

Adsorption System Based on Microlith[®] Technology and Its Progress in Fuel Cell, Spacecraft, and Chem-bio Warfare Defense Applications

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Abstract

The development of energy efficient, reduced-weight, regenerable adsorption systems for removal of environmental contaminants, such as CO₂ in spacecraft cabin air, chemical/biological warfare agents (CWA), and toxic industrial chemicals (TICs) is an area of continuing interest to NASA and Department of Defense, respectively. Precision Combustion, Inc. (PCI) has designed and developed a sorption technology using a bed consisting of metal meshes coated with a zeolite or molecular sieve sorbent. The metal mesh elements, trademarked and patented as Microlith[®] by PCI,¹ can be coated with various zeolite/sorbent materials and can effectively adsorb a number of contaminants of interest. The Microlith[®] elements also have the potential for direct electrical heating. 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.

Another application of this Microlith[®]-based sorption technology is for efficient hydrogen sulfide (H₂S) removal from the reactor exhaust stream in a fuel reforming-fuel cell system. PCI developed zinc oxide (ZnO) coating process on the Microlith[®] substrates with a high loading, and demonstrated that ZnO powder deposited on Microlith[®] is capable to reach its nearly full capacity with high bed utilization. Our test showed that a fresh ZnO-coated Microlith[®] system can achieve a H₂S sorption capacity of up to ~33 wt.% (i.e., 0.33 gram of H₂S uptake per gram of ZnO). This is a significant improvement from a typical H₂S sorption capacity of 10-20 wt.% obtained using commercial ZnO pellets/extrudates.

This paper describes the design, development, and optimization of the Microlith[®] sorption technology for each of the above applications. We will also discuss the results obtained from various sorption tests, such as using CO₂, water, and trace chemical contaminants for NASA cabin air cleaning/Environmental Control and Life Support System (ECLSS) application as well as using TICs for chem-bio warfare defense application. Additionally, the development of this technology for sulfur (e.g., H₂S) removal in reforming/fuel cell application will be addressed.

Introduction

Adsorption processes have been of interest in many application fields, such as filtration of environmental contaminants, cabin air revitalization in spacecraft and spacestation, and adsorption of toxic industrial chemicals (TICs). For example, adsorber units have been

used for cabin air quality control on all crewed spacecraft, and are expected to continue to remain at the forefront of spacecraft cabin air quality control technologies. As mission durations increase and exploration goals reach beyond Earth orbit, the need for regenerable adsorption processes for continuous removal of CO₂ and trace chemical contaminants becomes paramount. In another application, a regenerable adsorber system can be implemented in fielded military individual and collective protection systems against chemical/biological warfare agents and toxic industrial chemicals.

NASA-supported research at PCI has demonstrated that zeolites deposited on Microlith metal mesh elements can effectively adsorb a number of the contaminants of interest, such as CO₂, ethanol, and ammonia. The inert Microlith substrates and the use of a binder during deposition of the zeolites on them result in volumetric sorbent loadings that are considerably lower than the conventional carbon bed and packed bed systems. However, the unique ability to *directly resistively heat* the Microlith metal mesh support allows for rapid periodic regenerations via direct internal heating.^{2,3} Therefore, the weight and volume of the conventional adsorber subassemblies can potentially be reduced by implementing zeolites supported on Microlith and by employing periodic sorbent regeneration.

Furthermore, 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 application. This capability allows for weight, volume, and logistic savings. For example, in the NASA program, we have fabricated and delivered an integrated adsorber unit capable of removing both CO₂ and trace chemical contaminants within the same bed by coating two sections of the Microlith with two different zeolites. Both the trace contaminant control system (TCCS) and carbon dioxide removal assembly (CDRA) performance was demonstrated within the volume envelope of the current CDRA unit, thus eliminating the need for the current TCCS unit entirely. Using a similar design concept, we designed, fabricated, and delivered adsorber systems for removing TICs to Air Force Research Laboratory (AFRL).

We recently examined the performance of zinc oxide (ZnO) powder coated on Microlith[®] substrates for removing sulfur, such as hydrogen sulfide (H₂S), from the product stream of fuel processors. To date, on-board fuel processing of liquid fuels, such as diesel and JP-8, has been investigated by many research groups and of interest to both government and industry as a potential option to supply hydrogen for either polymer electrolyte membrane (PEM) or solid oxide fuel cell stacks for auxiliary power unit (APU) systems.⁴⁻⁹ The sulfur compounds from the logistic fuels, however, can poison both the fuel reformer catalyst and the fuel cell catalyst. For example, the sulfur level in the H₂-rich stream needs to be less than 100 ppb_v prior to entering a typical PEM fuel cell stack.⁴ Therefore, sulfur compounds from the fuels have to be removed before the H₂-rich stream is sent to the fuel cell stacks. Depending on the application and the sulfur concentration in the logistic fuels, the sulfur removal can be performed either in the liquid phase (i.e., prior to the fuel reformer units) by implementing liquid-fuel sulfur sorbent materials or in the gas phase by passing the reformat stream through a ZnO-based unit.¹⁰⁻¹⁵ The results from our sulfur tests showed that the ZnO-coated Microlith substrates were able to reach nearly full ZnO capacity with high bed utilization while maintaining a low pressure drop across the bed.

Microlith[®] Technology

The Microlith technology consists of a series of ultra-short-channel-length, low-thermal-mass metal meshes as shown in Figure 1. It replaces the long channels of conventional monoliths with a series of short-channel-length substrates. Whereas in a conventional honeycomb monolith a fully developed boundary layer is present over a considerable length of the device, the very short channel length characteristic of the Microlith substrate avoids boundary layer buildup. Since heat and mass transfer coefficients depend on the boundary layer thickness, avoiding boundary layer buildup enhances transport properties. The Microlith can also pack more active surface area into a small volume, providing increased adsorption area for a given pressure drop. The effectiveness of the Microlith technology and the long-term durability of PCI's proprietary zeolite and catalyst coatings have been rigorously demonstrated in applications such as exhaust after-treatment,¹⁶ space station cabin air cleaning,^{2,3,17,18} catalytic combustion,¹⁹ and fuel processing.²⁰⁻²³

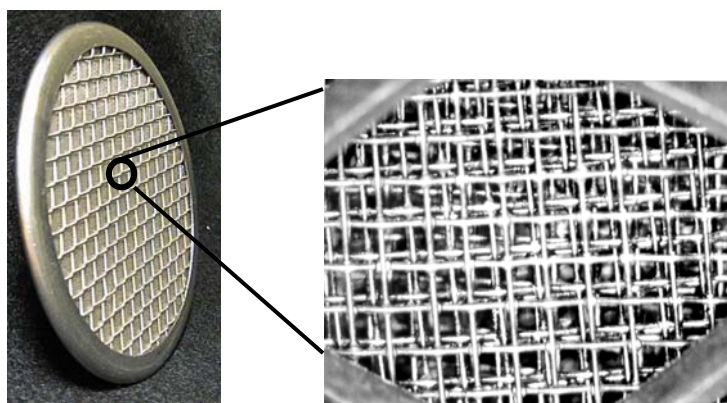


Figure 1. Example of a Microlith substrate assembly.

During the zeolite and other sorbents coating development, several washcoat formulations were evaluated in an effort to produce an adherent coating. The Microlith substrate geometry poses unique challenges in the development of adherent coatings. The washcoats must be easily applied, and the resulting coatings must have a high degree of adhesion and cohesion and must be sufficiently abrasion resistant in order to withstand routine handling and multiple thermal cycles. At the same time, the formulation must retain the desired chemical and physical characteristics of the zeolite and ZnO. The washcoats were applied to the Microlith substrates using methods developed at PCI that allow for rapid application of sorbent coatings to large volumes of substrate. Scanning electron microscopy (SEM) micrographs of the sorbent-coated Microlith substrates are shown in Figure 2 and Figure 3. SEM analyses indicated uniform coatings with complete coverage. Furthermore, the cross-sectional SEM view (Figure 3) indicated that the coating was adherent and that the thickness of the coating was relatively uniform on the surface of the substrate. A few cracks were observed in the coating layer due to cutting of the Microlith substrate prior to SEM cross sectional analysis.

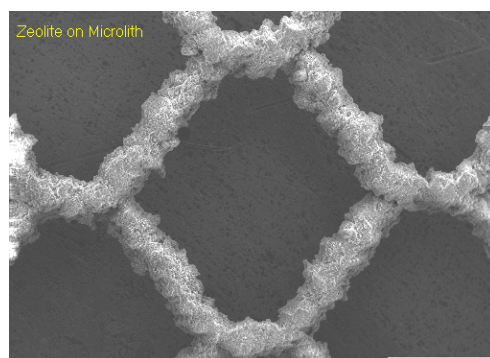


Figure 2. Surface-scan SEM micrograph of sorbent-coated Microlith.

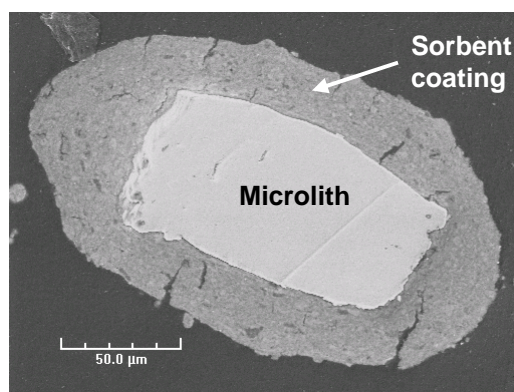


Figure 3. Cross sectional SEM micrograph of the sorbent-coated Microlith substrate.

Regenerable Adsorber Units Design and Fabrication

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 mesh serves as an insulating layer 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.^{3,24} 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 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 Figure 4. 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.

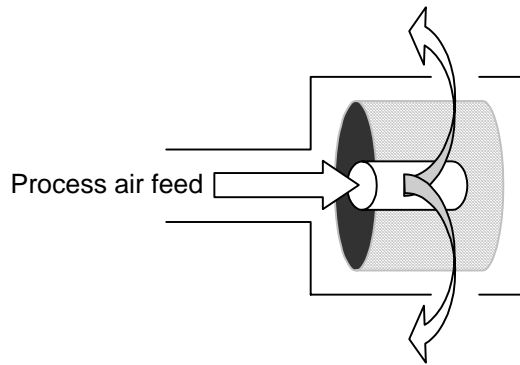


Figure 4. A simplified Microlith[®]-based radial flow adsorber design consisting of a “jelly-roll” coil of sorbent-coated Microlith screens and sorbent-coated insulating meshes.

Figure 5 shows detailed 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 AFRL. A computational fluid dynamic (CFD) simulation software package 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).

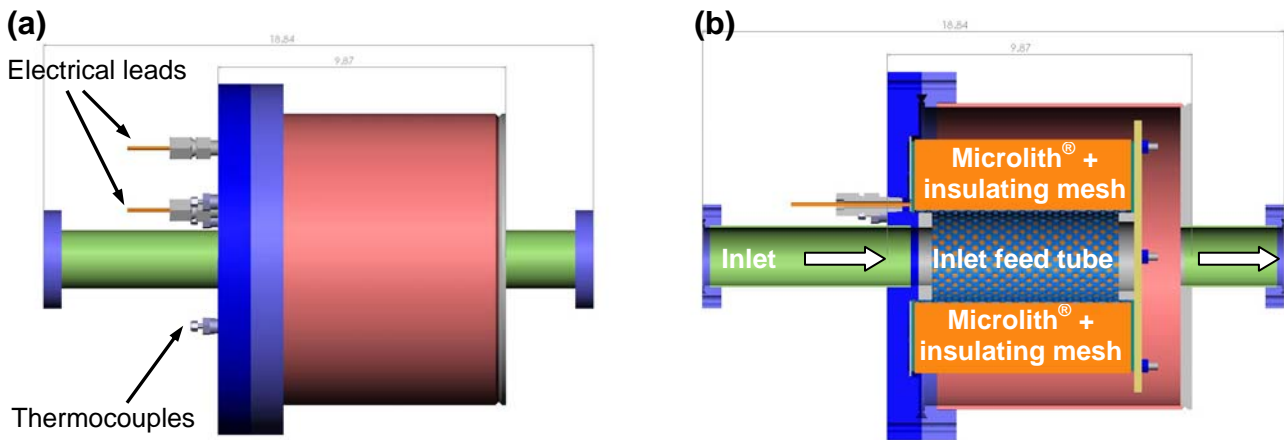


Figure 5. (a) External and (b) Internal cross-section views of the Microlith[®]-based radial flow adsorber design concept.

Finally, Figure 6 shows the Microlith-based regenerable adsorber unit after integration with the external housing. The adsorber unit was connected both to a vacuum line for the regeneration process and to a process air feed line for the sorption test.



Figure 6. Microlith[®]-based regenerable adsorber unit rated for 5-cfm flow rate after integration with the external housing.

Results and Discussion

(i) H₂O, CO₂, and trace chemical contaminants sorption for NASA cabin air/ECLSS application

Recent development testing supported by NASA successfully demonstrated “proof of concept” as well as a one-person prototype demonstration of coated adsorbents on the Microlith substrate for a regenerable CO₂ removal and trace contaminants control (TCCS) application. The adsorber unit was exposed to a contaminant-laden airflow containing ethanol, acetone, toluene, ammonia, and CO₂, and the adsorption capacity was evaluated via multiple sorption/regeneration cycles up to at least 500 hours of operation. For a dual function CO₂ removal/TCCS unit, the appropriate lengths of the Microlith and insulating layers in the “jelly roll” coil were coated with the desired amounts of the preferred sorbents as determined from bench-scale tests.

This adsorber unit was targeted to operate at an approximate nominal contact time of ~1.8 sec. This corresponded to a volumetric flow rate of ~5 cfm through the annular sorbent-coated coil volume of 4235 cm³, which was the total volume of the “jelly roll” coil (CO₂ and trace contaminant removal segments). Part of the coil volume was devoted to CO₂ sorption by coating the appropriate length of the Microlith substrate and the insulating mesh with Molecular Sieve 5A (MS5A), and the remaining part of the coil was designated for the trace contaminants removal. The TCCS sorbents coating was used for the removal of ethanol, acetone, ammonia, toluene, and dichloromethane (DCM). This design was estimated to be sufficient for maintaining the exit concentration of the trace contaminants significantly below their inlet levels during the sorption cycle, whereas the regeneration requirement of the CO₂ removal segment determined the cycle time for the adsorber unit.

Prior to testing, the adsorber unit was heated under vacuum until the lowest registering thermocouple reached ~230-240°C. The total flow rate of the feed process air was ~5 cfm, and the component concentrations were 3500 ppm CO₂, 500 ppm ethanol, 10 ppm NH₃, 11 ppm acetone, 8 ppm toluene, and 5 ppm DCM. During the regeneration process, the electrical lead terminals (Figure 5a) were connected to a wall AC power supply regulated by a

variac. Power requirements were examined for direct electrical heating of the “jelly roll” coil based on estimates of the total energy required to heat the mass of sorbent washcoats and to supply the heat of desorption (dominantly that of CO₂, but also that of the trace contaminants). Assuming a 45-minute sorption/regeneration cycle, ~110–150 watts would be required depending upon the final target regeneration temperature (230–300°C).

Figure 7 represents the results from the first three prototype testing cycles. The data in the figure indicate poor performance but improving from cycle to cycle, suggesting the need for a better process to remove moisture and/or other sorbed species initially to establish a dry, clean surface effective for sorption. After the fourth cycle, the adsorber unit was regenerated more aggressively by flowing dry nitrogen at ~5 slpm through the unit for ~4.5 hours at the regeneration temperature. After cooling the unit in a dry nitrogen flow to room temperature, sorption cycle 5 was then performed and the results are shown in Figure 8.

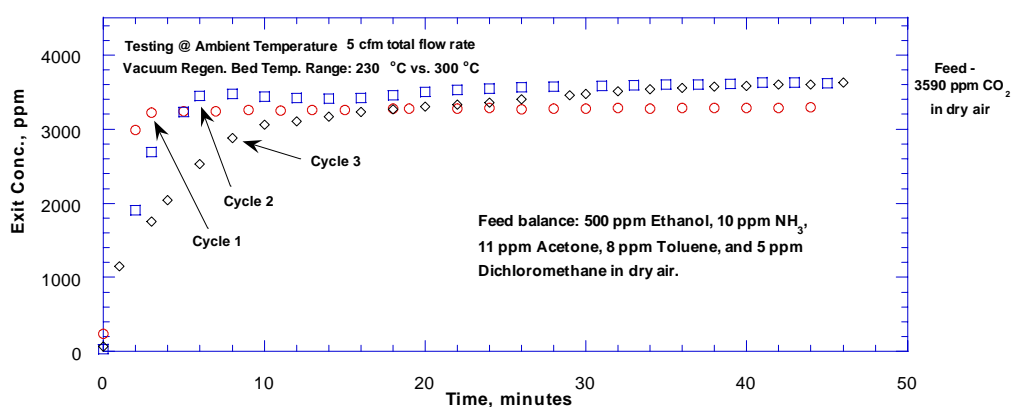


Figure 7. Cycle-to-cycle variation of the CO₂ exit concentration as a function of time observed during the performance test of the CO₂/trace contaminants removal unit.

The results from sorption cycle 5 in Figure 8 indicated 3 wt% CO₂ sorption capacity based on the MS5A washcoat (i.e., 0.03 gram of CO₂ sorbed per gram of MS5A washcoat), and a 45-50 minute cycle time during which ~55% of the delivered CO₂ was adsorbed. The marked improvement in CO₂ sorption capacity compared with the first three cycles underscored the need to establish the correct initial sorbent surface conditions and dryness to achieve proper performance. During this cyclic test, the concentrations of trace contaminants in the exit gas at the end of each cycle were well below their inlet levels. With the exception of ethanol, the exit concentrations of trace contaminants were zero. The ethanol exit level was only 5 ppm 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. Measurements indicated that the pressure drop of the unit was <0.5 inch of water at 5-cfm process air flow rate. Furthermore, the sorbent coatings on both the Microlith substrate and the insulating mesh showed good adhesion and durability after multiple thermal cycles during the sorption and regeneration cyclic test.

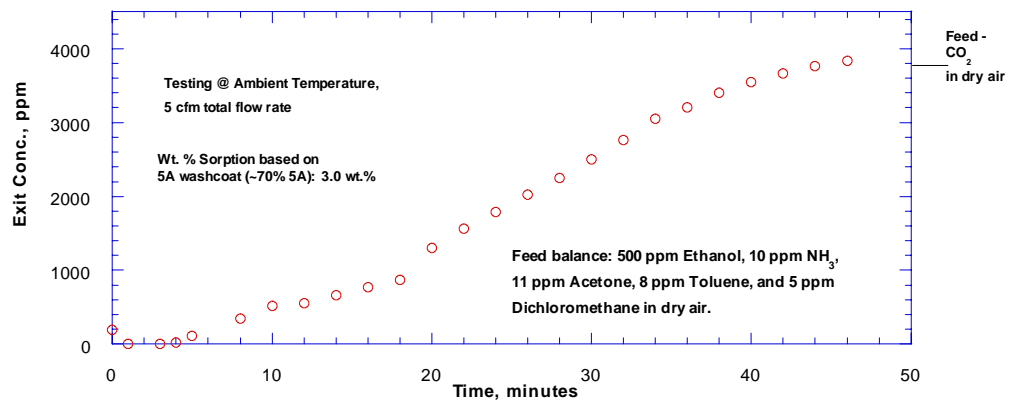


Figure 8. Exit concentration of CO₂ as a function of time obtained from the sorption cycle 5 test of the CO₂/TCCS prototype unit. Cycle 5 of the sorption test was performed following a more aggressive regeneration process to ensure a dry, clean surface effective for sorption.

In the current CO₂ removal assembly located in the ISS, the process air is passed through a water removal unit prior to the CO₂ and trace chemical contaminants removal. Drying of the process air is desirable in order to mitigate the effect of water co-adsorption on the CO₂ and trace chemical contaminants adsorption process and to collect the water to be returned to the cabin for recycling and crew use. PCI has recently developed a Microlith-based regenerable water removal unit by coating both the Microlith substrate and the insulating mesh with zeolite 13X.

Figure 9 shows the results obtained from the cyclic sorption test performed at PCI (Figure 9a) and from a single water sorption test performed at NASA (Figure 9b) using 5-cfm process air feed at room temperature. The water vapor inlet concentration for the cyclic test (Figure 9a) was ~12,100 ppm_v (50% RH, 21°C, 1 atm). At the end of each cycle, the water removal unit was regenerated for 20 minutes at 200°C by applying direct resistive heating while flowing dry air at 20 slpm. The subsequent sorption test was performed after the unit was cooled to room temperature by flowing dry air at 5 cfm. The results indicated that the sorption capacity remained unchanged at 4.2 wt.% (based on the zeolite 13X washcoat) after four sorption cycles with 20-minute cycle time. For the water sorption test performed at NASA, the water vapor inlet concentration was ~8300 ppm_v (34% RH, 21°C, 1 atm). Although the sorption test at NASA was performed prior to a prolonged regeneration process in dry nitrogen, the unit still gave a good performance by removing ~95% of water vapor in the first 17 minutes and was able to maintain the water removal at more than 80% in the remaining 45-minute test.

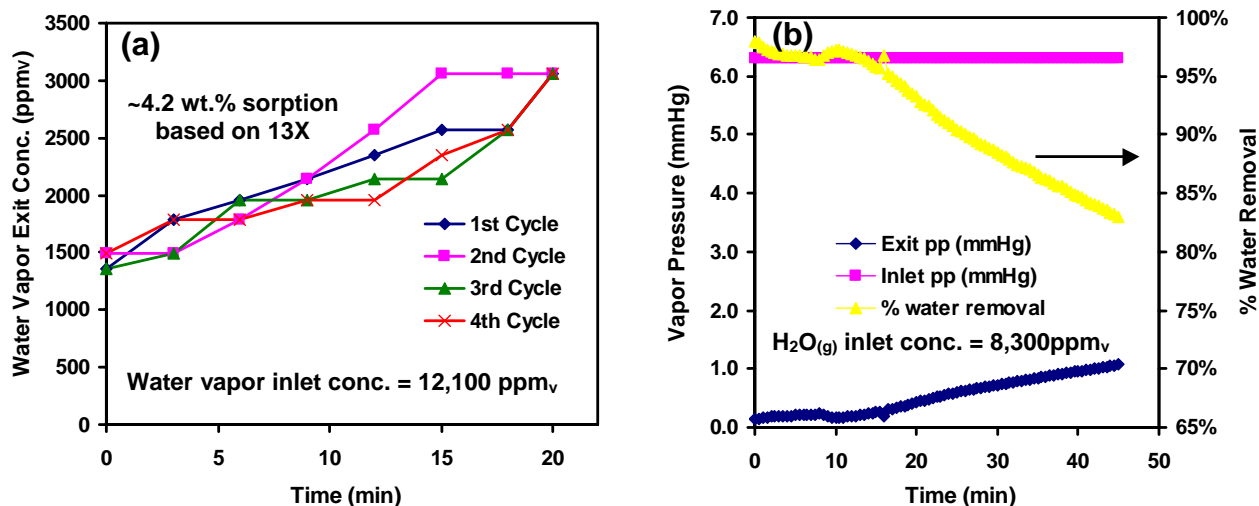


Figure 9. (a) Cycle-to-cycle variation of water vapor exit concentration as a function of time obtained from the cyclic test and (b) Inlet and exit water vapor pressure as well as the % water removal as a function of time from a single water sorption test.

(ii) TICs sorption for chem-bio defense application

Four prototype adsorber units with a design concept similar to that of the NASA 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 and two units for removing TICs. Two sets of filters working in tandem (i.e., an ESA-based system) are used. Process air will first be 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 10 shows the preliminary results of TIC adsorption 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 and insulating mesh developed by PCI. Based on the % weight change results for each sorbent material shown in Figure 10a, we estimated the volumetric sorption capacity (lb of TIC sorbed per ft³ of sorbent) for each material (Figure 10b). 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. For a dual-function TICs removal unit, the appropriate lengths of the Microlith and insulating layers in the “jelly roll” coil were coated with the desired amounts of the preferred sorbents. Currently, sorption cyclic tests are being performed at AFRL to evaluate the TICs sorption capacity and to compare the Microlith-based regenerable adsorber performance with the other carbon-based sorbent systems over multiple sorption/regeneration cycles.

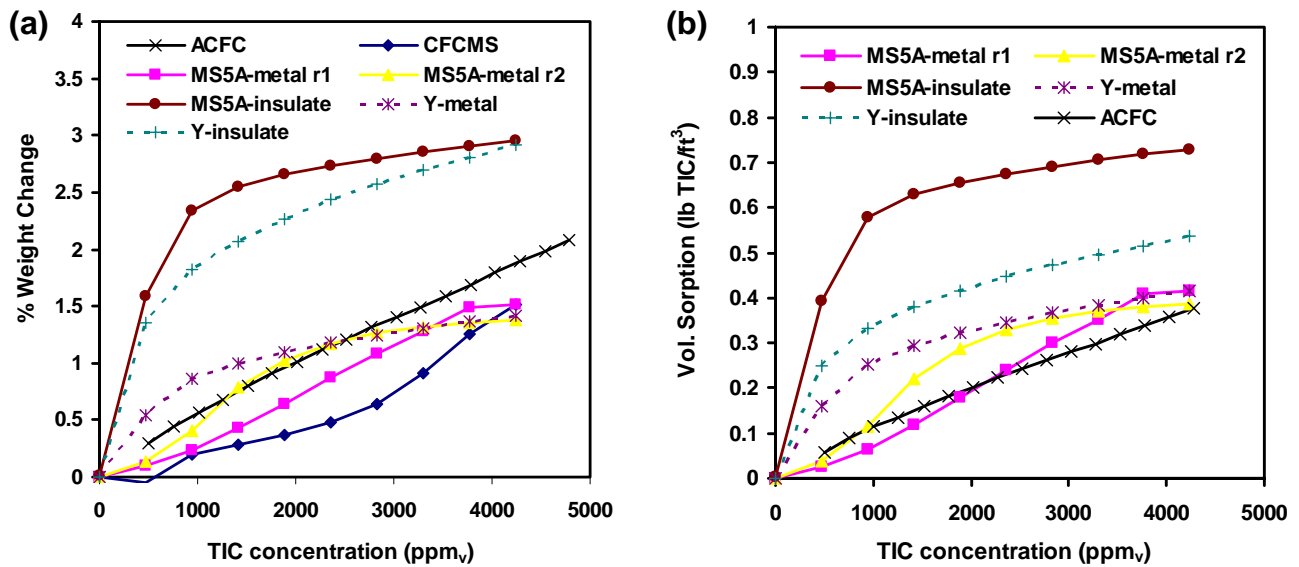


Figure 10. TIC adsorption isotherms: (a) % weight change and (b) volumetric sorption capacity (lb of TIC sorbed per ft³ of sorbent) as a function of TIC concentration obtained for various sorbent materials.

(iii) H₂S sorption for fuel reforming-fuel cell application

Recently, PCI has developed a formulation and an application method for coating the ZnO powder on PCI's Microlith substrate. We then performed H₂S sorption tests on the ZnO-coated Microlith substrate to evaluate its sulfur sorption capacity, regenerability, and cycle-to-cycle variation. The sulfur sorption testing employed a linear stack of ZnO-coated Microlith screens, which was inserted into a stainless steel tube.

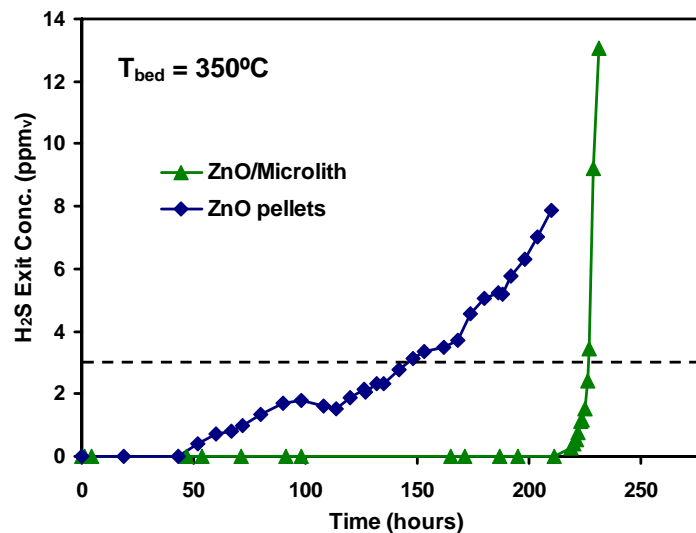


Figure 11. H₂S exit concentration as a function of time obtained from two H₂S sorption tests. The H₂S inlet concentration was 115 ppm_v. The H₂S breakthrough level (3 ppm_v) is indicated by a dashed line.

Figure 11 shows the H₂S outlet concentration as a function of time obtained from the H₂S sorption tests on: (i) ZnO coated on Microlith substrate and (ii) ZnO pellets bed under the same operating conditions (Table 1). In these tests, a surrogate gas mixture containing 115 ppm_v H₂S was flowed into the ZnO beds. The temperature of the beds was maintained at 350°C. The surrogate gas was a blend of 23.4 mole % H₂, 16.4% CO, 19.9% H₂O, and 40.3% N₂, which simulates PCI's ATR reformat stream composition when operating with 500 ppm_w sulfur fuel. For the ZnO pellets experiment, the ZnO bed was 1/8 inch long ZnO pellets packed in a stainless steel tube, 1 inch in diameter and 1.01 inch long. In these tests, the concentrations of H₂S at the inlet and outlet streams were measured using a gas chromatograph (GC) equipped with a flame photometric detector (FPD) and were checked periodically using Drager tubes.

Table 1. Detailed test conditions and the resulting H₂S sorption capacities for H₂S sorption tests on: (i) ZnO-coated Microlith and (ii) ZnO pellets.

	ZnO/Microlith	ZnO pellets
Gas composition (mole %)	23.4% H ₂ , 16.4% CO, 40.3% N ₂ , 19.9% H ₂ O	23.4% H ₂ , 16.4% CO, 40.3% N ₂ , 19.9% H ₂ O
Total gas flow rate	0.633 slpm	0.880 slpm
V _{eff} or V _{bed}	V _{eff} = 0.57 in ³ = 9.4 cm ³ V _{bed} = 0.90 in ³ = 14.7 cm ³	V _{bed} = 0.78 in ³ = 13.0 cm ³
Bed Temperature	350°C	350°C
ZnO weight	4.011 gm	17.974 gm
Inlet H ₂ S concentration	Drager analysis: 115 ppm _v	Drager analysis: 115 ppm _v
H ₂ S uptake	1.405 gm	1.248 gm
Breakthrough time	227 hours	146 hours
Wt.% H₂S sorption (3 ppm_v breakthrough)	33.0 wt.% (based on ZnO)	6.95 wt.% (based on ZnO pellets)
Vol. sorption capacity (gm H₂S / V_{bed})	0.096 gm/cm³	0.096 gm/cm³

The resulting weight percent H₂S sorption capacity for the ZnO/Microlith was ~33.0 wt.% based on the ZnO (i.e., 0.33 gram of H₂S uptake per gram of ZnO). This is almost five-fold higher capacity than the ZnO pellets capacity obtained at the same operating conditions. The H₂S breakthrough occurred after ~227 hours of continuous exposure to 115 ppm_v H₂S. This high wt.% sorption capacity may be due to the presence of well-dispersed ZnO particles on the surface of Microlith, and thus the ZnO active surfaces are more accessible for the reaction with H₂S. The volumetric sorption capacity for this ZnO/Microlith system based on the bed volume was 0.096 gram H₂S uptake/cm³, which is expected to be lower than the volumetric sorption capacity for ZnO pellets systems due to the presence of inert Microlith substrate. However, the unique ability to *directly resistively heat* the Microlith metal mesh

support allows for rapid periodic regenerations via direct internal heating, which can potentially reduce the weight and volume of the H₂S removal beds.

The most recent H₂S sorption test on ZnO pellets indicated a breakthrough after 146 hours of continuous exposure to 115 ppm_v H₂S, resulting in a H₂S sorption capacity of 6.9 wt.% based on the ZnO pellets (i.e., 0.069 gram of H₂S uptake per gram of ZnO pellets). This corresponds to a volumetric sorption capacity of 0.096 gram H₂S uptake/cm³, which was surprisingly low and was the same as the capacity for the ZnO/Microlith bed. More experiments still need to be performed on these beds to evaluate and to compare their performance.

Summary

Ongoing efforts with NASA and DoD have shown the potential to implement sorbent-coated Microlith substrates for various sorption applications, such as water removal, CO₂ 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 allows for 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 a more periodic regeneration. Since these programs were primarily a science- and technology-based discovery effort, there was limited scope for rigorously exploring economic feasibility and long-term performance.

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, CO₂/trace contaminant removal, and TICs removal, and the performance results indicated good sorption capacity with the ability to apply direct internal heating. Additionally, we have further developed this Microlith-based sorption technology for efficient sulfur removal (i.e., H₂S) in the reforming/fuel cell application. The results from our sulfur tests showed that the ZnO-coated Microlith substrates were able to reach nearly full ZnO capacity with high bed utilization while maintaining a low pressure drop across the bed.

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