

Performance of Silica Gel in the Role of Residual Air Drying. Part II.

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Removal of carbon dioxide (CO₂) is a necessary step in air revitalization and is often accomplished with sorbent materials. Since moisture competes with CO₂ in zeolite sorbent materials, it is necessary to remove the water first. This is typically accomplished in two stages: “bulk” removal and “residual” drying. Silica gel is used as the bulk drying material in the Carbon Dioxide Removal Assembly (CDRA) in operation on ISS. There has been some speculation that silica gel may also be capable of serving as both bulk and residual drying material to reduce system mass and Foreign Object Debris (FOB). Previous research tested silica gel alone as drying material. However, the silica gel volume used was not comparable to the current amount used on the CDRA. Therefore, the tests were repeated with the new silica gel volume. This paper discusses the fabrication and assembly of the modified canister to accommodate the new volume, the testing, and the evaluation of the test results.

Nomenclature

| | | |
|-----------------------|---|--|
| <i>ARC</i> | = | <i>Ames Research Center</i> |
| <i>ARREM</i> | = | <i>Air Resource Recovery and Environmental Monitoring</i> |
| <i>BaRDD</i> | = | <i>Bulk and Residual Dryers Downselect</i> |
| <i>CAS</i> | = | <i>Cabin Air Simulator</i> |
| <i>CDRA</i> | = | <i>Carbon Dioxide Removal Assembly</i> |
| <i>CO₂</i> | = | <i>Carbon Dioxide</i> |
| <i>CRCS</i> | = | <i>Carbon Dioxide Removal Compression System</i> |
| <i>DP</i> | = | <i>Dewpoint</i> |
| <i>FOD</i> | = | <i>Foreign Object Debris</i> |
| <i>ISS</i> | = | <i>International Space Station</i> |
| <i>MBAD</i> | = | <i>Membrane Bulk air Dryer</i> |
| <i>MSFC</i> | = | <i>Marshall Space Flight Center</i> |
| <i>NASA</i> | = | <i>National Aeronautics and Space Administration</i> |
| <i>NRAD</i> | = | <i>NovelAire Residual Air Dryer</i> |
| <i>PSA</i> | = | <i>Pressure Swing Adsorption Twin Towers Air Drying System model HR7</i> |
| <i>POIST</i> | = | <i>Performance and Operation Issues Testbed</i> |
| <i>RAD A</i> | = | <i>Residual Air Dryer, Unit #A</i> |
| <i>SCFM</i> | = | <i>Standard cubic feet per minute</i> |
| <i>SG</i> | = | <i>Silica gel</i> |

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SLPM = *Standard liters per minute*
TSA = *Temperature Swing Adsorption*

I. Introduction

The CDRA has the function of removing carbon dioxide (CO₂) on the International Space Station (ISS), along with the Russian Vozdukh. The CDRA's on orbit performance maintains the CO₂ partial pressure at < 5.3 mmHg for up to 9 crewmembers. Flight experience has previously demonstrated a vulnerability to Foreign Object Debris (FOD) migration in microgravity. This was due to zeolite particulate material which could block valve closure or lead to excessive drop in pressure due to accumulation on filters. Description of this failure mode and hardware changes to address these issues can be found in El Sherif and Knox¹. Additional CDRA modifications are described in Reysa, et al.².

The CDRA uses both silica gel and zeolite (13X) to dry the air before it enters the CO₂ removal process. The silica gel removes the bulk of the water while the zeolite acts as a residual dryer³. It was suggested that the silica gel alone might provide sufficient air drying, allowing the removal of the zeolite 13X and thereby reducing associated FOD risk. The resultant design may potentially be simpler and of lower mass. An activity was initiated within the Air Resource Recovery and Environmental Monitoring (ARREM) project to study the engineering suitability of this approach. Previous results from this activity have been reported; this paper presents results from additional tests. Studies on sorbent materials are also being conducted; see, for example, Knox, et al³.

A Summary of the CDRA configuration

The CDRA uses molecular sieves operating in a thermal/pressure swing cycles to selectively remove CO₂ from ISS air. As the CO₂ removal bed is also sensitive to water, the entering cabin air is first sent through a desiccating section before entering the CO₂ removal bed for CO₂ adsorption. The air then routes through a desorbing desiccant bed, returning clean, humid air to the cabin. Adsorption and desorption switches half cycles every 144 minutes.

The desiccant bed contains a quantity of silica gel, surrounded both upstream and downstream by 13X molecular sieve. The 13X layers serve as residual drying material and can also protect the silica gel from the occurrence of condensed water droplets. The entire section dries the air, flowing at 20 SCFM, to a dewpoint of -90°C. If the silica gel is to be sufficient without the molecular sieve, it must be able to adsorb sufficient water to reach the desired dewpoint and then desorb during the other half cycle, and sustain these levels during normal operation. The objective of this continuing research is to experimentally test the silica gel ability to act both as a bulk and a residual dryer. The data from these tests will be used by Marshall Space Flight Center (MSFC) for modeling purposes.

II. Experimental Set-up

A. Prior Work

This team previously reported on the rack assembly and performance data of the original ARC silica gel desiccant bed¹. It was found that breakthrough began after about one hour of exposure to a simulated cabin environment. This was not expected, as data from a 2006 test showed that breakthrough did not occur even after over an hour of exposure. The test canister was disassembled, and it was found that the quantity of silica gel contained was only about 245 in³, about 30% less than the expected 363 in³. This could, at least in part, explain why breakthrough occurred earlier than expected. Additional silica gel was provided, and the container was modified to accommodate the additional desiccant.

B. The Modified Silica Gel Apparatus

There are three silica gel test canisters used at MSFC: the CDRA, the POIST, and the Life Test. Previously ARC used the Life Test canister filled with 245in³ of Grace Davidson B125 silica gel. However, the volume of silica gel inside (245in³) was not comparable to either the POIST or the CDRA silica gel volume. Therefore, ARC dismantled the Life Test canister and fabricated a new standoff to accommodate a the new silica gel volume (363in³). Figure 1 showed some pictures of the dismantling and reassembly process. Beside modifying the new standoff, four RTDs were inserted around the edges of the canisters and are imbedded into the sorbent. These RTD readings will provide feedback as to how uniform the temperature profile is during bakeout and possibly give indications of flow issues.

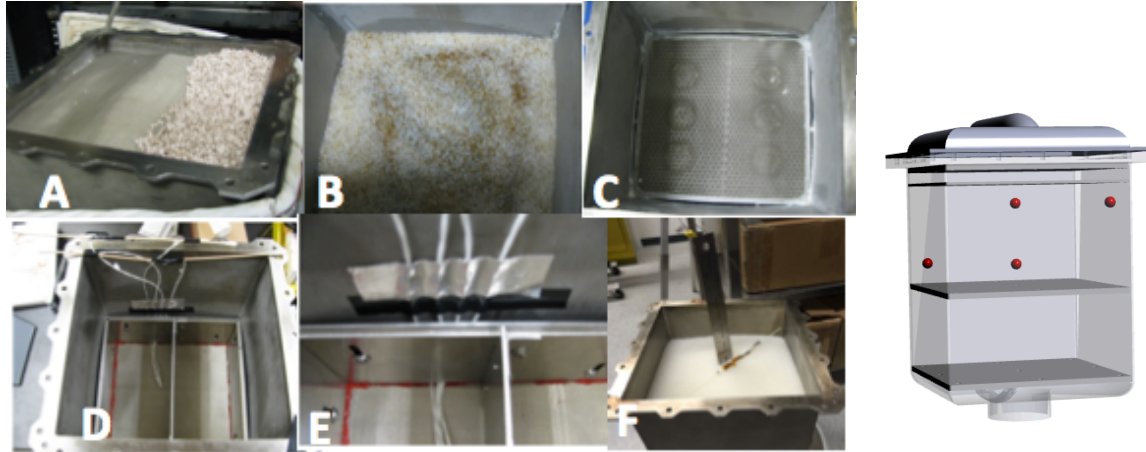


Figure 1: Pictures of the Life Test canister with (A) the Sorbead WS layer and screen, (B) a layer of the silica gel beads after bake out, (C) the middle screen surrounded by RTV sealant, (D) the new standoff installed, (E) the four RTDs to be imbedded into the sorbent layer, and (F) the silica gel layer. (right) A Solidwork diagram showing the location of the installed RTDs inside the sorbent layer (drawing per Joseph Lowman).

Table 1 outlined the layered material and associated depth and volume. The bottom of the canister contained only the standoff. In comparison to the CDRA bed, the standoff volume is where the 13X zeolite would be. The silica gel is filled at the top half of the canister, similar to the CDRA set-up. The standoff were fabricated with adjustable depth such that glass beads can be added at the bottom to resolve flow distribution/channeling issues, if needed. At the adsorption inlet, there is a layer of Sorbead WS® material to protect the SG from swelling by humid air. Table 2 lists the updated specifications for the ARC Life Test canister.

Table 1: A table listing the filling/packing order of the silica gel into the Life Test canister.

| Component | FY 14: Ames Life Test-Volume | FY 14:Ames Life Test-height | FY 15: Ames Life Test-Volume | FY 15: Ames Life Test-height |
|---|------------------------------|-----------------------------|------------------------------|------------------------------|
| bottom of the canister | | | | |
| | in ³ | in | in ³ | in |
| screen with RTV | | 0.04 | | 0.04 |
| Standoff | | 2.78 | | 3.76 |
| Spring Screen with RTV | | 0.251 | | 0.251 |
| Silica Gel | 245.496 | 3.53 | 363.45 | 4.92 |
| screen | | 0.02 | | 0.02 |
| | | | | 0.00 |
| Sorbeads | 49.80 | 0.71 | 49.80 | 0.73 |
| spring screen | | 0.251 | | 0.251 |
| gap from top edge of canister, without gasket | | 0.2035 | | 0.2035 |
| top the canister | | | | |
| Total height | 295.30 | 7.79 | 413.25 | 10.19 |

Table 2: A table listing the silica gel canister specifications

| Parameters | Specification |
|---|--|
| Bed geometry | square rectangular box |
| Canister Depth (in)* | 10.19 |
| Canister Width (in)* | 8.24 |
| Total SG volume (in³) | 413.24 |
| Total canister Volume (in³)* | 691.88 |
| Material | SOLOBEAD SG B125 Silica Gel (beaded) |
| Average Particle Size | 1.8mm |
| Sorbent main application | Used for Dehydration, and purification of |
| Specific Properties | Dehydration, low dust and attrition, low pressure drop |
| Maximum regeneration temperature (°C)* | 155.00 |
| Bulk Density at 20°C (68°C°(kg/m³))** | 200-850 |
| Density at 20°C (68°F(g/cm³))** | 2.17-2.20 |
| * data provided by MSFC. | |
| ** data based on GRACE Davison Silica Gel B125 Specification Sheet | |

A picture of the experimental apparatus and the process flow are shown in **Figure 2**. The test apparatus consists of five subsystems used for testing: (1) the Pressure Swing Adsorption (PSA) system; (2) The Cabin Air Simulator (CAS); (3) the Silica Gel bed; (4) the heater; and (5) the National Instrument/Labview control and data acquisition system.

The PSA system (Twin Towers HR7) supplies dry air of approximately -90°C at 20 SCFM for bake-out. To simulate ISS cabin air conditions, the CAS input air at 20SCFM, 8°C dewpoint, and 22°C. and Additional insulation was added (Lightweight Melanine, 1" thick) to help accommodate uniformity of temperature through the bed. Dewpoint sensors (Vaisala DMT348, GE Series 5 M2 Panametrics Probe, and a Michell Stirling Engine chilled mirror), thermocouples, RTDs, pressure transducers, and mass flow meters (FCI ST75V and Alicat MCR) were placed in various locations of the test apparatus to monitor and control the process.

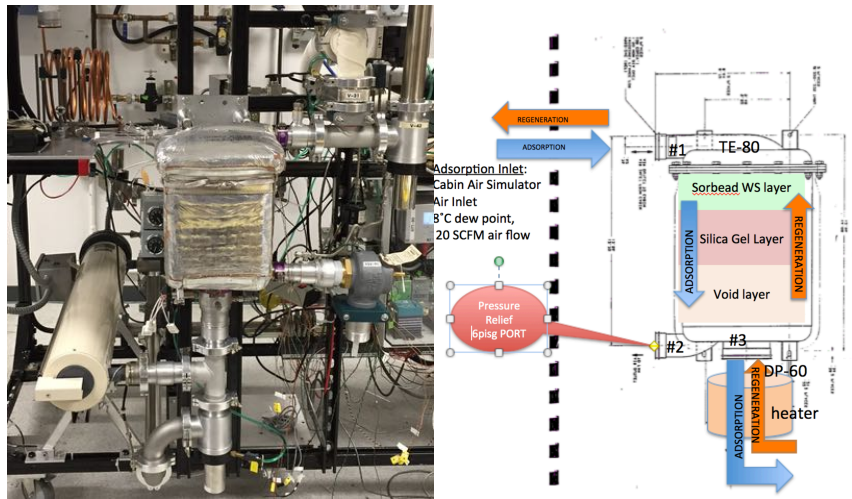


Figure 2: (left) A picture of the silica gel test set-up. (right) A diagram showing the orientation of the Silica gel bed and the flow direction for regeneration/bake-out and adsorption/breakthrough test.

C. Testing

After assembly, a total of three breakthrough tests were completed. Before each breakthrough test, the bed was baked out for at least 10 hours with PSA air at the -90°C and a 155°C exit temperature. The PSA air system copper piping had small leaks and the inlet dewpoint was not always maintained at -90°C for previous runs. The maximum inlet temperature and the bake-out outlet temperature set-points were based on a product information sheet from

GRACE Davison. It is planned that later tests will employ a variation of the temperature profile that is currently used by the CDRA and for the Bulk and Residual Dryer Down-select activities in 2013^{5,6}.

III. Results and Discussion.

The overarching objective of the silica gel test task is to determine if the silica gel sorbent itself can be used as both a bulk and a residual water removal. In order to achieve this task, the silica gel bed would need to be exposed to the same cyclic operation as the CDRA bed. Instead of conducting multiple scenarios of hardware cyclic tests, modeling was considered as an option to assist in determining the various possible test scenarios. Hardware testing then would be used as a validation process. Unfortunately, since the original baseline data for the CDRA silica gel bed were not available; therefore breakthrough tests were done on the ARC Life Test bed in order to provide data to MSFC for modeling purposes.

A. Breakthrough results and comparison

Three breakthrough tests were conducted and the dewpoints curves are shown in Figure 3. Table 3 summarized the historical data at MSFC, the preliminary modeling data, and test data at ARC. According to these graphs, the test (test #6) with the lower volume (245in³) breakthrough 33 minutes whereas the test at the larger volume (363in³) breakthrough at 75 minutes (test #103, “D” in Figure 4). With 44% reduction in volume, the bed breakthrough 67% before.

Comparing the breakthrough time with the MSFC 2006 data, the ARC breakthrough time is less than the 155 minutes (Figure 5.) The discrepancies may be due to several factors. One, the system set up is not the same. For example, the MSFC 2006 was conducted under cyclic operation. The bed contained a layer of 13X and WS sorbent. The ARC bed has a void layer. Two, ARC encountered considerable issues with dewpoints sensors issues that made it difficult to obtain reliable dewpoint data. Some of these operational issues are discussed below.

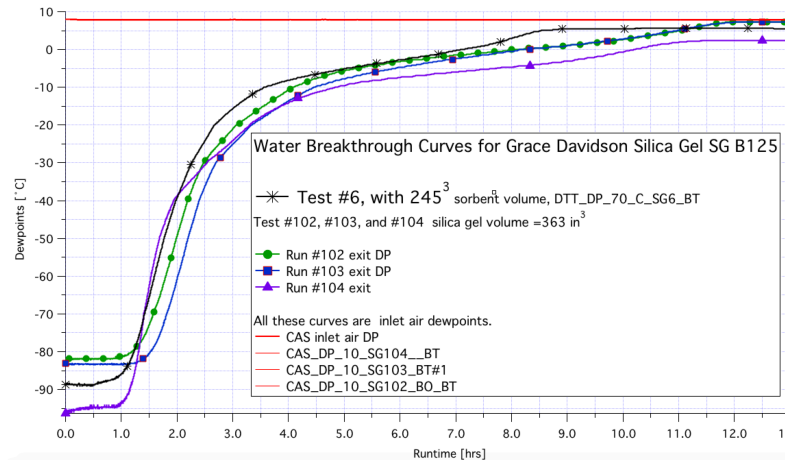


Figure 3: (Left) The breakthrough curve showing breakthrough results for both the previously used low volume of silica gel and the adjusted volume.

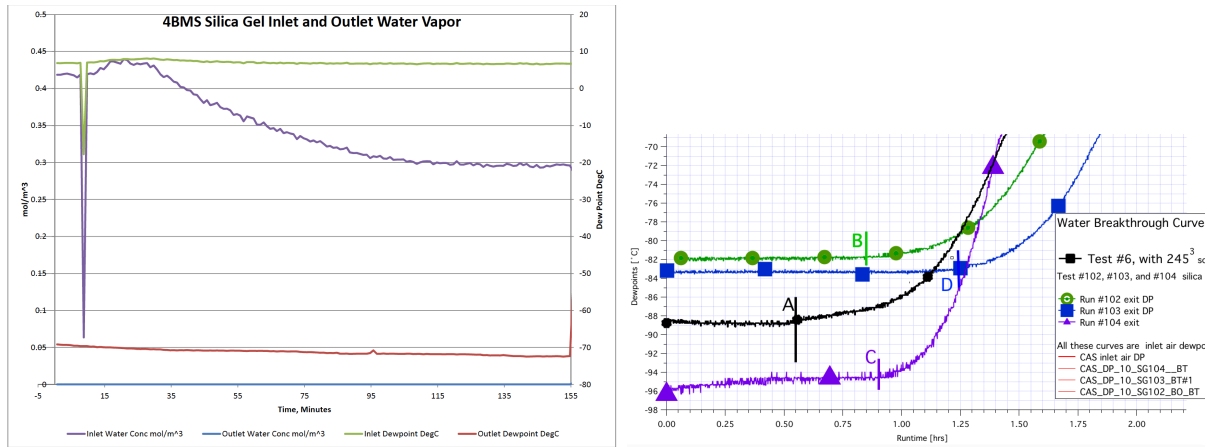


Figure 4: (left) Graphs comparing the breakthrough curves conducted at MSFC in 2006 during cyclic testing. (right)The graph showing the zoomed in section at the lower dewpoints region. Here, the different points A, B, C, and D show the different breakthrough times for the different runs.

Table 3: The table summarizing the breakthrough times comparison between the MSFC 2006 cyclic test, Rob Coker modeling data, and ARC test data.

| | Bed configuration | Volume | width | depth | CDRA-3 Silica Gel layer length | Porosity | breakthrough times based on the dew points curve | |
|-------------------------------|-------------------|--------|-------|-------|--------------------------------|----------|--|---------|
| | | | | | | | hrs | minutes |
| MSFC 2006 cyclic test | POIST-4 | 363 | 8.24 | 8.44 | 5.23? | tbd | 2.6 | 155 |
| Modeling data based on CDRA-3 | CDRA-3 | 407.8 | 8.25 | 8.45 | 5.85 | 38 | 2.8 | 168 |
| Modeling based on ARC data | CDRA-4 | tbd | 8.25 | 8.45 | 5.85 | tbd | 1.8 | 119 |
| run 6 | Life Test | 245 | 8.24 | 8.44 | 3.53 | tbd | 0.55 | 33 |
| run 102 | Life Test | 363 | 8.24 | 8.44 | 6.13 | tbd | 0.95 | 57 |
| run 103 | Life Test | 363 | 8.24 | 8.44 | 6.13 | tbd | 1.25 | 75 |
| run 104 | Life Test | 363 | 8.24 | 8.44 | 6.13 | tbd | 0.9 | 54 |

*modeling data provided my Rob Coker at MSFC

B. Data and Operational Issues

Some of the operational and data collection issues were encountered that may have contributed to the unexpected breakthrough results are: the reliability of dewpoint sensors , incompletd bake out due to non-uniformmity in heating, and insulation thickness.

1. Inadequate heating during bake out

Previously, it was believed that inadequate heating due to the air gap in the canister contributed to the incomplete bake out. Bake out is done by passing 20SCFM of heated (to 155°C exit at the bed outlet) dry PSA (with 100-400ppm CO₂) air through the bottom of the canister (the air gap) for at least 12 hours or until the inlet and outlet dewpoints converges. In previous test, there were no temperature probes imbedded in the sorbent layer. Therefore, it was not possible to determine if there were flow distribution issues in the bed. However, when the modified volume was installed, temperature probes were placed inside the sorbent to evaluate the bake out procedure. During bake-out the four temperature readings do vary at ramp up, but stabilized to one as time progresses.

2. Temperature rise and insulation thickness

Upon examination of temperature distribution data, readings of the RTDs imbedded in the sorbent during breakthrough, puzzling questions arised as to why these temperatures rose initially at breakthrough. Figure 5 shows an example of the temperature rise for run #103. Some speculations for the reason for this rise maybe due to: (1) Not

allowing the bed and the associated plumbing to cool completely before breakthrough; (2) the added insulation prevented heat from dissipating, and (3) the heat of adsorption. To resolve the cooling issue, in run #104, the bed was allowed to cooled for 12 hours overnight before breakthrough test was conducted, see Figure 7. The temperature rise at the inlet is similar in both run #103 and run #104.

It remains to be determined if the various parameters (heat of adsorption, insulation thickness, pressure drop, etc.) may contribute to this temperature rise at the inlet. Therefore, it is inconclusive if the experimental set-up contributed to the earlier breakthrough, or are there other contributing factors that is unknown at this time. One consideration would be that regeneration is done with PSA air. Recently, it was found that the CO₂ content of PSA air varies with time approximately 100-400ppm. Could this have contributed to the less than ideal bake-out conditions? Further testing and analysis need to be completed using nitrogen as the bake-out gas and/or vacuum to ensure that the sorben is fully desorbed.

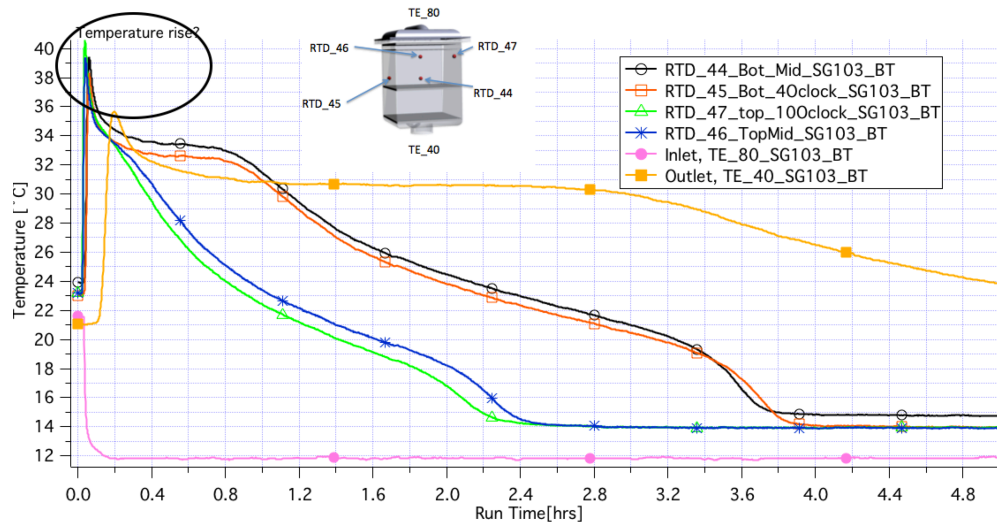


Figure 5: The graph for run #103 showing the initial temperature rise of all the RTD readings imbedded in the sorbent breakthrough.

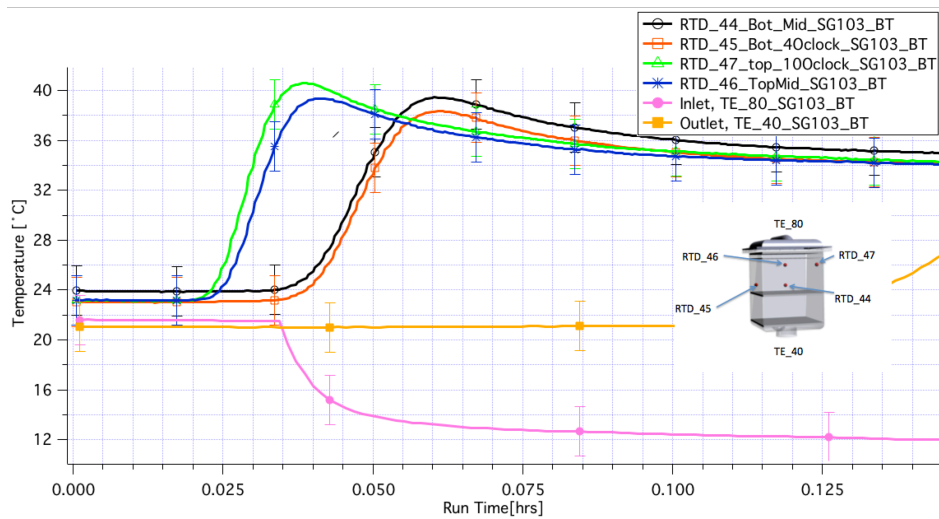


Figure 6: The zoomed in section Figure 5 showing the temperature rise with error bars of +/- 2 degrees Celcius for RTD readings.

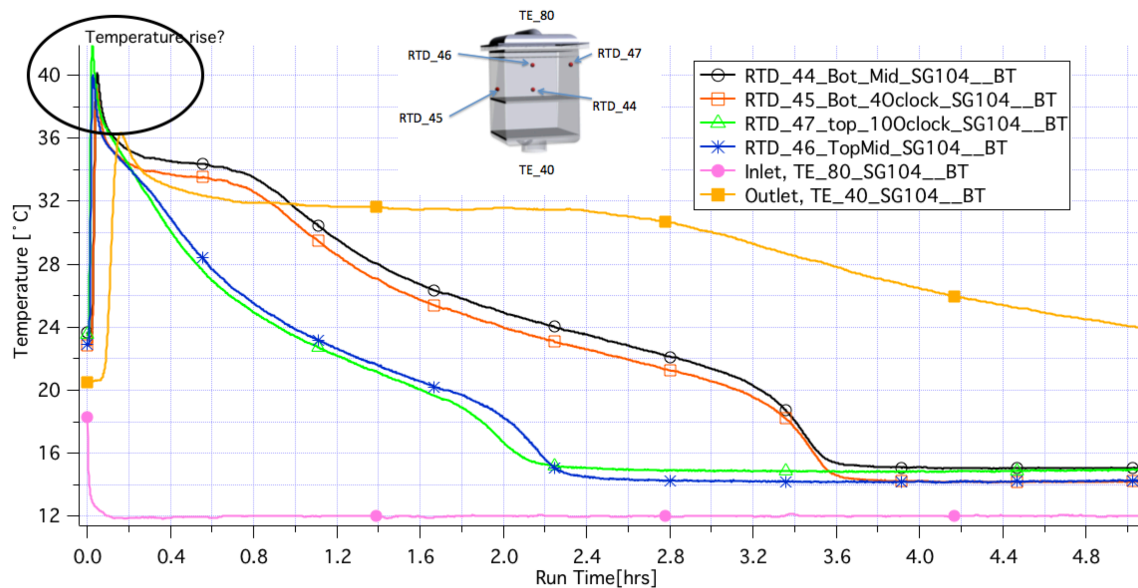


Figure 7: The graph for run #104 showing the initial temperature rise at the inlet of the bed during breakthrough.

3. Dewpoint measurements

Dewpoints sensors were used to validate the PSA air dry condition for bake out as well as monitor dry conditions at the inlet and outlet of the bed. Several issues related to dewpoints measurements are : (1) the maximum temperature the dewpoints sensor can tolerate; (2) the responds time from high humidity to low humidity; (3) the respond time from low humidity to high humidity; and (3) the dewpoint sensors ranges. We placed several dewpoints from different manufactures in series and parallel to both determine the offset as well as obtain accurate data. The discussion of the variability and reliability of the dewpoints measurements is beyond the scope of this work. We, however, continue to explore a variety of dewpoints sensors for measurement our air stream.

Conclusion

The first step in characterizing the SG bed behavior, breakthrough testing, is currently not conclusive, as it appears inconsistent with past data. But since the prior data was performed in a cyclic manner, this difference may be due in part to operational differences. The silica gel was the same material, but came from a different manufacturing lot. The SG canisters were different in that the canister used in the recent tests had no 13x layers, whereas the 2006 data had 13x layers, similar to the CDRA configuration (the probes were placed directly before and after the SG layer.) Based on the breakthrough graphs, intial breakthrough occurs well before two hours and complete breakthrough by 8 hours. Additional testing will be performed to verify repeatability, and multiple cycle tests may contribute insight. Improvements have been made to the experimental apparatus to obtain more reliable results,including better insulation at the SG bed, fixing leaks in the PSA air line, resolving dewpoints sensors noise, and preconditioning dewpoint sensors. Further testing will help determine whether or not the SG bed accommodate both the bulk and residual drying functions.

Acknowledgments

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