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Update of the Ground-Based Liquid Amine Horizontal Contactor Test System

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As NASA continues to pursue longer durations of crewed space flight missions, the importance of a robust carbon-capture system is becoming more evident. Currently, NASA relies on a packed bed of zeolite pellets to remove carbon dioxide from the International Space Station cabin air. While this is a proven technique, NASA and the Environmental and Life Support System community are continuously looking for alternatives that are lighter, take up less volume, draw less power, and are more reliable. One method being investigated is a system using a liquid amine to absorb carbon dioxide from the cabin air stream. Because the liquid must be in contact with cabin air in a microgravity environment, possible exposure to crew must be mitigated. This can be achieved with the use of V-shaped channels which use capillary action to keep the liquid contained within the channel. Since amount of CO_2 removed is a function of sorbent surface area, the contactor liquid surface area will need to be designed and sized to account for the CO_2 removal requirements per crew member. Ground-based test data to date has evaluated vertical contactor channels, which have a delta from microgravity performance due to the gravity effect in the vertical orientation. Therefore, a new horizontal channel contactor design, operable in any gravity or absence thereof, was built and tested. An analytical model of this new design was also developed in Aspen Custom Modeler. Both tools will be used to further understand the fluid characteristics, CO_2 absorption, and scale up requirements for the overall liquid amines CO_2 removal system.

Nomenclature

ACM = Aspen Custom Modeler
ARC = Ames Research Center
CO_2 = carbon dioxide
DGA = diglycolamine
ISS = International Space System
NASA = National Aeronautics and Space Administration
PPM = parts per million
SCFM = Standard Cubic Feet per Minute
SLA = stereolithography

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I. Introduction

WITH the ever-evolving growth of humanity's technological advancements that rely on a high consumption of energy, the release of carbon dioxide (CO_2) into Earth's atmosphere has become extremely prevalent. Energy consumption across the United States has increased rapidly over the last century, with CO_2 emissions from combustion-based and other industrial processes accounting for close to 89% of the energy sector's greenhouse gas emissions in 2021.[1] A high concentration of CO_2 gas buildup, whether in Earth's atmosphere or any enclosed environment, can become harmful to those who are subjected to the chemical compound. In concentrations of 1,000 to 2,000 parts per million (PPM), humans typically feel some level of drowsiness. Further increasing the CO_2 PPM level from 2,000 through 5,000 PPM will cause headaches and/or sleepiness, leading to the loss of attention, increased heart rate, and slight nausea.[2] These health concerns are attentively evaluated when the sealed environment, such as submarines, crewed spacecraft, or galactic habitats, are subjected to constant CO_2 emissions. Thus, the need to mitigate CO_2 in said environments is of high priority to those pursuing to design such systems.

NASA's goal of extending human presence deeper into space and to the Moon for sustainable long-term exploration and utilization requires that humans travel extreme distances in a sealed environment. This requirement calls for a robust carbon dioxide capture system for closed atmosphere use, which needs to be easily integrable into crewed spacecraft modules or lunar habitats. While different crewed mission scenarios will have different requirements, they will typically share a common philosophy: lighter, low volume systems with low power draw are seen as favorable when down selecting subsystem architectures. This philosophy will of course apply to the carbon dioxide capture system that will be used. Currently, there a few different ways of designing such system, with one promising way being the use of liquid amines.

The use of liquid amines for CO_2 removal is most prevalent in multiple terrestrial applications, such as CO_2 removal from flue gas and from the closed environment of submarines. Both of these industries use amines in similar ways: an aqueous amine solution is held in a liquid-to-air contactor, where the amine exothermically reacts with the CO_2-rich air stream. When heated, this solution releases the captured CO_2. Because this sorbent is liquid, capture and release can happen continuously, with the amine flowing between the contactor and regenerator rather than using batch reactors. Due to oxidation and sensitivity to thermal degradation, the amine is typically replaced every few months.[3] Although this issue exists, amines do offer an advantage over the current state of the art carbon dioxide capture system that is currently running on the International Space Station (ISS). The current system uses a solid sorbent swing bed process, where one of the beds is adsorbing CO_2, and the other is desorbing in parallel – the beds are then cycled once Bed A is at full adsorption capacity, and Bed B has desorbed all its CO_2. As previously stated, an amine system can run in a continuous loop, so there is no thermal cycling needed. This also means that an amine system will not need to have two separate "beds." For example, if a system that is scaled for four crew members needs 1 m^2 of amine surface area to capture 4 $kg/CO_2/day$, it will not need an extra 1 m^2 to be able to absorb and desorb at the same time. This does not mean that the volume, mass, and power draw of an amine system will be half of a cyclic bed system – those variables are dependent on the flux and capacity of CO_2 that the amine is able to uptake. Thermal cycling that requires the need to wait for hardware to cool down or heat up is also not a concern with an amine system. Solid sorbents are regenerated at temperatures around 200°C, while amines can be regenerated at much lower temperatures, around 75-115°C. These characteristics can lead to power consumption savings over a swing bed system. Continuous CO_2 release from the amine system is also an advantage, as it would eliminate the need for a downstream CO_2 management system. The upstream desiccation subsystem could also potentially be eliminated due to the fact that amines can absorb CO_2 in the presence of water, and humidity would help maintain the aqueous sorbent concentration. Amines also have the advantage of minimal pressure drop compared to solid sorbents, as there is no tortuous path caused by a packed bed. Therefore, the blower used could potentially be smaller than those needed for a solid sorbent bed, leading to a lower power demand.

While the use of liquid amines is optimized for Earth based usage, amine usage in microgravity is still a relatively new topic of research. Liquid containment in microgravity is one of the main concerns when transitioning to extraterrestrial use. One way to go about containing liquid in microgravity is with the use of wedge-shaped channels that utilize capillary action to contain the liquid and allow for a direct air-liquid interface without mixing.[4] This concept has been tested previously at Johnson Space Center, with V-shaped channels being placed in a vertical orientation, maximizing surface area in a volume by allowing amine to flow on either side of a corrugated sheet.[5] Initial testing of this idea was conducted through the Capillary Structures for Exploration Life Support flight experiment,[6] showcasing that the wedge channels for viscous liquid flow in microgravity is a viable solution. Alongside the development of a test bench for testing the vertical orientation of the wedges, a model was created in Aspen Customer Modeler (ACM) to simulate the mass transfer characteristics of the amine solution being used. An optimized form of the vertical contactor design was then sent to and tested at Ames Research Center (ARC), where data was collected and compared against the simulated results. It was shown that the high liquid flow rate required to flow through the vertical wedges minimized the residence time of the air stream in the wedges, impeding the amine solution from properly absorbing CO_2. There was also very little tuneability of the liquid flow rate across the vertical wedge due to the effect of gravity in this orientation.[4] This paper will discuss the development of a horizontal contactor system and the subsequent hardware and modeling effort involved. Multiple liquid flow rates and air flow rates were tested to determine the flux and capacity of a 65wt% diglycolamine (DGA) and 35wt% water solution within a wedge contactor designed for microgravity and partial gravity applications.

II. Horizontal Contactor Tray

A. Horizontal Contactor Bifurcated Channel Manufacturing

As shown in previous publications, a horizontal contactor was designed with a bifurcated manifold to deliver liquid to each wedge.[4] Figure 1 below showcases the bifurcated design of the initial horizontal contactor iteration. The reasoning behind the bifurcation was for liquid to reach each wedge at the same time with minimal working liquid volume, allowing for uniform liquid delivery to all 1,024 wedges of the contactor tray. A subscale stainless-steel contactor tray was printed using selective laser melting to test the manifold channel size, which was shown to produced reliable flow. Although the stainless steel sample print worked, this success proved to be difficult to replicate using resin-based printing. The geometry of the contactor to maximize contact area and reduction of liquid dead volume minimized the allowable space for the internal channels. This optimization resulted in the diameter of each bifurcated channel to be ~0.040”. Although it is technically possible to achieve this resolution from stereolithography (SLA) 3D printers, the channels were not all able to fully resolve.

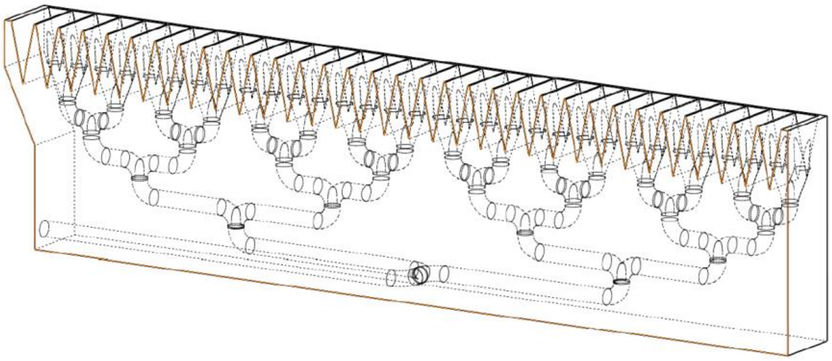

Figure 1. Internal Bifurcation for liquid delivery to V-Shaped Channels.

One potential cause is the viscous resin not being able to be completely cleared when cleaning the contactor tray after printing. Clearing of the channels involved injecting isopropyl alcohol into the tray to reduce the amount of excess resin in the wedges and channels before curing. When the contactor was cured, there was leftover resin in the channels that ended up solidifying and evidently blocking liquid flow once tested. Because of these apparent blockages, achieving flow equilibrium across the bifurcated channel contactor was not trivial. Each wedge received liquid at inconsistent time scales, up to a 10-minute difference depending on the flow rate of the pump. At higher pump flow rates, ~25 mL/min, the wedge channels were able to be filled quickly, on a time scale of less than 2 minutes, while at slower flow rates, such as 5 mL/min, the channels took up to ~12 minutes to completely fill. Individual rows of wedges would fill up first, typically the middle rows directly above the inlet and outlet of the contactor, before overflowing into the rows next to them.

Eventually, liquid equilibrium would become stable, but only for a few minutes as the blocked bifurcated channels would have trouble evacuating the liquid from the V-shaped channels, eventually leading to the tray overflowing. There were other issues with this iteration of the horizontal contactor as well, with prints warping and material chipping. Two materials were tested at this stage. Accura ClearVue resin, which was prone to material fractures, causing the trays to be too fragile to use, and Accura AMX rigid black, which was prone to warpage, causing problems when trying to align the trays with the housing (discussed later in Section 4).

B. Addition of Teardrop and Crosstalk

After experiencing the issues with the bifurcated liquid delivery method, the contactor tray was updated to include a teardrop-shaped, trough-style liquid delivery method. The teardrop shape was optimized to be easily printable using the SLA method and maintain the same microgravity liquid control as the wedges. The first prototype printed in AMX black is shown in Figure 2, but ultimately Acura 60 resin was chosen rather than ClearVue or AMX black due to its better printability, material properties, and better quality control from the manufacturer. Liquid delivery to the wedges became significantly more uniform, but the updated design was still unable to consistently deliver liquid to all wedges of the same row evenly. The trough-style is not optimized for simultaneous fluid delivery, therefore another fluid management solution was needed. A "crosstalk," developed by and proprietary property of IRPI in Wilsonville, OR was therefore also implemented to equalize any dissimilar filling of the wedges. The crosstalk is essentially a through hole unique to each row of wedges, allowing for liquid to travel between all wedges of the same row. This solution allowed for each row to fill uniformly relative to one another, creating a quick-priming liquid delivery solution to each wedge.

C. Equilibrium Verification with Sugar Water

After verification that uniform liquid flow across the updated teardrop manifold/crosstalk wedge contactor was achievable with water, a solution of sugar and water was created to mimic the viscosity of the 65vol% DGA solution. A 50wt% sucrose solution with a viscosity of 9.87 cP was used. With this liquid solution, pump speed was characterized for flow equilibrium across the channels. It was shown that the optimized contactors could handle up to 25 mL/min across the inlet and outlet at this liquid viscosity. A higher flow rate was not tested due to pump limitations, but it is likely that the teardrop manifold/crosstalk wedge configuration could achieve equilibrium across the wedges with a higher flow rate.

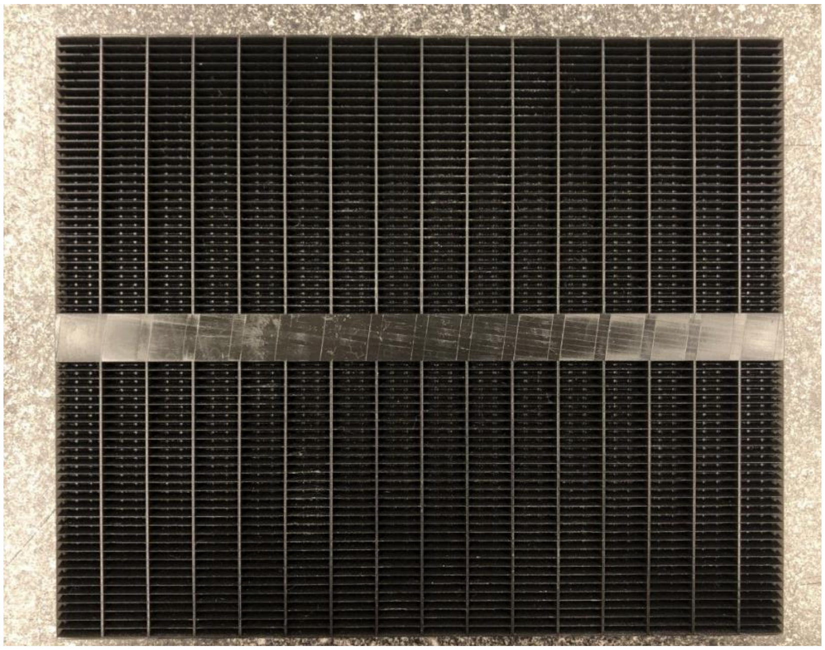

Figure 2. Top-down View of Contactor Tray Printed in AMX Black.

III. Contactor Test Stand

In order to fully test the horizontal contactor wedges with liquid amines, the test stand that was previously used to house the vertical contactor had to be modified. A detailed discussion of the design work for the horizontal contactor test stand can be seen in Chu et al., 2021.[4] After design work was completed, the housing components were manufactured by the NASA ARC Manufacturing Division. Figure 3 below shows the updated test stand for the horizontal contactor configuration, with various components called out. Six contactor trays fit inside the housing, achieving a total of up to 6,144 wedges and liquid surface area of 0.23m^2. Other than the new contactor, the gear pumps were replaced with peristaltic ones to operate at a lower liquid flow rate. The rest of the functionality remains the same as with the vertical contactor, with room air supplemented with CO_2 blowing through a diffuser, across the contactor, then exhausted to a fume hood. Meanwhile, DGA is pumped from an inlet reservoir, across the contactor, then to an outlet reservoir as the contactor is only half of the regenerable system liquid loop.

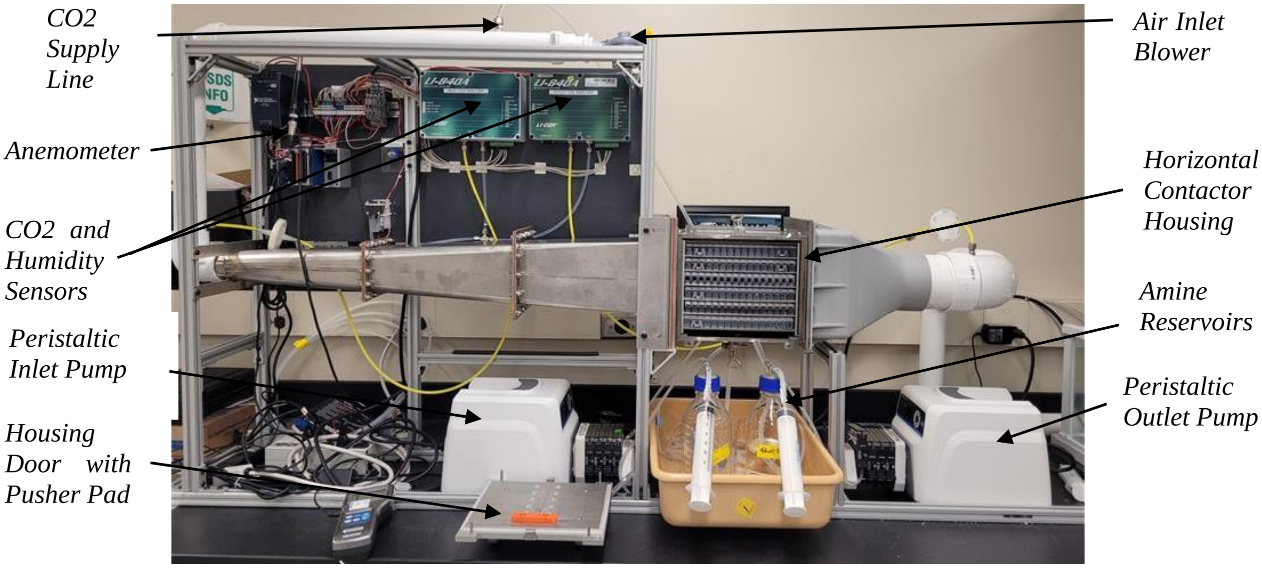

Figure 3. Updated Contactor Test Stand Assembly.

A. Addressing Manufacturing Concerns

Leak testing using water commenced once the horizontal contactor housing was installed onto the test stand. A few components ended up developing a slight leaking of water after a 24-hour test – notably the latch mechanism for the door and sliding guides for the internal contactor rack that needed a drilled and tapped hole to be installed. 3M Super Silicon sealant was used on these areas after a material compatibility test was done with DGA on the sealant. A drain port was also added to the housing to allow for any liquid that pooled on the floor of the housing to be safely cleaned. Once the sealing was completed and leaks eliminated, a 3D printed rack that holds the contactors within the sheet metal housing was inserted and testing with one contactor in the assembly commenced. The contactor uses 3/8” NPT luer lock fittings as a quick disconnect system – these fittings were engaged with “pusher pads” (shown in Figure 3), essentially a small shim attached to the door that presses on the face of the contactor tray, forcing it to align to and engage the luer lock check valve on the rear face of the housing. Therefore, DGA would not leak from the connection points when trays were removed from the contactor for cleaning or modification, as the check valve would close when the door was disengaged and removed from the housing assembly. The pusher pad to contactor to luer lock NPT fitting interface posed a few issues when attempting to run the sucrose solution through the test stand. Because NPT fittings use tapered threads, they engage at a certain height of the thread length, meaning the fitting does not seat entirely flush on the face of the contactor tray. This caused tolerance issues as each tray’s NPT fittings were tapped at different heights, meaning the pusher pads on the door need to be different thicknesses, causing sealing issues between the tray and bulk head fittings. Also, the 3D-printed AMX black rack holding the trays in place was revealed to have warped to an unusable state, both from compression of the door and from 3D printing large flat surfaces in plastic being a known challenge. Therefore, a stainless steel sheet metal rack was designed to replace it, using laser cut parts that are attached through tabs and slots. Figure 4 below shows the assembled rack, minus several shelves.

Figure 4. Sheet Metal Rack for Horizontal Contactors inside housing. *Luer lock quick disconnects and airflow channels can be seen.*

B. Initial Test of Contactor in Test Stand

After addressing all of the manufacturing concerns surrounding the interface between the contactor and test stand housing, a fully functioning contactor assembly was completed. Operational checkouts were performed with water plumbed to one tray, then testing of the horizontal contactor was underway. One multi-channel peristaltic pump was hooked up to in-line flow meters on the inlet and outlet that had a maximum flow rate of 50 mL/min. Therefore, flowrates could be adjusted in real-time to achieve steady state flow. While 50 mL/min was anticipated to have sufficient operational margin and the desired flow rate with water was easily achieved, the higher viscosity amine solution struggled to hit the desired flow rates. A maximum of only 3 mL/min was achieved with this setup with the DGA solution, therefore the flow meters were removed from the test stand and a second peristaltic pump installed so that the contactor inlet and outlet was directly controlled by dedicated individual pumps. In order to get an accurate flow rate from the peristaltic pumps, the speed of the pump was correlated to flow rate across the tray in mL/min by running the amine solution of 65wt% DGA and 35wt% water across the contactor and into a graduated cylinder. This was done multiple times at different pump speeds, with the data being used to create a correlation curve to convert the desired flow rate in mL/min to pump speed in RPM.

At this stage, it was found that using the pump to prime a tray, i.e. fill a dry tray so that the wedges are fully filled and fluid flow can achieve steady state, took on average nearly 30 minutes. In order to have uniform flow across all wedges more efficiently, the contactor trays were primed using syringes on the inlet and outlet lines, allowing for all the wedges to be filled with the DGA solution prior to the inlet and outlet pumps being turned on. This method allowed for the control of liquid height in the channels before starting the pumps, as maintaining a liquid level close to the top edge of the tray is ideal. A slightly higher surface area is able to be achieved with the liquid level being closer to the top of the wedges, as well as a better contact between the incoming air stream and the DGA solution.

IV. Experimental Setup & Results

After addressing all the operational concerns, the horizontal contactor test stand was run at an array of parameters to determine optimal performance. A design of experiments was synthesized to test different air and liquid flow configurations - air flow rate was tested at 6 SCFM, 13 SCFM and 26 SCFM, while liquid flow rate was tested at 3 mL/min and 10 mL/min for all 3 air flow rate cases. The data from the different air and liquid flow rates were used to determine the effect of residence time of both the CO_2-rich air stream and liquid sorbent stream to the flux and capacity capabilities of the 65wt% DGA, 35wt% water solution. Results also helped determine the operational scale of the contactor system itself. CO_2 levels of both the inlet and outlet air supply were measured using LiCor 840A sensors. Humidity, while not actively controlled for this testing, was also measured on both the inlet air supply and outlet air supply, but faulty humidity sensor calibration led to data not being reported in this paper. CO_2All air flow and liquid flow combinations were tested on with 1 contactor tray plumbed into the liquid flow loop. When running only 1 contactor tray, there were still 6 total trays within the housing to keep the air flow across each test consistent. For all runs, the CO_2 inlet concentration was kept as close to 2600 PPM as possible without using PID control. This concentration was selected to simulate the ISS CO_2 concentration and have data relevant to future planned spacecraft environments. Table 1 below showcases the amount of CO_2 captured and calculated CO_2 flux per the given parameters with the use of a single tray in the contactor. As of this publication, only one tray has been used in the contactor with future plans of running 3 and 6 trays, respectively. Fresh DGA solution was made prior to every run, with an amount of 500 mL being used to run the test for each data point. At 3 mL/min, this volume allowed for ~2.5 hours of testing, while at 10 mL/min the same volume allowed for ~50 minutes of testing.

Table 1. CO_2 Flux and Amount Captured in Relation to Number of Trays, Air, and Liquid Flow Rates.

Number of Trays	Air Flow Rate (SCFM)	Liquid Flow Rate (mL/min)	CO_2 Captured (kg/day)	CO_2 Flux (kg/day/m^2)
1	6	3	0.063	1.634
	13		0.082	2.115
	26		0.119	3.073
	6	10	0.059	1.528
	13		0.085	2.191
	26		0.122	3.149

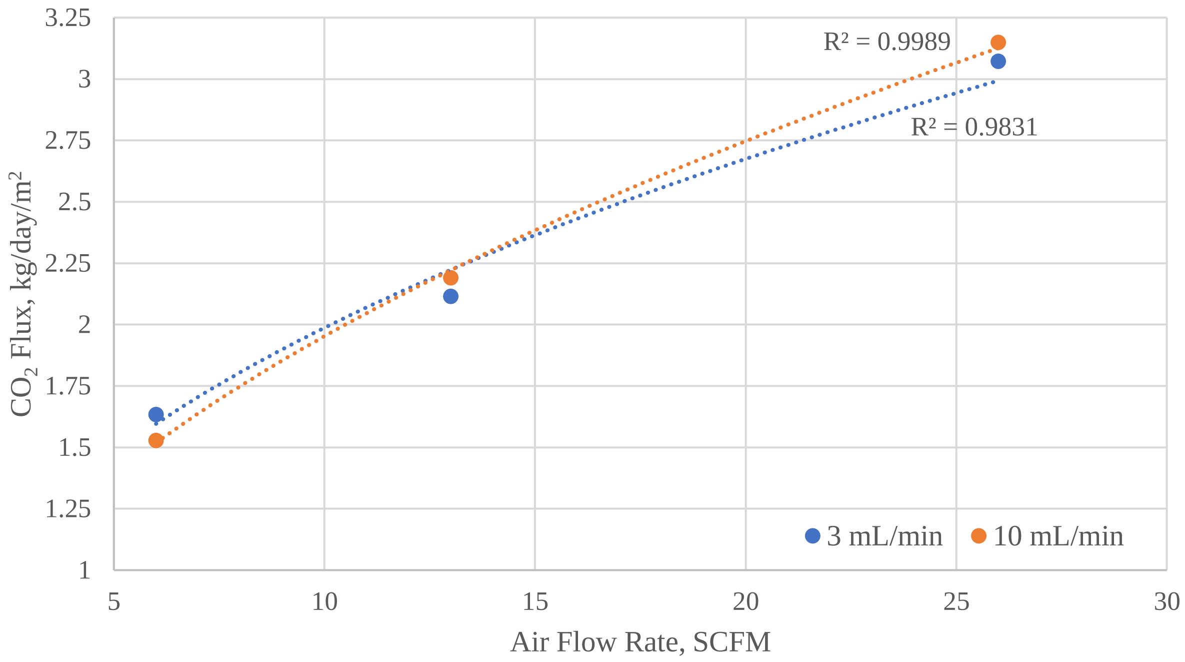

Figure 5. CO_2 Flux trends for 3 mL/min and 10 mL/min liquid flow rates.

Considering the values in Table 1 and resulting trends plotted in Figure 5, it was shown that in general the higher liquid flow rate has better performance, and performance increases with increasing air flow rate. However, the change in performance between the two liquid flow rates is nearly negligible, therefore air flow rate is the main performance driver. Only a few data points are available, but the increase of performance with increasing air flow rate seems to be decaying, and therefore increasing airflow much beyond 26 SCFM would not be beneficial. Interestingly, at the lowest air flow rate tested the lower liquid flow rate performed better. This result may show a shift in mass transfer dynamics, where longer liquid residence time is desired at lower air flow rates to allow for more diffusion. Whereas at higher air flow rates, a quicker refresh of the contacting liquid surface is better.

Overall, the optimal performance of a single tray contactor system at 2600 PPM of CO_2 in the air stream is at 26 SCFM air flow rate and 10 mL/min liquid flow rate, thus far measured to capture 0.122 kg of CO_2 per day per contactor with a flux value of 3.149 kg/day/m^2. These values would relate to 1.27 m^2 of surface area needed to capture 4 kg/CO_2/day if scaling linearly to multiple trays from single tray performance. If multiples of the existing contactor trays were used, this would equate to 33 total trays. The change in performance based on airflow rate shows that wedges arrayed in series such that a higher airflow rate is blown across results in better performance than if the wedges were in parallel, further splitting the airflow. This observation should be carried forward into future designs.

V. Horizontal Contactor Modeling

A chemical model of the Liquid Amine system was built in Aspen Custom Modeler (ACM). The model simulates a single absorbing tray in series with a single desorbing tray. Each modeled tray contains 32 individually calculated channels. An assumption was made that within each row of channels the channels are nearly identical; however, each sequential row will be different on both composition and flow. The model further breaks down each channel to 20 nodes for both the gas and liquid phases. The model uses a series of mass and energy balances to track temperature and absorption, or desorption. The model utilizes ACMs built in properties packages for water and DGA to see realistic differences in the fluid properties as absorption occurs across the length of the channel, and so creates a more realistic flow profile for the liquid. The flux of CO_2 between the gas phase and liquid surface is assumed to be very fast, and is therefore at equilibrium, but a secondary slow step exists, either diffusion from a surface film or chemical reaction between the DGA and CO_2, which controls the overall absorption rate by Equation 1 below. The long-term equilibrium concentration of CO_2 in various concentrations of DGA is well documented and the difference between the equilibrium concentration ($C_{CO_2}^{Eq}$) and real concentration ($C_{CO_2}^{Real}$) is used as a driving force with a kinetic parameter (k) which is then fit to real data. Thus far the model has shown reasonably good agreement with the test setup, but the data set is currently limited, so as more data is created the model should be reevaluated.

$$r = k\left(C_{CO_2}^{Eq} - C_{CO_2}^{Real}\right) \tag{1}$$

VI. Discussion & Future Work

The results presented thus far have shown that a subscale amine system with horizontal liquid contactors for use in microgravity has been built and tested, proving to be a suitable system for space-bound applications. Assuming the technology scales linearly, a full-scale, 4-crew amine system utilizing the V-shaped contactors and an amine solution of 65wt% DGA and 35wt% water would require 1.27 m^2 of surface area. This amount of surface area would equate to roughly 0.063 m^3 of volume, taken up by 33 horizontal contactors if using a similar housing design used by the current unoptimized subscale system. Subsequent hardware to complete the liquid amine system, such as a degasser, condensing heat exchanger, and liquid heat exchanger/mixer would be required, adding to the overall amount of mass, volume, and power of the system.

A degasser setup is still under development for the subscale system. In order to heat the amine solution to optimal temperatures of 85°C and up,[5] a metal horizontal contactor tray was 3D printed out of GrCop-42. This material is suitable for this application as its thermal stability and heat transfer characteristics allow it to conduct and retain heat well, meaning heaters can be embedded directly into the tray to keep the temperatures of the amines within the optimal range. Embedded heaters can also be used in unison with tape heaters on the inlet line of the degasser system, allowing for the amine solution to be heated before entering into the wedges. The embedded heaters, and potentially a floating pad heater above the wedges, can be used to keep the temperature of the amine constant and prevent loss from condensation while it is within the degasser.

Other solutions to heating the CO_2-rich amine are also being investigated, including the use of microwaves and heat pipes. Analysis of the usage of microwaves for heating our particular amine solution is in the early stages, it has shown some promise with monoethanolamine.[7] Microwave heating is appealing because it has the potential to reduce the power consumption of the degasser. Further analysis is needed to verify by how much the power consumption could actually be reduced as well as calculate the volume and mass of this type of system. It also may be possible to embed heat pipe channels within the horizontal contactor degasser tray, as 3D printed heat pipes are a technique being studied already.[8,9] This would require a complex system of channels that would have to be able to be resolved by the 3D printing process in close proximity to the existing liquid delivery channels. Further work is needed to verify the possibility of this technique in terms of manufacturing. If manufacturable, printed heat pipes could prove to be a viable solution to quickly transfer heat to the degasser, reducing the overall degasser system volume, mass, and power draw.

Once testing of the subscale system with multiple contactor trays is completed and degasser installed, the system will be automated to run in a continuous loop for extended periods to test both hardware reliability and sorbent lifetime. It would be ideal for the automated system to have a feedback loop to keep the liquid height optimal within the V-shaped wedges in order to maximize performance and minimize off-nominal events, such as bubble ingestion. Ways of enabling the feedback loop so must be investigated for feasibility, and may include the use of strain gauges, liquid flow rate sensors on the inlet/outlet ports, or some other method of accurately gauging the amount of liquid in the contactor.

Outside of the development of the subscale ground-based system, there have been developments on other fronts of the technology related to the liquid amine system. CapiSorb, a flight experiment used to test capillary flow for the use of liquid containment in microgravity, launched on SpaceX CRS-27. This experiment will test the fundamental idea behind hardware components unique to the liquid amines system used in the ground-based system by using a non-toxic simulant liquid. The results gleaned from this experiment will prove to be a huge stepping stone for the V-shaped wedge system for liquid containment. Carbonic anhydrase is also being developed in parallel with the horizontal contactor test stand, using directed evolution to augment amine CO_2 capture kinetics. Alternate sorbents are being characterized as well, which will be further discussed elsewhere.[10]

V-shaped wedge channels have proven to be a functional way of providing open air-liquid contact, with ground-based test stand data proving that the wedges can be utilized in an amine based carbon dioxide capture system effectively. Data that was presented in this paper will be utilized moving forward with the design of a full-scale amine based carbon dioxide capture system for microgravity applications.

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