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Graduated in Materials Engineering

**Air Gap Membrane Distillation Process
Enhancement for Hypersaline Feed Solution by
Application of Vacuum**

Dissertation for obtaining the Master degree in Membrane Engineering

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Air Gap Membrane Distillation Process Enhancement by Application of Vacuum for Hypersaline Feed Treatment



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Abstract

This study investigated how the process performance of air gap membrane distillation be enhanced by the application of vacuum to treat hypersaline feed solutions. A vacuum-enhanced air-gap membrane distillation (V-AGMD) configuration was set up and sub-ambient pressure was applied in the air gap to remove non-condensable gases and reduce conductive heat transfer for enhancing the flux and thermal efficiency. The process was evaluated in comparison to the AGMD process while varying operational parameters such as the mean temperature (T_m), difference of temperature in heating and cooling streams (ΔT), flow velocity, and absolute air gap pressure. Initially, tap water was used as feed to set a baseline for the analysis of vapor pressure reduction incurred in hypersaline feed. Two commercial flat sheet PTFE membranes (pore size 0.1 μm and 0.2 μm) were tested. In the hypersaline feed experiments, two air gap widths (0.80 mm and 1.15 mm) were assessed in both ambient and vacuum conditions. Within the scope of this work, the operational conditions which yielded a reasonable trade-off between flux and thermal efficiency were considered optimal. These conditions were T_m 60 °C, ΔT 16 °C, flow velocity 0.1 m/s and absolute air gap pressure of 500 mbar for tap water experiments. For hypersaline feed experiments, the optimal conditions were T_m 60 °C, ΔT 16 °C, flow velocity 0.07 m/s and absolute air gap pressure of 350 mbar. A quantitative comparison between AGMD and V-AGMD setup showed that, for the determined optimal conditions using tap water, the flux improved by 37.6 %, and thermal efficiency improved by 17.7 %. For the hypersaline feed, the V-AGMD setup showed an enhancement of the flux by 279.5 % and thermal efficiency by 149.9 %. The results of this work proved that improving the performance of AGMD process is possible by deaeration of the airgap using vacuum and that V-AGMD is a viable option for treatment of hypersaline feeds.

Keywords: Vacuum-enhanced air-gap membrane distillation (V-AGMD), flat sheet membranes, thermal efficiency, hypersaline feed treatment

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Abbreviations

AGMD	Air Gap Membrane Distillation
CPC	Concentration polarization coefficient
CF	Concentration factor
DCMD	Direct Contact Membrane Distillation
GOR	Gained output ratio
HDH	Multi-effect humidification–dehumidification
LEP	Liquid entry pressure
LVE	Liquid-vapor equilibrium
MD	Membrane Distillation
MVC	Mechanical vapor compression
MDC	Membrane distillation crystallization
MSF	Multi-Stage Flash
MED	Multi-Effect Distillation
NCGs	Non condensable gases
OVP	Optimal vacuum pressure
PR	Performance ratio
PVDF	Polyvinylidene fluoride
PAC	Powdered activated carbon
PP	Polypropylene
PTFE	Polytetrafluoroethylene
PVDF	Polyvinylidene fluoride membrane
PE	Polyethylene
RO	Reverse Osmosis
SEC	Specific energy consumption
SRF	Salt rejection factor
SGMD	Sweep gas membrane distillation
TDS	Total dissolved solids
TPC	Temperature polarization coefficient
V-AGMD	Vacuum Air Gap Membrane Distillation
VMD	Vacuum membrane distillation
VOCs	Volatile organic compounds
ZLD	Zero liquid discharge

Nomenclature

J (kg/m ² .h)	Permeate flux
η_{th} (%)	Thermal efficiency
δ (m)	Membrane thickness
δ_F (m)	Thickness of boundary layer on feed side
δ_P (m)	Thickness of boundary layer on permeate side
T_{bF} (K)	Temperature in bulk feed
T_{mF} (K)	Temperature on membrane surface at feed side
T_{bP} (K)	Temperature in bulk permeate
T_{mP} (K)	Temperature on membrane surface at permeate side
h_F (W/(m ² .K))	Heat transfer coefficient on feed side
q_L (W/m ²)	Heat transferred because of volatile component evaporation
N (mol/m ² K)	Vapor flux
ΔH_{vap} (J/mol)	Latent heat of vaporization
q_P (W/m ²)	Heat transfer across the permeate side of the membrane
q_m (W/m ²)	Heat transfer via gas-filled pores
λ_i (m)	Mean free path of the vapours transferred
d_p (m)	Membrane pore size
S (g/kg)	Salinity
p_{sol} (mbar)	Vapor pressure above solution
p_w^o (mbar)	Vapor pressure of pure water
x_w	Molar fraction of water in solution
a_w	Water activity
γ_w	Water activity coefficient
$P_{v,(T)}$ (mbar)	Saturation vapor pressure at temperature T
H_{Ei} (W)	Heat in evaporator inlet
H_{Eo} (W)	Heat in evaporator outlet
Q_{lossE} (W)	Heat losses

Chapter 1: Introduction

1.1 Background and motivation

With the world's eye set on sustainable development, factors such as growing world population, drastic climate change, and increase in natural disasters have taught an important lesson: resource management is key to sustaining life on Earth [1], [2]. Water is one such resource that serves as a linchpin for a healthy and balanced ecosystem. Although about 70 % of the Earth's surface is covered with water, only 2.5 % is fresh water[3] from which 1 % is easily accessible [4]. More than 20 % per person of this accessible water has declined globally over the last 20 years [5]. With the existing climate change scenario and 40 % of the world population already residing in arid areas or where freshwater is scarce [6], it is predicted that by 2030 a global water deficit of 40 % will be incurred [7] and more than 700 million people could be displaced [8]. Such alarming statistics call for further development of technologies for desalination, recycling, and reuse of water resources to meet future needs especially for potable water. At present, there are many desalination technologies under study as shown in Figure 1.1.

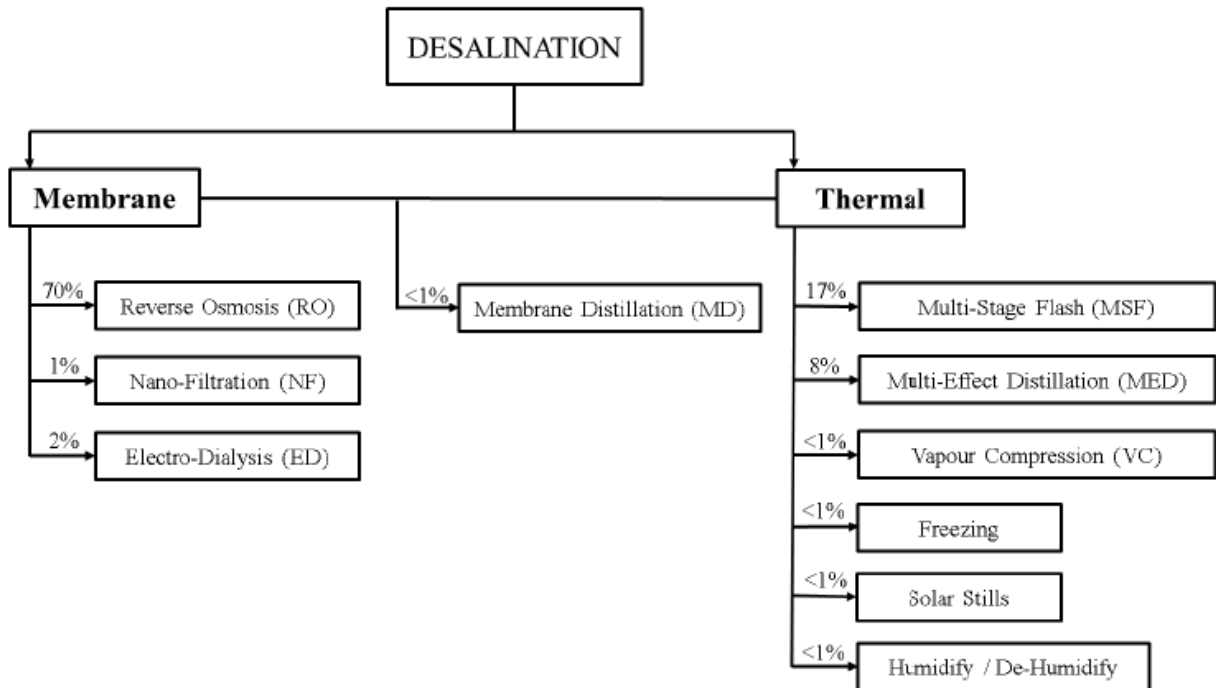


Figure 1.1: Desalination techniques [9]

Not all the techniques in Figure 1.1 dominate global water production. As per the International Desalination Association (IDA), desalination plant capacity in the world reached 99.8 million m³/day in 2017 [10], [11]. The most common commercially used technologies for potable water include pressure-driven separation by reverse osmosis (RO) and thermal distillation by multi-stage flash (MSF)

and multi-effect distillation (MED) [12]. There is, however, a growing concern with regards to these separation processes as they posit a negative impact on the environment especially the marine and coastal ecosystems owing to dumping of concentrated and chemical discharges. For Zero discharge liquid (ZLD) process, RO can be employed to treat incoming wastewater stream, but it is limited by the upper bound of salinity concentration as depicted by Figure 1.2. In the same plot, it can also be observed that the concentrators or crystallizers which treat the RO retentate consume high energy. Therefore, it opens research avenue for new technologies. Other concerns associated with conventional technologies include salting of groundwater, high propensity for membrane fouling, and requirement of high robustness and mechanical strength of membranes used [12], [13]. Although the progress is slow, the research focus has now shifted on decreasing dependence on these processes and finding innovative solutions for countering the disadvantages and limitations of conventional desalination processes. One promising process is that of Membrane Distillation (MD). It is a hybrid technology that combines both membrane technology and thermal distillation and can be applied for many separation processes; be they industrial or desalination in nature. It is attractive majorly because it can be used to treat hypersaline solutions and its advantage of operating at low-temperature levels of under 80 °C which is considered non-usable waste heat in most industries [4], [14], [15]. Although the capability of using solar thermal energy or waste heat and producing drinkable water from concentrated streams from other separation processes makes MD seemingly a cost and energy-efficient and environment-friendly process, it is still a novel technology and remains under development. Work regarding its operational optimization, membrane fabrication, and countering of its drawbacks requires research.

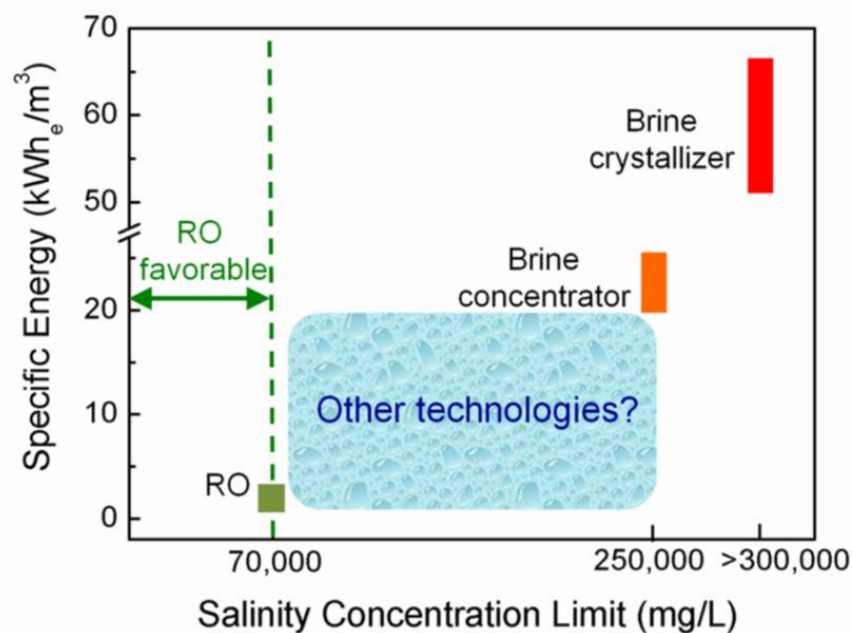


Figure 1.2: Operating ranges of salt concentration in a conventional ZLD system with a potential scope for new technologies [16]

1.2 Objectives

This project was focused on the experimental and analytical investigation of a novel MD configuration namely Vacuum Air Gap Membrane Distillation (V-AGMD) to assess the effect of employing vacuum for the treatment of hypersaline solutions under different operational conditions such as temperature and fluid velocities. Initially, the objective was to carry out baseline experiments using tap water as feed solution and analyze how the key performance indicators such as flux and thermal efficiency vary with experimental conditions. This was followed by using a hypersaline feed solution as feed. In a V-AGMD setup, analysis of these parameters was then carried out with one selected air gap pressure after which the pressure was then subsequently varied. As this project is embedded within the initial phase of the public funded research project HaSiMem, based on the findings, an outlook and recommendation for the continuation of the work is given as a conclusion.

1.3 Structure of report

The report starts with a comprehensive literature review carried out for the project. It includes all necessary information that laid a base for theoretical understanding before the experimental work was started. Chapter 3 then discusses the materials used and the methodology employed to achieve the objectives of this thesis project. It is followed up by Chapter 4 in which a comprehensive discussion of obtained results is done and the final chapter concludes while giving recommendations and shedding light on future perspectives of the MD process.

Chapter 2: Literature Review

This chapter summarizes the literature study done for this thesis project. First, it reviews the evolution of MD as a viable water treatment process followed by its definition and comparison with other processes and relevant fields of application. After establishing that, fundamentals regarding MD are discussed followed by a review of the most common MD configurations used for the treatment of hypersaline feed and why the novel V-AGMD configuration is a balanced solution.

2.1 Evolution of membrane distillation

Today membrane distillation is a fast-growing research topic for desalination, but this was not the case always. Bodell defined the MD process and filed for a patent in 1963 followed by another patent in 1964 by Weyl and the first paper was published in 1967 by Findley [17], [18]. In the following years, MD did not garner much commercial interest because of the low flux obtained compared to other processes such as RO until the 1980s when advances were made to commercialize MD membranes. The notable names in this regard include Gore and Associated Co. the Swedish National Development Co. and Enka AG [17]. Previously, MD had different industrial names such as transmembrane distillation, membrane evaporation, and thermo-pervaporation (PV) owing to dependence on a liquid-vapor equilibrium and the need for external energy to obtain the necessary latent heat of vaporization of the feed [17]. In 1986, however, a workshop on MD was held in Rome to attribute a unique name for the process and hence ‘Membrane Distillation’ was coined [17]–[19]. From a commercial viewpoint, MD is still to make a solid niche, but because of it being an effective and practical separation technique, the academic community is striving to overcome the associated problems. Figure 2.1 shows the growth of MD publications since 2000.

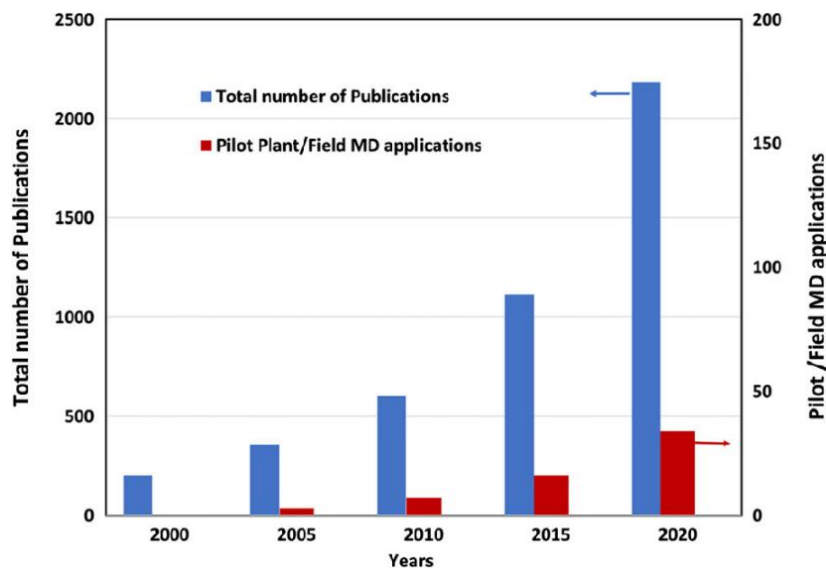


Figure 2.1: Growth of MD publications [20]

2.2 Definition and comparison with other water treatment processes

Membrane distillation is a non-isothermal separation process that employs a porous hydrophobic membrane that does not change the liquid-vapor equilibrium (LVE) of the species in the feed [17], [19]. This membrane is in contact with hot fluid on the feed side and a cooling fluid flows on the permeate side. A transmembrane vapor pressure difference is established because of this temperature gradient and serves as the primary driving force [21]. Figure 2.2 (a) represents the schematic of the MD principle.

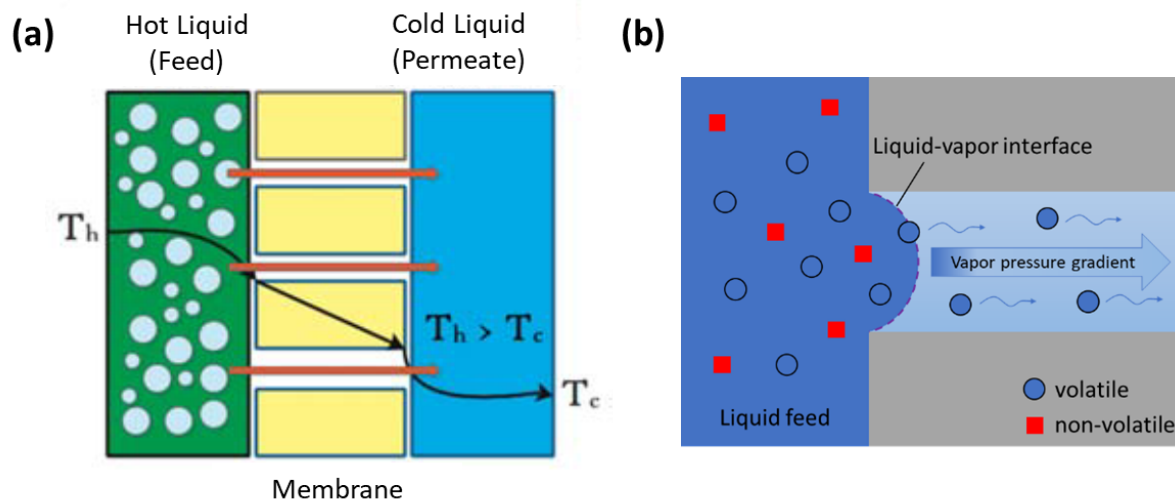


Figure 2.2: Schematic representation of (a) MD principle [18], (b) MD evaporation at Liquid vapor interface [9]

Figure 2.2 (b) shows that because the membrane is hydrophobic, the liquid feed does not penetrate the membrane pores thus a liquid-vapor interface is formed and if a transmembrane vapor pressure gradient exists, volatile feed components evaporate and the vapors formed are forced to go through the membrane pores towards the permeate side [9].

Figure 2.2 (a) and (b) not only help to understand the basic concept of MD but also to draw a comparison with other conventional wastewater treatment processes. The driving force depicted in Figure 2.2 (a) makes MD unique for saline water treatment. Generally, the feed temperature requirement for MD is within range of 40-85 °C as opposed to the conventional distillation process, which operate at higher temperatures. This implies that MD can potentially be coupled with low-grade waste heat and renewable sources of energy [22], [23]. Waste heat with low enthalpy is not expected to have such an important role in other processes such as Multi-Stage Flash (MSF) or Multi-Effect Distillation (MED) [24]. The low operating temperature and pressure requirement for MD increases process safety and reduces the overall equipment cost for the process in comparison with the conventional processes while also making it an environmentally friendly process [14], [25], [26]. MD can also operate at feed salinities where a process like RO is limited. It can be used to treat a hyper saline feed with concentrations of up to 70-300 g salt/kg solution [27], [28]. For such a high concentration the required osmotic pressure is

approximately 300 bar, and the conventional spiral-wound RO process operates under 70 bar. Therefore, for such cases, the air gap membrane distillation (AGMD) configuration of MD serves as a viable option [28]. Furthermore, complete rejection of non-volatile feed components (such as salt), colloidal species, and macromolecules at low temperatures is another aspect where MD stands above other processes [22], [24], [28]. Unlike MD, common processes like RO and NF suffer from limits caused by concentration polarization, so it can be employed when the demand is for high retentate concentration or high permeate recovery factor. Figure 2.3 shows a comparison of MD and RO whereby transmembrane fluxes were measured for an orange juice concentration test. After 17.5 °Brix solute concentration, there is a drastic drop in RO flux owing to an increase of osmotic pressure with concentration. Although the MD flux also decreases but the drop is not that drastic. The drop is attributed to increased viscosity of the feed solution and reduction of the activity coefficient.

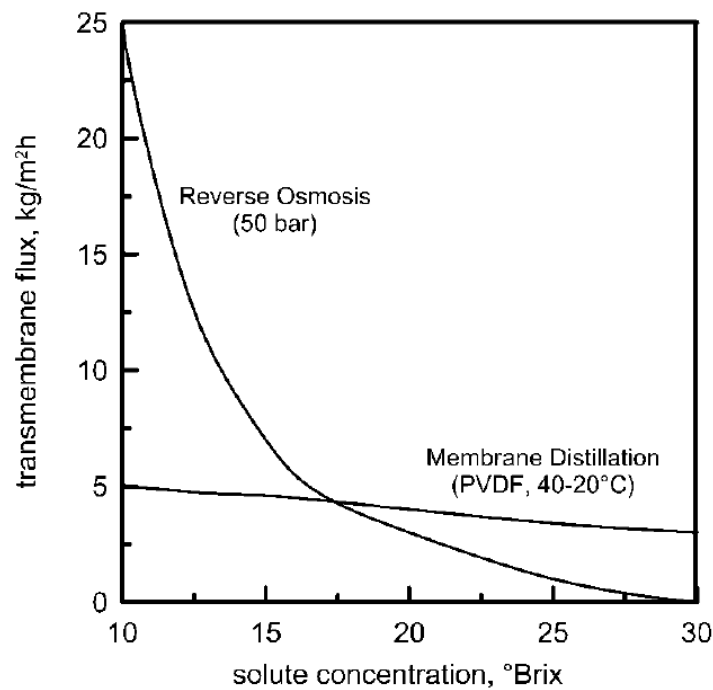


Figure 2.3: Comparison of RO and MD fluxes for concentrating orange juice [24]

MD can also be integrated with other techniques like MED and bioreactors to enhance water recovery, decrease the amount of concentrate to be disposed of, and for improving the overall energy efficiency [18]. An example of integrating MD with the conventional process is found in recent literature. Thermal membrane distillation was coupled with RO and a sensitivity analysis was carried out by Huang *et al.* and it was observed that the heat to power demand ratio for the hybrid system was less than that for conventional RO setup. The freshwater and fuel consumption dropped by 21 % and 1.7 %, respectively [29]. In another study, MD was coupled with RO in a hybrid system for desalination as MD is less sensitive to high brine concentration relative to RO [17], [30]. As per the results, the recovery ratio of the hybrid setup was 87 % which was higher than that obtained in MD and RO individually. Standalone,

in MD and RO, the recovery ratio was found to be 77 % and 40 %. For applications where purified water was used for a boiler, the MD permeate of high quality could be used in place of RO permeate. In the mining industry where the brine discharge and water recovery ratio from RO is limited, MD is a viable alternative [18]. Henceforth, it is established from the literature study that MD can be more economical than other thermal processes in zero liquid discharge (ZLD) applications.

For MD, in contrast with other common pressure-driven separation techniques, the possibility of using plastic equipment is higher so there are fewer chances of erosion of equipment [14], [24]. One of the requisites for MD is employing highly hydrophobic membranes, therefore, scaling, and organic fouling of hydrophobic membranes is not as serious as for hydrophilic membranes employed in other processes. Hence, pretreatment requirements for MD are not that stringent or relatively expensive [14], [25]. Furthermore, as the membrane is not influencing the LVE, its only purpose is to act as a barrier even under low pressure and temperature conditions, and the membrane mechanical properties are relatively more flexible than for other treatment processes. Additionally, the large surface area available allows high vapor concentration [14]. Another advantage that MD has over other water treatment processes is that since the vapors have to diffuse over a short distance from evaporation to condensation, the membrane is thin and compact with high (m^2/m^3) ratios [14]. MD has some disadvantages when compared with other separation processes. The availability of MD-specific membranes is an issue and so is the risk of membrane wetting higher from the liquid in the feed or with hypersaline feeds. Another problem is the low flux obtained when compared with other membrane separation processes [14]. Table 2.1 shows a qualitative comparison of MD with other established desalination technologies such as RO, MED, MSF, and mechanical vapor compression (MVC).

Table 2.1: MD compared with other separation techniques across key performance metrics [31]

Metric	MD	RO	MED	MSF	MVC
Energy Efficiency 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆
High-Salinity Feedwaters 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆
Small-Scale Operation 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆
Utilizing Low-Grade Energy 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆
Fouling Resistance 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆
Rejection of Non-Volatile Neutral Solutes 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆
Low Pretreatment 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆
Low Lifetime Costs 	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆	☆☆☆☆

2.3 Fundamental concepts of membrane distillation

Before reviewing the findings from literature about MD configuration (V-AGMD in the scope of this project) it is important to get a basic know-how of the common MD configurations, the membranes used in the MD process, the mass and heat transfer as well as the heat recovery concept and in general how saline feed affects the MD process.

2.3.1 MD process configurations

Membrane distillation can be carried out in several process configurations, each bearing some pros and cons. In general, the vapor pressure difference between the feed and permeate side is the driving force across the membrane. The feed solution is in contact with the membrane always, and henceforth, defines a certain vapor pressure concerning the liquid-vapor interface at the feed side. On the permeate side, however, the vapor pressure gradient can be manipulated in several ways such as [9]:

- i. Lowering the temperature on the liquid-vapor interface
- ii. Application of vacuum
- iii. Using a sweeping gas
- iv. Applying a stripping solution on the liquid-vapor interface

These approaches have given rise to some basic and other novel MD configurations. The more conventional ones being direct contact membrane distillation (DCMD), vacuum membrane distillation (VMD), air gap membrane distillation (AGMD), and sweep gas membrane distillation (SGMD). These 4 conventional configurations have some common features: The hot feed is in contact with the microporous hydrophobic membrane and only the vapor diffuses to the cooler side. This vapor can be of water as in the case of desalination or some volatile solute present in water mixed with water vapor. For all four configurations, the pressure and temperature at the feed side are kept moderately low in the range of 50-80 °C. The lower bound is at 50 °C, as lower temperatures would not give significant vapor pressure at the membrane interface on the feed side, while temperatures higher than 80 °C would be relatively more energy-intensive [32]. A maximum bound of 80 °C means low-grade waste can be utilized for heating the feed if heat recovery is being done. Furthermore, the modules use PP as the main component and beyond 80 °C it can start to melt. PVDF can be used as an alternate option, but it is more expensive than PP. The major difference between these configurations is whether the passing vapor is cooled within the membrane module or externally. Some novel configurations are stemming from these conventional ones such as osmotic membrane distillation, permeate gap membrane distillation, feed gap air gap membrane distillation, material gap MD (MGMD), and conductive gap MD (CGMD) [21]. Figure 2.4 shows the schematics of the four conventional configurations.

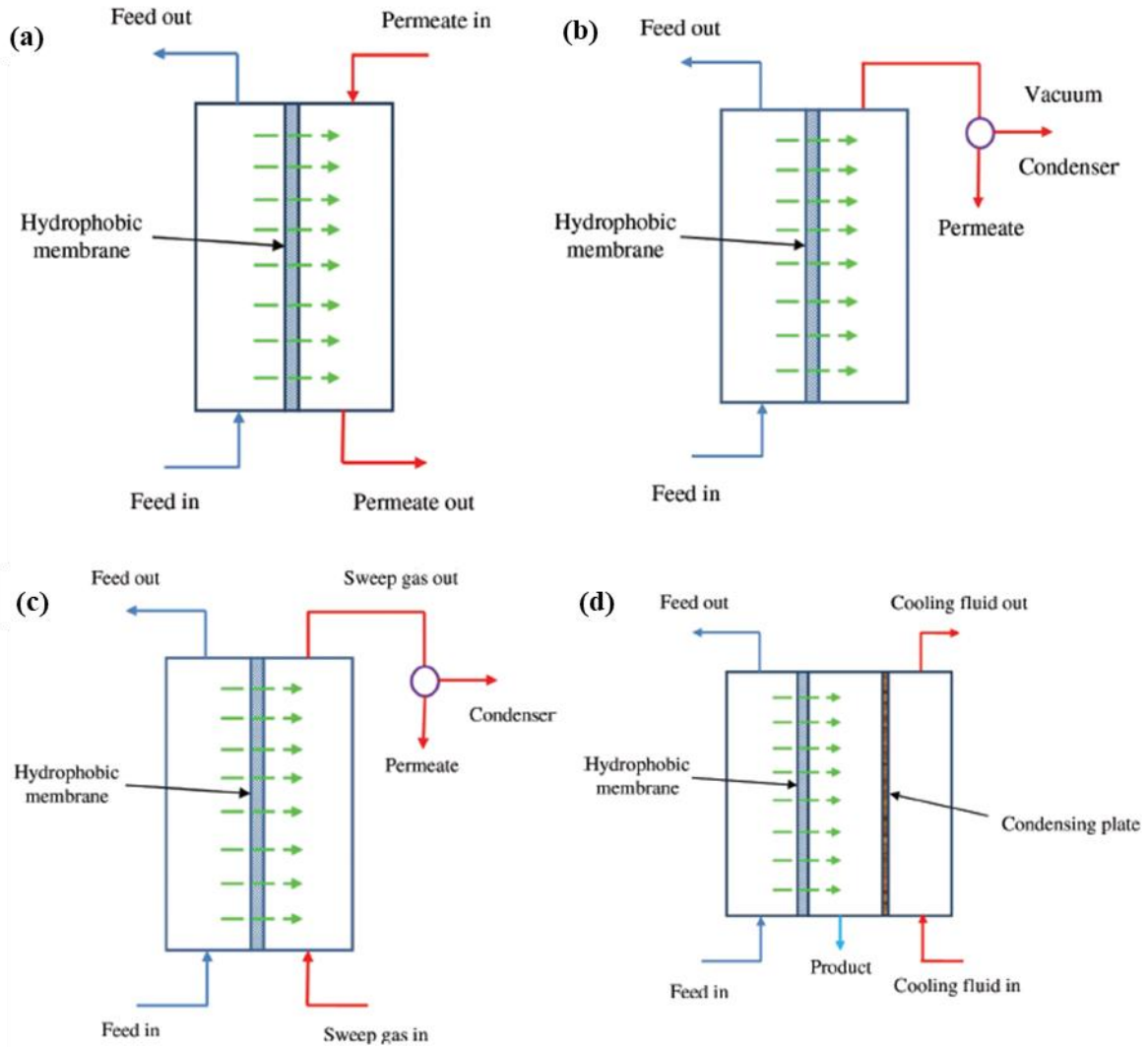


Figure 2.4: Common MD configurations (a) direct contact membrane distillation, (b) vacuum membrane distillation, (c) sweep gas membrane distillation, (d) air gap membrane distillation [28]

2.3.1.1 Direct contact membrane distillation (DCMD)

Being the oldest and most widely studied configuration of MD, DCMD is primarily used for desalination and concentrating aqueous solutions because it is easier to handle the setup [33] and has adequate flux [28]. As shown in Figure 2.4 (a), both the feed and permeate are in direct contact with the hydrophobic membrane which prevents the condensed vapor to permeate via the membrane matrix thereby maintaining the liquid-vapor interface [34]. The diffusion path for the membranes is restricted to the pores of the membranes only, thereby decreasing the mass and heat transfer resistance [35]. The permeate acts as a coolant and is recirculated inside the membrane module where the distillate is mixed with the coolant. Recirculation is done to completely condense the vapor inside the module [34], [36]. Although the DCMD setup is easier to handle and usually gives high flux values, low efficiency is

observed because a large amount of heat is lost by conduction through the membrane as both the liquids are in direct contact with it [37], [38]. Operating at high temperatures can lower the heat losses but not eliminate them [32]. Owing to this reason, commercially other MD configurations are gathering interest. The main fields of application for DCMD are desalination [39] and concentrations of aqueous solutions in the food industry [38]. DCMD has also been used in the textile industry for its cost-effective water treatment [40].

2.3.1.2 Vacuum membrane distillation (VMD)

VMD is one of the most favourable MD configurations. In this setup, as schematically represented in Figure 2.4 (b), the membrane is placed between the hot feed and a vacuum side whereby a vacuum pump is used for withdrawing the vapor by exerting a vacuum pressure on the permeate side. This pressure is kept less than the saturation pressure of the volatile components in the hot feed solution. It is done to maintain a driving force for the separation to take place as it allows for a higher partial pressure gradient [28], [37]. The hydrostatic pressure across the membrane should be less than the LEP so that the liquid feed does not penetrate the membrane pores and wet it. In VMD this is a major issue to be addressed. The difference between the hydrostatic pressure and the LEP creates a liquid-vapor interface on the membrane at the feed side so the bulk feed evaporates owing to the lower pressure applied at permeate side. The vapor pass through the membrane pores and condense outside the module with the use of an external condenser [28], [37]. The attractiveness of VMD lies in the application of vacuum which not only provides a driving force but also leads to low heat conduction (and hence high thermal efficiency) thereby yielding high distillate flux in comparison with other MD configurations [37], [38]. Furthermore, VMD is characterized as less demanding in terms of membrane mechanical properties and achievement of higher salt rejection rates for non-volatile solutes. The applied vacuum, however, also posits disadvantages such as the increased risk of membrane wetting and the need for supplementary electrical power for the vacuum pump [41]. The main applications of VMD are VOCs separation from aqueous solutions [28] and for water purification [42], [43].

2.3.1.3 Sweep gas membrane distillation (SGMD)

SGMD is also referred to as air stripping MD and, as shown in Figure 2.4 (c), encompasses a gas barrier between the membrane and the cold surface which leads to higher efficiency because of the minimized heat losses. This gas barrier sweeps the membrane, enhancing the heat transfer coefficient and resulting in a high permeate flux. Compared to the DCMD setup, SGMD gives better evaporation efficiency and flux values [37], [44]. Just like in VMD, an external condenser is incorporated in the SGMD setup. The sweeping gas is used as stripping gas which collects the vapor at permeate side for their condensation outside the module using the external condenser. To produce a significant amount of permeate, large volumes of sweep gas need to be used along with external condensers which can increase the overall

cost of the SGMD process, therefore, making it a less attractive option on a commercial scale [18]. To address the issue of low permeate obtained, a study was carried out and it was reported that coating the membrane with polydimethylsiloxane or the application of hollow fibers can result in higher flux values [45]. Some applications for SGMD include desalination, wastewater treatment, concentration of feed, and biorefining. However, it is acknowledged that the energy consumption and optimization require further investigation [46].

2.3.1.4 Air gap membrane distillation (AGMD)

AGMD is another variant of MD configuration in which the air gap gases are stationary and not sweeping over the membrane as in the case of SGMD. It was the first set up to be tested at pilot scale [47]. In this setup, an air gap is kept between the membrane and the condensing plate/film. The air gap section is also the permeate section as shown in Figure 2.4 (d). The cooling liquid used for condensing the vapor flows on the other side of the cooling film. Owing to the difference in temperature, the feed solution evaporates at the liquid-vapor interface, passes through the hydrophobic membrane and the air gap, and eventually condensing on the cool surface of the impermeable film. Gravity makes the liquid permeate move towards the bottom of the air gap from where it is extracted so that the air gap does not fill up [9]. One of the main advantages of the AGMD setup is the lower temperature polarization and conductive heat losses because it acts as an insulating layer while being stationary in gap [28]. This makes AGMD an energy efficient option. In literature, thermal efficiency values as high as 70-99 % are reported and thus more energy efficient than DCMD [48]. However, this advantage incurs a problem of the additional mass transfer resistance which is directly proportional to the thickness of the air gap. This mass transfer resistance is largely attributable to the non-condensable gases present in the air gap. Using the AGMD setup, it is easier to detect membrane wetting or leakage in the system because the permeate is obtained from the air gap without any physical separation. Furthermore, as the cool stream and hot vapor are separated due to a condenser, internal latent heat recovery is possible. In this configuration, by using the integrated cooling plate, multi effect or multi-stage membrane modules can be set up [34], [49].

2.3.1.5 Vacuum enhanced air gap membrane distillation (V-AGMD)

When all the common MD configurations are compared, it can be observed that DCMD has a high flux and simple module design but faces high conduction heat losses. While AGMD presents a solution by introducing an insulating air gap, it gives low permeate flux values. In summary, DCMD has high flux and AGMD has relatively high thermal efficiency [28]. V-AGMD which is a derivative of the AGMD setup aims to strike a balance between high flux and thermal efficiency.

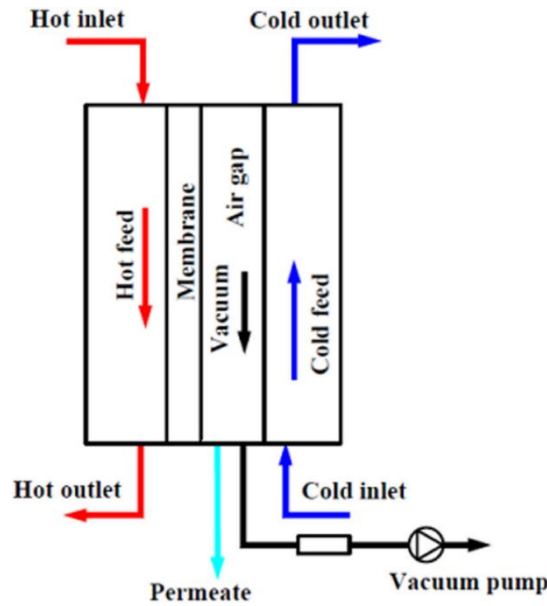


Figure 2.5: V-AGMD set-up schematic [50]

Figure 2.5 shows the V-AGMD setup. The inspiration for this setup stems from the aim to optimize the mass transfer rate in AGMD by overcoming the mass transfer resistance incurred in AGMD due to the presence of NCGs in the membrane pores and air gap. One way the air gap resistance can be overcome is by employing a vacuum pump for deaeration.

One major issue associated with V-AGMD is that because of vacuum generation in the air gap, the limiting liquid entry pressure (LEP) decreases, and the chances of membrane pore wetting increase as compared to the basic AGMD configuration. The goal of this thesis project is to build on these findings from the literature and attempt to optimize the V-AGMD process by varying the operational parameters to obtain high flux. Table 2.2 compares the advantages and disadvantages of some of the main MD setups described before and highlights their main areas of application [17], [18].

2.3.2 Membranes used in MD

Membranes are an essential component of the MD process, with hydrophobicity being an important property. Therefore, these are made from polymers with low surface energies [18]. Generally, the materials used are PTFE, PP, and PVDF.

Figure 2.6 shows a comparison of the hydrophobicity of these materials found in the literature. Hydrophobicity is a function of and in direct relation with the water contact angle. Other important requirements include the membranes to have low thermal conductivity and low resistance to mass transfer. It is important in setups like DCMD where the resistance contribution by the membrane is most important to determine the flux obtained. The membranes should also have inertness to chemical mediums such as acids and bases and be stable at high temperatures.

Table 2.2: Properties of common MD configurations

MD configuration	Area of application	Advantages	Disadvantages
DCMD	- Desalination of seawater - Arsenic removal from aqueous solution - Dye effluents treatment - Crystallization	- Considered at commercial scale - High permeate flux - Operation simplicity - Least required equipment - Simplest MD configuration	- High conductive heat loss - Suitable for volatile organics and undissolved gasses
VMD	- Desalination of seawater - Alcoholic solution treatment - Aroma compounds recovery - Textile wastewaters treatment	- Commercial scale applicability - High permeate flux - Negligible conductive heat loss	- High risk of pore wetting - Process complexity - Higher fouling - Additional requirement of vacuum pump and external condenser
SGMD	- Desalination of brackish water - Azeotropic mixtures separation - Treatment of wastewater - Removal of VOC	- Barrier reduction of the mass transport through forced flow - Suitable for removing volatile components and dissolved gasses - Lower thermal polarization	- Risk of temperature polarization - Process complexity (Large condenser needed) - Low flux
AGMD	- Desalination of seawater - Fruit juices concentration - Azeotropic mixtures separation - Removal of VOC	- Low conductive heat loss and high flux - Simple process - Low risk of temperature polarization - High set-up flexibility - Less susceptible to fouling	- Lower flux than DCMD and VMD - Additional resistance to mass transfer - Hard module designing - Lowest output ratio

Membranes for MD should be thin having large pore size and porosity but narrow pore size distribution and low pore tortuosity [14]. This is important to have high permeability of the membrane and a narrow distribution range ensures the prevention of wetting of larger pores [51].

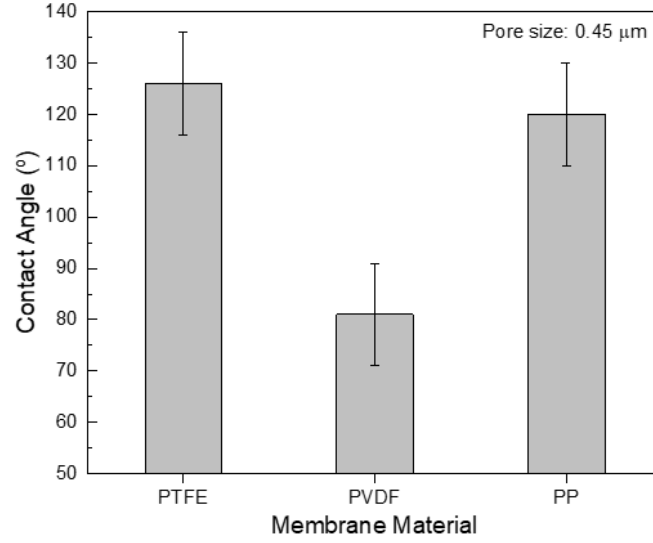


Figure 2.6: Contact Angle for common membrane materials [52]

Another significant characteristic of the membrane is the liquid entry pressure (LEP). Figure 2.7 depicts the capillary depression phenomena which helps to understand the concept of LEP. When the absolute pressure difference from the liquid feed phase to the vapor phase across the membrane ($\Delta p_{(l)-(g)}$) is adequately low, the liquid feed cannot enter the membrane pores because of the surface tension. When the surface tension is unable to resist the hydrostatic pressure and hence the liquid-vapor interface, a point comes when the liquid enters the membrane pores. This minimum hydrostatic pressure that prevents the liquid feed from penetrating the membrane pores is called the LEP. Therefore, for membranes to maintain their separative functionality, a high LEP is desired which can be attained with a highly hydrophobic membrane having a small maximum pore size [9], [18]. In summary, Eq. (2.1) should be obeyed.

$$\Delta p_{(l)-(g)} < LEP \quad (2.1)$$

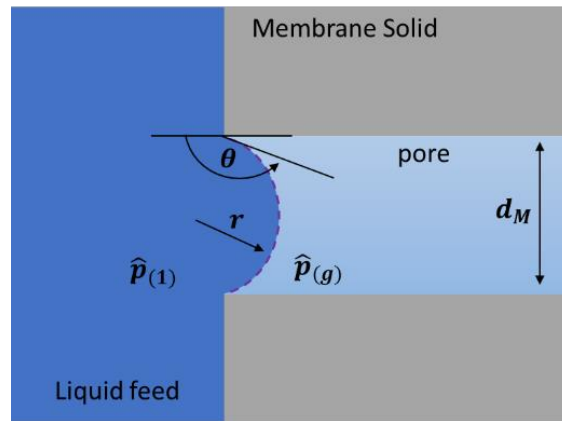


Figure 2.7: Schematic representation of the capillary depression phenomena [9]

A study showed that the effect of membrane backing depends on the structural design of the backing. The backing provides mechanical stability to the membrane and aids in turbulence. From the perspective of thermodynamic effects, backing increases the diffusion path length and influences the effective cross-sectional area for diffusion as well as provides an additional thermal resistance network [53]. Zhang *et al.* discovered that backing with a more open porous structure enhanced the membrane performance [51].

From the different studies reviewed, it can be concluded that most of the microporous commercial membranes used by researchers are flat sheet or hollow fiber membranes with a porosity range of 60–85 %, made from hydrophobic PTFE, PVDF, and PP polymers with membrane pore size in the range of 0.1-1 μm , and membrane thickness in the range 60–200 μm with minimum LEP value of 0.31 bar [28]. As a result of the review, it was also understood that at present, there are no commercial membranes made specifically for MD, and optimization of the membrane characteristics in that regard needs to be done. Table 2.3 shows the commercial membranes recently being used for MD.

Table 2.3: Commercial membranes used for MD (adapted from [17])

Membrane commercial code	Membrane type	Producer	Material	Porosity (%)	Membrane thickness (μm)
TF200	Flat sheet	Gelman	PTFE/PP	80	178
GVHP	Flat sheet	Millipore	PVDF	70–75	110
MD020TP2N	Tubular	Enka Microdyn	PP	75	1550
Celgard X-20	Tubular	Hoechst Celanese Co.	PP	35	25
G-4.0-6-7 (discontinued)	Flat sheet	Gore-Tex Sep GmbH	PTFE	80	100
PP 50/200	Hollow fiber	Accurel Membrana	PP	0.5	200
3 MA	Flat sheet	3 M Corporation	PP	60	91
Donaldson 6501	Flat sheet	Donaldson filtration solutions	PTFE	84	70
Clarcor QL218	Flat sheet	Clarcor Industrial Air	PTFE	-	40

2.3.3 Transport mechanisms in MD

It is imperative to understand how the mass and heat transfers take place in MD, in general, to work on improving the process. A detailed discussion of transport mechanisms can be found in the Ph.D. thesis

of Daniel Winter and a research article published by Liu *et al.* [9], [50]. In this report, a brief review of both heat and mass transfer is done.

2.3.3.1 Heat transfer

The mechanism of heat transfer in MD process comprises of the three following steps:

- i. Heat transfer through the thermal boundary layer at the feed side
- ii. Heat transfer across the membrane
- iii. Heat transfer through the thermal boundary layer at the permeate side

As explained in 2.3.1, for all the MD configurations, the hot feed is in direct contact with the membrane (of thickness δ) leading to the formation of a thermal boundary layer next to the membrane (of thickness δ_F). As the temperature on the permeate side is different from that on the feed side, the temperature gradient another thermal boundary layer is formed adjacent to the membrane surface but on the permeate side (of thickness δ_p). Within the thermal boundary layer, the temperature reduces from that in feed bulk (T_{bF}) to the membrane surface on the feed side (T_{mF}), and on the other side, it increases from bulk permeate (T_{bP}) to membrane surface on the permeate side (T_{mP}). As the MD process depends on the vaporization of a feed component, latent heat should be transitioned from bulk feed to the membrane surface on the feed side and via the thermal boundary layer on that side. The heat transfer coefficient (h_F) plays an important role in the transfer of heat across the thermal boundary layer. The heat flux between the bulk feed and membrane surface on the feed side is given by Eq. (2.2).

$$q_F = h_F (T_{bF} - T_{mF}) \quad (2.2)$$

On the membrane surface, when vaporization takes place, the latent heat of vaporization is transferred to the permeate side along with the stream of vapors:

$$q_L = N \times \Delta H_{vap} \quad (2.3)$$

where q_L (W/m^2) is the heat transferred because of evaporation of the volatile feed component, N ($mol/m^2 K$) is the vapor flux via the membrane pores and ΔH_{vap} (J/mol) represents the latent heat of vaporization.

Apart from this heat transfer, at the permeate side, heat transfer occurs across the membrane at that side (q_p) and also through gas-filled pores q_m . The heat transfer coefficient of the membrane plays a key role and is dependent on the gas which fills the pores and also the material of the membrane [17]. Eq. (2.4) represents the heat transfer at permeate side.

$$q_m = h_m (T_{mF} - T_{mP}) \quad (2.4)$$

One of the challenges faced during MD operation is that of temperature polarization in which there exists a temperature gradient between bulk feed and membrane surface at liquid/vapor interface. When the volatile component in the feed vaporizes, the bulk feed temperature decrease but the vapor temperature increases which leads to a reduction of the temperature difference, and hence the permeate flux drops [17], [54]. Temperature polarization coefficient (TPC) is given by:

$$TPC = \frac{T_{mF} - T_{mP}}{T_{bF} - T_{bP}} \quad (2.5)$$

where subscript m and b denote membrane and bulk.

2.3.3.2 Mass transfer

Generally, the mass transfer in MD comprises of three theories:

- i. Knudsen flow theory
- ii. Viscous flow theory
- iii. Molecular diffusion theory

In MD the volume of pores is much higher than the penetration area of membrane matrix, therefore, surface penetration is ignored [17]. Furthermore, since one of the requisites of the membranes in MD is that it must be hydrophobic, so any mass transport via the membrane matrix can be ignored. The governing mass transfer mechanism in an MD module is depicted by the Knudsen number calculated as [17]:

$$K_n = \frac{\lambda_i}{d_p} \quad (2.6)$$

where λ_i is the mean free path of the vapors transferred via the pores with a size of d_p .

In the case when Knudson number greater than 1, it is more likely for the vapors to collide with the pore wall than with one another. For such a situation the permeability through pores is given by [17]:

$$\beta_{k,m} = \frac{2}{3} \frac{\varepsilon d_{p,m}}{\tau \delta} \left(\frac{2}{\pi MRT} \right)^{\frac{1}{2}} \quad (2.7)$$

where $d_{p,m}$ represents a uniform pore size of membrane and δ , ε , τ , and are membrane thickness, membrane porosity, and membrane tortuosity, respectively.

For cases when the Knudsen number is less than 0.01, the governing mass transfer mechanism is molecular diffusion and thus the membrane permeability is given by Eq. (2.8) [17].

$$\beta_{M,m} = \frac{\pi}{4RT} \frac{PD}{P_{air}} \left(\frac{d_{p,m}^2}{\tau\delta} \right) \quad (2.8)$$

where P_{air} , P , and D represent the air pressure inside a pore, the total pressure within a pore, and the diffusion coefficient, respectively.

For cases when hydrostatic pressure is applied on the membrane with pore sizes greater than the mean free path for vapors, the governing mass transport mechanism will be that of viscous flow. It is more likely for the vapors to collide with each other than with the pore wall and for such a case with assumption of uniform pore size of membrane $d_{p,m}$, the permeability is given by the following equation [17]:

$$\beta_{V,m} = \frac{\varepsilon}{32RT} \frac{P_m}{\mu} \left(\frac{d_{p,m}^2}{\tau\delta} \right) \quad (2.9)$$

where P_m and μ are the average pressure of the pores and viscosity of transferred vapors, respectively.

Another challenge faced during MD operation is that of concentration polarization.

With the evaporation of the volatile component, the non-volatile component accumulates next to the membrane and hence a concentration gradient (concentration polarization) exists between bulk solute concentration and membrane surface [28]. Concentration polarization coefficient is given by:

$$CPC = \frac{C_{mF}}{C_{bF}} \quad (2.5)$$

2.3.4 Heat recovery

Heat recovery is an important concept, especially for module scale MD setups. In MD, a lot of heat is transferred via the membrane, either as conductive or latent heat. MD setups also make use of the heat recovery concepts in conventional thermal desalination techniques e.g., sensible heat recovery and latent heat recovery.

For sensible heat recovery, the coolant is heated continuously as it flows along the condenser channel by gaining heat from both latent heat and conductive heat transfer. When the coolant temperature at the condenser outlet exceeds the temperature of the cold feed stream, partial heat recovery can be done by preheating the cold feed stream before the application of heat via an external source. Sensible heat recovery can be employed for DCMD, AGMD, and PGMD setups and Figure 2.8 (a) depicts this process.

For latent heat recovery, as illustrated by Figure 2.8 (b), first the feed is externally heated to evaporate, thus the vapors which are produced give out latent heat to the condensation surface. This latent heat causes heating and evaporation of feed in the later stages owing to the temperature gradient established. The overall thermal efficiency, in this case, is dependent on n – the number of stages.

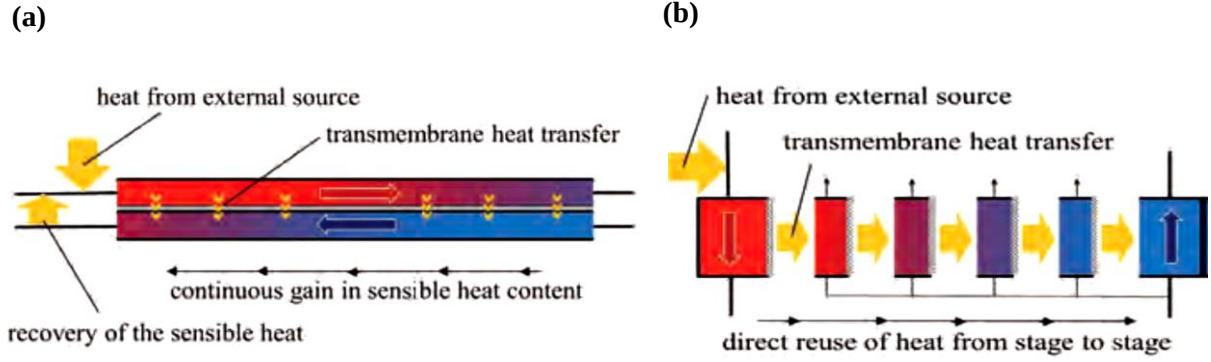


Figure 2.8: (a) Sensible heat recovery (cf. MSF), (b) Latent heat recovery (cf. MED) [9]

2.3.5 Effect of salt on MD process

One of the goals of this thesis project was to optimize V-AGMD for hypersaline feed treatment. Therefore, it was important to understand how salty feed impacts the process in general.

When a salt (solute) is dissolved in water (solvent) it dissociates into its ions and the measure of salt amount in the solvent indicates its concentration. Salinity (S) is measured in g/kg and defined by the ratio in Eq. (2.6).

$$\text{Salinity} = \frac{\text{Mass of salt dissolved}}{\text{Total mass of solution}} \quad (2.6)$$

Conductivity is a measure of the number of ions available in solution to conduct electricity and is usually in direct relation with the feed concentration but can be in inverse relation too, depending on the feed composition and how each of the salt components behaves upon dissolving with respect to temperature. In a closed system, vapor pressure is the pressure exerted by the vapors in equilibrium with their condensed phases at a given temperature. Vapor pressure is in inverse relationship with solute concentration and expressed by Raoult's law in Eq. (2.7). This law however applies only for ideal solutions whereby there exists a linear relationship between mole fraction and vapor pressure. At higher concentrations, saline water deviates from ideality and the term activity needs to be incorporated. The activity of water is defined by Eq. (2.8) as the ratio of water vapor pressure to the vapor pressure of pure water at the same temperature. Activity thereby is a function of both salt concentration and temperature.

$$p_{sol} = x_w \cdot p_w^o \quad (2.7)$$

$$a_w = \frac{p_{sol}}{p_w^o} = \gamma_w \cdot x_w \quad (2.8)$$

where p_{sol} is the vapor pressure above solution, p_w^o is the vapor pressure of pure water, x_w is the molar fraction of water in the solution, a_w is the activity of water, and γ_w is the water activity coefficient.

In MD, the water flux depends on the vapor pressure difference between the feed and permeate side [22]. When the concentration of salt increases in the feed, the vapor pressure gradient decreases, and the water flux then becomes proportional to the difference between water vapor pressure in the feed side and that on the distillate side. Vapor pressure reduction is a colligative property, and the vapor pressure can be estimated using Antoine Eq. (2.9).

$$P_v(T) = \exp\left(23.1964 - \frac{3816.44}{T - 46.13}\right) \quad (2.9)$$

where $P_v(T)$ is the saturation vapor pressure at temperature T .

From the literature review, it can be concluded that there are three reasons for water flux decrement as the feed salinity increases [22]:

- i. Decrease in water activity as salinity increases
- ii. Decrease in mass transfer coefficient owing to concentration polarization
- iii. Decrease in heat transfer coefficient because of reduction in the temperature of the membrane surface

All these reasons can be related to the understanding developed about how the physical parameters discussed above affect the vapor pressure gradient at the membrane surface. Figure 2.9 shows the difference of vapor pressure between two temperatures as a driving force of MD for pure water ($S = 0$ g/kg) and a NaCl feed solution ($S = 150$ g/kg). This graph depicts the reduction in the driving force due to the salt. For pure fresh water and at a temperature difference of 10 K between the two points 80 °C and 90 °C a vapor pressure difference (Δp) of ~ 0.22 bar is observed across the membrane. This gets reduced to ~ 0.14 bar for the salty feed which corresponds to at least 36.4 % reduction in the driving force.

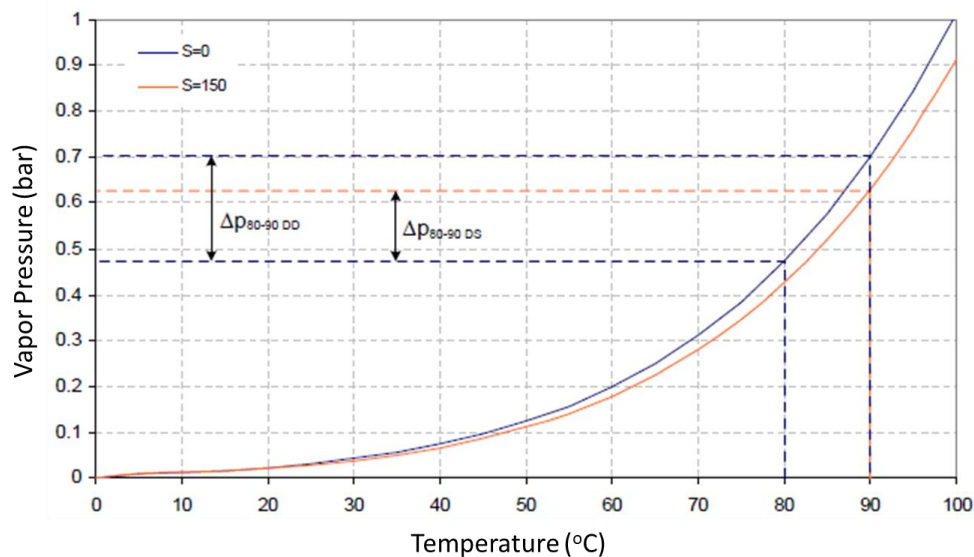


Figure 2.9: Influence of salinity on the correlation between temperature and vapor pressure [55]

It can be concluded that the flux directly depends on the transmembrane temperature difference (ΔT) and to achieve the desired flux, the vapor pressure-lowering effect of all the components present in the feed should be considered [56]. Relating this with MD, it is known that as the feed concentration increases, Δp (the driving force) and water activity decrease so the MD performance is influenced negatively. This is because a fraction of the supplied heat energy is utilized to overcome the vapor pressure reduction. As a result, the permeate flux and thermal efficiency drop.

2.4 Fields of application

MD is slowly establishing its place in the market with regards to several applications because of its unique properties as discussed in section 2.2. Among those, the most attractive characteristics of MD would be the high separation factor and dealing with water as a key component of physical and chemical processes. At present, MD is being used in the pharmaceutical [57], nanotechnology, mining, and environmental industries [31]. The ability to be used as a single process or in integration with other separation techniques as the last step has increased the domain of MD applications, with some notable ones being [17], [31]:

- i. Desalination of groundwater, seawater, brine, and brackish water supplied by other units
- ii. Removal of volatiles from alcohols, fruit juice, benzene, and halogenated VOCs using VMD and SGMD
- iii. Industrial wastewater treatment including volatile acid recovery from effluents, treatment of radioactive waste, the concentration of non-volatile acids, treating textile industry effluents, and recovering salt by membrane distillation crystallization (MDC)
- iv. For the medical and pharmaceutical industry, preparation of distilled, pure, and ultra-pure water
- v. To dealcoholize fermented beverages and enhancement of ethanol production using DCMD
- vi. Producing concentrates of liquid food e.g., mandarin juice, sucrose solution, apple juice, and whey

It is noteworthy to mention here that the most significant application of MD is the desalination of hypersaline wastewaters to produce potable water or to make wastewater safe for discharging back into the environment [20], [25], [58], [59]. In theory, MD guarantees 100 % rection of any biological matter, colloids, and non-volatile solutes that are prevalent in water sources. It has been reported in the literature that the electrical conductivity of MD products can be as low as $0.75 \mu\text{S/cm}$ [60], [61], and total dissolved solids (TDS) concentration as low as 0.6 ppm [17], [61].

2.5 Challenges for commercialization of MD and future prospects

Although MD has unique fields of application which makes it a promising separation process there exist some challenges that hinder its commercialization on a large scale. Some of these are fouling, the high propensity of inorganic scaling, high energy consumption and, membrane stability [18], [32]. Unavailability of MD-specific membranes on a large scale is another challenge as currently hydrophobic microfiltration membranes are used for studies on MD [18], [31]. Other challenges include conductive dissipation (especially in DCMD) [37].

The key to breaking barriers for commercialization of MD lies in:

- i. Fabrication and use of membranes specifically for MD process that combat fouling. For example, omniphobic membranes which repel both water and oil can widen the application of MD to the treatment of wastewater containing low surface tension constituents or composite membranes that resist organic fouling more than just hydrophobic membranes. Fabrication of highly porous thin membranes with high thermal conductivity and low tortuosity are needed to counter the effects of temperature and concentration polarization [62].
- ii. Effective module design catering to specific MD requirements for proper mixing on both feed and permeate side can help combat temperature and concentration polarization effects.
- iii. Integration of MD with other separation processes to yield high thermal efficiency.
- iv. Conduct MD modelling to analyze concentration polarization effect more effectively and to fabricate novel membrane architectures.
- v. Investigation of MD membranes over long-term operation to evaluate consistency of performance metrics.
- vi. Overall performance enhancement with a combination of new membrane materials and smart module design.

2.6 Hypersaline feed and MD

As highlighted in section 2.4, MD is an emerging technology to treat hypersaline solutions. It has garnered commercial interest over the years for desalination applications as depicted by the surging numbers of referred papers and patents [32]. A key application domain for MD has been identified as using a zero liquid discharge (ZLD) process chain for hypersaline effluents [63]. A pilot unit from the company Memsys was used to treat hypersaline groundwater in a hybrid setup of MD facility and multi-effect humidification–dehumidification (HDH) unit for establishing a ZLD process. The salinity of the incoming feed to MD unit was at 6.3 % TDS. The MD brine contained 10.2 % TDS, and both the units attained TDS rejection greater than 99.9 %. For MD, the specific energy consumption (SEC) was 260 kWh/m³ [63].

Hussain *et al.* reviewed field testing of MD technology and highlighted primary applications as seawater desalination, brine desalination coupled with thermal desalination plants, desalinating hypersaline groundwater, and concentration for hypersaline brines. The results of different pilot investigations of MD technology are summarized in

Table 7.1 in the Appendix as adapted from field evaluations done by Hussain *et al.* [20].

The effect of varying operational parameters for MD to treat saline feed has been studied both on lab and pilot scales. Despite that, the results of different MD configurations found in the literature study cannot be directly compared because the test conditions such as flow rate, temperature, and membrane characteristics vary in each study. Furthermore, the lab-scale experiments differ from those carried out on the pilot scale in terms of heat recovery done at the pilot scale as opposed to independent heating/cooling loops of evaporator inlet and condenser outlet streams on the lab scale. Also, the flow velocities on the pilot scale are lower than that for the lab-scale owing to a larger membrane surface area.

As this thesis project dealt with experimenting with V-AGMD for both tap water and high saline feed, the literature review encompasses the study of V-AGMD and other MD configurations done at lab scale primarily, but some pilot scale studies were also reviewed to gauge the overall efficacy of MD with regards to the treatment of highly saline feed. First, the common MD configurations used for hypersaline feed treatment will be reviewed, while identifying their drawbacks, and finally V-AGMD will be established as a potential solution by reviewing literature where it has already been assessed.

2.6.1 Common MD configurations for highly saline feed treatment

2.6.1.1 DCMD for treating saline feed

Ameen *et al.* investigated the effectiveness of the DCMD setup for treating highly concentrated brine feed (up to 200 g/L NaCl) [64]. A PTFE membrane with an average pore size of 0.22 μm , active surface area of $1.6 \times 10^{-3} \text{ m}^2$, and 78 % porosity was used in the experiments. For fixed feed salinity of 35 g/L, varying the feed flow rate and temperatures resulted in a linear increase of flux with the flow rate. At 65 °C, the flux increased from 9.2 kg/m².h to 15.5 kg/m².h when the flow rate was increased from 0.3 L/min to 1.07 L/min. This increase was attributed to better fluid mixing and decreased temperature and concentration polarisations at boundary layers. For different feed salinities but with a constant flow rate of 1.07 L/min, the feed temperature was varied from 45 °C to 65 °C. It was observed that the flux increased from 10 kg/m².h to 21.7 kg/m².h as the temperature was increased due to an increase in vapor pressure difference and hence the driving force.

As compared to deionized water, in salt solutions the vapor pressure rise is not that much because of the dissolved salts so there is a trade-off on an increase in driving force that can be obtained by increasing feed temperature. The feed concentrations were also varied to see the effect on the flux; from 0 g/L to 200 g/L NaCl at 60 °C. The feed flow rate was varied from 0.3 to 1.07 L/min. It was observed

that for a feed flow rate of 1.07 L/min, the permeate flux reduced from 17.3 kg/m².h to 13.2 kg/m².h as the feed concentration was increased from 0 g/L to 35 g/L. This reduction was more prominent when feed concentration was increased to 100 g/L and 200 g/L as the flux decreased to 10 kg/m².h and 6.8 kg/m².h, respectively. The authors reasoned that the flux decreased because of the low water activity of saline solutions and the occurrence of concentration polarization due to which the salt collected at the surface of the membrane, preventing the passage of vapors and hence increasing the mass transfer resistance [64].

Bush *et al.* carried out a study to investigate the effects of fouling or scaling on the performance of DCMD for the concentration of a hypersaline brine collected from salty water bodies [65]. A 150 µm PP membrane with pore size 0.22 µm and 70 % porosity was used. To evaluate scaling by pure NaCl, synthetic solutions of 200 g/L NaCl were used and maintained at either 50 °C or 70 °C. The concentrate with distillate was kept constantly at 30 °C until either the distillate conductivity exceeded 2 mS due to membrane wetting or the flux dropped to zero owing to scaling of the membrane surface. Both feed and distillate flow rates were maintained at 1.6 L/min. Water flux for this feed concentration was observed to be 10 L/m².h, even after several scaling events. The flux decreased to 7.5 L/m².h with a feed concentration of 330 g/L over some cycles. Below this concentration salt rejection higher than 99.9 % was observed so authors attributed this decline in flux to reduced water activity at high salt concentrations. Beyond 330 g/L scaling contributed to a rapid decrement of flux to zero [65]. When similar experiments were carried out on brine solution from a lake, the initial water flux was 8.3 L/m².h but declined by an additional 3 %, after which the flux reduced to zero in the next three hours.

Bouchrit *et al.* also investigated DCMD capability to treat hypersaline feed solutions [66]. A PVDF membrane was used with a pore size of 0.22 µm, an effective area equal to $3.2 \times 10^{-3} \text{ m}^2$ and a porosity of 62 %. The temperatures set for hot and cold streams were 59 and 20 °C with both feed and permeate velocities set to 0.046 ms⁻¹. RO brine was used as feed and when its concentration increased, the flux decreased from 8.43 to 4.06 kg m⁻² h⁻¹. From the experiments, the author concluded that the DCMD process can be employed to concentrate the solution and reach CF more than four times, and that concentration corresponded to the solution being super saturated [66].

Li *et al.* also conducted DCMD experiments to treat saline feed [67]. PTFE membrane without backing was used with an active area of $1.4 \times 10^{-3} \text{ m}^2$. The feed concentration was varied from 0 to 4 mol/L, and it was observed that when the salt concentration is higher than 2 mol/L, the permeate flux decreased. The flux dropped from 46 kg/m².h to 34 kg/m².h when the feed concentration was increased up to 2 mol/L. When it was set at 4 mol/L, the permeate flux further dropped down to 18.5 kg/m².h [67]. Table 2.4 summarizes the findings from the literature study regarding the treatment of hypersaline feed using DCMD.

Table 2.4: Summarized review of DCMD for hyper saline feed treatment

Membrane material	Membrane area (m ²)	Feed temp. (°C)	Feed flow (L.min ⁻¹)	Salinity (g/L)	Flux (Kg.m ⁻² .h ⁻¹)	Ref.
PTFE	1.6 x 10 ⁻³	45	1.07	0	10	[64]
		65			21.7	
			0.3	35		
			1.07		15.5	
		60	1.07	0	17.3	
				35	13.2	
				100	10	
				200	6.8	
PP	1.36 x 10 ⁻²	50	1.6	200	10 L.m ⁻² .h ⁻¹	[68]
				330	7.5 L.m ⁻² .h ⁻¹	
PVDF	3.2 x 10 ⁻³	59	-	143.14	4.06	[66]
PTFE w/o backing	1.4 x 10 ⁻³	60	1.07	0 mol/L	46	[67]
				2 mol/L	34	
				4 mol/L	18.5	

2.6.1.2 VMD for treating saline feed

Chang *et al.* studied VMD for desalination by investigating scaling and wettability of a commercial hollow fiber PP membrane with a pore size (0.1 µm) in comparison with that of larger pore size (0.2 µm) under vacuum conditions [69]. A membrane bundle consisting of 30 fibers with an effective area of 0.0082 m² and a length of 28 cm was used. The feed flow rate was maintained at 0.3 L/min. Three feeds were used: pure water (distilled water), 3.5 wt% sodium chloride (NaCl) solution, and brackish water with feed temperature kept at 55 °C and 60 °C. A vacuum of 3 kPa absolute was maintained in the system. The experiment was also repeated using another bundle of PP membranes consisting of four fibers with an effective area of 0.0085 m² and a length of 25 cm. The commercial membrane with 0.1 µm pore size fouled later than the 0.2 µm one but it had lower flux performance. Larger pore size led to faster surface precipitation and crystallization resulting in wetting of membrane and hence higher flux. In contrast, the membrane pores with higher surface hydrophobicity were less prone to being blocked even under the driving force due to vacuum application [69].

Karanasio *et al.* carried out experiments to study the separation of synthetic brackish water by vacuum membrane distillation employing two hollow-fibre PP membrane modules [70]; M1B with effective surface area 0.067 m² and M1A with effective surface area 0.087 m². The feed consisted of 3000 mg/L NaCl, the crossflow velocity in the modules 0.2 m/s, feed temperatures varied from 42.3–65.7 °C and

vacuum conditions being 60–130 mbar. It was observed that in module M1A, the permeate flux increased from 2.4 kg/m².h to 5.1 kg/m².h and for module M1B, the flux improved from 5 kg/m².h to 10.2 kg/m².h under vacuum conditions [70].

Safavi *et al.* investigated the separation of hypersaline feed using VMD. PP membrane with a pore size of 0.2 µm and effective area 9.1x10⁻³ m² was used. Several operational parameters were varied such as feed concentration (100, 200, and 300 g/L), feed temperature (25, 40, and 55 °C), feed and flow rate (15 and 30 mL/s), and vacuum pressure (40, 60, 80, 100 and 120 mbar) [71]. When the temperatures and feed flow rates were kept constant, it was observed that increasing vacuum decreased the permeate flux. For feed concentration of 100 g/L, as the vacuum pressure increased from 40 mbar to 120 mbar, the flux decreased from 11.2 kg/m².h to 5.7 kg/m².h. Increase in feed concentration from 100 g/L to 300 g/L, for a flow rate of 30 mL/s and constant vacuum pressure, the permeate flux decreased from 13 kg/m².h to 9.3 kg/m².h as the vapor pressure in feed got reduced. Authors attributed the decrease in flux to the formation of an extra concentration boundary layer adjacent to the temperature boundary layer which decreased the overall driving force for the process. Increasing feed temperature from 25 °C to 55 °C for feed salinity 100 g/L the flux increased from 9.5 kg/m².h to 14 kg/m².h. Conditions optimized for obtaining the highest permeate flux of 14.4 kg/m².h were feed flow rate of 30 mL/S, feed temperature of 55 °C, feed concentration of 100 g/L, and vacuum pressure of 40 mbar [71]. A summary of the literature review regarding VMD for saline feed treatment is given in Table 2.5.

Table 2.5: Summarized review of using VMD for hyper saline feed treatment

Membrane material	Membrane area (m ²)	Feed temp (°C)	Feed flow rate (Lh ⁻¹)	Salinity	Vacuum Pressure (mbar)	Flux (Kg.m ⁻² .h ⁻¹)	Ref.
PP	0.0082	54.85	18	3.5 wt% NaCl	30	0.93	[69]
		59.85				4.79	
	0.0085	54.85				1.48	
		59.85				6.12	
PP	0.067	42.3-	0.2 m/s cross flow velocity	3 g/L	60-130	2.4-5.1	[70]
	0.087	65.7				5-10.2	
PP	9.1 x 10 ⁻³	40	108	100 g/L	40	11.2	[71]
					120	5.7	
		55			40	13	
						9.3	
						9.5	
						14	
		25					
55							

2.6.1.3 AGMD for treating saline feed

To test MD for concentrating hypersaline brine, Schwantes *et al.* tested a spiral wound AGMD module with sodium chloride solutions ranging in concentration from 0 to 240 g/L by blowing low-pressure air in the air gap channel to mitigate membrane wetting. Sparged air in the air gap channel led to small reductions of 1.2 % and 4.1 % in the flux and GOR respectively but the wetting phenomena were reduced. This was attributed to the avoidance of stagnant distillate in the air gap. The authors also affirmed that with increasing brine concentration a downward trend for flux, GOR, and thermal efficiency was observed [20], [63].

Alkhudhiri *et al.* experimented to determine the effect of changing the feed concentration of different salts such as NaCl, MgCl₂, Na₂CO₃, and Na₂SO₄ for an AGMD setup [72]. Three PTFE membranes with different pore sizes were used: TF200 (0.2 µm) TF450 (0.45 µm) and TF1000 (1 µm). Throughout the study, the temperature and flow rate conditions for feed and condenser streams were maintained at [50 °C, 1.5 L/min] and [10 °C, 8.5 L/min], respectively. For the case of NaCl and TF200, when the feed concentration was increased from 5.84 g/L to 180 g/L, apart from a rise in specific energy consumption, a permeate flux reduction from 10.08 kg/m².h to 4.68 kg/m².h was observed. For membranes with bigger pore sizes and the same feed salinity of 180 g/L, the flux increased from 4.68 kg/m².h (TF200) to 6.48 kg/m².h (TF450) and 7.28 kg/m².h (TF1000) [73].

Sanmartino *et al.* conducted a study in which a 5 g/L NaCl feed was to be concentrated using AGMD setup [74]. PTFE membranes, TF200 (pore size 0.2 µm) and TF450 (pore size 0.45 µm), both with PP backing and an active surface area of $5.5 \times 10^{-3} \text{ m}^2$ were used. Condenser temperature was maintained at 25 °C, the flow rates at 100 L/h, and the thickness of feed and air gap channels were kept at 4.4 mm and 3 mm, respectively. It was observed that initially, with 65 g/L feed concentration and temperature of 82 °C, both TF200 and TF450 gave high flux of 34 kg/m².h and 47 kg/m².h, respectively. Over time, as the feed became concentrated, the flux dropped without compromising the product quality indicating that the salt rejection remains constant for both membranes. The gradual drop in permeate flux was attributed to a decrement in driving force as the feed became concentrated and increased concentration polarization and salt crystallization at the surface of both membranes. The experiment was continued even after the feed saturation level was surpassed but at a lower feed temperature of 45 °C. Permeate flux of 17 kg/m².h for TF200 and 36 kg/m².h for TF450 was observed. At all times, TF450 gave higher flux values thus it was commented that TF450 showed relatively higher flux because of greater pore size [74].

Another study was conducted by Khalifa *et al.* to investigate the treatment of saline feed using AGMD setup by varying both design and operational variables [49]. Key parameters such as the inlet-outlet temperatures, feed salinities, air gap width, and flow rates were varied. A PTFE membrane with a pore size of 0.45 µm and an active surface area of $2.29 \times 10^{-3} \text{ m}^2$ was used. The effect of feed salinity was studied by varying feed concentration from 0.075 g/L to 50 g/L against different feed temperatures. The feed and condenser flow rate were set at 3 L/min with coolant temperature at 20 °C. For feed

temperature of 40 °C flux reduced from 10 kg/m².h to 8.3 kg/m².h and at 80 °C it reduced from 66 kg/m².h to 62.7 kg/m².h. For test conditions of 3 mm wide air gap, feed and coolant temperatures of 80 °C and 20 °C, respectively, and coolant flowrate of 3 L/min, as the feed flow rate was increased from 1 L/min to 5 L/min, it was observed that the flux increased from 51 kg/m².h to 72 kg/m².h. For feed salinity of 75 mg/L, coolant temperature of 15 °C, and air gap width of 3 mm the feed temperature was increased from 40 °C to 80 °C and the permeate flux increased from 11 kg/m².h to 72 kg/m².h. This was attributed to increased driving force with the rise of temperature as MD is a temperature-driven process. The effect of reducing the mass transfer resistance across the air gap was also studied by reducing the width of the air gap from 7 mm to 3 mm at feed temperatures of 40 °C and 80 °C. The permeate flux was observed to increase from 4 kg/m².h to 9 kg/m².h and 35 kg/m².h to 70 kg/m².h, respectively [49]. A summary of the literature findings regarding AGMD for saline feed treatment is given in Table 2.6 .

Table 2.6: Summarized review of using AGMD for hypersaline feed treatment

Membrane material	Membrane area (m ²)	Feed temp (°C)	Feed flow rate (Lh ⁻¹)	Salinity	Flux (Kg.m ⁻² .h ⁻¹)	GOR	Ref.
LDPE	7.2 with 6 spiral envelopes and 1.5m channel length	80	400-1100 and velocity 2.9-8.1	0.6-5 M	2.8-6.2	2.7-1.4	[75]
	25.9 with 12 spiral envelopes and 2.7m channel length		400-1100 and velocity 1.5-4.1		0.8-2.0	6.6-4.7	
PTFE	8.3	80	300	0–240 g NaCl /kg solution	1.8	2.6	[63]
PE	24	80	500	35 g/L	1.0	6.1	[76]
	7.2				3.4	2.3	

2.6.2 V-AGMD for treating highly saline solutions

In the literature, not much has been documented whereby a V-AGMD setup was used to treat hypersaline feed. This is because process optimization under vacuum conditions is still underway. Being a variant of the AGMD configuration, vacuum-assisted AGMD was first mentioned as a patented

invention with configuration such that it allows for effective recycling of the latent heat within the system, and therefore, reducing the energy consumption [32], [77].

Andrés-Mañas *et al.* conducted a study at Plataforma Solar de Almería's solar desalination test facilities and concluded that membrane distillation using multi-envelope spiral-wound modules in V-AGMD operation is the best technology for the hypersaline treatment. The modules differed in terms of the channel length (1.5 and 2.7 m hence different effective membrane surface areas of 7.2 m² and 25.9 m², respectively). Both exhibited good performance in a wide range of feed salinities. For the shorter module, the flux doubled, and specific thermal energy consumption was halved with the V-AGMD setup but independent of the feed salinity. For the longer module, however, these key performance indicators improved with the salinity of the feed (0.6 – 5 M), especially beyond 3 M. Authors imply that using the V-AGMD module, GOR of 1.2 can be achieved with SEC lower than 515 kWhm⁻³ [75]. It was observed that with an increase of feed salt concentration, the salt rejection decreased from 99.8 % to 97.9 %. The distillate flux obtained using V-AGMD was better than that for AGMD by 31 % owing to the removal of non-condensable gases and hence decreased mass transfer resistance in the air gap. High GOR values in the range 8.5-13.5 were also observed [20].

Andrés-Mañas *et al.* also repeated the same experimental conditions as mentioned for the V-AGMD setup before but without the application of vacuum at the permeate side (hence AGMD). A comparison of the result findings is summarized in Figure 2.10. The feed temperature, flow rate, salinity, and membrane material were the same for all experiments and to compare the flux and GOR values, the average of the ranges obtained was used. It was noticed that V-AGMD outperforms AGMD in both aspects of flux and GOR. This is in line with the theoretical reasoning as stated in section 2.3.1.4.

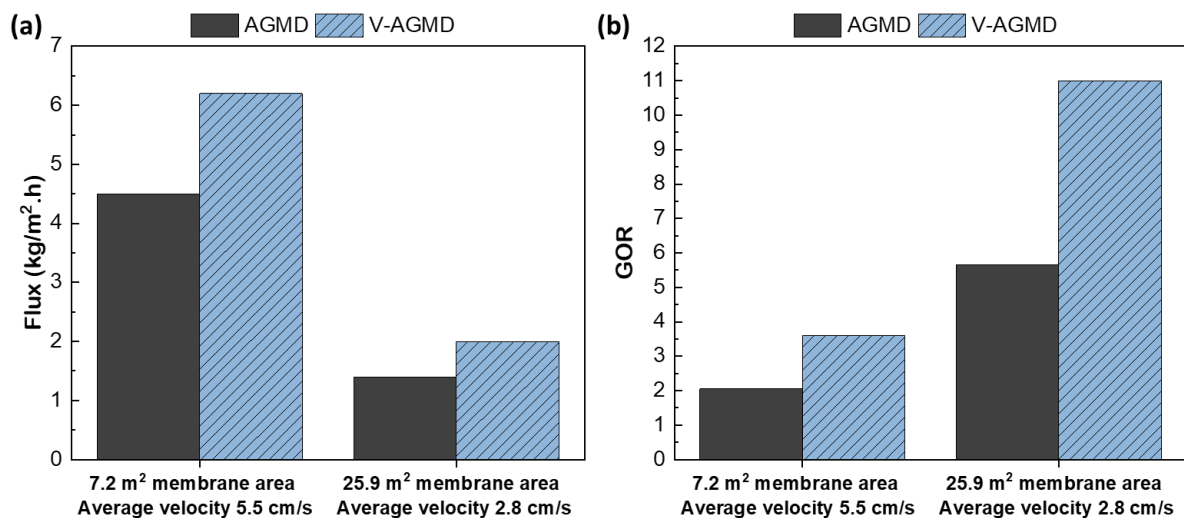


Figure 2.10: Comparison of AGMD and V-AGMD (a) Flux, (b) GOR [75]

The efficacy of vacuum-assisted AGMD was employed in an experimental study whereby a hybrid process was set up consisting of adsorption via powdered activated carbon (PAC) and V-AGMD. PAC was used to remove the surfactants in the wastewater for mitigating the wetting of the membranes used. Increment of the vacuum pressure and its relation to the effect of PAC on mitigation of membrane wetting was also studied. As predicted, higher vacuum pressures decreased the time for onset of wetting [78]. This highlighted one of the main challenges faced with regards to V-AGMD which is increased propensity of wetting with the application of higher vacuum.

Eryildiz *et al.* conducted a study on three different real wastewaters (rich in boron with a concentration range of 2500–6200 mg/L) using V-AGMD configuration. The experimental goals included evaluating the impact of parameters such as the vacuum pressure, membrane material, and pore size as well as boron concentration. The membrane material used were polypropylene (PP) and polytetrafluoroethylene (PTFE) with pore sizes of 0.20 and 0.45 μm . The main takeaway in terms of using V-AGMD was that the permeate water flux rose with increased vacuum pressure (from 0.02 to 0.08 bar) owing to mass driving force; the transmembrane steam pressure gradient [79].

A study on optimal vacuum pressure (OVP) in V-AGMD was also published in 2017, the underlying principle being that since V-AGMD is a combination of both AGMD and VMD, for AGMD to dominate, there needs to be an OVP in the airgap [80]. The experimental setup shown schematically in Figure 2.11 comprised of a double pipe AGMD module with hydrophobic PVDF hollow fiber membrane and tested with tap water.

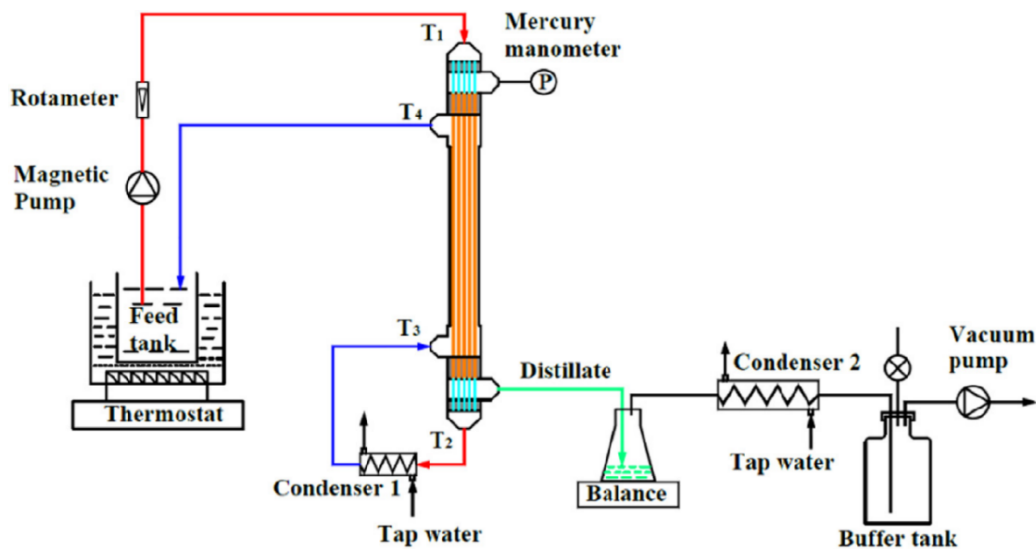


Figure 2.11: Schematic representation of the experimental setup for V-AGMD [50]

The VA-AGMD process performance was assessed by the permeate flux (J) and gained output ratio (GOR). It was found that by increasing the vacuum pressure, flux and GOR both increased but after a certain point increasing the vacuum pressure reduced the J and GOR. The authors attributed this to the

fact that as the pressure increased, so did the vapor pressure difference because of the removal of the non-condensable gases (NCGs) and according to the Antoine equation the permeate flux and GOR increased as well. Beyond a certain point though, condensation did not occur due to a rise in the vacuum pressure which led to the saturation pressure of cold feed. It was concluded that the GOR of the VA-AGMD process was enhanced by about 20–50 % as compared to AGMD, and there was no significant effect on the specific energy consumption. The condensation of water vapors ceases when the OVP value is higher than a specific value, therefore, it must be carefully selected to avoid this issue. Furthermore, based on the findings that for temperature values 70 °C, 80 °C, and 90 °C, the OVPs in the air gap could reach 0.08 MPa, 0.06 MPa, and 0.04 MPa. The authors concluded that it is imperative to select an appropriate vacuum pressure as per the operating conditions [80].

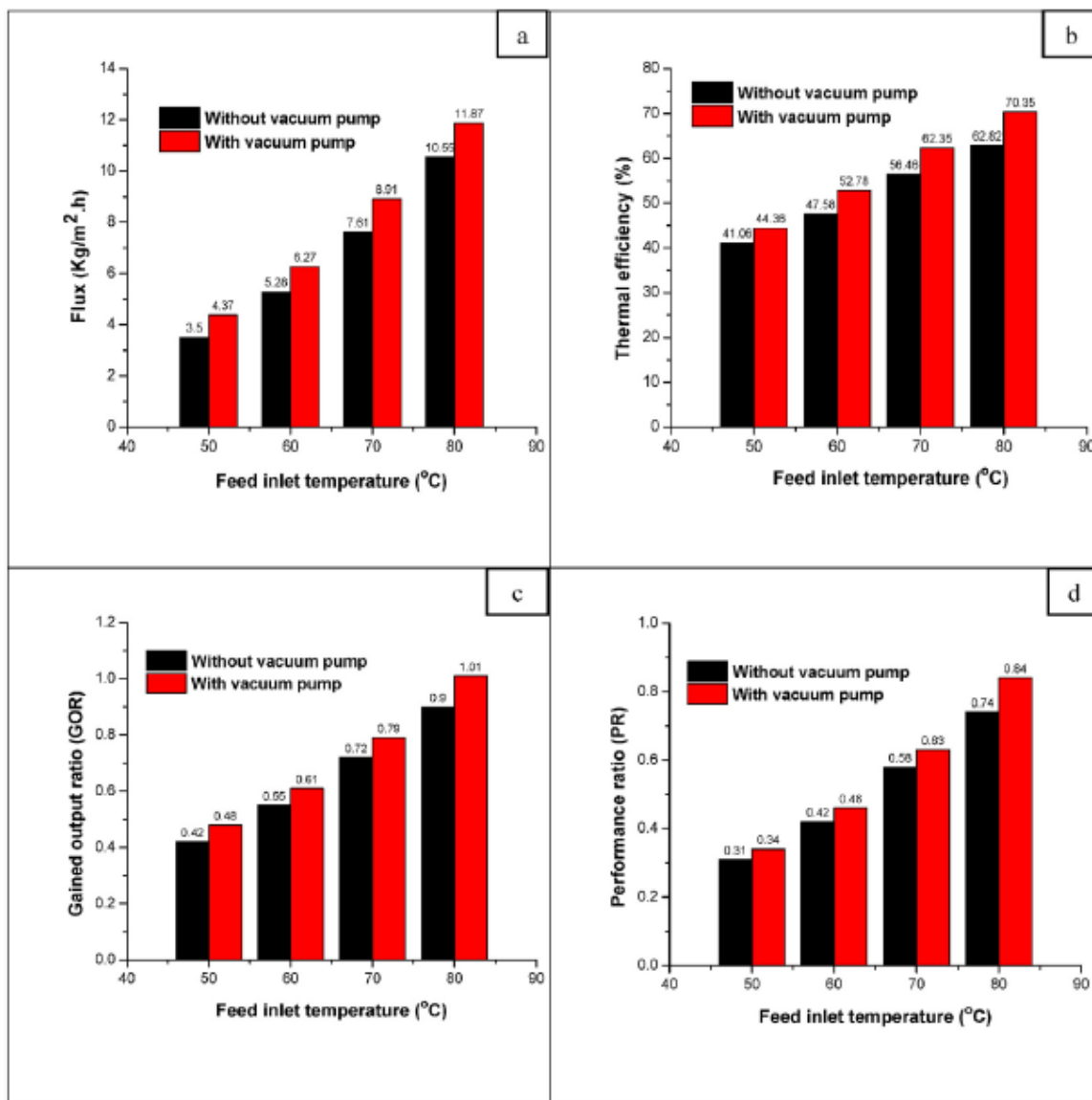


Figure 2.12: Effect of varying feed temperature on performance metrics [81]

Another study followed in which the performance of AGMD was assessed using a supplementary vacuum pump [81]. The underlying principle of the study was the understanding that the NCGs present in the air gap contribute to major mass transfer resistance [24] and that to decrease negative concentration and temperature polarization as well as water bridges effect, the NCGs should be removed by decreasing the pressure in the air gap [81], [82]. Two PTFE modules were arranged in a series configuration to minimize the requirement for heat energy and to improve the permeation flux and amount of heat recovered. In a module, two types of membranes were set alternately. The hydrophobic microporous PTFE hollow fiber membrane acted as the evaporator channel and the non-porous PTFE hollow fiber membrane served as the condensing channel. Operational parameters such as the salt concentration, flow rate and temperature of the feed, and temperature of cooling water were monitored to assess the performance of V-AGMD in comparison with AGMD [81]. The performance metrics were permeate flux (J), GOR, Thermal efficiency (η), and the performance ratio (PR).

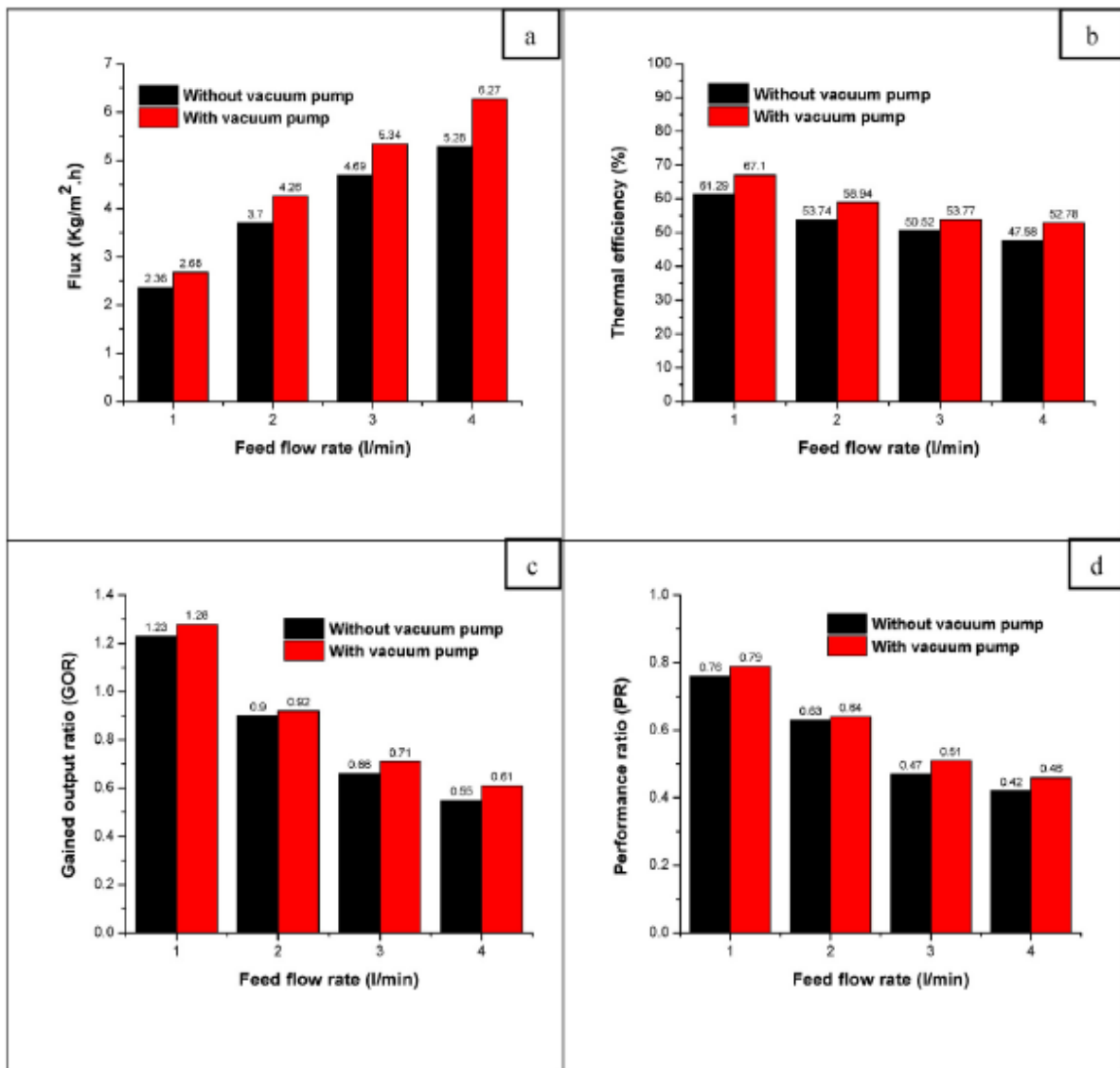


Figure 2.13: Effect of varying feed flow on performance metrics [81]

The effect of different feed temperatures with and without application of vacuum for a feed flow rate of 4 L/min and cooling water temperature of 20 °C was observed as shown in Figure 2.12. The effect of varying another independent operational parameter, different feed flow rate, with and without application of vacuum for feed inlet temperature of 80 °C and cooling water temperature of 20 °C was also observed as shown in Figure 2.13. As seen in Figure 2.12 and Figure 2.13 all the performance metrics are enhanced using the supplementary vacuum pump.

An experimental and simulation study was carried out to investigate AGMD performance under sub-atmospheric conditions by Alsaadi *et al.* Flux improvement was predicted and a comparison of the results was made with experimental conditions. Conclusively, the results depicted that upon elimination of the non-condensable gases present in the air gap, the flux improved by more than threefold when the air gap pressure was kept at saturation pressure of the feed temperature [47]. With the assistance of a mathematical model, the authors also proved that good control over the air gap pressure is possible by connecting the AGMD modules in series and operating in sub-atmospheric conditions. This is due to a reduction in feed temperature by evaporation. A summary of the literature findings regarding V-AGMD for saline feed treatment is given in Table 2.7.

Table 2.7: Summarized review on V-AGMD for saline feed treatment

Membrane material	Membrane area (m ²)	Feed temp (°C)	Feed flow rate (Lh ⁻¹)	Salinity	Vacuum Pressure (mbar)	Flux (Kg.m ⁻² .h ⁻¹)	GOR	Ref.
LDPE	7.2 with 6 spiral envelopes and 1.5 m channel length	80	400-1100 and velocity 2.9-8.1	0.6-5 M	150-200	3.7–8.7	4.6–2.5	[20], [75]
LDPE	25.9 with 12 spiral envelopes and 1.5 m channel length	80	400-1100 and velocity 1.5-4.1	0.6-5 M	150-200	1.1–2.9	13.5–8.5	
PVDF	1.2 x10 ⁻³	60	36	50 g/L NaCl	0-800	28 L/m ² .h	-	[78]

Table 2.7: Summarized review on V-AGMD for saline feed treatment (continued)

Membrane material	Membrane area (m ²)	Feed temp (°C)	Feed flow rate (Lh ⁻¹)	Salinity	Vacuum Pressure (mbar)	Flux (Kg.m ⁻² .h ⁻¹)	GOR	Ref.
PTFE	4.4 x 10 ⁻³	70	84	%1 (w/v) NaCl solution	200-800	160 L/m ² .h	-	[79]
PVDF	4.8 x 10 ⁻²	70	12	Tap Water	0-1000	Highest 8.8	-	[80]
		80				Highest 9		
		90				Highest 12		
PTFE	First through module 1 of area 3 m ² then module 2 of 2.57 m ²	60	60	253mg/L NaCl	50-100	2.68	-	[81]
			120			4.26		
			180			5.34		
			240			6.27		
PTFE	5 x 10 ⁻³	80	90	Red sea water	300	45	-	[47]

From the literature review it can be concluded that research on V-AGMD is essential to optimize the process. The feed temperature, fluid velocities and vacuum pressure employed in the literature needs further work whereupon optimum operational parameters can be finalized for high flux and resource recovery. V-AGMD strikes balance between the pros and cons of two common configurations DCMD and AGMD.

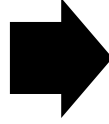
Based on the findings from literature review, this project aims to assess the effect of temperature difference (ΔT), mean temperature (T_m), flow rates and absolute air gap pressure both for tap water and saline feed.

Chapter 3: Materials and Methods

3.1 Overview of the experimental plan

The experimental work was planned in 2 stages to assess the performance of the V-AGMD process.

Running Tap Water experiments to set base line for any other feed solution for V-AGMD experiments



Using a synthetic saline feed to assess the performance for Vacuum application

First, for a set of test conditions, tap water was used as feed and several curves were obtained as a baseline to analyze the reduction in flux owing to the addition of salt in the feed. Then a synthetic feed solution was produced and tested in appropriate test conditions.

3.2 Test set-up

All experiments were carried out in MD-lab with a lab-scale test setup as shown in Figure 3.1. It was constructed by SolarSpring GmbH. The setup construction is flexible to any change in operational or design parameters. All MD configurations (with flat sheet membranes) can be accommodated within the auxiliary unit, and it can also be used to test a module, test plate, or test cell. Furthermore, the membrane, channel thickness, and spacer type can be changed too to carry out studies on heat and mass transfer, scaling/fouling potential as well as membrane aging tests and upscaling calculations. The operational parameters can also be controlled remotely via a PLC program connected over Wi-fi.

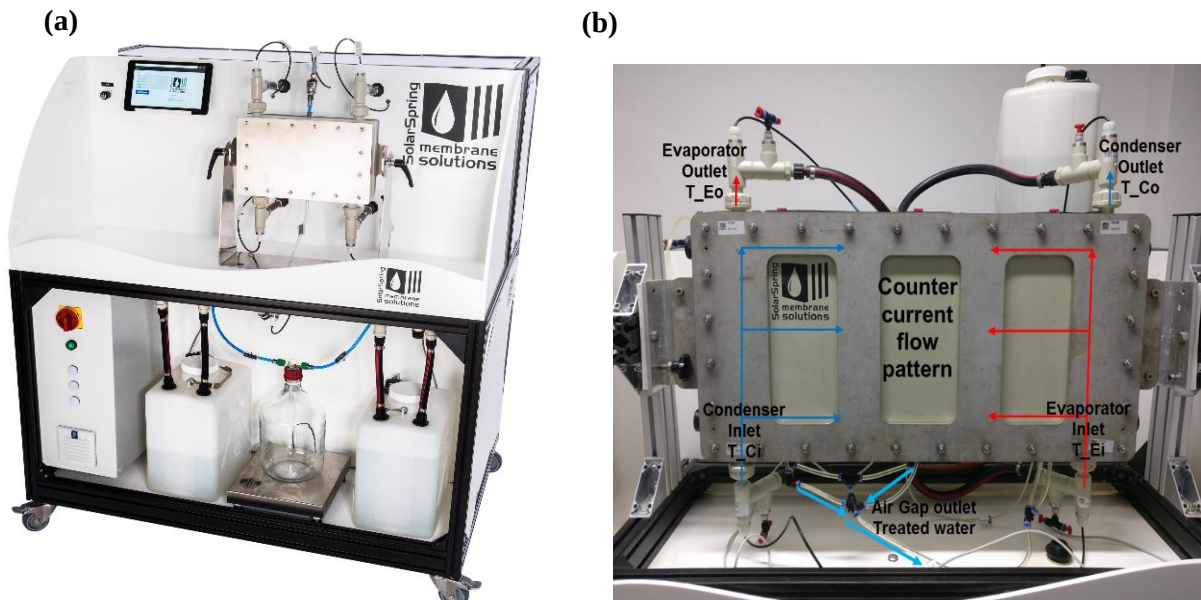


Figure 3.1: MD set-up manufactured by SolarSpring GmbH (a) test cell installed, (b) Test plate without insulation

For the first stage of the project, a test plate was used for the experiments. For the second stage, to control the vacuum better and productively utilize the time, experiments were continued in a test cell. Size specifications of both test modules are given in Table 3.1.

Table 3.1: Specifications of flat sheet testing modules

Test module	Dimensions (m)	Membrane surface area (m ²)
Plate	0.67 x 0.31	0.2077
Cell	0.25 x 0.15	0.0375

Both the test modules consist of 30 mm inner and outer frames made of PP. These frames hold together the inlets and outlets of the hot and cold loop as well as for the product. The evaporator and condenser channel thickness was 2.15 mm. Both channels were created using a 2.15 mm thick Tenax T2 spacer to facilitate transport by providing turbulence to the flow. Air gap thickness was changed using a spacer of two kinds as per the requirement of the two stages in the experimental plan. The purpose of using spacers with suitable geometry and material was not only to maintain the channel width but also to ensure appropriate crossflow velocity of the fluids to minimize concentration and temperature polarization effect. A 100 μ m thick PP film was used to separate the air gap channel from the condenser channel. The membrane used for stages 1 and 2 was manufactured by W.L. Gore & associates and Donaldson filtration solutions Deutschland GmbH, respectively. The properties of both membranes will be discussed in section 0. In both stages, the fluid flow pattern for the test plate and test cell was counter current as depicted in Figure 3.1 (b).

The V-AGMD set-up was done in the following steps. A pictorial representation of the test assembly is given in Figure 3.2.

- i. The evaporator channel is constructed. PP sealing is placed through screws onto the PP plate followed by the Tenax T2 spacer.
- ii. The membrane is placed onto the evaporator channel with the backing facing the condenser side as per literature findings.
- iii. The air gap sealing comes on top of the membrane followed by the spacer to create a channel of desired thickness (different for both experimental stages).
- iv. The film is placed followed by the sealing and spacer for the condenser channel. The PP plate for the cool side is then placed on top.
- v. The inner and outer frames are provided with stainless steel plates of 10 mm thickness in front and back for mechanical support. These stainless-steel plates are locked using 28 sets of nuts and bolts with a torque of 10 Nm, starting from the centre of the test plate, and going towards the edges in a crisscross fashion, ensuring that the membrane and sealing gaskets spread uniformly to ensure leak-proof sealing.

- vi. All the inlet and outlet hoses of heating, cooling and feed loops are connected to their respective inlets and outlets on the PP plates.
- vii. The distillate can be collected from 6 ports available at the bottom of PP plates of the test plate and 1 port at the bottom of the test cell. The unused inlets and outlets, if any, are sealed.

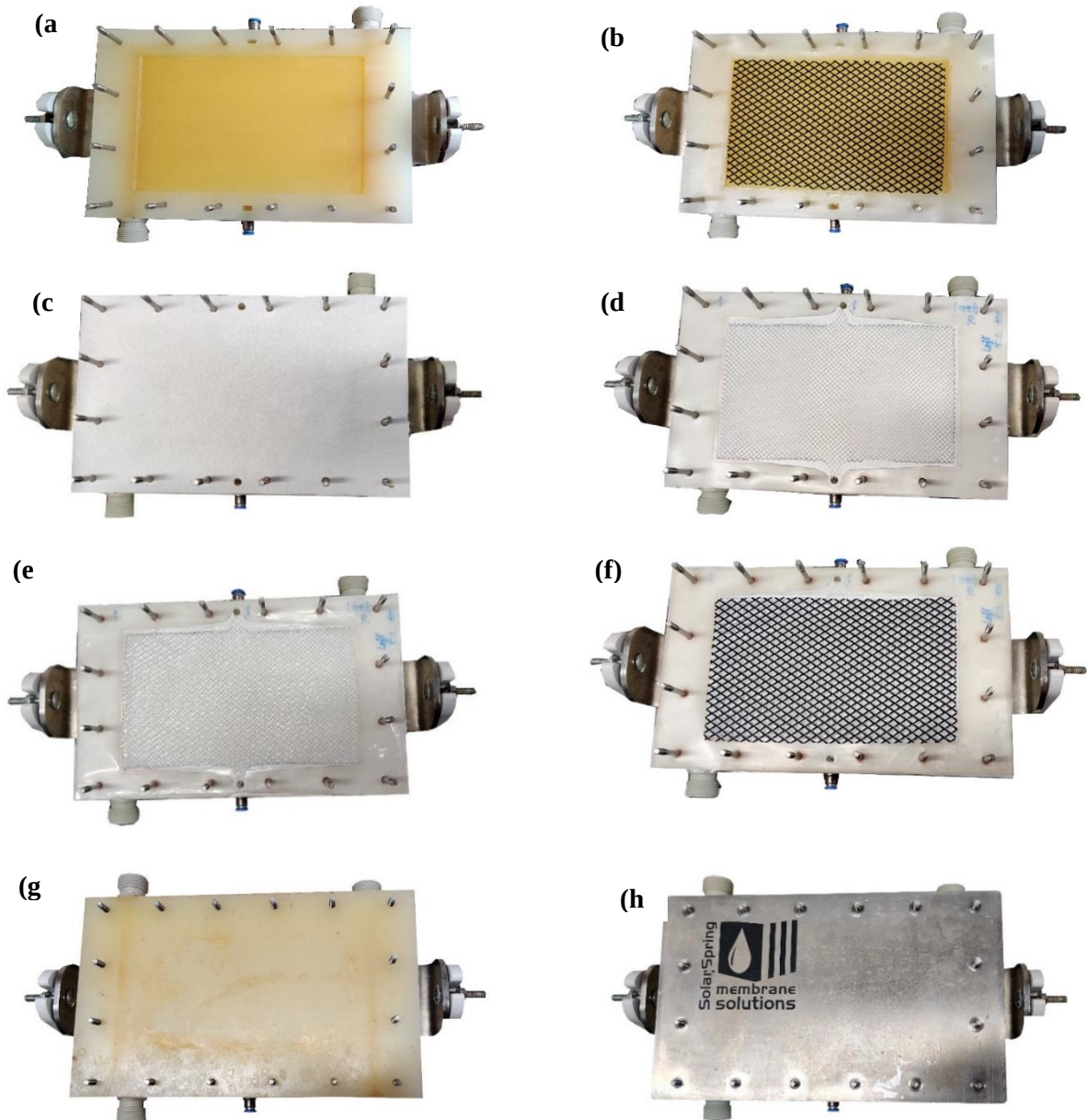


Figure 3.2: Test cell assembly for V-AGMD setup

Figure 3.3 depicts the V-AGMD experimental setup. Both test plate and test cell are connected in separate hydraulic loops for heating and cooling each with a centrifugal pump to maintain the flow rate of the feed-in heating circuit and tap water in the cooling circuit. Heat exchangers in both loops heat and cool the fluids in circuits and PT100 class A temperature sensor monitors the temperatures of all

inlets and outlets of the setup. The temperature of the evaporator inlet (T_{ei}) and condenser outlet (T_{co}) are set in the software to control the ΔT during the experiments. Conductivity meter for both distillate and feed measures the conductivity of both fluids. The product outlet is connected to a 20 L vessel which also has a pipe connection from a vacuum pump. A pressure sensor was connected to the product outlet using a T connector and that enabled monitoring of the vacuum pressure in the setup. The vessel which collects the flux is placed on a weight balance that is connected to the PLC. Table 3.2 contains technical details of sensors and equipment used in the setup.

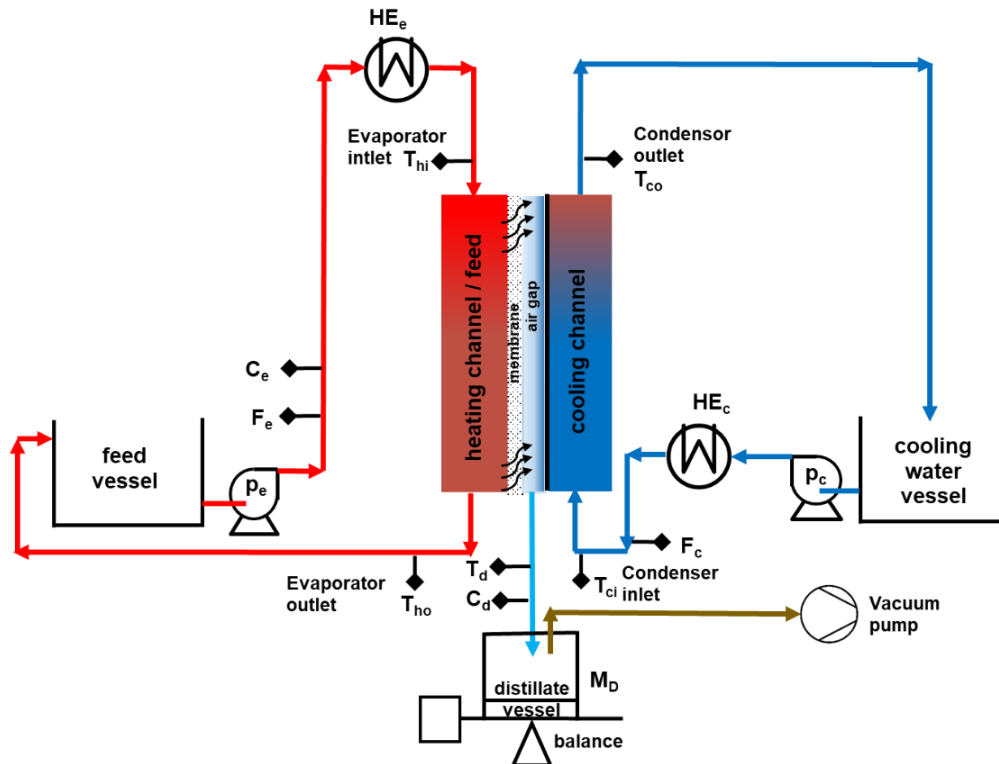


Figure 3.3: Schematic of V-AGMD set up in MD lab

3.3 Material selection and test conditions

From the literature review, one of the major findings was that MD performance is influenced by the operational parameters as well as the selection of materials for experimental usage. To assess the effect of vacuum on MD, it was imperative to ensure that any other factor did not undermine the process parameters.

3.3.1 Material selection

For the first and second experimental stage, the membrane used was provided by W.L. Gore & associates and Donaldson filtration solutions Deutschland GmbH, respectively. Summary of membranes used is given in Table 3.3.

Table 3.2: Details of sensors and equipment in MD lab

Component	Producer	Type	Accuracy	Name in schematics
Conductivity meters	Bürkert GmbH, Ingelfingen, Germany	8228	$\pm 2\%$ of measured value, range (100 $\mu\text{S/cm}$ -2 S/cm)	C_h
Conductivity meter distillate	Jumo, Fulda, Germany	BlackLine CR-EC	$\leq 2\%$ of measuring range (0-2000 $\mu\text{S/cm}$)	C_d
Temperature sensors	TC direkt, Mönchengladbach, Germany	Pt100 Klasse A	$\pm (0.15 + 0.002 \cdot t)$	All the temperature sensors
Volume flow	Krohne, Duisburg, Germany	Optiflux 4050C / IFC050C	0.5 % of the measured value $\pm 1\text{ mm/s}$	F_h, F_c
Heating and cooling pumps	Iwaki, Saitama, Japan	RD40 24VDC	-	P_e, P_c
Vacuum Pump	KNF	N 938.50.KT.18	-	Vacuum Pump
Balance distillate	Soehnle, Backnang, Germany	2890.04.040	1 gram for 0-32 kg	M_d
Heat exchangers	Kelvion, Nobitz-Wilchwitz, Germany	GNS220H-8-XCR	-	HE_h, HE_c
Cooling unit	Huber, Offenburg, Germany	Unichiller 025-H OLÉ	$\pm 0.2\text{ }^\circ\text{C}$	-
Feed temperature controller	Tool-temp, Sulgen, Switzerland	MP-888	-	-

Table 3.3: Properties of membranes used in the experiments

Manufacturer	Membrane thickness (μm)	Backing thickness (μm)	Pore size (μm)	Porosity (%)
W.L. Gore & associates	76	280	0.2	80
Donaldson filtration solution	70	184	0.1	84.4

Owing to previous work done at SolarSpring GmbH, it had already been optimized that the Tenax T2 spacer works best to provide appropriate turbulence and least mass transfer resistance for the evaporator and condenser channel. Similarly, experiments had been done by previous interns using Conwed and Intermaas spacer. Building on previous results, they were chosen because of their positive influence on MD performance. Conwed spacer was used for stage 1 and Intermaas for stage 2. A summary of the spacer properties is given in Table 3.4.

Table 3.4: Properties of spacers used in the experiments

Manufacturer	Reference no.	Material	Thickness (mm)	Mesh size (mm)	Mesh arrangement	Angle of attack
Intermas	6246	PP	1.15	3*3 & 4*4	Diamond	90°
Conwed	XOC530	HDPE/PP	0.80	2.8*2.8	-	90°
Tenax	T2	-	2-2.3	-	-	-

For the second experimental stage, 40.5 kg of a synthetic saline feed solution was made, the composition of which was similar to the leachate of a potassium salt mine. Salts were acquired from Carl Roth GmbH. Table 3.5 shows the salts used and their quantities by weight.

Table 3.5: Synthetic feed solution composition for experimental stage 2

Salt	MgCl ₂ .6H ₂ O	MgSO ₄ .7H ₂ O	KCl	NaCl	CaCl ₂	Water
Weight (g)	13723.5	3027	2338.0	2421.4	2.8	18987.8

3.3.2 Test Conditions

For the first stage, experiments were run using tap water to set a baseline for the next experimental stage. In it, to test the effect of mean temperature (T_m), a difference of temperature (ΔT) between the incoming feed fluid ($T_{\text{Evap in}}$) and condenser fluid going out ($T_{\text{Cond out}}$), vacuum pressure, and fluid flow velocities in evaporator and condenser channels on the flux and thermal efficiency of the setup, several experiments were carried out. While one parameter was assessed, all others were kept constant. For the second stage, the effect of all these parameters was assessed again and the initial conductivity of the feed was 168 $\mu\text{S/cm}$. Table 3.6 summarizes the test conditions for both stages. At the start of the experiments, all the connections were checked for leakages, and then the setup was insulated using Styrofoam paddings. Depending on the change in temperature or flow rate, each experiment point was run for at least four hours to ensure that all the operational parameters had attained a stable condition. In the case of saltwater experiments, when the product conductivity went beyond 2 mS/cm, the membrane was considered wetted and then replaced with a new one.

Table 3.6: Summary of the experimental conditions

Parameter under assessment	Constant parameter	Variation values	Unit
Temperature difference (ΔT)	Flowrate, T_m , Feed and Vacuum Pressure	Stage 1: 8, 16 Stage 2: 12-28	$^{\circ}\text{C}$
Mean temperature (T_m)	Flowrate, ΔT , Feed and Vacuum Pressure	Stage 1: 40, 60, 68 Stage 2: 50, 60, 68	$^{\circ}\text{C}$
Fluid flow velocity (m/s)	ΔT , T_m , Feed and Vacuum Pressure	Stage 1: 0.05-0.11 Stage 2: 0.07, 0.09	m/s
Air gap pressure (mbar)	Flowrate, T_m , Feed, and ΔT	Stage 1: 200, 500, 750, 1000 Stage 2: 300-600 and 1000	mbar

3.4 Data Acquisition and Handling

Both the test plate and test cell are connected to a PLC computer. All operating parameters (in our case T_{Evapin} and T_{Condout}), vacuum pressure, flow rates for evaporator and condenser, etc. can be easily changed from this computer installed in the MD lab and is connected to the company server via Wi-Fi. Data set points can be changed from the or also remotely from the office or at home. As the sensors take in the changing values throughout the experiment, the real-time output values from all sensors are displayed on the screen and the computer records the readings every 30 seconds. In the settings page, the shutdown of the system can be controlled in case of high pressure, high conductivity, over-heating, empty feed, or condenser tank as well as in case the distillate vessel fills beyond the set point. A record of all readings is downloaded from the computer in the form of a '.csv' data file which is then imported into a template file made in MS Excel. The template file is used to plot graphs of desired parameters over a selected time. From the graph, once the parameters have stabilized and the flux is constant, an average is calculated for the flux, distillate output, temperatures, flow rates, vacuum pressure, and conductivity of feed and distillate. This procedure is repeated for all the points and then fed into a results file where the data is analyzed.

3.5 Calculation methodology

3.5.1 Flux

The flux is calculated using the formula:

$$J = \frac{M_d}{A_m t} \quad (3.1)$$

where J is the permeate flux in $\text{kg/m}^2\cdot\text{h}$, M_d is the weight of the distillate collected, A_m is the effective surface area of the membrane and t is time in seconds.

3.5.2 Thermal efficiency

The thermal efficiency is calculated using the formula:

$$\eta_{th} = \frac{\text{Latent heat transferred } (\dot{Q}_l)}{\text{Total heat transferred } (\dot{Q}_{trans})} \times 100 \quad (3.2)$$

For this project, a template (Figure 7.1) was used to calculate thermal efficiency values by inputting the required values of hourly distillate, flow rates, and temperatures of hot and cold loops. This template was already made as part of another project and is currently in use by the company. Detailed analysis of this template is out of scope of this project. Nonetheless, Table 3.7 contains the formulas from the template used for the calculation of quantities which are further used to find thermal efficiency. Here H_{Ei} is the heat in the evaporator inlet, H_{Eo} is the heat in the evaporator outlet, and Q_{lossE} accounts for heat losses.

Table 3.7: Formulas used for thermal efficiency calculations in the template

$Q_l(W) = \frac{\text{Hourly distillate flow}}{1000 \times 3600} \times \Delta H_{vap}$	(3.3)
$Q_{trans}(W) = H_{Ei} - H_{Eo} - Q_{lossE}$	(3.4)
$H_{Ei}(W) = \dot{m}_e \times c_p \times T_{ei}$	(3.5)
$H_{Eo}(W) = (\dot{m}_e - \dot{m}_{dist}) \times c_p \times T_{eo}$	(3.6)
$Q_{lossE}(W) = \left(\frac{TR}{1 + TR} \right) \times Q_{loss,total}$	(3.7)
$Temperature\ Ration\ (TR) = \frac{\left(\frac{T_{Ei} + T_{Eo}}{2} \right)}{\left(\frac{T_{Ci} + T_{Co}}{2} \right)}$	(3.8)
$Q_{loss,total}(W) = H_{Ci} + H_{Ei} - H_{Co} - H_{Eo} - H_{distout}$	(3.9)
$H_{Ci}(W) = \dot{m}_c \times c_p \times T_{Ci}$	(3.10)
$H_{Co}(W) = \dot{m}_c \times c_p \times T_{Co}$	(3.11)
$H_{distout}(W) = \dot{m}_{distil} \times c_p \times T_m$	(3.12)
$T_m(^{\circ}C) = \frac{T_{Ci} + T_{Co} + T_{Ei} + T_{Eo}}{4}$	(3.13)

Chapter 4: Results and Discussion

The experiments were done in two testing facilities; a so-called test plate and a so-called test cell which are different in terms of their dimensions. Section 4.1.1 discusses how and why the two testing modules were compared before continuing the experiments in the test cell. Section 4.1.2 first presents an analysis of the influence of the characteristic operational parameters, namely the mean temperature, the flow velocity, and the temperature difference for specific vacuum pressure using tap water, and then how these parameters vary for different vacuum pressures. Section 4.1.3 presents a similar analysis for using hypersaline feed solution and discusses the effect of spacer thickness and feed conductivity. For both tap water and saline feed, performance is also compared with ambient pressure in the air gap (AGMD setup). For the setup used, it was determined that running one experimental point for minimum 4 and maximum 6 hours led to a stationary output. Running one point for this long a time, prevented need for repetition or statistical comparison and any deviation obtained is attributed to associated instrumental error. As observed in Table 3.2, each component in the setup has an associated instrumental error. It ranges from a minimum of 0.5% of the measured value for the volume flow meters and maximum of $\pm 0.2^{\circ}\text{C}$ for the cooling unit. Cumulatively, the error propagation results in deviations within one experimental point that is run. Since that is common for all experimental points, the use of error bars in the plots is avoided.

4.1.1 Lab study to compare the performance of two V-AGMD modules under vacuum conditions

The first stage of the experiments was carried out in a test plate module (TP) but after several experiments, an issue of vacuum loss arose so it was decided to continue the experiments in a smaller test cell (TC), which has a different method of sealing. To make the results comparable, it was important to ensure that the rate at which heat energy is introduced in both test plate and test cell was similar. For comparability, the results were normalized via flow velocity and compared as depicted in Comparison of fluid velocity in Test plate and Test Cell.

Table 4.1: Comparison of fluid velocity in Test plate and Test Cell

Fluid flow rate (L/h)	Velocity in TP (m/s)	Velocity in TC (m/s)
70	0.036467	0.075366
100	0.052096	0.107666
150	0.078145	0.161499
200	0.104193	0.215332

It was found that there was a 3.5 % difference between fluid velocity for a flow rate of 70 L/h in the test cell and 150 L/h in the test plate and a 3.3 % difference between fluid velocity for a flow rate of 100 L/h in the test cell and 200 L/h in the test plate. Thus, a flow rate of 70 L/h and 100 L/h for the test cell experiments was chosen while for the velocity of 0.05 m/s, extrapolation of data was done to find corresponding flux and thermal efficiency values. In the next step, 12 experiments that were already done in the test plate were repeated in the test cell using the same conditions for mean temperature (T_m), ΔT , air gap pressure (abs), and feed velocity. Using the results already obtained in the test plate, flux values were extrapolated and interpolated for the actual T_m obtained in the test cell for each experiment and compared with the flux obtained in the test cell. Figure 4.1 shows the results and Table 7.2 in the appendix shows the detailed experimental conditions for the experiments carried out. It was observed that the flux values obtained with TC and TP are within $\pm 5\%$ of each other. As a result, it was concluded to carry out the rest of the experiments in the test cell.

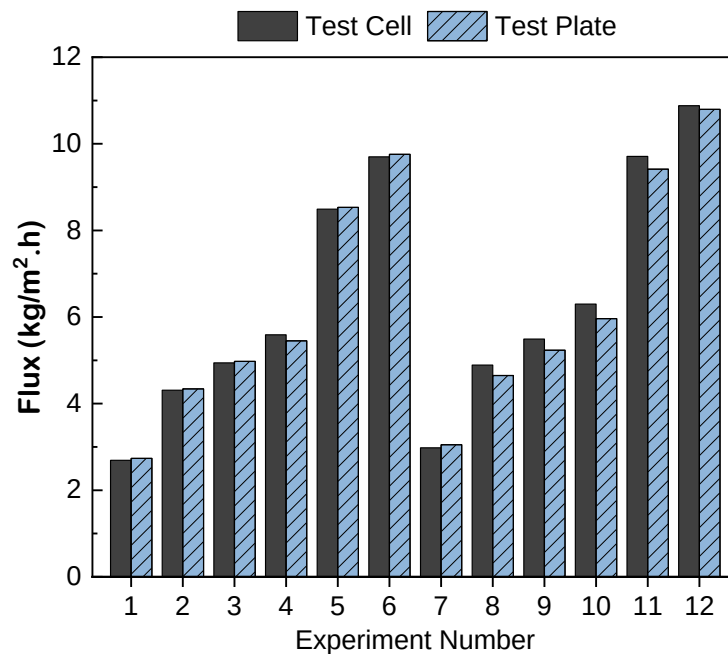


Figure 4.1: Comparison of flux between TP and TC V-AGMD setup under air gap pressure of 500 mbar (abs) using tap water as feed

4.1.2 Performance characterization of V-AGMD setup with tap water

4.1.2.1 Influence of flow velocity in the hot and cold channel for a specific air gap pressure

The velocity of feed in the evaporator channel and the cooling fluid in the condenser channel affect the performance of the V-AGMD setup. The temperature of these fluids maintains the ΔT across the membrane so the velocity in the hot loop (evaporator inlet and condenser outlet) plays a significant role in the amount of distillate collected per unit time per unit area of the membrane (flux). Velocity in the hot loop also impacts the turbulence in the flowing fluid. Same flow rate values were input for both

hold and cold channels in the setup. Figure 4.2 (a) shows the impact of fluid flow velocity on the flux. It can be observed that as the flow velocity increases, so does the flux. This positive influence is observed for both ΔT 8 °C and ΔT 16 °C. For ΔT 16 °C, the flux increases from 6.95 kg/m²·h to 8.49 kg/m²·h as the flow velocity increases 0.05 m/s to 0.075 m/s and the highest distillate flux of 9.71 kg/m²·h is obtained for the highest flow velocity of 0.11 m/s. A similar trend is observed for ΔT 8 °C experiment. As the velocity increases the Reynolds number increases too which implies relatively high turbulence for the fluid, Furthermore with increase in velocity, additional heat energy is introduced into the system at a faster rate which increases both the bulk and interfacial temperature difference across the membrane surface. This increase in temperature elevates the driving force as the vapor pressure difference increases and hence the rate of evaporation at the membrane feed interface is higher (refer to Antoine Eq. (2.9)). Resultantly, the distillate flux also increases.

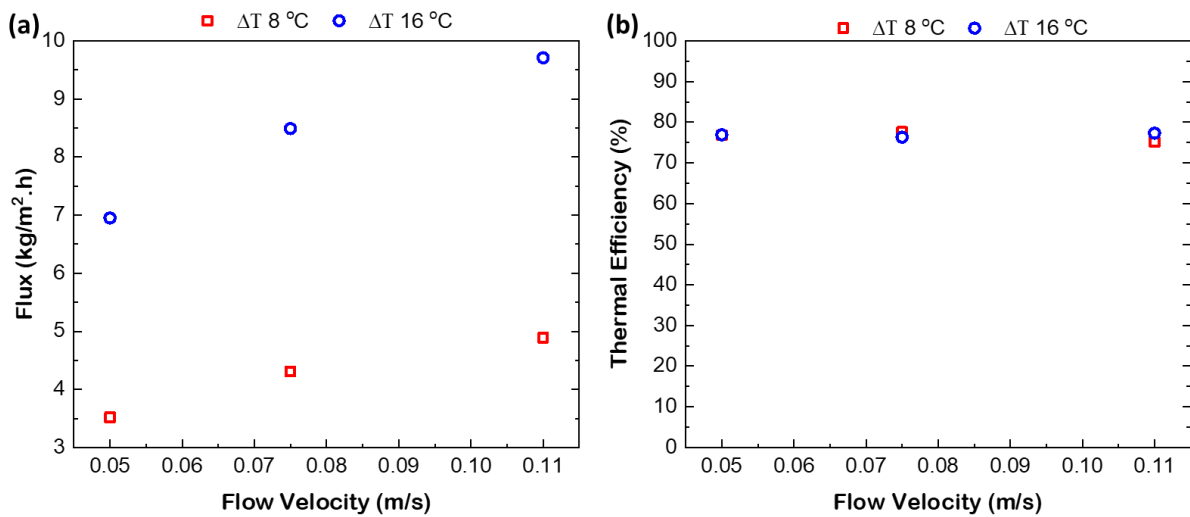


Figure 4.2: Influence of varying flow velocity on (a) distillate flux, and (b) thermal efficiency for different ΔT at T_m 60 °C and air gap pressure 500 mbar (abs) using tap water as feed

Figure 4.2 (b) depicts the impact of changing flow velocity for different ΔT at 500 mbar absolute air gap pressure and T_m 60 °C on the thermal efficiency (η_{th}) of the system. As explained in section 3.5.2 the thermal efficiency is the ratio of latent heat of vaporization to the total heat which is transferred across the membrane. In Figure 4.2 (b) it can be observed that the thermal efficiency is approximately 76 % for all experimental points with minor deviation due to instrumental error of temperature sensor at velocity 0.11 m/s. This independence of thermal efficiency of the flow velocity can be explained in this context that with an increase in velocity and hence energy supply, the latent heat available or vaporization also increases. Thus, as the distillate flux increases proportionally to the increased heat influx into the system, the ratio of latent heat to total heat transferred through the membrane remains constant. An increase in flux owing to high flow velocity does not impact the thermal efficiency.

4.1.2.2 Influence of Mean Temperature (T_m) for a specific air gap pressure

The mean temperature obtained in the setup is calculated according to Eq. (3.13). This mean temperature is controlled by inputting temperature values for the evaporator inlet (hot stream inlet) and condenser outlet (cold stream outlet) using the PI controller as described in section 3.2. Figure 4.3 (a) depicts how flux varies with increasing mean temperature at two ΔT values of 8 °C and 16 °C at absolute air gap pressure of 500 mbar and flow velocity in both hot inlet and cold outlet set as 0.075 m/s. From the results, it can be observed that for ΔT 16 °C, as the mean temperature increases from 38.8 °C to 58.5 °C, the flux increases from 5.59 kg/m²·h to 8.49 kg/m²·h and the highest flux of 9.7 kg/m²·h is obtained at mean temperature 66.3 °C. A similar positive influence of mean temperature on flux is observed for ΔT 8 °C. This surge in distillate can be understood from the resultantly increasing vapor pressure difference across the membrane at higher mean temperatures. Antoine's Eq. (2.9) shows this exponential relationship between temperature and vapor pressure and Figure 2.11 graphically shows the relation that as temperature increases, so does the vapor pressure. It can also be deduced that the increase in vapor pressure is more significant at higher temperatures. Hence the flux values at both ΔT 8 °C and ΔT 16 °C are low at low mean temperatures of around 40 °C and 60 °C as compared to T_m of 68 °C.

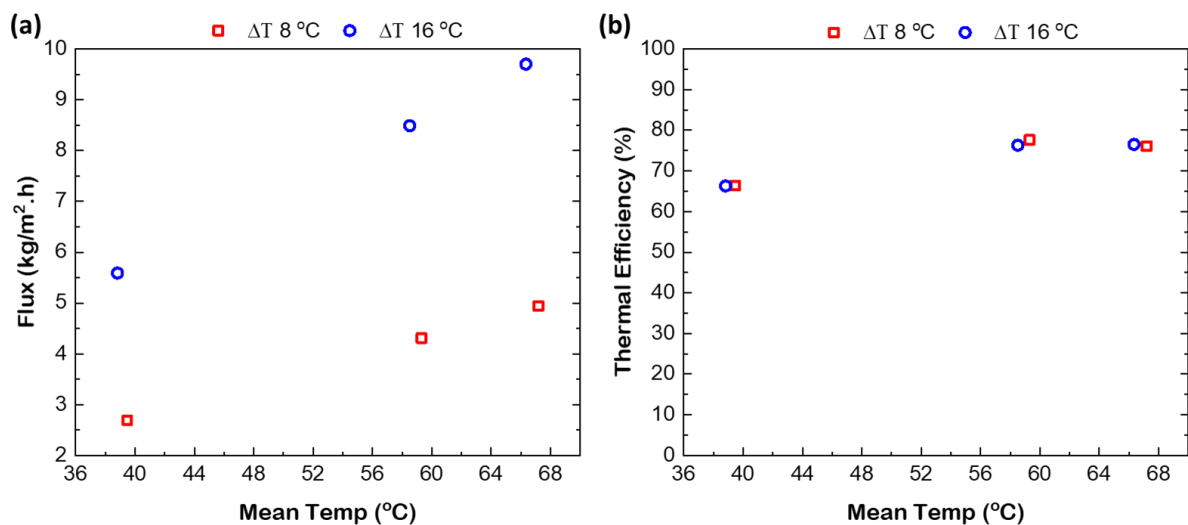


Figure 4.3: Influence of varying mean temperature on (a) distillate flux, and (b) thermal efficiency for different ΔT at flow velocity 0.075 m/s and air gap pressure 500 mbar (abs) using tap water as feed

Figure 4.3 (b) shows the influence of T_m on thermal efficiency. For ΔT 16 °C, the thermal efficiency increases from 66.3 % to 76.5 % as the T_m rises from 38.8 °C to 66.3 °C. As the mean temperature increases, so does the vapor pressure difference between heating and cooling streams, therefore, the fraction of latent heat transfer increases which results in increased thermal efficiency.

4.1.2.3 Influence of temperature difference between hot and cold channels (ΔT) for a specific air gap pressure

The effect of changing ΔT on permeate flux is observed in Figure 4.4 (a) for a mean temperature of approximately 60 °C, the absolute air gap pressure of 500 mbar, and at different flow velocities. A general trend of increase in flux with an increase in ΔT is observed for all flow velocities. For flow velocity of 0.11 m/s, flux increases from 4.89 kg/m²·h to 9.71 kg/m²·h as the ΔT increases from 8 °C to 16 °C. For a fixed mean temperature, a larger ΔT means a greater difference between the heating and cooling streams. This difference translates to a difference in vapor pressure. For example, the temperature conditions for hot in and cold out at T_m 60 for ΔT 8 °C are 64 °C and 56 °C, respectively, while for ΔT 16 °C they are 68 °C and 52 °C, respectively. As the driving force for MD is the vapor pressure difference between the heating and cooling channels, with the rise in ΔT , the vapor pressure difference between both the streams rises and hence leads to higher distillate flux. This variation of vapor pressure can be graphically seen in Figure 2.11 where it is observed that for the same T_m , a larger ΔT generates a higher difference of vapor pressure. A larger ΔT also implies a higher mass transfer coefficient across the membrane and the airgap which contributes to higher flux as backed by equations (7.2) and (7.3) in section 7.4 of the appendix.

Figure 4.4 (b) shows that for different ΔT values, the thermal efficiency remains almost similar around 77 % for both the values of ΔT . This can be attributed to the increase in permeate flux which is in proportion to the increase in temperature difference between the heating and cooling streams.

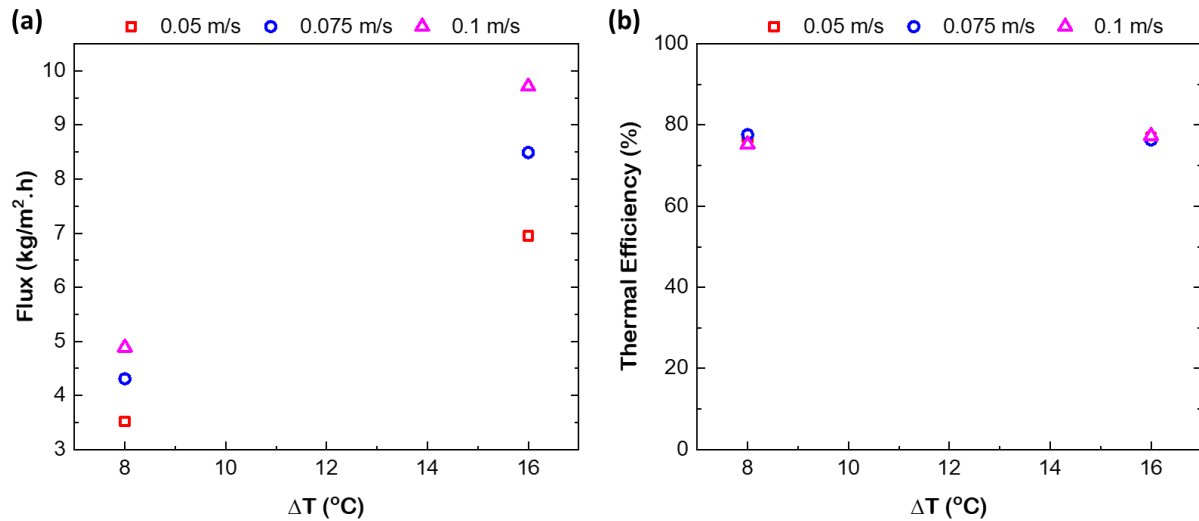


Figure 4.4: Influence of varying ΔT on (a) flux, and (b) thermal efficiency for different flow velocities at T_m 60 °C and air gap pressure 500 mbar (abs) using tapwater as feed

4.1.2.4 Influence of varying air gap pressure

After carrying out experiments at a specific air gap pressure, the results of experiments carried at the same operational conditions, but different air gap pressures (absolute) were carried out. As the absolute air gap pressure is decreased, vacuum in it increases. Figure 4.5 depicts the influence of varying the air gap pressure in the V-AGMD setup with ΔT 16 °C and flow velocity of 0.075 m/s. A general trend of flux increment with decreasing air gap pressure is observed for all mean temperatures of around 40 °C, 60 °C, and 68 °C. For a T_m of 68 °C, the flux increased from 8.8 kg/m²·h to 9.7 kg/m²·h as the absolute air gap pressure decreased from 750 mbar to 500 mbar. The highest flux of 10.90 kg/m²·h was obtained at an air gap pressure of 200 mbar. The effect of T_m is also clearly visible as the flux for T_m 40 °C is lower than flux obtained at any air gap pressure for T_m 60 °C and T_m 68 °C. This positive influence of decreasing the air gap pressure on flux is also seen in the consolidated summary plot in Figure 4.6. As air gap pressure reduces, the amount of NCGs decreases which implies there is less mass transfer resistance so flux increases. This result is also backed by findings in the literature. In a Ph.D. thesis study done by Daniel Winter [9], model development was done to understand combined mass transfer across membrane and airgap. The equation representing this is given by Eq. (7.1) in section 7.4 of the appendix. As seen in this equation, as the air partial pressure decreases and water vapor partial pressure increases, the difference of total pressure increases which leads to a higher molecular diffusion coefficient and hence higher flux. Furthermore, by decreasing pressure, the evaporation rate is enhanced which translates to higher flux values.

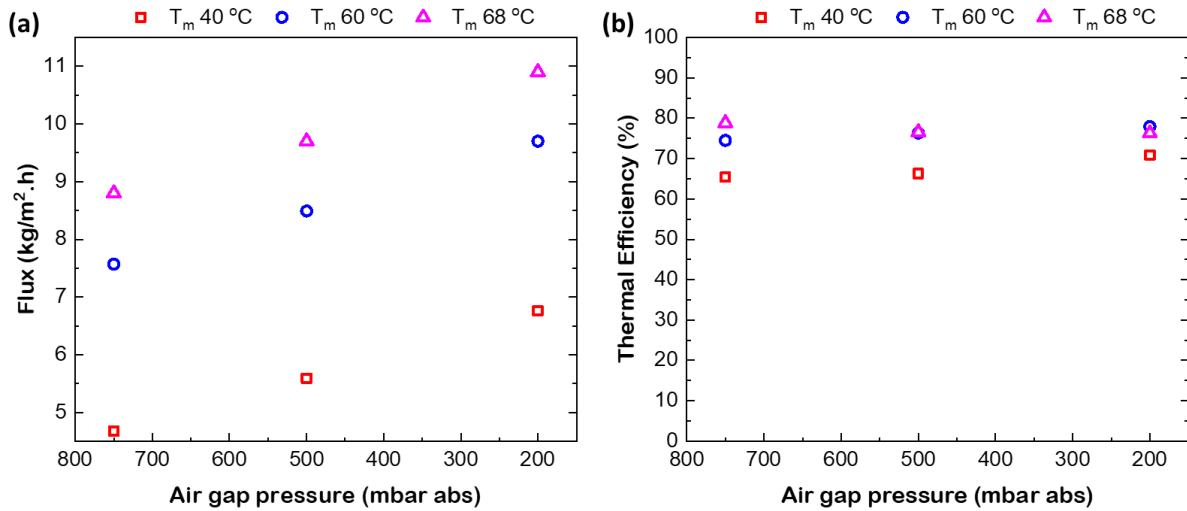


Figure 4.5: Influence of varying air gap pressure on (a) flux, and (b) thermal efficiency for different T_m at ΔT 16 °C and flow velocity 0.075 m/s using tap water as feed

Figure 4.5 (b) and the consolidated summary plot in Figure 4.7 show the effect of varying air gap pressure on the thermal efficiency of the system concerning different mean temperatures. As the air gap

pressure is decreased, it is observed that the thermal efficiency improves for the same T_m . This is because the removal of air decreases the conductive heat transfer which at the same time enhances the latent heat transfer. It implies that the ratio of latent heat to total heat transferred does not remain constant. It increases as the air gap pressure is reduced.

This result is further explained by a literature finding. Eq. (7.7) in section 7.5 of the appendix is obtained from a Ph.D. thesis study by Daniel Winter [9]. It depicts that the total heat transferred consists of latent heat transfer, conductive heat transfer, and radiation exchange across the airgap. The equation also shows that the latent heat transfer is directly proportional to the total flux across the membrane and air gap.

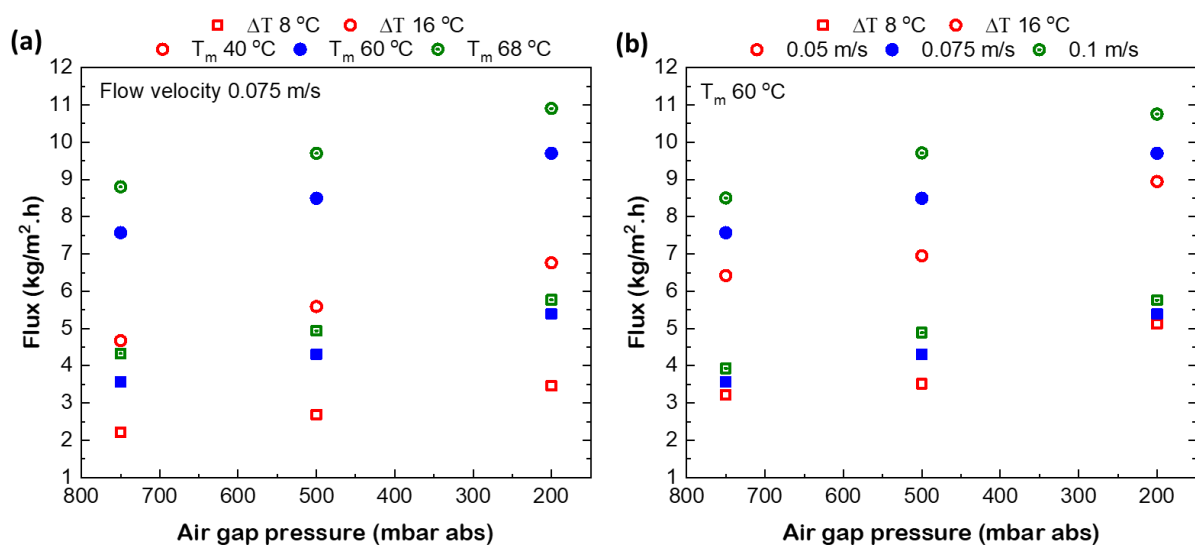


Figure 4.6: Summary plot to analyze effect of (a) varying air gap pressure on flux with T_m and ΔT changing for constant flow velocity 0.07 m/s, and (b) ΔT and flow velocity changing for constant T_m 60 °C using tap water as feed

Under vacuum conditions, the flux improves so latent heat transfer also improves. Also, depending upon the degree of deaeration, the transferred conductive heat decreases which reduces the total heat transferred and as mentioned before, increases the thermal efficiency. A slight dip in thermal efficiency for T_m 68 °C at air gap pressures 500 mbar (abs) and 200 mbar (abs) is noticed but the values are within ± 3 % of each other and the drop is attributed to inaccuracies associated with the instrumental error.

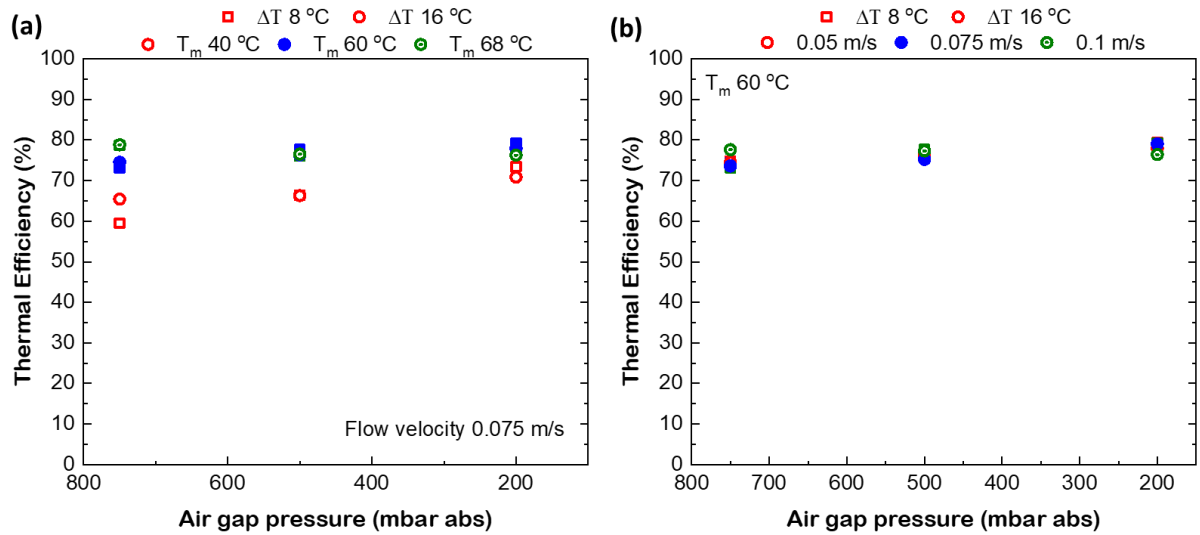


Figure 4.7: Summary plot to analyze effect of (a) varying air gap pressure on thermal efficiency with T_m and ΔT changing for constant flow velocity 0.07 m/s, and (b) ΔT and flow velocity changing for constant T_m 60 °C using tap water as feed

4.1.2.5 Comparison with ambient pressure (AGMD)

Figure 4.8 draws an analytical comparison between AGMD and V-AGMD setup in terms of performance metrics of flux and thermal efficiency. The main objective of this thesis was to investigate enhancement of MD using vacuum and Figure 4.8 clearly depicts that. Experiments were done under ambient conditions and compared with the results already obtained by employing vacuum. It is observed that in comparison to ambient condition, using vacuum enhances both flux and thermal efficiency. Reducing the air gap pressure improves the flux and thermal efficiency due to increased mass transfer driving force and decreased conductive heat transfer.

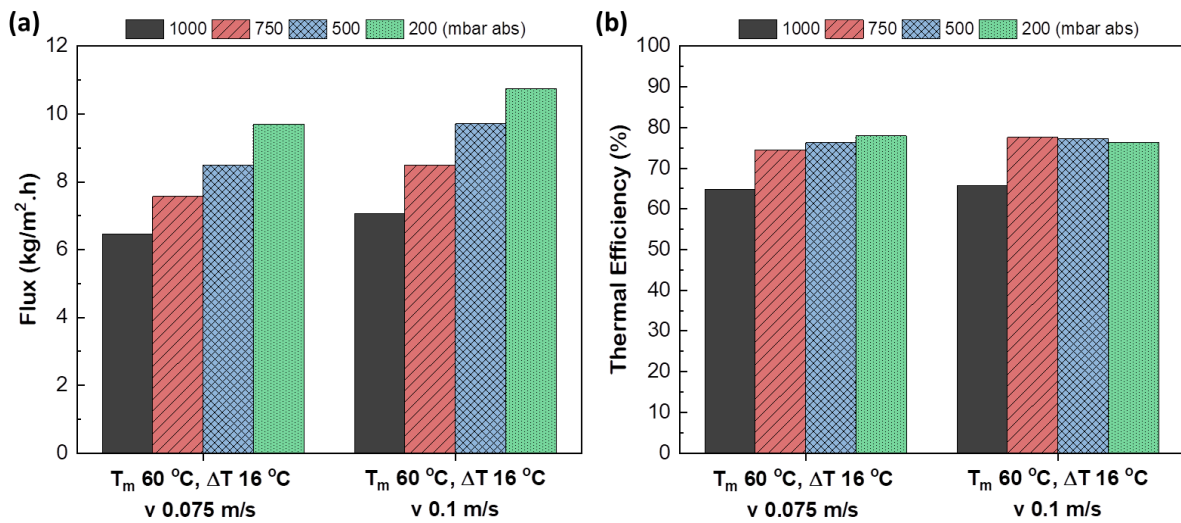


Figure 4.8: Comparison of V-AGMD and AGMD (a) flux, and (b) thermal efficiency ranging from air gap pressure 1000 mbar (abs) to 200 mbar (abs)

4.1.3 Performance characterization of V-AGMD setup with hypersaline feed

After investigating the enhancement of MD performance metrics by using vacuum and tap water, a hypersaline feed was used. Generally, dilute solutions conduct less electricity because of the lower number of ions but for the feed solution used in these experiments, it was experimentally determined that high conductivity implied low concentration. This was attributed to a high concentration of MgCl_2 in the feed as it shows inverse relation of concentration and conductivity after 0.6 wt%. For the initial concentration of 312.7 g of salt/kg solution, it was experimentally verified that the feed solution crystallized in the range 25 °C to 30 °C at the given concentration. Therefore, a heating rod was used to ensure a uniform feed temperature of 50-60 °C during the experiments. The feed salinity was controlled using the PID controller to stay in the feed concentration range under study (conductivity of 163 ± 10 mS/cm). It was done by adjusting the settings for amount of distillate pumped back to the feed vessel to avoid making the feed too concentrated or dilute.

4.1.3.1 Selection of operational conditions for hypersaline feed experiments based on baseline results

The following experimental results were obtained by using tap water as feed. They gave a baseline for comparison to the experiments using hypersaline feed. It was already observed how the parameters ΔT , T_m and flow velocity influence the performance with the variation of air gap pressure, so in the next step the operational parameters were selected considering the effect of salt ions on the process as described in section 2.3.5. Another factor kept in consideration was that the probability of membrane wetting increases with the presence of salts in the feed because of reduced surface tension of feed that comes in contact with the membrane thereby reducing the liquid entry pressure (LEP).

Air gap pressure of 750 mbar (abs) was deemed insufficient to overcome the reduction in driving force while 200 mbar (abs) increased risk of membrane wetting because of high suction force associated. The need for using high ΔT arose because the minimum required driving force to produce a flux is higher with the saline feed. Since the ΔT was increased, T_m 40 °C was not suitable because crystallization of salt had to be avoided. Furthermore, because of apparatus limitation and the high density (2.6 g/cm^3) and viscosity of the feed, flow rate of 70 L/h and 100 L/h caused high pressure in the evaporator inlet. To avoid apparatus damage, low feed flow rates had to be chosen. Summary of the operational conditions for the second experimental stage are given in Table 3.6 in section 3.3.2.

4.1.3.2 Influence of spacer thickness

Influence of spacer thickness was evaluated, and the result of this comparative study is summarized in Figure 4.9. First, the experiments were carried out using a CONWED spacer of 0.8 mm thickness at ambient air gap pressure, ΔT 24 °C, T_m 60 °C, flow velocity of approximately 0.07 m/s and average feed conductivity 162.64 mS/cm. It was observed that no flux was obtained because the mass transfer

driving force had reduced significantly in addition to the resistance provided by the non-condensable gases present in the air gap. At 400 mbar (abs) a flux of 0.8 kg/m².h was recorded for ΔT 20 °C, T_m 60 °C, flow velocity of 0.07 m/s and average feed conductivity of 170.41 mS/cm. It was also observed that after 4 hours, because of the low pressure, the membrane and condenser film ballooned, compressing the air gap which led to its blockage.

Next, the air gap thickness was increased using an Intermaas spacer of 1.15 mm thickness. First an experiment was conducted for ambient air gap pressure, ΔT 24 °C, T_m 60 °C, flow velocity of 0.07 m/s and average feed conductivity of 169.93 mS/cm. Then an experiment was conducted at 400 mbar absolute pressure in the air gap and for ΔT 20 °C, T_m 60 °C, flow velocity of 0.07 m/s and average feed conductivity of 163.34 mS/cm. Flux values of 2.53 kg/m².h and 1.71 kg/m².h were observed, respectively. Here the flux obtained at ambient air gap pressure is higher because of the higher ΔT and relatively low feed concentration. It was also observed that a higher thermal efficiency of 29.9 % and 24.5 % was achieved when the wider Intermaas spacer was used for ambient air gap pressure and 400 mbar (abs), respectively.

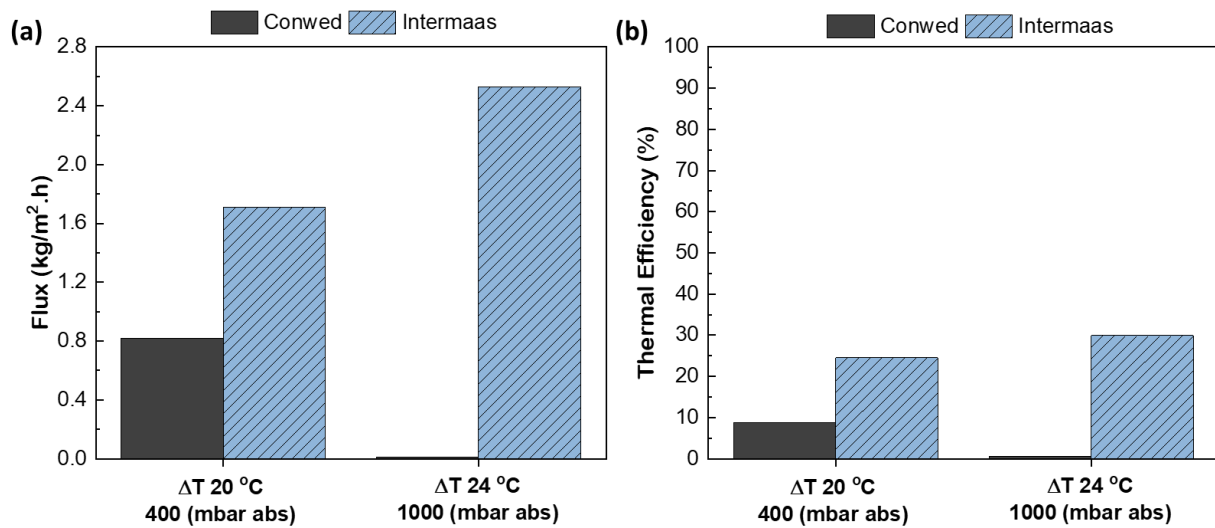


Figure 4.9: Comparison of (a) flux, and (b) thermal efficiency using spacers of different thickness

The reason suggested for this improved performance can be understood with the help of Figure 4.10. When a wider spacer is used, the temperature difference across the air gap (until the condenser surface is reached) is higher resulting in greater transmembrane temperature gradient and higher driving force. However, as the air gap pressure is reduced, flux is obtained only until the suction force does not compress the air gap. Henceforth, it is suggested to further investigate the effect of increasing air gap width using thicker spacer to find a balance point.

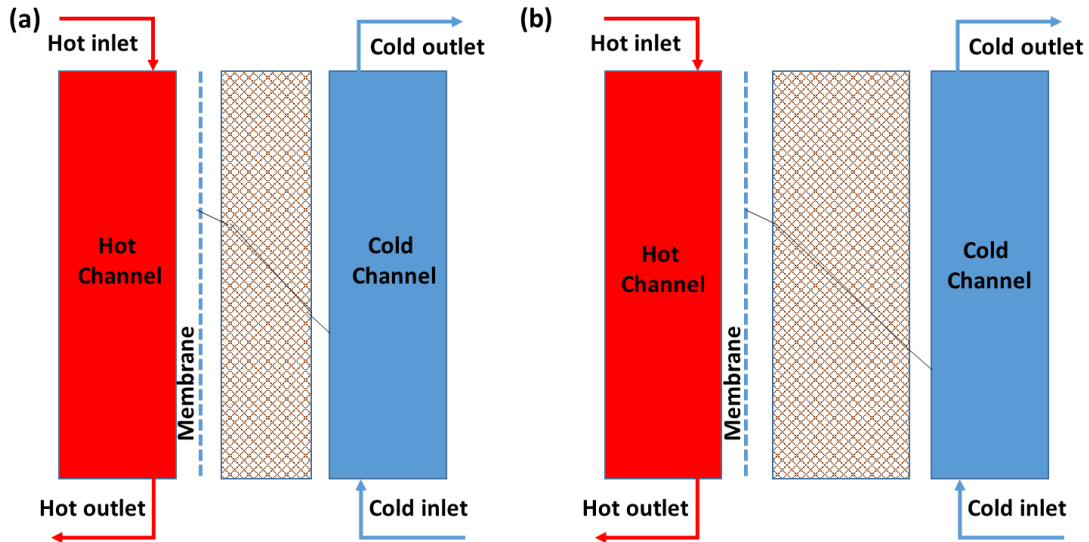


Figure 4.10: Approximated temperature profiles for (a) thin spacer, and (b) thick spacer

4.1.3.3 Influence of flow velocity in hot and cold channel for ambient pressure in the air gap

It was observed that as the feed velocity increased, for both ΔT 16 °C and 24 °C, the flux also improved. At ΔT 24 °C, the flux increased by approx. 57 % from 0.39 kg/m².h at 0.07 m/s to 0.92 kg/m².h at 0.091 m/s. A similar trend was observed at ΔT 16 °C. As the velocity increases, improved Reynolds number translates in more turbulent flow of the fluid. Furthermore, an additional heat energy is introduced into the system which increases both the bulk and interfacial temperature difference across the membrane surface. This increase in temperature difference increases the driving force as the vapor pressure difference is elevated. Therefore, the rate of evaporation at the membrane feed interface is also higher. Resultantly, the distillate flux increases as observed in Figure 4.11 (a).

Figure 4.11 (b) depicts the impact of changing flow velocity for different ΔT ambient pressure and T_m 60 °C on the thermal efficiency (η_{th}) of the system. It can be observed that the thermal efficiency is less than 30 % for all experimental points which is quite low. This can be attributed to the fact that for hypersaline solutions, only a small fraction of heat is actually utilized as driving force because the driving force is consumed by the reduction in vapour pressure (refer to Figure 2.11) leading to the low thermal efficiency values. Thermal efficiency is generally independent of flow velocity and ΔT as explained for the tap water experiments in section 4.1.2.1. Hence, the $\approx$ 5 % difference between thermal efficiency values as the flow velocity and ΔT changes is attributed to inaccuracies associated with instrumental errors of the temperature sensors and the cooling unit, therefore, it is suggested to repeat these experimental points.

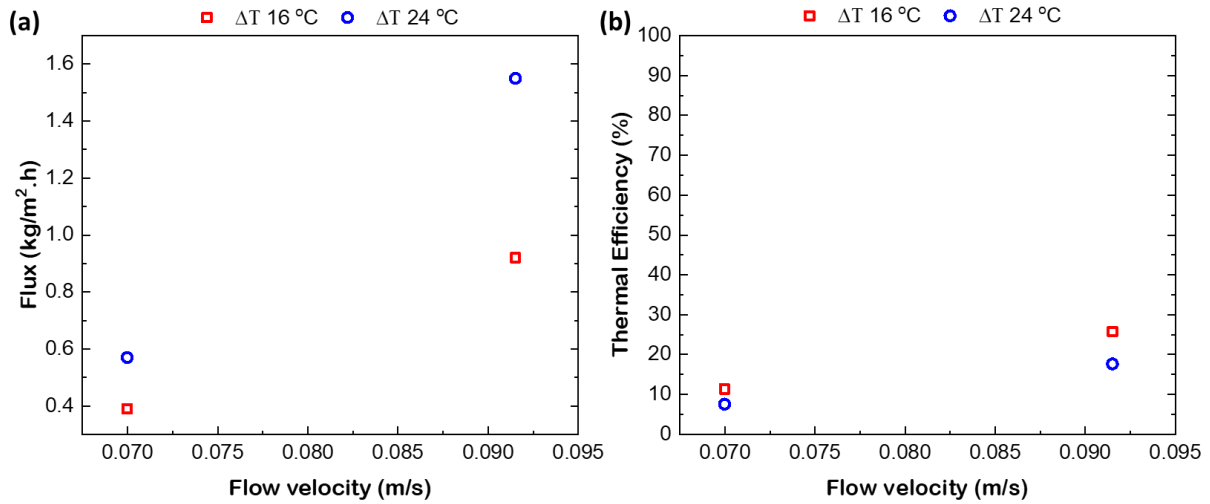


Figure 4.11: Influence of varying flow velocity on (a) flux, and (b) thermal efficiency obtained using hypersaline feed for ambient air gap pressure, $T_m = 60\text{ °C}$, and at $\Delta T = 16\text{ °C}$ and $\Delta T = 24\text{ °C}$ with an average feed conductivity of 157.8 mS/cm

4.1.3.4 Influence of temperature difference between hot and cold channels (ΔT) for ambient air gap pressure

The effect of changing ΔT on permeate flux is observed in Figure 4.12 (a) for a mean temperature of 60 °C , ambient air gap pressure and flow velocity of 0.07 m/s and average feed conductivity of 159.6 mS/cm . Similar to what was observed for tap water experiments, a general trend of increase in flux with increase in ΔT is observed. From $0.28\text{ kg/m}^2\cdot\text{h}$ at $\Delta T = 14\text{ °C}$, the flux rises to $0.74\text{ kg/m}^2\cdot\text{h}$ at $\Delta T = 28\text{ °C}$. It can be concluded that the effect of increasing ΔT is the same even with vacuum. An interesting point to notice here is that when tap water was used as feed, under ambient air gap pressure, flow velocity 0.075 , $T_m = 60\text{ °C}$ and $\Delta T = 16\text{ °C}$ the flux was $6.45\text{ kg/m}^2\cdot\text{h}$ which is higher than the flux obtained even for $\Delta T = 28\text{ °C}$ for hypersaline feed. Again, this can be reasoned with the reduction in vapor pressure difference and hence the driving force as depicted in Figure 2.11.

Figure 4.12 (b) shows that for different ΔT values, the difference between the thermal efficiency values remains within $\pm 4\%$. The deviations are attributed to inaccuracy associated with the instrumental error. This phenomenon of independence of thermal efficiency from ΔT which was observed and explained for tapwater experiments is still valid with the application of vacuum.

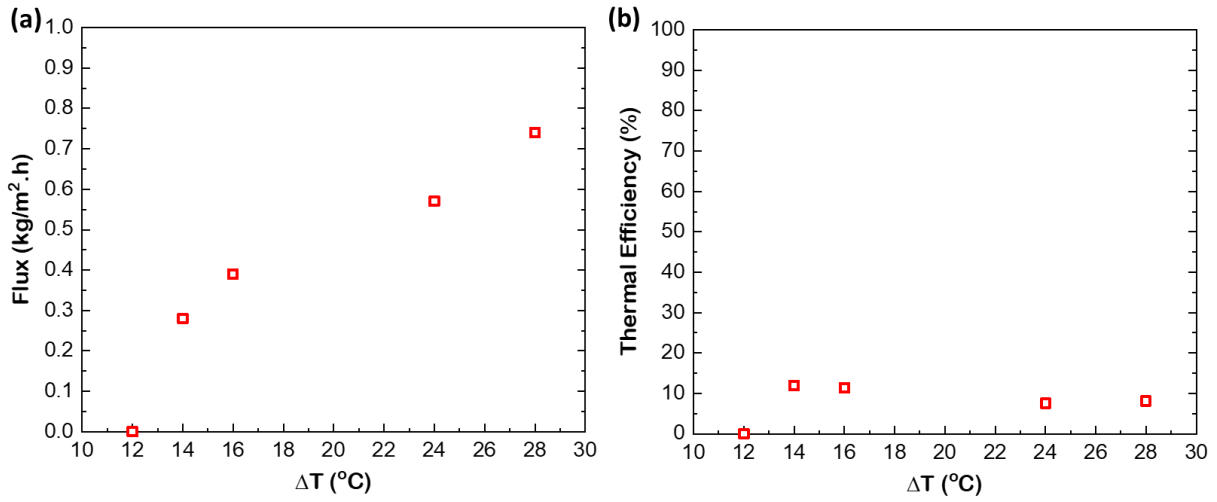


Figure 4.12: Influence of changing ΔT on (a) flux, and (b) thermal efficiency for T_m 60 °C, ambient air gap pressure, and 0.07 m/s flow velocity with an average feed conductivity of 159.6 mS/cm

4.1.3.5 Influence of Mean Temperature (T_m) for sub-ambient air gap pressure

Figure 4.13 (a) depicts how flux varies with increasing mean temperature for two flow velocities (0.075 m/s and 0.091 m/s.), at ΔT 16 °C and an air gap pressure of 400 mbar (abs). For flow velocity 0.07 m/s, the flux increases from 0.54 kg/m²·h at 49.3 °C to 1.91 kg/m²·h at 67.1 °C. A similar positive influence of mean temperature on flux is observed for flow velocity 0.09 m/s although when we compare the flux values at T_m 59.3 °C and 67.1 °C, we notice that the increment is not as pronounced as for flow velocity 0.07 m/s for the same values of T_m so the experimental point at T_m 67.1 for flow velocity 0.09 m/s is suggested to be repeated to affirm the trend. Nevertheless, the general positive effect of increasing mean temperature on flux which was observed for tap water experiments is found to also be valid for the hypersaline feed under vacuum conditions.

Figure 4.13 (b) shows the influence of T_m on thermal efficiency. For flow velocity 0.07 m/s, the thermal efficiency increases from around 12 % to 47 % as the T_m rises from 49.3 °C to 67.1 °C. The phenomenon of a positive influence of increasing T_m on thermal efficiency observed for tap water experiments holds valid for hypersaline feed. This trend is valid for a flow velocity of 0.09 m/s with the exception of the deviation at T_m 67.1 °C. The thermal efficiency drops here because of the relatively low flux value obtained at this T_m and as explained before, it is suggested to repeat this reading. The deviation is attributed to inaccuracy associated with the instrumental error.

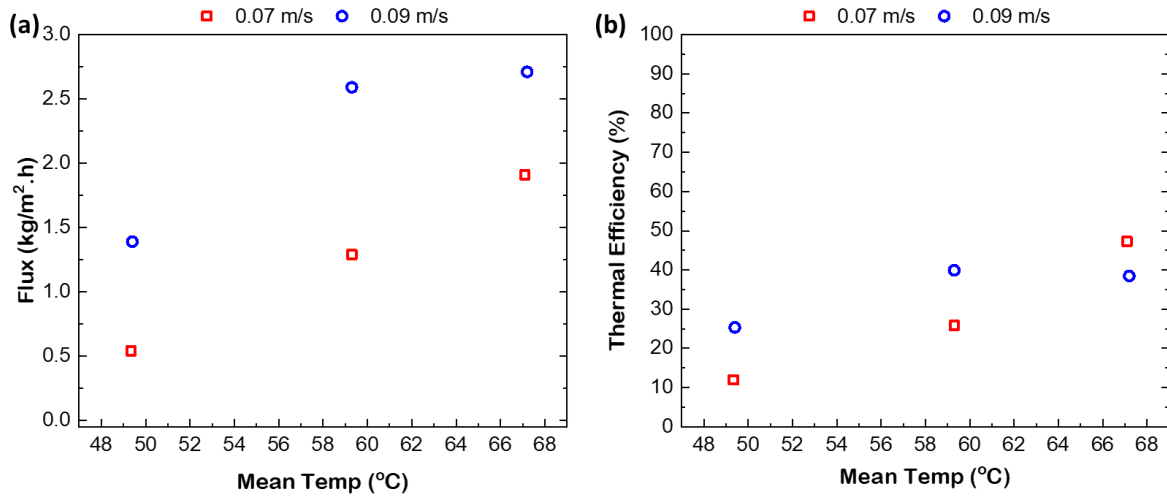


Figure 4.13: Influence of changing mean temperature on (a) flux, and (b) thermal efficiency for flow velocity of 0.07 m/s and 0.09 m/s at air gap pressure of 400 mbar (abs) and ΔT of 16 °C with an average feed conductivity of 168.3 mS/cm

4.1.3.6 Influence of varying air gap pressure

The effect of varying air gap pressure on the flux obtained is seen in Figure 4.14 (a) while keeping flow velocity, ΔT , and T_m constant at 0.07 m/s, 16 °C and 60 °C, respectively. A general trend of flux enhancement is observed with decreasing air gap pressure. At 600 mbar (abs), the removal of NCGs from the air gap is not that efficient so a low flux of 1.11 kg/m²·h is observed although it still fits the trend. The highest flux of 1.48 kg/m²·h is obtained when the pressure in the air gap was 350 mbar (abs). Figure 4.14 (b) shows the effect of varying air gap pressure on the thermal efficiency of the system concerning constant flow velocity, ΔT , and T_m . Thermal efficiency increases from 21.8 % at 600 mbar (abs) to 28.4 % at 350 mbar (abs).

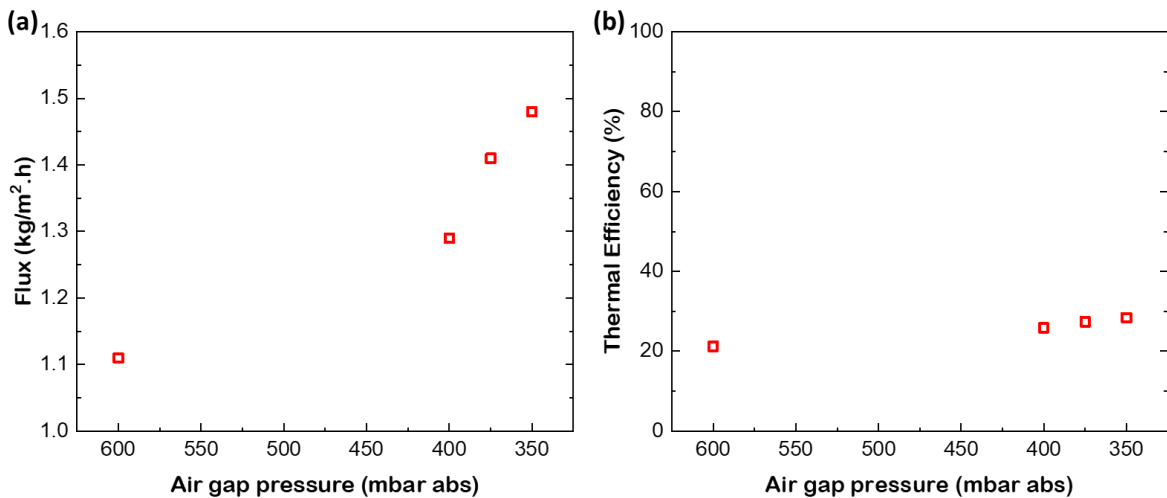


Figure 4.14: Influence of varying air gap pressure on (a) flux, and (b) thermal efficiency with ΔT 16 °C, flow velocity 0.07 m/s, and T_m 60 °C with an average feed conductivity of 168.3 mS/cm

A comparative analysis is drawn for experiments carried out at the same conditions for all operational parameters except the air gap pressure as given in Figure 4.15 for two flow velocities: 0.07 m/s and 0.09 m/s. It is observed that for both flux and thermal efficiency, the positive effect of the applying vacuum as observed and explained for the tap water experiments stays valid even for hypersaline feed experiments.

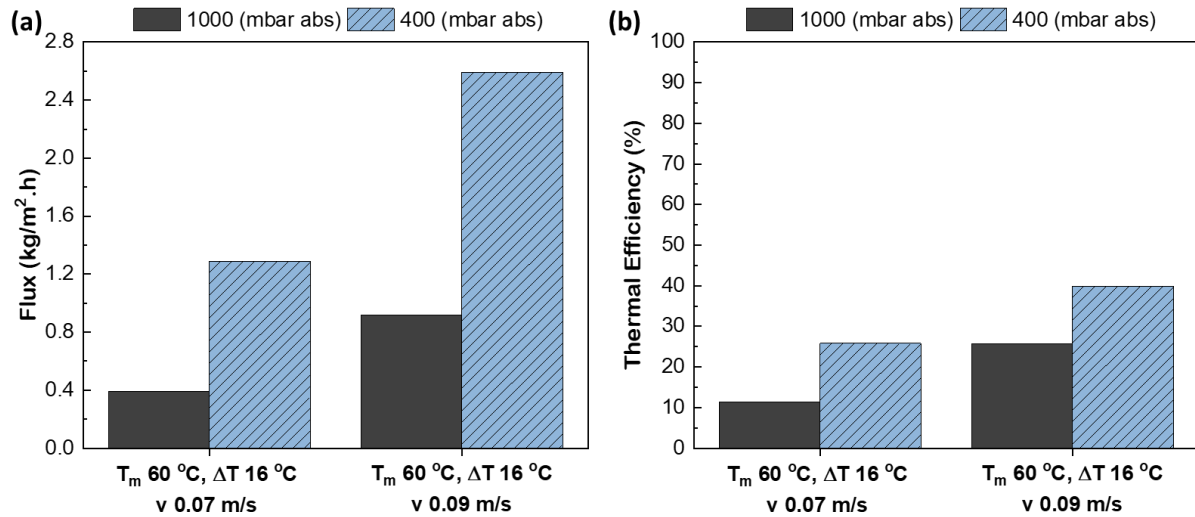


Figure 4.15: Influence of varying air gap pressure on (a) flux, and (b) thermal efficiency for feed with an average conductivity of 163.7 mS/cm at $T_m 60\text{ }^{\circ}\text{C}$, $\Delta T 16\text{ }^{\circ}\text{C}$ for flow velocities of 0.07 m/s and 0.09 m/s

In summary, it can be concluded that for hypersaline feed an additional fraction of energy is needed to overcome the reduction in driving force due to the presence of salt. However, with the lowering of the diffusion resistance by application of vacuum in the air gap, the resistances faced reduced so that more energy is available to overcome the reduction in driving force due to salt. Henceforth the performance metrics improve.

4.1.3.7 Influence of feed concentration (feed conductivity)

The influence of feed concentration was analyzed for obtained flux. It was experimentally determined that, unlike the normal cases where a high concentration would denote high conductivity of solution due to the presence of more ions that can conduct electricity, the hypersaline feed solution used had the opposite trend. A higher concentration was represented by low conductivity. This is attributed to MgCl_2 which is the main component of the feed solution and shows an interesting behavior of decreasing conductivity after a certain wt% concentration [83], [84]. A graphical representation of MgCl_2 behavior can be seen in Figure 7.2 in subchapter 7.6 of the appendix. Theoretically, it was expected that high flux will be obtained at high conductivity (low concentration) and the results obtained verified this as shown in Figure 4.16. For both sets of experimental conditions, high flux values were observed for high

conductivity values as it represented dilute solution. It can also be seen that at ΔT 24 °C the flux values for ambient pressure are more than air gap pressure of 325 mbar (abs) with a lower ΔT of 16 °C. For ΔT 24 °C at 325 mbar (abs), higher flux is predicted as per the findings of this thesis project. To further verify it, it is suggested to carry out an experiment at ΔT 16 °C, T_m 60 °C and air gap pressure 325 mbar (abs) to compare with the experiment done at ambient pressure. Nonetheless, within the scope of each experimental reading the same trend of, low flux values at high feed concentration (low feed conductivity) and high flux values at low feed concentration (high feed conductivity) is observed.

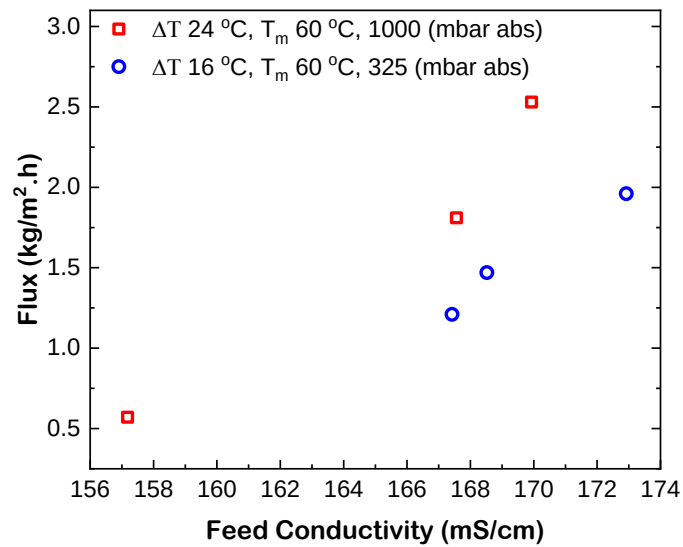


Figure 4.16: Influence of feed conductivity on obtained flux at flow velocity 0.07 m/s and different experimental conditions for T_m , air gap pressure and ΔT

Chapter 5: Conclusion and future perspectives

The aim of this thesis was to study process enhancement in air gap membrane distillation (AGMD) for hypersaline feed treatment using vacuum. Initially, the effect of employing vacuum by using tap water as the feed solution was investigated. This provided a baseline before changing the feed to a hypersaline solution and evaluating how the application of vacuum affects the performance metrics. For both experimental stages, lab-scale experiments were carried out using a PTFE membrane. Operational parameters such as mean temperature T_m , a difference of temperature between heating and cooling stream (ΔT), flow velocity and pressure in the air gap were varied. The experimental results were consistent with the theoretical expectations. The operational conditions which resulted in reasonable tradeoff between flux and thermal efficiency values were considered optimal within the scope of experiments carried out. These were T_m of approximately 60 °C, ΔT of 16 °C, flow velocity of 0.1 m/s and vacuum pressure of 500 mbar (abs) for tap water experiments. While T_m of 60 °C, ΔT of 16 °C, flow velocity of 0.07 m/s and vacuum pressure of 350 mbar (abs) were the optimal conditions for hypersaline feed experiments. For the case of tap water, a quantitative comparison between AGMD and V-AGMD setup showed that for the set of operational parameters considered optimal, the flux improved by 37.6 %, and thermal efficiency improved by 17.7 %. When compared with the highest vacuum employed, the flux improved by 52.4 %, and thermal efficiency enhanced by 16.3 %. As for the hypersaline feed, for the set of operational parameters concluded optimal, in comparison with AGMD setup, the V-AGMD setup showed flux improvement by about 279.5 % and thermal efficiency enhancement by 149.9 %. The effect of vacuum is more pronounced for hypersaline solution because the vapor pressure has been reduced to such an extent that ambient conditions produce marginal results, thus on a relative scale, the results obtained under vacuum are high. Therefore, it could be concluded that the removal of non-condensable gases by using vacuum improves the flux by decreasing the mass transfer resistance faced in the air gap.

Apart from enhanced flux, creating a vacuum in the airgap also reduces conductive heat transfer which improves the thermal efficiency. As a result of these benefits incurred, sub-ambient pressure in the air gap makes available more heat to counter vapor pressure reduction for the case of saline feed. Therefore, V-AGMD is a viable option for hypersaline feed treatment.

Furthermore, it can be concluded that the data obtained from the experimental work in the scope of this thesis project can be used to validate a simulation model for upscaling from lab scale to pilot scale. For the first experimental stage, the performance of PTFE membrane by GORE performed consistently but due to commercial unavailability of this membrane, a Donaldson 6501 PTFE membrane was used for experimental stage 2. Analysis of its consistent performance helped conclude that it can be used for future work using a V-AGMD setup.

Considering future perspectives, some recommendations are made from the scope of this project:

- **Study the variation of air gap width**

For hypersaline feed treatment, a spacer of thickness of greater than 1.15 mm should be used to vary the air gap width and the optimum conditions should be found until, which by using vacuum, the gain in mass transfer driving force compensates for the loss of insulation.

- **Study the effect of vacuum with heat recovery in pilot-scale module**

Since the efficacy of using vacuum has been established, and on lab-scale heat recovery loops were not possible so it is suggested to study the cumulative effect when a vacuum is used and simultaneously heat is recovered. Thermal efficiency values obtained in such a case will be interesting to secure applications for the V-AGMD setup.

- **Fabricate MD specific membranes and test in V-AGMD setup**

For hypersaline feed treatment, the problem of membrane wetting is recurring because of the high propensity of inorganic fouling caused by the salts. It is suggested to explore the vista of surface modification of membranes to deal with inorganic scaling faced for hypersaline feeds. Furthermore, it is recommended to carry out V-AGMD tests using omniphobic membranes and also to delegate some research on membrane fabrication. This will not only relieve the company from dependence on manufacturers in the market but research in this regard can help make a breakthrough regarding the issue of fouling and scaling of MD membranes.

- **Carry out further research on the suitability of V-AGMD for novel applications**

The viability of V-AGMD for hypersaline feed treatment can be concluded from this project. It is further recommended to assess the impact of vacuum for novel applications such as breaking azeotropic mixtures to know more about its applicability since the use of AGMD for azeotropic separation is already found in the literature.

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Appendix

7.1 Pilot investigations of MD technology

Table 7.1: Different pilot investigations of MD technology [20]

Vendor	Year	Membrane material	Membrane configuration	Application	Feed TDS mg/L	Product water TDS (mg/L)	MD flux (L/m ² .h)	Recovery (%)	GOR	Energy consumption (kWh/m ³)
Xzero	2014	PTFE	AGMD	Thermal brine	68529	1472	2.5	NA	0.74	1031
Memsys	2016	PTFE	VMD	Hypersaline ground water	62592	<5	5	40	2.5	260
Aquastill	2016	LDPE	AGMD	Sea water	35000	50	1.0	5	6-7	90-95
Memsys	2016	PTFE	VMD	Brackish water	1000	2	1.6-2.5	NA	NA	ND
Memsys	2017	PTFE	VMD	Saline water	15000	NA	1.7	76	2.0	340
Fraunhofer	2017	PTFE	AGMD	Sea water	35000	<1	1.1	4-10	3.18	207
Solarspring	2018	PTFE	AGMD	Saline water	240000	128	0.7	NA	3.64	200-800
Memsys	2018	PTFE	VMD	Sea water	25600	<2	8.5	36	3.3	200
Solarspring	2019	PTFE	AGMD	Saline water	35000	<1	1.5	NA	NA	NA
Aquastill	2020	LDPE	VEMD	Hyper saline solution	25000-292000	508	1-3	NA	8.5-13.5	48-77

7.2 Operation parameters for comparison of test plate (TP) and test cell (TC)

Table 7.2: Detailed experimental conditions for experiments done to compare TP and TC performance under same conditions

Experi- ment	Flow rate L/h	ΔT	T Evap In (°C)	T Cond Out (°C)	Average Pressure (mbar)		Flux (kg/m ² .h)		Tm (°C)		Flux predicted from test plate exps (kg/m ² .h)	% Difference
#					Test plate	Test Cell	Test plate	Test Cell	Test plate	Test Cell		
1	70	8	44	36	500.58	508.14	2.64	2.69	38.58	39.48	2.735032	-1.67
2	70	8	64	56	509.74	506.52	4.32	4.31	58.18	59.31	4.339279	-0.68
3	70	8	72	64	485.4	511.86	4.82	4.94	65.93	67.19	4.976771	-0.74
4	70	16	48	32	501.34	508.83	5.19	5.59	37.02	38.83	5.449478	2.51
5	70	16	68	52	484.99	509.3	8.08	8.49	56.22	58.51	8.531366	-0.49
6	70	16	76	60	489.16	504.26	9.43	9.7	63.85	66.34	9.757544	-0.59
7	100	8	44	36	496.24	506.77	2.82	2.98	38.86	39.62	3.05197	-2.42
8	100	8	64	56	498.07	500.81	4.65	4.89	58.48	59.95	4.647875	4.95
9	100	8	72	64	-	493.87	-	5.49	-	67.4	5.2327	4.69
10	100	16	48	32	499.93	508.83	5.72	6.3	37.57	39.15	5.96388	5.34
11	100	16	68	52	495.81	495.92	8.42	9.71	56.03	58.84	9.413568	3.05
12	100	16	76	60	493.26	509.52	10.5	10.88	64.56	66.73	10.795896	0.77

7.3 Template for thermal efficiency calculation

Figure 7.1: Snapshot of template used to calculate Thermal Efficiency for V-AGMD setup

[illegible]

7.4 Important mass transfer equations from literature

A detailed insight into mass transfer is provided in the doctoral thesis of Daniel Winter [9]. Some equations that back this project's results are given below whereby 1 and 0 denote hot cold side respectively.

- Combined mass transfer across membrane (M) and airgap (AG) is given by:

$$\begin{aligned} j_{M-AG} &= j_{M-AG,d} + j_{M-AG,v} \\ &= C_{M-AG,d}(p_{w,1} - p_{w,0}) + C_{M-AG,v}(\hat{p}_1 - \hat{p}_0) \\ &= \left[\frac{1}{C_{M,d,Kn}} + \frac{1}{C_{M,d,mol}} + \frac{1}{C_{AG,d,mol}} \right]^{-1} (p_{w,1} - p_{w,0}) + C_{M,v}(\hat{p}_1 - \hat{p}_0) \end{aligned} \quad (7.1)$$

where $p_{w,0}$ and $p_{w,1}$ are the water vapor pressure on the cold permeate side and the hot feed side of the membrane, respectively. $C_{M,d,Kn}$ is the mass transport coefficient for the Knudsen diffusion mechanism.

- Mass transfer coefficient describing ordinary molecular diffusion $C_{M,d,mol}$ in a micro-porous membrane:

$$C_{M,d,mol} = \frac{M_w}{R\bar{T}_{1-0}} \frac{\varepsilon_M}{\tau_M \delta_M} \frac{\bar{p}_{1-0} D_{wa}^o}{\bar{p}_{a,1-0,ln}} \quad (7.2)$$

where δ_M , ε_M and τ_M are the thickness, porosity, and tortuosity factor of the membrane. M_w is the molecular weight of the permeating water vapor, $\bar{T}_{1-0}$ is the mean membrane temperature and D_{wa}^o is the ordinary diffusion coefficient water vapor in air.

- Additional mass transfer coefficient owing to molecular diffusion in the air gap $C_{AG,d,mol}$ represented by:

$$C_{AG,d,mol} = \frac{M_w}{R\bar{T}_{1-0}} \frac{\varepsilon_{AG}}{\delta_{AG}} \frac{\bar{p}_{1-0} D_{wa}^o}{\bar{p}_{a,1-0,ln}} \quad (7.3)$$

where ε_{AG} is the air gap spacer voidage and takes into account the reduction in the cross-sectional area due to the addition of the solid spacer. The path length of the diffusion is represented by the air gap thickness (δ_{AG}).

- Total pressure as function of air pressure and water vapor pressure:

$$\hat{p} = p_a + p_w \quad (7.4)$$

where $\hat{p}$ is the total pressure, p_w is the pure water vapor pressure and p_a is the air partial pressure.

- Logarithmic mean partial pressure of air in membrane represents the quantity of air in the pore volume and can be evaluated using:

$$\bar{p}_{a,1-0,ln} = \frac{p_{a,0} - p_{a,1}}{\ln\left(\frac{p_{a,0}}{p_{a,1}}\right)} \quad (7.5)$$

7.5 Important Heat transfer equations from literature:

A detailed insight into thermal efficiency calculations is provided in the doctoral thesis of Daniel Winter [9]. Some equations that back this project's results are adapted as follows:

- Thermal efficiency for combined membrane and airgap setup:

$$\eta_{th} = \frac{\dot{q}_{latent}}{\dot{q}_{total}} \quad (7.6)$$

- Whereby the heat transfer through combined membrane (M) and airgap (AG) is given by:

$$\begin{aligned} \dot{q}_{total} &= \dot{q}_{latent} + \dot{q}_{conductive} + \dot{q}_{radiation} \\ &= j_{M-AG} \Delta h_v + \left[\frac{\delta_M}{\lambda_M^*} + \frac{\delta_{AG}}{\lambda_{AG}^*} \right]^{-1} \Delta T_{1-0} + \frac{\varepsilon_{AG} \sigma}{\varepsilon_M^{-1} + \varepsilon_w^{-1} - 1} (T_1^4 - T_0^4) \\ &= k_{M-AG} \Delta T_{1-0} \end{aligned} \quad (7.7)$$

Where δ and λ represent thickness and effective thermal conductivity and k is the integral heat transfer coefficient.

7.6 Conductivity vs Concentration – the behavior of MgCl_2

Table 7.3: Electrical Conductivity κ in mS/cm for the Indicated Concentration in Mass Percent [84]

Salt	0.5%	1%	2%	5%	10%	15%	20%	25%	30%	40%	50%
MgCl_2	8.6	16.6	31.2	66.9	108	129	134	122	98		
NaCl	8.2	16.0	30.2	70.1	126	171	204	222			
KCl	8.2	15.7	29.5	71.9	143	208					
CaCl_2	8.1	15.7	29.4	67.0	117	157	177	183	172	106	
MgSO_4	4.1	7.6	13.3	27.4	42.7	54.2	51.1	44.1			

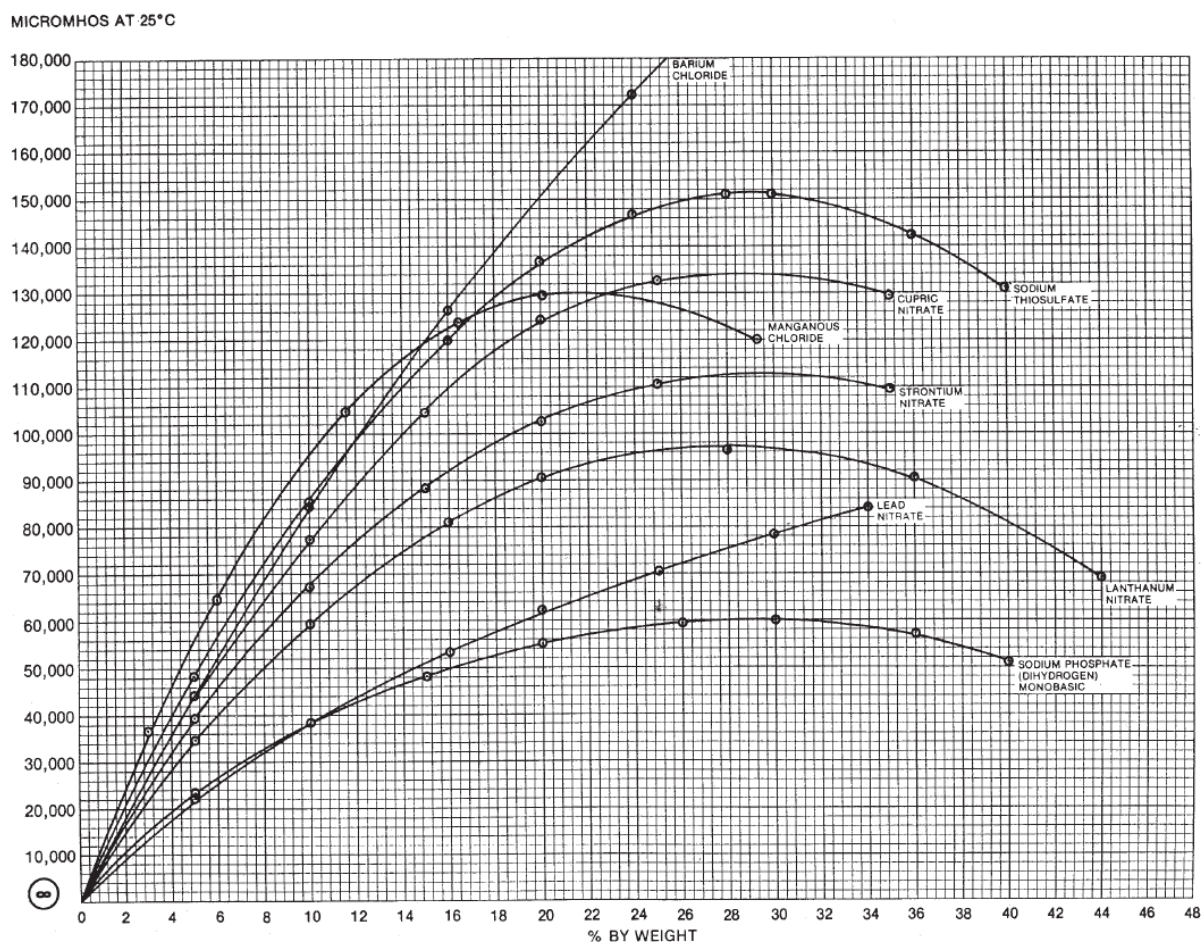


Figure 7.2: Conductance variation with wt% for electrolytes [83]