Use of Microbubbles to Mitigate Scaling in Membrane Distillation

by

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ABSTRACT

Membrane fouling, especially inorganic fouling, is a significant obstacle to treating highly saline brine using membrane distillation (MD). In this study, microbubbles (MBs) were injected into the feed tank of a lab-scale direct contact membrane distillation (DCMD) system, and its effect on permeate flux over time was examined. A synthetic inland reverse osmosis (RO) brine with a high scaling tendency was used as a feed solution. Results showed a sharper flux decline in the absence of MBs compared to when MBs are continuously injected into the feed tank. The introduction of MBs reduced the formation of salt precipitations on the membrane surface, which was the primary cause of the decline in flux. The use of intermittent MBs injection instead of continuous MB injection was evaluated as a way to reduce energy consumption; with a 15 min MBs injection every 2h, similar benefits were found for intermittent injection compared to continuous injection, indicating that providing MBs continuously is not needed to mitigate scale formation. These results show that MBs can be a potential chemical-free method to prevent scaling in desalination systems treating high saline solutions.

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CHAPTER 1

INTRODUCTION

1.1 Water scarcity

The shortage of clean water is a problem that emerged in recent decades due to improvements in living standards and the rapid growth in the world's population (Boretti and Rosa 2019). Physical water scarcity occurs when there is insufficient water to meet all needs, affecting more than 1.2 billion people. Economic water scarcity is induced by a shortage of human capacity or a shortage of investment in water, affecting more than 1.6 billion people (Molden 2013). High salinity water represents about 97 percent of the total water on the planet. It is available to almost all countries, making desalination an option for securing water supply for water-stressed nations (Elsaid et al. 2020). Seawater Desalination is an energy-driven process that aims to convert seawater to freshwater, producing concentrated brine as a byproduct (Figure 2). Desalination is used in more than 175 countries around the globe, producing around 100 million cubic meters per day (MCM/d). Fifty percent of the production is in the middle east and north Africa (MENA) (Elsaid et al. 2020; Jones et al. 2019).

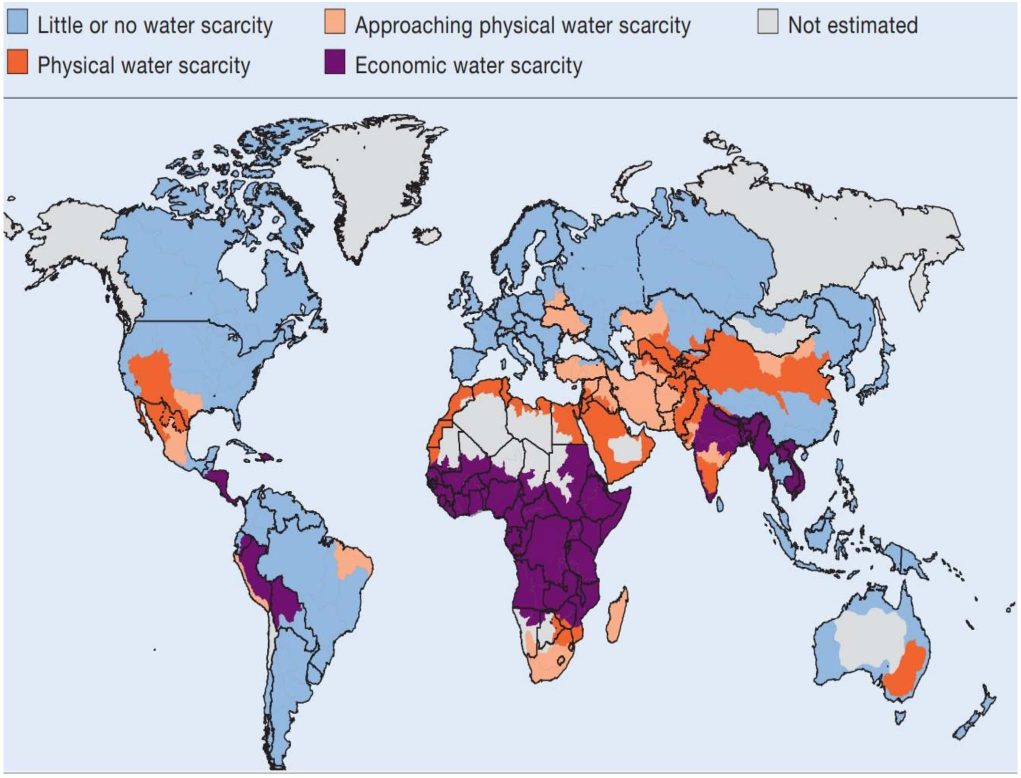

Figure 1: Physical and economical water scarcity in the world (Molden 2013).

1.2 Desalination technologies

The two main categories of desalination technologies are thermal and membrane technologies (Elsaid et al. 2020). Thermal desalination was the leading technology used for seawater desalination from the 1950s to 1970. Thermal technologies were widely operated in the middle east, especially in the Gulf Cooperation Council (GCC) countries. Thermal technologies simulate the natural water cycle, where it uses thermal energy to evaporate seawater and then condense it to produce freshwater (Petersen 2017). These processes are usually preferred when the salinity and temperature of the feed are high, have a low energy cost, and when it can use waste energy from a nearby energy plant. The two most used thermal technologies are multi-effect distillation (MED) and multi-stage flash distillation (MSF) (Panagopoulos and Haralambous 2020). In MED, vapor from each stage is condensed in the next stage, giving up its heat to drive more evaporation. MED is better in terms of thermal performance than MSF, but suffers from high scaling propensity (Mezher et al. 2011). It was replaced with MSF. MSF coupled evaporation and condensation steps to recover the latent heat of evaporation for reuse to heat the incoming water.

However, thermal processes are energy-intensive compared to membrane technologies (Ali et al. 2018). Membrane-based desalination uses a semipermeable membrane that selectively passes pure water while retaining salts on the feed side of the membrane. The most used membrane technologies are reverse osmosis (RO), Nanofiltration (NF), and Electrodialysis (ED), respectively (Jones et al. 2019). In the case of RO, which is the most used process in seawater desalination, a hydraulic pressure higher than the osmotic pressure of seawater is added so pure water only can pass through the membrane. The state-of-the-art in RO desalination is able to achieve, using thin-film composite polyamide membranes, >99.5% salt rejection and from 50% for seawater desalination to close to 90% water recovery for brackish water desalination (Werber, Osuji, and Elimelech 2016). However, this also means that between 10 to 50% of the treated water volume ends up as high salinity waste brine that needs to be disposed of. Brine from seawater desalination is less of a problem than inland desalination since it can be disposed of back to the ocean. Brine from inland desalination is usually disposed of in wells or evaporated using evaporation ponds (Ahmed et al. 2000). Methods to dispose of brine from inland desalination are unattractive due to their high capital cost (Qiu and Davies 2012). This is why it is essential to develop better technologies to manage the brines.

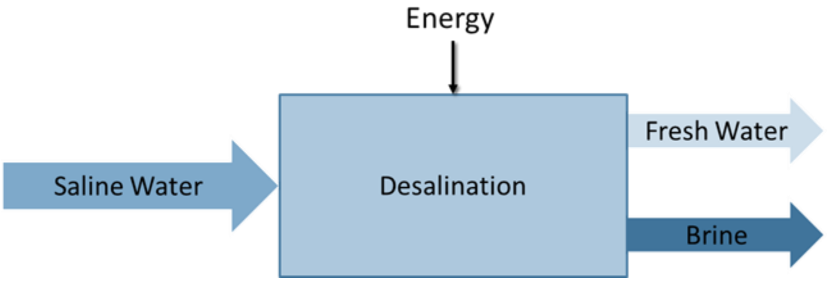

Figure 2: Energy is needed to produce fresh water out of saline water. Brine a concentrate of the feed solution is a byproduct of that process.

1.3 Brine treatment

Brine is generally disposed of using various methods such as deep-well injection sewer discharge, surface water discharge, and evaporation ponds (Panagopoulos, Haralambous, and Loizidou 2019). Brine's high total dissolved solids content may contain, in addition to salts, dangerous pretreatment chemicals (antiscalants, flocculants, and coagulants) and microbial contaminants. Studies have shown negative environmental impacts of brine on groundwater and soil quality, and the marine environment (Sadhwani, Veza, and Santana 2005).

Brine treatment systems can be an environmental alternative to disposal methods. Brine treatment processes aim is to recover more freshwater and avoid brine disposal to the environment. Due to osmotic constraints, commercial desalination technologies like MSF, MED, and RO are not appropriate for brine treatment. MSF/MED are highly energy-intensive and must be constructed from highly corrosion-resistant materials like titanium. RO can treat feedwater up to a specific salinity (70,000 mg/L) (Davenport et al. 2018). Several arising technologies, including forward osmosis and membrane distillation

(MD)/Membrane crystallization, show great potential to treat high salinity brine(Quist-Jensen et al. 2016).

Vapor pressure-driven membrane separation processes such as MD are being advanced as a strategy to tackle the high salinity brines produced by RO desalination (Sweity et al. 2013; Adham et al. 2013). These processes use the difference in vapor pressure as the driving force to move water vapor through a hydrophobic porous membrane (Alkhudhiri, Darwish, and Hilal 2012). The main advantage of using vapor pressure-driven separation compared to hydraulic pressure as in RO is that vapor pressure is not significantly affected by salinity, which allows MD to achieve very high-water recovery and treat highly saline waters (Ricceri et al. 2019). In addition, MD can operate at low operating temperature as it is unnecessary to heat feed water to the boiling point to achieve separation. It can also be integrated with low-grade or renewable sources of energy, such as solar energy, geothermal energy, or natural temperature gradients, to reduce the energy cost for desalination (Alrehaili et al., 2020; Rice et al., 2020; Sarbatly & Chiam, 2013).

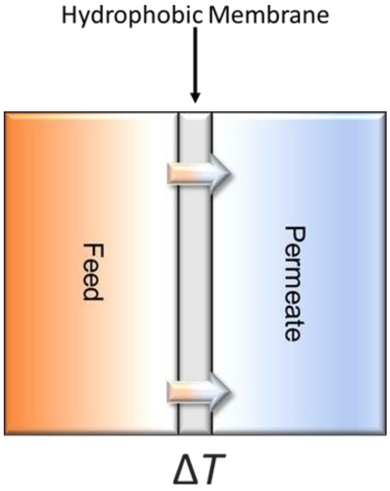

Figure 3: MD uses the difference in temperature between the feed side(hot) and the permeate side(cold) as a driving force to drive water vapor through a hydrophobic membrane

1.4 Scaling

As water recovery is pushed further and further in MD desalination, membrane fouling by inorganic scaling becomes inevitable. Inorganic scaling is caused by the precipitation of insoluble salts such as calcium sulfate ($CaSO_4$), calcium carbonate ($CaCO_3$), and silica on the membrane surface (Wang Y. 2005). The layer of inorganic scaling effect membrane hydrophobicity and slows the transport of water vapor across the membrane (Chen et al. 2021). Therefore, negatively impacting the permeate flux and permeate quality (Warsinger et al. 2015). Scale formation rate is dependent on several factors, including feed temperature, flow conditions, water composition, and the degree of supersaturation (Nghiem and Cath 2011).

Two nucleation events can cause scaling after the solution becomes supersaturated. The two nucleation events depend on solution temperature and solution saturation. The formation of salt crystals in bulk is called homogeneous nucleation, while the formation of salt crystals on the membrane surface is called heterogeneous nucleation (Warsinger et al. 2015). Homogeneous nucleation is less common in desalination processes than heterogeneous nucleation because it takes more time to occur (Warsinger et al. 2015). Another reason is that homogeneous nucleation usually happens at higher concentrations compared to heterogeneous nucleation (Warsinger et al. 2015).

1.5 Scaling mitigation

To mitigate membrane scaling, antiscalants can be added to the feed water to hinder inorganic scaling formation if the pretreatments are insufficient to eliminate scaling (Alkhatib, Ayari, and Hawari 2021). It is essential to note that antiscalant addition during RO pretreatment can negatively affect a later RO brine treatment that uses precipitation to separate potential scale-forming minerals. Coagulation or oxidation is needed to remove antiscalant prior to perception of RO concentrate (Greenlee et al. 2010). It has been reported that antiscalants can induce biofouling formation since they can be nutritious to bacteria. Biofouling can affect water flux and salt precipitation through the membrane surface (Sweity et al. 2013; Greenlee et al. 2010).

Because of these limitations of antiscalants, chemical-free solutions to scaling control are gaining interest, particularly in off-grid settings where chemical management can be challenging. Air microbubbles (MBs) are tiny bubbles with a diameter of 10–100 µm with various unique properties that distinguish them from ordinary air bubbles, such as a longer lifetime to dissipation, negatively charged surface, and high specific area (Sakr et al. 2022). Macrobubbles instantly burst on the surface, while microbubbles can last several minutes before disappearing underwater. According to (Takahashi et al., 2007), when microbubbles(10-60 µm) were injected into a water tank, it gave the solution a milky appearance. It took around 5 min after the generator was switched off for the solution to be fully transparent. MBs are used in diverse applications, including water treatment, biomedical engineering, and agriculture (Agarwal, Ng, and Liu 2011). Using MBs is cheaper and more environmentally friendly than chemicals such as oxidants and disinfectants (Tekile et al., 2017). MBs are used to separate low-density particles in the dissolved air flotation process and oxidize the organic matter present in wastewater (Tekile et al., 2017). Removal of surface-active compounds by air flotation has been used to control pore wetting in MD desalination (Rajwade et al. 2020). During the past few decades, membrane cleaning using air bubbles has been a rapidly growing technology. The addition of air bubbles can reduce the concentration polarization (CP) of feedwater (Dayarathne, Choi, and Jang 2017). According to (Goosen et al. 2004), it has been proven that adding air bubbles with cleaning chemicals can help wash RO membranes by agitating the fouling layer.

The various types of microbubbles are pressurized-dissolution, electro flotation, spiral liquid flow, venturi, and ejector methods (Kim et al., 2018). One of the generating techniques is an ejector-type microbubble generator. In an ejector-type microbubble generator, the gas needed to form microbubbles is automatically sucked by the law of energy conversion. So, a compressor to provide air is not necessary (Arumugam, 2015). Figure 4 shows the interior design of an ejector-type micro air bubble device. The generation of air microbubbles generator requires a high-pressure water flow to create a cavitation pocket. A controlled gas flow is then injected into the attached vapor cavity. The gas then splits up into a cloud of very finely dispersed microbubbles (Nakatake et al., 2013).

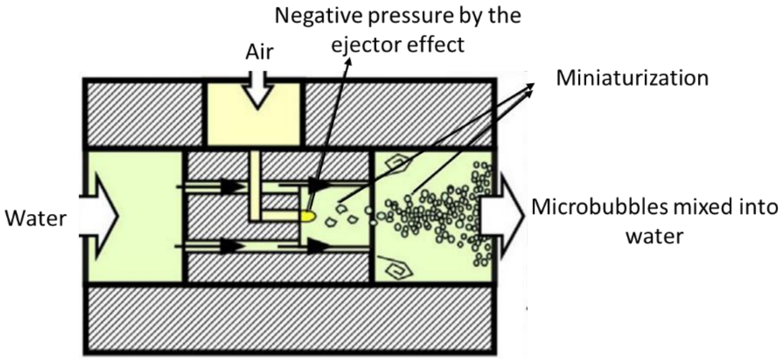

Figure 4: The interior design of an ejector-type micro air-bubbles device (Nakatake et al., 2013).

1.6 Knowledge gap and research needs:

Membrane fouling, especially inorganic fouling, is a significant obstacle to treating highly saline brine using MD. An environmentally friendly way to mitigate inorganic fouling in desalination systems is by injecting MBs to help agitate the membrane surface. Previous studies (Table 1) tested this using an RO system using single salts solutions containing either $CaCO_3$ or $CaSO_4$. Another study used a vacuum membrane distillation (VMD) system (6 hours operation time) using a simple synthetic seawater brine. However, MBs were not tested in MD systems treating more complex brines, such as those originating from inland brackish water desalination. These brines, although lower in salinity, contains a wide variety of inorganic species with high scaling potential that can alter the efficiency of MBs added to the feed water. This work aims to study MBs effect on permeate flux over time in a flat sheet direct contact membrane distillation (DCMD) system using a complex groundwater brine.

Table 1: Previous studies that used MBs. For the RO system, feed flow ranged between (0.3-0.6 L/min) and permeate flow (0.025 – 0.15 L/min), while for the VMD system, feed velocity ranged between (0-23-0.69 m/s) and permeate pressure (100-1000 Pa).

Ref	System	Membrane area(cm^2)	MBs pump pressure (Mpa)	Gas intake (mL/min)	bubbles diameter (um)	Main findings
(Dayarathne et al., 2019)	RO	4	-	0.2	0.09-0.9	• MBs increase membrane permeability. • Continuous application of MBs is more effective than MBs pulsation addition.
(Ye et al., 2019)	VMD	1.2	0.1-0.4	40	15-70	• MBs effectively alleviate membrane scaling and improves permeate flux

1.7 Research Hypotheses and Objectives:

The main hypotheses of this project were:

1-MBs improve the performance of MD in terms of higher permeate flux and lower flux decline over time.

2-Continuous injecting of MBs can improve the performance of MD in terms of higher permeate flux and lower flux decline more than intermittent MBs injection.

3-MBs reduce scale formation by agitating membrane surface.

The following research objectives were pursued to verify the above hypotheses:

1-Compare the performance of the system in the absence of MBs and when MBs were injected Continuously.

2-Compare the performance of the system with continuous MBs injected and when MBs were injected intermittently.

3-Study membrane scaling on the membrane surface using a scanning electron microscope (SEM).

CHAPTER 2

MATERIALS AND METHODS

2.1 Feed water:

In this study, calcium chloride dihydrate ($CaCl_2.2H_2O$), magnesium chloride hexahydrate ($MgCl_2.6H_2O$), sodium chloride (NaCl), potassium bicarbonate ($KHCO_3$), sodium bicarbonate ($NaHCO_3$), sodium sulfate (Na_2SO_4), boric acid (H_3BO_3), sodium metasilicate pentahydrate ($Na_2SiO_3.5H_2O$), strontium chloride hexahydrate ($SrCl_2.6H_2O$), barium chloride ($BaCl_2$), copper sulfate pentahydrate ($CuSO_4.5H_2O$), ferrous sulfate heptahydrate ($FeSO_4.7H_2O$), zinc sulfate heptahydrate ($ZnSO_4.7H_2O$), sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$), nickel(II) nitrate hexahydrate($NiNO_3.6H_2O$) potassium dichromate ($K_2Cr_2O_7$). We added 3.4 mL/L of polyacrylate antiscalant (Vitec 5100). The RO brine recipe is a replicate of an RO plant brine in Shalateen, Egypt. The Shalateen recipe is calculated based on elemental analysis of the feed, permeate, and brine in Table 2.

Table 2: Feed, permeate and brine elements concentrations from an RO plant in Shalateen, Egypt.

Species	**Feed(mg/L)**	**Permeate(mg/L)**	**Reject(mg/L)**
Ca	993.6	5.0	2531
Mg	421.3	1.5	468.9
Na	5600	184	6200
K	30.0	2.0	38
HCO_3	79.3	18.3	91.5
SO_4	1383.9	38.4	1851.3
Cl	10745.4	266.2	14097.2
Al	0.177	<0.01	<0.01
B	1.616	0.940	1.754
Ba	0.043	0.002	0.045
Cd	0.001	<0.0008	<0.0008
Co	<0.001	<0.001	<0.001
Cr	0.026	<0.01	0.027
Cu	0.033	<0.009	0.046
Fe	0.597	<0.02	0.107
Mn	0.007	0.006	0.011
Mo	0.011	<0.003	0.010
Ni	0.010	<0.002	0.007
Pb	0.011	<0.006	<0.006
Si	13.8	<0.02	19.2
Sr	50	0.1	62.2
V	<0.01	<0.01	<0.01
Zn	0.031	0.004	0.006

The chemicals in Table 3 were mixed with deionized (DI) water and antiscalant to prepare the RO brine feed solution. pH for the solution was adjusted to 7.2. All chemicals were bought from Sigma–Aldrich Corporation. The change in water chemistry was investigated using Visual MINTEQ software.

Table 3: Synthetic Inland RO brine recipe that replicates an inland RO plant reject in Shalateen, Egypt.

Chemical compound	**Concentration (mg/L)**	**Chemical compound**	**Concentration (mg/L)**
$CaCl_2.2H_2O$	9302.38	$SrCl_2.6H_2O$	189.24
$MgCl_2.6H2O$	3923	$BaCl_2$	0.07
NaCl	13477.54	$CuSO_4.5H_2O$	0.17
$KHCO_3$	97.30	$FeSO_4.7H_2O$	0.53
$NaHCO_3$	44.33	$ZnSO_4.7H_2O$	0.03
Na_2SO_4	2737.44	$Na_2MoO_4.2H_2O$	0.03
H_3BO_3	10.03	$NiNO_3.6H_2O$	0.03
$Na_2SiO_3.5H_2O$	145.24	$K_2Cr_2O_7$	0.16

2.2 Membrane:

A hydrophobic PTFE membrane (Polytetrafluoroethylene) bought from (Ji'an Qingfeng Filter Equipment Material Co. Ltd, China) was cut into small pieces (7.62 cm × 3.175 cm) to fit into the membrane cell used. The PTFE membrane has a pore size of 0.2 µm and a thickness of 130 mm ± 15.

2.3 MBs generator:

CARMIN D2 single MBs generator was bought from (Ylec Consultants Fluid Mechanics, France) (item number 002 18 02 01 02). MBs were injected into the feed side of a DCMD system. A high-pressure pump (Hooshing 12V 60W Micro Electric Diaphragm Water Pump 5 L/min 116 PSI) was used to pump feed water to MB's generator to generate bubbles in the feed tank (Figure 5). A pressure meter was used to assure the system was operating at a specific pressure.

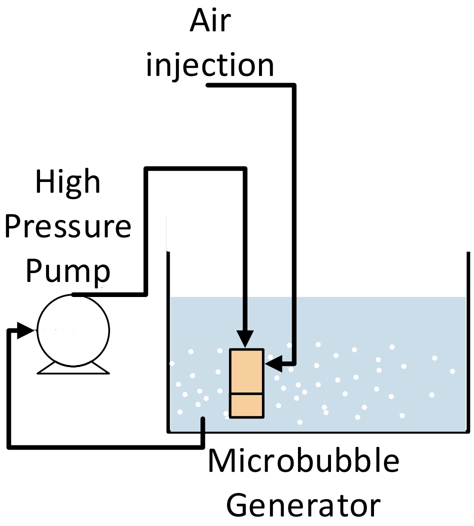

Figure 5: High-pressure pump was used to circulate feed solution from the feed tank to the MBs generator back to the feed tank. Pressure=3.6 bar and flowrate=2.1 L/min.

2.4 Experimental procedure:

Bench-scale experiments were done using custom DCMD systems (Figure 6) in a membrane cell having active membrane dimensions of (7.62 cm × 3.175 cm). In DCMD experiments, feed water was supplied at a rate of 0.8 L/ min, and a temperature of 64 °C, while the distillate side was supplied at a rate of 0.6 L/ min and a temperature of 22 °C (ΔT= 42 °C). DI water was used for the distillate side, while a synthetic RO brine solution was used as feedwater. The feed and distillate temperatures were kept constant by recirculating the water through glass coils placed in a hot or cold-water bath, respectively.

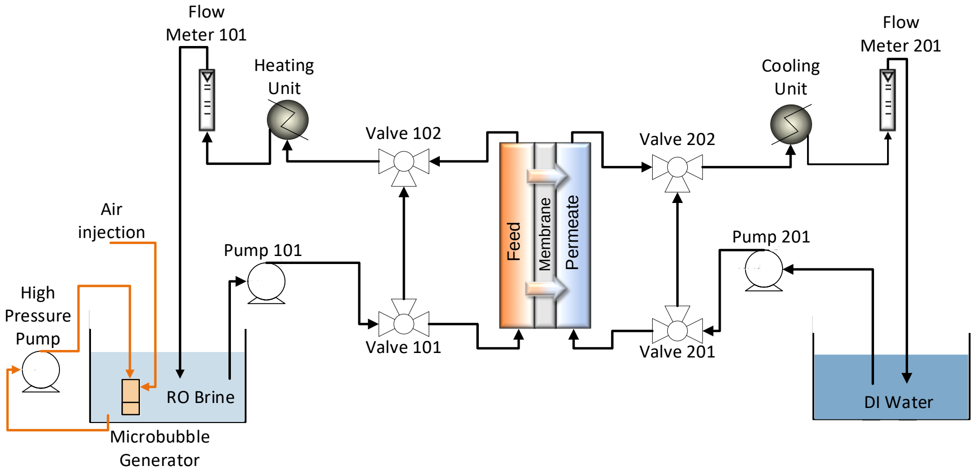

Figure 6:101 and 102 units represent the feed side, while units 201 and 202 represent the permeate side. 101 and 102 valves were used to control feed flow direction, while 201 and 202 were used to control the permeate flow direction. 101 and 201 are gear pumps used to transport feed and permeate solution respectively. Orange lines represent the MBs cycle.

The DCMD system was operated in three conditions:

1-without MBs (-MBs): The system operated under normal conditions without injecting MBs.

2-Continuous MBs (+MBs): Bubbles were injected for the whole duration of the experiment.

3-intermittent MBs (+15MBs): every two hours, bubbles were injected for 15 mins, which was done for the first 15 hours of the experiment.

2.5 Membrane characterization:

After the experiments, fouled membranes samples were dried, cut into small pieces, and then coated with gold in a vacuum. An SEM (SEM/FIB Focused Ion Beam - Nova 200 NanoLab) was used to acquire micrographs of the top surface of the fouled membrane.

CHAPTER 3

RESULTS

3.1 Precipitation Modeling:

Salt precipitation in the synthetic feed solution was calculated using Visual Minteq equilibrium modeling at different concentration factors to understand the type of inorganic species that could precipitate and accumulate on the membrane during MD operation. The software predicated salts precipitation of salts at feed concentrations 1-3 times the initial composition. Based on these results, salt precipitation was expected in the DCMD system. The main precipitating species is gypsum ($CaSO_4 \cdot 2H_2O$), with a modest amount of calcite ($CaCO_3$) and chrysotile ($Mg_3Si_2O_5(OH)_4$) present at higher salt concentration factors.

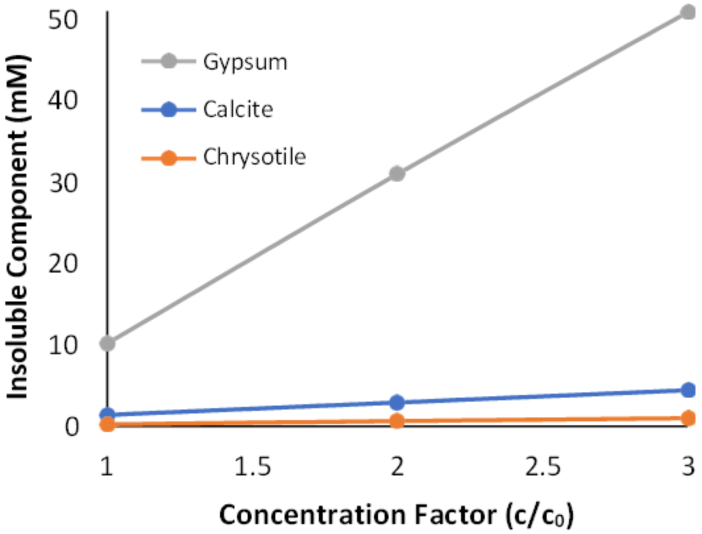

Figure 7: Equilibrium modeling of precipitated salts for synthetic RO brine using Visual MINTEQ software.

3.2. Normalized flux:

Bench-scale DCMD experiments treating RO brine water were performed using a ΔT of 42 °C between the feed (62 °C) and the distillate (22 °C). Synthetic RO brine was used as feed solution because of its high scaling tendency compared to low salt concentrations solutions. There was no notable change in permeate conductivity or ΔT when bubbles were injected compared to without bubbles. Conductivity and ΔT for (+MBs) were (1.6-3.56 µm/cm) and (42-43°C), while for (-MBs) (1.56-3.8 µm/cm) and (42-43.4°C). As shown in (Figure 8), (-MBs) flux was sharper than (+MBs), and (+15MBs). Flux decline in the DCMD system is usually attributed to membrane fouling concentration polarization and membrane pore wetting. With the introduction of MBs, the decline in flux was alleviated, particularly at higher concentrations factors, and the performance was almost identical at the beginning of the experiment. It seemed that the introduction of MBs reduced the formation of salt precipitations on the membrane surface,

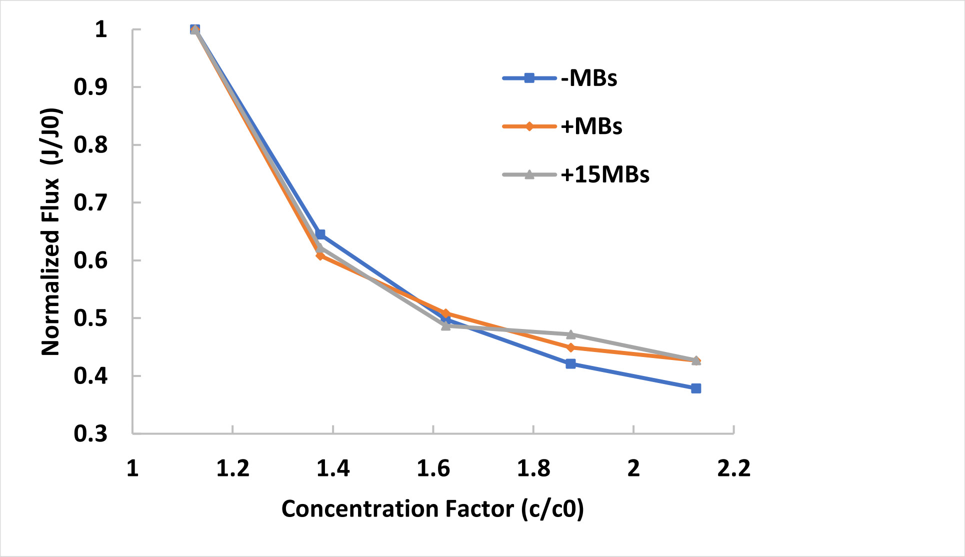

Figure 8: Comparison of normalized flux behaviors as concentration factor increases for three operation conditions -MBs, +MBs, and +15MBs.

which was the major reason for the decline in flux (-MBs). SEM images of the fouled membranes (Figure 9B) (Figure 9C) showed that a layer of scaling covered the membrane surface.

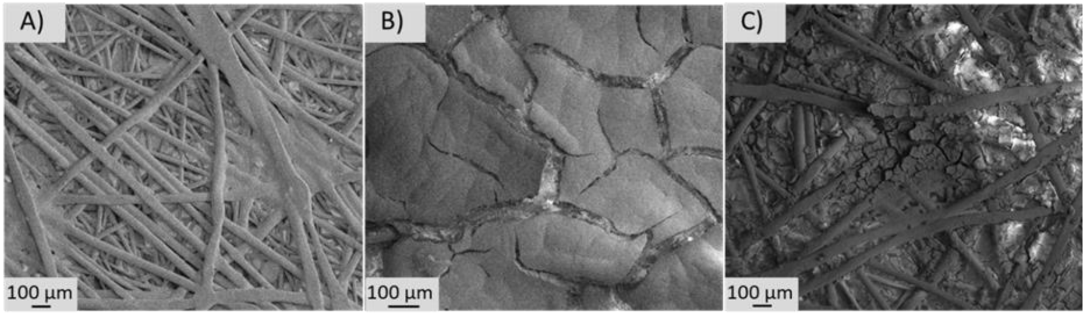

Figure 9:SEM micrographs (top) of (A) pristine PTFE (B) fouled PTFE after 24h of Operating -MB (C) fouled PTFE after 24h of Operating +MB.

3.3 Permeate flux:

As shown in (Figure 10), the continuous injection of MBs affected flux permeate negatively, particularly at the beginning of the experiment. We discovered that continuously operating with bubbles created dead zones (air gaps) on the membrane surface affecting the process performance. When bubbles were injected for a short period

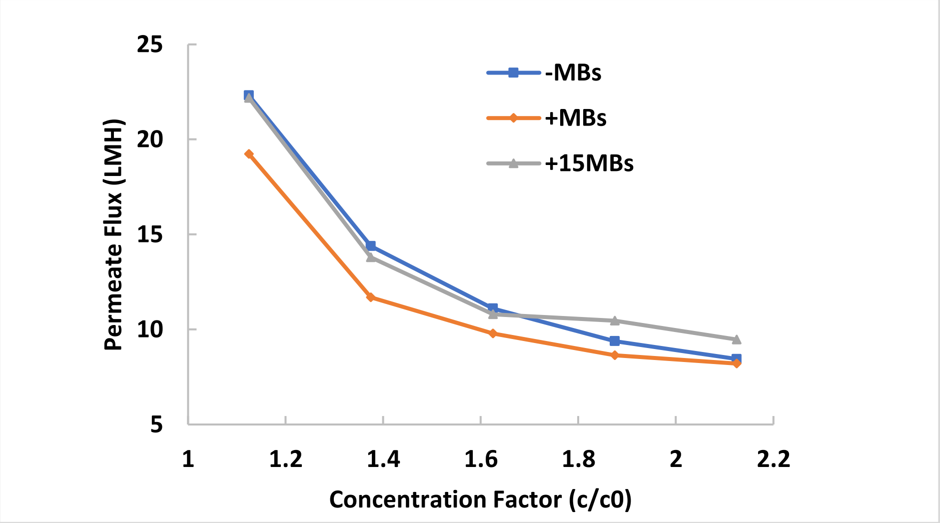

Figure 10 : Comparison of permeate flux behaviors as concentration factor increases for three operation conditions -MBs, +MBs, and +15MBs.

(15 min every 2h) there was no effect on permeate flux These results indicate that having intermittent MB injection can provide similar benefits of scaling mitigation without the detrimental impacts on permeate flux.

CHAPTER 4

CONCLUSION

This study shows that MBs can potentially alleviate and delay membrane scaling, especially at higher concertation factors. Continuous injection of bubbles helped reduce flux decline compared to without bubbles, but it reduces flux permeation as continuous injection of bubbles created dead zones (air gaps) on the membrane surface, affecting the process performance. Intermitted injection of bubbles helped reduce flux decline over time and maintain flux permeation values. The use of intermittent MBs injection instead of continuous MB injection achieved better permeation flux and lower energy consumption in comparison to continuous MB injection. These results show that MBs can be a potential chemical-free method to prevent scaling in desalination systems treating high saline solutions. Future studies can focus on studying NBs injecting, which could help reduce the effect of air gaps on the membrane surface and understand the effect of temperature and air injecting on the process performance.

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APPENDIX A

BUBBLES SIZE USING NANOPARTICLE TRACKING ANALYSIS

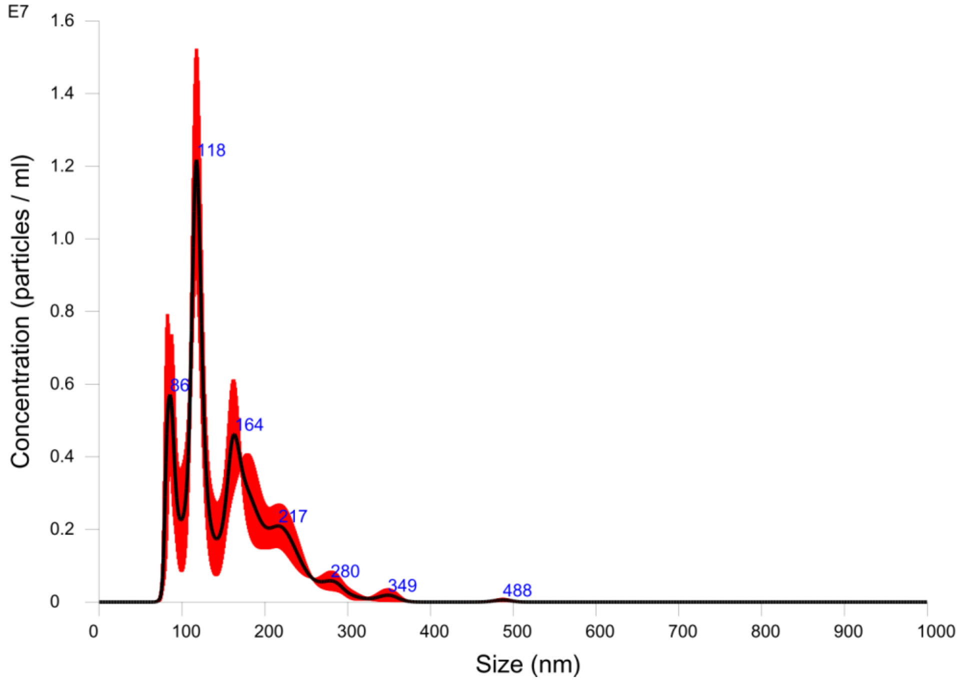

Figure A1 : Averaged FTLA Concentration / Size for the experiment

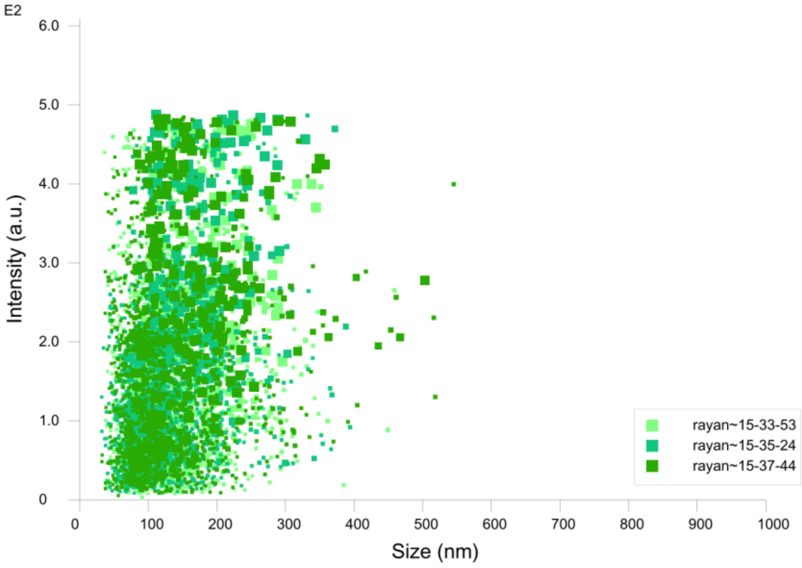

Figure A2: Intensity / Size graph for the experiment

APPENDIX B

DEAD ZONES EXPERIMENTS

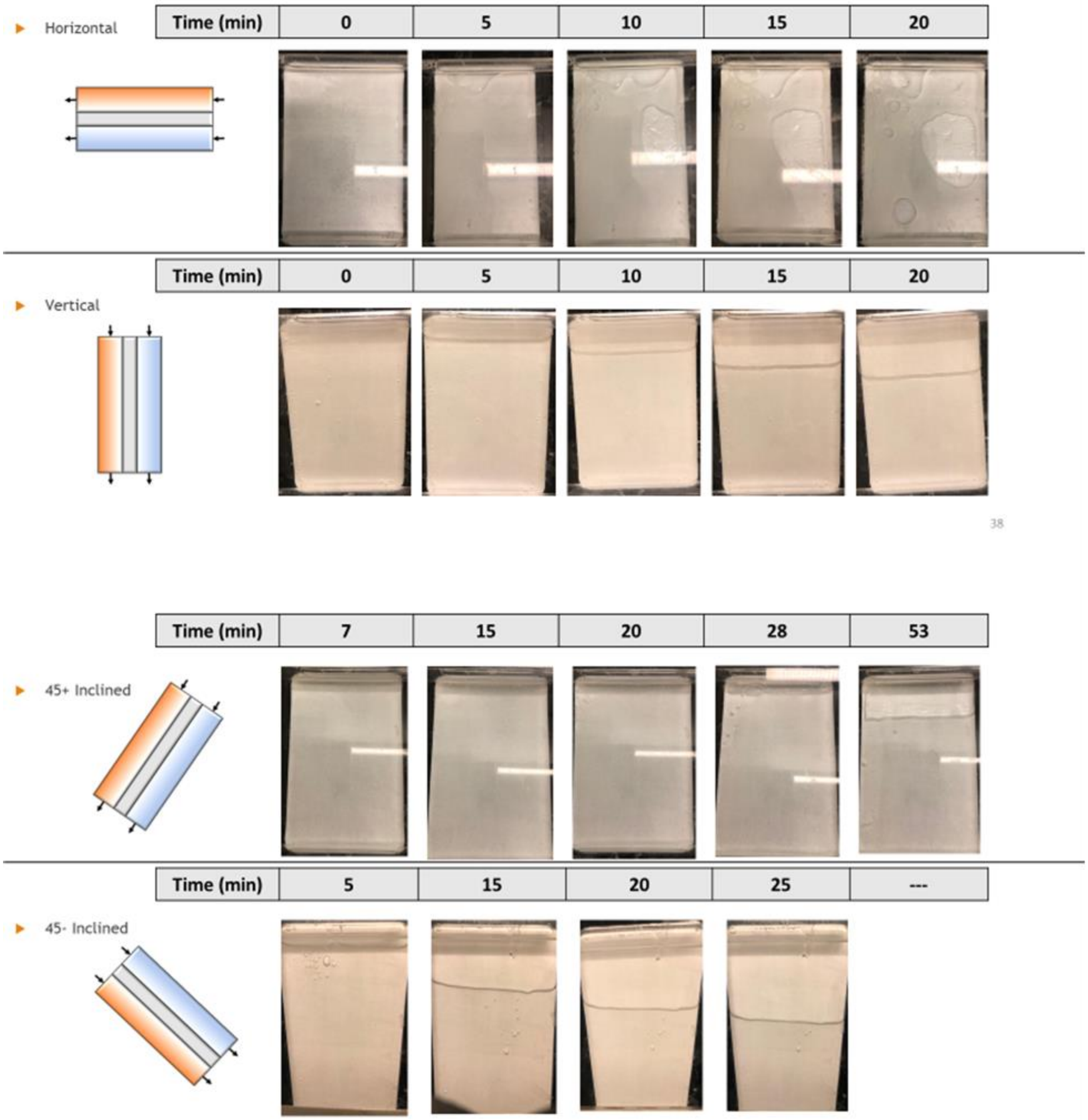

Figure B1: Effect of generating bubbles on creating dead zones on the membrane surface using different tilt angles. DI water was used as a feed solution. The microbubble pump was on, creating bubbles in the feed solution. Each experiment took around 30 min.