

# Microlith-based Structured Sorbent for Carbon Dioxide, Humidity, and Trace Contaminant Control in Manned Space Habitats

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To support continued manned space exploration, the development of atmosphere revitalization systems that are lightweight, compact, durable, and power efficient is a key challenge. The systems should be adaptable for use in a variety of habitats and should offer operational functionality to either expel removed constituents or capture them for closed-loop recovery. As mission durations increase and exploration goals reach beyond low earth orbit, the need for regenerable adsorption processes for continuous removal of CO<sub>2</sub> and trace contaminants from cabin air becomes critical. Precision Combustion, Inc. (PCI) and NASA – Marshall (MSFC) have been developing an Engineered Structured Sorbents (ESS) approach based on PCI’s patented Microlith<sup>®</sup> technology to meet the requirements of future, extended human spaceflight explorations. This technology offers the inherent performance and safety attributes of zeolite and other sorbents with greater structural integrity, regenerability, and process control, thereby providing potential durability and efficiency improvements over current state-of-the-art systems. The major advantages of the ESS explored in this study are realized through the use of metal substrates to provide structural integrity (i.e., less partition of sorbents) and enhanced thermal control during the sorption process. The Microlith technology also offers a unique internal resistive heating capability that shows potential for short regeneration time and reduced power requirement compared to conventional systems. This paper presents the design, development, and performance results of the integrated adsorber modules for removing CO<sub>2</sub>, water vapor, and trace chemical contaminants. A related effort that utilizes the adsorber modules for sorption of toxic industrial chemicals is also discussed. Finally, the development of a 4-person two-leg ESS system for continuous CO<sub>2</sub> removal is also presented.

## Nomenclature

$C$	= Celsius
$c$	= concentration
$c_0$	= feed or inlet concentration
$cfm$	= cubic feet per minute

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<i>cm</i>	=	centimeter
<i>d</i>	=	diameter
<i>ESA</i>	=	electrothermal swing adsorption
<i>g</i>	=	gram
<i>GSA</i>	=	geometric surface area
<i>h</i>	=	hour
<i>in</i>	=	inch
<i>kg</i>	=	kilogram
<i>L</i>	=	length
<i>m</i>	=	meter
<i>mg</i>	=	milligram
<i>min</i>	=	minute
<i>ml</i>	=	milliliter
<i>pp</i>	=	partial pressure
<i>ppm<sub>v</sub></i>	=	part per million by volume
<i>psia</i>	=	pound per square inch absolute
<i>RH</i>	=	Relative Humidity
<i>slpm</i>	=	standard liter per minute (21°C, 14.7 psia)
<i>V</i>	=	volume
<i>W</i>	=	Watts
<i>wt %</i>	=	percent by weight

## I. Introduction

Adsorption processes have important applications in air quality engineering, such as filtration of environmental contaminants, cabin air revitalization (AR) system in spacecraft and spacestation, and adsorption of toxic industrial chemicals (TICs). For example, an adsorption-based approach has been used for cabin air quality control on all crewed spacecraft, and is expected to continue to remain at the forefront of spacecraft cabin air quality control technologies. Removal of CO<sub>2</sub> and trace contaminants is crucial for the spacestation because these substances can be harmful to the crew if they are allowed to buildup over time. In the spacecraft applications, these chemicals can be adsorbed via several options, such as pellet bed canisters and charcoal beds. As mission durations increase and exploration goals reach beyond low Earth orbit, the need for regenerable adsorption processes for continuous, in-situ removal of CO<sub>2</sub> and trace chemical contaminants from the cabin air becomes important.

The implementation of zeolites, molecular sieves, and silica gel in the adsorption process is of interest due to their high efficiency to selectively remove chemical contaminants as well as their chemical inertness and non-flammable properties. Furthermore, these sorbent materials can be readily regenerated via either thermal swing adsorption (TSA) or pressure swing adsorption (PSA), and therefore, they are suitable for a continuous control of CO<sub>2</sub> and trace contaminant levels during long duration spacecraft missions. These regenerable adsorber systems do not have to be replaced during mission duration, and can be smaller and lighter than the disposable adsorber beds. Currently, packed beds of sorbent pellets are mostly used in the adsorption systems; however, recent studies have shown that these materials can be easily fluidized and/or eroded, due to both thermal cycling and mechanical vibration, and can generate fine particulates that bypass the downstream mesh filters.<sup>1,2</sup> This results in particulates buildup in downstream pumps, blowers, and other components, and has been problematic in some aerospace applications. Furthermore, these packed beds of pellets create a large pressure drop across the adsorption system resulting in high parasitic losses.

In an effort to develop alternative approaches to packed bed systems, NASA-supported research at Precision Combustion, Inc. (PCI) has demonstrated that zeolites and molecular sieves coated on Microlith<sup>®</sup> metal mesh elements (patented and trademarked by PCI) can effectively adsorb a number of the contaminants of interest, such as CO<sub>2</sub>, ethanol, and ammonia.<sup>3</sup> The inert Microlith substrates and the use of a binder during coating of the zeolites and other sorbent materials on them result in volumetric sorbent loadings that are considerably lower than the conventional carbon bed and packed bed systems. Typically, the volumetric sorbent loadings obtained for the Microlith-based sorbent beds are about 30-35% of the loadings reported for the packed bed systems. However, the unique capability for direct resistive heating of the Microlith metal mesh support permits rapid periodic regenerations via direct internal heating with low power requirement, which confers a clear advantage over a packed bed of pellets that can only be regenerated using an external heater. Furthermore, the high heat transfer rate of the Microlith substrate results in relatively uniform temperature distribution across the sorbent bed and fast transient

response during the regeneration process, compared to a more sluggish thermal response of the packed bed of pellets. Therefore, the weight and volume of the conventional adsorber subassemblies can potentially be reduced by implementing zeolites supported on the Microlith substrate and by employing periodic sorbent regeneration. Additionally, the implementation of the Microlith substrates, with a large void fraction, reduces the pressure drop across the adsorption system when compared to a similar packed bed unit.

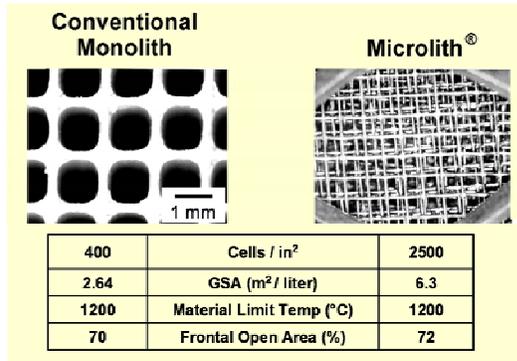
To date, PCI has successfully developed multiple Microlith-based AR systems that have been tested at NASA – Marshall (MSFC) for functional verification and performance assessments. Examples include high temperature catalytic oxidizers (CATOX) designed for retrofit into the Trace Contaminant Control System (TCCS) of the International Space Station (ISS), a combined Trace Contaminant and CO<sub>2</sub> removal adsorber, and most recently three subscale adsorbers (i.e., 1-crew unit) for a subsequent removal of water vapor, Trace Contaminants, and CO<sub>2</sub> to provide the complete function of a closed-loop trace contaminant and carbon dioxide removal system. The subscale adsorber modules have been tested at MSFC and have shown to meet functionality needed to support lunar surface closed-loop operations. This technology, consisting of sorbent coated onto an expanded metal substrate that serves as both support structure and heating element, has proven to provide quick, thorough, and efficient regeneration, surpassing the capabilities of a traditional packed bed configuration.

In order to obtain continuous removal of CO<sub>2</sub> in a closed-loop space, a two-leg system is required so that one leg functions to treat the incoming process air while the alternate leg regenerates. Recently, PCI has designed and developed a pair of adsorber units consisting of water removal module and CO<sub>2</sub> removal module to obtain full-scale (i.e., 4-crew unit) CO<sub>2</sub> removal functionality. A Microlith-based regenerable water vapor removal module was deemed necessary in order to remove humidity and thus mitigate the significant negative effect of humidity on CO<sub>2</sub> sorbent effectiveness and adsorber performance. Full-scale adsorber modules, comprising two CO<sub>2</sub> removal modules and two water vapor removal modules, rated for 20-cfm process air flow rate (i.e., 4 kg of CO<sub>2</sub> removed per day which is equivalent to a 4-crew metabolic load) were delivered to MSFC for further tests to validate performance and to optimize adsorber design and operating conditions. All of these modules are based on PCI's patented Microlith technology (coated with the appropriate sorbent materials) and are equipped with the internal resistive heating capability. In this paper, we discuss the design, development, and performance results of subscale adsorber modules for removal of water vapor and CO<sub>2</sub>. A related effort that utilizes similar adsorber modules for sorption of TICs is also presented. Finally, the development of 4-crew, two-leg regenerable water and CO<sub>2</sub> removal modules, including design optimization and sizing based on the available subscale data, is discussed.

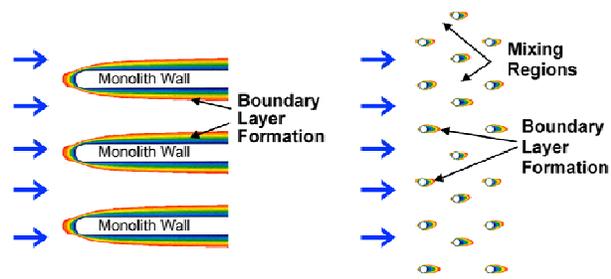
## II. Microlith Substrate Technology

The adsorbers described here are based on PCI's patented Microlith<sup>®</sup> technology (trademarked by PCI).<sup>4</sup> The Microlith substrate consists of a series of ultra-short-channel-length, sorbent or catalytically coated metal meshes with very small channel diameters (Fig. 1). The mesh-like substrate provides very high heat and mass transfer coefficients, low thermal mass, and extremely high reaction rates. The use of this kind of reactor, where the reacting stream is passed through the active substrate at extremely high space velocity, is generically termed a short contact time (SCT) approach. Whereas in a conventional honeycomb monolith, a fully developed boundary layer is present over a considerable length of the device, the ultra-short-channel-length Microlith substrate minimizes boundary layer buildup, resulting in remarkably high heat and mass transfer coefficients compared to other substrates (e.g., monoliths, foams, and pellets). The Microlith substrate also provides about three times higher geometric surface area over conventional reactors (e.g., monoliths) with equivalent volume and open frontal area. Furthermore, the Microlith substrate can pack more active surface area into a small volume, providing a substantial increase in adsorption area for a given pressure drop. Finally, PCI's proprietary sorbent coatings, with high surface area washcoats, allow for high sorbent utilization with rigorously demonstrated mechanical and performance durability.

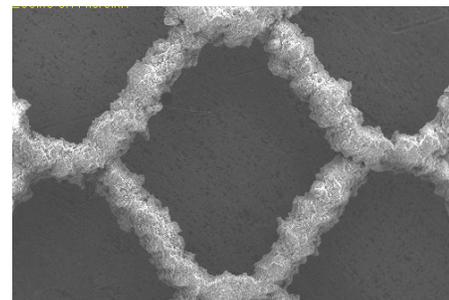
The heat and mass transfer coefficients depend on the boundary layer thickness. For a conventional long channel honeycomb monolith, a fully developed boundary layer is present over a considerable length of the active surface, thus limiting the rate of reactant transport to the surface of active sites. This is avoided when short channel length screens are used. A Computational Fluid Dynamics (CFD) analysis (Fig. 1) using Fluent illustrates the difference in boundary layer formation between a monolith and Microlith screens. Finally, PCI's proprietary sorbent coating formulations and application methods, with high surface area washcoats, allow for reasonably low sorbent usage with rigorously demonstrated long-term mechanical, thermal, and performance durability.



**Figure 1. Physical characteristics of conventional, long honeycomb monolith and Microlith substrates, and CFD analysis of boundary layer formation for a conventional monolith and three Microlith screens.**



The use of Microlith substrates with high heat and mass transfer rates, high surface area, and low pressure drop has a significant impact on adsorber performance and size as compared to the pellets or monoliths based adsorbers. The effectiveness of the Microlith technology and long-term durability of PCI’s proprietary zeolite and catalyst coatings have been systematically demonstrated in different applications. These include exhaust aftertreatment,<sup>5</sup> trace contaminant control,<sup>6-9</sup> adsorption of TICs and sulfur,<sup>10</sup> catalytic combustion,<sup>11</sup> partial oxidation of methane,<sup>12,13</sup> liquid fuel reforming,<sup>14</sup> CO preferential oxidation, and water-gas shift reactors.<sup>15</sup> A scanning electron microscopy (SEM) micrograph of the coated Microlith substrate is shown in Fig. 2. SEM analysis indicates uniform coatings on the substrate with complete coverage.



**Figure 2. Surface-scan SEM micrograph of the coated Microlith substrate.**

Summary of benefits for the Microlith-based adsorption approach is as follows:

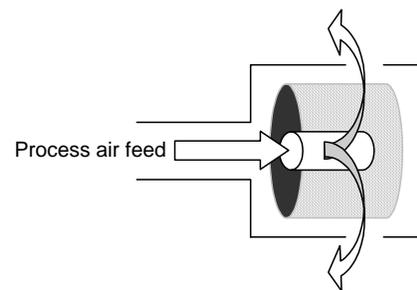
- Compact, lightweight, high efficiency adsorbers with greater selectivity for removal of specific contaminants.
- Higher mass transport for a more efficient utilization of sorbents.
- Higher heat transfer rates for greater temperature uniformity.
- Reduced pressure drop.
- Flexibility in designing and shaping the reactor (e.g., radial or planar configurations).
- Easily scalable for higher throughputs (i.e., modular design).
- Fast transient response with rapid startup and ability to withstand thermal shocks and multiple thermal cycling during adsorption-desorption sequences.
- Demonstrated long-term mechanical and performance durability.

### III. Results and Discussion

#### A. Design and Fabrication of Regenerable Adsorber Module

The original electrothermal swing adsorption (ESA)-based adsorber design consisted of a linear stack of alternate layers of sorbent-coated Microlith screens and sorbent-coated insulating meshes. The insulating mesh was utilized to isolate the Microlith screens electrically when applying the resistive heating during the ESA regeneration mode. Implementing this linear-stack design, however, presented practical difficulties.

Subsequently, a unique adsorber design concept consisting of a “jelly roll” coil in a radial flow configuration was implemented.<sup>1,9</sup> The radial design was based on a two-layer “sandwich” system consisting of a continuous layer of sorbent-coated Microlith screens and a corresponding insulating layer coated with the same sorbent material. Similar with the previous design, the insulating



**Figure 3. A simplified Microlith-based radial flow adsorber design consisting of a “jelly-roll” coil of sorbent-coated Microlith screens and sorbent-coated insulating meshes.**

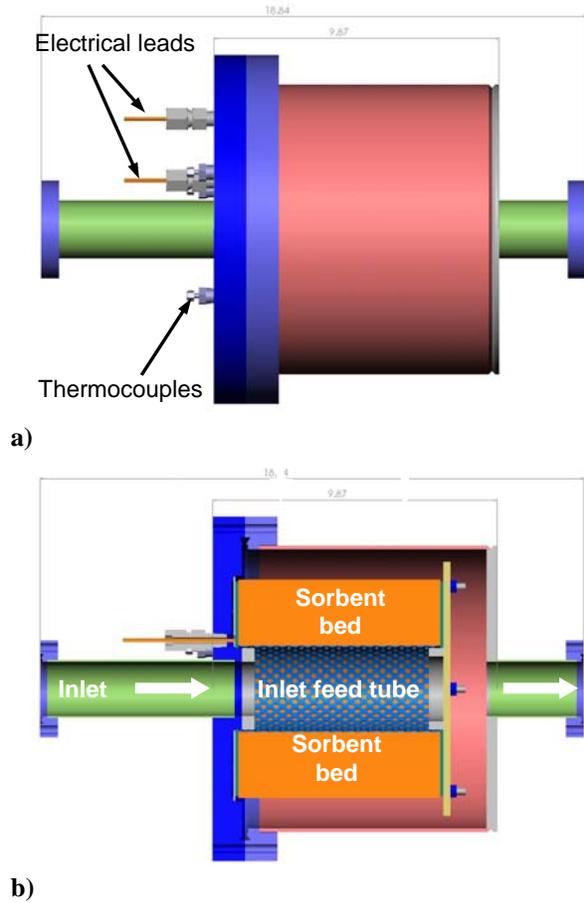
mesh serves as an insulating layer for isolating the Microlith layer electrically. Upon coiling the two-layer system around a centerline feed tube (i.e., a “jelly-roll” coil), the radial flow configuration can be achieved as illustrated in Fig. 3. A preliminary calculation indicated that the radial flow arrangement provided volumetric sorbent loadings that are at least comparable to a linear stack of screen elements. Furthermore, from the electrical and hardware assembly vantage points, a continuous length of coated screens largely mitigated the complicating issues encountered with a stack of screens, such as shorting and reactant channeling. Therefore, direct electrical heating of the metal Microlith substrate to regenerate the sorbent could be implemented more readily in the radial flow arrangement.

Figure 4 shows the external and internal cross-section views of a Microlith-based radial flow adsorber unit with internal resistive heating capability. To date, PCI has delivered several adsorber units with this design concept rated for 5-cfm flow rate to NASA and Air Force Research Lab (AFRL) for performance testing. CFDesign, a CFD simulation software package available at PCI, was used to study the flow pattern of process air within the adsorber unit. The unit design and sizing were optimized based on the modeling results in order to obtain a uniform flow distribution in the radial flow configuration and to avoid the presence of recirculation zones while minimizing the total housing volume (i.e., increasing the overall volumetric efficiency).

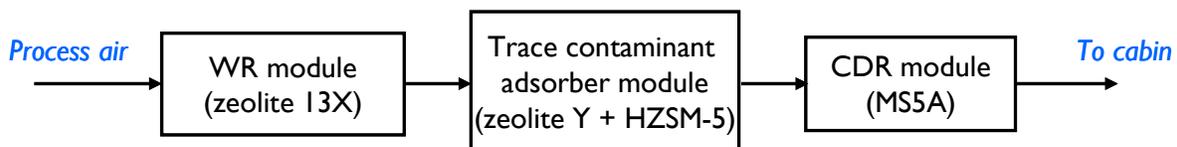
During the zeolite and other sorbents coating development, several washcoat formulations were evaluated in an effort to produce an adherent coating. The washcoats were applied to the Microlith substrates and insulating meshes using methods developed at PCI that allow for rapid application of sorbent coatings to large volumes of substrate.

**B. Moisture, CO<sub>2</sub>, and Trace Contaminant Sorption for NASA Cabin Air Application**

A total of three subscale adsorber modules, consisting of one water vapor removal (WR) module, one CO<sub>2</sub> removal (CDR) module, and one trace contaminants removal module, were delivered to NASA for initial performance testing. During the tests, process gas containing water vapor and CO<sub>2</sub> was first directed to the water removal module where the water vapor was removed. The removal of water vapor is necessary since the zeolite sorbents inside the CO<sub>2</sub> and trace contaminants removal modules are relatively hydrophilic and the presence of water vapor will significantly reduce the sorption capacity and effectiveness of the subsequent removal modules. Figure 5 illustrates the adsorber modules in series for removal of CO<sub>2</sub> and trace contaminant in manned space habitats. In this application, a pair of these adsorber sets will be working in tandem, one set of adsorbers will be in adsorption mode while the other set will be in regeneration mode.



**Figure 4. (a) External and (b) Internal cross-section views of the Microlith-based radial flow adsorber design concept.**



**Figure 5. Atmosphere revitalization system concept consisting of three adsorber modules in series for removal of CO<sub>2</sub> and trace contaminant in manned space habitats.**

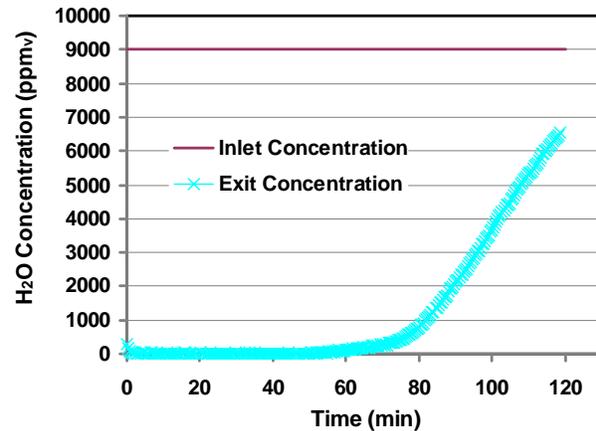
In order to obtain the performance curve (i.e., breakthrough profile) for each adsorber module, sorption tests were performed both by PCI (in collaboration with AFRL) and at MSFC using the WR module and the CDR module, separately. During performance test performed for PCI by AFRL, the WR module, consisting of a zeolite 13X sorbent bed, was exposed to process air at 5 cfm consisting of water vapor at ~9000 ppm<sub>v</sub> (i.e., 39% RH). The exit concentration of the water vapor was monitored as a function of time and the breakthrough curve is plotted in Fig. 6. This adsorption test was performed at ambient condition.

The WR adsorber module indicated high bed utilization, where the H<sub>2</sub>O concentration breakthrough in the outlet stream was observed after ~60-min of sorption. For this particular NASA application, breakthrough is reached when the exit concentration of the water vapor equals to ~100

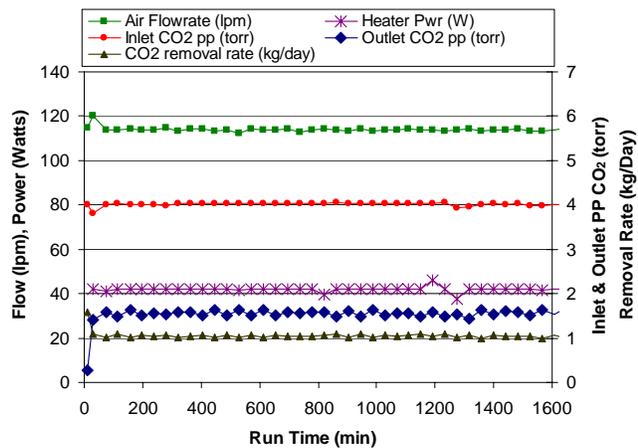
ppm<sub>v</sub> (i.e., ~45°F dew point) This preliminary test was discontinued after 120 minutes. The breakthrough profile showed a rather steep increase, which validated the high bed utilization of the “jelly roll” sorbent coil inside the adsorber module. The saturation capacity of the sorbent bed was ~21 wt.% (i.e., 21 g of water uptake per 100 g of zeolite 13X). About 60% of the bed capacity was utilized at breakthrough and up to ~93% capacity was used at c/c<sub>0</sub> of 0.5. The sorption capacity test of the WR module was only performed using air at a maximum relative humidity of 39%. Lower humidity level resulted in a longer breakthrough time, as expected. Since this particular WR module was designed to treat air consisting of up to ~40% RH, we expect that the exposure of the unit to air with higher humidity level will significantly reduce the breakthrough time, requiring a more frequent regeneration process.

Separately, the CDR adsorber module, consisting of a Molecular Sieve 5A (MS5A) sorbent bed, was tested at MSFC by passing process air stream at a flow rate of 114 slpm (i.e., 4 cfm) with an inlet CO<sub>2</sub> partial pressure of 4 torr. The cyclic test was performed at a 20-min half cycle, where adsorption mode and regeneration mode were carried out alternatively at 20 min interval. The adsorption process was performed at ambient condition. Figure 7 shows the performance of the CDR adsorber module during the 1600-min operation, which corresponds to ~40 full adsorption-desorption cycles. The test results indicated that the performance of the CDR adsorber module was stable after exposure to multiple thermal cycles, capable of consistently removing the targeted 1 kg of CO<sub>2</sub> per day (i.e., subscale, 1-crew unit) at an average heater power of 40 W. The 20-min half cycle was determined to be sufficient for the upstream WR module to remove water vapor from the feed air and to maintain the dew point of the process air entering the CDR adsorber module at -45°F. The specific power consumption for the Microlith-based CO<sub>2</sub> removal module was calculated to be 960 W-h/kg of CO<sub>2</sub> removed, which was much lower compared to the specific power of 2,174 W-h/kg CO<sub>2</sub> reported for the packed bed system.<sup>16</sup>

The third module was developed for removal of the trace contaminants from the cabin air. At the submission of this paper, the results obtained from performance testing performed at MSFC were not yet available. However, PCI has separately performed preliminary tests prior to developing the subscale trace contaminants removal module. The sorbent coating was tested for the removal of ethanol, acetone, ammonia, toluene, and dichloromethane (DCM). The design was estimated to be sufficient for maintaining the exit concentrations of the trace contaminants significantly below their inlet levels during the sorption cycle, whereas the regeneration requirement of the CO<sub>2</sub> removal segment



**Figure 6. Inlet and exit water vapor concentrations as a function of time from a single water sorption test.**



**Figure 7. Average inlet and outlet CO<sub>2</sub> concentrations, CO<sub>2</sub> removal rate, and average heater power during cyclic test of the subscale CDR module at MSFC.**

determined the cycle time for this adsorber unit.

In the preliminary test, the total flow rate of the feed process air was maintained at ~5 cfm, and the component concentrations were 3700 ppm<sub>v</sub> CO<sub>2</sub>, 500 ppm<sub>v</sub> ethanol, 10 ppm<sub>v</sub> NH<sub>3</sub>, 11 ppm<sub>v</sub> acetone, 8 ppm<sub>v</sub> toluene, and 5 ppm<sub>v</sub> DCM. The inlet concentrations of the trace contaminants were determined to be half of their Spacecraft Maximum Allowable Concentrations (SMAC),

except for NH<sub>3</sub> which was introduced at full SMAC due to the detection limit of the analysis equipment. Figure 8 represents the results from the prototype sorption testing, showing the CO<sub>2</sub> exit concentration as a function of time during the 50-min adsorption cycle. The prototype implemented in this preliminary study consisted of a combination of MS5A, zeolite Y, and HZSM-5 sorbent beds, and is different from the CDR module and the trace contaminant removal module being tested at MSFC. The results in Fig. 8

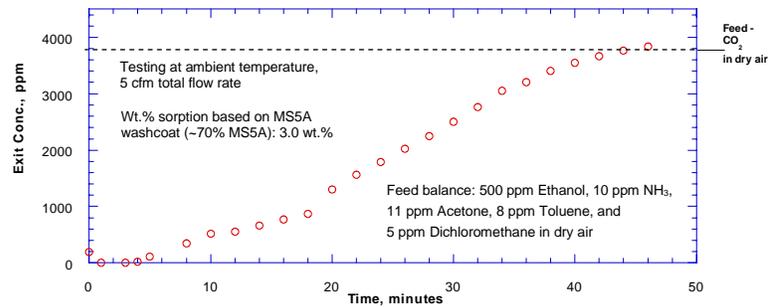
indicated 3 wt% CO<sub>2</sub> sorption capacity based on the MS5A washcoat (i.e., 0.03 g of CO<sub>2</sub> sorbed per g of MS5A washcoat), and a 50 min cycle time during which ~55% of the delivered CO<sub>2</sub> was adsorbed. During this test, the concentrations of trace contaminants in the exit gas at the end of adsorption cycle were well below their inlet levels. With the exception of ethanol, the exit concentrations of other trace contaminants (i.e., NH<sub>3</sub>, acetone, toluene, and DCM) were essentially zero. The ethanol exit level at the end of the test was only 5 ppm<sub>v</sub> or 1% of its inlet concentration. While these end-of-cycle trace contaminant levels might rise over multi-cycle operation of the prototype, our previous durability studies suggest that they will equilibrate at levels appreciably lower than their inlet concentrations. No change in pressure drop across the adsorber module was observed after multiple thermal cycles.

Furthermore, the sorbent coatings on both the Microlith substrate and the insulating mesh showed good adhesion and durability after multiple thermal cycles. The coating adhesion and durability was evaluated by separately conducting a thermal cycle test of identical sorbent-coated Microlith and insulating mesh layers (which were not assembled inside an adsorber housing), and was analyzed qualitatively and quantitatively. In this test, the sorbent coated samples were placed in an oven and the temperature was brought up to 200°C at a rate of 10°C/min and was held at 200°C for 30 minutes, which was similar to a typical temperature sequence during the regeneration of PCI's adsorber modules. For qualitative analysis, the adhesion and coating durability before and after 120 thermal cycles were compared by tapping and rubbing the samples, and trying to observe any resulting "dusting". For quantitative analysis, the sample weight before and after 120 cycles (and after tapping and rubbing of the sample) was measured. Based on the sample weight at the end of the thermal cycle test, the % coating weight loss was calculated. In this study, the % coating weight loss was defined as the amount of sorbent coating lost due to thermal cycle divided by the initial amount of sorbent coating. The qualitative analysis indicated minimal dusting after the thermal cycle test, and the quantitative analysis showed ~3 wt.% coating weight loss.

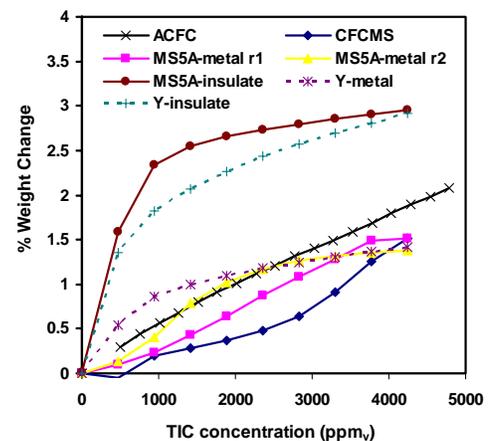
### C. Adsorber Module for Chem-bio Defense Application

Four prototype adsorber units with a design concept similar to that of the NASA subscale adsorber systems were developed for AFRL for testing as filters against toxic industrial chemicals (TICs). The four adsorber units consisted of two water removal units (i.e., WR modules) and two units for removing TICs. Two sets of filters working in tandem were used to implement the electrothermal swing adsorption concept. Process air was first passed through the water removal unit in order to reduce the effect of water co-adsorption on the TIC filter and thus maximizing the sorption capacity for TIC components.

Figure 9 shows the preliminary results of TIC adsorption



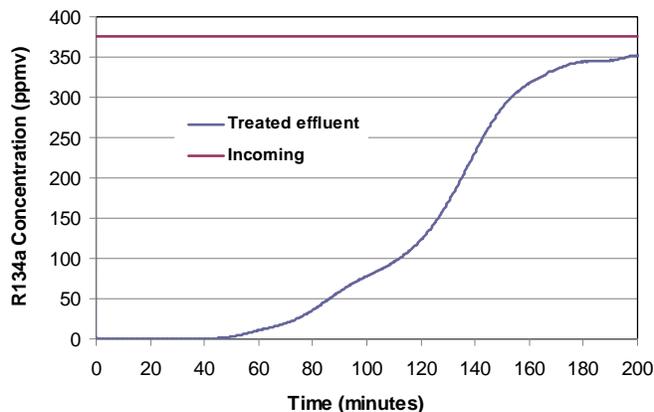
**Figure 8. Exit concentration of CO<sub>2</sub> as a function of time obtained from the preliminary sorption test of the CO<sub>2</sub> and trace contaminants prototype adsorber modules.**



**Figure 9. TIC adsorption isotherms obtained for various sorbent materials.**

isotherms obtained from various sorbent materials, including (i) activated carbon fiber cloth (ACFC) supplied by American Kynol, (ii) carbon fiber composite molecular sieve (CFCMS) developed by Oak Ridge National Laboratory (ORNL), and (iii) sorbent-coated Microlith substrate (MS5A) and insulating mesh (MS5A) developed by PCI. The results indicated that MS5A coated on insulating meshes gave the highest sorption capacity, much higher than the capacities obtained by the zeolite Y-coated insulating meshes and by ACFC. In general, the sorption capacity was higher on the coated insulating meshes than on the coated metal substrate since more sorbent materials (i.e., MS5A and zeolite Y) could be coated on the insulating meshes due to its lower open area. For a multifunctional TICs removal unit for removal of multiple TIC simulants in the dried feed air, the appropriate lengths of the Microlith and insulating layers in the “jelly roll” coil were coated with the desired amounts of the preferred sorbents (i.e., combination of MS5A and zeolite Y) to construct a mixed-bed sorbent configuration.

Figure 10 shows the performance curve (i.e., breakthrough profile) of a water removal (WR) and TIC simulants removal (CFR) adsorber set when flowing ~375 ppm<sub>v</sub> R134a (i.e., 1,1,1,2-Tetrafluoroethane as TIC simulant) in dry air at 5 cfm flow rate. Using dry air as the feed gas, the adsorber set was able to maintain practically zero ppm of R134a in the exit gas up to ~50 minutes prior to breakthrough. Based on the preliminary isotherm tests, we expect that most of the R-134a adsorption occurred in the MS5A portion due to its higher sorption capacity compared to the zeolite Y sorbent. Also, exposing the system to inlet process air consisting of R-134a at 39% RH resulted in minimal change to the performance of the adsorber modules as the upstream WR module was able to remove the water vapor from the feed air.



**Figure 10. R134a (TIC simulant) exit concentration as a function of time observed during the single sorption test of the WR and CFR adsorber set.**

#### D. Scale-up and Design of Full-scale Adsorber Modules

In order to meet the requirements of the full-scale system, PCI scaled-up and sized the required adsorbent beds for the CO<sub>2</sub> removal module and the moisture removal module based on the sub-scale test data furnished by MSFC. The full-scale sorbent bed design was similar to the subscale modules, which was based on the radial flow configuration and “jelly roll” coil sorbent bed. Subsequently, a previously developed adsorber design concept consisting of a “jelly roll” coil in a radial flow configuration was implemented. The CO<sub>2</sub> sorbent bed was scaled-up and sized to be able to perform CO<sub>2</sub> sorption for at least 30-min half cycle with an inlet CO<sub>2</sub> partial pressure of 4-5 torr at a 20-cfm process air flow rate and to provide a target CO<sub>2</sub> removal rate of 4 kg/day (equivalent to the metabolic CO<sub>2</sub> generation rate of 4 crew). The moisture sorbent bed was scaled-up and sized to be able to remove residual water vapor from 20-cfm process air at -12°C dew point.

Once the volumes of the sorbent bed for the CO<sub>2</sub> removal module and the moisture removal module were finalized, several housing configurations were developed and CFD modeling was performed using CFDdesign to evaluate the flow pattern of the process air inside the full-scale adsorber housing modules. CFD analysis was performed to determine the optimum housing design and sizing for achieving uniform reactant flow distribution in the radial flow configuration without the presence of recirculation zones. An optimum overall volumetric efficiency was also targeted by maximizing the ratio of the sorbent bed volume to the housing volume.

In this full-scale adsorber hardware design, the centerline of these radial flow adsorber modules was occupied by a 2.5" diameter feed tube; hence, based on the required sorbent bed volumes for the full-scale system, the dimensions of the CO<sub>2</sub> sorbent bed (i.e., “jelly roll” coil coated with MS5A) and the moisture removal bed (i.e., “jelly roll” coil coated with zeolite 13X) were estimated. Then, for each adsorber module, the housing dimension was optimized to achieve maximum overall volumetric efficiency with uniform reactant flow distribution and minimal recirculation zones. As a starting point in this CFD simulation effort, the housing dimension was calculated based on the required size of the sorbent bed and by setting the  $L_H/L_C$  (i.e., length of the adsorber housing per length of the sorbent bed) and  $d_H/d_C$  (i.e., diameter of the housing per diameter of the sorbent bed) to be the same with the ones implemented in the subscale housing design. The optimum adsorber housing geometries for the CO<sub>2</sub> removal module and the moisture removal module were then obtained from the CFD analysis. These dimensions were

implemented in the actual housing fabrication and adsorber assembly. By performing further CFD simulation studies, the overall volumetric efficiency (i.e.,  $V_{\text{sorbent}}/V_{\text{housing}}$ ) was increased from  $\sim 0.32$  (used in the subscale adsorber modules) to  $\sim 0.52$ . The inlet and outlet tubes outside of the adsorber housing were 1.5" diameter and the centerline feed tube diameter within the housing was 2.5" to allow room for electrode terminal fittings.

All of the CFD simulation results indicated a fully-developed laminar flow entering the adsorber module through a 2.5" centerline feed tube, followed by a radial flow of process air (with a very low velocity magnitude) from the axis to the outer diameter of the "jelly roll" sorbent bed. The results also showed good bed utilization and uniform flow distribution at the recommended housing configurations for both CO<sub>2</sub> removal and H<sub>2</sub>O removal modules.

As discussed previously, the use of the inert Microlith substrate to support the zeolite sorbents resulted in a reduced volumetric sorption capacity compared to a pellet bed. To narrow the gap in sorption capacity, an important goal was to increase the sorbent volumetric loading on the Microlith substrate and the insulating mesh (i.e., "jelly roll" sorbent coil) while maintaining good coating adhesion as well as mechanical and thermal durability. This can be done via several approaches, e.g., modifying coating formulations, optimizing washcoat application parameters, and using different supporting substrates (i.e., new insulating mesh with less open area).

To date, the overall volumetric sorbent loadings on the Microlith substrates have been increased by  $\sim 10\text{-}15\%$  with minimal effect on the coating adhesion. Furthermore, using the new insulating mesh materials (with less open area vs. the one used in the subscale development effort) and the new washcoat formulation, the overall volumetric sorbent loadings on the insulating meshes were increased by  $\sim 13\text{-}20\%$ . The results give potential benefits to the overall adsorber performance (e.g., longer adsorption time, less exposure to thermal cycles, lower overall power consumption, and size benefits) due to the higher sorbent density and thus a higher expected volumetric sorption capacity (i.e., g of CO<sub>2</sub> or H<sub>2</sub>O uptake per cm<sup>3</sup> of adsorber unit).

During the sorbent coil assembly, up to eight thermocouples were placed between the "jelly roll" coil layers for temperature readout during the actual adsorption and regeneration process. These thermocouples were placed at different radial and axial locations within the sorbent bed. After assembly of each "jelly roll" sorbent coil was completed, electrodes were installed at proper locations to enable electrical resistive heating for the sorbent regeneration process. After the "jelly roll" sorbent coils were fabricated, the assembly of the full scale adsorber modules was carried out by integrating the "jelly roll" sorbent coils into the NASA-provided aluminum housing parts. Figure 11 shows the Microlith-based regenerable adsorber module for the CO<sub>2</sub> removal after integration with the aluminum external housing parts.



**Figure 11. Full-scale Microlith-based adsorber module rated for 20 cfm process air. An 18" wooden ruler is shown to provide scale.**

#### IV. Conclusion

Ongoing efforts with NASA and DoD have shown the potential to implement sorbent-coated Microlith substrates for various sorption applications, such as water removal, CO<sub>2</sub> removal, TIC filtration, and sulfur removal. From these programs, PCI has been able to develop coating formulations and robust application methods for several different sorbents, such as MS5A, zeolite Y, and zeolite 13X, with resulting coatings that are adherent and able to withstand multiple thermal cycles. Additionally, the ability to coat different sections of Microlith mesh substrates with different zeolite/sorbent washcoats for removing different chemicals offers a system benefit for combining two or more adsorber assemblies into a single adsorber unit and for tailoring an adsorber system based on the requirements. This capability results in weight, volume, and logistic savings.

Furthermore, the ability to directly, resistively heat the Microlith substrate offers the potential for relatively rapid periodic regenerations instead of the longer thermal cycles typical of packed bed adsorbers. As a result, a regenerable adsorber using zeolite/sorbent coated on Microlith can reduce the system weight and volume compared to the conventional packed bed configurations by using more frequent regeneration cycles.

To date, PCI has designed, developed, and delivered several regenerable adsorber units with radial flow concept to NASA and AFRL. These units have been tested for water removal, CO<sub>2</sub>/trace contaminants removal, and TICs removal, and the performance results indicated good sorption capacity with the ability to apply direct internal heating for the regeneration cycle. PCI has also designed, developed, and delivered to MSFC four full-scale adsorber

modules, comprising two CO<sub>2</sub> removal modules and two moisture removal modules, rated for 20-cfm process air flow rate. The two-leg system was targeted to provide continuous removal of CO<sub>2</sub> from cabin air at a rate of 4 kg/day, equivalent to the metabolic CO<sub>2</sub> generation rate of 4 crew. Performance testing and operational validation of these modules are currently ongoing at MSFC.

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