University of South Alabama

Undergraduate Elective Class: Technical Evaluation of Methods to Recover Gas from Liquids in Low Gravity

T. Grant Glover, Kevin N. West, Brooks D. Rabideau (Faculty Advisors)
John Clearman, Stephanie Goff, Jon Hastings, Mary Hudson (Student Group)

Final Report

* Author to whom correspondence should be addressed. glover@southalabama.edu

Abstract

This document is the final report resulting from the work conducted by undergraduate students at the University of South Alabama during the 2019/2020 academic year. Currently, on the International Space Station a solid adsorbent material is used to remove CO_2 from cabin air; however, liquid absorbent/ reactive based systems are appealing because they may offer greater CO_2 capture efficiency. When liquids are used to capture CO_2 a regeneration step is required to release the CO_2 from the fluid for subsequent processing. During regeneration aerosolized droplets of water and liquid amines are produced creating a 2-phase fluid stream. Because the CO_2 stream is to be sent to a Sabatier reactor for conversion into methane, the droplets of the water and amines must be removed, necessitating the need for a

vapor/liquid separation process in a microgravity environment. This class was tasked with designing a system to separate the aerosolized droplets from the gas stream. An ionic liquid was assumed to be the amine based absorber fluid that was used to capture the CO_2, which is a key assumption because the ionic liquid has vanishingly low vapor pressure. The lack of volatility of the ionic liquid eliminates the need to manage an aerosolized amine resulting in only an aerosolized water air separation. To solve this challenge an undergraduate class used systems engineering to design and construct a hydrophobic screen separation apparatus for a 2-phase, vapor/liquid flow. The apparatus was constructed, but initial testing was not possible due to the closure of the University labs due to the corona-virus pandemic.

Introduction

Air revitalization is a vital aspect of life support in closed system environments and is one of NASA's mission priorities. A number of technologies have been used to remove CO_2 from enclosed environments, such as adsorption swing systems, liquid amine absorption/reaction systems, and chemical reaction canisters; however, unlike applications on Earth, applications on micro-gravity are complex. However, the possibility of a liquid based absorption system for CO_2 removal is appealing, and NASA has conducted their own research by spending 7.5 man hours aboard the International Space Station examining the use of a regenerable organic-liquid amine CO_2 sorbent system (CapiSorb). This experiment demonstrated the uses of a network of "waterfalls" to bring a liquid sorbent in contact with air, allowing CO_2 molecules to adsorb onto the liquid. Broadly, the subsequent regeneration of the absorber fluid is challenging because heating the fluid can result in the formation of aerosolized droplets of amines and water. With the recovered CO_2 stream being sent to a Sabatier Reactor for recycling, the presence of water and amines is problematic. Therefore, a method to remove the aerosolized droplets and collect the water and amines is required.

Like air revitalization, there have been a number of technologies proposed for gas/liquid phase separation in zero or microgravity.[1,2] Although both creating intimate contact between gases and liquids (gas absorption, stripping, distillation, etc.) and affecting their separation (flash tanks, settling tanks, disengagement devices) are well matured technologies in the field of chemical engineering, all of these common techniques for mixing/separations process rely on gravity to be effective, taking advantage of the large density difference between gases and liquids. However, in a microgravity environment, such as the ISS, density differences do not result in separation for quiescent mixtures. Without the force of gravity, other forces must be employed to achieve separation.

Dean separates the various types of zero/microgravity separations processes into two broad categories: static and rotary.[1] The static systems rely on either the inertia of the liquid phase (vortex), surface tension/hydrophobilicity/hydrophilicity (wick, slurper, hydrophobic/hydrophilic) or a combination of both forces to accomplish the separation (elbow wick). The rotary methods utilize the inertia of the fluid, either the natural inertia of the fluid flowing through a turbine (turbine rotary), inertial imparted via a motor (motor driven rotary), or a combination of the two (integral fan rotary). Each of these methods has advantages and Dean outlines the conditions under which each is effective.

During the Fall semester the class spent time reading Dean and other references to complete a tradeoff analysis of the various methods concluding that the simplicity of the static hydrophobic/hydrophilic separator shown in Figure 1 was the most preferred solution.[1] As shown in Figure 1, the key variable in this process is the hydrophobic material chosen to complete the separation along with the flow rate and the size and quantity of the aerosolized droplets introduced to the system.

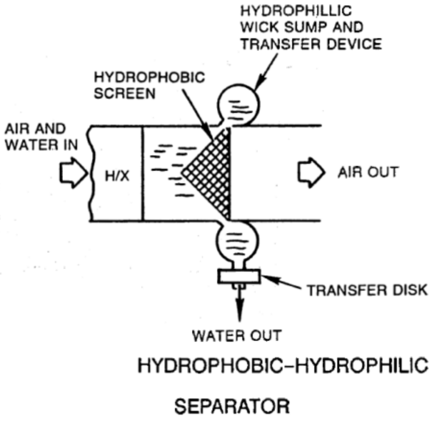

Figure 1. Graphic from Dean[1] showing a static hydrophobic/hydrophilic separator.

Figure 2 shows a representation of the apparatus envisioned based on the figure from Dean. At the inlet, gases are introduced into a tube (clear PVC to observe the flow) using a rotameter and the liquid is introduced via an atomizing nozzle. After sufficient distance to ensure that the liquid is evenly distributed in the gas, the two-phase flow is then passed across the hydrophobic barrier where the liquid is channeled to a sump, allowing the vapor to pass through. The vapor is then heated to vaporize the residual liquid and the liquid content is determined via a humidity detector. The amount of liquid in the effluent as measured via the humidity detector can then be compared to the inlet conditions to determine the amount of liquid removed, thus quantitatively measuring the efficacy of the 2-phase separation.

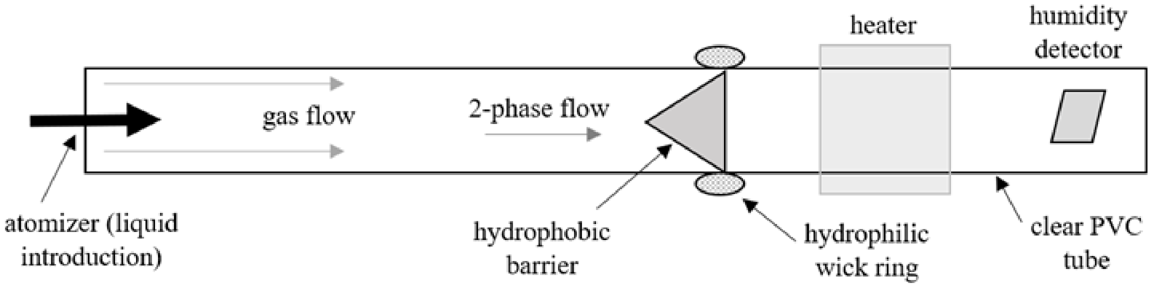

Figure 2. Schematic of the proposed testing apparatus to be formally designed and constructed by the students.

System Design and Results

Detailed discussions with the class based on Figure 1 and 2 produced a series of questions regarding the selection of the different components that were required and how the process would be physically completed. To resolve these questions a concept exploration focused on a pipe based separation system was completed as shown in Figure 3. In 3a the class considered the use of two pipes, one over the other, holding the conical separation cone (referred to as the hydrophobic barrier in Figure 2). This concept was explored further resulting in Figure 3b where the pipe over pipe concept was abandoned in favor of some type of apparatus that could hold the cone in a single pipe. This idea was then discussed and Figure 3c was produced showing that incoming air containing water that would be separated and a series of lines connected to a pump would be used to remove the liquid water from the pump. However, at this stage the physical interface of the cone-shaped hydrophobic separator, the pipe, and the pump was unknown.

To define the interface, the student team leader worked through a basic design in CAD as shown in Figure 3d. The design was based on rupture disks commonly maintained in industrial piping. With the rupture disk concept defined, the student then worked to alter the rupture disk design to allow for the connection of a series of pipes as shown in Figure 4a and then 3D printed a prototype part for review (Figure 4b).

To hold the hydrophobic screen in a conical shape, a 3D printed dome like structure was created as shown in Figure 5. The assembled sump and hydrophobic screen holding apparatus is shown assembled in Figure 6b. In 6b a 3D printed ring is shown that holds the conical screen holder in place.

With the separations component designed, a system or test stand was needed to evaluate the concept. However, even with this much definition to the system design there were still a large number of unknowns. For example, regarding the spray nozzle there was uncertainty about the type of nozzle, the spray pattern shape/width, the total liquid flow rate, and if any of these variables could be varied during testing. Questions also arose about the hydrophobic wick ring and how it would be used to collect the water stream. Also, it was concluded that a blower would be required to pull the air and aerosolized droplets through the separations process. Each of these questions were addressed as the design moved forward.

As a starting point for designing the full test stand, a drawing was produced first by hand and then in CAD as shown in Figure 7a. From this drawing the apparatus was constructed as shown in Figure 8, which is similar to the CAD drawing shown in Figure 7. Figure 8b shows the 3D printed hydrophobic screen holder and sump with pipe fittings connected to accept a series of pipes and valves as shown in 8d. Figure 8c shows the inline heater that was selected for the process. This heater is down stream of the hydrophobic screen and was selected to vaporize all of the remaining aerosolized water droplets that were not collected by the sump. The vaporization of these remaining droplets is

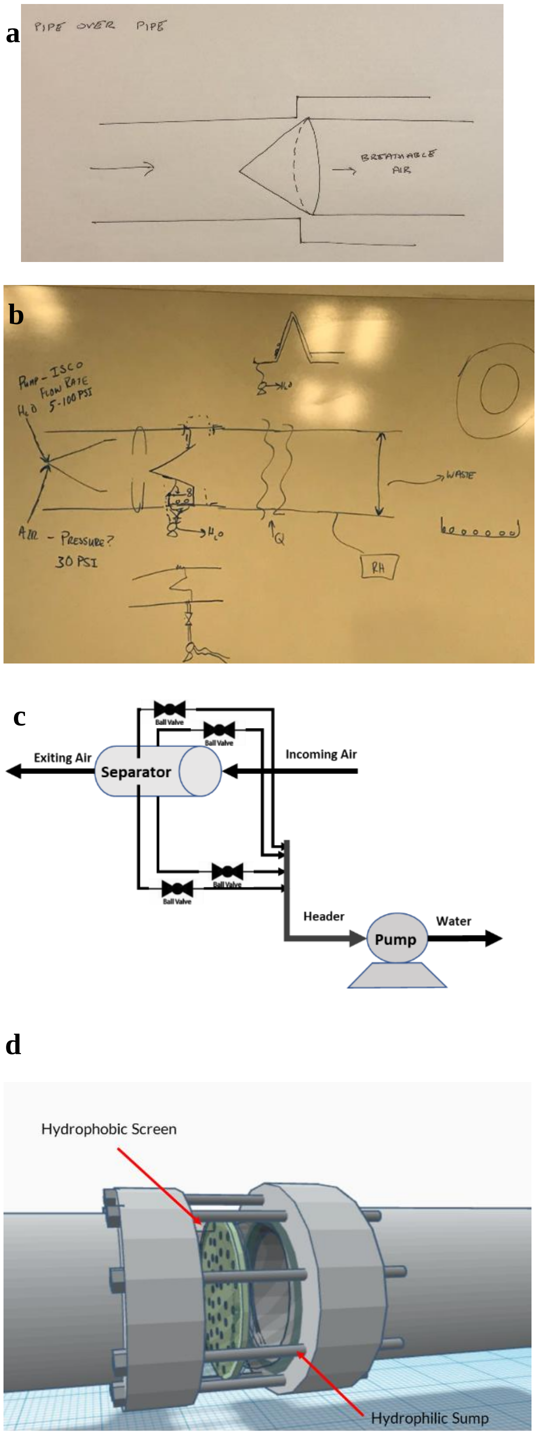

Figure 3. (a) Initial student design based on Figure 2 (b) refined student design (c) system sump design (d) detailed sump design based on a rupture disk.

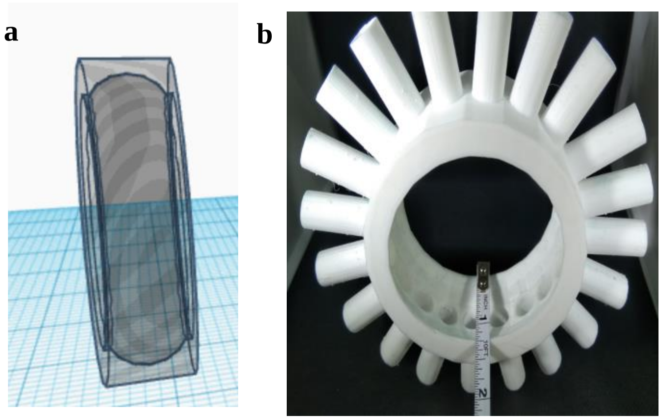

Figure 4. (a) Initial ring like rupture disk in CAD (b) resulting 3D printed part after adding pipes to basic rupture disk

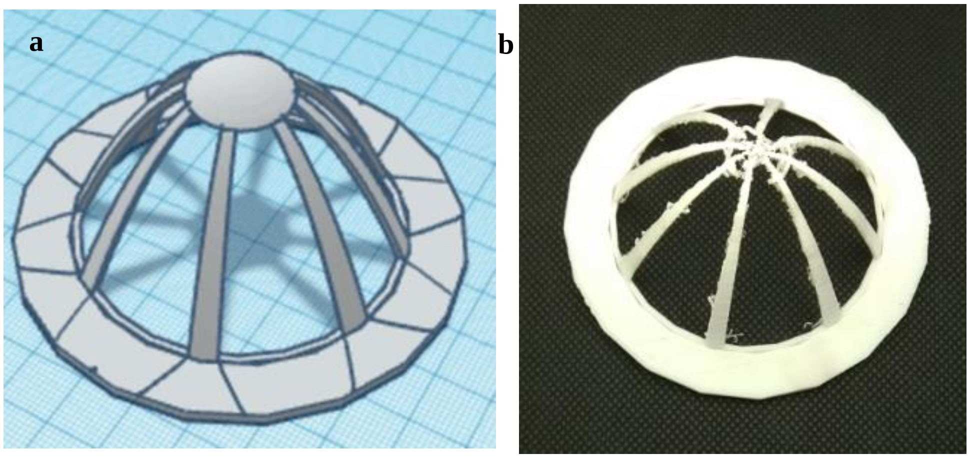

Figure 5. Cone shaped hydrophobic screen holder (a) drawn in CAD and (b) 3D printed

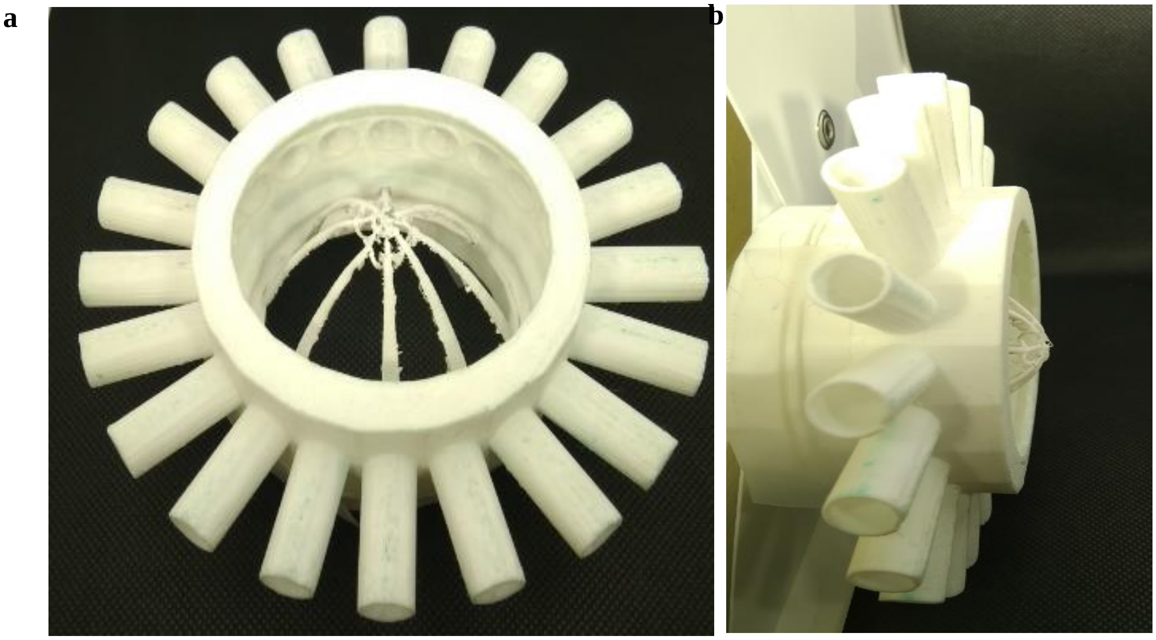

Figure 6. Assembled hydrophobic screen holder and sump device (a) viewed directly from top (b) cross section showing backing ring holding hydrophobic screen holder in place.

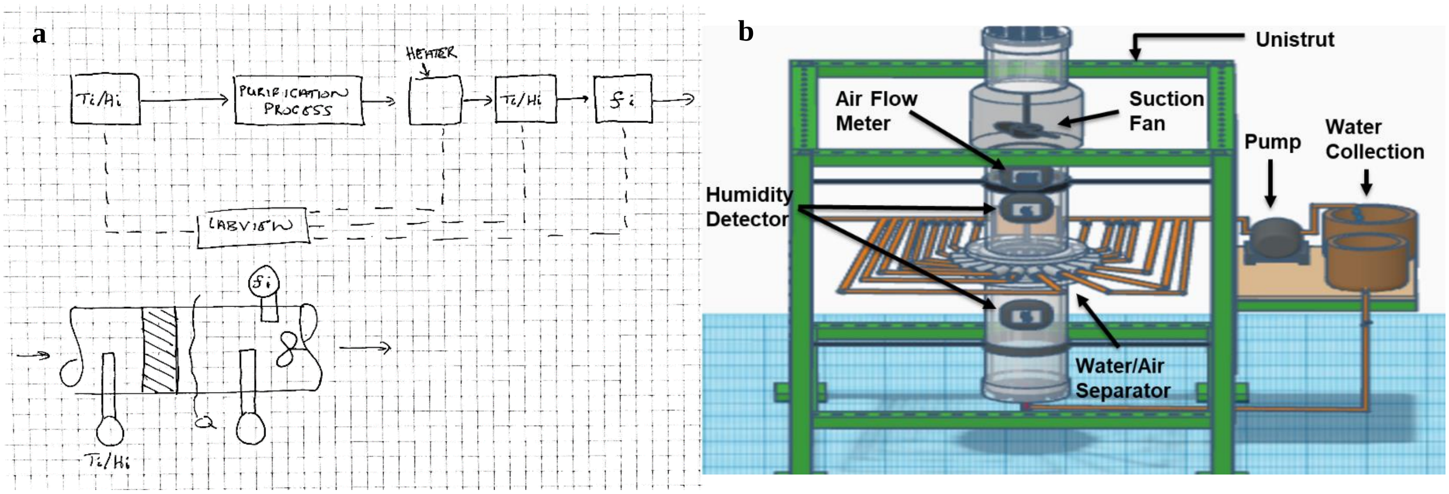

Figure 7. (a) Initial drawing of system controls and (b) subsequent drawing in CAD.

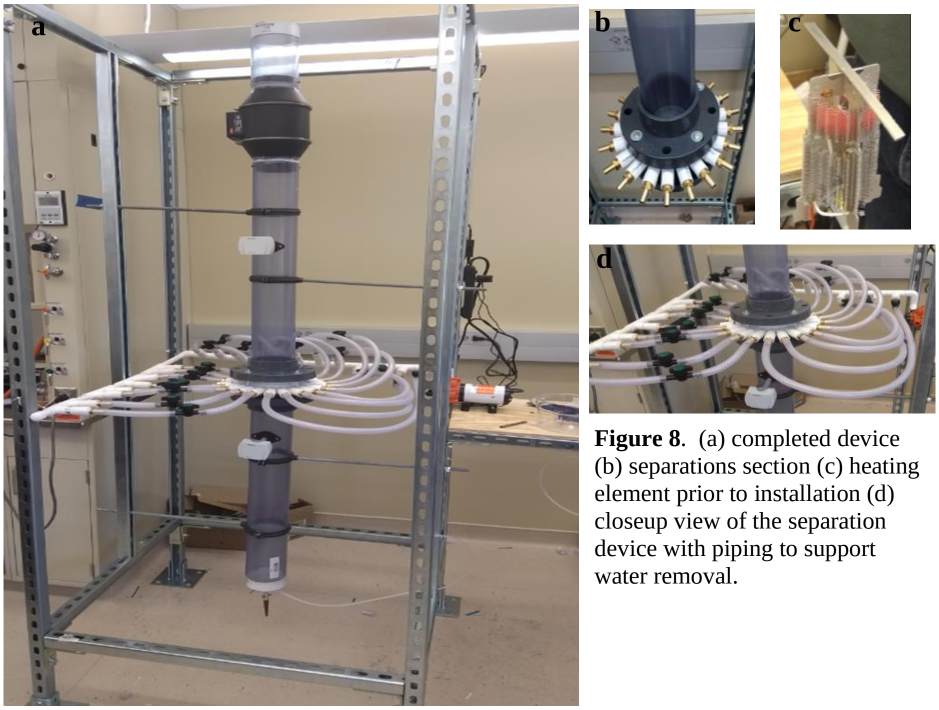

Figure 8. (a) completed device (b) separations section (c) heating element prior to installation (d) closeup view of the separation device with piping to support water removal.

important because a humidity meter occurs just after this heater and the expected humidity meter input was water vapor and not aerosolized droplets. Therefore, to ensure an accurate humidity reading, which is necessary to close the water mass balance, all of the aerosolized droplets need be completely vaporized. Many different heaters were considered, but in general, none were a good fit for this application with many being too powerful and others not fitting well in the pipe. It was determined that the heater from a consumer hair dryer would provide the correct amount of heat. With the assistance of an electrical engineer, the heating element was removed from the air dryer and controls put in place to utilize it as an inline heater.

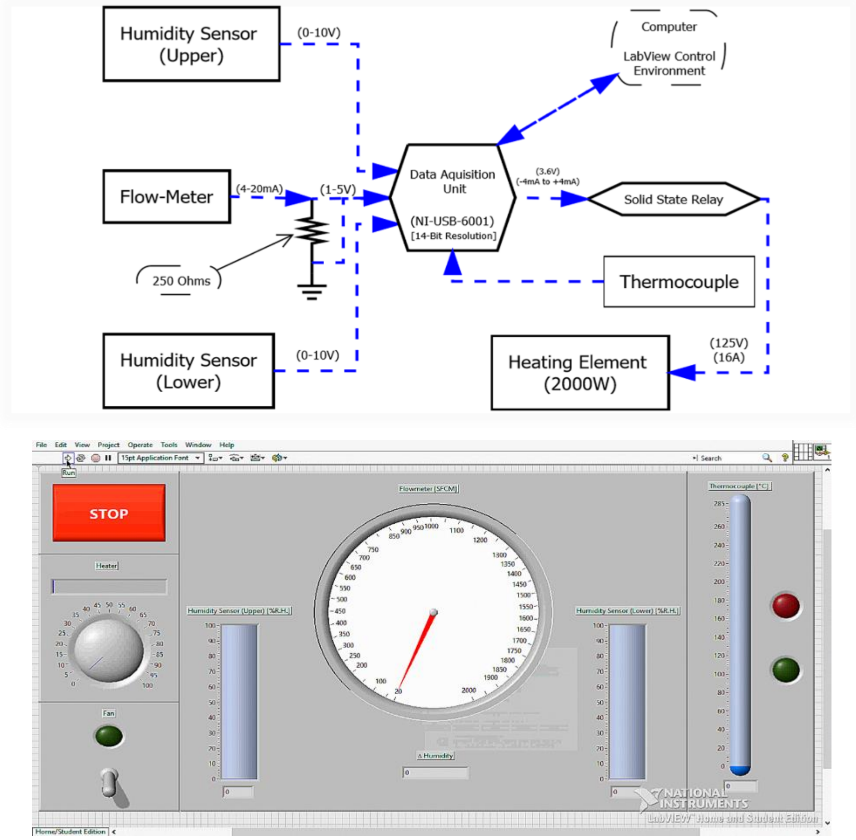

Figure 9. (a) Block flow diagram for control system (b) LabVIEW interface for used to operate the apparatus.

To develop the controls hardware a diagram was again produced as shown in Figure 9a, and with a hardware design determined, a LabVIEW interface was designed as shown in Figure 9b. The LabVIEW control interface also provided data recording and plotting of the recorded humidity.

After meeting with NASA and providing an update on this project, engineers at Ames Research Center recommended that a standard operating procedure be developed, and thus, a start-up and shut down procedure and an emergency shutdown procedure were all developed.

While the system was being built, the hydrophobic screen material was synthesized as shown in Figure 10.[3] This material was made by a senior student in chemical engineering as part of their undergraduate honors thesis who was not directly involved as a X-hab team member. The concept uses a textile dye material that has been modified with a long chain alkane group instead of a chromophore. In this way the material is dyed with using known textile dye chemistry to impart a hydrophobic property. Figure 10 shows a an aqueous drop containing blue coloring placed onto the base cotton fabric and a similarly sized drop placed on the hydrophobic fabric. The impact of the alkane dye is readily observed with the beading of the droplet on the cotton surface.

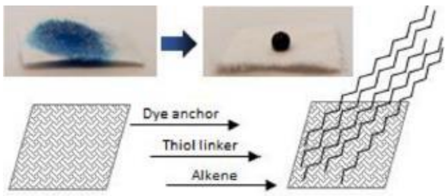

Figure 10. Reactive dye chemistry used to produce the hydrophobic screen.[3]

With the apparatus completed initial flow testing was completed that included operating the blower and spraying water into the column. Both the variable flow rate and the water injector volume were varied to determine to observe the impact on the aerosolized droplet flow. Theses initial tests were completed without a hydrophobic screen in place. After this test work was stopped due to COVID-19.

COVID-19 Impact

The project was stopped at this point due to a COVID-19 and associated University of South Alabama mandated lab closure. A final update was provided to NASA detailing the COVID-19 impact on approximately 24 April 24 2020. It is anticipated that additional work to finish the project will be attempted during the summer of 2020 depending on lab availability and a subsequent update to NASA will be provided if such work takes place.

References

(1) Dean, W. C. *Zero Gravity Phase Separator Technologies - Past, Present and Future*; SAE Technical Paper 921160; SAE International: Warrendale, PA, 1992. https://doi.org/10.4271/921160.

(2) Pour, N. B.; Thiessen, D. B. A Novel Arterial Wick for Gas–Liquid Phase Separation. *AIChE Journal* **2019**, *65* (4), 1340–1354. https://doi.org/10.1002/aic.16499.

(3) Brown, A.; Bozman, M.; Hickman, T.; Hossain, M. I.; Glover, T. G.; West, K. N.; West, C. W. Superhydrophobic Functionalization of Cotton Fabric via Reactive Dye Chemistry and a Thiol–Ene Click Reaction. *Industrial & Engineering Chemistry Research* **2019**, *58* (50), 22534–22540. https://doi.org/10.1021/acs.iecr.9b03258.