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Lunar Applications of Direct Contact Membrane Distillation For Regolith And Hydroponic Water Processing

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LUNAR APPLICATIONS OF DIRECT CONTACT MEMBRANE DISTILLATION FOR REGOLITH AND HYDROPONIC WATER PROCESSING

by

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ABSTRACT

Water management has always been a critical issue facing lunar missions, since its uses are as numerous as they are vital. Using ionic liquids has been proposed for the purpose of processing the metal oxides in lunar regolith into oxygen and metals. The process used dilutes the ionic liquid. Recycling the ionic liquid solution after regolith processing is essential to reduce materials required in resupply missions and to supply oxygen. Water will also be needed for lunar farms to both grow food and assist in maintaining a habitable atmosphere. The use of direct contact membrane distillation (DCMD) technology is a method of water purification that was tested for viability in both concentrating ionic liquid streams and in hydroponic salt based nutrient streams. It was found that the ability of PTFE and PVDF based polymer membranes to separate solutes from water held potential in concentrating aqueous solutions and in recovering water from aqueous solutions. This was tested using a bench scale DCMD system to perform the operation at the temperatures of 50 °C, 65 °C, and 80 °C. The results were tested using UV-Visible spectroscopy to determine concentrations. Through this method, the benefits and limitations of PTFE and PVDF membranes were found when operating at various temperatures with either aqueous ionic liquid solution or simulated hydroponic nutrient solutions.

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Author ___________Mark J. Wong___________

Date ________4/15/23_________________

DEDICATION

I would like to thank all my friends and family for providing me with the support needed to complete this thesis and project.

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ACKNOWLEDGMENTS

I want to acknowledge and thank Dr. Joan Lynam for giving me this opportunity and her continued support throughout these projects. I also wish to thank Dr. Sven Eklund for his help in analyzing my samples and teaching me how to and allowing me to operate laboratory equipment, particularly the UV-Visible spectrophotometer and the other equipment present in the Carson-Taylor chemistry building. I also want to thank my fellow graduate student lab mates, Viral Sagar, Mohammad Tarikuzzaman, and Lauren Mekalip. I also would like to thank the undergraduate researchers who contributed to these projects.

CHAPTER 1

INTRODUCTION

1.1 Background Information

1.1.1 Direct Contact Membrane Distillation

Direct contact membrane distillation (DCMD), is a novel method of separating water from aqueous solutions. It has found purposes ranging from desalination plants to dairy processing. The system is based on a temperature differential between two aqueous solutions being passed on top of each other with a thin porous hydrophobic membrane [Tai, 2019]. During the process, the temperature gradient results in a vapor pressure differential. The porous polymer membrane allows for water vapor to form from the high temperature stream and condense into the low temperature secondary stream. This, in theory, allows for only water to pass through the membrane as the carrier solution is repulsed by the hydrophobic membrane or the solute molecules are too large to pass through membrane pores. The primary advantage of DCMD processes is that they do not require the aqueous solution to be fully vaporized. This reduces the complexity of the operation significantly, thus reducing the construction size and energy requirements. This is in direct contrast to the most common method of aqueous separation, in which the majority of the liquid is vaporized and condensed leaving a solute rich base and water rich condensate. Methods used to accomplish this operate by either providing energy, most commonly in the form of heat, to the solution to vaporize large portions of the solution, or reducing the atmospheric pressure through vacuum systems to allow for vaporization at a lower temperature [Tai, 2019]. These operations, however, have major downsides as heat based vaporization is extremely energy intensive and requires cumbersome infrastructure. Vacuum base methods are reliant upon a complex system of interconnected pumps that are all subject to variations and their own individual process control systems and possible failure points.

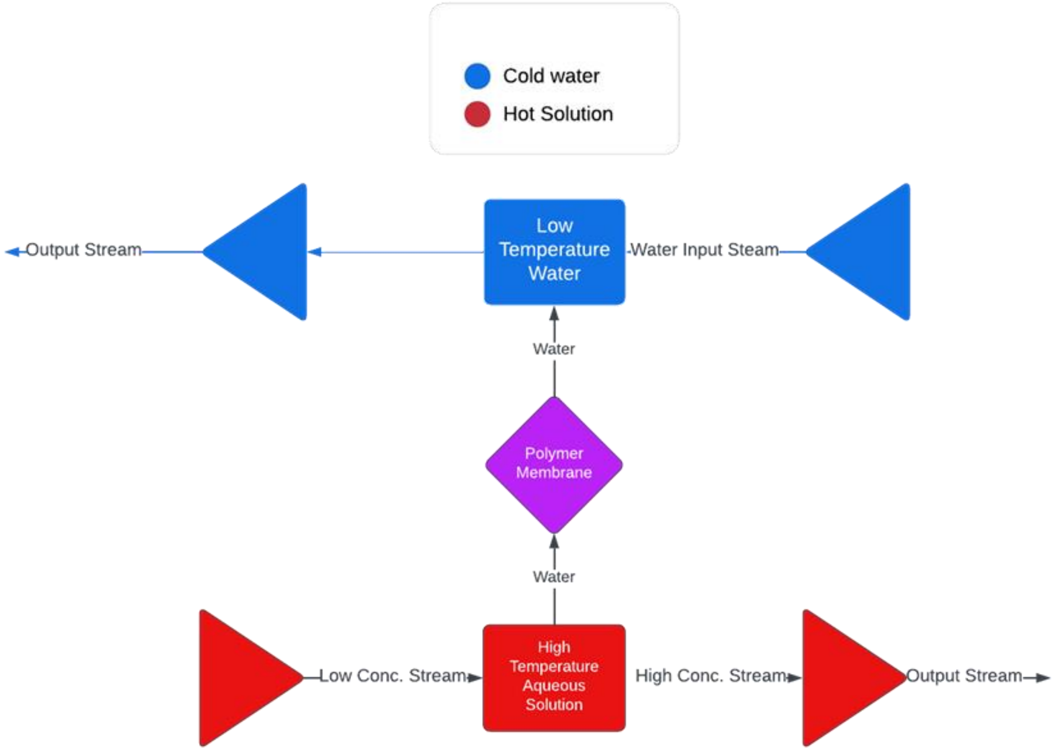

Figure 1-1: An Overview of the DCMD process.

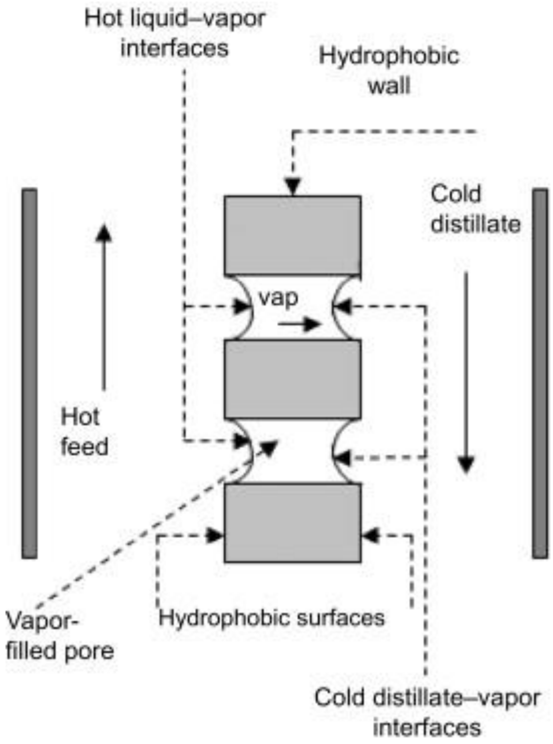

Figure 1-2: Detailed diagram of the DMCD vapor exchange process top view (Pal, 2015).

1.1.2 Membrane Distillation

Membrane based distillation has been developed and shown results since the 1960's [Kabay, 2022], however this method has always suffered from the issue of polymer technology. The crux of the process is reliant upon the use of a hydrophobic polymer based membrane, and its effectiveness will drastically change the effectiveness of the process. While advances in membrane and polymer technology have increased the viability of membrane based systems, there are still issues within the technology that must be addressed. Issues include the costs and quality of the membranes. Quality issues in the membrane result in degradation of the membrane, at rates dependent upon the solution being processed and the quantity of the solution being pumped across the membrane. Currently, research is needed to understand how membranes transfer both energy and mass with respect to varying molecule size [Kabay, 2022].

1.1.3 UV-Visible Spectroscopy

UV-Visible spectroscopy is a measurement tool that operates by sending a thick stable beam of light through a sample and measuring the change in the strength of the light beam on the opposite side of the sample. The beam used will change through a selected range of wavelengths. Depending on the compound being tested there will be a change in absorbance at different wavelengths. As concentration increases the absorbency will increase. This allows for concentrations in samples with compounds that absorb light to be quantifiably measured.

1.2 Extraterrestrial Challenges

As humanity proceeds to reach for the stars, we face many challenges to overcome. The biggest of these challenges are the basic life support systems that will be required to ensure the survival of astronauts for the long time periods required for extended missions. A functional life support system at the minimum must provide the capability to provide solutions to three problems; the need for oxygen, the need for water, and the need to remove carbon dioxide [Jackson, 2016]. The ability to meet these challenges is a primary barrier to extraterrestrial expansion. While recycle processes are invaluable, no process can be 100% efficient and as a result there will always be the need to accrue new water and new oxygen. While oxygen can be synthesized using water via electrolysis in a manner similar to that which is used on modern military naval vessels, liquid water is a rare resource in our local region of space, with the majority of lunar water being concentrated in poles in the form of ice [Feldman, 2001]. Various methods have been proposed to handle this issue. One promising solution has been to process metal oxides to obtain the embedded oxygen and use that to react with hydrogen to create usable water [Schwandt, 2012]. This method is supported by the discovery that the earth's moon contains large quantities of metal oxides such as iron and titanium oxide [Papike, 1982].

While a less critical issue, food production will be a hurdle that once overcome will increase the viability of off-world exploration. The issue of soil has long been problematic. Experiments have been conducted and shown some results in growth in lunar soil, particularly with the prospect of genetically modified plants [Paul, 2022]. A popular method to address the issues faced is to use hydroponic and aquaponic systems. The key difference between the two is whether other organisms are used to regulate water quality, as in aquaponics. These systems would allow for minimal soil to be used as a base for the plants, while also producing increased yields for similar spatial requirements. However, this does incur increased energy use compared to conventional farming methods [Barbosa, 2015].

1.3 Membrane Separations and Extraterrestrial Applications

Membrane separations offer the ability to separate water from streams in a manner that is energy efficient with low spatial requirements [Barbosa, 2015]. Membrane separations do not require large-scale vaporization, meaning that complex and large distillation tower systems may not be needed for successful water reclamation. Membrane separations also provide the advantages offered by vacuum distillation without the complications of this kind of system, since recycled or waste heat sources could be used. In this way, the amount of extra energy needed to be imputed into the system could be reduced [Tai, 2019].

A new approach to solving oxygen requirements on the moon is to use ionic liquids to process the metal oxides common in lunar regolith into water using the oxygen form the metal oxides and hydrogen that can be recycled. The use of ionic liquids have allowed for the operating temperature to be dropped to below 200 °C [Barrios, 2011]. Water, however, will permeate and dilute the ionic liquid output stream. The ionic liquid solution must then be processed to remove the water from the solution before re-use. The water can then be processed using electrolysis to produce oxygen gas and allow the hydrogen to be recycled back into the process. Membrane based distillation was this study's proposed method to complete this process.

Hydroponic solutions proposed for lunar missions have the advantage of producing both food crops while simultaneously regulating the oxygen and carbon dioxide concentrations of the air [Fu, 2016]. Issues facing hydroponic systems are the regulation of the nutrient stream feeding the plant. The nutrient solution consists of various salts that are usually present in living soil. This usually includes the some of the 17 elements commonly used to support plant growth: carbon, hydrogen, potassium, phosphorus, calcium, nitrogen, magnesium, iron, copper, sulfur, manganese, zinc, boron, chlorine, molybdenum, nickel and oxygen. The balance of the microbiome within the root system of plants must be maintained properly as well. If the nutrient concentration is too high the root systems can be damaged or undesirable organisms such as mold can proliferate through the system. If the nutrient concentration is too low the plants will have decreased yields and may not grow properly at all [Asao, 2012].

For terrestrial operations this can be accomplished by routinely dumping water and flushing the system with fresh water and restarting the system's nutrient system. In a lunar environment this would not be a preferable solution to the issue. Instead a new form of regulating the concentration of the nutrient system would be needed. Membrane based distillation offers the potential to remove water from the stream thus allowing for it to be concentrated while staying in the process and lessening the need to purge the system.

CHAPTER 2

MATERIALS AND METHODS OXYGEN PRODUCTION FROM LUNAR REGOLITH

2.1 Material Preparation

For the scope of the project conducted from September of 2021 to August of 2022 it was assumed that the operation to process the metal oxides from lunar regolith was already conducted and successfully resulted in a diluted ionic liquid solution. The solution's initial target concentration was 20%. This was chosen as it was the concentration tested in previous studies [Barrios, 2011].

2.2 DCMD Trials

The solutions were processed by using direct contact membrane distillation (DCMD). The setup was done by having a heated aqueous ionic liquid solution pumped on the bottom of a polymer membrane while cooled water was pumped on the top layer of the polymer membrane. The temperatures of both the cooled and heated solutions were kept constant by the use of water baths. The temperature of the cooled water was kept constant with a water bath kept at 5 ℃. The heated water bath temperature varied at 50, 65 and 80 ℃. Two runs were conducted at each of these temperatures for two hours at a feed rate of 200 ml per min on each side of the membrane. After all three temperatures were tested twice the membrane was changed from a Polytetrafluoroethylene (PTFE) to a polyvinylidene (PVDF) polymer based membrane (Sterlitech, Kent, WA, USA: PTFE

Flat Sheet Membrane, Polypropylene Netting Backer, Laminated, 0.45 Micron, Sepa, Lot#: J000014897 11-1; Novamem Flat Sheet Membrane, PVDF100, 0.1 Micron, MF, Sepa, Lot#: 624096PVDF100).

2.3 Sampling Method

During DCMD the solutions were sampled at 30 min intervals by removing 50 ml samples from the ionic liquid solution. This was done both initially before the process began and after as well. The final water was saved as well to test for transfer of ions across the membrane. Testing was done by using two methods, conductivity and UV-Visible spectroscopy analysis.

The mass of the cool side water before and after the run was taken as well. This was done in order to determine the total flux of the water over the time span of the experiment.

2.4 Testing Materials

During DCMD the solutions were sampled at 30 min intervals by removing 50 ml samples from the ionic liquid solution. This was done both initially before the process began and after as well. The final water was saved as well to test for transfer of ions across the membrane. Testing was done by using two methods, conductivity and UV-Visible spectroscopy analysis.

The mass of the cool side water before and after the run was taken as well. This was done in order to determine the total flux of the water over the time span of the experiment.

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Conductivity and Absorption Calibration

The effects of membrane polymer material and feed temperature on the ability to concentrate ionic liquids was explored and tested.

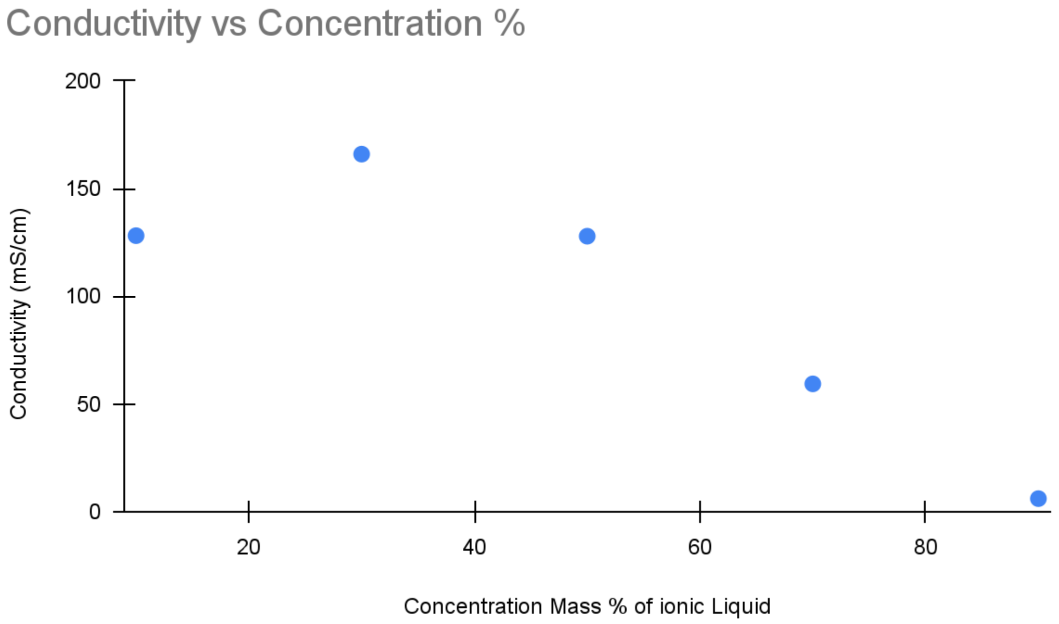

Figure 3-1: Calibration curve created to establish conductivity to mass percentage of ionic liquid relationship.

It was found that concentration of ionic liquid and conductivity would result in a nonlinear relationship. This resulted in a polynomial relationship between the conductivity of the solution and the concentration of the solution. This method was also found to have a large variation within it and therefore the UV-Visible spectroscopy method was used in conjunction with conductivity to provide more reliable data. The conductivity was used therefore in an ancillary role to the UV-Visible spectroscopy.

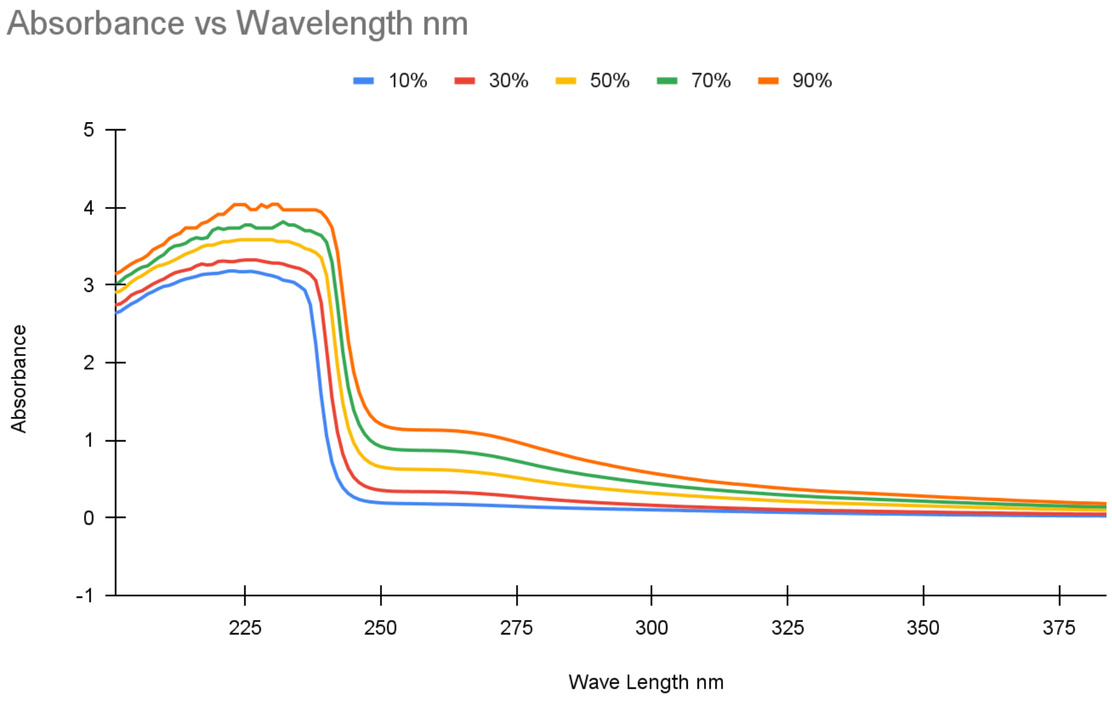

Figure 3-2: Calibration curve created to establish the relationship between mass percentage of ionic liquid and light absorption.

The calibration curve for the UV-Visible spectroscopy showed a typical curve that would be expected for a solution made primarily of two non-water components. This resulted in two peaks both in the ultraviolet range. The first peak was at 238 nm and the second was at 265 nm. The first peak at 238 nm was neglected as all samples would result in an absorbance peak above 2, a condition that degrades the quality of the data, as the light sensor begins to become overloaded. The second peak at 265 nm was instead used as the primary measuring tool.

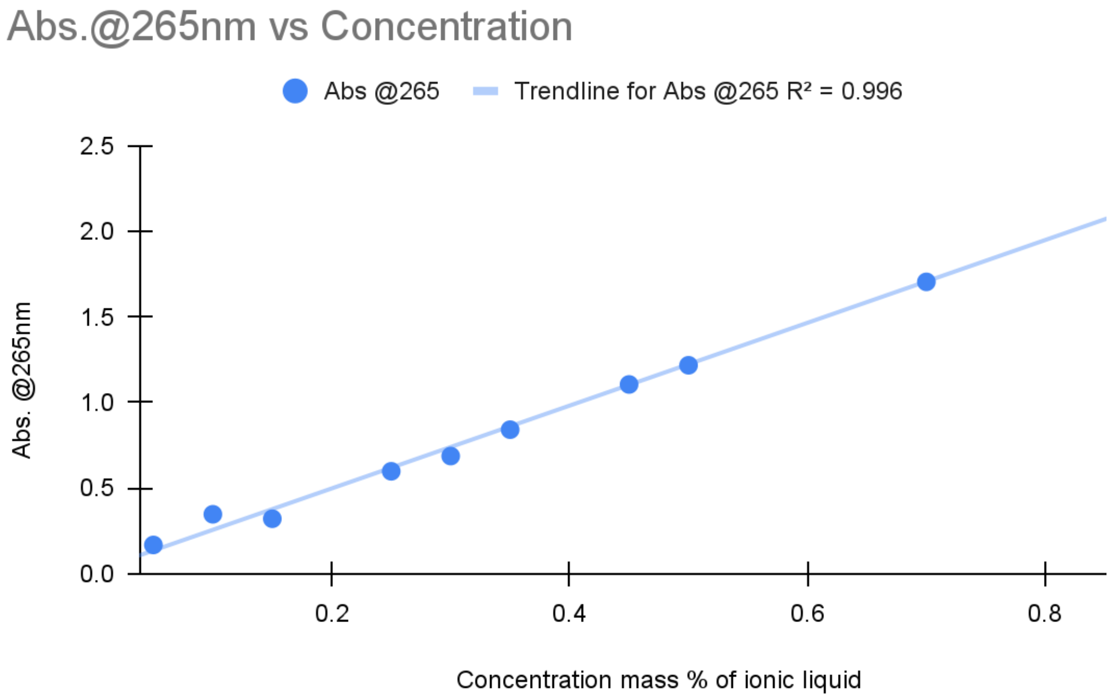

Figure 3-3: Calibration curve measuring the absorbency of light at 265 nm at five different mass percentage of ionic liquid.

A chart was then made using the absorption of light at 265 nm for both calibration curves and combined. It found that the concentration of ionic liquid and respective absorbance was linearly related. This allowed for all calculations that needed to be performed to be considered directly linearly proportional.

3.2 Experimental Analysis

UV-Visible spectra was then obtained for all samples taken during the experiment's 12 trials. For the sake of manageable brevity not all 12 trials will be discussed.

Table 3-1: Total average change of solution properties with PTFE membrane at various temperatures.

Temperature (°C)	Total water exchange (g)	Average flux 9g/m^2*hr)	Change in Concentration
50	4.61	0.16	3.71%
65	14.98	534.82	12.52%
80	44.66	1595.00	22.83%

Table 3-2: Total average change of solution properties with PVDF membrane at various temperatures..

Temperature (°C)	Total water exchange (g)	Average flux (g/m^2*hr)	Change in Concentration
50	15.58	556.25	1.44%
65	3.83	136.79	2.16%
80	22.47	802.32	17.67%

Table 3-1 and **Table 3-2** show the overall change in the ionic liquid solution based upon the material of the membrane and the temperature of the run. Important to note is that during the trials for PTFE it was found that after ninety minutes of operation the change in the solution was negligible at low temperatures and the operation was only carried out for 90 mins. This time was increased to 2 hours when the data indicated a significant increase in time was required to reach significant changes as the temperature of the feed was increased.

The difference in results is most evident when comparing the two membranes. The PTFE membrane was overall significantly more effective at transferring the water from the feed stream. **Table 3-1** and **Table 3-2** demonstrate that at the 80 °C the average water flux across the membrane was nearly double that of the PVDF. It can also be seen that the concentration of the ionic liquid also changed more with the use of the PTFE membrane as opposed to the PVDF membrane.

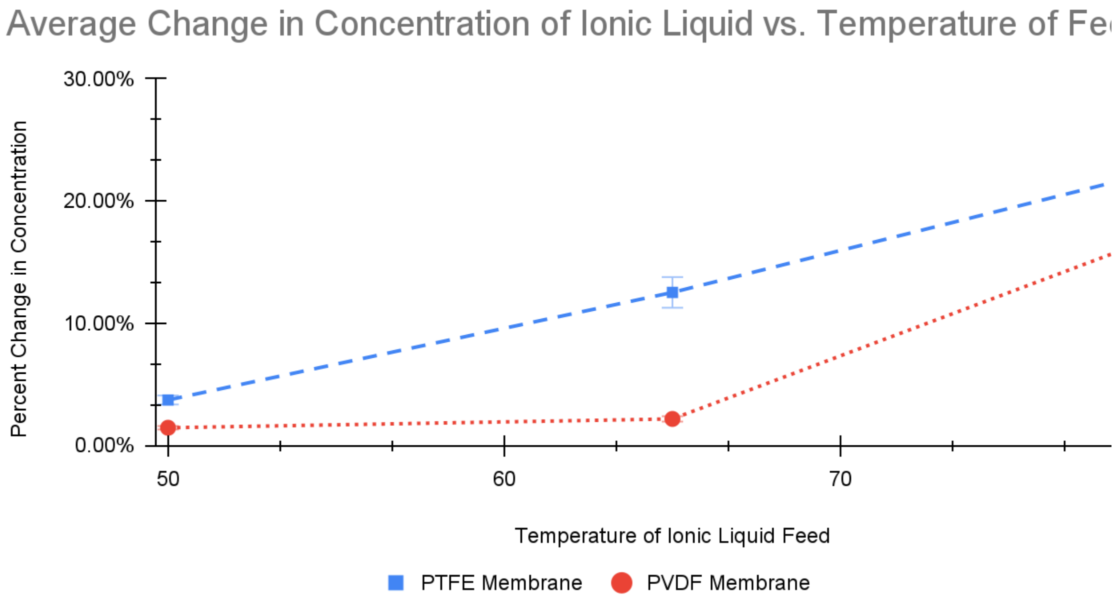

Figure 3-4: Effect of temperature on the change in concentration of ionic liquid solution.

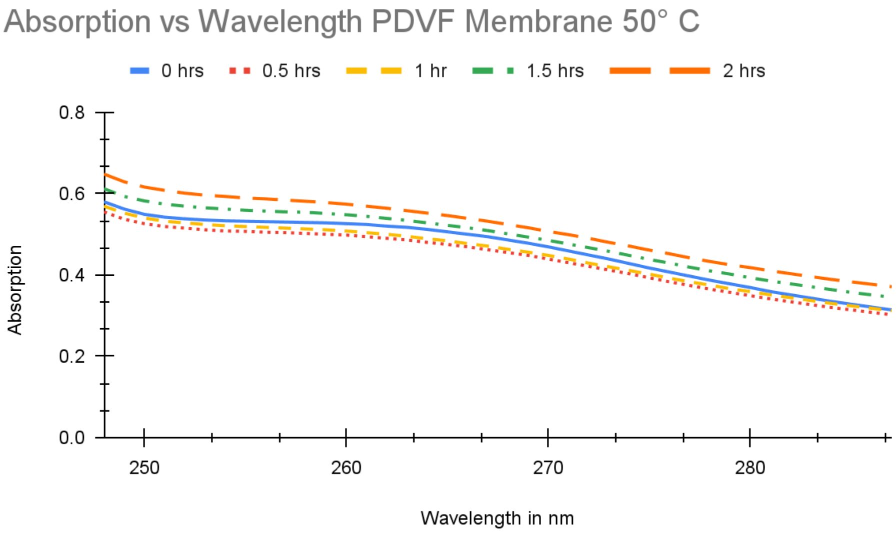

Figure 3-5: Absorption spectra of ionic liquid solution at various operation times for a PVDF with feed temperature of 50°C.

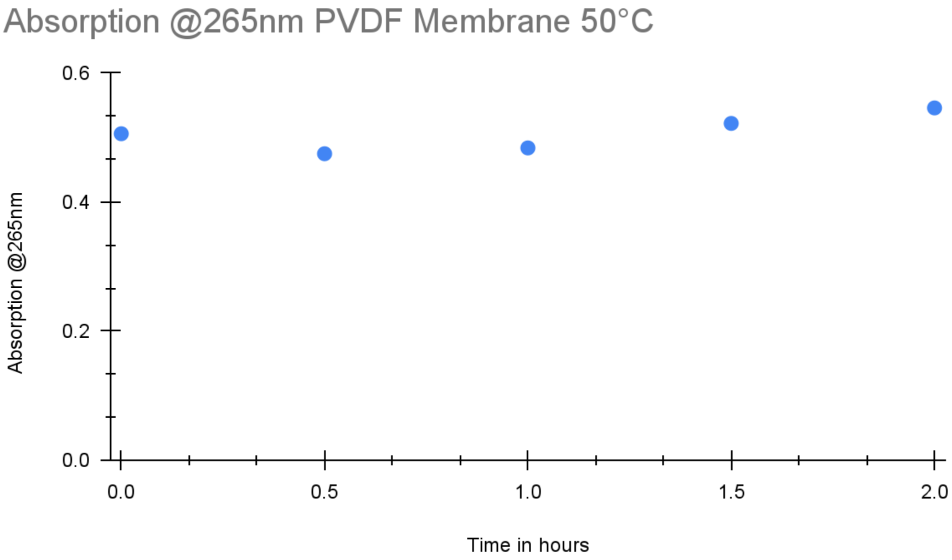

Figure 3-6: Figure 3-6: Absorption of light at 265 nm, of ionic liquid solutions at various operation times for a PVDF membrane with feed temperature of 50°C.

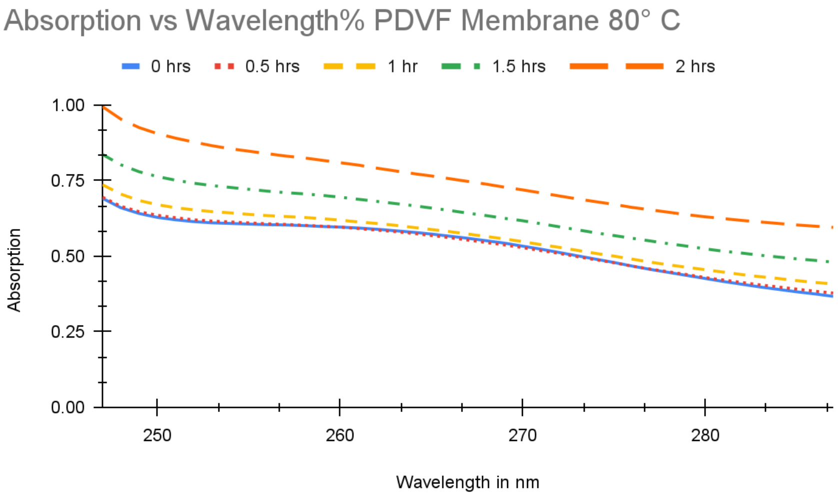

Figure 3-7: Absorption spectra of ionic liquid solutions at various operation times for a PVDF membrane with feed temperature of 80°C.

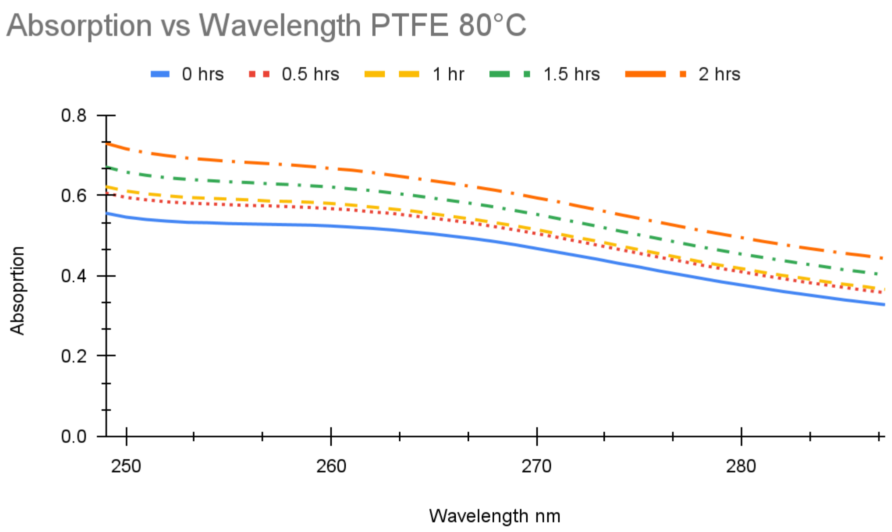

Figure 3-8: Absorption spectra of ionic liquid solution at various operation times for a PTFE membrane with feed temperature of 80℃.

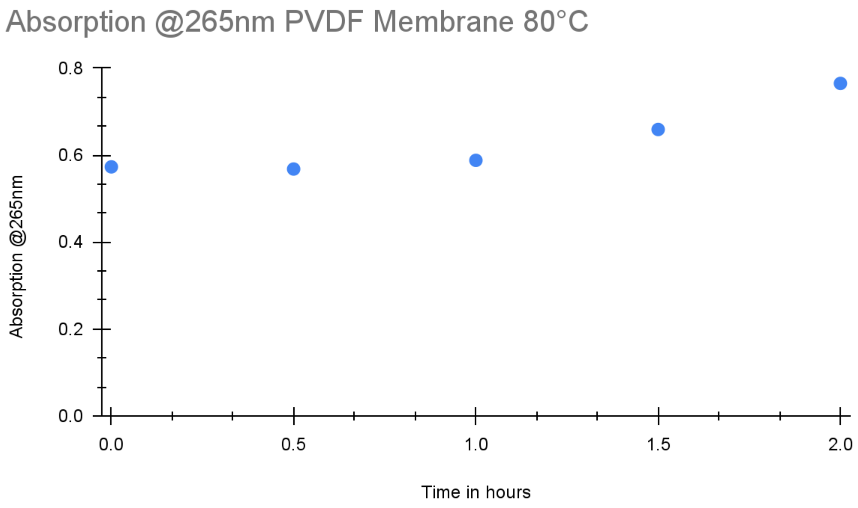

Figure 3-9: Absorption of light at 265 nm, of ionic liquid solution at various operation times for a PVDF membrane with feed temperature of 80℃.

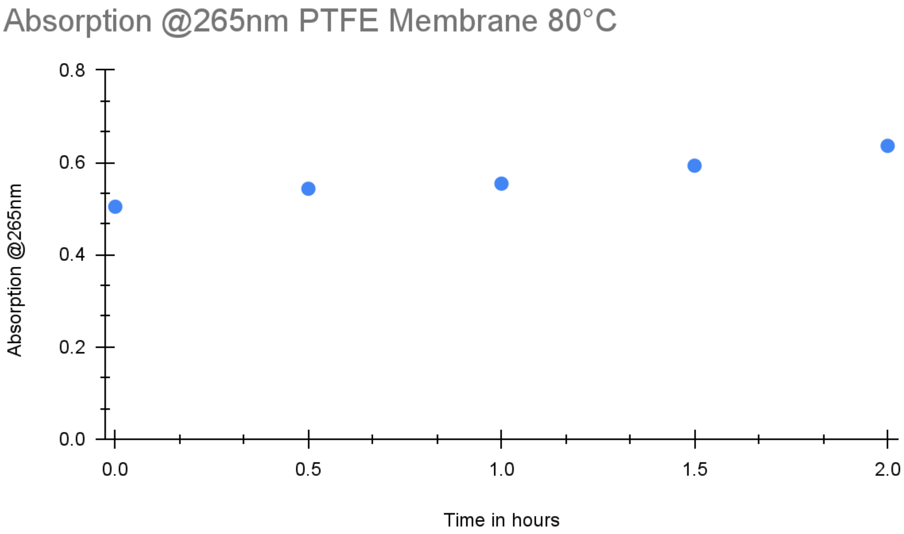

Figure 3-10: Absorption of light at 265 nm, of ionic liquid solution at various operation times for a PTFE membrane with feed temperature of 80°C.

Figure 3-4 demonstrates that both the PVDF and PTFE membranes at high temperatures transfer water across the membrane in a polynomial manner. However the PTFE membrane is significantly more linear. PTFE membranes transfer water at a higher rate at equivalent temperatures then their PVDF counterparts. This does come with the downside of increased permeate contamination. As can be seen from both **Figure 3-9** and **Figure 3-10** both membrane types result in some decrease in concentration at some point in their respective curves. This resulted in suspected contamination of the water supply that was being used as one of the product streams. This was confirmed by testing the conductivity of the final water product stream. A possible explanation for this difference is the pore size difference between the two membranes. The PTFE membrane used had a pore size of 0.45 microns and the PVDF membrane had a pore size of 0.1 microns. This difference could explain the change in contamination rates of the distillate.

Table 3-3: Change in water conductivity based on feed temperature and membrane type.

Feed Temp (°C)	Δ Conductivity PTFE (us/cm)	Δ Conductivity PVDF (us/cm)
50.0	165.0	14.8
65.0	1162.0	85.8
80.0	1560.9	259.4

Table 3-3 demonstrates that the cost of increased water recovery from the PTFE membrane is the increase in contamination for the water product stream. While this contamination was low it would still represent a consideration for usage in long term water purification methods. This also suggests that across the membrane there is some transfer rate of ions that is outpaced by the transfer of the water vapor. It is likely that there is a minimum operating temperature differential required between the two streams needed to ensure that the rate of water permeating through the membrane outpaces the rate of the solute.

CHAPTER 4

CONCLUSION

4.1 Conclusion in Ionic Liquid Concentration

Overall, the tests performed demonstrate the potential for DCMD technology to be used to operate in a water recovery system for ionic liquids after use in lunar regolith processing. Both membranes tested offered advantages and disadvantages for use in water recovery depending on the end purpose of the water permeate. For purposes in which the water quality can be seen as flexible the PTFE membrane operating at a high temperature would be the preferable option. These DCDM operating conditions would offer a larger change in concentration of the ionic liquid, allowing for a faster recovery period before it can be reused in the initial process. However, this choice would be made at the cost of contaminating the permeate at a significantly faster rate than its PVDF counterpart. The downside of water contamination would need to be accounted for. In situations where the water quality must be maintained as high as possible, such as for drinking water, then the PVDF membrane would be the preferred method. This membrane resulted in decreased water recovery rate at the benefit of a decreased rate of contamination of the permeate. It is clear however that both membranes offer viability in the ability to recover water from post process ionic liquids, resulting in a product stream of increased concentration.

4.2 Future Work

More studies should be performed to study the life span viability of a variety of membrane materials. This study showed the viability of PTFE and PVDF membranes for performing the task of recovering water from ionic liquid streams. If put into practice for lunar missions, resupply of ionic liquid and membranes could be reduced. Even so, a significant surplus of membranes will be required. This can only be defined by understanding the effective life spans of the membranes. This study had each membrane operate with approximately 12 hours per membrane. In an industrial level application, these membranes will need to be tested with the ionic liquid on the order of days, if not weeks, of continual operating time.

CHAPTER 5

MATERIAL AND METHODS LUNAR HYDROPONICS

5.1 Material

Current hydroponic systems are designed around the use of a base solid that the plants can attach themselves to, and a nutrient rich stream to supply them with the required nutrients to grow effectively. The atmosphere and the nutrient stream must supply 17 elements for the plants to grow effectively: carbon, hydrogen, potassium, phosphorus, calcium, nitrogen, magnesium, iron, copper, sulfur, manganese, zinc, boron, chlorine, molybdenum, nickel and oxygen [Asao, 2012].

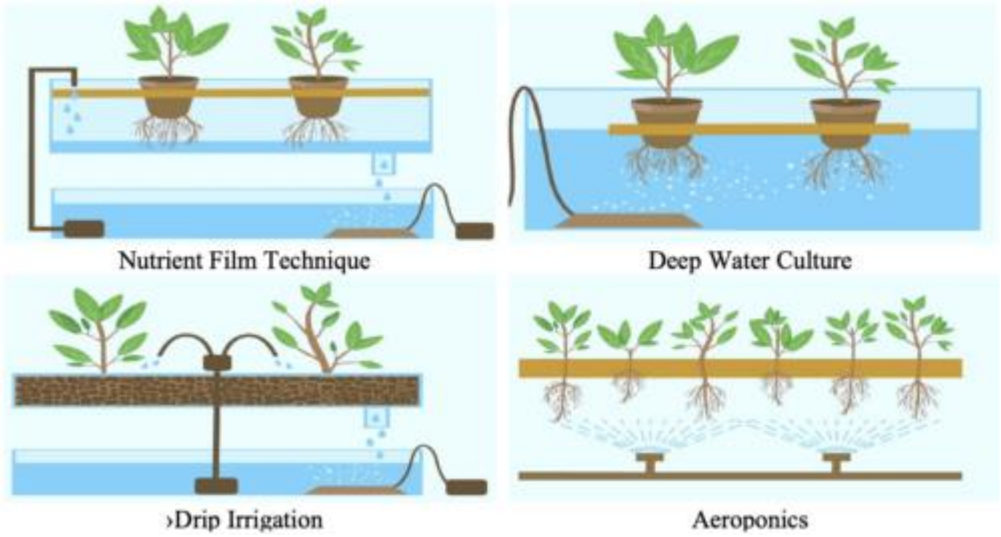

Figure 5-1: An illustration of various methods of hydroponic systems [Geetali, 2022].

While various methods exist for how to do this combination exactly, the fundamental requirements do not change. One of the more challenging for lunar expeditions will be the stabilization of the nutrient supply. While working with limited water supplies, maintaining a proper nutrient supply will prove challenging. As the plants consume the nutrients out of the solution the existing output solution must then be concentrated by a mixture of adding additional nutrients and water recovery. The hypothesis to be tested is that the use of DCMD technology can offer a potential solution to stabilizing the nutrient solution by removing water from the solution.

5.2 Solution Preparation

To test this hypothesis, a mock nutrient solution was made to test three common and readily testable salts that are used in hydroponic systems: nitrogen, phosphorus, and potassium. This solution was created by using ammonium nitrate (Flinn Scientific inc., Chicago, IL, USA, Ammonium Nitrate, Catalog# A0056), sodium dihydrogen phosphate monohydrate (hBARSCI, Rochester, NY, USA, Labatory-Grade Sodium Phosphate, Monobasic, Anhydrous, The Curacted Chemical Collection), and potassium carbonate (hBARSCI, Rochester, NY, USA, Lab-Grade Anhydrous Potassium Carbonate, The Curacted Chemical Collection). A large stock solution was created to target a concentration of 10 ppm. This solution was then referred to as a mother solution and used to create subsequent trial solutions.

5.3 Experimental Method

Samples of the mother solution were taken in 450 ml portions. These solutions were then processed using a bench scale DCMD setup. The solution feed was preheated for 5 mins in a water base set to the temperatures of 50, 65, and 80 °C. Two runs were performed at each temperature for a two hour duration each. During the trials, an initial sample was taken and follow up samples were taken every half hour until the end of the two hour testing phase, with each sample having a target volume of 50 ml. The cool side water was 200 ml initially and it was weighed before and after the experiment. It was chilled in a water base set to 5 °C using a chiller. A final water sample was taken at the end to test for contamination of the product water.

5.4 Testing Methodology

Testing was done using UV-Visible spectroscopy, and chemical reagents used to cause a color shift in the nutrient solution. All three tested components do not show on UV-Visible spectroscopy under normal conditions. However, as they are commonly tested for in commercial and civilian applications, many kits are produced that use various chemical reagents to cause a color phase shift (Hanna instruments, Woonsocket, RI, USA: Phosphate (as orthophosphate, PO43-, Range: (0.00-1.00 mg/L)(0.0-5.0 mg/L)(0-50 mg/L) Method: checker disc, 100 tests; Nitrate Chemical Test Kit, HI 3874-100; Thomas Scientific,Pittsburgh, PA, USA: VISO ECO POTASSIUM-1 kit(~60 test) UN3316, Manufacturer #. 391032) . This results in a complex being formed with the tested compound, this compound is a colored solution that can be tested. While generally these are used with the naked eye and a reference chart to determine the concentration of the compound they can also be tested using a UV-Visible spectrophotometer if the protocols are modified appropriately. This provided two benefits, the first being a method that allows for repeatable measurement to be taken and qualitatively and quantitatively measured. The second being that each reagent would not react with the other compounds allowing for testing to be done to isolate the concentration of each compound.

To modify the testing protocols for each testing kit two actions were taken. The first was to convert all powered reagents in a liquid aqueous solution that would simultaneously account for the needed dilution of the sample, and the testing volumes to be reduced to match that of the testing cuvette. As each kit's protocol requires varying volumes of sample, ranging from 0.78 ml to 17 ml these needed to be adjusted to match a standardized testing method. This was chosen to be 3 ml as it was the standard volume used for the cuvettes. The solid reagents were dissolved in water treated with reverse osmosis and kept for a maximum of 48 hrs before being replaced. A five point calibration curve was created for each of the components using a range of concentrations between 5-25 ppm with 5 ppm intervals.

CHAPTER 6

RESULTS AND DISCUSSION

6.1 Preliminary Data

The initial data from the trials was found by taking the change in the mass of the water of the cold flux stream and comparing it between trials.

Table 6-1: Water recovered from DCMD process at different feed temperatures and membrane polymers.

Feed Temperature (°C)	Water Recovered (g), PVDF	Water Recovered (g), PTFE
50	78.65	38.46
65	105.355	70.115
80	107.755	110.82

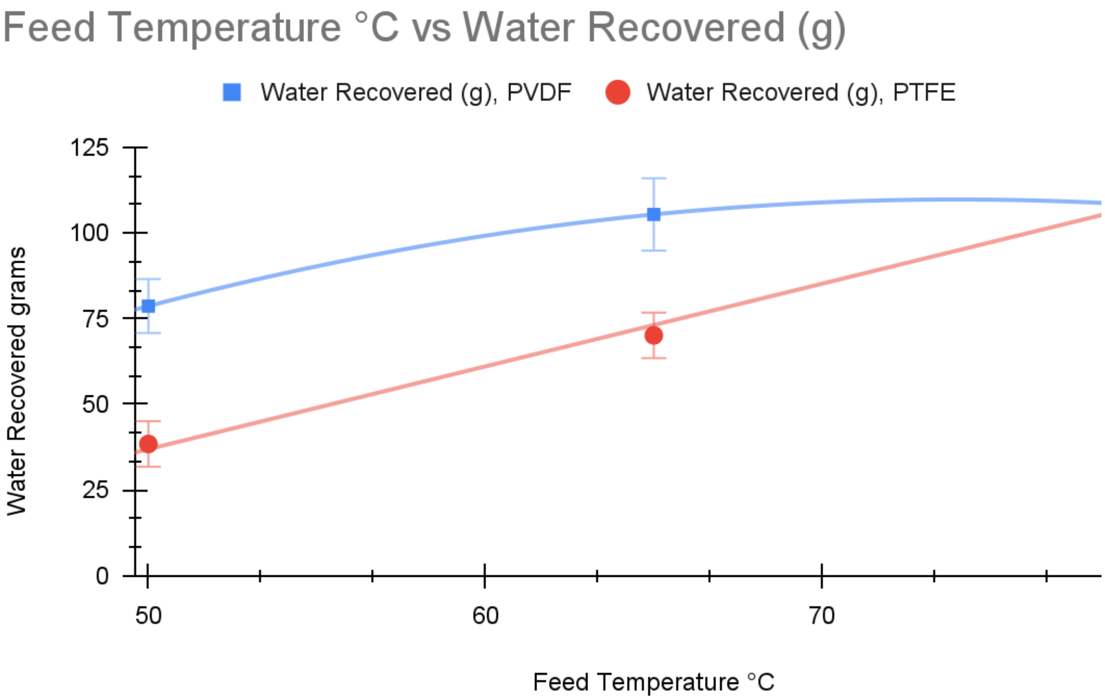

Figure 6-1: A graph of water recovered from DCMD process at different feed temperatures and membrane polymer.

As seen in **Figure 6-1** the PVDF membrane was found to be better at water recovery than the PTFE membrane, however a curve rather than a straight line fits the data best. This compares to the PTFE counterpart that performs in a linear manner with the lower temperatures resulting in a dramatically reduced water recovery of only 48.9% of the water recovered from the PVDF membrane at 50 ℃. While water recovery improves for the PTFE membrane at 65 ℃, it is still only 66.6 % of its PVDF counterpart. However, at 80 ℃, the PTFE membrane surpasses the PVDF membrane in water recovery. The linear nature of the PTFE trend suggests that this trend could continue further and testing at higher temperatures may be warranted.

6.2 Calibration of UV-Vis Spectroscopy

The initial five point calibration curve was done for all three tested components, nitrate, potassium, and phosphate. This initial calibration resulted in the determination that the nitrate testing would not be viable for quantitative measurements as it could not produce an absorbance of less than 2 without such an extreme dilution that it would make the accuracy of the test questionable.

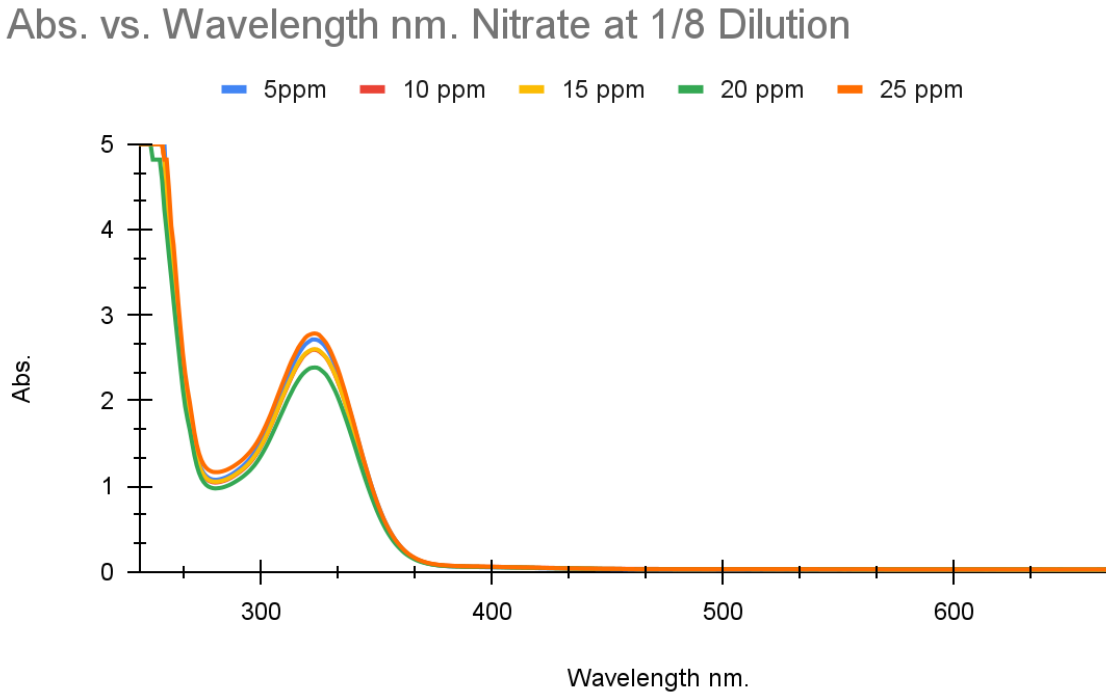

Figure 6-2: Light absorbency spectra of nitrate at ⅛ dilution and various concentrations and wavelengths.

Table 6-2: Change in Nitrate using different temperatures and membranes*

*Nitrate concentrations were below detection limits of the kit, this resulted in large inaccuracies in resulting data

Feed Temp.(°C)	Nitrate Change PTFE	Nitrate Change PVDF
50	2.10%	-2.04%
65	-13.07%	-160.44%
80	-62.49%	18.08%

Figure 6-2 shows that throughout the tested spectrum of 200 nm to 800 nm there is no area of the spectrum where the reagent reacts and produces an absorption of less than 2 reliably. This was noticed originally at the testing protocol of 50% dilution. As a result, further dilutions were done until it was at ⅛ of the sample strength. This still produced results that would overload the reliability of the sensor. As a result, the valley at a wavelength of 280 nm was chosen, however it was still unreliable data and was not used for more than qualitative analysis.

The calibrations for potassium testing resulted in a cloudy solution that had very minor peaks but instead had an overall absorption of light in a broad spectrum. A dilution of ½ was built into the test protocol to account for the need to keep the absorption below 2. As a result, a second calibration was done ranging from 2.5-12.5 ppm in increments of 2.5 ppm.

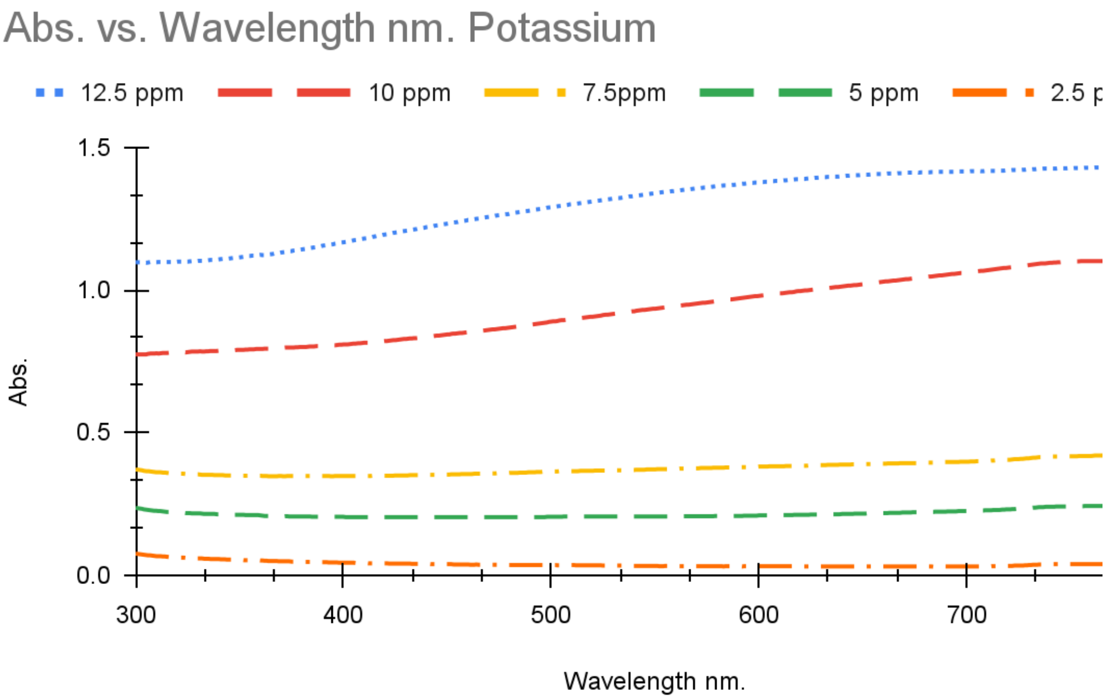

Figure 6-3: Absorption spectra of five point potassium concentration calibration curve.

As **Figure 6-3** shows, the potassium testing solution resulted not in a major peak but instead a general broad spectrum absorption of light. There was a small peak noticed at a wavelength of 754 nm. This was used as the baseline testing measurement for the subsequent trial samples.

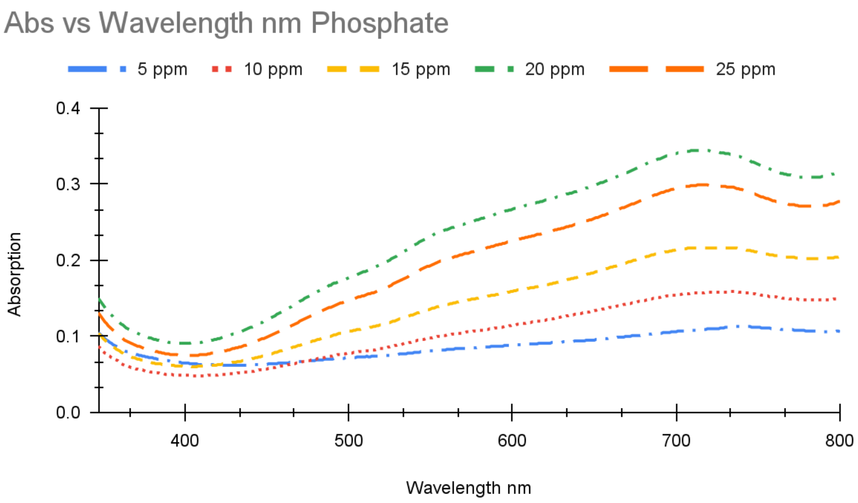

Figure 6-4: Absorption spectra of five point phosphate concentration calibration curve.

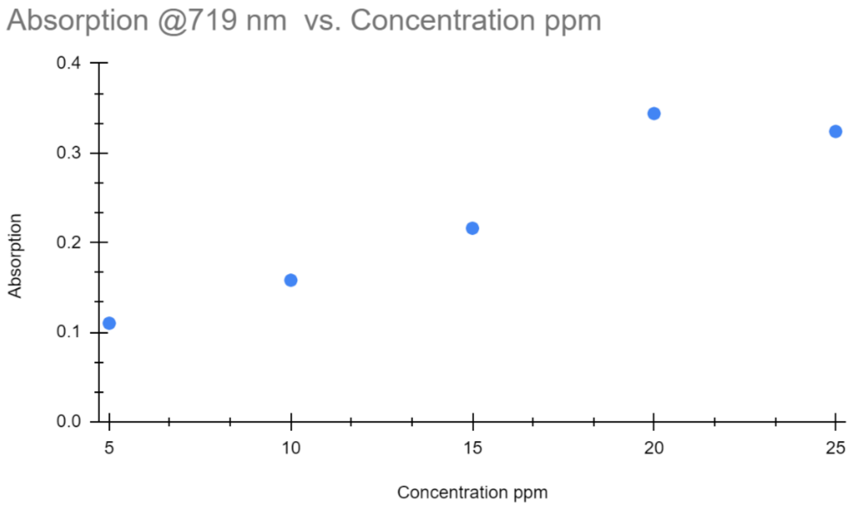

Figure 6-5: Absorption of phosphate at 719 nm of a five point concentration calibration curve.

As can be seen from **Figure 6-4** a distinct peak can be seen at the wavelength of 719 nm; this was therefore chosen as the wavelength that would be used to test the trial samples. An issue was noted in that at higher than 20 ppm the test solution would not react properly with the sample, resulting in an apparent decrease in absorbance. This issue was accounted for when testing the solutions by doing an estimated maximum possible change in concentration calculation by using the change in water volume found previously.

6.3 Experimental Samples

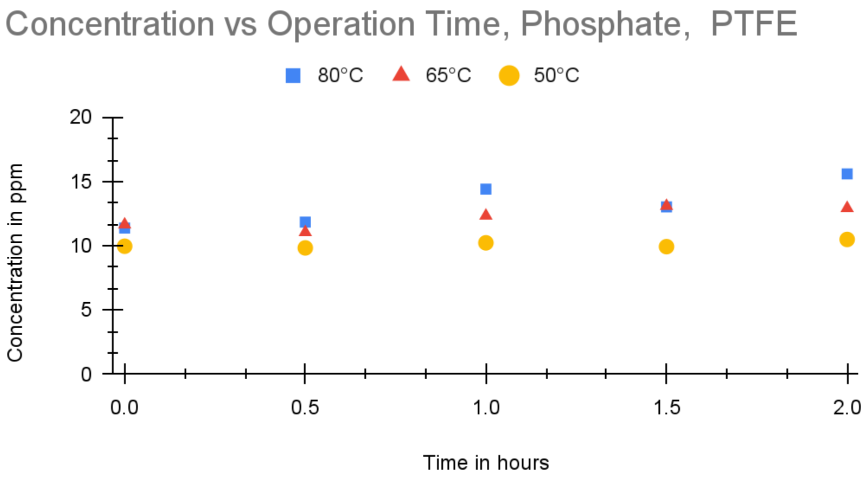

Figure 6-6: Effect of DCMD operational time on concentration of phosphate using a PTFE membrane.

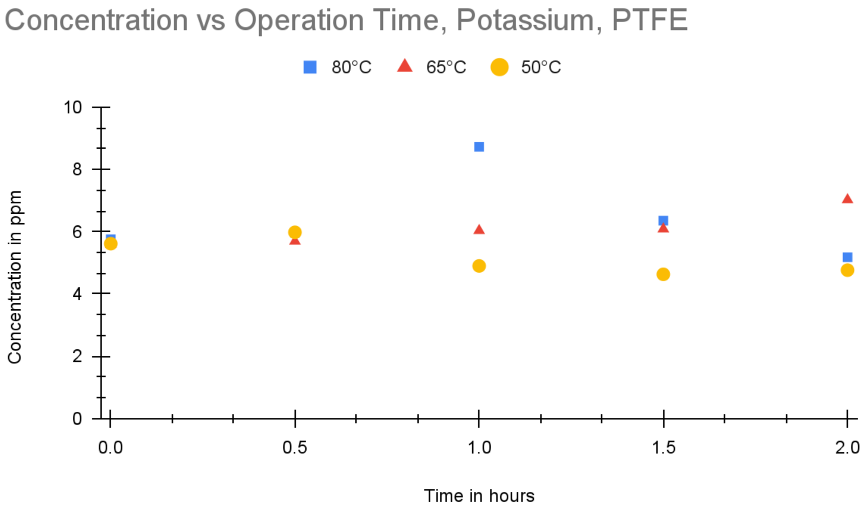

Figure 6-7: Effect of DCMD operational time on concentration of potassium using a PTFE membrane.

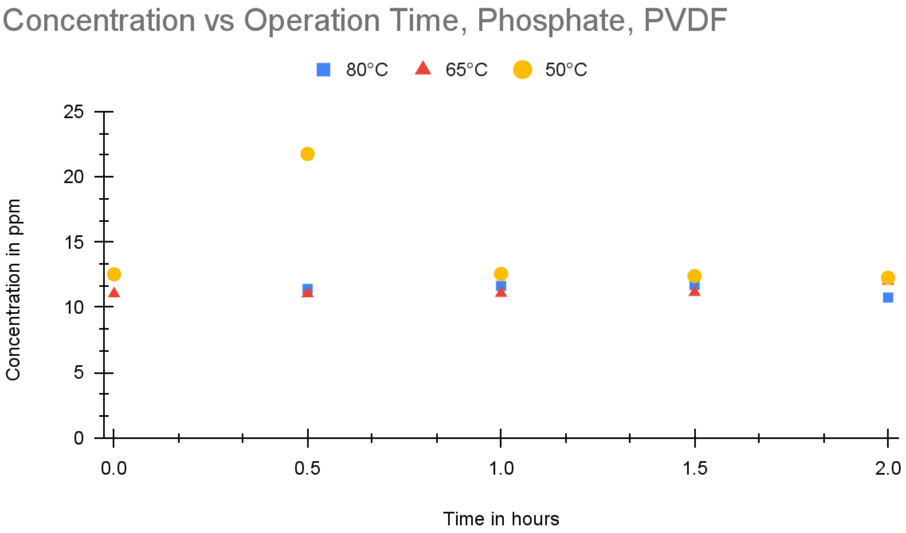

Figure 6-8: Effect of DCMD operational time on concentration of phosphate using a PVDF membrane

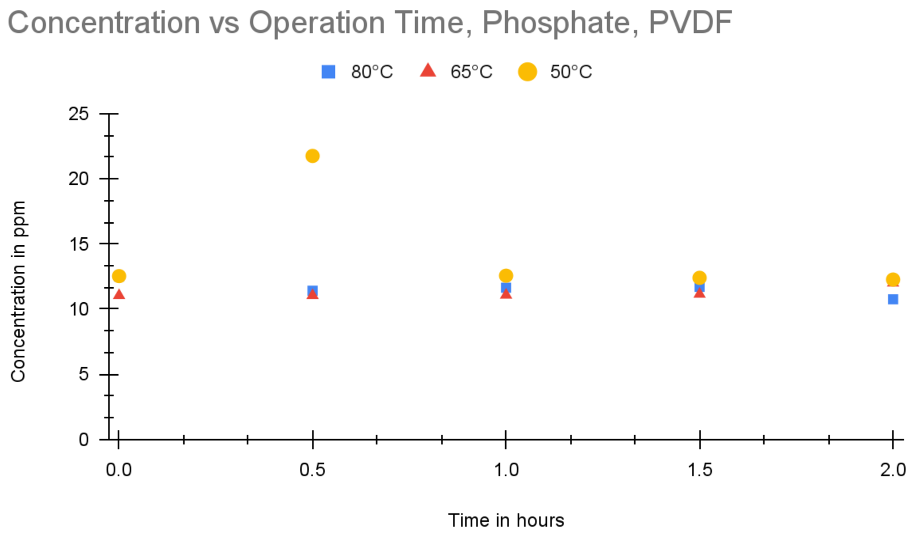

Figure 6-9: Effect of DCMD operational time on concentration of potassium using a PVDF membrane

Table 6-3: Change in concentration of final nutrient ions at different feed temperatures using a PTFE membrane

Feed Temp. (°C) PTFE	Phosphate Change	Potassium Change
50	5.28%	-15.11%
65	11.02%	25.45%
80	36.99%	-10.01%

Table 6-4: Change in concentration of final nutrient ions at different feed temperatures using a PVDF membrane

Feed Temp. (°C) PVDF	Phosphate Change	Potassium Change
50	-2.10%	4.95%
65	8.63%	28.96%
80	-13.95%	-1.28%

Table 6-3and **Table 6-4** demonstrate that there is mass transfer being done across the membrane and that it is successful to a degree at concentrating the nutrient solution. However, it can also be seen that the overall concentration of ions in some instances is dropping while water is still being transferred across the membrane. These tables miss the nuance that the corresponding **Figure 6-6**, **Figure 6-8**, and **Figure 6-9** show. From the figures it can be seen that there seems to be a clear pattern of concentration. The concentration will increase rapidly and then decrease until increasing again. This is strikingly similar to the effects of a process control integral regulator attempting to regulate.

6.4 Cross Membrane Ion Transfer

This could be explained by a small amount of diffusion across the membrane of the ions that occurs once the concentration gradient becomes great enough. As the feed solution loses water to the distillate the concentration of the feed increases until the rate of diffusion of the ions out paces the rate of distillation. To test if this was a viable possibility, tests were run on some of the water samples taken from the end of trial runs. It was found that there were detectable concentrations of phosphate across the membrane. The concentrations found ranged from 1.91 ppm to 4.67 ppm. This demonstrates that for these membranes the ions were able to pass through the membranes in a sizable quantity. This is also supported by data from the previous project where it was found that there was contamination of the distillate from the ionic liquid being tested.

In this application however this could prove advantageous. As the oscillating nature of the concentration implies that there is a balance point between temperature, water flux, and concentration that could be achieved. This could be used as a method to accomplish the original objective of regulating the concentration of the aqueous nutrient solution.

CHAPTER 7

CONCLUSIONS AND FUTURE WORK

7.1 Conclusions

Both the PTFE and PVDF membranes have shown the potential to recover water from nutrient streams and concentrate them. This comes with the issue of contamination of the distillate water. With the nutrient salts this appeared to be in an oscillating pattern that must be taken into account.

Due to the issue of contamination of the distillate two factors must be considered separately based upon the desired usage of the water supply. For a priority of water recovery the PVDF membrane has shown better results in achieving greater water recovery rates compared to its PTFE counterpart. This assumes, however, that operations occur below 80 °C. Above this temperature the PTFE membrane will produce better results. However if the goal is to stabilize and concentrate the nutrient stream then the PTFE membrane operated at a middle temperature between 50°C and 80°C would be advantageous. This combination appears to offer the best results for concentration of the nutrient stream as well as recovering water. While this method results in approximately a 33.45% loss in water recovery, it does result in an additional 20% increase in concentration when performed at 65 °C.

7.2 Future Work

More work is required to test the effect of membrane pore size on the rate of ion contamination of the distillate. This will prove to be an issue that needs to be better understood in order to properly employ DCMD technology on a large scale. This information could also prove useful to the industries that are trying to make large scale desalination plants with a similar process. Desalination has been practiced at small scale using DCMD, and could prove to be beneficial to any coastal area with the looming threat of climate change and unsteady fresh water supplies.

Additionally further work should be conducted on the effects of initial concentration of the solution at the start of the DCMD process. The contamination effect seen may have to do with the concentration gradient changing as the DCMD process is performed. This could be tested and verified in better detail with a dedicated study. This information could also prove useful to rural communities and their waste water treatments systems, since many of the same compounds commonly used for hydroponic systems are used to fertilize farmland. Such studies could provide an option to help rural communities cheaply process water that has been plagued by excess fertilizers, such as Minnesota [Anderson, 2016].

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