Preliminary Testing of Electrolytic Silver Ion Generation for Spacecraft Potable Water Systems

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Anodic dissolution of silver electrodes, or "silver electrolysis," is being investigated as a means of imparting biocidal silver into potable water on exploration spacecraft. This method of water disinfection is used commercially in hospitals and marine vessels, but the spacecraft application poses unique challenges such as low water conductivity, increased importance of electrode longevity, and an interface with other systems that may be sensitive to any particles released in the electrolysis process. Testing has shown that due to the low conductivity of the water, the electroactive area of the cell can be scaled linearly to achieve a target electrolysis current despite the general non-linearity of electrochemical systems. More importantly, however, the low conductivity dictates that a large electroactive area and minimal electrode gap be employed in the design. Furthermore, thermodynamic considerations suggest that a low applied voltage is necessary to avoid undesired electrode reactions that could negatively impact long-term performance. Preliminary testing of long-term electrode health suggests that anodic oxide formation may not present as significant a challenge as anticipated; however, further testing in a system closed to the atmosphere is required to rule out the influence of atmospheric CO_2 on the pH of the process water. Comparison of filtered and unfiltered silver concentration measurements indicates that release of particles larger than 0.2 μm was negligible in this preliminary test. A test article has been developed to facilitate further testing of silver electrolysis, which continues to be a candidate technology for spacecraft potable water disinfection.

Nomenclature

A = electrode area
d = electrode spacing (gap)
DI = deionized water
I = current
ICP-MS = inductively coupled plasma mass spectrometry
ISE = ion-selective electrode
IX = ion exchange
κ = solution conductivity
η = overvoltage (voltage in excess of null potential)
ppb = parts per billion
R_{pol} = polarization resistance
R_{soln} = solution resistance
SBIR = Small Business Innovation Research
WPA = Water Processor Assembly
XPS = X-ray Photoelectron Spectroscopy

I. Introduction

Ionic silver at concentrations between 200-400 ppb is being considered as a replacement for iodine as the residual biocide in future spacecraft potable water systems. Specifically, a technology is sought to replace the iodine-imparting resin in the product water segment of the water processor to be used on future long-duration missions. It is

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assumed that such a water processor would be very similar to the Water Processor Assembly (WPA) currently in use on the International Space Station. Thus, assumed conditions for silver introduction technology are taken from what is known about the WPA; namely that the product water is essentially deionized (DI) water and that the processing of the water occurs at a flowrate of approximately 100 mL/min. Replacing iodine with silver requires an in-line method to accurately and consistently introduce the silver into water under these conditions. Furthermore, the method should require no maintenance (including replacement of consumables) over a three-year mission. Silver electrolysis has the potential to meet these requirements: it is easily controlled and monitored via electrical parameters, the output concentration is proportional to the electrolysis current, and a single set of electrodes would contain enough silver to last many years. However, potentially-significant challenges must be overcome before a silver electrolysis reactor can be reliably integrated into a next-generation water purification system. These challenges include difficulty operating in low conductivity water, the potential to develop an oxide film on the anode that resists further electrolysis, and the possibility that the effluent water quality will be negatively impacted by the electrolysis process.

Electrochemical processes require ionic species in solution in order to allow current to flow between the electrodes, and electrolysis is typically performed in very conductive solutions. In contrast, the product water of the WPA is essentially deionized water,[*] whose very low conductivity of ~0.056 µS/cm is based solely on the self-ionization of water. Minerals (e.g. $CaCO_3$ and $MgCO_3$) could be added to the water upstream of the silver electrolysis reactor, but this would entail adding a consumable to the system. Furthermore, the impact of minerals in the water on other systems that use potable water, such as the spacesuit, the toilet, and the oxygen generator, would have to be fully assessed. Finally, mineralization generally increases pH, which may negatively impact the performance of the reactor itself (as discussed below). As such, water additives have been avoided during preliminary reactor development. Another technique for increasing conductivity is to place a conductive, water-permeable membrane in the inter-electrode gap. This approach was attempted by Reactive Innovations, LLC in their development of a silver electrolysis reactor under a Small Business Innovation Research (SBIR) grant; in this case it was deemed unsuccessful due to the membrane's tendency to promote the formation of dendritic silver bridges that shorted out the cell.[1] Aside from increasing conductivity, the only ways to reduce the reactor's solution resistance are to increase the electrode area and/or decrease the space between them. The appropriate electrode geometry and applied voltage are therefore critical in establishing the current that yields the desired silver concentration at the nominal water processor flowrate. However, because the reactor's overall resistance comprises a polarization resistance in addition to the solution resistance, the current-voltage relationship generally does not vary linearly with electrode geometry. Empirical data is necessary to inform proper reactor design.

Past studies of silver electrolysis for long-term water disinfection have attributed a degradation in system performance to anodic oxide formation. It was cited by Reactive Innovations as a major obstacle in their development of a membraneless reactor; specifically, they experienced degradation of cell performance due to increased resistance after 18 hours of operation. Other reports on silver electrolysis, though not focused on oxide formation, have cited the phenomenon as a possible explanation for sub-optimal performance.[2-4] One of these reports states that mechanically cleaning the electrode surfaces was necessary after only 50 minutes of operation, and the formation of unwanted byproducts was also evident in a low current efficiency of 50%.[4] In some systems, oxide formation has been combatted by periodically reversing the polarity of the electrodes, such that the anode becomes the cathode and vice versa. When a silver electrode switches from anode to cathode, the anodically-formed oxide film is cathodically reduced, cleaning the electrode. Polarity reversal was employed in the final Reactive Innovations SBIR design and is also used in some commercial silver generators.[5] However, implementation of polarity reversal was not entirely successful in the Reactive Innovations system and mechanical electrode polishing was still required.[1] Furthermore, polarity reversal does not address the underlying inefficiency (and thus silver dosing inaccuracy) that results from unwanted side reactions (e.g. oxide film formation). It is critical to understand the fundamental phenomenon of oxide film formation and identify operating conditions that minimize its occurrence.

Thermodynamic considerations suggest that a low applied voltage and a neutral or slightly acidic pH could mitigate or even completely avoid the oxide formation. In their paper describing the development of a potential-pH diagram (i.e. Pourbaix diagram) for silver, Delahay, et al., state that "electrolysis of solutions of silver ion Ag^+ under sufficiently low current density with a silver anode leads to anodic dissolution of silver as Ag^+ exclusively … without

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[*] After catalytic oxidation and gas separation, water is passed through an anion exchange resin followed by a mixed cation/anion exchange resin (collectively "IX resin") before iodine is imparted to the water. The IX resin theoretically removes all ionic contaminants, such that pure water of neutral pH and ultra-low conductivity results. Conductivity of the product water between the IX resin and iodine-imparting resin is not measured and thus not known empirically. Future testing will include a similar IX resin, and conductivity will be measured immediately downstream.

any risk of secondary reactions."[6] They also state that "electrolysis of solutions of silver ion Ag^+ under high current density, with a sufficiently high anodic potential, may lead to a deposition of black peroxides AgO and Ag_2O_3 on the anode." Furthermore, the electrode passivation under these conditions is incomplete and the electrode may continue to dissolve. Finally, the formation of a highly resistive, brown film of Ag_2O is thermodynamically favored during electrolysis of silver in alkaline solution.[6-7] Therefore, the present research seeks to identify a voltage and pH range over which oxide formation (particularly silver(I) oxide) is minimized.

Finally, it is imperative that a silver electrolysis reactor not only impart the correct concentration of silver ion, but that it also not have any negative impact on the process water. One such impact would be the release of silver particles resulting from uneven dissolution of the anode. The particles could clog filters or otherwise disturb processes in downstream systems that are sensitive to influent water quality (such as the spacesuit and the oxygen generator). Furthermore, the total content of silver in such a situation would be higher than desired because silver ions are generated at a fixed rate based on the current, while silver particles are generated at an unpredictable rate via the pseudo-mechanical mechanism described above. Silver particles have been observed in the effluent of silver electrolysis in past informal tests, both by visual inspection and by comparison of filtered and unfiltered inductively-coupled plasma mass spectrometry (ICP-MS). However, it is suspected that an unnecessarily high current density was at least partially responsible for silver particle release in these experiments. Therefore, adjusting the operating conditions may effectively mitigate the problem.

The possible drawbacks to silver electrolysis discussed here can be mitigated by careful design and operation of the silver electrolysis reactor. In this paper, we describe preliminary testing and the design of a test article that will enable further investigation into the aforementioned problems and associated mitigation strategies. That testing included an electrode polarization test to determine the required electrode dimensions for operation in low-conductivity water and a long-duration electrolysis test to characterize the impact of oxide formation and assess the overall feasibility of electrolysis, including an assessment of the extent to which particles were released in the process.

II. Methods

The electrolytic cell comprised high-purity (99.9%) silver electrodes suspended in 1 L of DI water. The silver electrodes were cut from a stock sheet of 0.64 mm-thick silver supplied by Rio Grande. Depending on the test case, electrode area was varied by immersing more or less of the electrodes, and conductivity was varied via addition of NaF (for a final concentration of up to 3.7×10^{-3} M in the polarization test) or $NaNO_3$ (for a final concentration of 3×10^{-4} M in the electrolysis test). Conductivity and pH measurements were made with Hach CDC401 and PHC101 probes, respectively. The cell was powered and controlled by a Gamry Instruments Interface 1010E potentiostat. A two-electrode cell was employed, such that all voltages were referenced from the anode to the cathode. Silver concentration measurements were made with an ion-selective electrode (ISE) from Electrochemical Devices, Inc. and an Orion Versa Star Electrochemistry Meter, while periodic off-line measurements were made using inductively-coupled plasma mass spectrometry (ICP-MS). ICP-MS measurements of unfiltered samples and samples filtered through 0.2 μm syringe filters were compared to distinguish total from "dissolved" silver, in keeping with EPA Method 200.8.[*,8]

III. Results

A. Electrode Polarization Test

Based on Faraday's law of electrolysis, imparting a silver concentration of 400 ppb at a flowrate of 100 mL/min requires a current of 0.6 mA. This polarization test was performed to determine the cell parameters (electrode dimensions, water conductivity, applied voltage) required to achieve that current. Over sixty polarization curves were generated using staircase voltammetry under varying operating conditions: electrode area (A) from 2 to 100 cm^2, electrode spacing (d) from 0.5 to 2.5 cm, and conductivity (κ) from ~1 to 400 μS/cm. Two of these polarization curves are shown in Figure 1, where Figure 1a is representative of the curves obtained at low solution conductivity and Figure 1b is representative of those at high conductivity. In Figure 1b, the plateau at approximately 0.2 mA/cm^2 is indicative of increased resistance, due to either anode passivation or, more likely, a mass transport limitation (at either anode or

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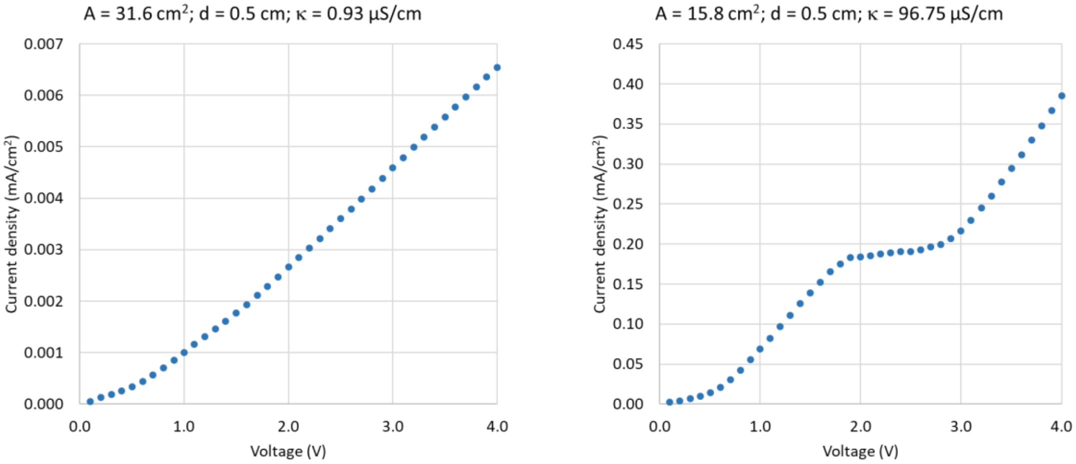
[*] The EPA method calls for 0.45 μm filtration for determination of dissolved analyte; 0.2 μm filtration is as convenient and slightly more refined.

cathode). In the latter case, the second upward inflection in the curve would indicate the onset of secondary reactions. Because either situation is undesirable, it is apparent from Figure 1b that applied voltages should not exceed 2 V.



(a) (b)

Figure 1. Representative Polarization Curves. *(a) Low conductivity. (b) High conductivity.*

To understand how the current-voltage relationship is impacted by conductivity, area, and spacing, the current measured at 1 V for each test case was plotted against the lumped parameter of cell conductance (Figure 2), which is the inverse of solution resistance, $R_{soln} = \frac{d}{\kappa A}$. Also plotted is the current-voltage relationship that would result if the polarization resistance were absent and only the solution resistance were significant. These lines are annotated as "ohmic predictions", for which the expected current is $I = \frac{\eta}{R_{soln}} = \frac{\eta \kappa A}{d}$, where the overvoltage η is the applied voltage in excess of the null voltage, which is estimated to be 0.4 V.[*] At high cell conductance, there is a significant divergence between the measured current and the ohmic prediction because the polarization resistance, R_{pol}, comprising the net resistive effect of charge transfer and mass transport, is significant due to the current density approaching its mass-transport-limited value. However, the ohmic predictions are reasonable at lower cell conductance values (Figure 2b-d) where $R_{soln} \gg R_{pol}$. This is relevant to the spacecraft application because the low conductivity of the potable water means that a silver electrolysis reactor would typically operate at a point much lower than its limiting current density.[†]

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[*] The null voltage was estimated as the average x-intercept of all of the polarization curves.

[†] If the WPA product water conductivity is intentionally increased to aid in the electrolysis process (e.g., by way of mineral addition), this assumption must be reassessed.

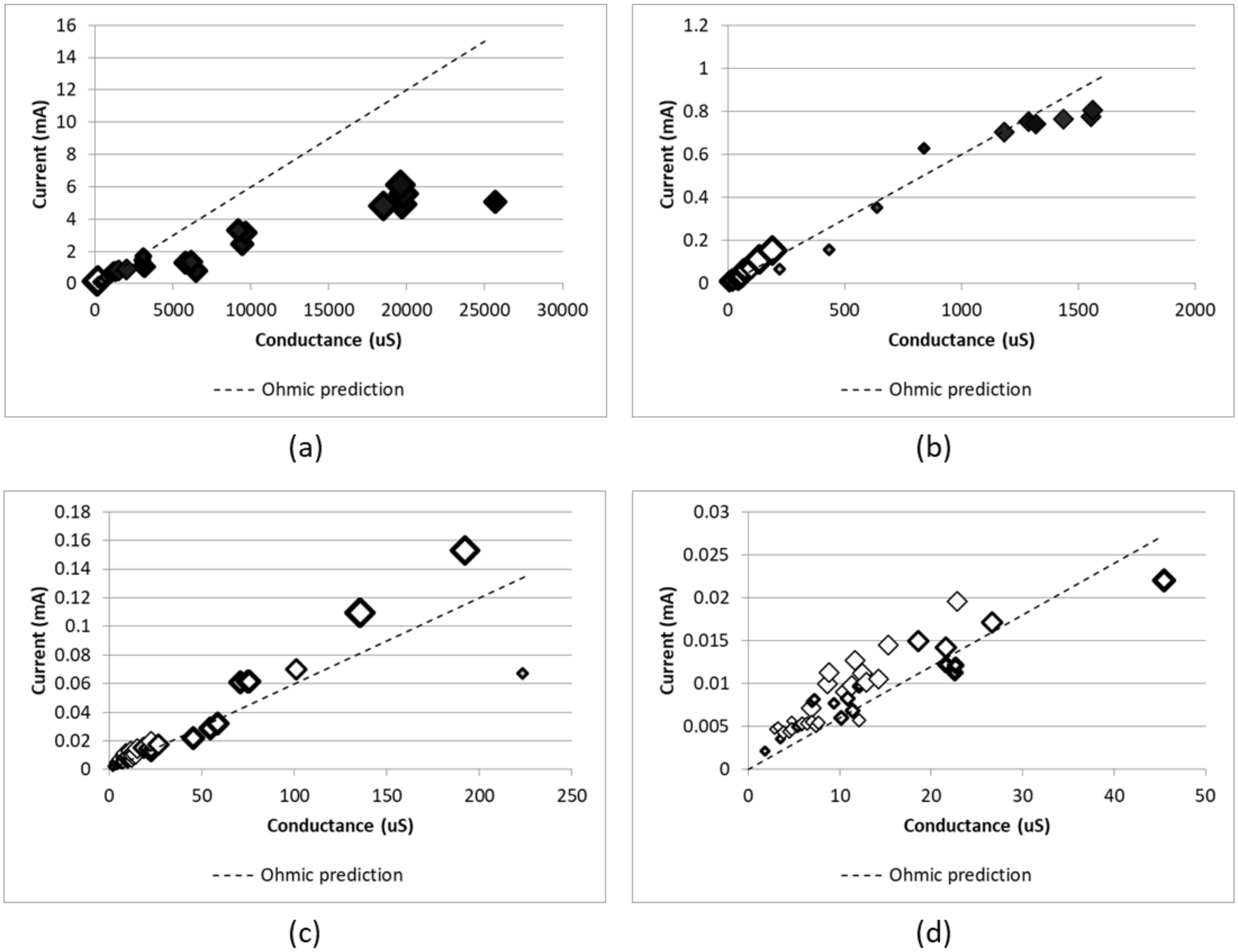

Figure 2. Current vs Conductance at 1 V. *Plot (a) shows the full set of data, and each subsequent plot (b-d) shows an inset of the previous plot. Data points are formatted according to the value of the test case parameters used to generate that data point: weight of border increases with reciprocal of spacing, size of data point increases with electrode area (not to scale), shading increases with conductivity.*

Data points in Figure 2 are formatted according to the test case parameters κ, A, and d to show that the linearity of the current-voltage-conductance relationship has no dependence on these parameters.* In other words, the current-voltage relationship varies linearly with conductance, regardless of how the cell conductance is achieved. Therefore, design of a silver electrolysis reactor can take advantage of the fact that the electrolysis current is proportional to the total electrode area and inversely proportional to the electrode gap. The ohmic prediction line in Figure 2b can be employed to estimate that a cell conductance of 1,000 µS is required to produce a current of 0.6 mA at 1 V of applied voltage.

Acceptable combinations of electrode area and spacing can then be determined from the cell conductance and expected water conductivity. For example, if one assumes a water conductivity of 1 µS/cm (typical of purified water in equilibrium with atmospheric CO_2) and an electrode gap of 0.5 cm, the required surface area would be 500 cm^2. On the other hand, if one assumes the conductivity expected in WPA product water (potentially as low as 0.056 µS/cm), the required surface area is roughly 18 times higher. However, as silver and hydroxide ions are generated via electrolysis, the conductivity of the water in the reactor will increase in the direction of flow, resulting

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* It should be noted that minor divergence from the ohmic prediction in many test cases is most likely primarily due to the difficulty in precisely controlling the test parameters, particularly electrode spacing.

in an average water conductivity in the reactor higher than that of ultra-pure water. Nevertheless, the average conductivity is still expected to be very low (on the order of 1 µS/cm), and a total electrode area of 500 cm^2 is likely a reasonable baseline for estimating overall reactor size. This electrode area can be packed into a large but reasonably-sized reactor by placing multiple electrodes in parallel: for example, one design solution results in an internal volume of 3 cm x 3 cm x 35 cm. Although an acceptable size for a silver dosing system has not yet been defined, it is assumed that these dimensions are close to the maximum.

If a reduction in reactor volume is needed, a number of design solutions may be considered, including: reducing the electrode gap, increasing the feed water conductivity, lowering the flowrate, altering electrode geometry/morphology to increase the effective surface area, and increasing the applied voltage. Reducing the electrode gap to 0.25 cm (half the previously-assumed value) could reduce the required reactor volume by a factor of nearly four, but the increased likelihood of cathodic silver deposition must be assessed. Increasing the water conductivity with consumable additives has disadvantages discussed earlier, but it is one of the most straightforward ways to decrease reactor size. Another possible method for increasing conductivity is to intentionally promote the permeation of carbon dioxide from the spacecraft cabin into the product water upstream of the reactor by way of a gas-permeable membrane. This would require no consumables and would have the added benefit of decreasing pH; however, the practical considerations of implementing such a scheme have not yet been assessed. Lowering the flowrate would allow the reactor to operate at a proportionally-lower current; however, the flowrate of the WPA is finely tuned for its catalytic oxidation reactions, such that significant changes in process flowrate would likely be unacceptable. Increasing the effective surface area of the electrodes could take the form of either roughening planar electrodes or employing three-dimensional electrodes, such as a silver mesh or foam. In either case, the difficulty in controlling the potential/current distribution across such electrodes makes it more likely for unwanted secondary reactions to take place. Finally, simply increasing the applied voltage is a means to achieve the required current without decreasing the solution resistance; however, doing so comes with the possible downside of allowing secondary reactions that diminish reactor efficiency and potentially passivate the anode. In summary, a number of potential options exist to address the problem of low water conductivity, but all require some amount of testing to determine their impact on reactor and overall system performance. The test described in the next section sought to elucidate the impact of high voltage on reactor performance.

B. Long-duration Electrolysis Test

In early phases of testing, several small-scale tests were performed with the goal of demonstrating the impact of applied voltage on anodic oxide formation. The majority of these tests were inconclusive because a significant charge has to be passed through the anode before oxide formation is detectable, and by the time this occurs, the pH and silver concentration of the water have shifted far from the values expected in a potable water system. In order to allow a test of sufficient duration while maintaining constant water conditions, a dual-headed peristaltic pump was used to flow water into and out of a 1 L beaker at the same rate in a continuously-stirred tank reactor configuration (Figure 3).

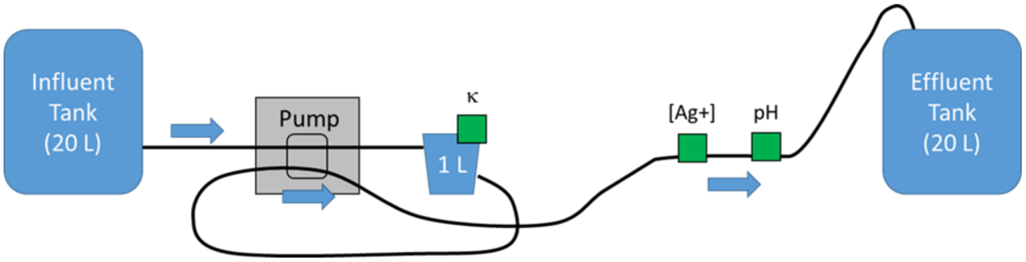

Figure 3. Functional Representation of Test Rig. *Green boxes indicate sensor placement.*

The cell was otherwise the same as previously described, with an electrode area of 8.7 cm^2 and spacing of 1 cm. It was operated at a flowrate of 400 mL/min and constant current of 1.49 mA; under these conditions, the expected effluent silver concentration is 250 ppb. The water conductivity was adjusted to approximately 12 µS/cm so that the voltage would be regulated near 8 V. This high voltage was selected to intentionally accelerate oxide formation in an effort to determine the minimum test duration required for parametric testing of oxide mitigation. The test was run for a cumulative duration of 36 hours, which is twice the duration at which the Reactive Innovations team experienced degraded performance.[*,1]

Figure 4 shows pictures of the inner faces of the two electrodes upon conclusion of the test. Formation of a black film on the anode is readily apparent. X-ray Photoelectron Spectroscopy (XPS) analysis suggests that the film is an oxide of silver but was inconclusive regarding the species of silver oxide. Based on the color of the oxide (black as opposed to brown), the lack of increased polarization resistance (discussed below), and the pH of the water in which the electrolysis was performed, the oxide is suspected to be AgO. The dull silver film on the cathode suggests that a portion of the silver ions redeposited there.

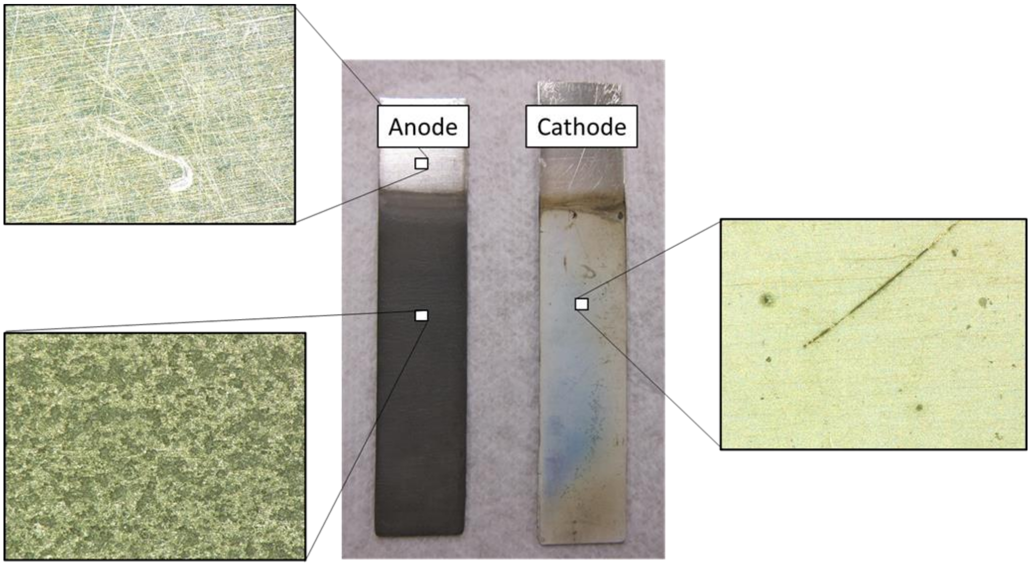

Figure 4. Pictures of Electrodes. *Insets show 200x optical microscopy of select regions.*

As shown in Figure 5, the voltage remained relatively steady throughout the test near the average voltage of 8.8 V, indicating the absence of increased resistance to current flow.[†] Furthermore, as shown in Figure 6, the silver concentration remained within 10% of the target concentration[‡] for most of the test, indicating that the ability to generate ionic silver to a target concentration was not significantly compromised by the development of the oxide. The ICP-MS data exhibits a subtle but consistent downward trend from 250 ppb at 6 hours to 221 ppb at 32 hours,[§] which may indicate a gradual decrease in current efficiency over the life of the anode. It is not clear whether this decrease in efficiency was due primarily to anodic side reactions or cathodic silver deposition; based on the photographs in Figure 4, both phenomena occurred to a great enough extent to be visible to the naked eye.

The overall good performance of the system is noteworthy, especially considering that the system was intentionally driven at a high voltage to stress it as much as possible. Over the course of the test, enough silver was generated to treat 225 days-worth of drinking water for one crewmember.[**] The fact that no obvious performance degradation was observed in that amount of time indicates that electrolytic silver ion generation is a viable approach for exploration missions and that oxide formation may not be as significant a concern as originally anticipated.

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[*] It should also be noted that the Reactive Innovations test was performed at 0.3 mA; thus, the total charge passed (and theoretically the total amount of silver produced) in this test was roughly ten times that in the Reactive Innovations test.

[†] Most of the variation in voltage was due to gradual variation in immersed electrode area caused by slight differences in the flowrate into and out of the beaker. In some cases, this caused sharp changes in voltage until the situation was corrected, such as that observed at 35 hours. Thus, variation in voltage was primarily an artifact of the test set up that will be eliminated in future testing by use of a flow-through reactor.

[‡] The target concentration is that which would be achieved with 100% faradaic efficiency.

[§] The filtered ICP-MS data is described here, but the total silver exhibited the same trend.

[**] Assuming 2.4 L/day consumption rate of water treated to 400 ppb silver concentration. 864 L of water was treated to 250 ppb in this test, which is equivalent to 540 L of water treated to 400 ppb.

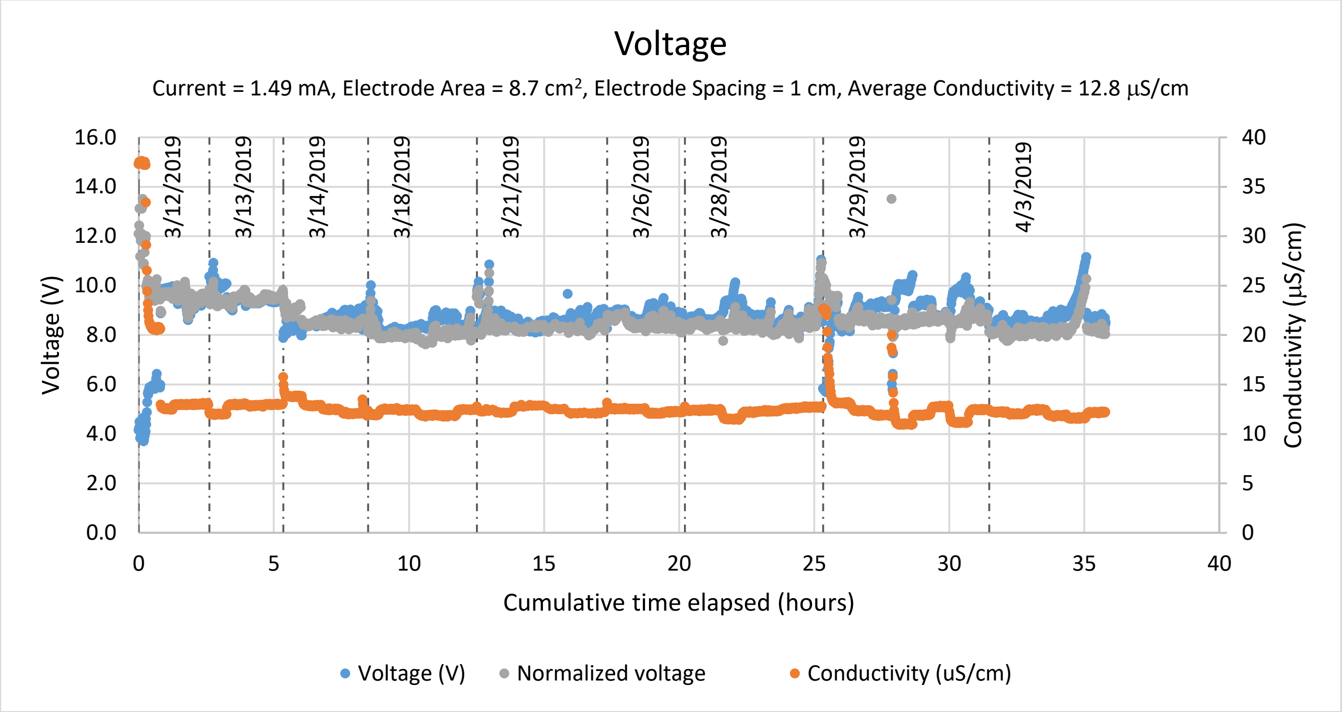

Figure 5. Voltage throughout test duration. *The normalized voltage accounts for fluctuations in the solution conductivity. It is calculated as* $\boldsymbol{V_{norm}(t) = V(t) * \frac{\kappa(t)}{\kappa_{avg}}}$

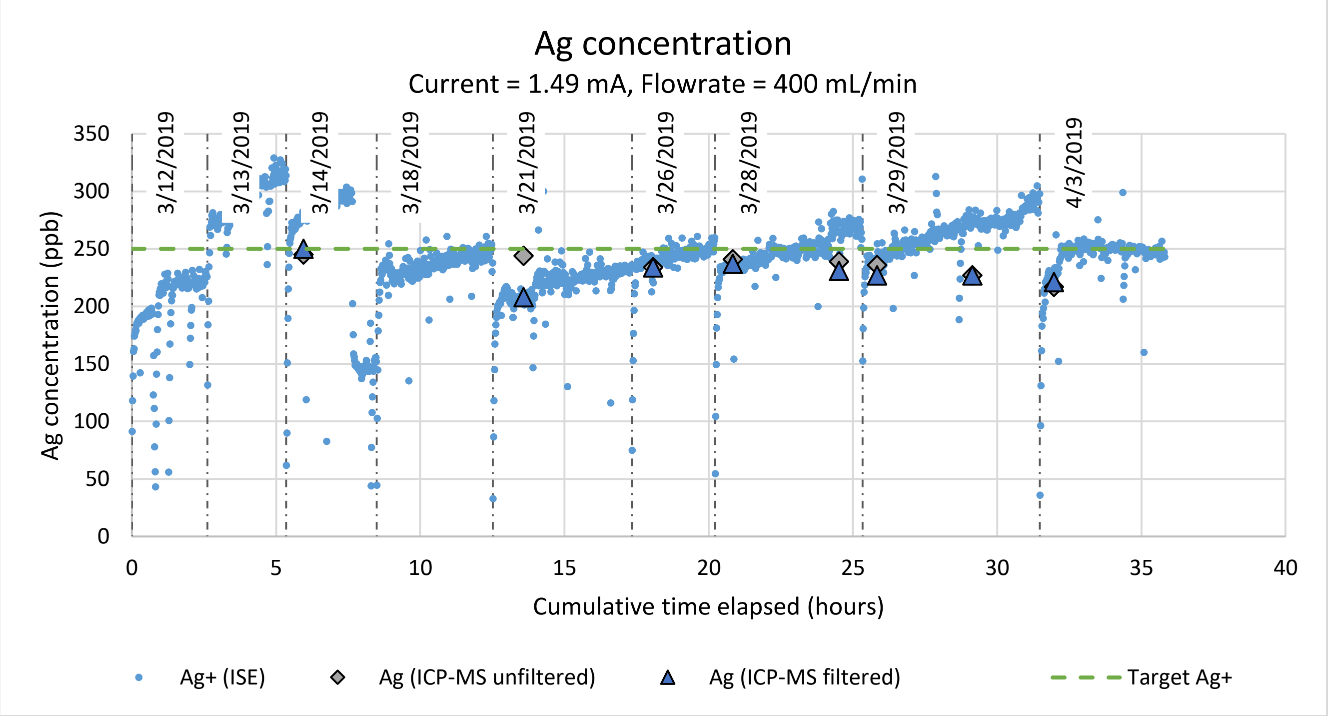

Figure 6. Silver concentration throughout test duration. *ICP-MS measurements were made on two samples taken at the same time, one of which was passed through a 0.2-micron filter to distinguish total versus "dissolved" silver. The ISE was calibrated at the beginning of each day, after which point it appears to exhibit an upward drift.*

Furthermore, the close agreement (in most cases) between the filtered and unfiltered ICP-MS data (Figure 6) indicates that no significant quantity of silver particles greater than 0.2 µm was liberated during this test. Comparison of ISE to ICP-MS measurement was intended to provide a means to indirectly detect any particles smaller than 0.2 µm, but the apparent drift of the ISE over the course of each day, which was most likely due to its continuous immersion, diminished the value of such a comparison. In future testing, off-line ISE measurements will be used to provide a better representation of the ionic silver concentration. Furthermore, future testing will seek to understand the operating conditions that promote or discourage the phenomenon of particle release.

The cause of the discrepancy in performance of the electrodes in this test versus the Reactive Innovations system is not immediately clear but could possibly be explained by differences in the operating conditions. For example, the design of the present test setup was simple and included a beaker of water in direct contact with lab air (and thus carbon dioxide), which results in potentially-significant buffering of the solution pH to lower values than are expected to occur in a closed system. The Reactive Innovations report does not discuss the pH nor the specific reactor design, but their reactor is assumed to have prevented direct contact with lab air, possibly resulting in a higher pH. The average pH in this test was 6.14 and the maximum pH was 6.83, whereas the pH expected in the effluent of a closed system is approximately 8.6. The open nature of the present test setup may thus have provided an artificial buffer against oxide formation. The next phase of silver electrolysis testing will use a flow-through reactor to provide test conditions more like an eventual spacecraft system than those afforded by the open-beaker setup described above.

IV. Future Testing

A silver electrolysis reactor test article has been developed to address the shortcomings of the previously-described test setup. The primary purpose of the test article is to facilitate parametric testing of the electrolysis process. The testing will seek to determine the operating conditions that result in the best performance (including avoidance of oxide formation and particle release) in addition to demonstrating the accuracy of silver dosing over a long duration in a representative environment. Secondary purposes include 1) providing a proof of concept for a specific reactor design and 2) providing a source of electrolytically generated silver solutions for other tests (e.g. materials compatibility and microbiological testing).

The test article comprises a flow-cell containing multiple parallel plate electrodes. The flow-cell accommodates up to 10 electrodes, at spacings of 0.25, 0.50, or 0.75 cm. The baseline configuration of six electrodes spaced 0.50 cm apart assumes a water conductivity of 1 µS/cm, a flowrate of 100 mL/min, and an applied voltage of 1 V. Other conductivities and voltages can be accommodated in testing by virtue of the alternate configurations in addition to the use of lower flowrates.

The reactor test article will facilitate future testing by providing greater control over electrode area and spacing and by enabling test conditions that are closed from the atmosphere. Initial test cases will assess the impact of surface finish and applied voltage to electrode longevity and particle release. Subsequent testing will investigate the impact of influent pH, conductivity, and dissolved oxygen concentration and the impact of polarity reversal frequency on reactor performance. Testing will also seek to determine whether cathodic silver deposition is a significant concern, and whether the electrode geometry (electrode area, configuration, and spacing) can be optimized to mitigate it.

V. Conclusion

Preliminary testing shows that silver electrolysis could be a feasible technology for in-line introduction of biocidal concentrations of silver into spacecraft potable water. The formation of a resistive oxide may not be a significant concern, especially if the cell is operated at a low voltage, but this must be evaluated at higher pH. Although the phenomenon of silver particle release was not directly studied, preliminary test results indicate that it may not be a significant problem at low current density. The largest hurdle to implementation appears to be the large reactor size required for operation in very low conductivity water. A smaller reactor would be achievable with moderate or high applied voltages or via in-line addition of conductive species (e.g. minerals or carbonic acid) upstream of the reactor, but these strategies need to be assessed for their impact on the performance of the reactor as well as downstream systems. Future testing will seek to optimize the design of a silver electrolysis reactor so that its mass and volume footprint can be minimized while still delivering an accurate silver concentration over a long duration with no adverse impact to effluent water quality.

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