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U.S. Army Contracting Command
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Subject: Option Final Report
Contract No. W911QY-20-P-0277

Dear Ms. McGraw:

Enclosed is the Option Final Report for the above-referenced SBIR Phase I project titled "Compact, Low-Power Water Harvester."

Please contact me (603-640-2405; *mgizenson@creare.com*) if you have any questions.

Sincerely,

A handwritten signature in black ink, appearing to read "Michael G. Izenon".

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1010340/btt

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DTIC

Submitted by email



Option Final Report (Data Item B001)

COMPACT, LOW-POWER WATER HARVESTER

SBIR Phase I Contract No. W911QY-20-P-0277

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Submitted to:

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Creare LLC
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January 10, 2022
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1 INTRODUCTION

1.1 BACKGROUND

Warfighters require over three gallons of drinking water per day for sustainment. Reducing the logistical burden of supplying this water is a priority for the Army. To meet this need, Creare is developing a compact, efficient device that will enable individual warfighters to produce drinking water by harvesting it from ambient humidity. Our Microchannel Water Extractor (MWX) is an innovative device that promises breakthrough performance by combining three innovative technologies:

1. Creare's proven technology for low-cost fabrication of microchannel heat and mass exchangers using proprietary hybrid additive manufacturing (HAM) technology (Figure 1). This technology has been well established for use in compact heat exchangers and will provide an excellent basis for a compact sorbent bed based on water sorption in nanocrystal metal-oxide framework (MOF) materials.
2. RTI International's (RTI) innovative, high-volume MOF materials (Figure 2). These high-performance sorbent materials and RTI's fabrication methods are well suited to provide thin coatings of MOF sorbent on the microchannel surfaces.
3. A rapid-cycle, dual-bed sorption system that efficiently uses heat generated by a small combustor to drive water sorption/desorption from two beds operating 180° out of phase.

Preliminary sizing calculations show that the MWX sorbent beds should be extremely compact and lightweight (< 2 kg total mass of sorbent beds) and efficient (about 50 g fuel per kg of water produced).



Figure 1. Microchannel Heat Exchanger Produced Using Creare's HAM Technology. Highly uniform array of microchannels enable high surface area density. Efficient coupling of MOF sorbents with process streams yields very rapid kinetics. Module in photo is 9 in. long.

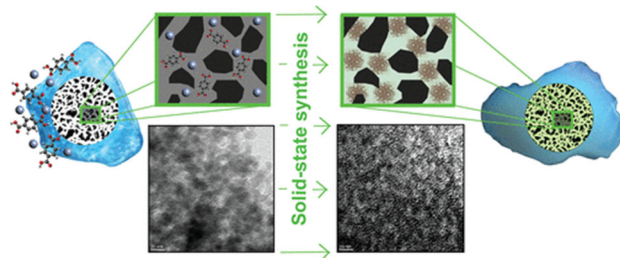


Figure 2. Nanocrystalline MOF Materials Produced by RTI. The MOF is well dispersed within mesoporous materials via novel "solid-state" synthesis. The mesoporous supports can be produced in thin layers on HXA surfaces.

1.2 SCOPE

In Phase I, we demonstrated MOF coating and fabrication methods for microchannel sorbent beds, measured cyclic water sorption performance, and produced a concept design and performance estimates for the MWX system. In the Option phase, we (1) measured the potability of the water produced by temperature-swing cycling of the MOF sorbent for compliance with TBMED-577, and (2) explored methods to improve MOF coating performance by growing MOF

crystals directly on MWX surfaces. Based on the information from both, we plan to update the MWX system layout design.

1.3 RESULTS: OPTION PHASE

Key results in the Option phase are as follows:

1. *MOF direct adhesion development.* RTI's process to grow MOF sorbent directly on stainless steel (SS) substrates shows promise as a way to increase MOF content in the MWX system without increasing size or mass. The surface preparation and processing steps need additional development to achieve a stable MOF layer suitable for water collection.
2. *Water quality tests.* Detailed water tests show no evidence of MOF material carryover into the collected water sample. Decreased pH and increased turbidity of the sample relative to the control suggest some modification of the water. If necessary, both can be remedied with a post-filter polishing step to bring the properties back within the TBMED 577 standard's acceptable range.

1.4 NEED FOR A COMPACT WATER HARVESTER

Water harvesting technology must overcome considerable technical challenges before a compact device will be attractive for use in the field. Existing sorption technology is too inefficient to enable a compact system that meets the Army's requirements.

1.4.1 Technical Challenges

Warfighters need a great deal of drinking water—over three gallons (roughly 30 lb_m) per day for sustainment. Carrying this much water on extended patrols or sorties is a significant burden. A small device that can harvest water from the atmosphere and produce drinking water would be a significant benefit. However, state-of-the-art technology for water extraction faces significant challenges.

One problem is that water vapor in the atmosphere has a very low concentration, particularly in cooler environments. Under the conditions specified in Topic A20-062 (20°F to 125°F, 40% RH), the absolute humidity can range from 35 g H₂O/kg dry air at high temperature to only 0.86 g H₂O/kg of dry air at the lowest temperature. As a result, the quantity of air that must be harvested to meet sustainment requirement varies tremendously depending on ambient conditions. Figure 3 shows the volume of air corresponding to the topic requirement of 14 L per day. While these volumes are manageable for higher temperatures (747 m³ at 100°F/40% RH), the volumes are enormous at the low end of the operating range (12,300 m³ at 20°F/40% RH). As a result, the device will require large throughput of air to meet requirements under all conditions, and high mass transfer efficiency is essential.

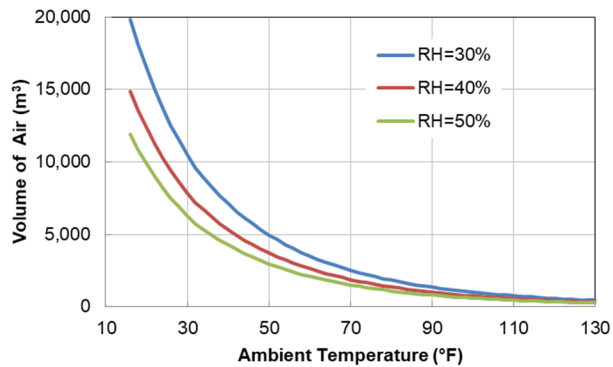


Figure 3. Volume of Air That Contains Enough Water Vapor to Meet Daily Sustenance Requirement for an Individual Warfighter.

Secondly, sorption and desorption of water are energy intensive. A sorption bed releases energy during sorption (roughly equivalent to water’s heat of condensation) and requires the same amount of heat addition during desorption. The heat transfer needed to maintain stable temperatures during these parts of the cycle must occur as efficiently as the mass transfer of water vapor into and out from the sorbent material. To enable rapid cycling, the sorbent bed must be assembled with features that enable extremely effective heat and mass transfer. State-of-the-art systems are based on relatively crude sorbent supports and large flow channels, both of which limit the rates of heat and mass transfer. This increases the time needed to cycle the bed and implies the need for larger beds to meet the Army’s water production requirement.

1.4.2 Requirements

The basic requirements for the water harvester were detailed in Topic A20-012 and listed in Table 1. In addition to these quantitative requirements, the design must be intrinsically safe (possess anti-microbial features, capable of being sanitized and/or disposable) and provide hygienic functionality and convenience.

| Requirement | Unit | Value |
|-----------------------------|-----------------|-----------|
| Water production rate | L/day | 14 |
| Ambient relative humidity | | 40% |
| Max fuel consumption | kg/day | 0.5 |
| System weight | lb _m | < 20 |
| Operating temperature range | °F | 25 to 125 |
| Production cost | \$ | < 100 |

1.4.3 State of the Art

Existing technology cannot meet the Army’s goals because cycle time is too slow, sorbent materials cannot be integrated with compact and efficient sorbent bed structures, and power consumption is high due to inefficient design of balance-of-plant subsystems.

Atmospheric water extraction is moving away from traditional dehumidification techniques such as fog harvesting and direct condensation (which are prohibitively expensive outside of humid environments) and toward novel application of sorbent materials.¹ Researchers have demonstrated feasibility of water harvesting by using a temperature swing bed approach, exposing air at different conditions (ranging from controlled laboratory settings to desert environments) to beds of sorbent material at low temperature, allowing the material to saturate with water, then heating the bed to release trapped water.^{2,3} Although functional for atmospheric water extraction, most sorbent-based approaches today struggle to exceed ~1 liter of water per kilogram of sorbent per day, significantly less than that required for the technology to trade well against alternatives. Key limitations in existing approaches include the following:

- Adsorption/desorption cycle times on the order of several hours, driven by kinetics and dictated by sorbent bed geometry.
- Sorbent materials structured as pellets or powder, which are difficult to integrate efficiently into heat exchangers.
- High power consumption due to inefficient design of supporting systems for heat and mass transfer.

1.5 MICROCHANNEL WATER HARVESTER

We propose to meet the Army’s requirements by developing microchannel sorbent beds for nanocrystal MOF materials. We will use these sorbent beds to build an innovative, simple system that provides rapid sorption cycling and continuous water production from a dual-bed, temperature swing system.

1.5.1 Compact, Efficient Water Extraction System

The design concept for Creare’s microchannel water harvesting system is proprietary and is described in the Phase I proposal and in separate reports to the Army. The overall architecture is a two-bed temperature swing system with heat for desorption provided by a small combustor and cooling for sorption and condensation provided by the flow of ambient air through the system. While one bed is heated to desorb water, the other is cooled to adsorb water from the atmosphere and condense water from the desorbing air stream.

1.5.2 Microchannel Sorbent Beds

Creare’s HAM technology enables us to produce high-performance, compact microchannel heat and mass exchange structures at low cost using methods that are ideally suited for integration

¹ Tu, Y., Wang, R., Zhang, Y. and Wang, J., “Progress and Expectation of Atmospheric Water Harvesting,” *Joule*, Vol. 2, No. 8, 2018, pp. 1452–1475.

² Kim, H., Rao, S.R., Kapustin, E. A., Zhao, L., Yang, S., Yaghi, O. M. and Wang, E. N., “Adsorption-Based Atmospheric Water Harvesting Device for Arid Climates,” *Nature Communications*, Vol. 9, No. 1, 2018, pp. 1–8.

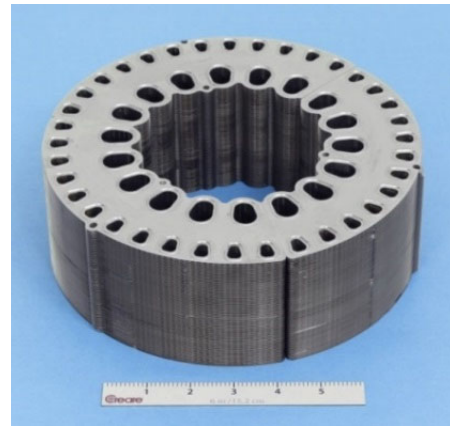
³ Hanikel, N., Prevot, M. S., Fathieh, F., Kapustin, E. A., Lyu, H., Wang, H., Diercks, N. J., Grant Glover, T. and Yaghi, O. M., “Rapid Cycling and Exceptional Yield in a Metal-Organic Framework Water Harvester,” *ACS Central Science*, Vol. 5, No. 10, 2019, pp. 1699–1706.

with MOF materials. We propose to use this approach to produce microchannel sorbent beds for high-efficiency water harvesting.

We have used HAM successfully to build compact, high-performance heat exchangers for heat recuperation in small gas turbine engines (Figure 4). Details of the HAM process are proprietary and have been described to the Army in other documents.



(a) Five 100-Plate Modules From the Recuperator of the Rolls-Royce 700 hp M250 Gas Turbine Engine



(b) Recuperator for UAV Turbines' 50 hp UTP-50R Turboprop Engine

Figure 4. Microchannel Heat Exchangers Produced by Creare

1.5.3 Innovative Nanocrystalline MOF Materials

RTI has developed nanocrystalline Zr-fcu-MOF-801, which has demonstrated good water sorption under constant humidified airflow at 10% RH and 25°C and good desorption at different temperature and 10% RH as well (Figure 5). Using isorecticular chemistry (computational modeling), RTI will design and construct novel MOFs with fcu and xhh topologies and with a suitable aperture size for water sorption.

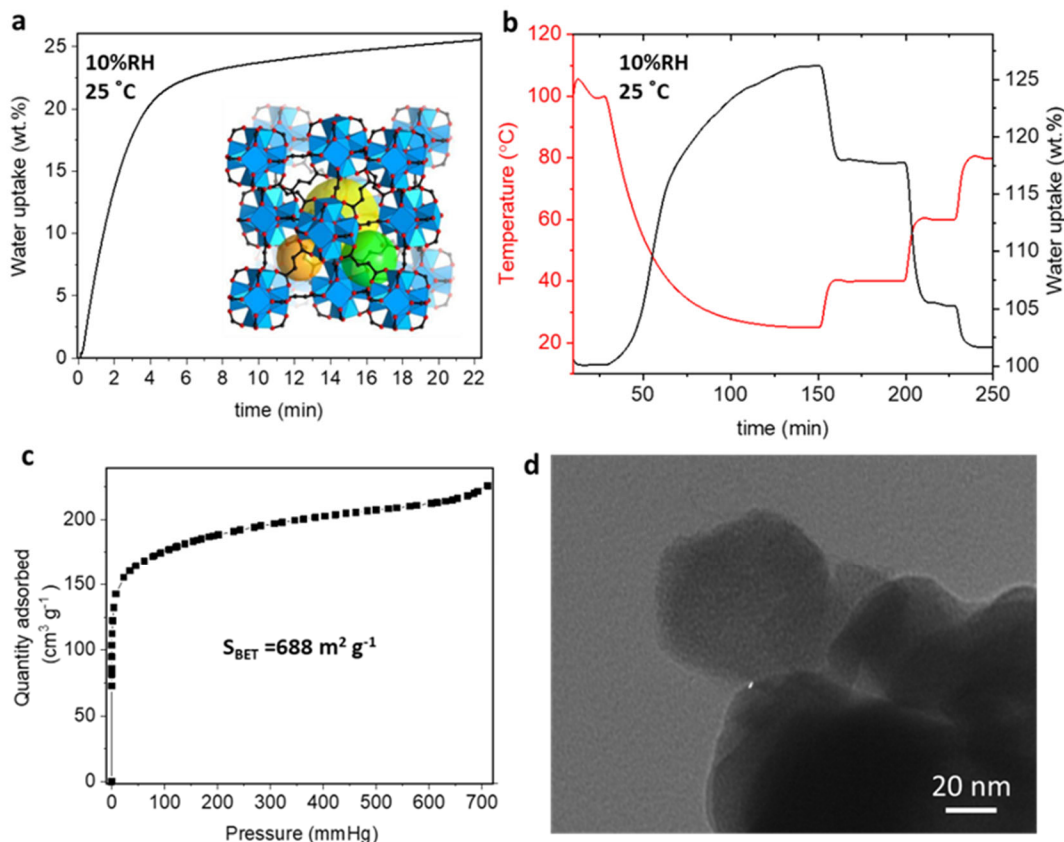


Figure 5. Water Sorption Properties of Zr-fcu-MOF-801. (a) Dynamic vapor sorption properties of Zr-fcu-MOF-801. (b) Dynamic vapor sorption properties of Zr-fcu-MOF-801: adsorption at 25°C and 10% RH and subsequent desorption at 40°C, 60°C, and 80°C and 10% RH. (c) Surface area of Zr-fcu-MOF-801. (d) TEM picture of Zr-fcu-MOF-801 nanocrystalline.

RTI has developed methods for applying coatings of MOF materials to metal substrates. The current state of the art is to use mesoporous polymer carriers, which can be applied in layers as thin as 250 μm . More advanced methods would grow MOF directly on the metal substrates following etching with HF. In the Base phase of Phase I, RTI applied MOF sorbents to sample substrates using the polymer approach. In the Option phase, we explored the direct growth approach, which promises to reduce mass transfer resistance and enable more rapid cycling.

1.6 SCOPING DESIGN OF MWX SYSTEM

To estimate the overall weight and airflow requirements of our MWX concept, we performed scoping calculations based on a projection of the state of the technology at the conclusion of Phase II of this program. The estimated size and performance depends on the amount of water that can be absorbed in the MOF and how efficiently the MOF can be applied in thin layers to the surface of the heat and mass exchanger beds. Details of the projected sorbent layers are proprietary and were described in the Phase I proposal. We focus our scoping efforts on the mass of the exchanger beds, as these will make up most of the system weight (additional system mass will come from fans, combustor, fuel bottle, water reservoir, and tubing as well as

filtration/purification if needed). Table 2 shows the parameters used and the results of this scoping analysis. Based on ambient conditions of 30°C and 40% RH, we conclude that the swing beds in a 14 L/day system would have a dry weight of 2.04 kg. To perform at this level for these ambient conditions, the beds would require 52.1 ft³/min of airflow, a relatively small amount easily supplied by a small computer cooling fan.

| | | |
|------------------------------|----------------------|------|
| Ambient Temperature | °C | 30 |
| Ambient Relative Humidity | | 40% |
| Water Production Rate | L/day | 14 |
| Estimated Total Mass of Beds | kg | 2.04 |
| Estimated Total System Mass | kg | 4.5 |
| Airflow needed | ft ³ /min | 52.1 |

Regarding consumables for the system, about 0.7 kg of fuel would need to be consumed to condense 14 L/day of water from the air (assuming a net heat of combustion of JP-8 at 43,200 kJ/kg and assuming no external heat sources are available). With a water latent heat of 2,200 kJ/kg, this means that 0.051 kg of fuel is needed per kg of water produced. Limiting the system to producing 10 L of water per day results in a minimum of 0.5 kg of fuel required.

2 OPTION PHASE I TECHNICAL OBJECTIVES

The primary objective of this project's Option phase is to advance the MWX system-level design through refinement of the components contributing to the design. To accomplish this objective, we established three contributing objectives:

1. *Develop an initial detailed design of the MWX system.* Starting from the preliminary design and analytical models produced in the Base phase of the project, we will focus on details of each of the component designs. Most of the detailed design effort will focus on the sorbent bed design.
2. *Perform trials of growing MOF material directly on the sorbent bed metal surfaces.* The approach is to directly apply the MOF material to the substrate surface without incorporating any adhesive component. Doing so should increase the water loading per mass of sorbent, and thus increase the performance of the system while reducing its overall weight. RTI plans to conduct initial trials of this method to determine its applicability to the MWX system.
3. *Evaluate the quality of the water harvested from the Phase I proof-of-concept system.* Our collaborators at Cascade Designs, Inc. (CDI) will be responsible for evaluating the water using the TBMED 577 water potability standard. For these tests, only a small amount of water (roughly 50 mL) is needed, which matches the scale of our Phase I proof-of-concept tests. Results of this test will provide information on the water quality resulting from harvesting with the proof-of-concept system. We will use this information in Phase II to develop appropriate filtration and purification components to include in the MWX system design.

3 OPTION PHASE I RESULTS

3.1 DETAILED SYSTEM DESIGN

Most of the preliminary design work was accomplished during the Base phase and during preparation of the Phase II proposal. No additional work occurred on this objective in the Option phase.

3.2 MOF APPLICATION TRIALS

RTI successfully grew MOF crystals directly on metal substrates. Additional work is needed to improve uniformity and adhesion.

To provide higher surface areas for the MOF crystallites to grow, RTI studied the preparation of etched SS surfaces under acidic conditions. They submerged 0.01×1 in. OD 304 SS coupons in an HF solution in a Teflon-lined autoclave, sealed and heated at a fixed temperature for 15 min, 60 min, and 90 min. They recovered the resulting etched coupons and gently washed them in deionized water to remove unreacted acid and etched particles. Using a digital thickness gauge, they determined the thickness of the coupons to have been reduced by 0.01 in. (15 min), 0.01 in. (60 min), and 0.00975 in. (90 min) after etching. In contrast to the small differences in coupon thickness observed after etching, the change in color of the coupons distinctly demonstrates the extent of etching on the surface of these materials (Figure 6).

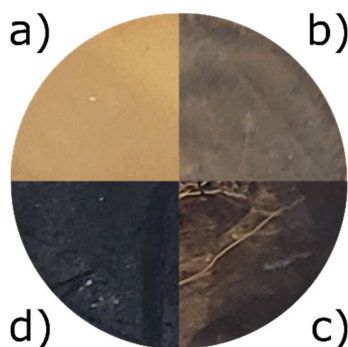


Figure 6. a) Pristine 304 SS Coupon. 304 SS coupon after b) 15 min, c) 60 min, and d) 90 min etching in an HF solution at fixed temperature.

To grow a crystalline coating of MOFs on the SS surface, RTI placed the etched coupons inside Teflon-lined autoclaves and added solvent, followed by MOF reagents. They sealed the autoclave and heated it to a fixed temperature overnight. Upon cooling to room temperature, they opened the autoclaves, decanted the reaction solution, and recovered the coupons with MOF crystals grown on the surface. They then gently washed these coupons in solvent and allowed them to air dry. Figure 7 shows a comparison of the FT-IR spectra for bulk MOF and the surface adhered MOF. The main features of the spectra align well, showing that the surface-adhered material is the expected MOF.

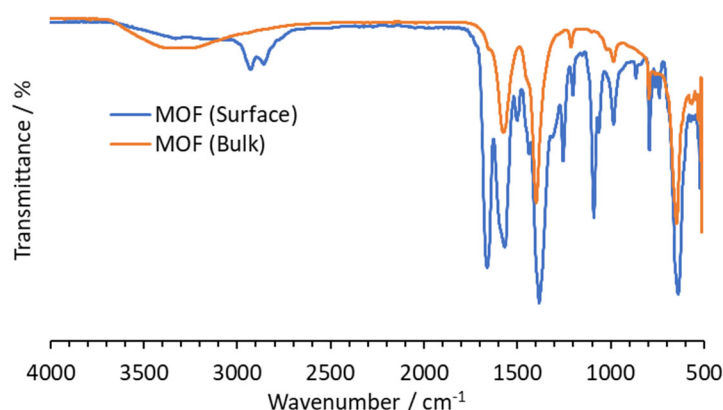


Figure 7. FT-IR Spectra of Bulk MOF and MOF Recovered From the Surface of the SS Coupons. Extra peaks observed in the blue plot are associated with adsorbed solvent.

RTI added organic acid to all reactions to control the nucleation of crystals on the surface of the coupon. They held the concentration and volume of acid added to the reaction constant throughout these experiments. To further control the nucleation and crystallite size of MOFs, they carried out a parallel set of reactions wherein they layered a basic mixture on top of the reaction solution and allowed it to diffuse throughout the reaction. They expected this addition of base solution to further control MOF crystal nucleation at the coupon surface. They observed limited crystal growth on the 15 min etched SS coupon. Indeed, regardless of the addition of acid and base, bulk crystal growth only occurred in solution with minimal crystal growth on the SS surface. Scanning electron microscope (SEM) images revealed a thin layer of microcrystals; Figure 8b) reveals a layer of crystals that have a cracked border on the SS coupon.

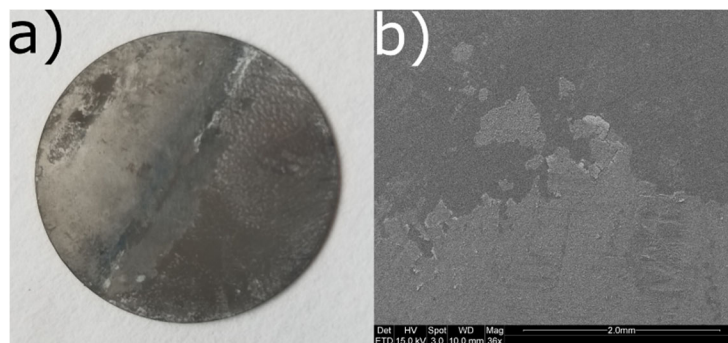


Figure 8. a) Photograph of MOF Grown on 15 Min Etched SS Coupon in Acid, b) SEM Image of Same

Microcrystalline growth on the 90 min etched SS coupons had better coverage than the 15 min etched coupon. However, after solvothermal reaction in acid/base, exposed regions of the SS began to exhibit buildup of rust where MOF crystals had not grown (Figure 9a)). SEM images reveal large microcrystals with structures that suggest impurities or defects.

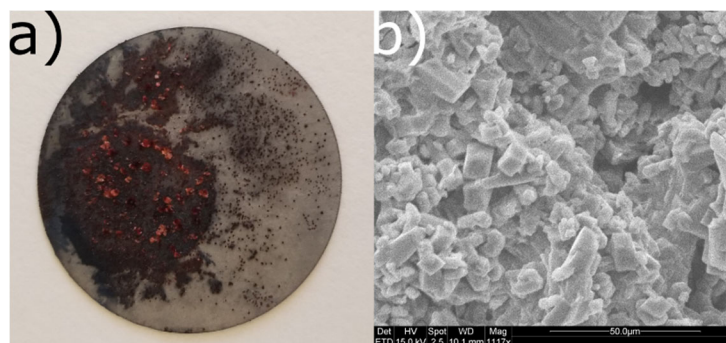


Figure 9. a) Photograph of MOF Grown on 90 Min Etched SS Coupon in Acid and Base, b) SEM Image of Same.

RTI observed good coverage of crystalline material on the surface of the 60 min etched SS coupons (Figure 10). Indeed, the coverage was not uniform but greater than that observed on the 15 min and 90 min etched SS coupons. Under only acidic conditions (Figure 10b) and c)), they observed MOF microcrystals with an approximate diameter of 1 of 3 µm.

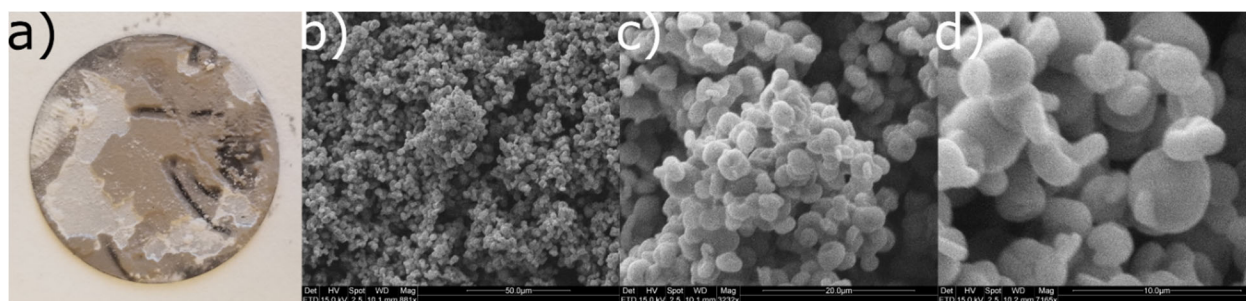


Figure 10. a) Photograph of MOF Grown on 60 Min Etched SS Coupon in Acid, b) and c) SEM Images of Same.

Similarly, with the addition of base to the reaction, RTI observed better coverage of crystalline growth on the 60 min etched SS coupon than for other etching times (Figure 11a)). In contrast to the reaction that only used organic acid, they observed significantly larger crystals throughout the surface (Figure 11b) and c)). These larger crystals exhibit several void defects on their crystal surface, affording opportunity to control the properties of these materials at SS surfaces.

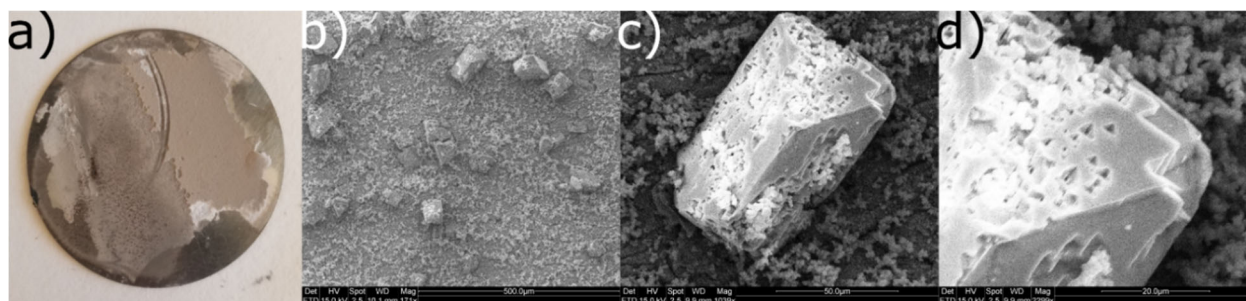


Figure 11. a) Photograph of MOF Grown on 60 Min Etched SS Coupon in Acid and Base, b) and c) SEM Images of Same.

To further probe the quality of the adhesion of these microcrystals to the etched surface, RTI employed a tape test wherein standard office tape was applied to the surface of the etched SS coupon and removed. Figure 12 reveals that for either experiment carried out on the 60 min etched coupons, microcrystalline MOF can be removed from its surface. However, for those MOFs grown in the presence of base (Figure 12b)), the tape test removed far less MOF crystals. A layer of MOF was still present where they applied the tape test to the coupon. This implies that multiple layers of MOF exist on the coupon surface with those grown in the etched spaces exhibiting better adhesion. Future work could focus on improving washing protocols to remove the poorly adhered outer layers.

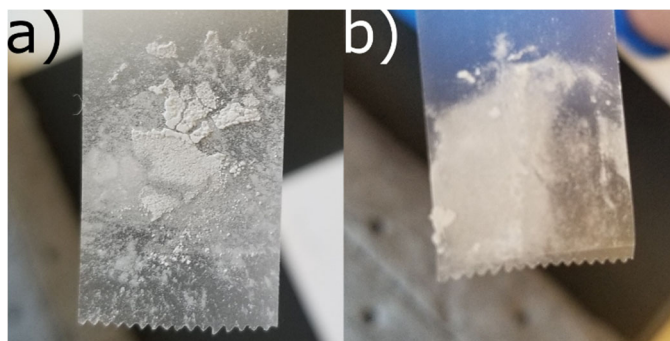


Figure 12. Tape Test Results of MOFs Grown on 60 Min Etched SS Coupons in the Presence of a) Acid and Base, b) Acid Only.

The MOF adhesion trial results show promise for the growth of MOF on etched SS. Etching the SS is a prerequisite to affording crystal growth on the surface; however, the presence of acids/bases in the MOF reaction resulted in corrosion of the SS coupons. Further work would focus on refining the etching time and temperature to afford an optimal surface on which to grow the MOFs.

3.3 HARVESTED WATER POTABILITY EVALUATION

We designed and assembled a test facility for collecting water from the MOF sorbent. Figure 13 shows a schematic of the test facility. Figure 14 shows a photo and points out the key hardware in the facility. In this facility, we chose to submerge the condenser and water collection flask in an ice bath as shown in Figure 15. Submerging our water collection flask in an ice bath serves two functions:

1. Ice water surrounding the flask helps to ensure all possible moisture is removed from the airstream passing through the condenser and flask.
2. Water collected in the flask will remain near 0°C, helping to inhibit potential bacteria growth.

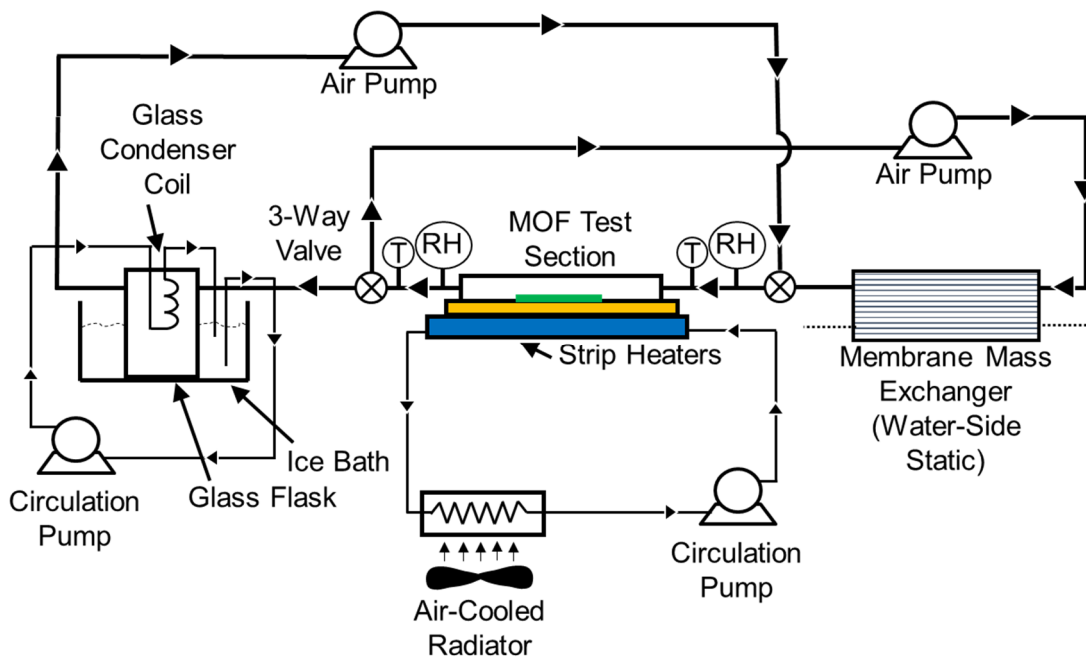


Figure 13. Updated Schematic of Our Closed-Loop Water Harvesting Test Facility

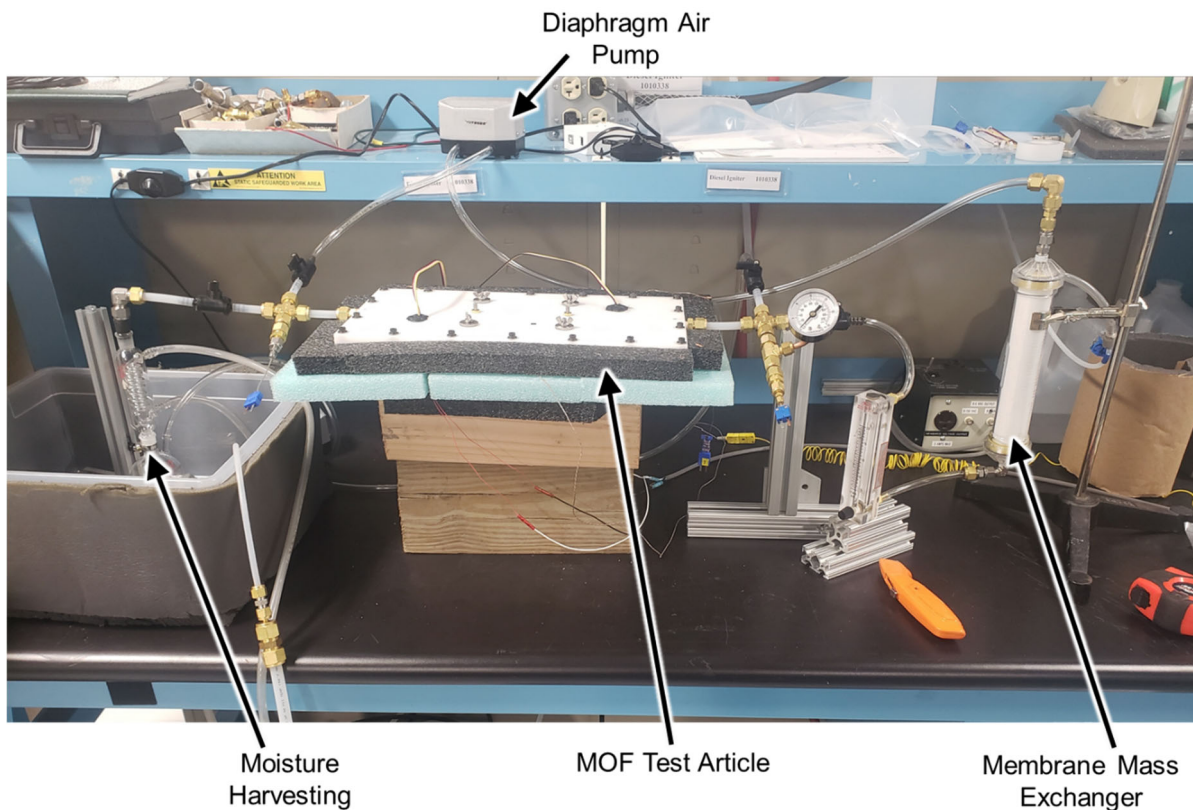


Figure 14. Photo of Our Water Harvesting Facility

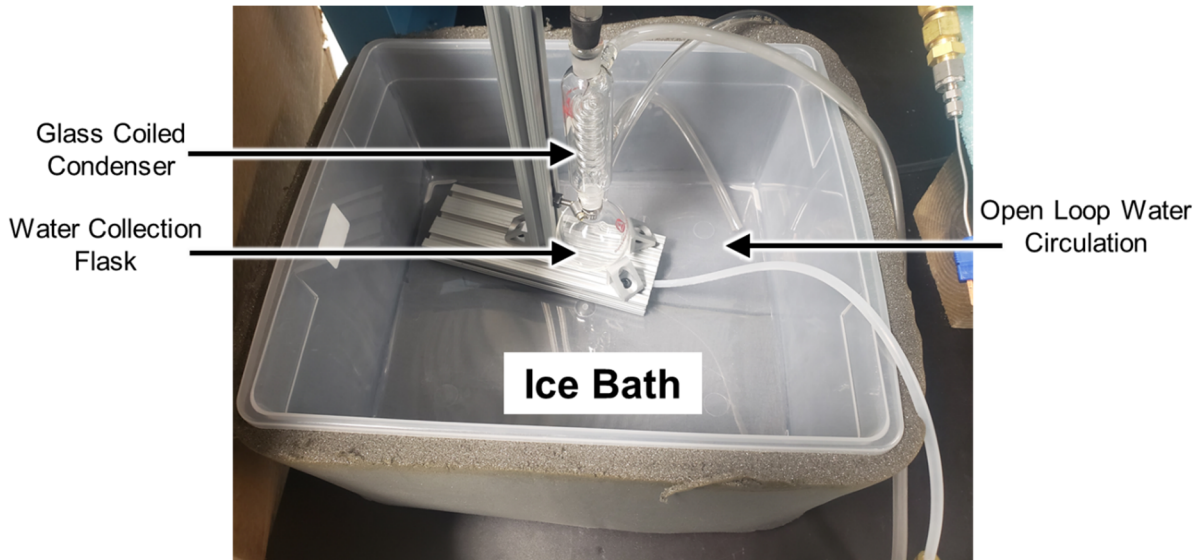


Figure 15. Our Glass Water Collection Hardware is Placed in an Ice Bath to Ensure All Possible Moisture is Extracted and to Reduce the Potential for Bacteria Growth.

As shown in Figure 16, cooling down the facility to create conditions favorable for adsorption was the rate-limiting factor for water production during initial testing. We added an air-cooled water loop to improve cooling of the test article and increase the rate of water production. Adding active cooling greatly accelerated the cycle rate. With this modification, the facility completes ~5 to 10 full sorption cycles per day, with each cycle producing ~100 to 200 mg of water.

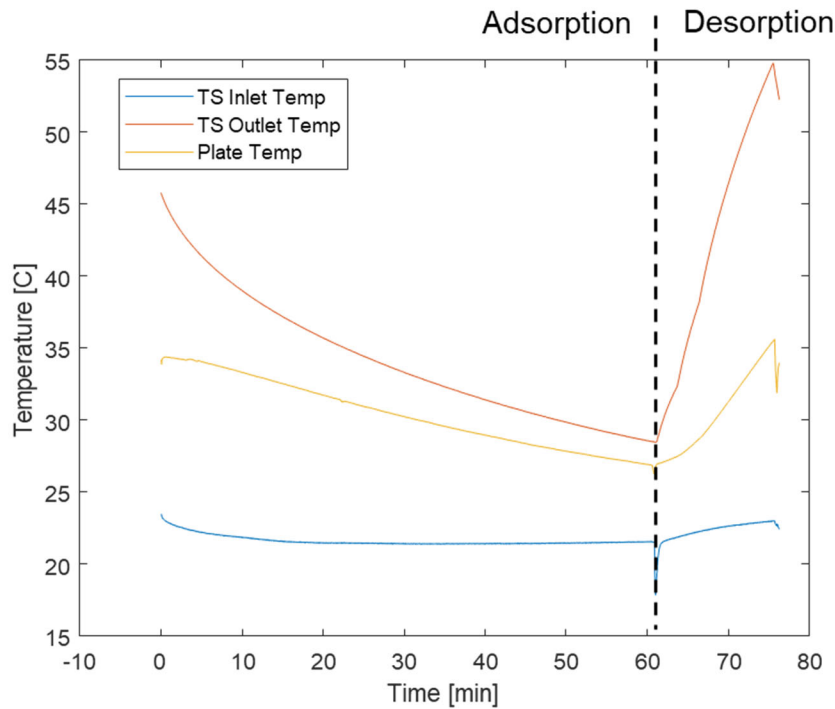


Figure 16. Sample Temperatures During a Single Adsorption and Desorption Cycle

Prior to initial water collection, we sanitized the internals of the water harvesting test facility to ensure a clean starting condition. To produce a control sample, we first collected water by evaporating from the membrane mass exchanger and condensing directly into the glass condenser (no adsorption/desorption with MOF-coated sample). We sent this control water sample to CDI for initial testing to ensure that we did not introduce any contamination into the test loop that could confound the MOF water potability assessment. We also sent CDI a sample of the distilled water used to charge the test facility. Along with the control sample, CDI also tested this charge water to ensure it does not contain any contaminants.

We then proceeded with cyclical adsorption and desorption using the Phase I MOF-coated sample. Figure 17 shows MOF-coated plate temperature over a period of three days. We produced a total of 15 mL MOF-collected water for sample testing by our collaborators at CDI.

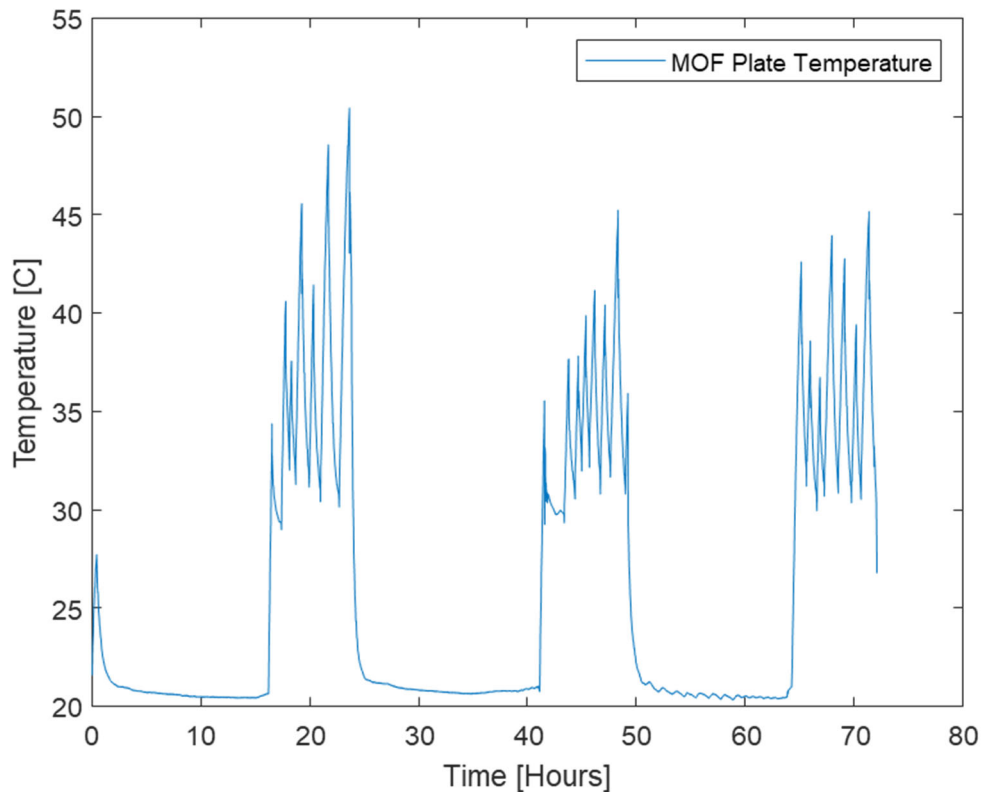


Figure 17. Multi-Day Test History of Our Water Production Facility

CDI tested the sample along with the two control samples to the TBMED 577 standard for short-term water potability outlined in the “Short-term potability military field water standards” (TBMED 577 standard; Table 4-2). Five different categories for characterizing the contaminants in drinking water comprise the TBMED 577 standard: physical properties, chemical properties, microbiological properties, chemical warfare agents and radiological contaminants. CDI’s water quality analysis only focused on the relevant physical properties and chemical properties.

The limited volume of water (15 mL) created by the Phase I prototype meant that there was not enough water available for measurements of several criteria listed under the physical, chemical, and microbiological properties. Since the water collected was not sampled when there were any chemical warfare agents or radiological species present in the intake air source, CDI did not attempt to analyze the harvested water from the Phase I prototype for these contaminants. The analysis undertaken by CDI aimed to show whether or not the harvested water from Creare's Phase I prototype complied with the physical and chemical properties of the TBMED 577 standard summarized in Table 3.


| Table 3. TBMED 30-Day Standard Analysis Properties and Applicability to the MOF-Collected Water Sample | | |
|--------------------------------------------------------------------------------------------------------|-----------------------|----------------------------------------------------------------|
| Parameter | TBMED 30-Day Standard | Sample Analyzed for This Parameter |
| Physical Properties | | |
| Color | < 15 CU | No – Insufficient Volume |
| Odor (PM evaluation and customer response) | Acceptable | No - Needs to be measured for the stream exiting the prototype |
| pH | 5 – 9 pH units | Yes – Measured at CDI |
| TDS | < 1,000 mg/L | Yes – Measured at CDI |
| Temperature | 15 – 22 °C | No – Needs to be measured for the stream exiting the prototype |
| Turbidity | < 1 NTU | Yes – Measured at CDI |
| Chemical Properties | | |
| Arsenic | < 0.02 mg/L | No – Insufficient volume |
| Chloride | < 600 mg/L | Yes – Measured at Amtest |
| Cyanide (as free cyanide) | < 2.0 mg/L | No – Insufficient volume |
| Magnesium | < 30 mg/L | Yes – Measured at Amtest |
| Sulfate | < 250 mg/L | Yes – Measured at Amtest |

In addition to the different water quality metrics listed in the TBMED 577 standard, the harvested water from Creare's Phase I prototype was also analyzed for the presence of zirconium. CDI tested the samples for zirconium to detect any of the MOF present in the water sample. The presence of zirconium in the harvested water would be a sign that the water harvesting material was becoming detached from the water harvester during operation.

Table 4 and Table 5 summarize the test results. The tests evaluated four different water samples defined as the following:


- Harvester Control is store-bought distilled water used to charge the test facility.
- Baseline/No MOF is store-bought distilled water that has been cycled through the test rig via direct evaporation and condensation to monitor leaching from the test rig loop.
- MOF is store-bought distilled water cycled through the test rig via adsorption and desorption in the MOF absorbent.
- MOF-Control is CDI's on-site deionized water source produced via packed-bed ion exchange resin filtration. CDI used MOF-Control water to dilute the MOF sample water 1:4 before sending it to Amtest Laboratories (Kirkland, WA) for third party testing in order to meet their absolute minimum volume requirements.

| Sample | pH | Conductivity (µS/cm) | TDS (mg/L) | Turbidity (NTU) |
|---------------------------|------|----------------------|------------|-----------------|
| TBMED 577 30-Day Standard | 5–9 | N/A | < 1,000 | < 1 |
| Harvester Control | 5.88 | 8 | 5 | 0.2 |
| Baseline/No MOF | 5.51 | 36 | 23 | 0.2 |
| MOF | 4.25 | 226 | 145 | 1.3 |
| MOF-Control | 6.41 | 4 | 3 | 0.3 |

 = PASS

| Sample | Magnesium (mg/L) | Chloride (mg/L) | Sulfate (mg/L) | Zirconium (mg/L) |
|---------------------------|------------------|-----------------|----------------|------------------|
| TBMED 577 30-Day Standard | < 30 | < 600 | < 250 | N/A |
| MOF | Non-Detect | 46.4* | 0.64* | Non-Detect |
| MOF-Control | Non-Detect | Non-Detect | Non-Detect | Non-Detect |

*Measured value was quadrupled to compensate for dilution factor

 = PASS

The MOF sample showed a measured TDS value of 145 mg/L, which is well below the maximum of 1000 mg/L. Additionally, the measured chloride, sulfate, and magnesium values were well below the thresholds outlined in the TBMED 577 standard. The water quality analysis showed

that zirconium was not detectable in the product samples via the EPA 200.7 test method, suggesting the MOF material remained intact during sample generation.

However, the measured pH of 4.25 was outside the required range specified in the TBMED 577 standard, along with the turbidity that was measured at 1.3 NTU (1 NTU is the requirement). The depressed pH value may be due an elevated CO₂ concentration in the adsorbed water as the MOF material tends to capture both water and CO₂ from the surrounding air. As such, there may be a need for the addition of a post filter to the system to bring the pH back into the TBMED standard's acceptable range. A microfiltration-based post filter will further ensure that there are no bacteria coliforms in the harvested water.

4 CONCLUSIONS AND RECOMMENDATIONS

4.1 CONCLUSIONS

Conclusions based on the Phase I Base and Option efforts are as follows:

- Growth of MOF sorbent directly on the MWX SS substrate appears to be a viable approach for increasing the amount of MOF on the coated plates.
- No MOF material was present in harvested water. pH and turbidity were outside the TBMED 577 requirements; however, CDI believes that a polisher post-filtration step can ensure compliance with TBMED 577 standards.

4.2 RECOMMENDATIONS

We recommend proceeding with the Phase II effort to develop and demonstrate the MOF water harvesting technology.

We also recommend considering additional funding for R&D focused on optimizing the MOF coating considering equilibrium water sorption characteristics, coating mechanical properties, and kinetics of water transport in the coating. This activity would consider a broader range of coating formulations than can be addressed in the Phase II project.

5 PHASE II PLANS

The overall goal of this program is to enable soldiers to produce drinking water in the field by harvesting moisture from the atmosphere. In Phase II, we will advance toward this goal by demonstrating a prototype water harvesting system using Creare's MWX technology. Table 6 shows the specific technical objectives of Phase II and our planned approach to achieve them.

| Objective | Technical Approach | Method for Demonstrating in Phase II |
|---------------------------------------|------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Prototypical Materials and Structures | MOF-801 sorbent coatings on compact heat/mass exchanger surfaces | <u>Sorbent</u> : RTI will formulate MOF coatings and apply them to SS MWX plates supplied by Creare. <u>Compact MWX</u> : We will produce a compact MWX core from the coated plates using HAM based on automated, precision laser welding. |
| Water Production Rate | Use as much sorbent as possible | The MWX sorbent beds are assembled from multiple modules. The Phase II prototype will contain one 100-plate coated module, which will produce 1/5 the total target water production rate (14/5 = 2.8 L/day). Additional coated modules can be produced and swapped in for post-Phase II demonstrations. To minimize NRE costs, we will use an existing crossflow recuperator design for the contactors. |
| Self-Contained | The Phase II prototype will operate independently | Components will be integrated into a close-coupled system with a compact form factor. All power and heat will be supplied by onboard batteries and fuel. |
| Compact and Lightweight | Efficient heat and mass transfer | Arrays of uniform, closely spaced channels for heat and mass transfer in the MWX core will enable efficient water scavenging with low pressure losses. Measurements of water production rate and prototype size and weight will provide direct validation. |
| Low Signature | Minimize fan noise and exhaust temperature | We will use low-noise commercial fans for airflow. Burner exhaust will be mixed with sorbent bed through-flow to reduce temperature. We will measure noise levels and the IR signature of the Phase II prototype to validate. |
| Full-Scale Air Handling Capacity | Fans and system flow resistance will be sized to meet water production requirement | Airflow requirements will be determined based on the range of expected environmental conditions and expected water harvesting efficiency of the MWX. Test setup will include methods for direct measurement of airflow through the system. |
| Detailed Performance Data | Prototype will accommodate test instrumentation | The prototype will be built with nonproduction features that will enable detailed performance measurements. We will measure the airflow rate, fuel consumption rate, temperatures, RH, and pressures at key points throughout the system. |
| Potable Water Production | System will include water purification steps | Incorporate water purification hardware supplied by CDI. Test product water for potability in CDI's test labs. |
| Test Conditions | Simulate operation under design-basis conditions | Air conditioning system will include RH, temperature, and flow rate control, plus a pressure-balancing/flow measurement system for system through-flow. |