

Microlith Based Sorber for Removal of Environmental Contaminants

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ABSTRACT

The development of energy efficient, lightweight sorption systems for removal of environmental contaminants in space flight applications is an area of continuing interest to NASA. The current CO₂ removal system on the International Space Station employs two pellet bed canisters of 5A molecular sieve that alternate between regeneration and sorption. A separate disposable charcoal bed removes trace contaminants.

An alternative technology has been demonstrated using a sorption bed consisting of metal meshes coated with a sorbent, trademarked and patented [1] as Microlith[®] by Precision Combustion, Inc. (PCI); these meshes have the potential for direct electrical heating for this application. This allows the bed to be regenerable via resistive heating and offers the potential for shorter regeneration times, reduced power requirement, and net energy savings vs. conventional systems. The capability of removing both CO₂ and trace contaminants within the same bed has also been demonstrated. Thus, the need for a separate trace contaminant unit is eliminated resulting in an opportunity for significant weight savings. Unlike the charcoal bed, zeolites for trace contaminant removal are amenable to periodic regeneration. This paper describes the design and performance of a prototype sorber device for simultaneous CO₂ and trace contaminant removal and its attendant weight and energy savings.

INTRODUCTION

Adsorption processes occupy an important niche in spacecraft environmental control and life support (ECLS) systems. Primary applications for adsorption processes exist in the area of cabin air quality control. Since the beginning of crewed space exploration, adsorption processes have been at the forefront for ensuring that cabin air is suitable for the crew to breathe by removing trace chemical contaminants and CO₂. As such, adsorption processes have been on board all crewed spacecraft – both U.S. and Russian. [2] It is anticipated

that adsorption processes will 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 becomes paramount.

The ability to remove trace chemical contaminants (e.g. alcohols, ketones, aromatics, CO, halocarbons, hydrocarbons, and ammonia) from cabin air is a necessary aspect of spacecraft life support systems such as that employed on board the International Space Station (ISS). Currently, this trace contaminant control system (TCCS) requirement is met on the ISS U.S. On-orbit Segment (USOS) by employing a 50 lb. bed of specially treated activated carbon and thermal catalytic oxidation. The fixed activated carbon bed is not regenerated. Due to its long life (>2 yrs.), it is simply replaced periodically. [3, 4]

NASA-supported research at PCI has demonstrated that zeolites deposited on Microlith metal mesh elements could effectively adsorb a number of the contaminants of interest. The inert Microlith substrate and the use of a binder during deposition of the zeolites on them results in volumetric sorbent loadings considerably lower than the carbon bed. However, the ability to directly resistively heat the metal mesh support offers the potential for relatively rapid periodic regenerations. Potentially therefore, the weight and volume of the current trace contaminant control subassembly (TCCS) could be reduced by as much as 75% by implementing zeolites supported on Microlith and employing periodic sorbent regeneration. The development of such a system as an alternative to the activated carbon in the TCCS was the early emphasis of the NASA's interest in the technology.

However, since cabin air is fed directly to the TCCS, humidity would likely have a significant negative impact on the performance of the zeolite sorbents. Drying agents are used to mitigate the effect of humidity on sorbent effectiveness and comprise a significant unit operation within the current carbon dioxide removal assembly (CDRA).

The CDRA, located in the ISS USOS, employs four fixed beds containing materials that first dry the process air and then remove the CO₂ in separate unit operations. The separate unit operations provide two benefits. First, the effects of water coadsorption on the CO₂ adsorption process are mitigated. Second, the water is returned to the cabin for recycling and crew use thus minimizing fresh water resupply requirements. The four beds alternate between adsorption and desorption cycles. Explained simply, the process air enters the adsorbing desiccant bed containing silica gel and zeolite 13X to remove residual moisture. Next, this very dry process air flows through the CO₂ adsorption bed that contains zeolite 5A. The air exiting the CO₂ adsorption bed then flows through the regenerating desiccant bed, entraining moisture, and exhausts to the cabin. The second CO₂ adsorption bed is heated and exposed to the vacuum of space to regenerate it. The flow alternates between the beds on a regular cycle. More details on the CDRA process may be found in Reference 5.

Upon studying the ISS's TCC and CO₂ removal process designs, it was decided to couple them in such a way to permit the CDRA's desiccant beds to mitigate the negative impact of humidity on the Microlith-based TCC system.

An extension of the CDRA/TCCS integration approach was the decision to pursue the possible replacement of the CDRA's packed bed for CO₂ removal with Microlith-supported 5A zeolite sorbent. Although the volumetric sorbent loadings on the Microlith are considerably lower than the 5A pellet bed (~30%), in conjunction with resistive heating of the metal mesh support, it nevertheless permits the potential for faster periodic regenerations. Engineering evaluation indicates that successfully integrating both the CO₂ removal and TCC processes using Microlith supported sorbents within the volume envelope of the current CDRA unit could eliminate the current TCCS unit entirely with attendant weight, volume, and logistics savings. Depending upon the design details and regeneration requirements, the integrated system potentially could offer power savings as well versus the current CDRA. This conceptual configuration is illustrated in Figure 1.

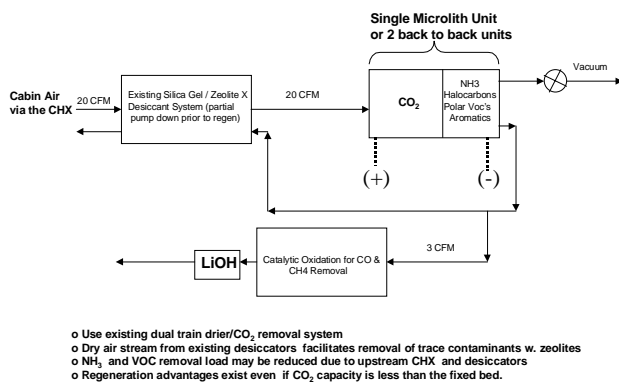


Figure 1: Conceptual schematic of CO₂/TCCS system

MICROLITH CATALYST TECHNOLOGY:

Microlith technology is a novel reactor engineering design concept consisting of a series of ultra-short-channel-length, low thermal mass metal monoliths as shown in Figure 2 below. This technology replaces the long channels of a conventional monolith 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. Also, the Microlith design can pack more active area into a small volume, providing increased adsorption area for a given pressure drop. Proprietary catalyst coating techniques have also been developed and the technology has been rigorously demonstrated in applications such as exhaust aftertreatment [6], space station air cleaning [7, 8], fuel processing [9] and catalytic combustion [10].

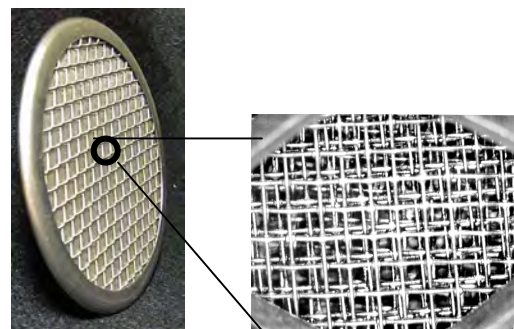


Figure 2: Example of a Microlith substrate assembly.

EXPERIMENTAL DETAILS:

TEST SETUP: A 2.75" diameter flanged housing was connected to an upstream plenum section through which dry air or nitrogen at room temperature flowed at the desired rate (typically 2 – 6 SLPM). A blend of the liquid phase trace contaminants in the appropriate relative proportions was introduced into this stream via a syringe pump operating at the proper rate to establish the desired concentrations in total flow. An NH₃/N₂ stream at the appropriate rate to establish the desired contaminant level was also introduced through a perforated circular tube just upstream of the housing containing the sorber elements.

TRACE CONTAMINANTS: Although a broad spectrum of contaminants (~125) have been identified for this application, only 18 of them represent ~88% of the total design load. Among them the key adsorption design drivers are acetone, dichloromethane (DCM), ammonia and ethanol [11]. Though a variety of single and multi component tests were performed, the "standard" multi component competitive sorption run employed 5 model compounds representing the component classes of greatest interest and delivered at concentrations

corresponding to 1/2 their spacecraft maximum allowable concentration (SMAC). The exception to this was NH₃, which was run at full SMAC to facilitate analysis. Thus, concentration in the “standard” runs at a total air flow of 5.9 LPM were: 500 ppm ethanol, 11 ppm acetone, 5 ppm DCM, 8 ppm toluene, and 11 ppm NH₃.

In all cases, exit concentrations were monitored as a function of run time, and the test was terminated when the exit concentration closely approached that of the inlet. A GC was used to measure the outlet concentrations of all components with the exception of NH₃, which was determined via chemiluminescence after catalytically converting it to NO_x.

CARBON DIOXIDE: Using premixed gas standards, Carbon dioxide was delivered at 3000 – 5000 ppm in air or nitrogen at 2 – 6 SLPM. “Standard” testing employed 3500 – 3900 ppm CO₂ (~1/2 SMAC) in dry air at 5.9 SLPM. A GC was used to monitor exit concentrations.

SORBENTS: Three zeolite sorbents supported on Microlith substrates were examined -- 50/1 SiO₂/Al₂O₃ HZSM-5 (Zeolyst CBV 5524G) and a 5.1/1 SiO₂/Al₂O₃ Y zeolite (Zeolyst CBV 400) for trace contaminant removal and 5A molecular sieve (calcium form of A) for CO₂ sorption. In addition, 5A molecular sieve supported on a woven glass fiber mesh was also examined. Typically, the washcoat contained ~ 60 – 80% zeolite and correspondingly 40 - 20% binder.

UPTAKE: In all cases, the uptake of a component was determined by the difference between the total amount of a component delivered over the course of the test and the area under its breakthrough curve to the corresponding time on stream.

SORBENT REGENERATION: For the scoping experiments described here, the mesh elements were not resistively heated. Instead, the housing containing the sorbent was isolated using flanged end caps with provision for a connection to vacuum and an external heater wrapped around the outside wall of the housing. Regeneration typically involved a pump down followed by heating to 200 – 230 °C under vacuum with a subsequent 2 – 2.5 hr hold interval.

TRACE CONTAMINANT TESTING:

AMMONIA SORPTION: Because NH₃ can be troublesome, several tests were conducted to assess its sorption behavior alone, the adequacy of the testing methodology, and the effectiveness of zeolites as candidate sorbents. Figure 3 illustrates data generated using the methods described above.

For a 50/1 SiO₂/Al₂O₃ ZSM-5 sample, the total sorption exceeds that expected based solely on the formation of NH₄⁺ ions associated with the tetrahedral framework Al, and therefore physisorption apparently contributes as well. 35-40% of the first cycle performance uptake could be attributed to the formation of NH₄⁺ ions and the

balance to physisorption. Second cycle sorption was ~70% of the first cycle which is consistent with the vacuum regeneration procedure completely restoring physisorption capacity but only partially restoring the ion exchange contribution (<20%). The close correspondence of the second and third cycle performance indicates that the vacuum regeneration treatment can reproducibly accomplish these full and partial restorations. Second and subsequent cycle sorption performance was considered to be adequate for achieving the low levels of NH₃ targeted.

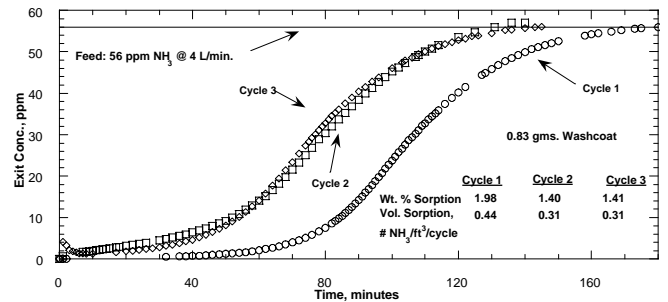


Figure 3: NH₃ Uptake - 50/1 SiO₂/Al₂O₃ ZSM-5 on Microlith @ 25 °C.

Similar experiments were also performed on Zeolite Y (CBV-400) supported on Microlith with differences between first and subsequent cycle performances evident for the same reasons discussed above. Second cycle performance for zeolite Y was comparable to that of second or third cycle performance of ZSM-5 on a weight % sorption basis. Higher volumetric sorption resulted with Zeolite Y due to the fact that the Microlith screens were more heavily loaded with washcoat. Thus, at similar loading, comparable performance would be expected by all measures for Y and ZSM-5 when sorbing only NH₃. These results illustrate the potential effectiveness of zeolites for removal of trace amounts of NH₃. They also indicate that observed first cycle NH₃ sorption levels can be misleadingly higher than that for subsequent cycles. Therefore, in the subsequent competitive sorption experiments (see below), the first cycle performance of zeolite loaded Microlith elements was regarded as a “pre-conditioning” cycle and second cycle performance was used to assess sorption behavior. Alternatively, the fresh sample was “pre-ammoniated” and vacuum regenerated prior to the first cycle testing.

COMPETITIVE SORPTION: Experiments were performed using an air stream containing 500 ppm ethanol, 11 ppm acetone, 5 ppm dichloromethane, 8 ppm toluene, and 11-13 ppm NH₃. These concentrations correspond to the half-SMAC levels for all constituents except NH₃, which, as noted above, was run at full-SMAC to facilitate analysis. Experiments were conducted in dry air in anticipation of implementation in the CDRA housing behind the CO₂ sorbent where dry conditions exist due to an upstream drier.

Because of the concentration disparity between ethanol and the other components, two types of experiments

were performed for each zeolite (Y and ZSM-5) to evaluate their competitive sorption behavior. First, to judge the sorption behavior of the lower concentration components in the presence of the large amount of ethanol, a small number of screens was employed such that ethanol broke through and equalized inlet and outlet concentrations fairly rapidly. For the rest of the components, breakthrough and the approach of the exit concentration to that of the inlet was slower and could be observed more accurately and in a practical amount of testing time. Second, to judge the sorption behavior of ethanol in the presence of the lower concentration components, a larger number of screens was employed such that ethanol breakthrough and approach of its exit concentration to that of its inlet level was slowed and could be observed more accurately and in a practical amount of testing time. In this case, with the exception of DCM, breakthrough for the much lower concentration components was slower and the approach of the exit concentration to that of the inlet was not fully achieved over the course of the test. Because of DCM's relatively poor sorption, breakthrough and the equalization of inlet and outlet concentrations could be observed when testing both the smaller and larger number of screens. Figures 4, 5 and 6 below illustrate the results for ZSM-5. Similar experiments were performed with zeolite Y.

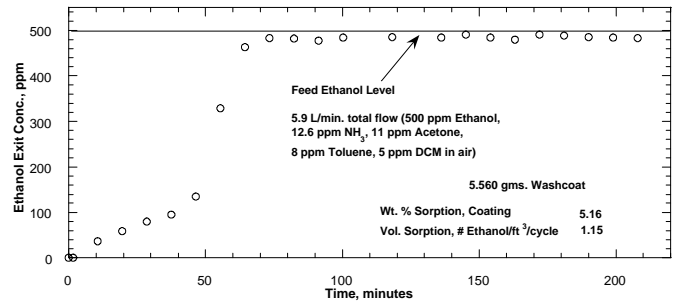


Figure 6: Competitive Ethanol Sorption - 50/1 SiO₂/Al₂O₃ ZSM-5 on Microlith @ 25 °C

Table 1:

Component	Zeolite Y (CBV-400)	ZSM-5 (CBV-5524)
	Amount Sorbed	Amount Sorbed
	wt % washcoat	wt % washcoat
Ethanol	9.55	5.16
Ammonia	0.60	0.15
Acetone	0.52	0.39
Toluene	0.19	>0.71
Dichloromethane	~0 - 0.035	0.09 - 0.2

Compared to sorbing ammonia only, the competitive NH₃ sorption data illustrate, as expected, a reduction in uptake for this component, considering the much higher feed concentration of ethanol. Also, the data indicates that zeolite Y would be preferred for ethanol, NH₃, and acetone sorption, while ZSM-5 is preferred for toluene and DCM sorption. In addition, it is evident that DCM sorption does not occur readily. As noted above, because of DCM's relatively poor sorption, breakthrough and equalization of inlet and outlet concentrations could be observed when testing both the smaller and larger number of screens; hence the dual entries in the table above. For zeolite Y, both results showed very low DCM sorption, though testing with the larger number of screens showed a somewhat greater uptake. Uptake of DCM was superior with ZSM-5, but variability was evident since testing with the smaller number of screens indicated a substantially larger sorption than did testing with the larger number of screens. These results enable a first pass estimation the relative and absolute amounts of ZSM-5 and Y required for a particular operating mode. Further refinement of the design requirements will result from actual testing of combined Y/ZSM-5 trace contaminant sorber. Note that any TCCS breakthrough also passes through the downstream catalytic oxidizer. After a discussion of the CO₂ sorption data in the next section, application of the results to the development of a preliminary design will be illustrated.

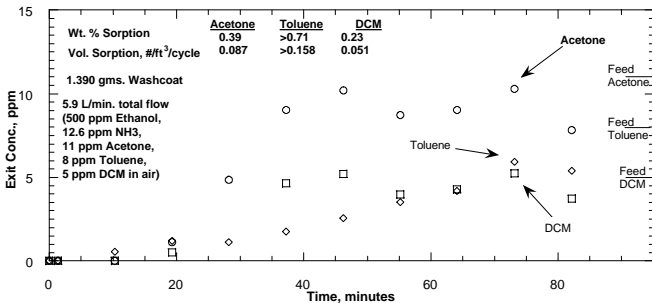


Figure 4: Competitive Sorption of Trace Species - 50/1 SiO₂/Al₂O₃ ZSM-5 on Microlith @ 25 °C.

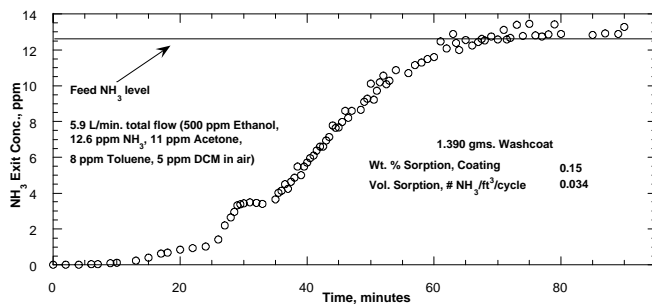


Figure 5: Competitive NH₃ Sorption - 50/1 SiO₂/Al₂O₃ ZSM-5 on Microlith @ 25 °C

CARBON DIOXIDE SORPTION: Results obtained using commercially available 5A pellets for CO₂ sorption are shown in Figure 7 below.

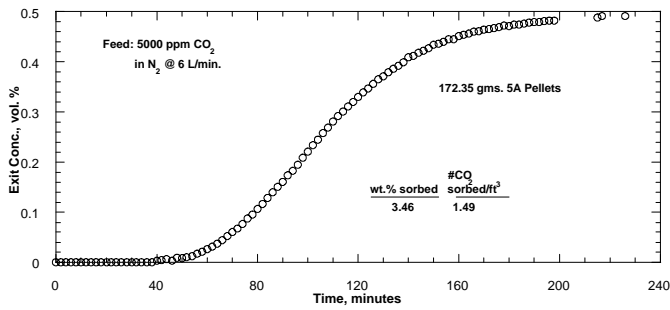


Figure 7: CO₂ Sorption – 5A Pellets @ 25 °C

The 5A pellet results are ~11% lower than what might be expected based on the pellet data reported in NASA/TM-1998-208752 for the 5A pellets currently employed in the CDRA. Similar performance was observed in a repeat sorption cycle after regeneration. The agreement is good considering possible differences in the zeolite contents (and/or binder types) in the pellets. (Typical binder levels are 20-50%, and often in the 30 – 40% range.) Results obtained when 5A was washcoated on Microlith (~109 mg/in²) are shown in Figure 8 below.

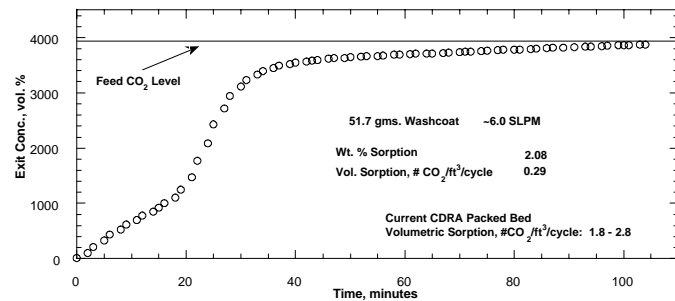


Figure 8: CO₂ Sorption – 5A on Microlith @ 25 °C

The Toth equation [12] is useful for estimating the effect sorbate partial pressure variations on sorbent capacity. It is given by $q = mP(b + P^b)^{-1/t}$ where m , b , and t are experimentally determined adjustable constants, q is the quantity sorbed and P is the component partial pressure. Based on 25 °C isotherm information for the Toth equation presented in the NASA TM noted above, at the lower feed CO₂ level employed in the Microlith test, ~88% of the uptake observed in the pellet sorption test would be expected (assuming 30% binder in both cases). Instead, the uptake is ~60% of that obtained with the pellets, and 68% of the expected level based on the Toth equation. The reasons for this greater than expected reduction in sorption capacity are unclear at present.

In addition to a lower “specific sorption” (i.e. wt% of the washcoat), as noted earlier, the presence of the inert Microlith substrate reduces the overall volumetric sorbent loading. Thus, at the washcoat levels employed in the test shown in Figure 8 above, the weight of sorbent per unit volume is ~30% of a pellet bed like that used in the current CDRA (assuming comparable binder levels).

Exploring the possibility that sorption performance might be influenced by the nature of the substrate, 5A was washcoated on a woven glass fiber mesh. Figure 9 shows the results for supported 5A sieve.

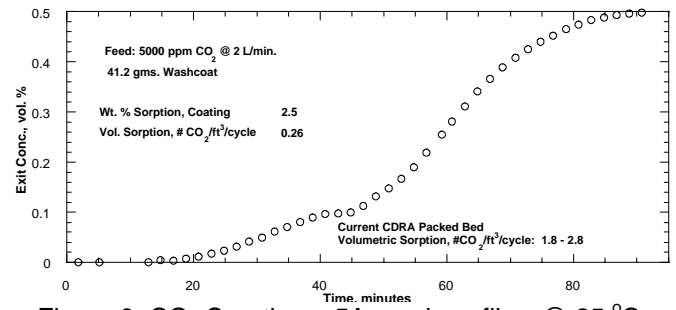


Figure 9: CO₂ Sorption – 5A on glass-fiber @ 25 °C

Since this test was conducted at the same feed CO₂ level as the pellet bed sorption test (Figure 7), the Toth equation correction need not be applied to establish the expected performance level, i.e. CO₂ uptake similar to that obtained with the pellets would be expected (again assuming 30% binder in both cases). The observed uptake is ~72% of the expected value which is consistent with the difference between expectation and observation noted above for 5A on Microlith (~68%). Thus, it would appear that the decrease in sorption capacity is not related to the nature of the substrate; rather, it may be associated with the washcoat application procedures. Similar to the Microlith case above, in this glass-fiber test, the weight of sorbent per unit volume is also ~30% of a pellet bed like that used in the current CDRA (assuming comparable binder levels).

PRELIMINARY DESIGN ESTIMATE: In the current CDRA, the linear velocity is 48.8 ft./min. at 20 cfm and the superficial mass velocity is ~210 lbs/ft²hr. Its gas hourly space velocity (GHSV) is 2125 hr⁻¹ with a nominal contact time of ~1.7 sec (~16.5” bed length). Each of the two canisters in the current CDRA system contains ~16,000 cc of 5A pellets. Ideally, the goal would be to have the Microlith based sorbent volume for both the CO₂ and TCCS functions comparable to that of the current CDRA. Assuming that the current CDRA bed length of 42 cm would be available for both CO₂ and trace contaminant sorption, for a square geometry with ~7.7” sides, the CO₂ sorbing elements would occupy ~85% of the available volume (~35 of the 42 cm. available). This volume would contain ~2500 gm of CO₂ sorbing washcoat with a sorption capacity of ~2.5 wt%. The shapes of the CO₂ sorption breakthrough curves indicate that, over the interval from time = 0 until the exit concentration closely approaches the inlet concentration, typically ~50% of the delivered species is sorbed. Because the bench scale tests operated at GHSVs and feed concentrations consistent with target values, calculations based on the observed breakthrough curves should provide reasonable estimates of the trace contaminant sorber cycle times in larger scale operation. Thus, at 20 cfm, calculations indicate that the CO₂ sorber elements would require regeneration approximately every 35 minutes.

As a result of the CO₂ sorber regeneration requirement, the TCCS sorption elements would also be regenerated every 35 minutes. The design issue for the trace contaminant removal section, therefore, is to determine the balance between each type of zeolite (Y and ZSM-5)

necessary to provide the required performance for a 35-minute cycle, and to see if they can be accommodated in the available volume behind the CO₂ sorber.

The remaining 15% of the volume would accommodate ~470 gms of Zeolite Y/ZSM-5 washcoated Microlith (metal, glass-fibre mesh) for trace contaminant sorption. For most of the sorbate / sorbent testing, the shape of the breakthrough curves indicate that, over the interval from time = 0 until the exit concentration closely approaches the inlet concentration, ~60% of the delivered species is sorbed. As discussed for CO₂ above, calculations based on the observed breakthrough curve behavior should provide reasonable estimates of the trace contaminant sorber size, as well as the relative amounts of zeolite Y and ZSM-5. Estimated bed lengths for the Y and ZSM-5 portions of the trace contaminant sorber are shown in the table below. Subject to the assumptions used, the results indicate that the 15% available volume behind the CO₂ sorption section should be sufficient to accommodate up to twice the required number of elements. Since DCM is difficult to sorb, this extra volume is desirable, and a 1:1 ratio of Y:ZSM-5 (3 cm each) should be a reasonable configuration.

Table 2:

Limiting Component	Ethanol	DCM
Cycle Time, min.	35	35
Min. Sorber Volume, cc	610	460
2x Min. Sorber Volume, cc	1220	920
Min. Bed Length, cm.	1.6	1.2
2x Min. Bed Length, cm.	3.2	2.4

REGENERATION POWER ESTIMATES: Power requirements were examined based on estimates of the total energy required to heat the mass of sorber components and to supply the heat of desorption (predominantly for that of CO₂, but also that of the trace components). Radiation losses were ignored since these are likely to be small for a well insulated system having a low ratio of external surface area to total sorber volume. Swinging two parallel sorber trains requires a 35 min. regeneration cycle to mesh with the 35 min. sorption cycle discussed above. The total required energy during heat up to the regeneration temperature is the sum of $mC_p\Delta T$ for the washcoat, substrate, and insulating layer plus the heats of desorption. The current CDRA uses ~960 W during a heat-up period of ~1.4 hr. Thereafter, a hold interval of ~1.0 hr. is maintained requiring an additional estimated 240 - 480 W.

The Microlith sorber system has about 55-60% lower mass vs. a pellet bed, and a lower overall estimated specific heat. Because of these characteristics, applying the same power currently used during heat-up of the CDRA should result in the achievement of the target regeneration temperature in ~20 minutes. This allows

for a potential 15 minute hold period, if necessary, to complete the regeneration at an expected power requirement of 240 – 480 W (depending upon the specific heat transfer characteristics of the final design).

CONCLUSIONS

Studies using Microlith supported zeolites for CO₂ and trace contaminant removal indicate the potential of this approach for accomplishing both functions in a volume equivalent to that currently employed for the CDRA. Weight and volume savings are achieved via the elimination of the separate charcoal bed currently used for TCCS. Additional savings should result from the expected lower weight of the combined Microlith CO₂ / trace contaminants system vs. the current CDRA pellet bed (but occupying the same sorbent volume). However, these advantages require more frequent regeneration of the CO₂ sorbent than the current CDRA pellet bed. Also, the trace contaminants sorbents require regeneration at the same interval as the CO₂ sorbent (the current charcoal bed is not regenerated). However, the physical characteristics of the combined CO₂/trace contaminant sorption system are such that more frequent regenerations and more rapid heat-up should be achievable at the currently used power levels. Since this program was primarily a science and technology based discovery effort, there was limited scope for rigorously exploring economic feasibility and long-term performance.

Combined CO₂/trace contaminant removal configurations based on the results and design approach outlined above remain to be tested. The design estimates above were based on the competitive sorption performance of Y and ZSM-5 tested separately. Thus, for example, ZSM-5 was regarded as having to manage the entire toluene and DCM removal load. Since the upstream zeolite Y will remove some of the toluene, and perhaps a small amount of the DCM, it is possible that an overall load reduction on the ZSM-5 could enhance its ability to remove DCM thus boosting performance. Similarly, removal of some trace components on the upstream 5A CO₂ sorber may reduce the load on the Y and ZSM-5 sorbents. Multi cycle performance with intermediate vacuum regenerations at effective time/temperature conditions, and estimated power requirements remains to be experimentally verified and is expected to lead to the fabrication of a combined CO₂/trace contaminant removal prototype.

ACKNOWLEDGMENTS

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