CO_2 Removal for the International Space Station – 4-Bed Molecular Sieve Material Selection and System Design

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Efforts over the past three years have focused on the study of candidate sorbent materials for use in a 4BMS molecular sieve system. The accumulation of knowledge has been invaluable for further decisions and for reflecting on the conclusions of past decisions. The goal of the next generation CO_2 removal system is continuous, failure-free operation for nearly 20,000 hours, but no complex life support system has yet reached this lofty goal. In addition to reliability, CO_2 removal performance improvements have been intensively studied. The achievements toward this end include highly detailed isotherm measurements which drive system simulations as well as testing physical design improvements. Looking back on the successes and failures of past systems, correlating data from long-duration tests, and carefully projecting future results are all needed for the success of the next system. This work intends to reveal the path we have taken and illuminate the steps to come for CO_2 removal life support with the 4BCO2 flight demonstration.

Nomenclature

4BCO2	=	4BMS Carbon Dioxide Scrubber Flight Demonstration
4BMS	=	4BMS Molecular Sieve
ASRT	=	Allied-Signal Research & Technology
CO_2	=	Carbon Dioxide
CDRA	=	Carbon Dioxide Removal Assembly
EXPRESS	=	EXpedite the PRocessing of Experiments to Space Station
NASA	=	National Aeronautics and Space Administration
MSFC	=	Marshall Space Flight Center
ISS	=	International Space Station
SG	=	Silica Gel
MS	=	Molecular Sieve
mmHg	=	millimeter of mercury pressure
COTS	=	Commercial off the Shelf
SLM	=	Selective Laser Melting
BER	=	Basic EXPRESS Rack

I. Introduction

BASED on Space Policy Directive-1, NASA's stated goal for the agency is to "advance the nation's space program by increasing science activities near and on the Moon and ultimately returning humans to the surface."[1] More recently, NASA's administrator has stated, "We will move forward to the Moon, this time to stay. And then we'll take what we learn on the Moon, and go to Mars."[2] At Marshall Space Flight Center (MSFC), these efforts are focused on producing an International Space Station (ISS) flight demonstration of the next-generation four-bed molecular sieve (4BMS) system known as the 4BMS CO_2 Scrubber (4BCO2).

Among NASA's long term goals is to have a long-duration crewed missions including a three year mission to Mars. Improving life support technologies is critical to ensuring mission success.[3] Existing technologies are insufficient with respect to reliability and performance vs. resource usage. 4BMS technology has operated in a partial

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closed-loop configuration onboard the ISS[4]. 4BCO2 is intended to prove the remaining concerns of reliability and performance have been mitigated.

The 4BCO2 flight demonstration was commissioned to prove that 4BMS technology could be a reliable CO_2 removal system for space exploration missions with no resupply and extremely limited sparing capabilities, such as a Mars Transport mission. The redesign is incorporating numerous changes based on lessons learned from CDRA, ground testing of 4BMS, sorbent characterization tests, and also incorporates chemical processing industry standard practices. The first aspect is the selection of a new sorbent to replace the custom CDRA CO_2 sorbent, ASRT 5A, which can no longer be manufactured. The second aspect is redesign of the components of CDRA to minimize or eliminate causes of sorbent dusting. The third aspect is to integrate state-of-the-art components into the functional test system for lifetime evaluation. This work will provide a brief summary of the work related to these three aspects that led to the decisions being implemented in the 4BCO2 flight demonstration, which is slated to begin operating on the ISS in 2020.

II. Redesign of 4BMS Technology

In 2016, the redesign goals for an exploration 4BMS were presented.[5] Table 1 is a reprint from that work. Nearly every feature described in Table 1 has been implemented in the 4BCO2 flight demonstration. A more detailed description of the analyses conducted in the intervening years and implementation of the design features follows.

Table 1. Exploration 4BMS reconfiguration rationale

Exploration 4BMS Reconfiguration	***Rationale***
Residual desiccant and CO_2 sorbent bed temp reductions	Depending on CO_2 concentration requirements, the desiccant beds may be oversized. In addition to mass and volume reductions, reduced residual desiccant can allow reduced CO_2 sorbent bed heater temperatures. High sorbent temperatures are linked to accelerated dusting.
Alternative high-speed blower	The current CDRA blower is limited to approximately 26 SCFM. An alternate blower with a similar form factor has been identified with considerably higher flow range and potentially less sensitivity to overspeed events. This blower will be tested for performance and durability.
Cylindrical sorbent beds	The CDRA beds have a square cross-section to maximize sorbent volume. As part of the redesign to achieve sorbent containment, some sorbent areas are no longer under spring pressure (used to maintain compaction). Fluidization and accelerated attrition may be the result. Returning to the industry standard of cylindrical sorbent beds insures complete containment and compaction.
Redesigned heater core	Current CDRA bed heaters have experienced multiple failures, and are designed for beds with a square cross section. The heaters will be redesigned for cylindrical beds. Part of the design process is to evaluate other heater types, such as the spiral-wound cartridge heaters used in the CRCS.
Modulated repressurization	Repressurization of the CO_2 sorbent beds is currently unmodulated. Standard industrial practice is to control the rate of a fixed bed pressure change to prevent rapid movement and attrition of the sorbent materials due to an inrush of air. The 4BMS will be reconfigured to modulate repressurization to reduce attrition. If the alternate CO_2 sorbent bed is used as the air source, an additional benefit would be the reduction of air-save vacuum pump power.
CO_2 sorbent bed layering	Zeolites with 2-3 times the CO_2 capacity, and greatly improved kinetics, compared to zeolite 5A have been tested by MSFC recently and have the potential to reduce bed height and increase system efficiency, which becomes more critical with lowered CO_2 levels and higher volumetric flow requirements. However these sorbents will need a protective layer of 5A to prevent water poisoning. Likely failure scenarios must be tested to insure appropriate protection.
Dust resistant valve	The scope of the current valve redesign was limited due to funding constraints. This effort would extend that redesign to protect all seals from dust intrusion. Local additive manufacturing capabilities would be used to fabricate prototypic valves, followed by bench testing and system testing in the ground test 4BMS systems.

A. Sorbent Selection for 4BCO2

Out of 14 candidate CO_2 sorbents obtained from various manufacturers, 2 stand-out candidates emerged as exceptional for attrition resistance: BASF 13X and Grace MS544 C 13X. These two candidates were shown to be robust in both dry and humid environments via long duration hydrothermal stability experiments.[6-8] Between these two, MS544 has flight heritage and was selected on that basis. These two materials were projected to produce dust at one-tenth the rate of ASRT or lower based on the rate of increasing pressure drop in the HST experiment shown in Figure 1.

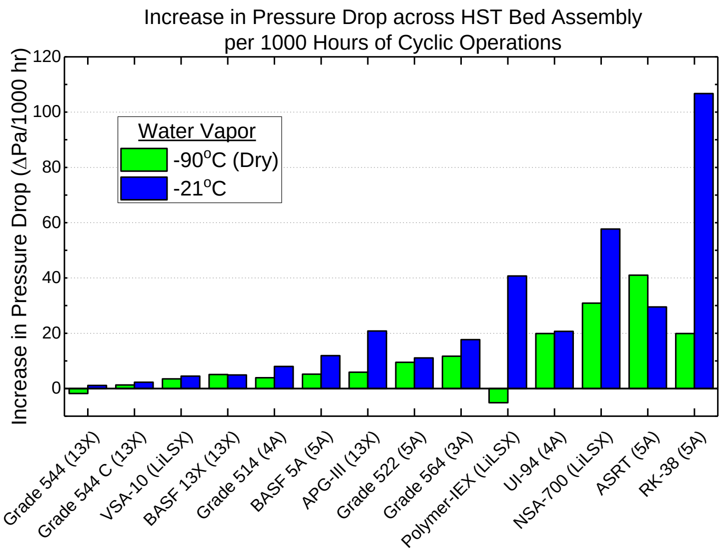

Figure 1. Rate of pressure drop increase which correlates to sorbent dust generation in sub-scale beds under dry and humid conditions subjected to thermal cycling.[5]

Pure component isotherms are shown in Figure 2 and breakthrough capacities were obtained to assess the potential performance of these materials.[9] To characterize desiccants, high fidelity isotherms were obtained for the water vapor adsorption on the bulk desiccant silica gel SG B125 and the residual desiccant zeolite MS544 13X from 25°C to 70°C as well as CO_2 isotherms for MS544 13X from 0°C to 200°C.[10-12] Breakthrough testing at 25°C was used with thermal and equilibrium models to fit linear driving force coefficients.[13] The isotherms were used to derive heats of adsorption.[14]

One of the key lessons learned is that 13X zeolite requires drying at 350°C to fully regenerate the CO_2 capacity. Since the adsorbent bed in CDRA and 4BCO2 is designed to heat only to 200°C, there is a natural concern about incomplete activation and reduced CO_2 removal performance. If sufficient mass of water vapor enters the bed to impair CO_2 capture performance, the thermogravimetric analysis (TGA) work as shown in Figure 3 was used to make the generalized prediction that nominal 4BMS operations would recover the removal performance quickly for 5A zeolites, slowly for 13X zeolites, and not at all for the very high CO_2 capacity LiLSX zeolites.[6] A possible system redesign was considered where the 13X sorbent is heated to 225°C or 250°C to recover nearly all capacity in one heating cycle as shown in Figure 3, but this small increase would require extensive material changes throughout the system. Without a redesign, the TGA testing indicated that repeated activation at 200°C would recover portions of the CO_2 capacity but it was not certain if recovery would be complete. The actual dry air entering 4BCO2 sorbent beds is significantly drier than most bottled gases, thus the expectation is that 4BCO2 will eventually recover full CO_2 capacity at normal operating temperatures.

Both CDRA and 4BCO2 are intended to process the cabin air onboard ISS where it is well established that trace contaminants are prevelant. These contaminants can cause negative performance changes in sorbents and on material surfaces. These trace contaminants were not detected in returned-from-flight zeolite samples, instead these compounds were captured in the layers of silica gel near the inlet of the desiccant beds.[15-17] Silica gel samples collected from flown CDRA beds contained many of these trace contaminants and showed significant performance reduction that seemed to correlate with contaminant concentration. However, causation could not be established, though as discussed later an alternate theory is under consideration. Ultimately, the same silica gel as currently used in CDRA, SG B125, was selected for 4BCO2 due to high attrition resistance and high water vapor capacity.

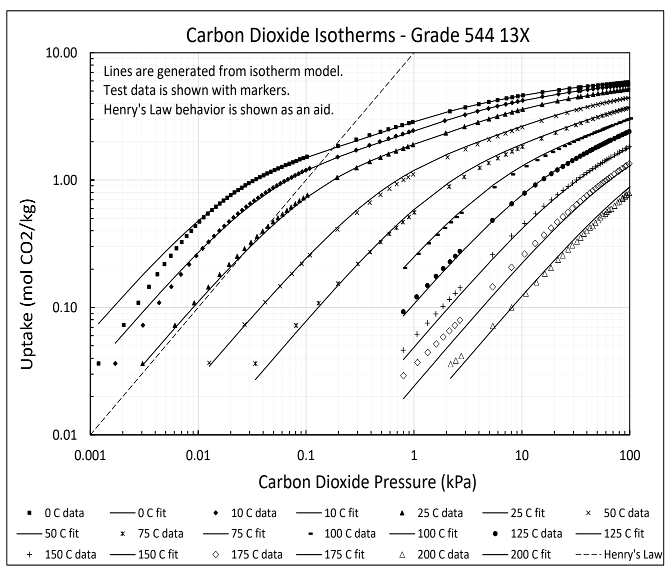

Figure 2. Pure component CO_2 isotherms at temperatures from 0°C up to 200°C.[6]

B. Computer Simulation of 4BCO2

Simulations were extensively used to optimize the design of the new system as well as support ongoing work for future designs.[18-21] Thermal modelling of the heater cores for CDRA and 4BCO2, such as shown in Figure 4, were

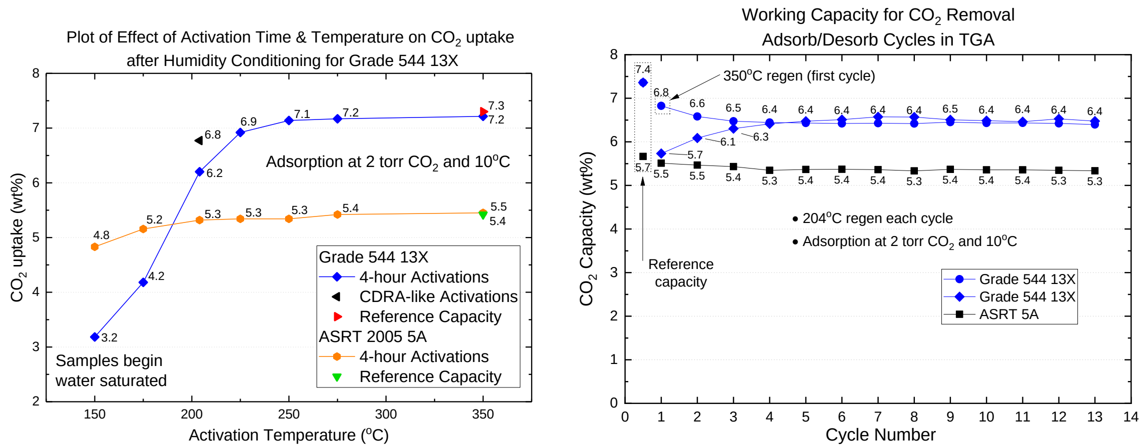

Figure 3. Effect of activation temperature on sorbent performance [left] and simulated 4BMS cyclic operation [right] for ASRT (5A) and Grade 544 (13X). Each point is the CO_2 capacity at end of a single adsorption cycle. Reduced capacity is attributed to trace water vapor in bottled gas supply to TGA.[4]

used to optimize power consumption and heater distribution.[20, 22] The redesigned heater core was simulated using 2-D transient models of sheet heaters, cartridge and fin heaters, and Selective Laser Melting (SLM) 3D printed monoliths. The selected design utilized heat spreading fins which are precision cut using wire Electrical Discharge Machining (EDM) to slide over cartridge heaters.

The 4BMS system simulation is a 1-D transient model of the full-scale system with coupled thermal and material transport and simulation of cyclic operations. Adsorption, particularly water vapor on 13X, can result in very stiff differential equations thus necessitating the one-dimensional approximations to maintain model speed. Simulation results correctly indicated that substantial performance gains could be realized by reducing the desiccant bed zeolite layer where competitive H_2O/CO_2 adsorption occurs. Quantification of the co-adsorption behavior was needed to achieve this optimization via the 4BMS model. CO_2 isotherm measurements with preloaded water on 13X were conducted with a custom instrument designed by Rubotherm, GmbH.[23] This data was used to fit a simplified co-adsorption isotherm that could be used in this model. The 4BMS simulation work produced several improvements, most notably the increased removal rate via reduction of 13X in the desiccant bed and optimization of flow rate, bed size, and cycle time for the case of 2 mmHg CO_2 cabin air.

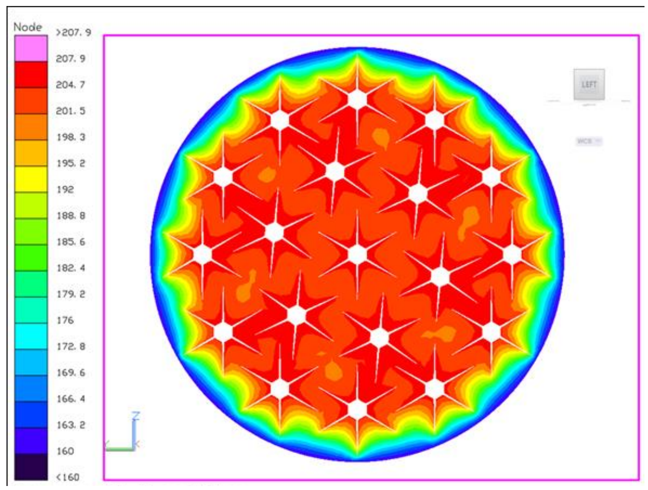

Figure 4. Simulated temperature cross-section of a heating 4BCO2 sorbent bed at the end of a half-cycle.[18]

Figure 5. Pleated filter element and filter slide designed for use in 4BCO2. Pleating increases dust capacity and reduces pressure drop.

C. System Design of 4BCO2

The redesign of 4BMS technology to eliminate failures due to sorbent dusting included addressing every known and potential cause of dusting acquired over years of 4BMS operations and from industry.[5] Besides addressing the dusting propensity of the sorbent itself via the sorbent selection process, numerous changes were made to the design, assembly, and operation of 4BCO2. The most apparent change is the switch from rectangular sheet metal beds to cylindrical beds which eliminates the wall deflection that results from vacuum cycling which causes sorbent movement and attrition. Cylinders also enable more reliable sorbent containment and compaction with internal spring-loaded plates. A maintainable, pleated filter element shown in Figure 5 was procured from Porvair Filtration Group Inc and integrated into the system to capture dust at the location which caused repeated dust-related failures in CDRA.

The second major change is to the heater core which in CDRA was a monolithic assembly within the sorbent beds using sheet heaters and aluminum to distribute heat. For bed packing, this assembly lead to obstructed views and no information on packing efficiency. The 4BCO2 heater core is designed with assembly in mind resulting in cartridge heaters with aluminum heater spreading fins cantilevered from a fixed support plate with clear lines of sight to closely monitor the packing process as shown in Figure 6. The packing process also utilizes a technique known as “snow storm” which effectively distributes sorbent beads and decreased void volumes.[24] These design and process changes nearly eliminate potential void spaces where dust would be generated due to attrition of moving sorbent particles.

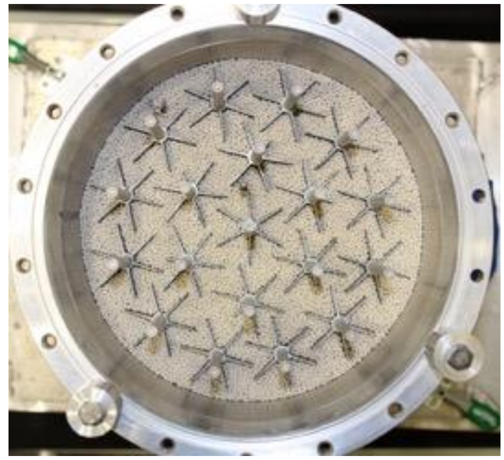

Figure 6. Partially filled sorbent bed for the ‘Linus’ prototype 4BCO2 system.

Several other changes were made to specific components and operating behaviors were changed from CDRA to 4BCO2. New valves are being designed at MSFC and initial testing shows very high tolerance to dust and equivalent operating lifetimes in excess of 9 years. These new valves have a small port to allow modulated or choked repressurization of the sorbent bed during half-cycle transitions as shown in Figure 7. Excessive air velocities generated during rapid repressurization with conventional valves are identified as a source of attrition due to the movement of particles. The new valves keep the velocity from exceeding the limit accepted in the chemical industry for attrition avoidance.[25] These new valves can also isolate the flow path ensuring leak tightness when necessary.

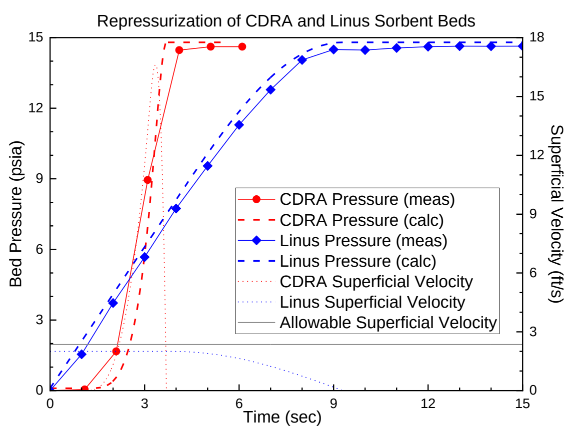

Figure 7. Measured repressurization profile with commercial ball valves and with the MSFC valve along with the maximum allowable superficial air velocity.

D. Operation of the 4BCO2 Prototype

The prototype 4BMS system for the 4BCO2 project is colloquially known as ‘Linus’ and is currently operating at MSFC. Linus is being used to map the performance envelope of the integrated hardware with regard to CO_2 removal and power consumption. In addition, Linus is being used to conduct specific tests of new hardware as it is integrated, refine the computer model, and expose any unforeseen interactions of the numerous changes. The instrumentation for Linus includes additional pressure, temperature, CO_2, and water vapor sampling.

Several positive changes have already been observed including improved cooling performance of the new precooler, reduced peak temperature of the outlet air, reduced dusting, reduced CO_2 holdup in the desiccant beds, and reduced overall bed mass. The precooler is a microtube heat-exchanger built by Mezzo Technologies which removes the heat generated by desiccation and from the air blower in order to improve the CO_2 capacity of the sorbent bed. One negative change has been a higher pressure drop through the desiccant beds versus the CDRA-like ground testbed. In addition to a smaller bed cross-sectional area, the improved packing procedures may have reduced the void fraction in the bed resulting in a higher pressure drop. To offset this pressure drop the airflow ducts with mitered elbows used in CDRA were replaced with optimal airflow ducts printed with metal Selective Laser Melting (SLM) 3D printing technology.

Several other components are being implemented in the design of 4BCO2 utilizing 3D printing (SLM) technologies. Nearly all air ducts inside of the system will utilize SLM production. The heater support plate would be a significant thermal leakage pathway if it was machined from aluminum stock. Instead, SLM enables the heater plate to be manufactured from Inconel or Titanium while remaining structurally robust, retaining a high open fraction for airflow, and having tortuous heat conduction paths.

Initial operation of Linus showed significant underperformance versus the 4.16 kg/day requirement as shown in Figure 8. It is unavoidable that sorbent will be exposed to ambient humidity during packing procedures. Subsequent bake out with facility dry N_2 purge removes most but not all water vapor. The previously discussed TGA testing of the CO_2 sorbent predicted this underperformance and also predicted that the performance would recover while conducting nominal operating cycles. The CO_2 removal performance did increase over the first few weeks of nominal operation, eventually achieving a steady-state close to predictions from the simulations. This indicates that, in the event of a water exposure event, normal operation alone can restore the system and no high temperature bakeouts or other risky actions are needed.

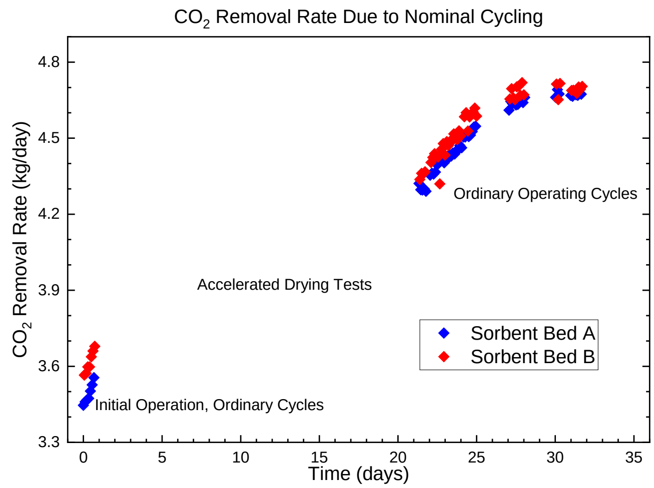

Figure 8. Recovery of CO_2 removal performance after initial assembly which had exposed dry sorbent to ambient humidity. The CO_2 removal was impaired but increased after numerous operating cycles to a steady-state.

One of the remaining unknown behaviors of CDRA was the cause of silica gel discoloration and performance degradation after many operating cycles. The possible causes of this silica gel degradation have been investigated extensively without a conclusive answer.[15, 17, 26] One theory is that it resulted from the desorption process which uses the heat stored in the thermally desorbed CO_2 sorbent bed to provide the driving force for water desorption in the desiccant bed. This is a fundamental function of 4BMS technologies, however due to the size and shape of the CDRA beds it lead to a condensing condition, which is shown in Figure 9 near the desiccant bed inlet. This location correlates to the position of most extreme silica gel degradation. Serendipitously, Linus does not exhibit the condensing conditions which were observed in CDRA and similar 4BMS testbeds. The smaller sorbent bed, lighter heater core, higher air flowrates, and shorter half-cycles all contribute to preventing the condensing conditions.

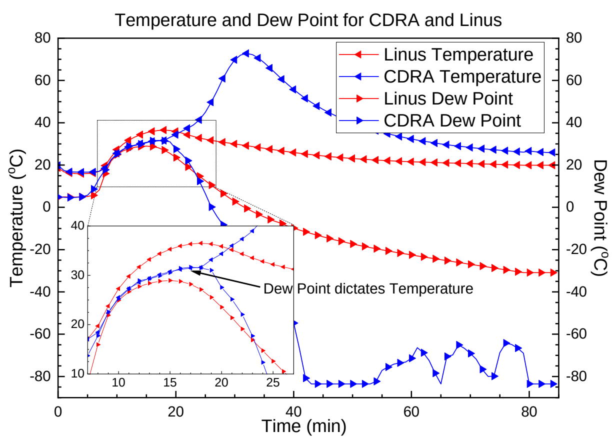

Figure 9. Temperature and dew point overlap indicating condensing conditions in the desorbing desiccant bed for the CDRA-like testbed whereas Linus does not exhibit this behavior.

Linus has accumulated over 1800 hours of normal operating time. The system has been inspected for dusting at two points to-date. The first inspection was conducted after 800 hours and traces of dust were found on the new pleated filter element and slide but no observable dust was found coating the metal surfaces outside the bed filters. The Linus sorbent beds both settled 2-3 mm which was attributed to residual compaction near the surfaces of the fins. Sorbent attrition and mass loss as dust was unlikely since the displaced volume exceeded the observable dust by orders of magnitude.

After the next 1000 hours, the filters were again removed and the beds inspected. The plates each travelled 1mm farther and 0.01(1) gm of dust was captured from each filter element, pictured in Figure 10. Overall, the measurable mass of dust per time is roughly 1% of ASRT from CDRA test, teardown, and evaluation measurements. This supports the premise that the dusting problem has been effectively solved.

E. Analysis and Data Reduction from Linus Testing

As previously discussed, over 1800 hours of testing has been conducted. The core test plans were designed to determine the performance envelope of the system. The primary factors affecting CO_2 removal rate which can be explicitly controlled are total flowrate, cycle time, and temperature setpoints. Implicit factors that depend on integrated operation onboard the ISS include: inlet CO_2 partial pressure, CO_2 capture efficiency, inlet temperature and dew point, cooling water temperature, and several sources of pressure drop. Testing with the Linus prototype system allows all of these factors to be explored in detail.

The first test series utilized components that closely matched CDRA and its related testbeds to provide the baseline performance. The system flowrate, inlet CO_2 concentration, and half-cycle time can be normalized to mass per half-cycle. Figure 11 shows the initial performance map highlighting the two primary effectors for varying system performance. Changing half-cycle time hardly changes the removal rate of the system whereas flowrate has a nearly linear relationship to removal rate. The dozens of other data points come from tests of the explicit factors such as inlet CO_2 partial pressure, cooling water, and/or vacuum.

Figure 10. Dust recovered after 1000 hours of operation from Linus removable filters (also pictured). The black trays provide an enhanced visual contrast to view the dust.

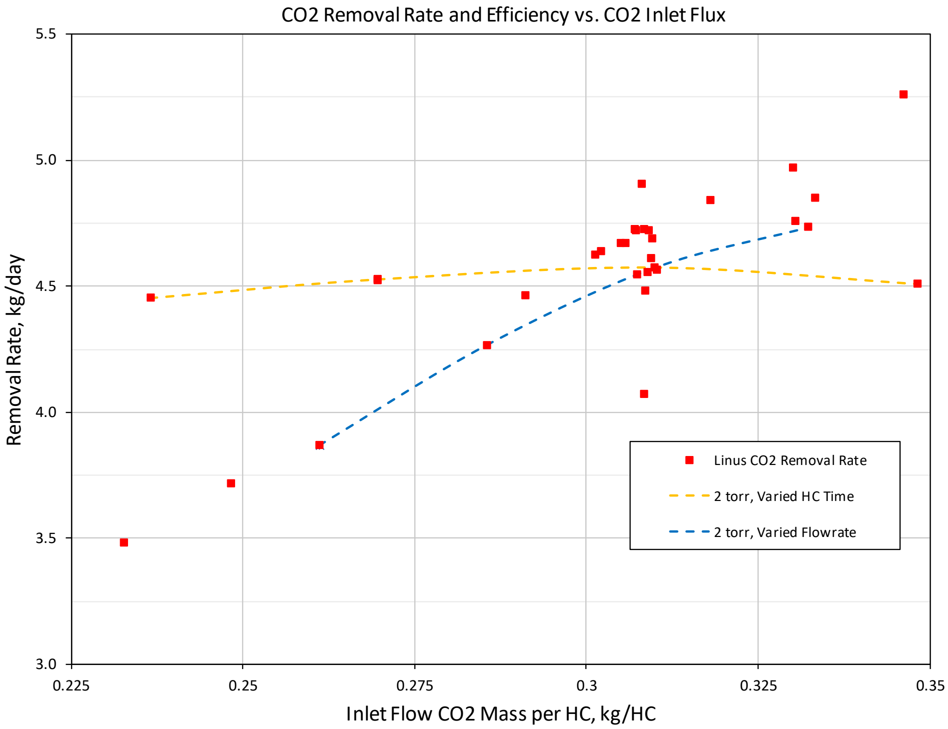

Figure 11. Performance map of Linus, the 4BCO2 prototype. Blue and yellow dashed lines connect two selected test series where in each a single factor is varied.

Based on the simulations and accumulated knowledge of CO_2 removal experts, several performance gains were theorized and then tested with the Linus testbed. One theory is that a thermal soak of the sorbent for a longer period above some essential desorption temperature will improve removal efficiency. Improved performance was realized with an increased power input to the heaters which increased bed temperature more rapidly.

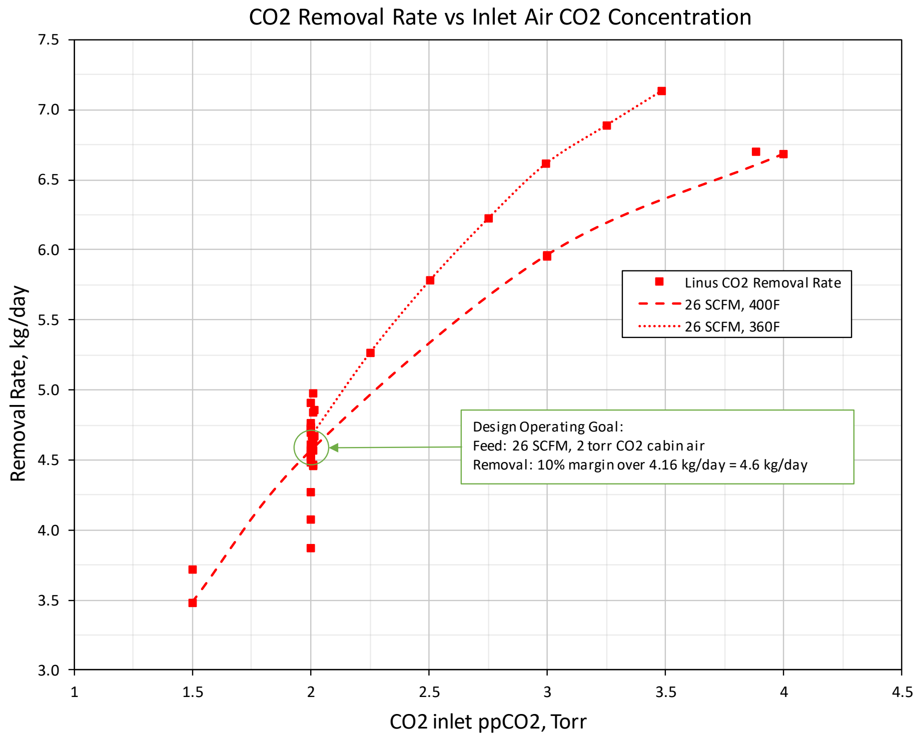

Figure 12. Performance map of Linus, the 4BCO2 prototype. Red dashed lines show two similar test series where the heater setpoint was set to 360°F and 400°F.

A second theory is the counter-intuitive reduction of sorbent bed regeneration temperature from 400°F to 360°F resulting in an increase CO_2 removal rate with results shown in Figure 12. The zeolite layer in the desiccant bed, which is necessary to remove nearly all residual water vapor, is also active as a CO_2 sorbent. Since the desiccant bed cannot be isolated to vacuum, the CO_2 held-up in this bed is denied to the sorbent bed and instead returned to cabin on the following half-cycle. Reducing sorbent bed temperature leaves more water vapor in the zeolite layer of the desiccant bed. The competitive adsorption between H_2O and CO_2 results in more CO_2 entering the sorbent bed, higher efficiency, and a higher removal rate. A plot combining all the discussed aspects in shown in Figure 13.

Naturally, the risk of operating with reduced temperature is insufficient drying. Any water breakthrough can impair downstream systems. Linus and 4BCO2 include a dew point sensor downstream of the desiccant beds to directly monitor the desiccated air for water vapor breakthrough. Further work with the Linus testbed can be used to guide the performance optimization of 4BCO2.

III. Conclusions

A. Conclusions for 4BMS Technology and 4BCO2

The primary directive for the 4BCO2 flight demonstration is to prove 4BMS technology can operate reliably and that sorbent dusting would not lead to shutdowns or require anything but brief maintenance. The results obtained to date from Linus strongly suggest that dusting has been solved and that 4BMS technology would be reliable for long-duration missions. The risk of silica gel degradation may also have been solved, albeit unintentionally, further ensuring long-term reliability. Ongoing work with the Linus testbed will enable the 4BCO2 system to experience performance gains after installation on board the ISS.

Operation of the Linus testbed has created a performance map at various conditions at and around the design point of 4 crew-equivalent CO_2 removal rate at 2 torr CO_2 cabin air. Higher removal rates can be achieved at higher CO_2 concentrations or by consuming more power to reduce half-cycle time and/or increase airflow. 4BCO2 can potentially return air directly to cabin which would reduce pressure drop and improve performance.

While the performance outlook for 4BCO2 is depicted as rosy, the system mass is not optimized as a compromise for an accelerated delivery. Also, 4BCO2 is required to be modified on station where it will integrate into both a Basic

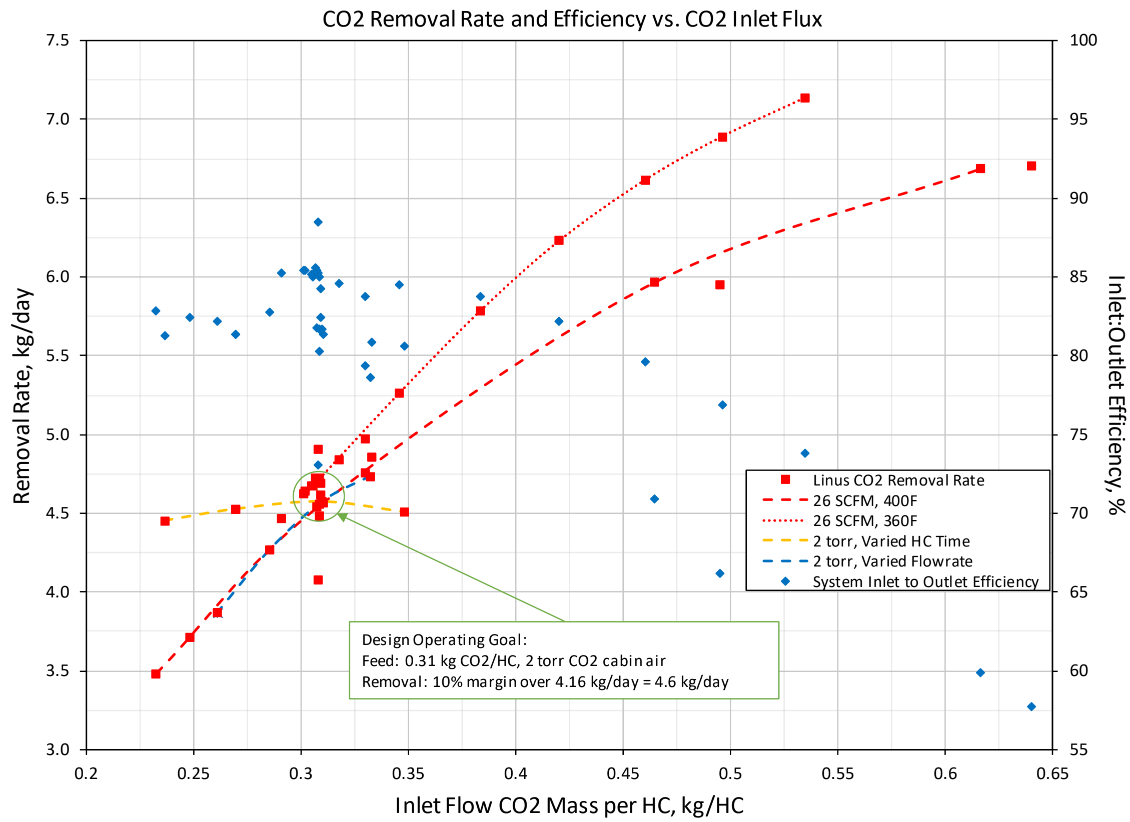

Figure 13. Performance map of Linus, the 4BCO2 prototype. Blue and yellow dashed lines connect two selected test series where in each a single factor is varied. Red dashed lines show two similar test series where the heater setpoint was set to 360°F and 400°F.

EXPRESS Rack (BER) as well as the Air Revitalization (AR) rack, should it be selected to replace CDRA. The system is also slated to be a testbed for a next-generation air blower and controller which will require significant on-orbit installation work.

B. Future Work for 4BMS Technology and 4BCO2

Upcoming work with the Linus testbed includes mapping the performance envelope for directly drawing inlet air from the cabin instead of conditioned air from module ventilation. Additionally, the improved water heat exchanger may be able to operate at reduced water flowrates. Further work can optimize the half cycles with regard to air save pump operation, heaters, and blower flowrate. Closed loop operation will also be tested to evaluate the effects on performance from the partial or complete loss of access to space vacuum desorption.

Numerous improvements are slated to follow the launch of 4BCO2 including: volume optimizations of the new valves, mass reductions and structural optimizations, improved electronics cooling, and noise mitigation features. Although scheduling will not allow these improvements to be implemented in the initial 4BCO2 flight demonstration, they would likely be part of designs for exploration or lunar missions should 4BMS technology continue to be selected. Software routines have been envisioned which would increase autonomy, minimize the need for operator attention, and be able to adapt to changing cabin air conditions.

Acknowledgments

The authors of this work would like to acknowledge the incredible efforts from the core adsorption science team to the 4BCO2 project management and everyone in between.

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