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Analysis of Performance Degradation of Silica Gels after Extended Use Onboard the ISS

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The teardown of two flight desiccant beds from the Carbon Dioxide Removal Assembly (CDRA) revealed significant discoloration of the silica gel near the bed inlet as well as a coincidental performance loss. This material was analyzed for the presence of chemical contaminants, physical porosity changes, and adsorption performance. The material characteristics are compared against the location in the bed from which they were sampled in order to develop profiles through the bed. Additional testing of the beds prior to teardown provided more data points. Possible mechanisms for the loss of capacity are provided though no root cause has been found. Extrapolation of the performance loss is used to estimate the required oversizing of the silica gel layer for long-term operation.

Nomenclature

CDRA = Carbon Dioxide Removal Assembly
DAB = Desiccant-Adsorbent Bed
ISS = International Space Station
POIST = Performance & Operations Issues System Testbed
4BMS = 4-Bed Molecular Sieve
TGA = Thermogravimetric Analysis/Analyzer
DSC = Differential Scanning Calorimetry
HC = Half-Cycle
GC-MS = Gas Chromatography – Mass Spectrometry
ICP-AES = Inductively Coupled Plasma – Atomic Emission Spectroscopy
BET = Brunauer, Emmett, and Teller method
MFC = Mass Flow Controller
D3-D6 = Silicon-Oxygen (Si-O) Siloxanes with 3 to 6 Cyclic Si-O Functional Groups
SG40 = Sylobead® SG 40 (Grade 40)
SGB125 = Sylobead® SG B 125 (SGB125)

I. Introduction

ONBOARD the International Space Station one of the CO_2 removal systems is the Carbon Dioxide Removal Assembly (CDRA). CDRA uses desiccant beds which contain silica gel as the bulk drying material. Upon return from orbit, materials used in the desiccant beds for the CDRA system were found to be significantly altered from original. This alteration was found to be three-fold: a readily visible translucent orange hue for a previously clear and colorless material, adsorption of various low vapor pressure trace contaminants found throughout the ISS cabin atmosphere, and, most importantly, reduced desiccant performance. This paper summarizes the efforts conducted to present to characterize the material changes and also predict whether it will have any negative impact on the performance of a system during a long-duration mission.

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Anecdotal past experiences have shown this discoloration to be present in both on-ground and in-orbit materials, though not in all tests. The Performance & Operations Issues System Testbed (POIST) test series in 2003 was run for extensive amounts of time and showed a similar discoloration, with photos of flight and POIST bed teardowns provided in Figure 1. Other flight bed teardowns have shown similar discoloration but samples were not made available for study. During the time where CDRA-3 beds were in use on station, siloxanes were identified as a major concern for atmospheric contamination. The effect of siloxanes was significant on other systems on the ISS, therefore it was decided to study the silica gels which are most directly exposed to ISS cabin air contaminants.

Two commercially available silica gels have been used as the bulk desiccant in the various CDRA iterations. Both materials were manufactured by Grace-Davison and are known as Silica Gel Grade 40 (SG40) and Silica Gel B125 (SGB125). SG40 is a granular material while SGB125 is a beaded material with superior structural resilience and virtually identical performance versus SG40.

At the time of this report, two pairs of desiccant beds have been disassembled and sampled for further study. To date, fifteen desiccant beds have been flown as part of Desiccant-Adsorbent Bed (DAB) units. Several studies on the samples from first pair of flight desiccant beds were conducted in 2013-2015. This also resulted in the first set of flight samples being severely depleted. GC-MS analysis identified numerous components, particularly siloxanes D3-D6, to be present in the samples from the end of the bed closest to the cabin air inlet/outlet. ICP-AES analysis identified compositional changes throughout the bed.

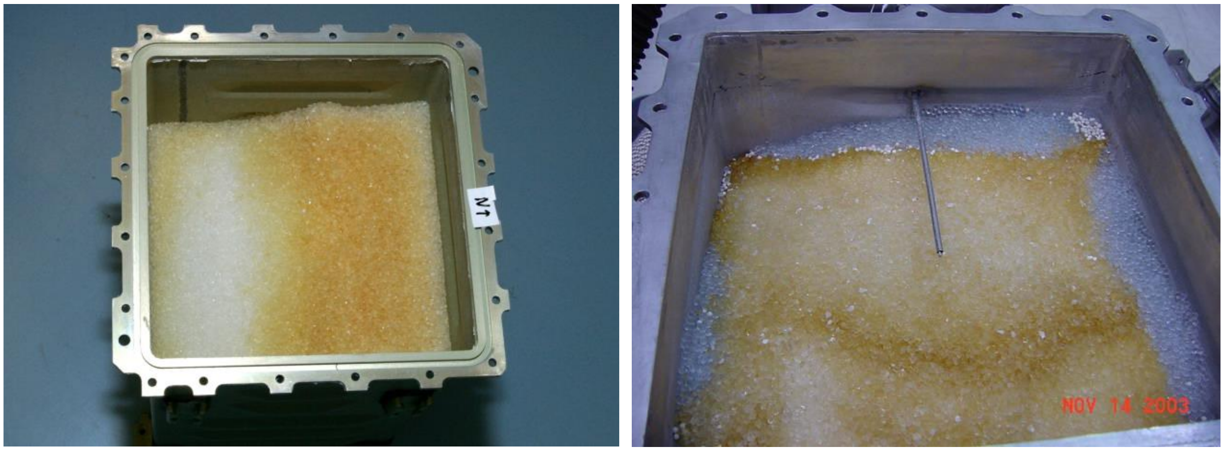

Figure 1: Photos of desiccant bed teardowns for flight (left) and POIST (right) showing discoloration of silica gel. Samples are removed layer-by-layer with a vacuum transfer line enabling sample collection at specific depths.

From these data points, a need to study the next available flight desiccant beds was identified. Test plans were developed to replicate the previous studies and determine new factors. In addition to GC-MS and ICP-AES analysis, BET analysis and newly developed water vapor adsorption analyses would be conducted. Flight DABs were returned from flight from CDRA-4 in 2015 for teardown and study.

II. Test Procedures and Test History

Analyses conducted by the Materials Test Lab at MSFC include: GC-MS, ICP-AES, and BET method adsorption. These tests follow standard procedures established by the Materials Test Lab. Adsorption tests conducted by the ECLSS branch at MSFC were designed for this test series.

After return from flight but before bed teardown, the first test was conducted on the desiccant bed D0003. This first test was conducted in three parts. First, the desiccant bed was installed in CDRA-4EU and tested with the improved instrumentation to determine if water breakthrough occurred. Second, the bed was thoroughly dried in an oven up to 175°C. Finally, the bed was challenged with a 10°C dew point water vapor stream for an extended period of time. After this test, the unit was dried and returned for teardown. Teardown was conducted via layer-by-layer removal of material with a vacuum hose. At specified depths, the removed material was recovered for testing.

The samples recovered from desiccant beds D0003 and D0004 from CDRA-4 were tested for water vapor adsorption performance. A SETARAM Sensys Evo TGA with a Wetsys water vapor generator, an auxiliary MFC, and a LICOR LI840a CO_2/H_2O gas analyzer were used to control and monitor the gas composition, temperature, and sample mass. Samples were activated at 50°C to mimic past testing, which dried samples at 45°C, with the reasoning that an activation temperature above nominal CDRA operation would potentially drive off adsorbed contaminants and alter the results. Samples are very small, typically 2 beads per measurement.

III. Results and Discussion

A. Chemical Analysis

Chemical composition analysis results from the CDRA-3 samples showed a correlation between siloxane content and loss of performance. Other correlations were noted between excess metal content and performance loss. The chemical composition analysis results from the CDRA-4 samples shows no correlation between siloxane nor metal content and loss of performance. There was no detectable siloxane content on all of the CDRA-4 samples except for a small amount on the Sorbead WS material sourced from D0003. It is possible that the adsorbed siloxanes would be lost to evaporation and permeation over time as this was observed in duplicate testing of CDRA-3 samples. The metal content was very consistent across the entire depth of bed D0003. As will be shown later, the adsorption performance strongly varies with sample depth, therefore any assumption of causation between performance loss and siloxane and/or metal content must be eliminated.

B. Porosity Analysis

Surface area and pore volume analysis via the BET method showed a consistent trend across sample depth. The BET method utilizes N_2 adsorption at 77K and enables study of the surface area, micropore volume, total pore volume, pore size distribution, and a number of other porosity properties. Figure 2 shows a summary of the properties for samples from D0004 compared with control samples. Surface area losses of up to 35% are measured which is slightly less severe than that measured for the samples from CDRA-3 of 45% loss. Surface area correlates with adsorption performance in virtually any situation, including this study. Pore volume correlates with total capacity which occurs when pores completely fill with sorbate molecules. Average pore width and micropore volume provide additional information on the size and shape of the pores. Surface area, pore volume, and micropore volume are found to increase and show a positive correlation with adsorption capacity. Average pore width shows the opposite trend but the negative correlation is consistent across the series of samples. The conclusion from this set of data is that the interconnected network of pores are altered in a manner that yields fewer pores and skews the structure towards more macropores. When pores are blocked causing a reduction in pore volume, the total adsorption capacity will be reduced. When the surface area and micropore volume are reduced, the adsorption performance at low partial pressures will be reduced.

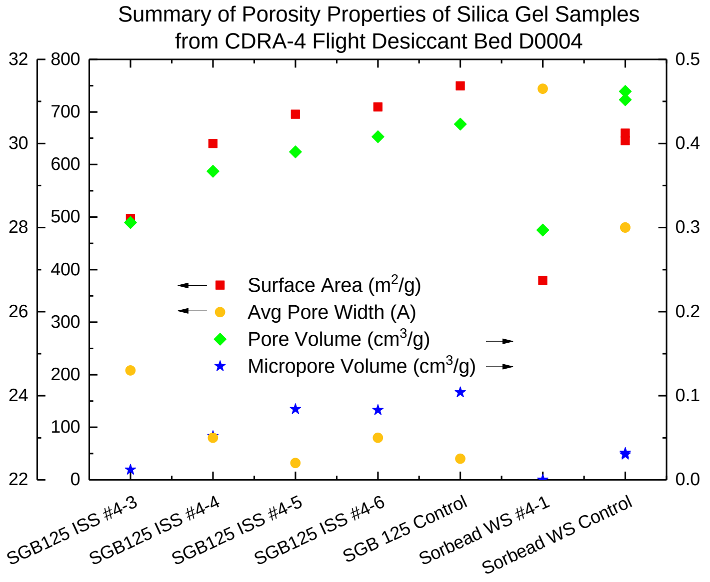

Figure 2: Summary of porosity properties for a series of samples from desiccant bed D0004 used in CDRA-4.

C. Water Vapor Adsorption at 25°C – Low Concentration (up to -2°C dew point)

In order to probe the effects of adsorbed contaminants in the materials, low concentration isotherms were measured on the available materials. At low concentrations, adsorption should occur via a layer-by-layer mechanism on the surfaces of the pores. This is as opposed to a pore filling mechanism, where water vapor condenses as a continuous liquid phase within the pore chambers. Therefore, any measured uptake of water vapor should correlate to the availability and affinity of the pore surfaces for water. Should hydrophobic contaminants, such as siloxanes, be present as a surface monolayer within the pore structure

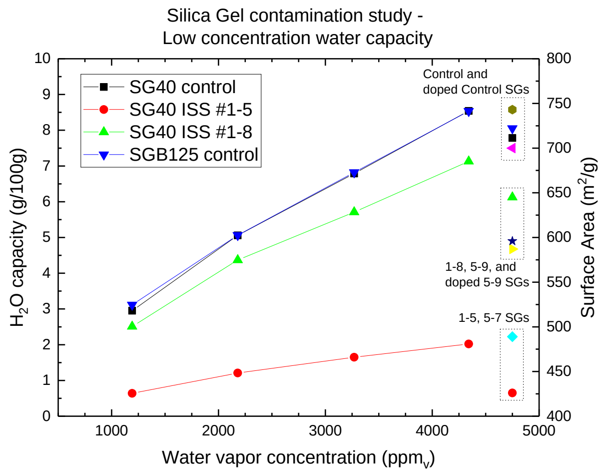

Figure 3: Results of testing on remaining S40 samples with comparison to control samples of SG40 and SGB125 as well as with all available past surface area analyses of the various samples.

then a marked reduction should be observed due to the reduced affinity whereas N_2 surface analysis should not show a major difference. If contaminants are present at higher quantities beyond a monolayer, then both water and N_2 capacity will be reduced. Alternatively, should the structure be compromised, then both the water vapor adsorption and N_2 adsorption analyses will show reductions.

In Figure 3, the first iteration of testing on the two remaining flight SG40 samples was compared with unused SG40 and SGB125 from drums kept in storage on the ground. The surface area values from a previous report were included to aid visibility of the two trends. The results in that report indicated a severe degradation of material performance and showed a correlation to both presence of contaminants and reduction in surface area. Initial results shown in this report support those correlations. A definitive conclusion could not be reached in this test series with the limited test material remaining from the CDRA-3 flight beds D0001 and D0005.

The flight desiccant beds used in CDRA-4 were returned and sampled. A slightly altered procedure was developed to test this series of SGB125 flight and control samples with all of the results shown in Figure 4. The results from porosity analysis and chemical analysis indicate that the best correlation to loss of water capacity is loss of porosity. This could indicate that either a physical pore closing process is responsible for the performance loss. Since no recoverable siloxanes were observed in the CDRA-4 samples, a contaminant coating of the surface can be discounted. From the measurements, a reduction of surface area by 45% results in a nearly 70% water capacity loss as shown by the difference between control and 1-5 samples while for the 3-3 and 4-3 samples show a 35% reduced surface area and a 50% capacity loss. One major caveat is that the 1-5 sample had a selection bias due to the few remaining granules.

The water vapor isotherms shown previously are reduced to two particular water vapor concentrations for easier comparison in the form of a column chart. Since the data points were not collected at exactly the same concentrations, values were normalized to 3250 and 5250 ppm by assuming a linear relationship between concentration and capacity near those levels. A simple look at the isotherms will justify this adjustment. Since each sample was tested at least twice, the results are reduced to mean and span values for each sample. Finally, correlating the notes taken during bed teardowns and with available surface area data yields the plots shown in Figure 5.

The trends are quite clear in this format. Surface area correlates strongly with capacity. The samples taken from the end of the desiccant bed closest to the cabin air inlet show the greatest loss of performance, while samples taken from positions that correspond to drier sections of the bed which also see hotter temperatures are more similar to control samples. All flight samples show at least a slight reduction in performance against control samples.

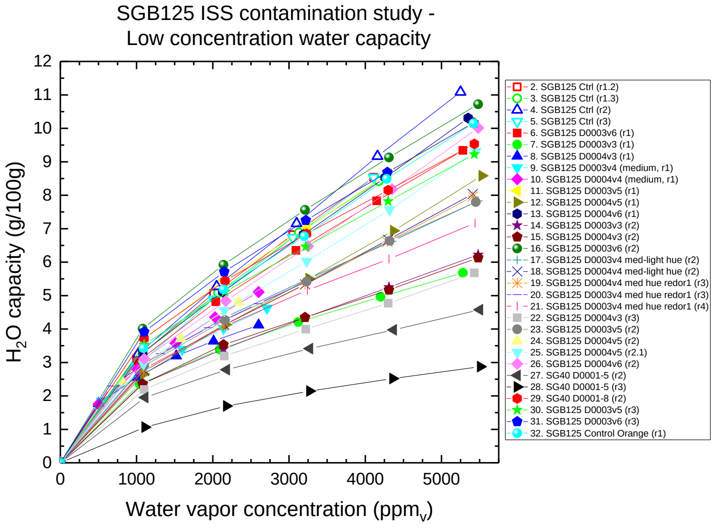

Figure 4: Isotherms obtained for silica gel samples obtained during low concentration water vapor tests.

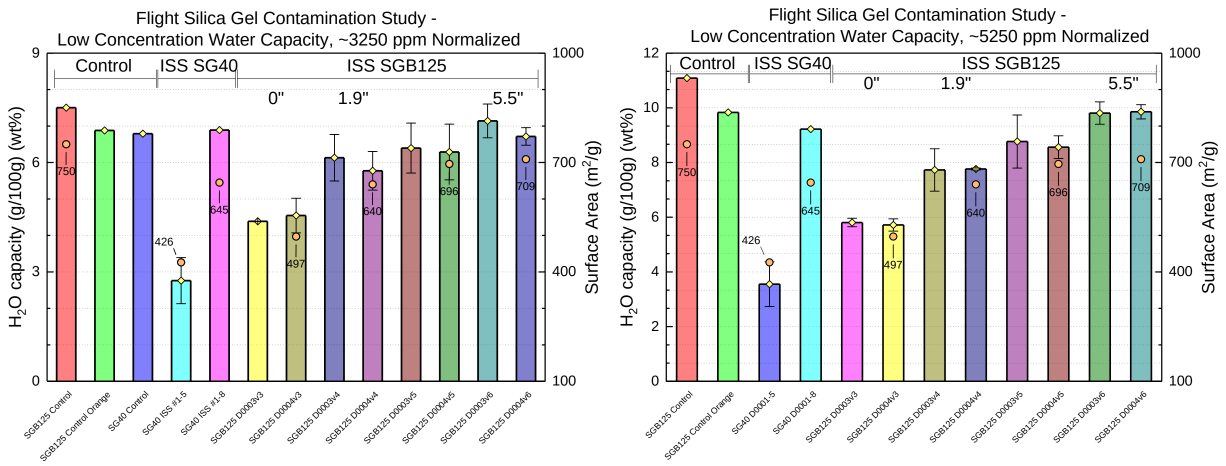

Figure 5: Results of water vapor adsorption test at low concentrations. Adsorption was conducted at 25°C and interpolated at 3250 and 5250 ppmv.

D. Water Vapor Adsorption at 25°C – High Concentration (up to 10°C dew point)

To affirm the working theory that the cause of performance loss is loss of surface area, a study of the materials at water vapor levels equivalent to 10°C dew point at 25°C sample temperature was conducted. At the time of these measurements, neither lower sample temperatures nor higher dew points could be obtained. The resulting isotherms are shown in Figure 6 and are connected with the previously obtained isotherms. At high capacities, a nearly linear extrapolation from lower water vapor concentration results is observed. The front of the bed is severely degraded by almost 50% from the control sample while the remainder of the bed shows a 15% to 25% reduction in capacity. This performance loss may be significant to system operation as the front of the bed adsorbs a large fraction of water during each cycle.

The normalized capacity values are shown in Figure 7 along with corresponding surface areas as reference. These isotherms do not show a distinct plateau at high concentrations, which would indicate complete saturation of the pores, thus no correlation can be made between total pore volume and total capacity.

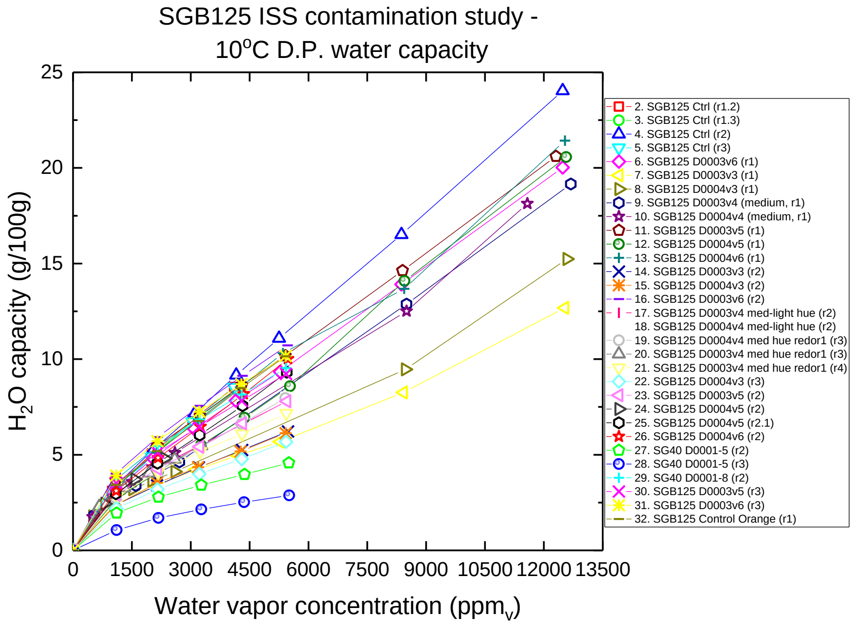

Figure 6: Isotherms obtained for silica gel samples obtained during low concentration water vapor tests.

E. Computer simulation of CDRA operation

An ongoing effort to precisely model the complex dynamics of a cyclic CO_2 removal system may be able to provide some insight. During desorption cycles of a desiccant bed, a water vapor concentration wave and a hot temperature wave are observed. The hot purge drives desorption by providing the endothermic desorption process with heat. Ahead of the hot purge a cold region develops due to some desorption occurring into the dry gas. These thermal

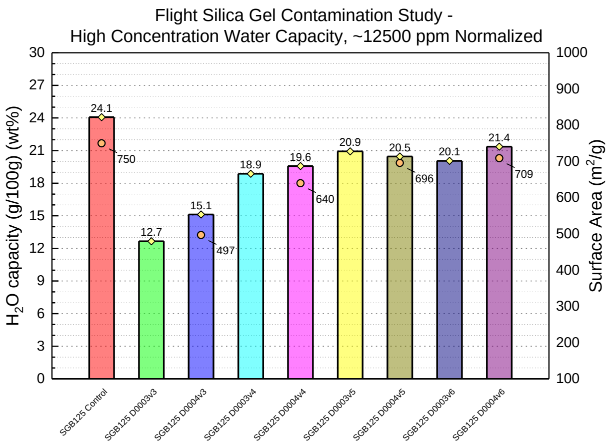

Figure 7: Results of one water vapor adsorption test at high concentrations which closely match the inlet concentration for CDRA. Adsorption was conducted at 25°C and interpolated at 12,500 ppmv.

and concentration waves progress through the bed at different rates with the concentration wave preceding the hot purge wave.

There may be a point where the cool, water-laden wave leads to a condition of 100% relative humidity. Contact of silica gel with liquid water is known to cause silica gel fracturing. The computer simulation does indicate a condition of 100% RH within the silica gel layer near the cabin air inlet end as shown in Figure 8. This 100% RH condition occurs within the first third of the bed as bounded by sample depths that showed the most degradation (D3-3 to D3-4 and D4-3 to D4-4). Perhaps this unique condition is a contributor to the silica gel degradation.

F. Test Data from 4BMS Operation

In addition to computer simulations of the next generation system, a full-scale test stand (4BMS-X test stand) is being used to validate against existing system behaviors and test new conditions. On many occasions during operation of this system, droplets of water were observed at the system outlet. Data from one of the most recent test series is shown in Figure 9. The data gathered during desorption of a number of test series show the exit dew point nearly parallel with a temperature probe (labelled desiccant bed inlet) embedded at a point that would be equivalent to between sampling points 3 and 4.

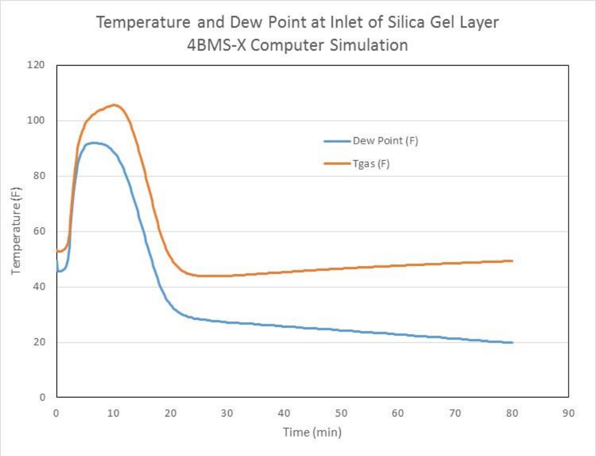

Figure 8: COMSOL simulation of the 4BMS system in operation showing the air temperature and dew point at the inlet of the silica gel layer of the desiccant bed during a desorption cycle. The two traces overlap indicating a condition of 100% RH.

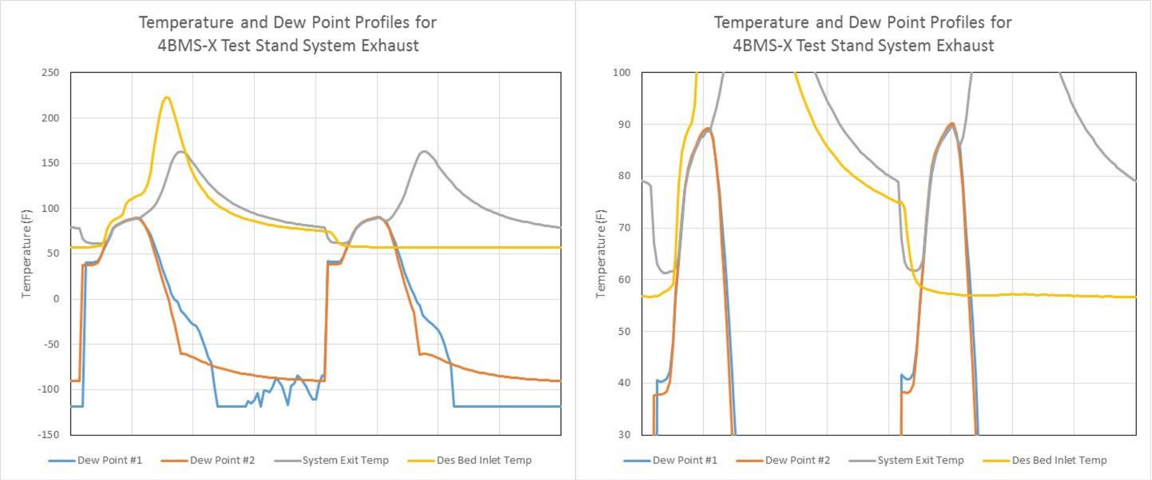

Figure 9: 4Xr3-61 test data showing two scales of the temperature at the inlet of one desiccant bed and the system outlet along with dew point measurements of the system outlet. Of particular note is the overlap of temperature and dew point curves at the early parts of a desorption cycle.

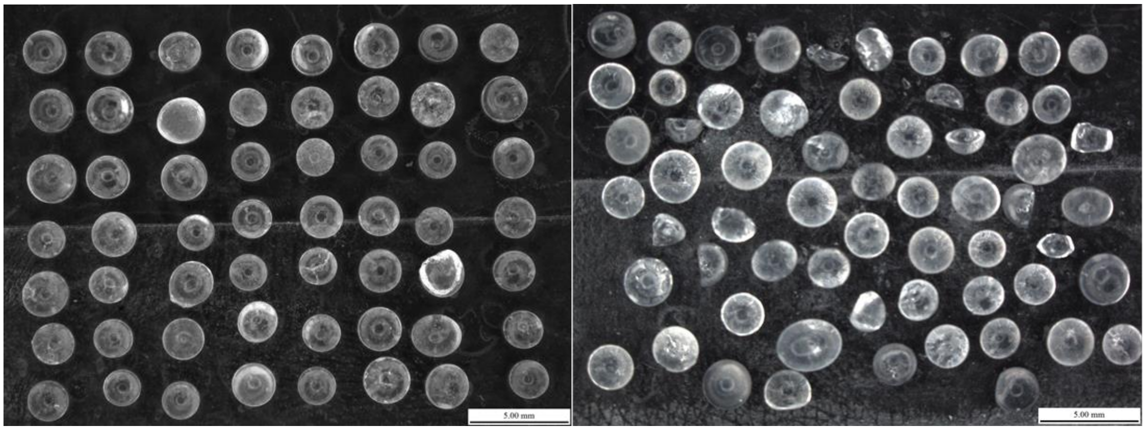

Figure 10: Photos of SGB125 from misting test report. Before [left] and after [right] misting.

Despite hundreds of half-cycles of operation, these desiccant beds did not have observable silica gel discoloration nor observable particle fracturing when beds were disassembled and emptied. No record of the exact number of half-cycles of operation presently exists. A procedural bed teardown and depth sampling was not required and therefore did not occur.

G. Silica Gel Misting

Based on manufacturer notes, SGB125 and SG40 particles break when exposed to liquid water. An internal test regarding misting-induced breakage was conducted and reported in 2015 by an ECLSS summer intern. Silica gel samples were conditioned with water vapor at 10°C dew point at ambient temperature then sprayed with a fine mist of liquid water. The results of that work was that silica gel beads fracture and show a slight loss of capacity. Figure 10 shows the broken SGB125 samples from this test. Since the test was conducted only once per sample, perhaps the effects on internal structure occurs only after repeated exposure and cycling.

No broken silica gel beads were mentioned in flight bed teardown notes, perhaps due to the difficulty in observing them. A small number of broken silica gel beads were found in the D3v3 and D4v3 vials but were only observed after a great deal of effort. Pictures of these broken beads are shown in Figure 11 and the pattern of breakage is similar to the control test with the major exception of orange discoloration. One conclusion that can be made is that the concentration wave poses a significant stress on the silica gel particles during each desorption cycle, sometimes to an extent similar to misting, and may be a cause of performance loss.

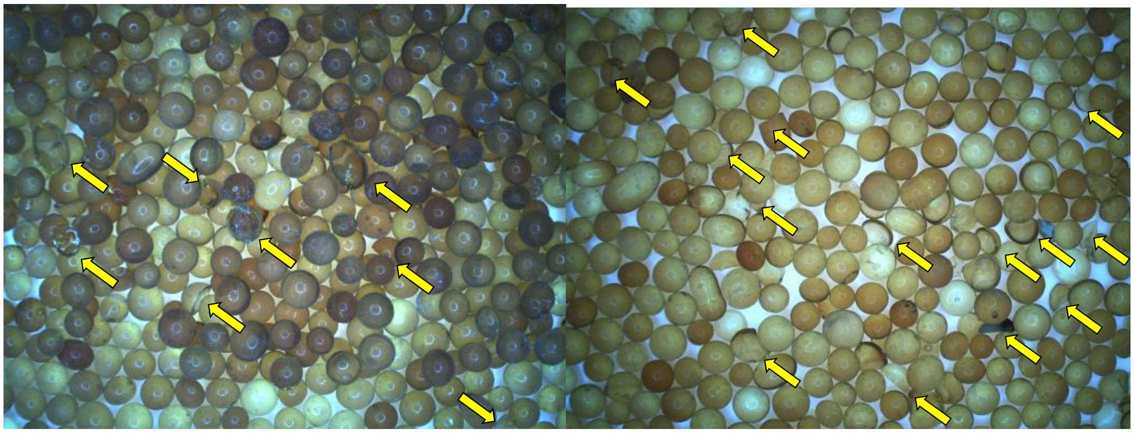

Figure 11: Photos of SGB125 sampled from the inlet of both CDRA-4 desiccant beds with arrows indicating some of the broken silica gel particles intermixed with the bulk.

H. Water Vapor Adsorption – Sorbead WS

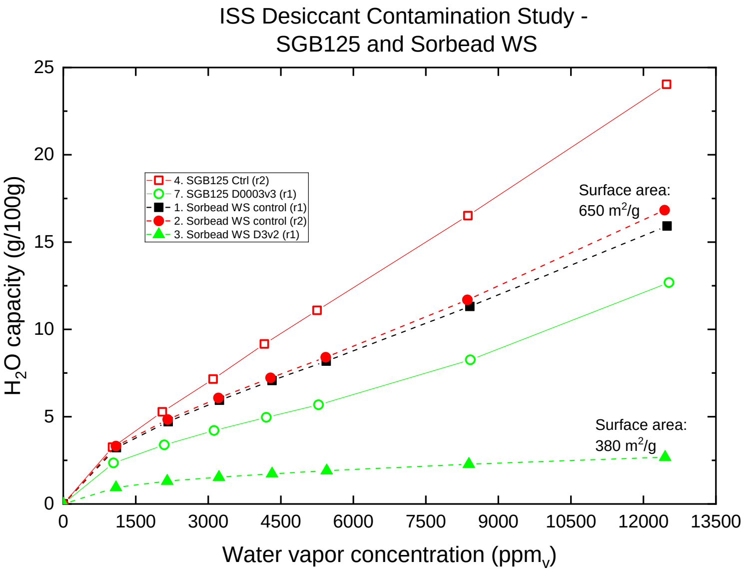

Figure 12: Water adsorption isotherm on two silica gels, SGB125 and Sorbead WS, showing the remarkable capacity loss of flight samples relative to control samples.

Both water vapor adsorption tests were conducted on the vials of desiccant that was sampled from the guard layer. The guard layer is 1" deep and composed of BASF Sorbead WS, a misting-stable silica gel with high robustness but reduced desiccation performance. The guard layer is the first sorbent layer exposed to incoming air and thus is expected to be most severely affected by possible contaminants. The BET surface area for control samples is 650m^2/g while it is 380m^2/g for flight sample #4-1. The same set of low and high dew points were conducted on this material. The results in Figure 12 clearly show roughly an 85% loss in performance for this robust material. This is extremely surprising considering the primary criterion for this material's selection was robustness. This observation indicates the mechanism which imparts particle robustness is different from the mechanism for pore stability.

I. Water Vapor Adsorption – Zeolite 13X

A new water vapor isotherm measurement procedure was developed to study the performance of the 13X zeolite used in the flight desiccant beds. This material is commercially available as MS544 from Grace-Davison and additional lots of the material are available at the MSFC test facility, though not the same lot as the flight lot. The results in Figure 13 show that the material sampled from flight bed D0004 is unchanged versus the control sample across a wide range of adsorption temperatures. New capabilities enabled simultaneous DSC operation which provides heat of adsorption data. This new data is shown, although it is noisy and imprecise. The overall expected downward trend and the magnitude encompassed by the data are in line with expected values.

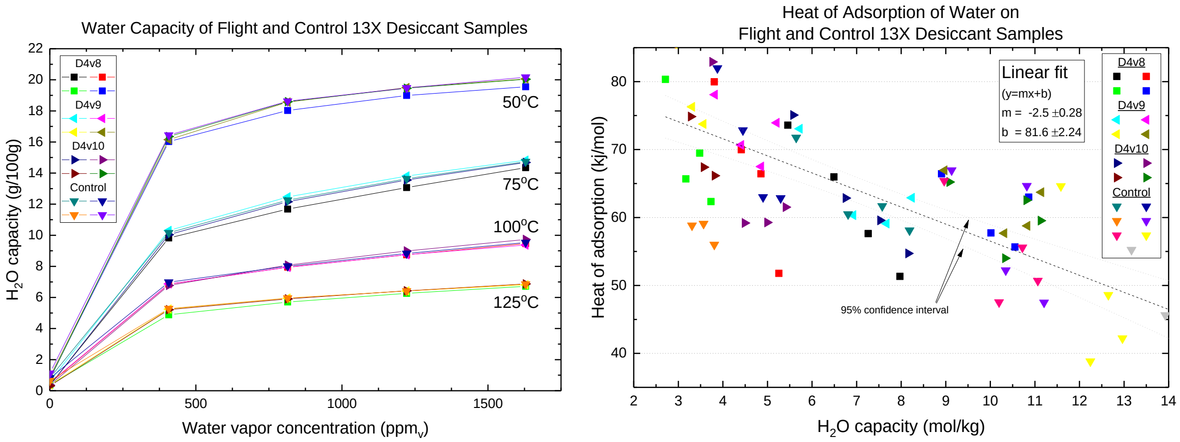

Figure 13: Water adsorption isotherms and heat of adsorption measurements on control and D0004 flight samples of zeolite 13X showing the seemingly imperturbable performance of the material.

J. Bed Oversizing Estimates

The natural goal of analyzing this data is to generate a prediction for desiccant bed sizing. A calculation yields 19,710 half-cycles for a 3 year mission with 80 minute half-cycles. Thus, a basis of 20,000 half-cycles will be used for estimates. The estimates for sample lifetime are largely extrapolations due to insufficient data and that fact that no CDRA unit has operated for 3 years or in excess of 6,000 half-cycles.

1. Estimate – Linear capacity loss from average bed performance

The first estimate is based on the reduced initial breakthrough time of CDRA-4 D0003 versus DevW and DevS from an internal report on the testing of the beds after return-from-flight but before teardown. The data in this report indicated that material degradation from use in flight had occurred. The ultimate capacity for the D0003 bed is roughly 85%, of the very similar DevS bed, as shown in Table 1. The first detectable breakthrough occurs at ~65% of the time of the ground-based units. These two values are the best available reference points for developing a bed sizing estimate. Since the purpose of these beds is to maintain extremely dry gas streams, the smaller of these two values is of more concern.

Table 1: Desiccant bed weights for flight bed D0003 and the two development beds used in the 4BMS test stand.

Bed Weights (lbs)	FltD0003 #2	DevS #2	DevW
Initial ‘Dry’ Weight	34.55	34.5	40.1
Final ‘Wet’ Weight	38.95	39.6	45.4
Increase in Weight	4.40	5.1	5.3

At conditions similar to the complete breakthrough of water vapor in a desiccant bed, fresh silica gel adsorbs water up to 38% by weight while zeolite 13X adsorbs up to 25% by weight and require significantly more heat to desorb. The beds are roughly 50/50 by volume and silica gel has a roughly 15% higher packing density based on packing masses from Cylindrical Breakthrough Test logs. If silica gel performance loss accounts for the entirety of total capacity reduction, then the expected average silica gel performance would be ~78% of control samples.

Extrapolations of initial breakthrough time assuming that the entire cause of the accelerated breakthrough is loss of silica gel capacity yields a linear relationship. At 100% performance, a breakthrough time of 297 minutes is observed, while at 78% performance, a breakthrough time of 190 minutes is observed. Since only 2 data points cannot predict a curve, a simple linear extrapolation is the only possible estimation. The design half-cycle time of the next generation 4BMS is 80 minutes, which would correlate to initial breakthrough occurring when the silica gel capacity drops to 55% of original levels. Assuming a linear rate of average capacity loss through the silica gel layer, this performance level would occur after roughly 9650 half-cycles.

For now, the 55% capacity value will be used as a cutoff for silica gel performance, below which water breakthrough would occur. Further refinement of this estimate will be approached via computer simulation.

2. Estimate – Two mechanisms for capacity loss are applied to halves of silica gel layer

A second estimate is that the high temperature used to regenerate the 13X causes a degradation of the Silica Gel. From POIST and 4BMS-X test data, the temperature of the silica gel reaches a peak of 170°C whereas the standard regeneration temperature is 155°C. It is known that exceedingly high temperatures on the order of 350°C will permanently damage the pores of silica gel by chemically removing hydrophilic hydroxyl groups[1]. This report activated silica gel at 200°C under vacuum for 2 hours without noticeable differences from the 120°C activation baseline. Development beds and flight bed D0003 have been activated per standard procedure in an oven at 190°C (or at 175°C depending on oven capabilities). Therefore, the case for this estimate is extremely weak.

Temperature measurements in the desiccant bed lacks resolution with depth of the bed (only layer inlet and outlet measurements are available). The hottest end of the bed reaches 175°C while the coldest end never exceeds 100°C, based on POIST data. Finally, 155°C is the standard activation temperature recommended by the manufacturer. Therefore, only a portion of the bed can be assumed to suffer thermally induced degradation. Since samples v5 and v6 from both beds show equivalent behavior and on average show 86% of the capacity of the control sample, then the assumption to be made is this is the post-thermal degradation capacity for the hotter half of the bed.

Extrapolating from this assumption, the silica gel layer is divided into two halves. The hotter half characterized by the v5 and v6 samples will be assumed to rapidly lose 14% of total capacity but remain at that performance level indefinitely. The colder half will be characterized by the v3 and v4 samples and will show a continuous loss of performance akin to the first and second estimations. This set of assumption essentially means that half of the bed will be nearly at peak performance for an indefinite amount of time and the estimated lifespan in half-cycles is over 42,500. This means the bed size does not need to be increased any further.

IV. Conclusions

Flight desiccant beds returned from use on station have shown a loss of performance. Attempts to quantify the extent, identify the cause, and predict the operating lifespan before failure are provided in this report. A threshold for drying failure, defined as detectable breakthrough of water vapor, was estimated to occur when the silica gel layer of a CDRA-4 desiccant bed was reduced to 55% of original capacity. This is a conservative estimate. The estimated additional mass of silica gel required to compensate for losses range from no additional mass needed to more than doubling the present amount.

Table 2: Summary of theorized causes of capacity loss in silica gel and estimated excess silica gel required to compensate for the losses of explored in this report.

Estimate Case	Basis	Projection – CDRA-4 configuration	Additional SG required to operate 3 years
1)	Whole bed breakthrough, linear loss of SG capacity only	Failure after 9650 HCs	Extra 107%
2)	Treatment of capacity losses split between halves	No failure	None

V. References

[1] Christy, Alfred A. "Effect of heat on the adsorption properties of silica gel." *International Journal of Engineering and Technology* 4.4 (2012): 484.

VI. Acknowledgements

Additional contributors include C. Doug Wingard, Richard Boothe, Timothy Giesy, and Matthew Kayatin.

VII. Executive Summary

Samples from bed teardowns from units used in CDRA-3 and CDRA-4 revealed significant discoloration of the silica gel near the bed inlet as well as a coincidental performance loss. This material was analyzed for the presence of chemical contaminants, physical porosity changes, and adsorption performance. The material characteristics are compared against the location in the bed from which they were sampled in order to develop profiles through the bed. Additional testing of the beds prior to teardown provided more data points. Possible mechanisms for the loss of capacity are provided though no root cause has been found. Extrapolation of the performance loss is used to estimate the required oversizing of the silica gel layer for long-term operation.

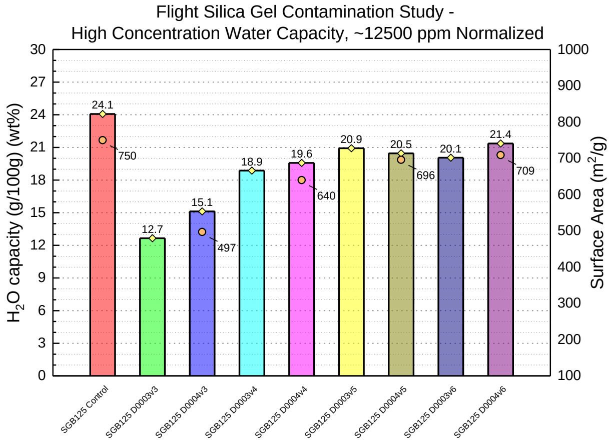

Figure 14 Results of one water vapor adsorption test at high concentrations which closely match the inlet concentration for CDRA. Adsorption was conducted at 25°C and interpolated at 12,500 ppmv.

Chemical composition analysis results from the CDRA-3 samples showed a correlation between siloxane content and loss of performance. Other correlations were noted between excess metal content and performance loss. The chemical composition analysis results from the CDRA-4 samples shows no correlation between siloxane nor metal content and loss of performance. There was no detectable siloxane content on all of the CDRA-4 samples except for a small amount on the Sorbead WS material sourced from D0003. Surface area and pore volume analysis via the BET method showed a consistent trend across sample depth.

In order to probe the effects of adsorbed contaminants in the materials, water vapor isotherms were measured on the available materials. Surface area correlates strongly with capacity. The samples taken from the end of the desiccant bed closest to the cabin air inlet show the greatest loss of performance, while samples taken from positions that correspond to drier sections of the bed which also see hotter temperatures are more similar to control samples. All flight samples show at least a slight reduction in performance against control samples. At high capacities, a nearly linear extrapolation from lower water vapor concentration results is observed. The front of the bed is severely degraded by almost 50% from the control sample while the remainder of the bed shows a 15% to 25% reduction in capacity. This capacity loss may be significant to system operation.

An alternative degradation mechanism is proposed. During desorption cycles of a desiccant bed, a water vapor concentration wave and a hot temperature wave are observed in test and computer simulation. These thermal and concentration waves progress through the bed at different rates with the concentration wave preceding the hot purge wave. This cycles the front layer of silica gel through its entire capacity range during each CDRA cycle. Broken SGB125 beads are observed in the sample vials consistent with exposure to liquid water droplets as observed in controlled misting tests. No sub-scale tests have successfully replicated the discoloration. Sorbead WS is also found to be severely degraded, despite its stability against misting and high pellet strength. The 13X zeolite used in the flight beds was found to be indistinguishable from control samples.

A conservative threshold for drying failure, defined as detectable breakthrough of water vapor, was estimated to occur when the silica gel layer of a CDRA-4 desiccant bed was reduced to 55% of original capacity. Four possible mechanisms are postulated from test data. The estimated additional mass of silica gel required to compensate for losses range from no additional mass needed to more than doubling the present amount. Certainly, more efforts are needed to understand the causes and mitigate risks for long-duration missions.