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Sorbent Structural Impacts due to Humidity on Carbon Dioxide Removal Sorbents for Advanced Exploration Systems

David Watson[1], James C. Knox [2], Phillip West[3],
NASA Marshall Space Flight Center, Huntsville, Alabama, 35812, USA

Christine M. Stanley[4] and Richard Bush.[5]
Jacobs ESSSA Group, Huntsville, Alabama, 35812, USA

The Life Support Systems Project (LSSP) under the Advanced Exploration Systems (AES) program builds upon the work performed under the AES Atmosphere Resource Recovery and Environmental Monitoring (ARREM) project focusing on the numerous technology development areas. The CO_2 removal and associated air drying development efforts are focused on improving the current state-of-the-art system on the International Space Station (ISS) utilizing fixed beds of sorbent pellets by seeking more robust pelletized sorbents, evaluating structured sorbents, and examining alternate bed configurations to improve system efficiency and reliability. A component of the CO_2 removal effort encompasses structural stability testing of existing and emerging sorbents. Testing will be performed on dry sorbents and sorbents that have been conditioned to three humidity levels. This paper describes the sorbent structural stability screening efforts in support of the LSS Project within the AES Program.

Nomenclature

MSFC = Marshall Space Flight Center
ISS = International Space Station
CDRA = Carbon Dioxide Removal Assembly
MTF = Mechanical Test Facility
HCS = Humidity Conditioning Test Stand
AES = Advanced Exploration Systems
ARREM = Atmosphere Resource Recovery and Environmental Monitoring
LSSP = Life Support Systems Project

I. Introduction

The Carbon Dioxide Removal Assembly (CDRA) was installed on the ISS in early 2001. Although it has provided the bulk of the CO_2 removal function for the ISS since that time, numerous equipment problems have arisen that required attention. Failed items such as valves and heater sheets have been redesigned or are currently undergoing redesigns that should resolve those problems for future applications. However there are also more persistent issues with ramifications for future spacecraft designs.

Zeolites currently used for capture of CO_2 are agglomerates of zeolite crystals and (typically) clay binder. The roughly cubic crystals (approximately 2 μm per side) are bound into spherical or cylindrical pellets (approximately 2

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[1] Mechanical Engineer, Environmental Control and Life Support Development Branch/ES62,
[2] Aerospace Engineer, Environmental Control and Life Support Development Branch/ES62.
[3] Mechanical Engineer, Environmental Control and Life Support Development Branch/ES62,
[4] Chemical Engineer, Environmental Control and Life Support Development Branch/ES62,
[5] Engineer, Materials Test Branch/EM10

mm in diameter the CDRA application). Pelletization with clay binder is required to reduce flow resistance through the fixed bed. An open structure inside the pellet is required to maximize CO_2 mass transfer into the pellet. However this open composite structure tends to have low resistance to attrition and is may be weakened by humidity and/or large temperature excursions. The pellet fragments resulting from attrition can migrate to the mesh screen retaining the sorbent, partially blocking the flow path and increasing differential pressure. Blower speed can be increased to regain the flow rate required for CO_2 removal performance, but at the cost of additional power. When screen blockage and the resulting flow restriction increases above equipment limits, replacement of the fixed bed or cleaning of the retention screen is required. The current ISS CDRA design includes features for screen replacement, however this requires considerable astronaut time which is highly undesirable. For future spacecraft, the vehicle architecture may not allow easy access to equipment as on the ISS, such that maintenance may become even less desirable.

A continuing effort to screen and characterize sorbents has been undertaken as part of the LSSP. Simple tests to screen existing and emerging sorbents for structural stability and working capacity can quickly identify sorbents with the highest potential. More detailed structural stability testing may then be conducted on selected sorbents. Previous work in the area of sorbent screening included pellet crush and bulk crush testing of dry sorbents and is documented in Ref 1 and Ref 2.

Dust production and sorbent fracturing are undesireable for packed bed applications on long term missions. The resulting fines eventually clog the screens and reduce air flow below that required to remove CO_2. The International Space Station (ISS) Carbon Dioxide Removal Assembly (CDRA) has shown a rapid increase in the differential pressure across one of the two CO_2 sorbent beds since replacement of the ASRT 5A zeolite with UOP RK-38 5A zeolite. A possible contributing factor for this rapid increase is introduction of humidity to the 5A material due to leakage or from other causes.[3] Recognizing the mechanisms for dust production and sorbent fracturing are important factors when selecting sorbent materials. As noted in earlier work[2], crush strength differs between dry and humidified sorbents. In order to provide a comprehensive comparison between candidiate sorbents, evaluation of bulk and pellet crush strength and attrition characteristics are necessary under both dry and humid conditions. A Humidity Conditioning Test Stand (HCS) was developed to enable conditioning the samples to differing humidities. Using the HCS will support these experiments to provide insight to the influence of adsorbed water on the structural integrity of sorbents. All of the tests discussed in this paper were performed at approximately -90 C dewpoint and at sorbent conditioned dewpoints of -21 and -14 °C (7.25E-05, 0.7, and 1.38 mmHg Inlet Vapor Pressure, respectively).

II. Sorbent Humidity Conditioning

A Humidity Conditioning Test Stand (HCS) was developed to humidify sorbent samples at the required dew point for testing. The schematic for the test stand is shown in Figure 1. A dry N_2 gas and a mixture of water vapor and N_2 gas are combined to produce a humid gas flow at the specified dew point prior to entering the process. The

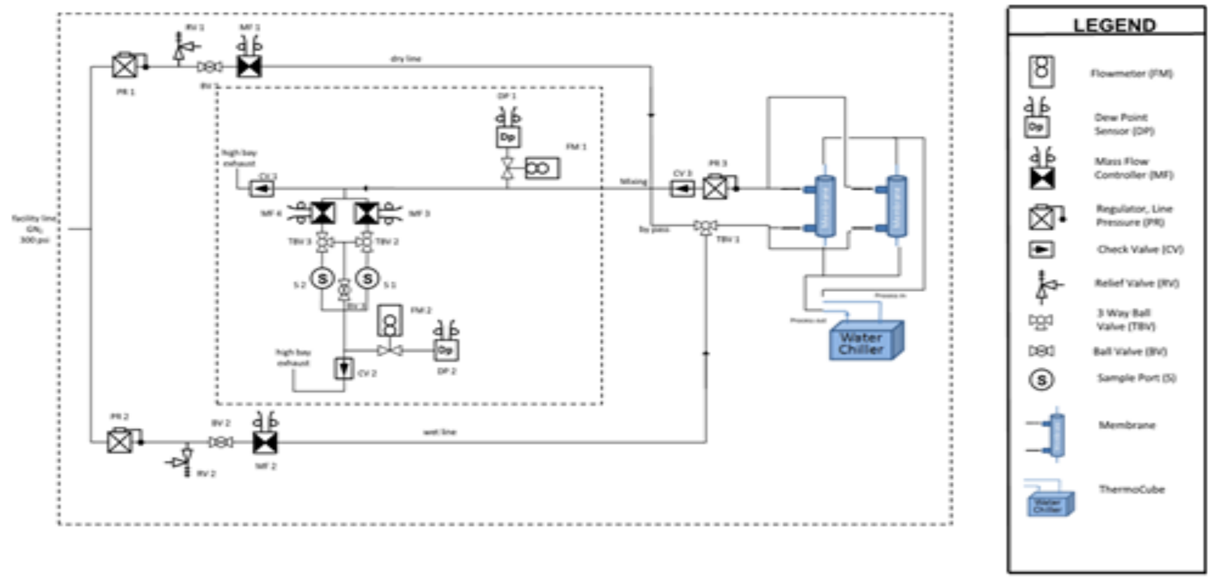

Figure 1. Humidity Conditioning Test Stand.

mixed gas flow enters the inlet dew point sensor and by-passes the sample canisters, exiting through an outlet dewpoint sensor. Once the inlet dew point sensor reaches the desired dew point, flow is redirected through the sample canisters. When the outlet dew point is equal to the inlet dew point, conditioning is complete.

III. Sorbent Structural Stability screening

A. Bulk Crush Testing

The bulk crush strength test outlined in ASTM Standard D7084-04, Standard Test Method for Detemination of Bulk Crush Strength of Catalysts and Catalyst Carriers, provides a method of evaluating the bulk crush strength of a bed of sorbent to determine the ability of the sorbent material to maintain its physical integrity. The bulk crush test differs from the pellet crush strength by better representing how a load applied to a packed bed can damage the sorbent material. Bulk crush testing of flight and non-flight lots of RK-38 and of ASRT 1995 were performed in accordance with the standard, with each sorbent conditioned at -90°C (dry), -21°C, and -14°C dewpoints.

1. Experimental

A Bulk Crush Test Cell was built as per the ASTM Standard, shown in Figure 2. The amount of each sorbent required to fill the test cell was determined pre-test, according to the Standard. Each sorbent tested was activated according to ES62-TCP-SORB-14-001, Test and Checkout Procedure for Sorbent Activation, transferred to a nitrogen-purged glovebox, and the required mass for testing was weighed from the sample. If required for a humidity-conditioned test, the sorbent was conditioned on the Humidity Conditioning Test stand (HCT) to the desired dewpoint and returned to the glovebox. Inside the glovebox, the test cell was packed with the sorbent sample using a snowstorm apparatus. The snowstorm is a cylinder with a mesh of fine wire across the opening that fits in the opening of the test fixture. The wire mesh helps to efficiently distribute the pellets as they are poured into the cell. The use of a snowstorm packing apparatus is a modification to the standard and was used to increase the density of material in the packed cell and to better simulate a bed that was packed using the snowstorm packing method typically used in beds that are prepared for life support processes. The test cell was subjected to various pre-determined crush loads for 30 seconds by MFSC's Mechanical Test Facility's (MTF) Instron 5569 electro-mechanical test frame. The sample was sieved to separate the dust from the remaining pellets. To increase the accuracy of the results, as well as to reduce the impact of the humid environment present at Marshall Space Flight Center, and insure repeatability, the pellets were re-activated using ES62-TCP-SORB-14-001 for the bulk material. The fines were dried by a Quantachrome MasterPrep vacuum degasser. The pellets and dry fines were weighed and the dry percent fines calculated.

As per the Standard, three runs were done to produce fines < 1%, and three to produce > 1% fines. The data points were fitted to a straight line and the fit was used to determine the crush load for 1% fines.

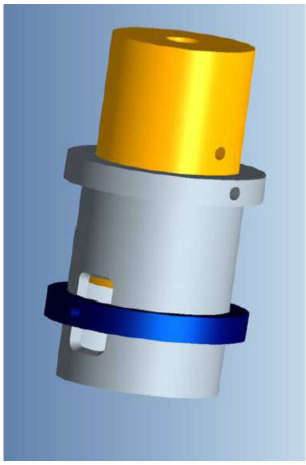

Figure 2. Bulk Crush Strength Test Fixture

2. *Results and discussion*

Figures 3, 4, and 5 show the test results of the three sorbents, ASRT 1995, a non-flight lot of RK-38 and the flight lot of RK-38. The flight lot of RK-38 showed slightly greater crush load required to produce 1% fines at all humidity levels than the non-flight lot of RK-38. At humidity levels above -90°C, though, the ASRT showed greater crush resistance than either sample of RK-38. Table 1 shows the crush force, in lbf, required to produce 1% fines for all three sorbents at all test dewpoints.

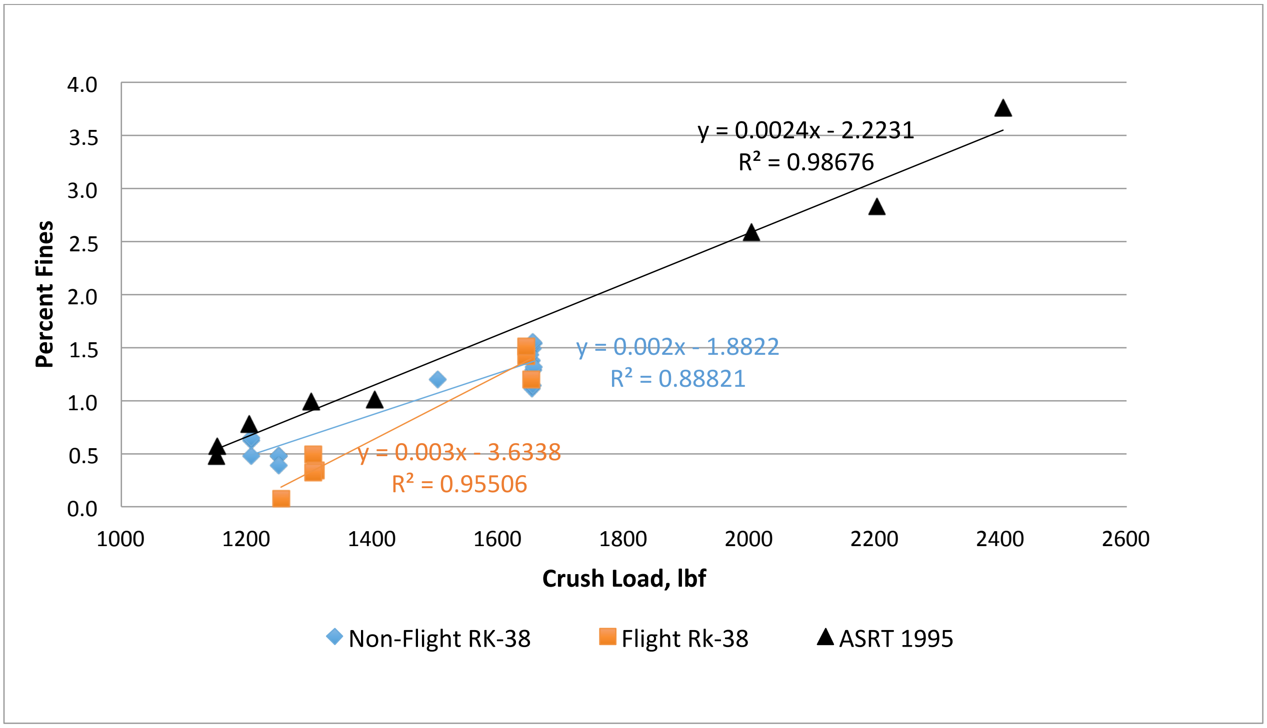

Figure 3. Percent Fines at -90°C Dewpoint

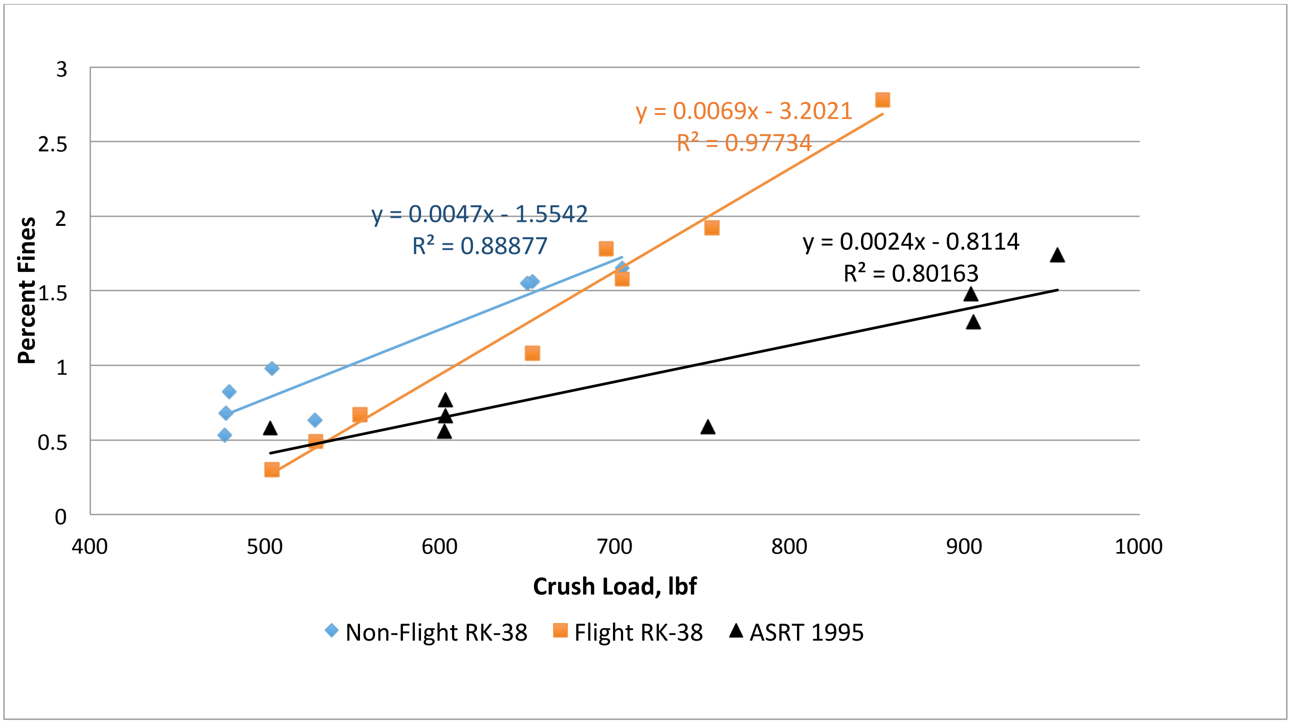

Figure 4. Percent Fines at -21°C Dewpoint

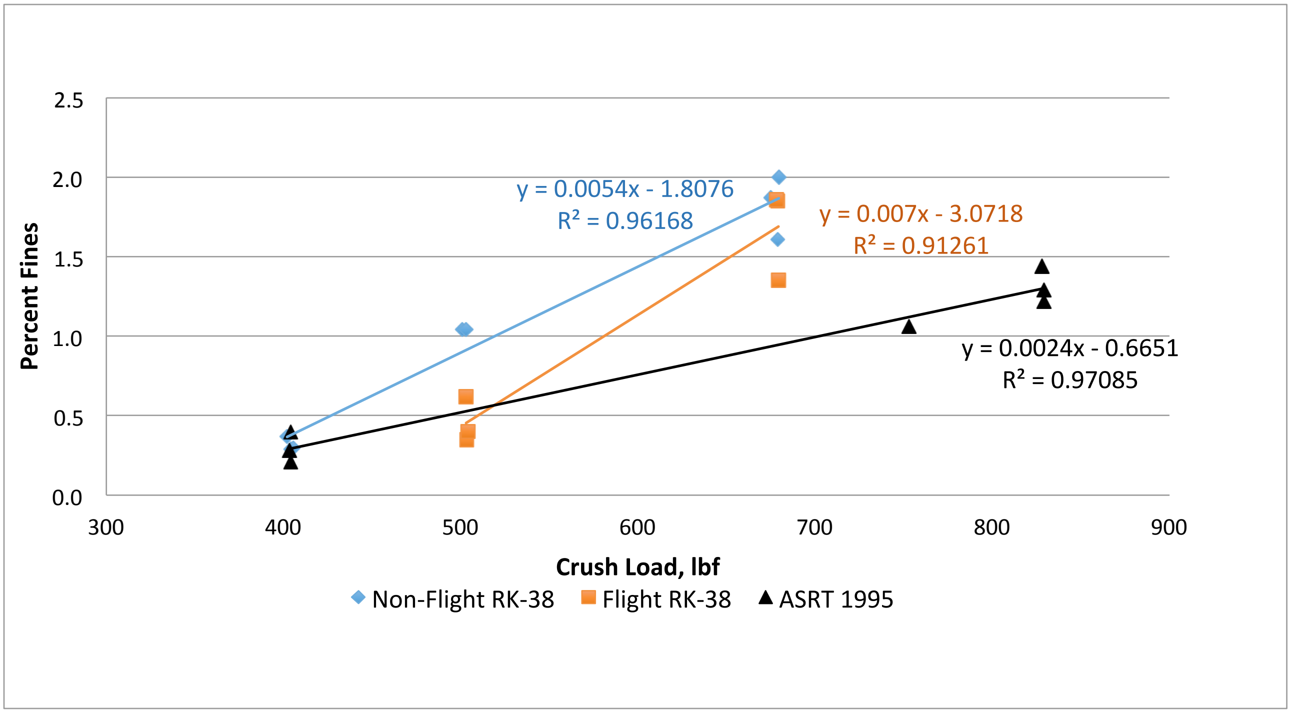

Figure 5. Percent Fines at -14°C Dewpoint

Table 1. Crush Force for 1% Fines

Dewpoint	Vapor Pressure, mmHg	ASRT 1995	RK-38 Flight	RK-38 Non-Flight
		Crush Load, lbf		
-90C	7.25E-05	1342.96	1544.60	1441.10
-21C	0.7	754.75	609.00	543.45
-14C	1.38	693.79	581.69	519.93

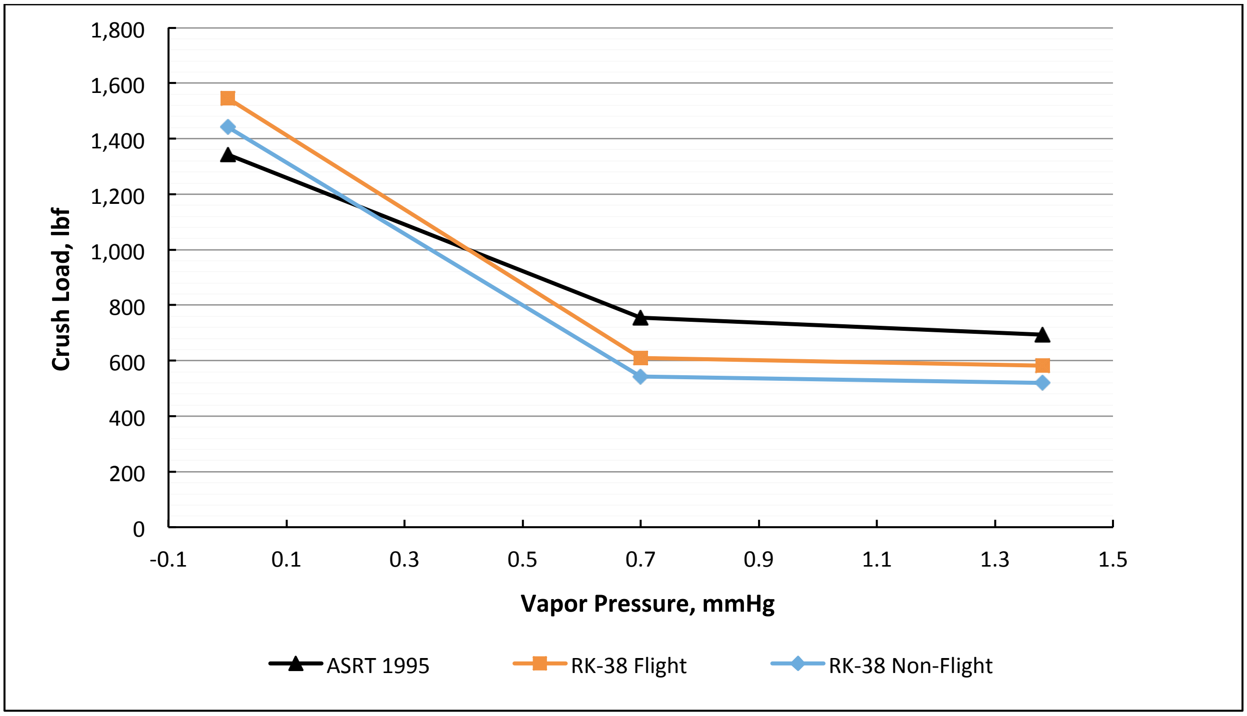

Figure 6. Bulk Crush Load for 1% Fines vs. Vapor Pressure

B. Single Pellet Crush Testing

1. Experimental

The Marshall Space Flight Center's MTF Instron 5569 electro-mechanical test frame was used to perform single pellet crush testing of various sorbent materials to gain information regarding the ability of a material to retain physical integrity during use. Tests were performed on pellet samples conditioned at all three dewpoints used in Bulk Crush and Attrition tests. This testing was performed in accordance with the ASTM standard D4179-11, Standard Test Method for Single Pellet Crush Strength of Formed Catalysts and Catalyst Carriers and, in modified form, D6175-03, Standard Test Method for Radial Crush Strength of Extruded Catalyst and Catalyst Carrier Particles. D6175-03 calls for reporting the crush force per unit length of an extruded sample, but for these tests, the sample length was not taken into account since in a packed sorbent bed the sizes of the extruded sorbents vary widely. It was assumed that the average crush strength measured by the testing of various sized pellets will correlate with bulk crush behavior.

The test apparatus and procedure is described in greater detail in Ref. 1 and shown schematically in Figure 7. One enhancement to the system is the addition of a small environmental control system that allows the sorbents to be stored in a humidity-controlled container before and during crush testing. The system consists of a dry nitrogen source that flows nitrogen directly through the container to maintain dryness of the sorbents, or through a dewpoint generator set to control the container dewpoint to the same as the conditioned sorbent. This insures that the sorbents remain at the proper conditioning, providing more accurate results. The design of the system is shown in Figure 8. In addition, in order to reduce variability due to additional adsorbed moisture, the following steps are taken at the MTF for testing of all samples:

1) Exposure of a pellet to the room air is limited to a maximum of one minute.
2) The platen is heated to 165 °F during testing.
3) A dry air purge is directed across the platen and pellet during testing.

Individual pellets from a representative sample of fifty pellets are crushed one at a time between two flat platens. Video capture was used to establish a dusting load criteria and a catastrophic event criteria for each sorbent. A load/displacement curve was generated for each test and the dusting load and the maximum load of the test was recorded. All dusting loads and maxium loads for the sample were averaged. To insure that the maximum loads being recorded were at catastrophic failure and not at points of limited fracturing, video confirmation was obtained for several test samples.

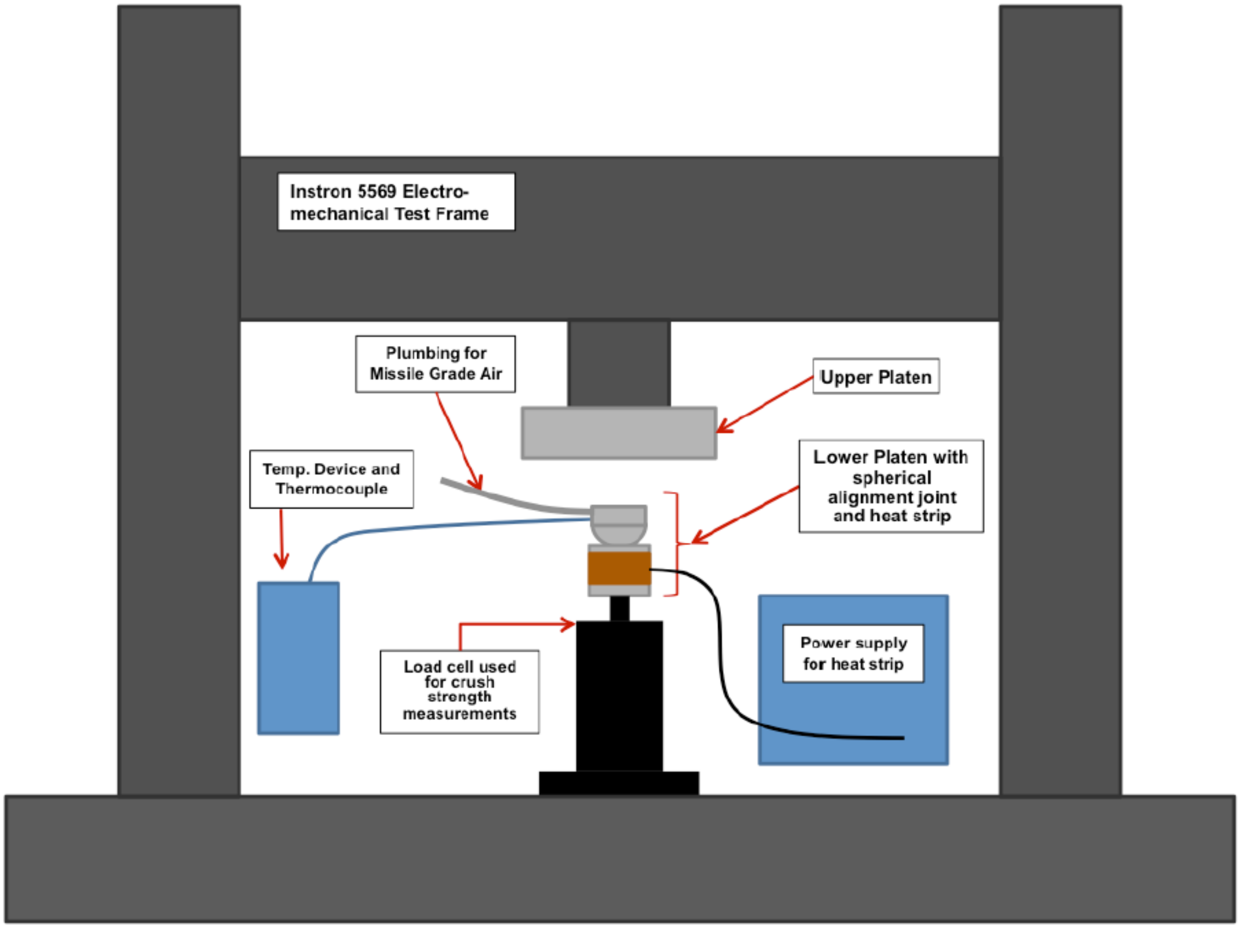

Figure 7. Apparatus for Pellet Crush Strength Testing

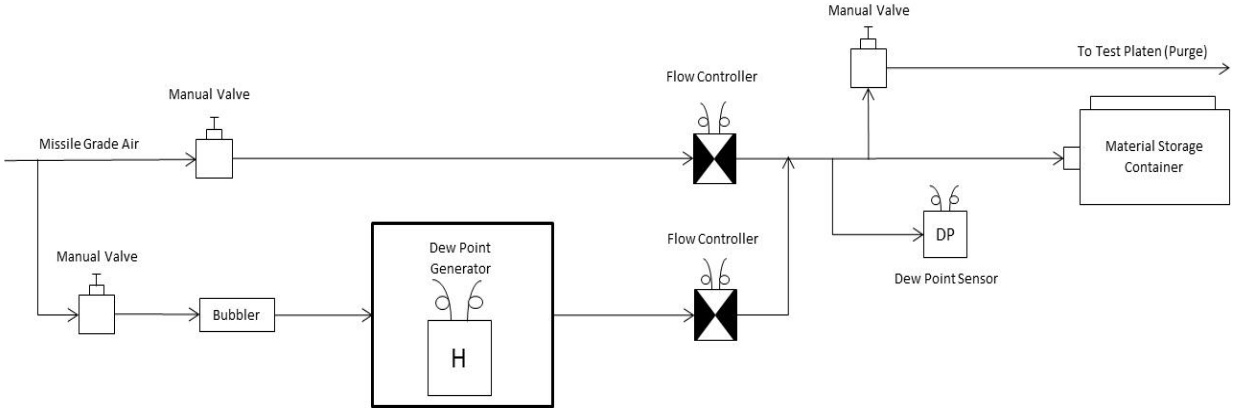

Figure 8. Schematic of EM10 test stand to maintain sorbent material conditioning for humid single pellet crush testing.

2. *Results and discussion*

Figure 9 shows the results of single pellet crush tests on ASRT 1995, a flight lot of RK-38, and a non-flight lot of RK-38. These tests showed that, at the dry condition, ASRT 1995 required slightly more load to initiate breakage than either of the RK-38 samples, with the non-flight lot of RK-38 showing greater crush resistance than the flight lot. At the wet conditions, the RK-38 lots were similar in crush resistance, with the ASRT still showing greater crush resistance than either. The results at the dry condition are inconsistent with the bulk crush results, which showed ASRT 1995 as the weakest sorbent and the Flight RK-38 as the strongest. Although pellet crush tests are preferred in that they consume less time and material, a means to directly compare results with pellets (cylindrical) and beads (spherical) particles has not been established. Until such a correlation has been developed, bulk crush tests are recommended since they provide an environment closer to the actual system operation.

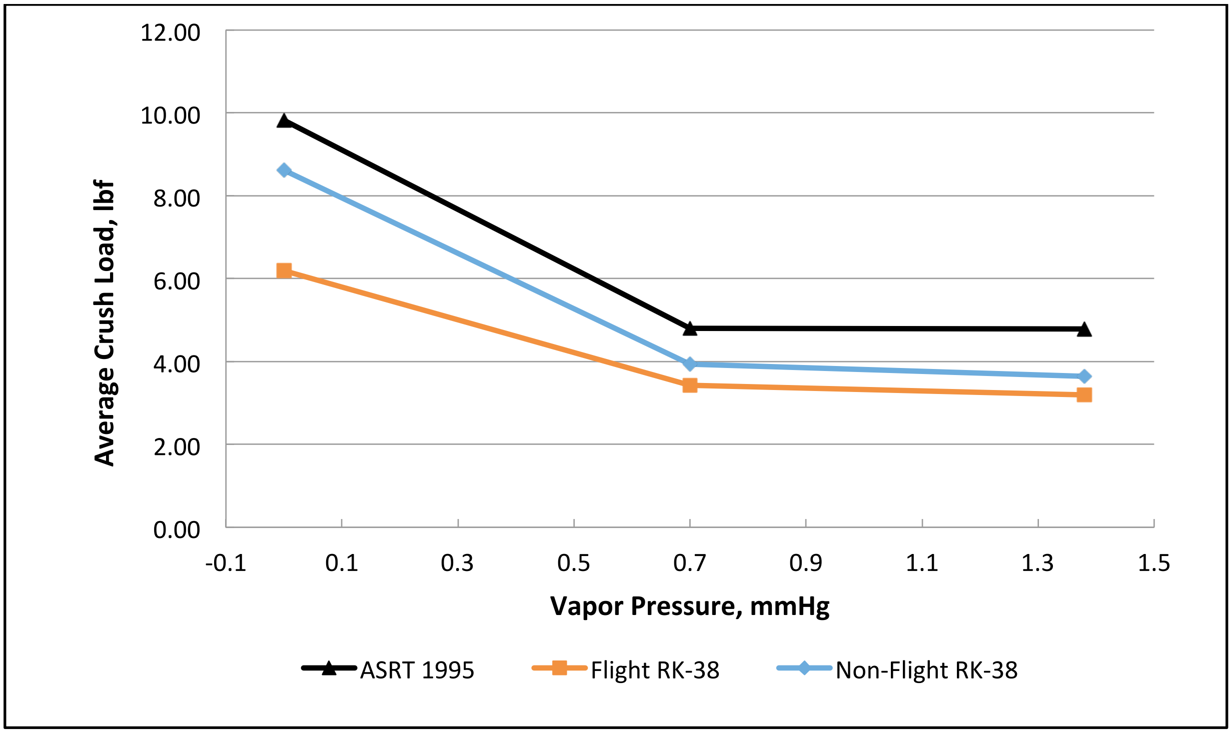

Figure 9. Single Pellet Crush Results

C. Attrition Testing

Attrition testing provides valuable information about a sorbent's propensity to produce dust which provide additional criteria to consider in sorbent selection for long-term missions. The attrition test method is based on ASTM D4058–96, Standard Test Method for Attrition and Abrasion of Catalysts and Catalyst Carriers.

1. Experimental

As with Bulk and Pellet Crush tests, a sample of sorbent was activated and a measured amount of activated sorbent, approximately 100 grams, was gently poured into a Materials Technology RoTab-AS abrasion test device, shown in Figure 10. The device was set to rotate 1800 cycles at one cycle per second. The resulting pellets and dust were separated through sieving and the pellets and dust re-activated and weighed. If a humidity conditioned test was to be performed, the sorbent was conditioned on the Humidity Conditioning Test stand (HCT) before loading in the attrition device. In all tests, the attrition device drum and the activated/conditioned sorbent were placed in a dry glovebox for sorbent loading.

Figure 10. Attrition and Abrasion Test per ASTM Standard D4058

2. *Results and discussion*

Table 2 tabulates the percent fines produced by the attrition tests. It can be seen that the ASRT 1995 produced fewer fines at the higher dewpoints, and about the same as the non-flight lot of RK-38 at the driest state. As with the bulk crush test, the flight lot of RK-38 produced, on average, the least amount of fines at the driest state, indicating greatest resistance to dusting. Figure 11 plots the average percent fines versus vapor pressure.

Table 2. Attrition Test Percent Fines

	Non-Flight RK-38 Raw Data			Flight RK-38 Raw Data			ASRT 2005 Raw Data		
Humidity Level, C	-90C	-21C	-14C	-90C	-21C	-14C	-90C	-21C	-14C
Vapor Pressure, mmHg	7.25E-05	0.7	1.38	7.25E-05	0.7	1.38	7.25E-05	0.7	1.38
% Fines	0.06 0.01 0.06	1.35 1.36 1.52	1.93 1.66 1.84	0.02 0.03 0.02	0.57 0.53 0.54	0.69 0.61 0.47	0.06 0.03 0.08	0.36 0.42 0.30	0.58 0.60 0.55
Avg % Fines	0.04	1.41	1.81	0.02	0.55	0.59	0.06	0.36	0.58

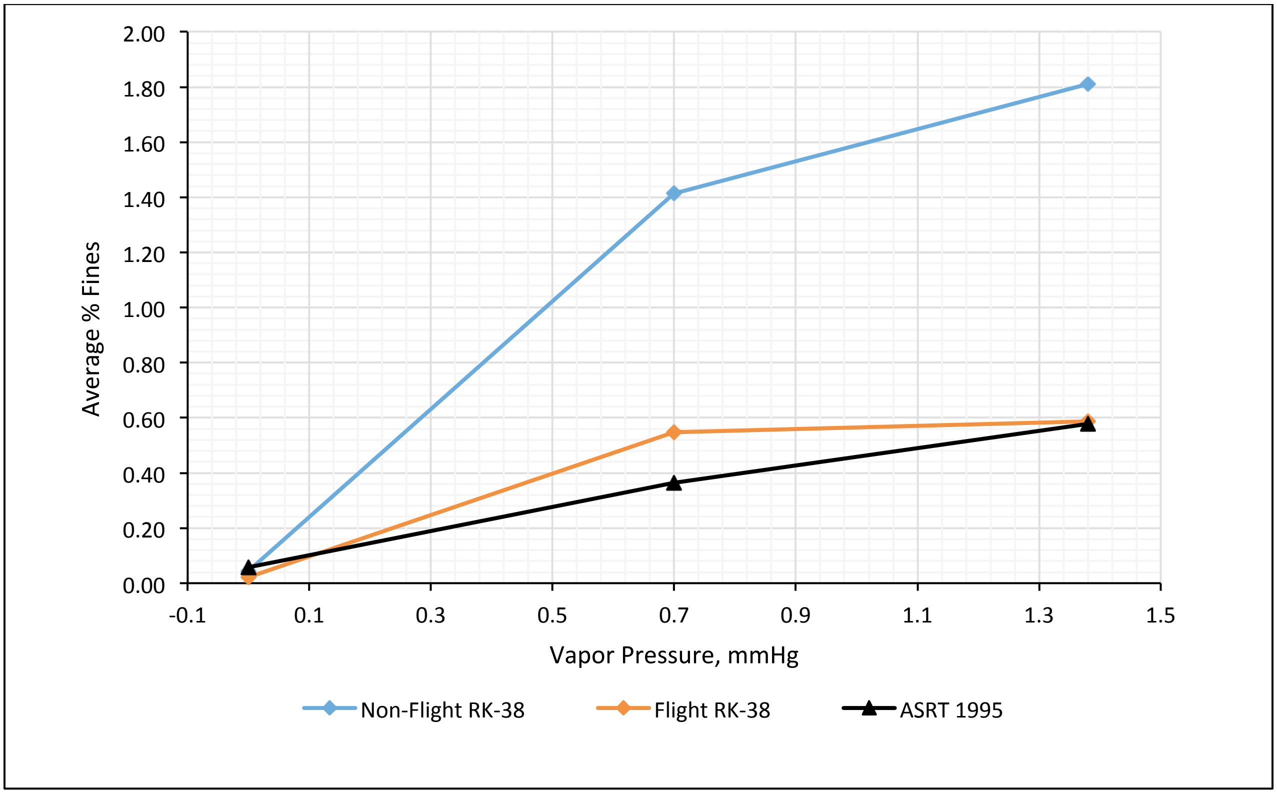

Figure 11. Average Attrition Percent Fines vs. Vapor Pressure

IV. Conclusion

The need for atmosphere revitalization systems to employ sorbents that perform satisfactorally even if exposed to some level of water vapor has been demonstrated by the issue of moisture exposure to the ISS CDRA. This paper described tests performed on three sorbents (ASRT 1995, a flight lot of RK-38, and a non-flight lot of RK-38), each conditioned at three humidity levels (-90°C, -21°C, and -14°C), and their performance compared. These and further tests with additional sorbents are expected to provide critical data to support optimized carbon dioxide removal systems for atmosphere revitalization systems used in future exploration vehicles.

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