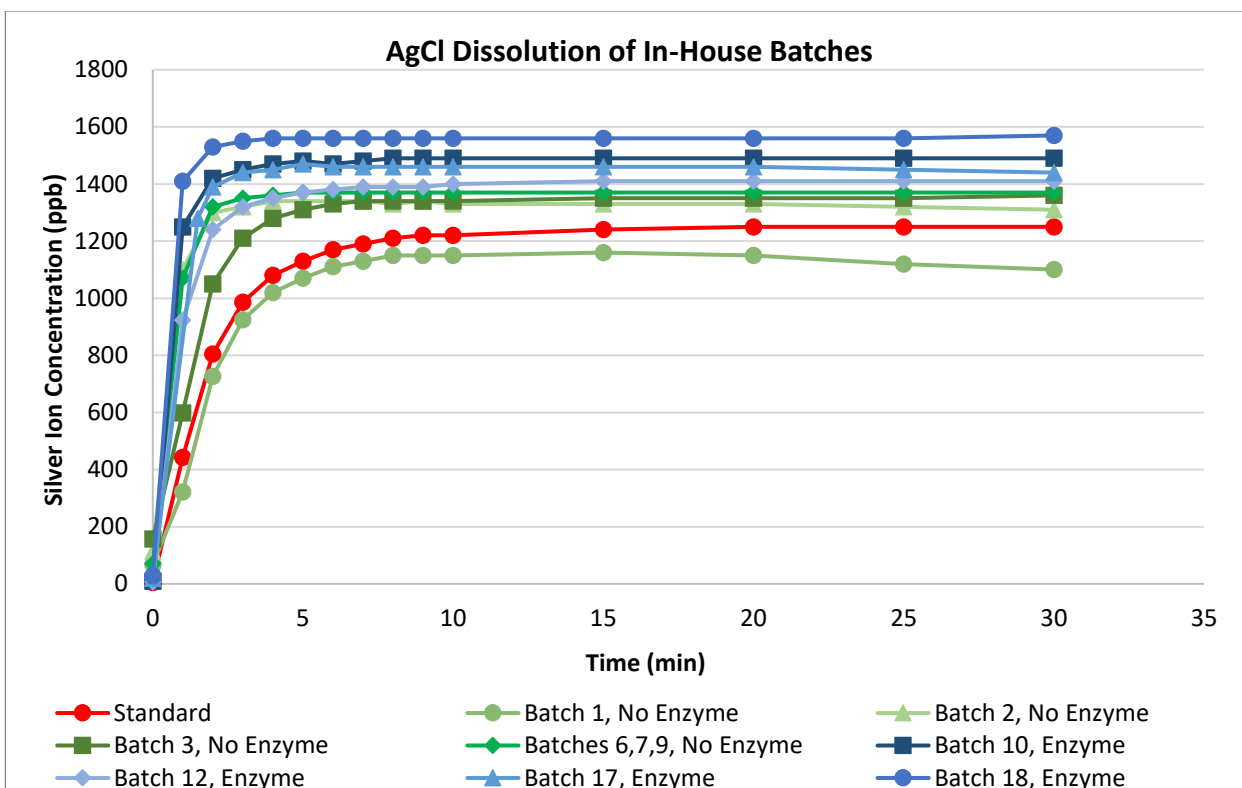


Figure 5. Dissolution test results of in-house AgCl nanoparticles. Green trends represent batches where no enzyme was used in the washing process, blue trends represent batches with enzyme used in washing. Red “standard” is a batch of particles known to perform well in AgFoam.

B. Initial Triplicate Test & Troubleshooting

Having determined a new and consistent method of AgCl production that outperforms our “standard” particles, no impacts on Ag^+ release rates were expected from AgFoams produced with these new particles. Unfortunately, once tested it was discovered that the particle dissolution rate was not the only factor responsible for producing quality foams. Through troubleshooting and developing new methods of incorporating the dried particles into the PU matrix, a successful method of synthesizing AgFoams with the in-house particles was determined and used to produce a new batch of 3 test foams with which to repeat the Triplicate Test. The details of this process are expanded upon below.

1. Product Variability Triplicate Test Method

AgFoams are first washed by recirculating DI water through the packaged AgFoams for 4 days. Water is recirculated in 24-hour increments through a 20L carboy, which contains AgCl to reduce silver depletion and is replaced with fresh water daily. Once washing is completed, the AgFoams are placed in the test stand shown in Figure 6. All components of the test stand are washed with soap and water and rinsed three times with DI prior to assembly. Each cartridge is connected to an independent tank and the tubing lengths and all fittings used in each cartridge system are identical to minimize differences in TOC leaching between the four systems. In the fourth slot, an empty cartridge is used to gather a control or blank TOC sample which can then be deducted from the AgFoam TOC effluent samples to show the amount of TOC being contributed by the PU foam alone. An upper limit of 50 ppb of TOC released by the foam was the target. More discussion on this target and previous efforts to minimize leaching can be found in ICES-2022-097.⁴

DI water is pumped through each cartridge at a flow rate of 0.1 L/min using independent peristaltic pumps. Effluent samples are taken every minute for the first 10 minutes, then once every hour. A separate effluent sample was taken each day for TOC analysis, at varying times into each test run to see if there is an impact on the TOC concentration due to down-time. This test is run in 8-hour periods, with varying amounts of down-time in between runs due to

weekends and holidays. The full test was designed to run for 20 days, 8 hours per day, for a total of 960 L of water treated. This is more than the approximate amount of potable water required for 1 crew member for 1 year (916 L). It is assumed that this test duration, combined with previously gathered 4-Crew-1-Year test data that was published in ICES-2024-33, is sufficient to determine the variability between different AgFoams.

For Ag⁺ analysis, a silver ISE (Orion Silver/Sulfide Electrode, Cat. No. 9616BNWP) was used for all samples with separate samples pulled weekly to cross-check with ICP analysis. TOC analysis was performed on a Shimadzu TOC-L CSH analyzer.



Figure 6. Test stand for Product Variability testing – Triplicate Flow-Through Test. From left to right, the cartridges are aligned: AgFoam 1, AgFoam 2, AgFoam 3, Control (no foam).

2. Initial Test Results

As stated briefly above, the AgFoams in the initial Triplicate Test did not perform as expected and therefore the test was terminated early. Figure 7 shows the results of this first test. Each AgFoam in this test fell below the target 200 ppb of Ag⁺ release, with release rates on the third day of testing leveling out to be below 50 ppb for all three AgFoams tested.

These results were surprising, given the high release rates seen in the particle dissolution tested, and led the team to troubleshoot other factors that may be causing this low release which is vastly less than what was seen from our previous AgFoam.

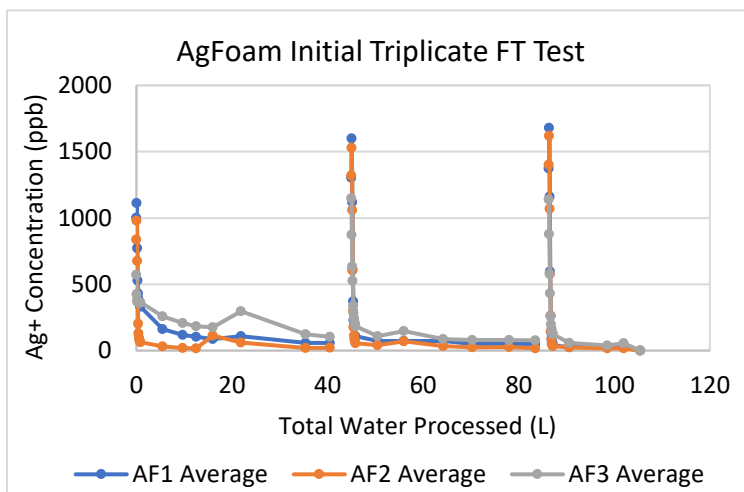


Figure 7. Silver ion release results for the initial Triplicate Flow-through test for the Product Variability task.

3. AgCl Moisture Level Test & Results

After looking back at potential differences between our first successful foam, the one used in the 4-Crew-1-Year-Flow-through Test that was completed in the year prior, the biggest potential difference noted was the moisture level within the AgCINPs themselves. The successful foam was synthesized using AgCINPs procured through the vendor Nanoshel. This was before changes in product quality led us to revisit in-house AgCINP synthesis. Unfortunately, the moisture level in this batch of particles was never quantified and was only ever noted as an observation. To determine if this could be a factor in increasing release rates, new AgFoams were synthesized with the following amounts of water added to the AgCINPs: 1g, 3g, 5g, and undried AgCINP cake (~3.5g H₂O).

For all but the undried particles, milli-Q DI water was weighed out and freeze dried AgCINPs were slowly added in while mixing by hand to achieve the most homogeneous mixture possible. The undried AgCINPs were taken directly from the cake of particles that forms at the bottom of the centrifuge tube after washing. Excess water was blotted off and a sample of the cake was reserved and weighed both wet and dry to determine the approximate amount of water in the cake AgCl used which was approximately 3.5g. Figure 8 shows the results of this test. As the amount of water incorporated the AgCINPs increased, the Ag⁺ amounts released by the AgFoams with those particles also increased. The particles with 5g of water added in had the highest release, in range of the release rates from the 4-Crew-1-Year AgFoam. It's also worth noting that, anecdotally, these particles appeared to be the most similar in consistency to the particles used in that successful foam based on images and observations.

With the results from this test, a new method of adding 5g of DI H₂O was adopted as the new technique for AgFoam synthesis and used to produce 3 new AgFoams for use in the Product Variability Triplicate Test. It's hypothesized that the thin film of water surrounding the AgCINPs aids in preventing a film of PU from forming on the particle surface. It may also cause the AgCINPs to become more dispersed in the hydrophilic zones of the PU structure rather than the hydrophobic zones. The AgFoams are also likely impacted by small changes in how well the particles are mixed into the polyol phase prior to foaming, since over-mixing would shear away the protective water layer while under-mixing would result in non-homogeneous dispersion of the particles in the foam matrix. The impacts are currently being explored but test results are not yet available.

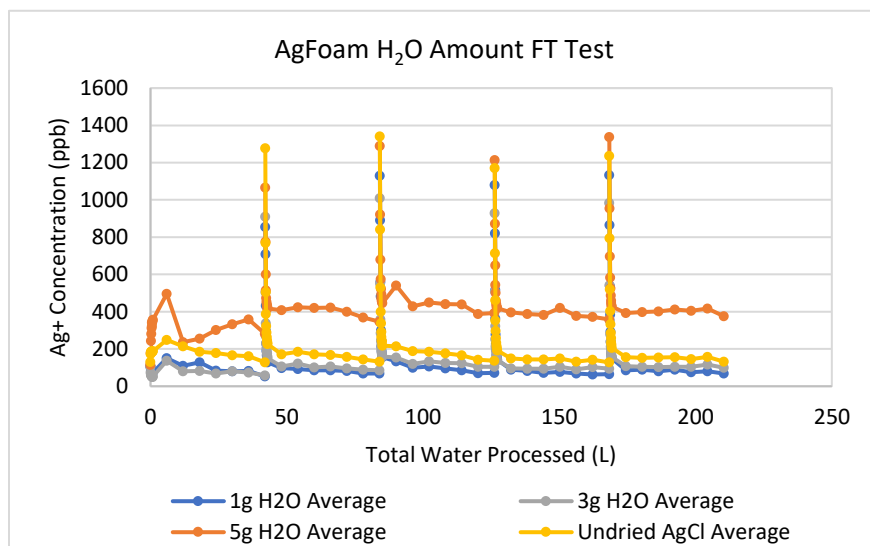


Figure 8. Flow-through Ag⁺ concentrations from AgFoams with various amounts of water added to the AgCINPs prior to synthesis.

C. Triplicate Test

The Product Variability Triplicate Test was completed using 3 new AgFoams synthesized using AgCINPs wetted with 5g of water and incorporated as described in Section III.B.3. The same test method outlined in Section III.B.1 was followed for this test. Testing ran for a total of 22 runs over the span of a month, with all but one run lasting for 8 hours. One test run was cut short due to an unexpected lab closure after start-up that resulted in a short, 2-hour run. In total, 1,016L of water was processed through each AgFoam and the empty control cartridge. The full results for the Ag⁺ release values are shown in Figure 9. Effluent TOC values are shown below in Figure 10 as corrected for TOC contributed by the environment.

In general, the 3 AgFoams followed the same trend throughout the full test. AgFoam 1 (AF1) had the highest Ag⁺ release rates, with AgFoam 2 (AF2) having the second highest and AgFoam 3 (AF3) having the lowest release rates. The difference in Ag⁺ effluent concentrations produced by each foam at any given time ranged from about 10-40 ppb, however the order of highest to lowest remained consistent throughout the test. Both AF2 and AF3 did drop below the 200 ppb target. AF2 dropped to a minimum of 189 ppb for a brief period, while AF3 consistently dosed at or just below 200 ppb, with a low point of 179 ppb. Since this was a test to determine the variability between

AgFoams, and since neither foam dropped well below the target, the testing was continued. It is possible that slight changes in how the particles were incorporated into the polyol are causing the release rates of the foams to decrease in the order that they were produced due to human error. Variability caused by channeling is unlikely and not considered problematic due to tight packaging of the PU foam and previous testing that utilized variable form factor with no change to Ag⁺ release rates.

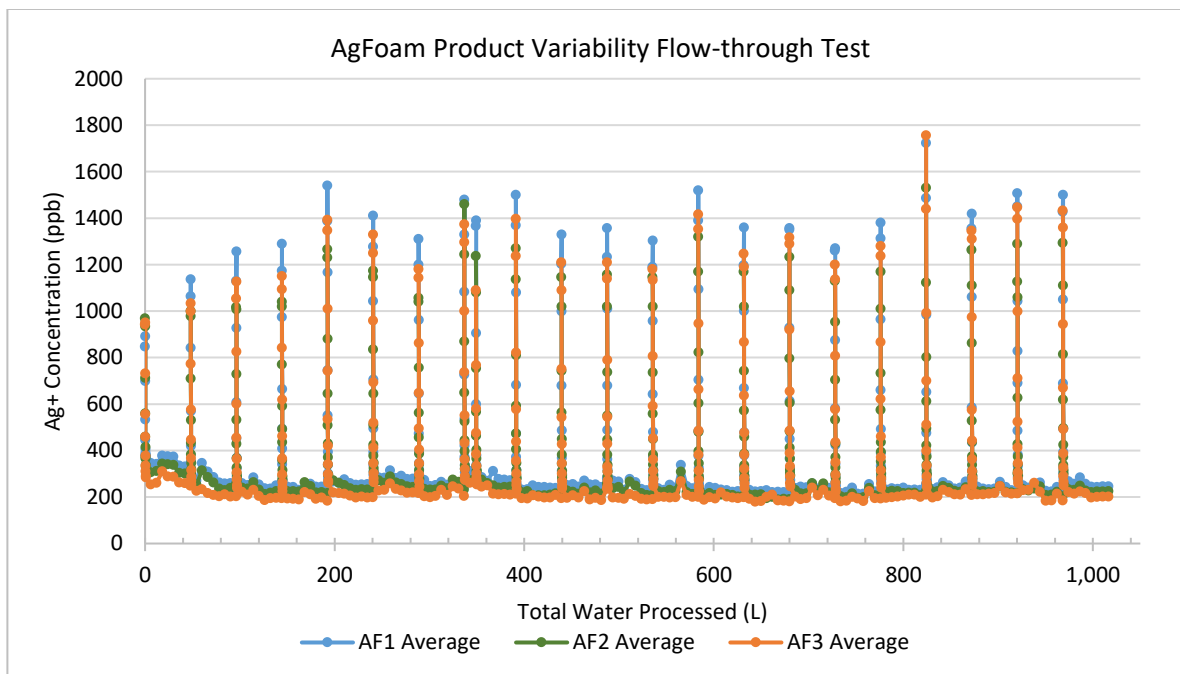


Figure 9. Ag⁺ release results from Flow-through testing for the Product Variability Triplicate Test.

TOC effluent samples were taken daily, with sampling times intentionally varied to gather data at different points into each run as the amount of soaking or down-time is expected to change the TOC concentration within the cartridge. Figure 10 provides a summary of the TOC data for each foam with the baseline system TOC subtracted to show the TOC contributed by the foam itself. Throughout the test, all 3 AgFoams performed well in terms of TOC leaching, with AF1 and AF2 consistently leaching less than the 50 ppb threshold. AF3 briefly leached above the threshold, this may have been due to increased wear in the tubing of that setup that eventually caused a split in a small section of tubing. TOC leachate values dropped back down once this was repaired.

The one exception was the sample taken on January 21st, omitted from the plot to avoid skewing the scale. This sample was taken within the first hour of operation after the AgFoams had been soaking over a long weekend, resulting in TOC leachate amounts of 591, 532, and 597 in AgFoams 1-3, respectively. A further study with samples taken in a similar fashion to the Ag⁺ samples taken during flow-through tests should be conducted to better understand the relationship between TOC leachate concentration and cartridge soak times.

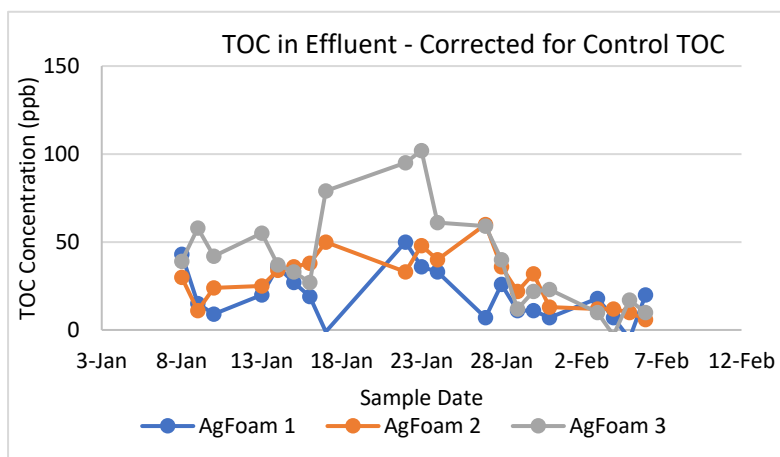


Figure 10. TOC effluent amounts from AgFoams 1-3, with Control cartridge effluent values subtracted out to show the amount of TOC released by the foams rather than the environment.

IV. MCV Testing

As shown in Figure 1, there are areas of water systems where microbial control is necessary such as the reject line of the water processing assembly (WPA). The reject line allows processed water that does not meet specifications to be directed back through the WPA. This means that the dirty and clean lines are hydraulically connected which requires the use of a microbial check valve (MCV) to ensure that bacteria cannot migrate across the physical check valve from the wastewater side to the product water side. The MCV is coupled with a mechanical check valve to prevent back flow of contamination from the wastewater bus to the potable water bus as depicted in the water processing assembly in Figure 1.

The current MCV system utilizes iodine biocide that is released from an ion-exchange material, but silver is being considered as the alternative biocide and would require a chemically compatible MCV. Feasibility testing of the AgFoam as an MCV began in late 2023 as a triplicate 3-month duration test. The first two replicates of this 3-month test were reported in ICES-2024-033, along with a detailed test method and summary of results. The third test concluded in mid-2024. A summary of the test method and triplicate results will be provided here to conclude this portion of the MCV test effort, however a review of the more detailed report from last year is suggested to those interested in better understanding the task.

A. Test Method

The experimental set up for the microbial check valve experiment can be seen in Figure 11. The setup includes two 10L carboys with spigots, one representing the dirty water (the WPA influent that contains microbes), and another representing the clean water (the WPA product water without microbes); for simplicity, microbial ersatz water was used to fill both carboys, but only the dirty side was inoculated with microbes. A cartridge containing AgFoam was placed in the middle of the two carboys and connected to them using Tygon tubing. There are two sample ports on the connecting tubing at each side of the AgFoam cartridge; there are also sample ports built into the lid of each carboy. There is a clamp on each side of the AgFoam cartridge, which was closed during sampling to prevent the forced flow of water through the foam. There is also an air vent with a 0.2 micron filter on the lid of each carboy to release pressure buildup while avoiding contamination from the room air. A control was also run in each test, which consisted of an identical setup with a Control Foam (PU foam without any AgCl) used in place of the AgFoam. To distinguish between the AgFoam and Control Foam set-up, sample numbers for the AgFoam test stand began with an “A” (A1-A4) while sample numbers for the Control test stand began with a “C” (C1-C4).

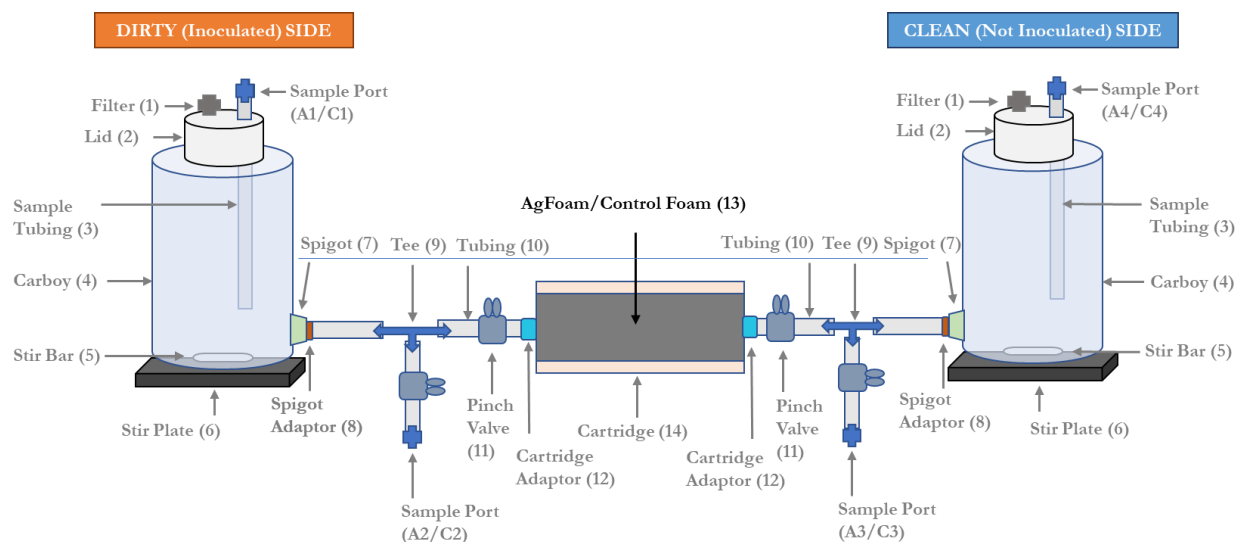


Figure 11. Microbial Check Valve Test Setup.

Ralstonia insidiosa, accession number 171870003-1, isolated from ISS WPA wastewater was used for the proof-of-concept testing as it showed the best growth in the test media of the common ISS WPA strains tested. A simplified WPA microbial ersatz was used for this testing; its composition is detailed in our 2024 ICES paper, number 33.

All components were sterilized before the start of each test using the most appropriate method for the material. Pre-test sterilization checks were performed on the media, bacterial culture, and the sterilized silver and control foams.

A brief Ag^+ flow-through test was also performed on the AgFoam to ensure it is still biocidally active. If any check in this process failed, the test stand was broken down and re-sterilized from the beginning.

Samples were pulled at the start of the test (T0) and at timepoints throughout the full 90 days with at least one timepoint per week from sample points A1-A4 and C1-C4. Samples from the bulk carboy solutions (A1, A4, C1, C4) were pulled for both microbial checks and chemistry checks, whereas samples from the tubing (A2, A3, C2, C3) only received microbial checks. All chemistry samples were split into two vials, one for total organic carbon (TOC) testing and one for inductively coupled plasma (ICP) testing.

B. Triplicate Results

The data from all three MCV 3-month tests is shown at the end of this section. Due to the repeatable nature of these results, a summary for each individual test is not provided here but a detailed summary for tests 1 and 2 can be seen in ICES-2024-033.

In all three test runs, the AgFoam successfully prevented bacterial crossover from the clean side (A1) to the dirty side (A4), as evident in the microbial test results reported in Figure 12 in the appendix. Although cell counts sometimes fluctuated on the dirty side for both the silver and control systems, in each test there remained a substantial population of bacteria on the dirty side of both systems indicating a sufficient challenge to the foams. In all three tests, the control system did experience microbial crossover from the dirty side (C1) to the clean side (C4), further illustrating the presence of a microbial challenge in each test. Crossover timepoints on the control side did vary for each test, with crossover occurring at day 21 (C3) and 28 (C4) in the first test, day 2 (C3 and C4) during the second test, and day 35 (C3 and C4) in the third test.

Figure 13 in the appendix shows the TOC and ICP data of Ni, S, Zn, K, Ca, P, Mg, Fe, and Ag are also plotted together with microbial counts (CFU/mL) for the A1 and A4 setup, and the C1 and C4 carboys at different time points. In all three tests the chemistry trends remain relatively consistent, with TOC and P levels decreasing as cell counts on the inoculated side increase or as soon as crossover occurs on the clean side. It is a consistent correlation with the simplified microbe ersatz, where P is the limiting nutrient while other elements are in large excess. Thus, P and TOC concentrations decrease while microbes grow. The TOC concentration continues to decrease during stationary phase where it is used as an energy source for the microbes. In A4, there is little change to the nutrient level, as no growth occurred in any of the three tests. The silver levels in A1 and A4 remain low, in the single ppb range, indicating that there is little to no dissolution of Ag^+ to the bulk solutions.

V. Conclusion

Product variability has been the focus for the AgFoam this year. Increasing material consistency is vital for the AgFoam as a silver doser. Product variability may also impact MCV performance, although it is expected that this use case is less sensitive to changes in product quality due to the long contact times.

As a silver doser, the silver ion release rates are the most easily influenced success criteria. AgCl particle size, cleanliness, and preparation can all have significant impacts on the final AgFoam quality. Dissolution testing has been shown to be a reliable way to test particle quality, however it fails to capture other AgFoam synthesis factors that deal with the way that these particles are incorporated into the polyurethane foam itself. This year, multiple troubleshooting exercises were performed to home in on the best method for replicating the success of previous AgFoams using in-house AgCINPs to eliminate vendor variability. Ultimately, adding a moisture barrier around the AgCINPs by simply wetting them prior to mixing them into the polyol phase led to a dramatic increase in performance over AgFoams made with similar, but dry, AgCINPs. A Product Variability Triplicate Test was performed with 3 new AgFoams using the new synthesis method, which showed limited variability in their Ag^+ release rates (10-40 ppb differences), treating a total of 1,016L over the course of 1 month. TOC leachate levels remained relatively low throughout the test, with an average leachate of 46, 52, and 66 ppb for AgFoams 1-3, respectively. Further TOC tests are necessary to understand how soak time influences the TOC leachate rates.

As an MCV, the 90-day triplicate proof-of-concept testing has been successfully completed. The AgFoam has proved to be capable of providing a sterile barrier between an inoculated tank and a clean tank in the simplified microbial WPA ersatz for 3 separate 90-day tests using an inoculation concentration of 1×10^6 of *R. Insidiosus*. With the success of this initial testing, a 1-Year duration static test that will incorporate a mixed culture of the most commonly seen bacteria and fungi on ISS is currently being prepared in triplicate.

Appendix

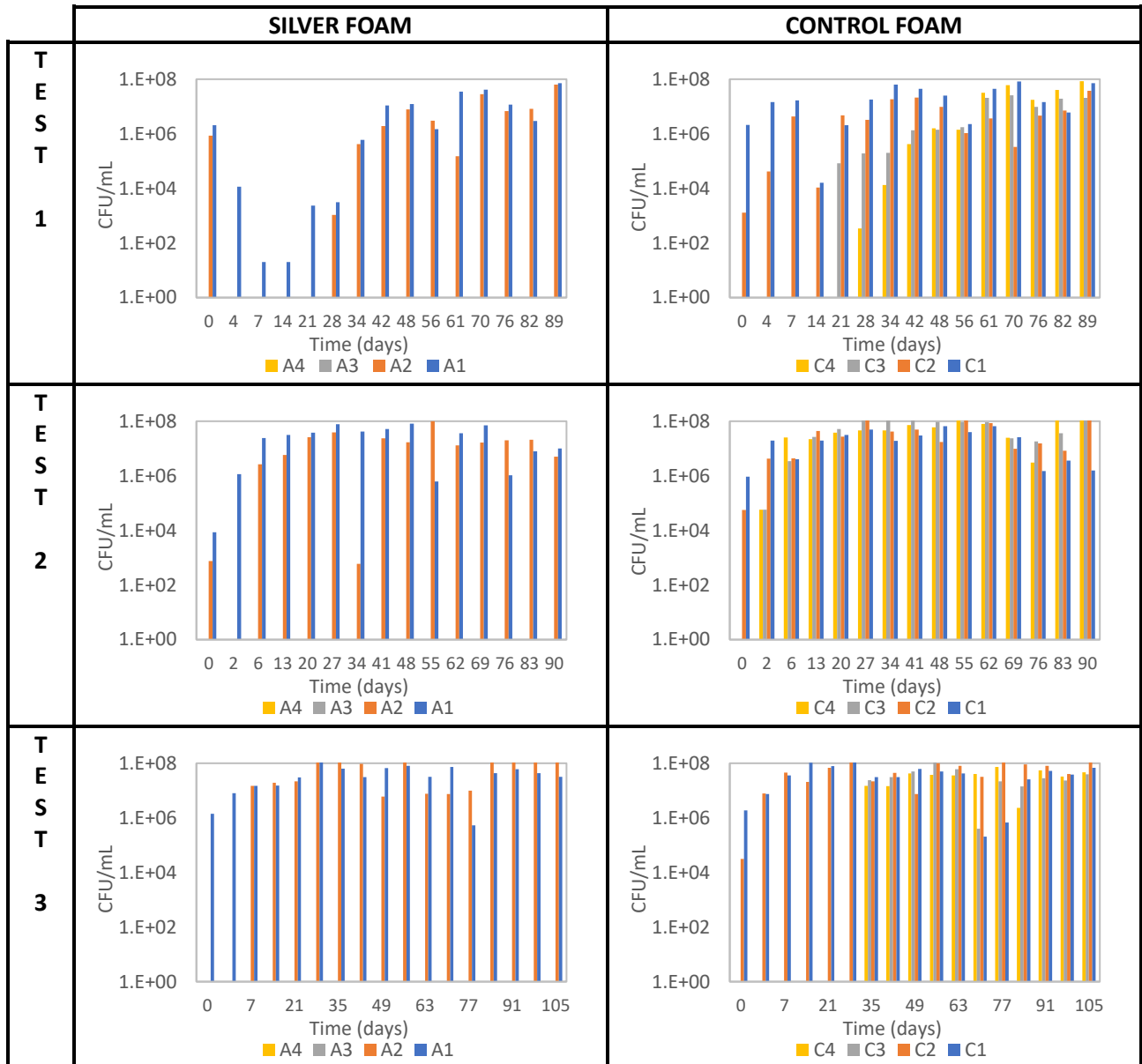


Figure 12. Microbial results from triplicate Single-strain 90-Day Tests

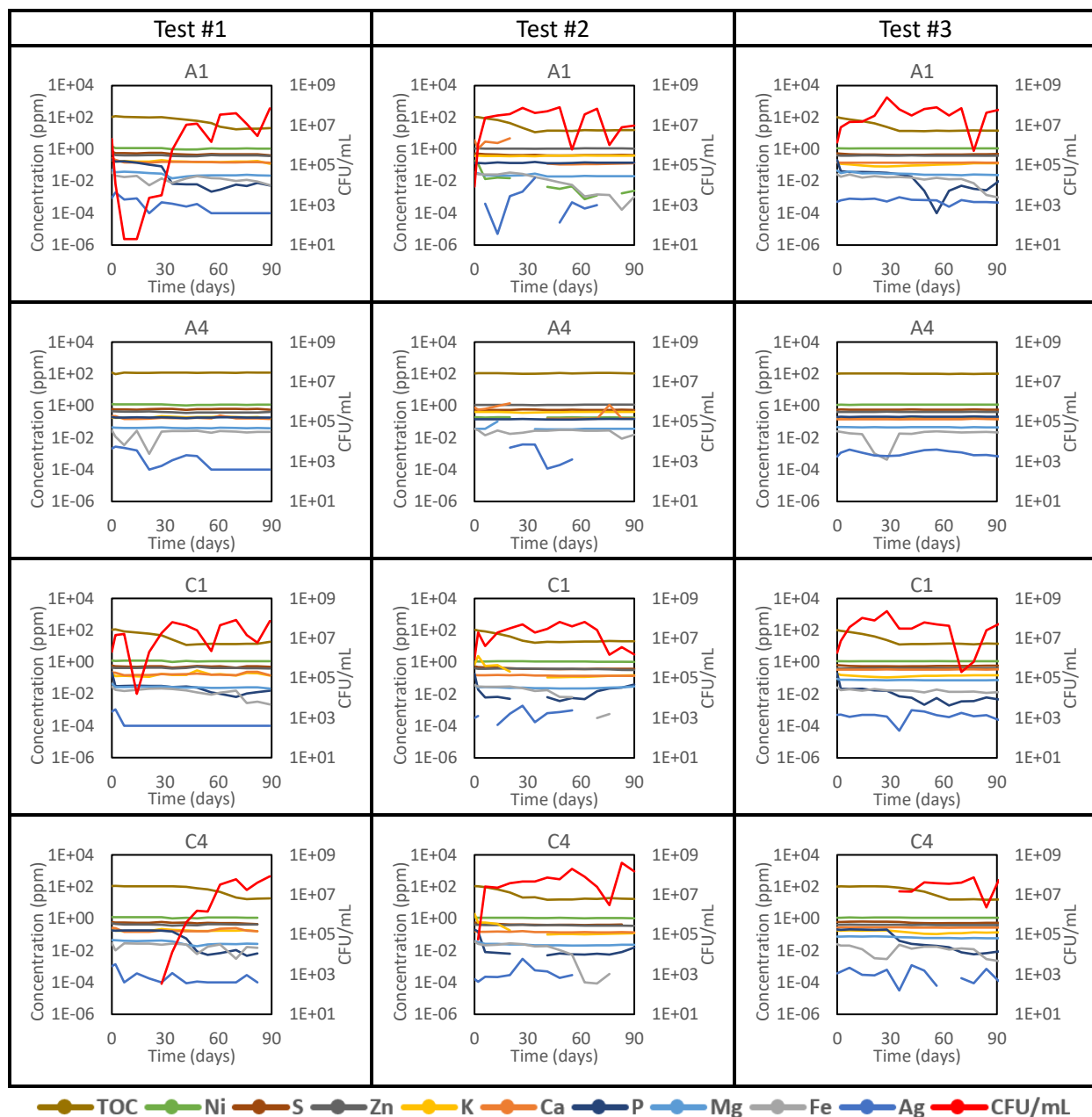


Figure 13. Microbial counts, TOC, and ICP water analysis data of Silver Foam carboys A1 (dirty) and A4 (clean) and Control carboys C1 (dirty) and C4 (clean) for triplicate 90-day tests.

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References

- ¹ Irwin, T., Li, W., Buhrow, J., Calle, L., and Callahan, M. “Silver Foam as Long-Term Passive Biocide for Potable Water Systems,” *49th International Conference on Environmental Systems*, ICES-2019-272, 7-11 July 2019, Boston, Massachusetts.
- ² Irwin, T., Li, W., Diaz, A., Calle, L., and Callahan, M. “Silver Foam: A Novel Approach for Long-Term Passive Dosing of Biocide in Spacecraft Potable Water Systems – Update 2020,” *International Conference on Environmental Systems*, ICES-2020-128.
- ³ Irwin, T., Li, W., Diaz, A., Calle, L., and Callahan, M. “Silver Foam: A Novel Approach for Long-Term Passive Dosing of Biocide in Spacecraft Potable Water Systems – Update 2021,” *50th International Conference on Environmental Systems*, ICES-2021-116, Virtual.
- ⁴ Irwin, T., Li, W., Diaz, A., Calle, L., and Callahan, M. “Silver Foam: A Novel Approach for Long-Term Passive Dosing of Biocide in Spacecraft Potable Water Systems – Update 2022,” *51st International Conference on Environmental Systems*, ICES-2022-097, 10-14 July 2022, St. Paul, Minnesota.
- ⁵ Irwin, T., Diaz, A., Gooden, J., Hummerick, M., Li, W., Azim, N., Essumang, D., and Callahan, M. “Silver Foam: A Novel Approach for Long-Term Passive Dosing of Biocide in Spacecraft Potable Water Systems – Update 2023,” *52nd International Conference on Environmental Systems*, ICES-2023-251, 16-20 July 2023, Calgary, Canada.
- ⁶ Irwin, T., Diaz, A., Gooden, J., Hummerick, M., Li, W., Azim, N., Essumang, D., and Callahan, M. “Silver Foam: A Novel Approach for Long-Term Passive Dosing of Biocide in Spacecraft Potable Water Systems – Update 2024,” *53rd International Conference on Environmental Systems*, ICES-2023-251, 21-25 July 2024, Louisville, Kentucky.
- ⁷ Williamson, J., Wilson, J., and Gleich, A. “Status of ISS Water Management and Recovery,” *51st International Conference on Environmental Systems*, ICES-2022-098, 10-14 July 2022, St. Paul, Minnesota.
- ⁸ Spacecraft Water Exposure Guidelines for Selected Contaminants, Vol 1., National Academies Press, Washington DC, 2004.
- ⁹ Ott, M., Almengor, A., and Harris, J., “FY19 Silver Disinfection,” NASA Johnson Space Center., Test Report 09302019.
- ¹⁰ Alternative drinking-water disinfectants: bromine, iodine and silver. Geneva: World Health Organization; 2018. Licence: CC BY-NC-SA 3.0 IGO.
- ¹¹ Adam, N., "Compatibility Study of Silver Biocide in Drinking Water with Candidate Metals for the Crew Exploration Vehicle Potable Water System," SAE Technical Paper 2009-01-2459, 2009.
- ¹² Nawaza, M., Hanb, M.Y., Kimc, Tschung-il, Manzoora, U., Amina M.T. “Silver disinfection of *Pseudomonas aeruginosa* and *E. coli* in rooftop harvested rainwater for potable purposes”, *Science of The Total Environment*, Volume 431, 1 August 2012, Pages 20–25.
- ¹³ Lu, J. X., and Murray, J., “Biochemistry, Dissolution and Solubility” *National Center for Biotechnology Information, U.S. National Library of Medicine* [online library], URL: <https://www.ncbi.nlm.nih.gov/books/NBK431100/> [cited 4 March 2024].
- ¹⁴ Marois Y, Guidoin R. Biocompatibility of Polyurethanes. In: Madame Curie Bioscience Database [Internet]. Austin (TX): Landes Bioscience; 2000-2013. URL: <https://www.ncbi.nlm.nih.gov/books/NBK6422/> [cited 4 March 2024].
- ¹⁵ Lamba, N., Woodhouse, K., Cooper, S.L., *Polyurethanes in Biomedical Applications*, CRC Press, New York, 1998.
- ¹⁶ E. Michalak, P. Nowak, A. Król-Gracz, and A. Dyonizy “Synthesis of Silver Halide Nanosols,” *International Congress on Advances in Applied Physics and Materials Science*, Vol. 121 (2012).
- ¹⁷ B. H. Crawford, “The preparation of ultra-fine grain photographic emulsions,” *J. Sci. Instrum.*, 31 333 (1954).