

# Membrane Distillation of Brine Wastes



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**Research Report No 63**

# MEMBRANE DISTILLATION OF BRINE WASTES

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## FOREWORD

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## EXECUTIVE SUMMARY

A significant proportion of potable water for rural communities is produced from brackish water sources by localised reverse osmosis (RO) operations. However, these inland RO plants typically recover only 70-75% of the feed water, the remaining 25-30% of the resource being discarded as saline RO concentrate. One possible remedy to this situation is to increase water recovery by passing the RO concentrate through an additional process like membrane distillation (MD).

MD is a promising, yet still emerging, technology for water desalination. MD differs from other membrane separation technologies in that the driving force for desalination is the difference in temperature either side of a membrane, rather than applied pressure as used in RO processes. In this type of operation, water vapour is not significantly reduced at high salt concentrations and therefore this technique is particularly useful for RO concentrate streams. Membranes used for a MD process are very hydrophobic, which provides a barrier for liquid water but allows water vapour to pass. As these membranes typically have large pores, relatively low temperature differences will produce surprisingly large amounts of desalted permeate. It is these relatively low temperature demands that make MD an attractive desalination process where low-grade thermal energy or solar heat sources are available. In addition to low energy consumption, MD also offers other benefits such as producing very high quality distillate, and the feed water not requiring extensive pre-treatment.

MD was first developed in the 1960s, but advances in the technology progressed very slowly due to perceived poor performance. This was partly due to non-optimal heat and flow conditions being used in early work, but also because of the generally unsuitable membranes available at the time. In the 1980s, MD research rose again and many novel MD modules were designed based on a better understanding of the mass and heat transfer processes involved. More recently, with fresh water resources becoming a problem world-wide, membrane manufacturers are now seeking to develop membranes specific for MD.

The goal of this study is to compare a selection of newly available polytetrafluoroethene (PTFE) membranes in a small MD apparatus against typical performance data reported in the scientific literature. Assessment of membrane performance takes the form of desalted water production rate produced from brackish or saline water. Laboratory scale equipment with a membrane size of 0.014 m<sup>2</sup>, along with a 0.35 m<sup>2</sup> hollow fibre element, were evaluated for overall water production rate and fouling tendency when exposed to a series of different feedwaters such as; pure sodium chloride, seawater and two different RO concentrate streams from existing plants.

Performance testing found that at brine feed temperatures of 60°C while maintaining the other side of the membrane at 20°C, water flux of 23.5 L/m<sup>2</sup>hr was achieved for laboratory scale flat sheet membranes and salt rejections of greater than 99.9% were typical. Various parameters that affect flux were found to be input temperature difference, the membrane support material and thickness. However, of significant influence to water flux were the heat and flow properties within the membrane module. Highly turbulent flow along the membrane face promotes the highest possible temperature difference on each side of the membrane, for a given set of temperature inputs, and therefore influences the rate at which water vapour will pass. Hence, the degree of turbulence within the membrane module greatly affects water flux.

Similar to other membrane filtration applications MD is sensitive to fouling, which is most likely exhibited as a lowering of flux or low salt rejection, or both. In MD applications that concentrate feed solutions to high salt concentrations, fouling could potentially be from precipitation of low solubility salts onto the membrane that causes pore blockage or reduces the membrane's effective area.

Fouling experiments were conducted using synthetic seawater feed and operated continuously for approximately three days without addition of chemical treatments or pH adjustment. It was concluded that operating the MD process in a low temperature mode, with a sub-ambient permeate cycle stream (inlets were 45°C and 5°C), may counter potential scaling problems associated with MD's elevated temperature operation. However, scaling that occurred from seawater at moderate concentrations appears to be readily removed from the membrane surface and benefits from operating at low temperature may be marginal. RO concentrate from a groundwater source obtained from Edenhope WTP containing approximately 3,300 mg/L TDS and 6.9 mg/L TOC operated for 3 days without addition of chemical treatments or pH adjustment. Over the duration of the experiment only minor flux

decline was observed. An initial flux of 23 L/m<sup>2</sup>hr reduced to 18.5 L/m<sup>2</sup>hr over 70 hours with no sign of scale on the membrane at the conclusion of the experiment.

Further fouling experiments were performed on groundwater obtained from the Botany Industrial Park (BIP) RO plant. The sample was from the secondary reject stream, comprised of 19,000 mg/L TDS with a TOC of 64 mg/L and was used without chemical treatments or pH adjustment. In contrast to the experiments with seawater feeds where only minor differences were observed in membrane fouling due to scale build up at high and low temperatures, there appears to be a significant difference in fouling from the BIP groundwater. The results showed that the initial flux of 21 L/m<sup>2</sup>hr was rapidly lost under high temperature conditions (without using any filtration schemes) until the membrane was totally covered with a recalcitrant foulant. Whereas a low temperature regime, with its initial lower flux of 16 L/m<sup>2</sup>hr, appeared to form larger amounts of loosely packed precipitate and was able to distil the RO secondary reject to greater than 67% water recovery.

This work demonstrates the potential benefits of MD processes for the minimisation of brine wastes. To further develop the technology the follow areas require investigation:

- Optimisation of the membrane module and spacer design to maximise water flux for a given input temperature difference. This was identified as a likely source of lower than expected flux.
- Identify the main foulants observed on the membrane and investigate strategies to combat fouling and/or membrane cleaning requirements. Removal of precipitate using in-line filtration may significantly improve long term performance. Simple pH control also may improve fouling performance.
- Determine what influence antiscalant chemicals used in conventional RO systems have on the operation of MD membranes with respect to scaling, fouling and flux.
- Scale up to semi-pilot dimensions to investigate limitations of larger membrane areas and include heat recovery equipment to determine economic feasibility of potential commercial installations.
- Undertake research into the extent of liquid water protrusion into membrane pores to predict optimal membrane thickness.
- Identify obstacles to further scale-up to a demonstration size MD plant

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## ABBREVIATIONS

AGMD	air gap membrane distillation
BIP	Botany Industrial Park
DCMD	direct contact membrane distillation
GE	General Electric
ID	inside diameter
LEP	liquid entry pressure
MD	membrane distillation
MS	Membrane Solutions
OD	outside diameter
PE	polyethylene
PP	polypropylene
PTFE	polytetrafluoroethylene
PVDF	polyvinylidene fluoride
RO	reverse osmosis
SGMD	sweep gas membrane distillation
TDS	total dissolved solids
VMD	vacuum membrane distillation

## 1 INTRODUCTION

### 1.1 Aim

The aim of this research project was to examine the feasibility of membrane distillation as a treatment technique to increase water recovery from reverse osmosis (RO) processes. Reducing the volume of concentrated salts generated significantly reduces the disposal footprint and construction costs associated with disposal infrastructure. Increasing overall water recovery ensures better utilisation of a limited water and environmental resource and can provide significant commercial benefits to industrial water users.

### 1.2 Background

A significant proportion of potable water for rural communities is produced from brackish water sources by localised reverse osmosis (RO) operations. However, these inland RO plants typically recover only 70-75% of the feed water, the remaining 25-30% of the resource being discarded as saline RO concentrate. Furthermore, this RO concentrate must find a disposal route which is becoming an increasingly expensive practice, and in light of chronic water scarcity, a particularly wasteful one. One possible remedy to this situation is to increase water recovery by passing the RO concentrate through an additional process like membrane distillation (MD).

MD is a promising, yet still emerging technology for water desalination. MD differs from other membrane separation technologies in that the driving force for desalination is the difference in vapour pressure across a membrane rather than total applied pressure as used in RO processes. As vapour pressure is not significantly reduced at high salt concentrations this technique is particularly useful for RO concentrate and other high salinity streams. Membranes used for MD processes are very hydrophobic which provides a barrier for liquid water but allows water vapour to pass. As these membranes typically have large pores (0.1 - 1.0  $\mu\text{m}$  pore size), relatively low vapour pressure differences will produce surprisingly large amounts of desalted permeate [1]. It is these relatively low temperature demands that make MD an attractive desalination process where low-grade thermal energy or solar heat sources are available. Incorporated as an adjunct to existing RO processes, MD appears to offer the greatest potential for the recovery of water that is presently considered unusable and is discarded.

In a MD process for desalting saline water, hot feed is passed over the surface of a porous hydrophobic polymeric membrane. Water is evaporated into the pores of the membrane at the brine–membrane interface and diffuses to the other (permeate) side of the membrane. Although there are different configurations of MD, the key difference is the technique used to draw away and condense the vapour (decrease the vapour pressure). Figure 1 illustrates the four commonly used configurations of MD [2] described as follows:

- A) Direct Contact Membrane Distillation (DCMD), in which the vapour is condensed by liquid in direct contact with the permeate side of the membrane and colder than the feed.
- B) Air Gap Membrane Distillation (AGMD), in which the permeated vapour migrates across an air gap before condensing on a cold surface nearby. Condensed permeate falls under gravity as product water.
- C) Vacuum Membrane Distillation (VMD), in which the vapour is drawn by simple vacuum and is subsequently condensed, if needed, in a separate device.
- D) Sweep Gas Membrane Distillation (SGMD), in which a gas stream is used as a carrier for the permeate vapour.

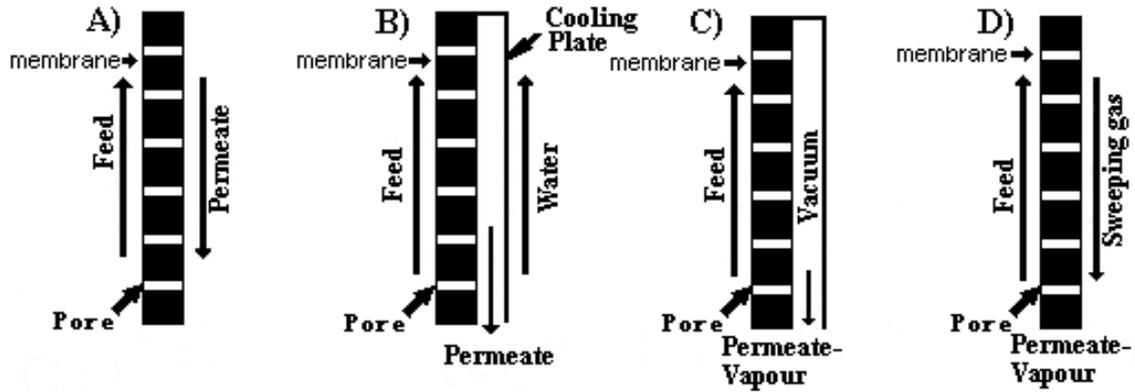


Figure 1 Configurations of various MD system

Advantages of membrane distillation over reverse osmosis or other thermal methods of desalination include:

- It produces very high-quality distillate. In most circumstances salt rejections of 99-100% are achievable.
- Water can be distilled at relatively low temperatures (i.e. 5 to 80°C). As the driving force for MD is temperature difference, very low feed temperatures can produce reasonably high rates of product water and may be more practical considering the nature of some water impurities (e.g. scaling issues at high temperature).
- Low-grade heat such as industrial waste heat, solar or desalination waste heat may be used.
- The feed water does not require extensive pre-treatment that is typically required for pressure-based membrane processes.

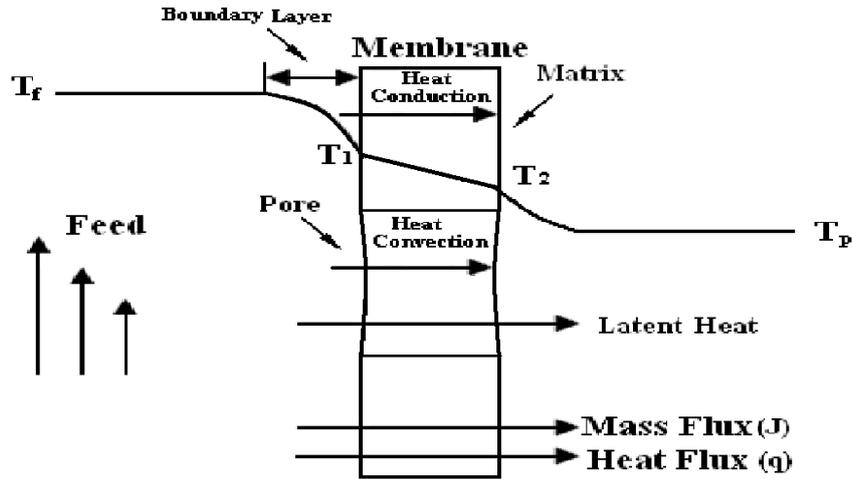
## 1.2.1 Performance Governing Features

The various MD configurations work slightly differently from each other, which in turn offer advantages and drawbacks in practice. This project considered only DCMD, primarily for its high performance and inbuilt water condensing ability.

### 1.2.1.1 Direct Contact and Air Gap MD

Both DCMD and AGMD work according to a temperature difference. The temperature profile and heat and mass transfer process is shown in Figure 2. Here, temperature of the feed,  $T_f$ , drops across the feed side boundary layer to  $T_1$  at the membrane's hot surface. The cold cycle temperature  $T_p$  increases across the permeate boundary layer to  $T_2$  at the membrane's cold surface.

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**Figure 2** Heat transfer and mass transfer through membrane

Therefore the driving force for distillation is the vapour pressure difference generated from the temperature difference  $T_1 - T_2$ . However, it is the degree of temperature equalisation from the bulk liquids to the membrane surface, i.e. the temperature drop from feed  $T_f$ , to  $T_1$  and temperature rise from cold inlet  $T_p$  to  $T_2$ , that limits desalination rates. This effect is known as temperature polarisation, and similar to concentration polarisation in a membrane filtration process, is a major limiting factor on the process. The temperature polarisation coefficient  $\tau$  is defined by [4]:

$$\tau = \frac{T_1 - T_2}{T_f - T_p} \quad (1)$$

MD developments in the 1960s, carried out in non-optimal conditions, slowed due to poor reported performance [3]. The temperature polarisation coefficient for the Handbury and Hodgkiess [3] system was estimated to be 0.32. Therefore, when the temperature difference between the centres of the hot and cold channels was 10°C (and therefore even higher input temperature differences), the actual temperature difference across the membrane was only 3.2°C. Although the worst effects of temperature polarisation can be controlled with high velocity turbulent flows within the module, this requires optimal module design and greater pumping capacities. However, high velocities on both sides of the membrane to counter temperature polarisation is limited by the threat of excessive pressure within the membrane module causing liquid feed water to pass through the membrane. In the 1980s, with the development of new membranes, MD research again increased in activity and many novel MD modules were designed based on a better understanding of the mass and heat transfer processes [5].

In DCMD, the hot and cold streams are in intimate contact with only a thin polymer membrane between. This results in superior permeate production but at the cost of greater heat and chilling demands of the system via high thermal conduction through the membrane. AGMD works in a similar way to DCMD, but with the added resistance of the air gap layer. The temperature polarisation effect described for DCMD is therefore also encountered in AGMD, but it is reduced on the permeate side by the insulating air-gap. Consequently the heating inputs of an AGMD system are significantly lower than a DCMD process, but water flux is similarly lower because of a very small temperature difference between  $T_1$  and  $T_2$ . From a practical point of view, AGMD requires an added cold surface making module design more complicated. This however has been used advantageously to harness the lost latent heat to preheat the feed water thus improving energy efficiency. This technique is used by a commercially available process known as Memstill [6], which is currently under demonstration and is discussed further in section 1.3.2.

### 1.2.1.2 Heat transfer

MD performance relies on the transfer of heat to the permeate side comprising both sensible and latent heat. The sensible heat transfer across the membrane consists of heat conduction in the membrane matrix and heat conduction through the pores of the membrane via stagnant air or vapour, while latent heat transfer is accompanied by the vapour mass transfer. Within the feed side of a membrane module, heat needs to be transferred from the liquid to the surface of the membrane and in the permeate side heat needs to be transferred from the membrane surface into the condensed liquid.

### 1.2.1.3 Mass transfer

The mass transfer in an MD process proceeds in three steps; water vaporises from the liquid/gas interface of the feed, vapour passes through membrane pores by diffusion and convection, and vapour condenses on the permeate side. Membranes employed for MD exert their influence on transmembrane water flux in three distinct ways. Firstly, water vapour molecules move only through the pores of the membrane, so the effective area for mass transfer is less than the total membrane area. Secondly, for most practical membranes the pores do not pass directly through the membrane, therefore the vapour transport path is greater than the thickness of the membrane. Thirdly, the inside wall of pores also increases resistance to diffusion by decreasing the momentum of vapour molecules. If the fluid dynamic conditions on both sides of a membrane are considered good (i.e. sufficiently turbulent flow and reasonable temperature difference), mass transfer through the membrane may be the limiting factor in producing a practical MD operation [7].

Schofield's model [8] and the Dusty-Gas model [9 & 10] are widely used for determining the mass transfer in MD. Both models suggest that mass transfer rate is proportional to the vapour pressure difference across the membrane [8]

$$N = C(P_{T_1} - P_{T_2}) = C\Delta P \quad (2)$$

where,  $N$  is the mass transfer rate or flux,  $C$  is membrane distillation coefficient that can be obtained experimentally, and  $P_{T_1}$  and  $P_{T_2}$  are the vapour pressures at  $T_1$  and  $T_2$ , respectively. Due to the relationship between vapour pressure and temperature, the above relationship shows that the flux is an exponential function of temperature difference. Furthermore,  $C$  is an important parameter to assess the performance of a membrane and will depend largely on the physical characteristics of the membrane.

### 1.2.1.4 Characteristics of Membranes

The membrane itself is not directly involved in selective transport. The membrane instead acts as a physical structure to contain the vapour interface and prevent liquid from entering the pores, much in the same way hydrophobic Styrofoam remains buoyant in water since it does not allow water to penetrate. Volatile compounds are however, transferred across the membrane via the pores according to vapour-liquid equilibrium principles [11]. Therefore, properties of membranes used for MD ideally should include:

- Thin film with low tortuosity. As permeate flux is inversely proportional to the diffusion length, membrane voids with a short mean free path offer a distinct advantage.
- Low surface energy of the membrane material, i.e. high hydrophobicity.
- Relatively large pore size. Mass transport is proportional to pore diameter, therefore large pore sizes are an advantage. However, the upper limit of usable pores sizes, for a given hydrophobicity is set by the minimum pressure that liquid water will enter the pores and pass through the membrane. This is known as the Liquid Entry Pressure (LEP).
- Low thermal conductivity of the membrane material. Minimising heat conduction across the membrane will maximise the temperature difference for a given heat input.
- High porosity. This is to make the most use of the membrane's characteristics by producing the maximum amount of product water from a given area of membrane.

### 1.2.2 Project Goals

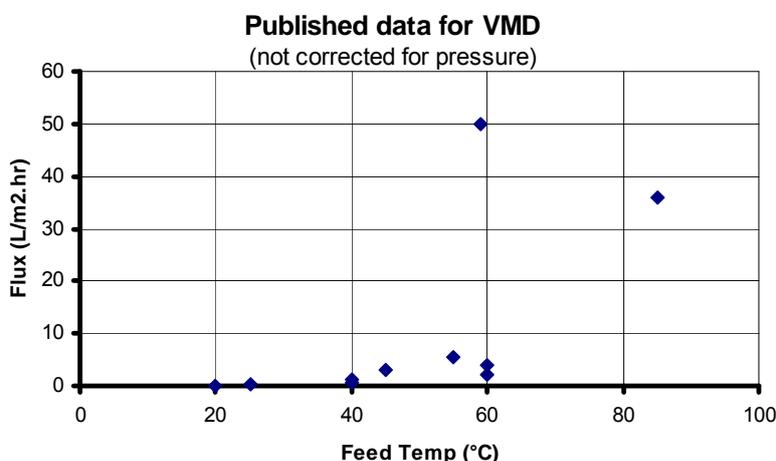
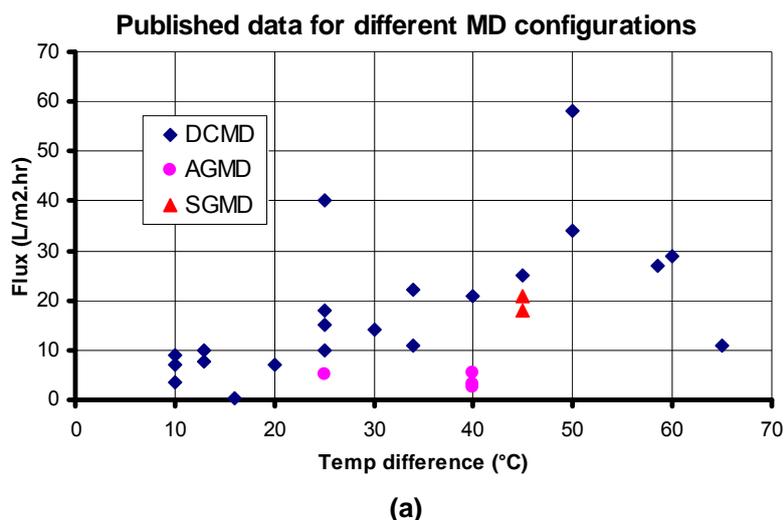
With the quality and quantity of fresh water resources becoming an ever greater problem world-wide, interest in low-cost desalination is growing. As a consequence, membrane manufacturers are now seeking to develop membranes specifically for MD. The goal of this study is to compare a selection of newly available membranes against typical performance data reported in the scientific literature. Assessment of membrane performance takes the form of desalted water production rate generated from brackish or saline water, with respect to potential water recovery options for RO concentrate streams. Laboratory scale equipment with a membrane size of 0.014 m<sup>2</sup>, along with a 0.35 m<sup>2</sup> hollow fibre element, were evaluated for overall water production rate and fouling tendency.

### 1.3 Literature Review

#### 1.3.1 Laboratory Scale Studies

A survey of published MD research [12-34] has found large variation in reported MD flux within each of the different configurations, therefore comparisons should be made within each of the MD modes. Flux measurements using a DCMD configuration, which theoretically should achieve higher flux than other MD forms and are studied most frequently, appear in the literature in the range of 3.6 to 40 L/m<sup>2</sup>hr, with the spread being strongly dependant on input temperature difference used.

The next most widely studied mode is VMD with flux values of 0.23 to 36 L/m<sup>2</sup>hr reported, with one study recording a flux of 50 L/m<sup>2</sup>hr. Sweep-gas MD appears to be investigated rarely but flux measurements of approximately 20 L/m<sup>2</sup>hr are reported. For the AGMD configuration, a few commercial organisations are developing the technology and already have equipment on the market (see below). Presumably, this mode has attracted commercial support due to the high energy efficiency of the AGMD process and its ability to capture latent heat to preheat the feed stream. Nevertheless, AGMD appear in the literature with flux in the order of 3 to 5 L/m<sup>2</sup>hr. See appendix 3 for a survey of published MD studies with flux, membrane type, input temperatures and associated data. Figure 3 displays flux data of many published studies against temperature difference used in their testing. Figure 3a shows water flux against temperature difference for the three MD modes that rely on a  $\Delta T$ , whereas Figure 3b show flux against inlet feed temperature of a VMD process.



**Figure 3 (b)** Flux data from published literature

Although the results are very encouraging, it should be noted that these research studies were performed on a laboratory or small pilot scale equipment, and that MD experiments often face difficulty when scaling-up to larger on-site processes. As stated above, temperature polarisation within a MD module limits flux or energy efficiency, and the elevation to larger equipment tends to exacerbate this effect.

### 1.3.2 Pilot Scale Studies

The Sandia National Laboratories (USA) [35] released an in-depth report regarding desalination techniques, both those currently available and technologies that are still in development. Together with the Bureau for Reclamation (USA) this organisation identified and evaluated two of the MD configurations – VMD and AGMD for possible further development.

In their report that dealt with the VMD process [36], porous hydrophobic hollow fibre polypropylene membranes were used for the desalination of brine solutions. These fibres were with or without nonporous/microporous coating of silicone polymer or a fluoropolymer. The number of fibres in a module varied between 78 and 6,000 (membrane area 0.023 to 0.96 m<sup>2</sup>). The hot water/brine feed temperature ranged between 40 and 91°C. Using a small parallel flow module having an ultra-thin silicone coating, at high feed velocities through the fibre lumen at 91°C a water permeation flux as high as 15 L/m<sup>2</sup>h was achieved. No leakage of salt or water was encountered in extended use of these modules in VMD configuration.

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In a later study, also sponsored by the Bureau for Reclamation (USA) [37], an AGMD process to desalinate brackish and saline water was investigated. This research was done on a commercially available MD module of 2.94 m<sup>2</sup> membranes area manufactured by Swedish firm Scarab HVR that produces complete air-gap MD systems. Heating and cooling for the MD module were supplied from the El-Paso solar pond. Desalted water production per unit area of membrane surface ranged from 1 to 6 L/m<sup>2</sup>hr in their study. Very high quality distillate was produced even from nearly saturated brine solutions. However, distillate quality was found to be primarily dependent upon the degree of wetting of the membrane. This study reported that wetting was a function of pressure drop and the system was relatively sensitive to pressure spikes through inattentive operation.

Another AGMD system that was not reported by the above bodies but which appears in international magazines is the Memstill technology developed by TNO, The Netherlands. In this process cold seawater flows through a condenser with non-permeable walls, gaining some heat from the condensing permeate. The flow then passes through a heat exchanger where additional heat is added to the feed and then into the membrane evaporator. The wall of the evaporator is a micro-porous hydrophobic membrane through which water vapour can diffuse and dissolved salts are retained. Keppel Seghers, who partnered the development of this technology, report that a series of pilot plants are in production, each delivering in the order of 3 L/m<sup>2</sup>h with low input energy. Plans are to report details of these trials at an international conference in 2008 (International Congress on Membranes and Membrane Processes 2008) [38].

## 2 MATERIALS AND METHODS

### 2.1 Equipment

This study sought to investigate desalination performance of MD processes with a series of membrane types, pore sizes and membrane areas. For small laboratory-scale experiments of flat sheet membranes in DCMD mode, a module with nominal membrane area of 0.014 m<sup>2</sup> was used. To test hollow fibre membranes, a cylinder type module was employed ("tube and shell") with an effective membrane area of 0.35 m<sup>2</sup>. These modules were installed one at a time onto a test apparatus configured with two controllable flow path circuits; one heated by an electrical heater, the other cooled by a refrigerated water bath. Permeate flux through the membrane was measured by monitoring the weight gain of the cooled water circuit, and salt rejection of the process was determined by monitoring the electrical conductance of this circuit. Section 2.1.2 and 2.1.3 below outlines the equipment used and measurements made.

#### 2.1.1 Membranes

Amongst the critical criteria for a membrane to function well in an MD application is high hydrophobicity, therefore PTFE membranes with their very high contact angle measurements are generally those of first choice. As such, PTFE flat sheet membranes were obtained from GE Osmonics (Minnesota, USA), Membrane Solutions (Shanghai, China), and Ningbo Chang-Qi Porous Membrane Technology (Ningbo, China). Most membranes were supplied bonded to a support such as non-woven fabric or scrim material (a thin, open weave polymer fabric-like material), however, unsupported PTFE membranes were also investigated. A hydrophobic hollow fibre membrane was also evaluated. A commercially available (GE Osmonics) hydrophobic microfiltration membrane of PVDF material was tested for MD performance as a comparison. The following sections list the membranes used and some of their physical properties.

**Table 1** Flat sheet membrane characteristics.

Material	Nominal pore size (µm)	Support material	Thickness (mm)		Provider	Contact Angle (°)	Labelled
			Total	Active layer			
PVDF	0.30	fibre	0.175	0.045	GE Osmonics	90 ± 5	GE 0.30 µm
PTFE	0.22	scrim	0.110	0.048	GE Osmonics	150 ± 10	GE 0.22 µm
PTFE	0.30	PP fibre	0.220	<0.004	Chang-Qi	142 ± 10	Ch 0.30 µm fibre
PTFE	0.42	scrim	0.115	0.010	Chang-Qi	135 ± 10	Ch 0.42 µm
PTFE	0.45	fibre	0.160	0.005	Membrane Solutions	141 ± 10	MS 0.45 µm fibre
PTFE	0.45	scrim	0.093	0.011	GE Osmonics	150 ± 10	GE 0.45 µm
PTFE	0.45	scrim	0.150	0.005	Membrane Solutions	141 ± 10	MS 0.45 µm
PTFE	0.45	unsupported	0.010	0.010	Membrane Solutions	n/a	MS 0.45 µm NS
PTFE	1.0	scrim	0.068	0.006	GE Osmonics	150 ± 10	GE 1.0 µm
PTFE	1.0	scrim	0.075	<0.004	Membrane Solutions	135 ± 10	MS 1.0 µm
PTFE	2.00	PP fibre	0.300	<0.004	Chang-Qi	140 ± 10	Ch 2.0 µm fibre

### 2.1.1.1 Flat Sheet Membranes

Table 1 lists the flat sheet membranes used in the study. Pore size was provided by the supplier and thickness measurements were made using a micrometer (the three thinnest membranes were recorded as <0.004 mm as accurate gauge measurements could not be made below 0.005 mm with the equipment available). An estimate of contact angle was made by measuring the angle of water droplets resting on the membrane surface. Actual effective membrane area was 0.014 m<sup>2</sup>.

Before flat sheet membranes were used in the MD apparatus a consistent pre-treatment procedure was followed to ensure the removal of preserving agents. Membranes were first completely wetted by soaking in ethanol followed by soaking in deionised water for a minimum of 16 hours. Immediately before use, membranes were again immersed in ethanol to displace as much water as possible then dried in a vacuum oven for 45 minutes at 34-45°C.

### 2.1.1.2 Hollow Fibre Membrane

A hollow fibre module was obtained from a supplier with membrane fibres already mounted in a rigid plastic pipe. Specifications of the hollow fibre module, provided by the supplier, are listed below.

Effective membrane area	0.35 m <sup>2</sup>
Effective fibre length	510 mm
Fibre O.D. / I.D.	0.63 / 0.3 mm
Nominal pore size	0.23 µm
Packing density	24%

### 2.1.2 DCMD Flat Sheet Module, 0.014 m<sup>2</sup> Membrane

The 0.014 m<sup>2</sup> flat sheet DCMD apparatus consisted of a GE Osmonics SEPA CF module holder with a polypropylene membrane module mounted horizontally. The module was equipped with a flow path cavity on each side of the membrane for hot feed and cold permeate flows. Each cavity was fitted with a mesh spacer of 0.3 mm depth to maintain uniform flow path dimensions and provide turbulent flow conditions over each face of the membrane. In all performance testing the active PTFE layer faced the feed (brine) stream.

Both hot feed and cold permeate cycles were driven by two digitally controlled peristaltic pumps in a counter-flow arrangement through the membrane module. The cavity dimensions and mesh spacers were identical for both sides of the membrane, so although flow rates were varied, feed and permeate flows were matched. The hot cycle drew feed solution from a reservoir, through a heater, to the 'hot inlet' of the membrane module. The 'hot outlet' was returned to the feed reservoir to complete the cycle. The cold cycle drew deionised water from a reservoir placed on an electronic balance, through a chiller, to the 'cold inlet' of the membrane module. The 'cold outlet' was returned to the permeate reservoir, via an electrical conductance meter, to complete the cycle. Heating and chilling was supplied by two programmable laboratory water baths. Reservoirs and all piping were of stainless steel construction and flexible hosing were PVC and silicon material. Figure 4 shows a photograph of the MD equipment with the 0.014 m<sup>2</sup> flat sheet module installed (reservoirs and heating and chilling baths not in view) and Figure 5 shows a schematic diagram of the apparatus.

Data from the MD apparatus was logged every 10 minutes, including four temperatures (hot feed in, hot feed out, cold permeate in, cold permeate out), weight gain of the permeate reservoir, electrical conductance of the permeate, and module inlet pressures.



Figure 4 MD apparatus with 0.014 m<sup>2</sup> flat sheet module installed

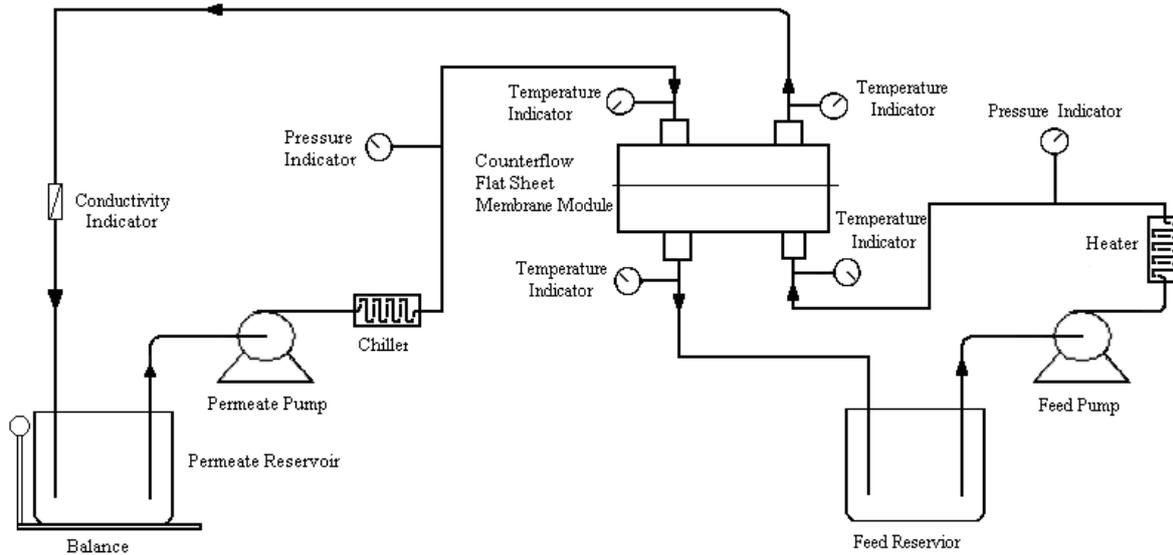
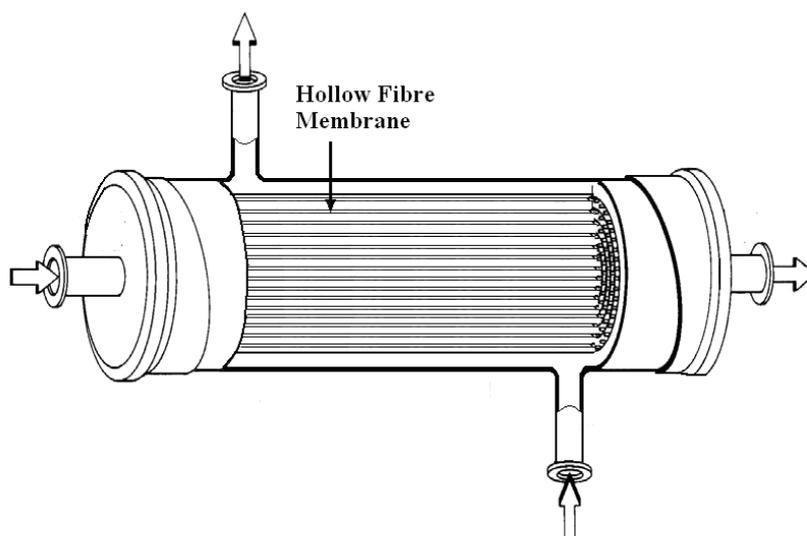


Figure 5 Schematic diagram of DCMD apparatus

### 2.1.3 DCMD Hollow Fibre Module, 0.35m<sup>2</sup> Membrane

A hollow fibre module was obtained that was equipped with approximately 378 fibres housed in a 25 mm polyethylene pipe with lumen inlet and outlet ports, and shell inlet and outlet ports, as displayed in Figure 6. The module was installed in the MD apparatus, in two counter-flow configurations; (1) brine feed into the fibres, cold permeate cycle across the shell side, and (2) cold permeate cycle into the fibre, brine feed across the shell side. The flow path was identical to that described in Figure 4 except the hollow fibre module directly replaced the flat sheet module in that diagram.

## MEMBRANE DISTILLATION OF BRINE WASTES



**Figure 6** Hollow fibre module

### 2.2 Feed solutions

Feed waters for desalination evaluation were prepared or obtained as outlined below:

Pure saline water (10,000 mg/L)

This feed solution was prepared by dissolving analytical reagent NaCl (Merck, Australia) in analytical grade deionised water generated from an Elix 3 Water Purification System (Millipore, USA)

Synthetic Seawater (35,000 mg/L)

This feed solution was prepared by dissolving Sea Salts (Sigma, Australia) in deionised water. See Appendix 2 for typical analysis.

RO Concentrate (3,300 mg/L)

This was obtained from Edenhope Water Treatment Plant, Edenhope. Analysis of the concentrate is listed in Appendix 2, and was reported to contain the antiscalant Flocon 135.

Botany Industrial Park groundwater (19,000 mg/L)

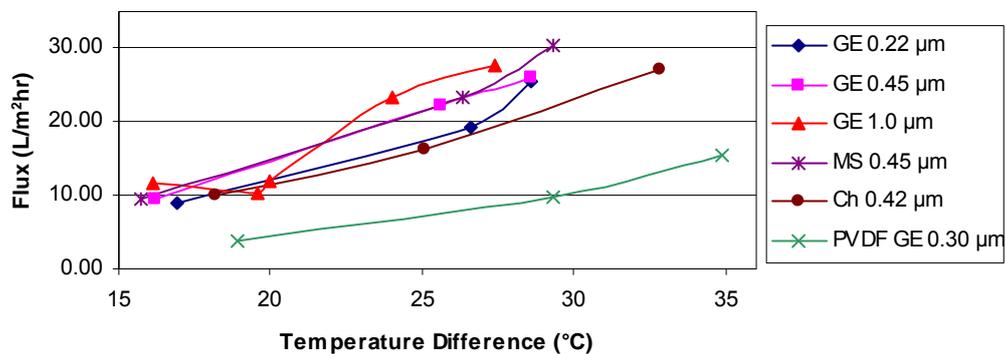
This was obtained from Orica's groundwater treatment plant in Botany, NSW. The sample was taken from the reject stream of the second stage of RO after a water recovery of approximately 90% had been achieved, and was reported to contain PermaTreat PC-191T antiscalant. The sample was filtered through No1 paper prior to use. Analysis of the filtered concentrate is listed in Appendix 2.

### 3 RESULTS AND DISCUSSION

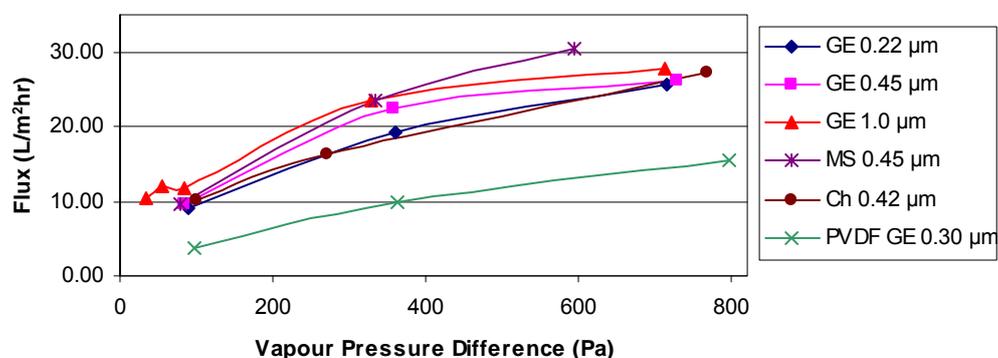
Within an MD process, water flux is known to be proportional to the vapour pressure difference on either side of the membrane, which is imposed by a temperature difference in DCMD. Further, in the DCMD configuration the temperature difference is obtained not only from input temperatures, but also the means of controlling temperature polarisation. A common method of deriving a sufficient  $\Delta T$  is to maintain as high a flow rate as possible across the membrane. However, high flow rates also generate high pressures within the module which lead to liquid feed entering the membrane pores and causing salt to pass to the permeate side. Therefore, membrane hydrophobicity and physical characteristics, along with flow rate through the membrane module and input temperatures are key parameters of MD performance. With these parameters in mind, the following series of experiments sought to explore the limitation of the new membranes with respect to salt rejection and overall water flux.

#### 3.1 Basic Performance Testing

Figures 7 & 8 display flux measurements of the various 0.014 m<sup>2</sup> flat sheet membranes tested with a feed solution of 10,000 mg/L NaCl at a series of different input temperatures under the same flow conditions. The same data are expressed as flux as a function of average temperature difference (Figure 7), and as a function of average vapour pressure difference (Figure 8). It is widely understood that application of a temperature difference across a MD membrane will induce water vapour to pass and some amount of permeate to be generated. Furthermore, developing significant temperature differences should lead to greater desalination production rates. However, the actual driving force for MD is the vapour pressure difference across the membrane, which is induced by this temperature difference. The relationship between flux and  $\Delta T$  shows the expected trend but with significant deviations, whereas the relationship with  $\Delta P$  shows a correlation closer to Schofield's model (2). This is reinforced by the trend-line of the GE 1.0  $\mu\text{m}$  data, which were performed at five temperature conditions.



**Figure 7** Flux vs temperature difference.  
Flow = 400 mL/min. Feed = 10,000 mg/L NaCl



**Figure 8** Flux vs vapour pressure difference.  
Flow = 400 mL/min. Feed = 10,000 mg/L NaCl

### 3.2 Effect of Membrane Support Material

The very thin film 0.014 m<sup>2</sup> flat sheet membranes used in the study were obtained bonded to some form of support material, presumably to protect the fragile active layer. However, the support material can impede flow along that membrane face and increase temperature polarisation on one side of the system. To test this, membranes of the same pore size with different support materials, and one unsupported membrane, were tested for water flux in the MD apparatus. Table 2 lists the flux and rejection results, which indicate the fibre backing appears to inhibit flux, possibly due to the above-mentioned temperature polarisation.

The unsupported membrane did not perform well in the apparatus. Within minutes of the experiment starting, and still at an initial 250 mL/min flow rate, both feed and permeate inlet pressures were at 30 - 40 kPa (normally inlet pressures are <10 kPa at 400 mL/min). At the conclusion of the experiment the membrane was found to have deformed and been forced into the mesh spacers by the impact of feed and permeate flows. Potentially, this could be overcome by a different module design but further work on unsupported membranes was not attempted.

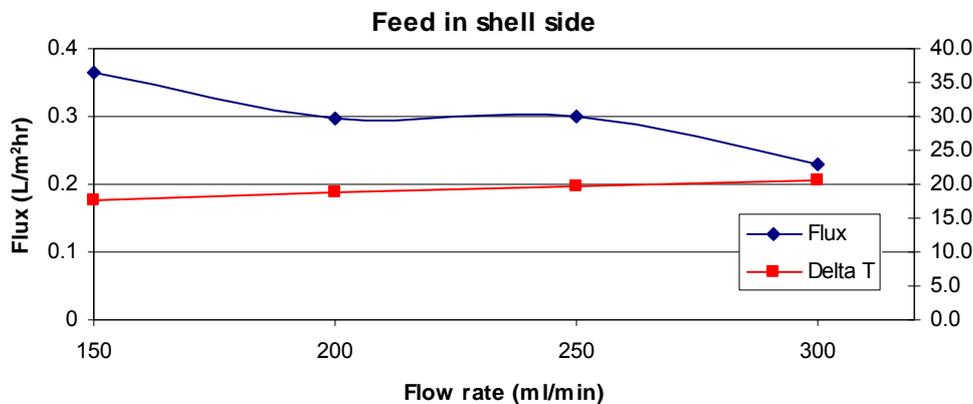
**Table 2** Flux using different support materials.  
 Flow = 400 mL/min. ΔT = 25°C ± 1 (input temps 60 / 20°C).  
 Note: For the unsupported membrane, flow = 250 mL/min.

Membrane type (all PTFE)	Support layer	Active layer thickness (mm)	Flux (L/m <sup>2</sup> hr)	% Rejection
GE 0.45 μm	scrim	0.011	22.3	99.99
MS 0.45 μm	scrim	0.005	23.4	99.62
MS 0.45 μm fibre	fibre	0.005	13.7	99.95
MS 0.45 μm NS	unsupported	0.010	11.6*	99.55

\* membrane deformed in test cell and blocked the flow path.

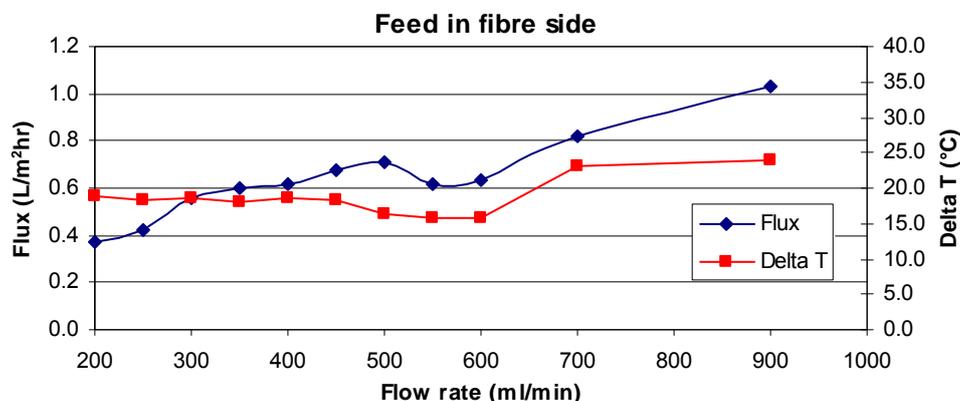
### 3.3 Hollow fibre module.

Hollow fibre membranes with their very high surface areas and compact module dimensions may be beneficial for MD applications. On the other hand, the narrow internal diameters (0.3 mm) would develop significant pressure when trying to attain useful flow rates, which would potentially wet the membrane. With this in mind, experiments using the hollow fibre module were initially configured so the permeate cycle was passed through the fibres and the hot feed was introduced into the shell side. Figure 9 shows flux and ΔT at the different flow rates attempted. With the particularly low water flux experienced in this configuration, feed into the fibre side and permeate cycle in the shell side was also tested. Figure 10 shows the results in this configuration with flow rates up to the capacity of the equipment. This resulted in inlet feed pressures of approximately 60 kPa, but salt rejection remained >99.8%.



**Figure 9** Flux vs flow rate for hollow fibre module (feed in shell side).  
 Input temps; Feed = 60°C, Permeate = 20°C.  
 Feed solution = 10,000 mg/L NaCl. All salt rejections were >99.8%

Reasons for these low water flux results may be attributed to several reasons. Temperature polarisation was a possible cause, so too was the generally thick wall dimensions of the fibres. The flow rate of 900 mL/min through the fibre's bore results in a linear velocity of 1.1 m/s, and although definitive velocities to overcome temperature polarisation are not known, this amount of flow would appear to be sufficient. However, when comparing the fibre thickness of 0.165 mm with those of flat sheet membranes (0.005 to 0.048 mm), the increase in vapour flow path is potentially of most significance.



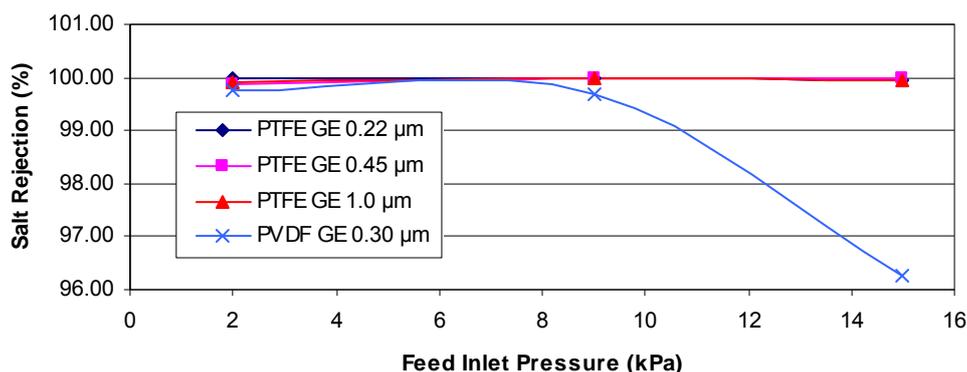
**Figure 10** Flux vs flow rate for hollow fibre module (feed in fibre side).  
Input temps; Feed = 60°C, Permeate = 20°C.  
Feed solution = 10,000 mg/L NaCl. All salt rejections were >99.8%

### 3.4 Membrane Hydrophobicity

Figure 11 shows the three 0.014 m<sup>2</sup> flat sheet PTFE membranes of different pore size, with contact angles measured to be 150 ± 10°, achieved salt rejections greater than 99.8% at all module pressures. Whereas a PVDF membrane, of similar pore size and a contact angle of 90 ± 5°, showed slightly lower but comparable salt rejection at lower pressures. However, at the higher flow rate (and module pressure of 15 kPa) some salt passage occurred resulting in a rejection of 96.3% after 5 hours. This was most likely due to membrane wetting from feed water intrusion. This level of rejection did not reach a plateau and would be expected to decline if the experiment continued. For reasons outlined in later sections (3.6), advantages to the MD process are gained by high feed and permeate flow rates through the membrane module. It was therefore appropriate to measure salt rejection of a selection of membranes under conditions of increasing flow rate. Three flow rate increments were carried out; 250, 400 and 550 mL/min, chosen within the limitations of the pumping equipment, that reached feed inlet pressures of 2, 9 and 15 kPa respectively. Both feed and permeate flow rates were mostly similar, with minor differences due to peristaltic pump dissimilarities.

The degree of hydrophobicity a membrane possesses will largely determine its suitability for an MD application. Highly hydrophobic membranes, indicated by a measured high contact angle, will resist wetting and oppose liquid water from entering the membrane pores. However, hydraulic pressure of the bulk water on either side of the membrane can overcome the inherent surface tension and allow feed water to pass through the membrane. As the membrane does not offer any resistance to salt passage, any feed water entering the pores will result in saline permeate and be seen as poor salt rejection. Therefore, the higher the membrane's hydrophobicity, the higher the pressure within a membrane module that can be tolerated before salt rejection is affected. These results indicate the newly available PTFE membranes are able to maintain high salt rejections at the higher pressures associated with desirable flow dynamics.

## MEMBRANE DISTILLATION OF BRINE WASTES



**Figure 11** Salt Rejection at various module flow rates.

Input temps; Feed = 60°C, Permeate = 20°C.

All membranes were from GE Osmonics. Feed solution = 10,000 mg/L NaCl.

### 3.5 Membrane Gauge

Hydrophobic membranes that offer the shortest possible path length for vapour to pass generate highest possible water flux, for a given set of thermal conditions. Therefore membrane thickness, or film gauge, was a parameter that required investigation. Table 3 shows water flux and rejection of seven 0.014 m<sup>2</sup> flat sheet membranes, tested for MD performance under the same temperature and flow conditions, listed in order of membrane gauge. The results indicate that some correlation exists between water flux produced and either membrane gauge or pore size. However, the experiments undertaken could not conclude which membrane characteristic was the greater influence. Unfortunately, methods of manufacture commonly used for porous films draw down membranes of small pore size to generate membranes of larger pore at a thinner film gauge. As such, membranes of fixed pore size at varying gauges could not be sourced. Similarly, membranes of constant gauge and different pore sizes were also unavailable at the time of this study.

**Table 3** Comparison of membrane gauge with flux and rejection.

Input temps; Feed = 60°C, Permeate = 20°C. Feed solution = 10,000 mg/L NaCl.

Membrane type (all PTFE)	Active layer thickness (mm)	Flux (L/m <sup>2</sup> hr) at temp.; Feed = 60°C, Permeate = 20°C	% Rejection
GE Osmonics 0.22µm	0.048	19.3	99.99
GE Osmonic 0.45µm	0.011	22.3	99.99
GE Osmonic 1.0µm	0.006	23.5	99.99
Membrane Solutions 0.45µm	0.005	23.2	99.64
Membrane Solutions 1.0µm	<0.004	-12	nil
Chang-Qi 0.30µm	<0.004	-16	nil
Chang-Qi 2.0µm	<0.004	-32	nil

Although a direct comparison of flux with pore size or gauge could not be made, the striking aspect is that very thin membranes appear to allow salt to pass from feed to permeate, irrespective of pore size. This was concluded by witnessing the same behaviour for all three of the thin film membranes tested. The observations were; as the experiment started the permeate conductivity increased rapidly indicating salt was passing from the feed, and the permeate reservoir lost weight indicating negative flux was occurring. Speculation was that feed and permeate streams were in contact through the membrane and liquid feed was mixing with the permeate. Furthermore, there appeared to be a net flow from permeate to feed due to osmosis. A possible explanation for this observation was that liquid

water routinely protrudes some distance into membrane pores without effect, but the use of very thin films allows the protrusion from both sides of the membrane to meet and ultimately mix. Further work is underway to confirm this by theoretical calculation, but is not currently available for this report.

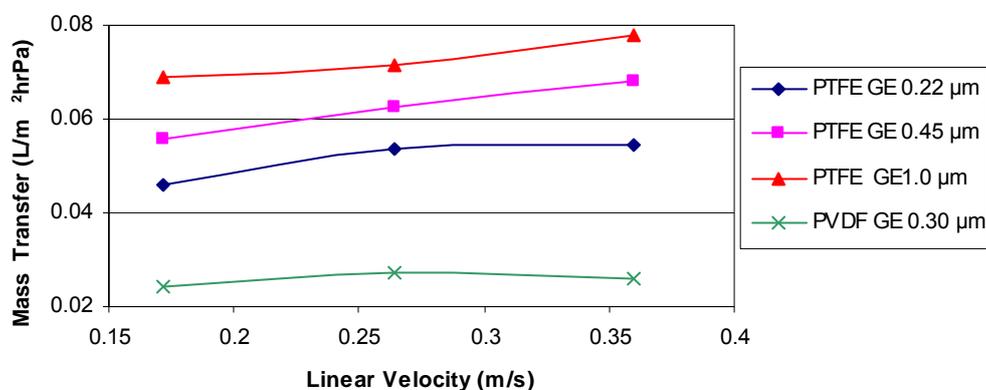
Another interesting observation of membrane gauge that was made during the course of these experiments was the apparent low durability of thin membranes. Whereas a single specimen of the GE Osmonics 0.45  $\mu\text{m}$  membrane (0.011 mm gauge) was used reliably in the MD apparatus in excess of 12 times, the equivalent 1.0  $\mu\text{m}$  membrane (0.006 mm gauge) failed after 5 uses. Inspection of this 0.006 mm membrane indicated feed water was passing through a wear-line where the module o-ring pressed against the active layer. Therefore, from this limited data set it can be surmised that PTFE membrane gauges in the range 0.010 mm are appropriate for MD applications. Whereas thicknesses much above that may limit water flux, and below that are potentially too delicate for extended use.

### 3.6 Role of Linear Velocity

As stated earlier, water flux of a DCMD system is proportional to the temperature difference at the membrane barrier layer (i.e. at both membrane faces) and the effect of temperature polarisation lowers that  $\Delta T$ . Therefore, successful DCMD operation requires an efficient method of moving the hot feed from the heating device to one face of the membrane, and cold permeate to the other. The method of choice is to provide highly turbulent flow across the both membrane faces. This is achieved by driving feed and permeate streams at high volumetric flow rates through mesh material placed against both faces of the membrane (known as cavity spacers). Therefore for a given spacer material, membrane barrier layer turbulence and temperature polarisation is related to linear velocity across the membrane.

Figure 12 shows the results of four different 0.014  $\text{m}^2$  flat sheet membranes tested in the MD apparatus at flow rates of 250, 400 and 550 mL/min, which represent velocities of 0.17, 0.25 and 0.35 m/s respectively (calculated using the Arden Buck equation, given in Appendix 1), based on module cavity and spacer volume measurements. The same feed temperatures were used for all these tests, and flux measurements were converted to mass transfer,  $\text{L}/\text{m}^2\cdot\text{hr}\cdot\text{Pa}$  (discussed in the section 3.1 earlier), to correct for slight differences of input temperatures. Results indicate that although the 0.45 and 1.0  $\mu\text{m}$  membranes demonstrate the expected rise in flux with increasing module velocity, the 0.22  $\mu\text{m}$  and the PVDF membranes may be flux limited by other conditions. As outlined in section 3.5 (membrane gauge) and section 3.2 (support layer) are characteristics that may restrict improvements in flux with an increase in  $\Delta T$  at the barrier layer.

The overall flat curves in Figure 12 may be symptomatic of generally poor turbulence conditions within the membrane module. As the upper limit of volumetric flow rate is governed by the pressure within the module that the membrane's LEP can withstand, the other parameter controlling turbulence is spacer design. Different mesh spacers were not explored for this study, and it remains an area that requires examination.



**Figure 12** Flux (mass transfer) vs velocity.  
Input temps; Feed = 60°C, Permeate = 20°C.  
Feed solution = 10,000 mg/L NaCl.

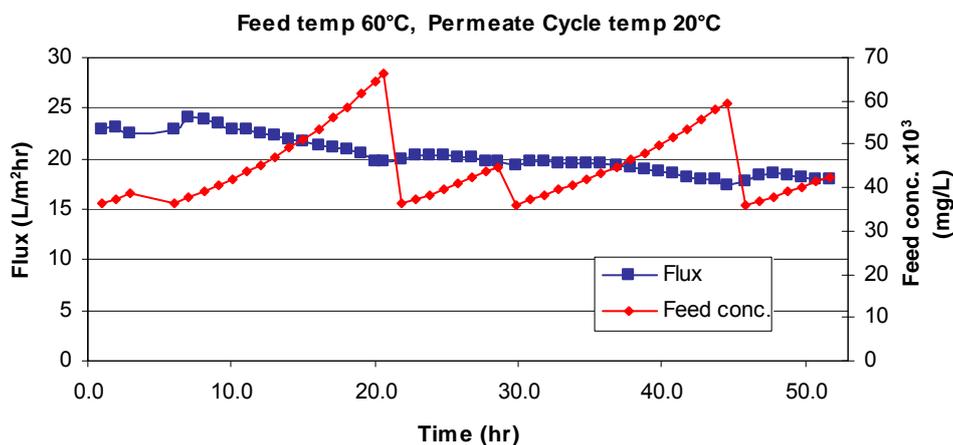
### 3.7 Tests with Real Waters and Fouling.

Similar to other membrane filtration applications MD is sensitive to membrane fouling, which is most likely exhibited as a lowering of flux or membrane wetting leading to low salt rejection, or both. In MD applications that concentrate feed solutions to high salt concentrations, fouling could potentially be from precipitation of low solubility salts onto the membrane that causes pore blockage or reduces the membrane's effective area.

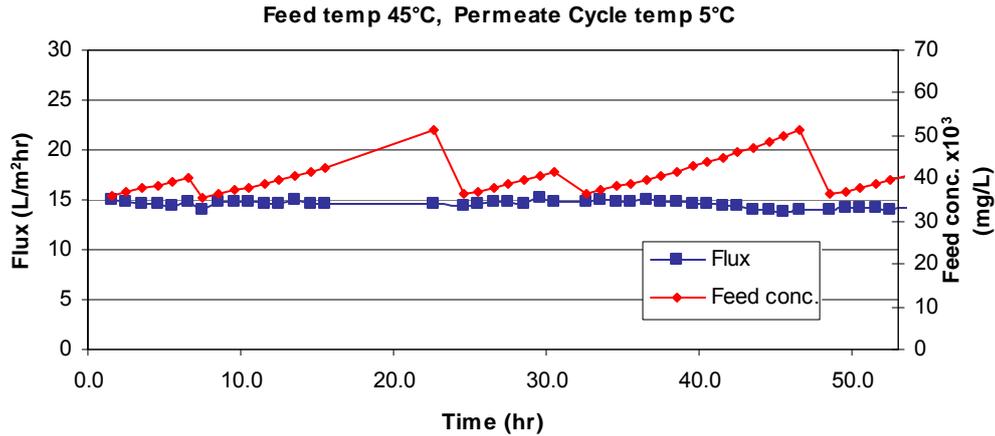
The following sections introduce realistic feed solutions in the form of synthetic seawater (35,000 mg/L) and two highly saline ground water sources obtained from operating RO plants. The membrane used for these fouling tests was the GE Osmonics 0.45  $\mu\text{m}$  PTFE flat sheet (0.014  $\text{m}^2$ ) with scrim backing. This membrane displayed good water flux and was found to be more durable than the 1.0  $\mu\text{m}$  equivalent.

#### 3.7.1 Sea Salts

To evaluate fouling potential, synthetic seawater was used as feed for the flat sheet DCMD apparatus and was operated continuously for approximately three days. Figures 13 & 14 show the flux profile at both high and low temperatures along with feed concentration, which was prepared from sea salts at 35,000 mg/L. One experiment used an inlet feed temperatures of 60°C, and 20°C for the permeate cycle. To demonstrate potential benefits of a low temperature MD regime, the second experiment used a feed of 45°C and a permeate cycle of 5°C. To extend the run length and to approximate constant feed, the permeate was periodically returned to the feed reservoir which can be seen by the return of feed concentration to the initial value. The higher temperatures generated an initial flux of 23 L/m<sup>2</sup>hr which declined to 18 L/m<sup>2</sup>hr over 2 days, producing a total of 14 L of permeate. However, the lower temperatures produced a flux of 15 L/m<sup>2</sup>hr, which declined slowly to 13.5 L/m<sup>2</sup>hr and produced 14 L over a slightly longer period. This confirms the principle that the lower vapour pressure difference induced from lower overall temperatures reduces flux, but may support the speculation that this also lowers the degree of scaling. Analysis of the sea salts is given in Appendix 2 but the concentration of the likely scalants were; calcium 400 mg/L, magnesium 1320 mg/L, sulphate 2660 mg/L and carbonate/bicarbonate 200 mg/L.



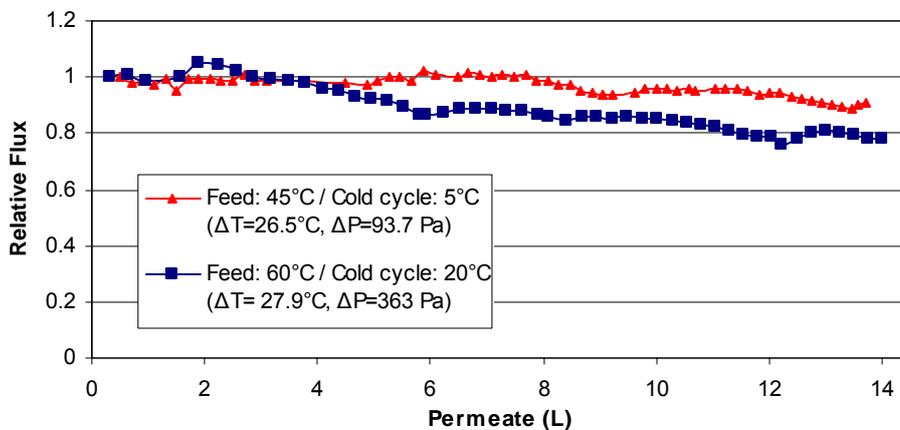
**Figure 13** Fouling associated with Sea Salts (high temp). Flux vs run length. Flow = 400 mL/min. Rejection > 99.99%



**Figure 14** Fouling associated with Sea Salts (low temp).  
Flux vs run length. Flow = 400 mL/min. Rejection > 99.99%

Naturally occurring saline water contains minerals that form sparingly soluble salts at relatively low concentrations. These salts can potentially precipitate on the MD membrane causing a loss of flux in a similar manner to scaling in RO processes. The most common RO scaling problems are caused by precipitation of calcium sulphate, calcium carbonate and silica [39]. Theoretically MD performance is not sensitive to high concentration feed, however, the presence of these sparingly soluble salts may lead to membrane fouling at moderate concentrations.

Figure 15 displays both experiments as relative flux versus permeate produced. This shows that only a small advantage is realised over the period tested, and further testing of longer duration is needed.



**Figure 15** Relative Flux vs Permeate produced from Sea Salts.  
Flow = 400 mL/min. Rejection > 99.99%

Observations of the membrane at the completion of these experiments revealed the presence of a pale cream coloured deposit across a larger proportion of the membrane face. Due to time constraints and the limited number of test membranes, further chemical analysis of this deposit was not performed. However, to assess potential cleaning methods, the membrane that was fouled after the high temperature experiment was soaked in deionised water overnight and returned to the test apparatus. Using the same feed and conditions, the flux was found to be 21 L/m<sup>2</sup>hr which represents a recovery of over half the lost flux. To examine if an acid clean would recover the remain flux, the membrane was soaked in 0.2% hydrochloric acid for 1 hour, water washed and returned to the apparatus. Again using the same feed and conditions, the flux was found to be 20 L/m<sup>2</sup>hr indicating the acid treatment achieved no further flux recovery.

It can be concluded that a low temperature regime MD process, with a sub-ambient permeate cycle stream, may counter potential scaling problems associated with MD's elevated temperature operation.

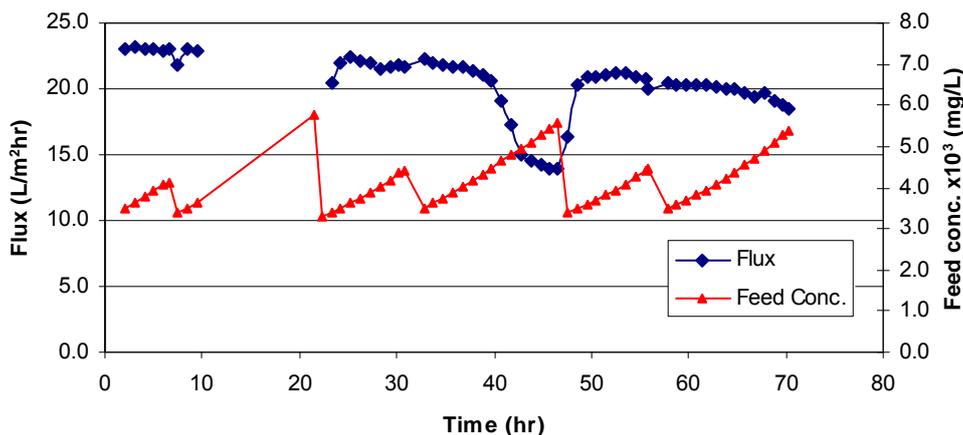
## MEMBRANE DISTILLATION OF BRINE WASTES

However, scaling that occurs from seawater at moderate concentrations appears to be readily removed from the membrane surface and benefits from operating at low temperature may be marginal.

### 3.7.2 Edenhope WTP RO Concentrate

The Edenhope WTP RO concentrate was found to contain approximately 3,300 mg/L TDS with potential scalant levels at; calcium 200 mg/L, magnesium 100 mg/L, sulphate 200 mg/L and carbonate/bicarbonate 350 mg/L (full analysis is listed in appendix 2) but would have contained some level of Flocon 135 antiscalant added in the normal operation of the WTP. With the MD apparatus configured with inputs feed at 60°C and permeate cycle at 20°C, the RO concentrate displayed an initial flux of 23 L/m<sup>2</sup>hr which declined to 18.5 L/m<sup>2</sup>hr in 70 hours (Figure 16). At the completion of this experiment the membrane was inspected for the presence of scale or other fouling but none were visible.

In Figure 16, between hours 10 and 20 of the experiment the data logging equipment failed but the MD apparatus continued to function correctly and a reasonably steady flux profile can be inferred over this period. However, between hours 40 and 50 a rapid flux decline was observed followed by a somewhat steady period then a rapid recovery, which occurred without any operator intervention. This artefact was attributed to an accumulation of air within the module that robbed membrane contact area, reducing flux, then purged of its own accord and the previous flux returned. Although there is no evidence of this supposition, the lack of visible fouling and the observation of air bubbles in the equipment tubing led to this conclusion.



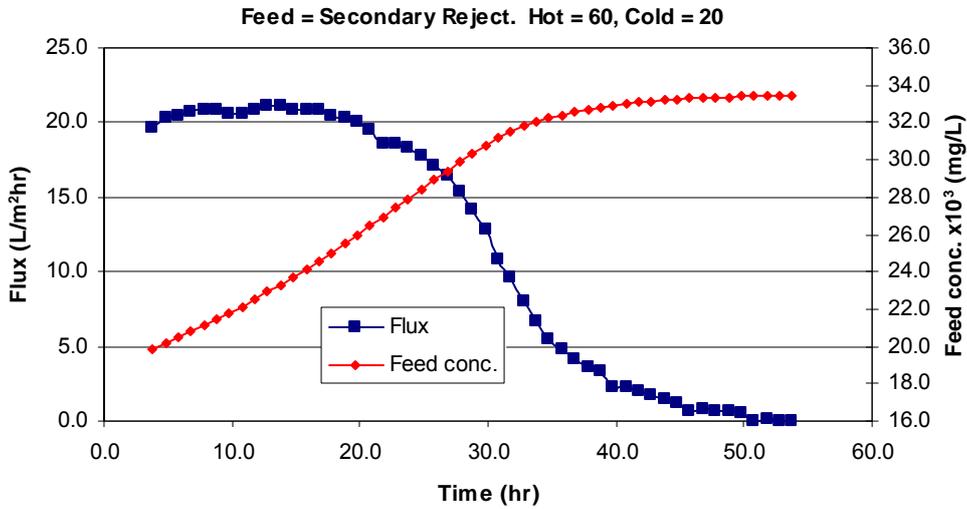
**Figure 16** Fouling associated with Edenhope RO concentrate.  
Flux vs run length. Flow = 400 mL/min. Rejection > 99.98%

### 3.7.3 Botany Industrial Park RO Concentrate

The Botany Industrial Park (BIP) RO plant secondary reject was a straw yellow colour, malodorous (characteristic of hydrogen sulphide), and was found to be approximately 19,000 mg/L TDS with potential scalant levels at; calcium 450 mg/L, magnesium 340 mg/L, sulphate 1500 mg/L and carbonate/bicarbonate 600 mg/L (full analysis is listed in Appendix 2) and would have contained some level of PermaTreat PC-191T antiscalant added in normal RO plant operations. In a similar manner to the sea salts fouling experiments, two different MD temperature regimes were tested. In light of the generally high concentrations of potential scalants in the secondary reject, relative fouling rates at each temperature may be substantially different. To test the full extent of this feed's concentration on MD performance, the feed reservoir was allowed to concentrate until permeate flux approached zero, or the feed volume diminished to the point it could not be pumped.

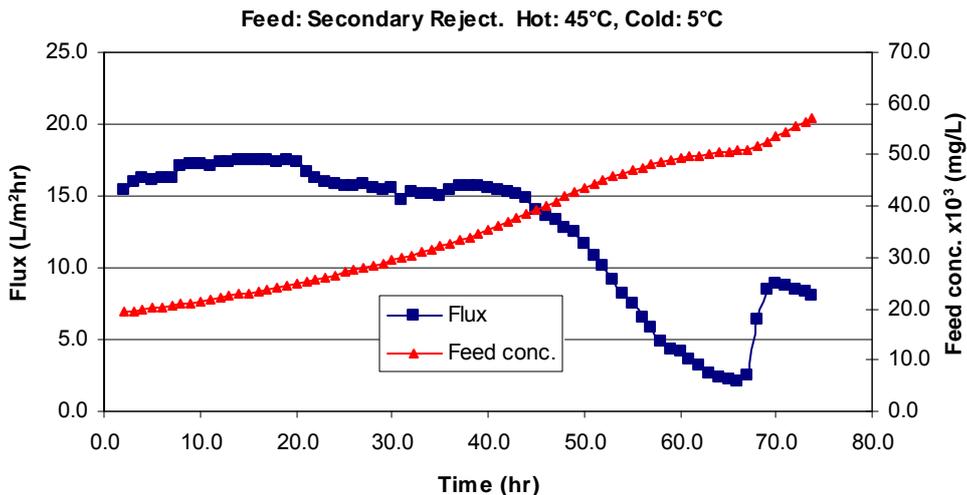
Figure 17 shows that initial flux at inlet feed and permeate cycle temperatures of 60°C and 20°C respectively was 21 L/m<sup>2</sup>hr but started to decline after 20 hours or when the feed concentration reached approximately 27,000 mg/L. The flux decline continued until at 50 hours no further permeate was produced and the feed concentration was in the order of 33,000 mg/L. Inspection of the

membrane at the completion of the experiment revealed a substantial amount of sticky brown material that adhered the cavity spacer to the membrane. Chemical analysis of this deposit will be performed but due to time constraints can not be included in this report.



**Figure 17** Fouling associated with BIP RO concentrate (high temp).  
Flux vs run length. Flow = 400 mL/min. Rejection > 99.6%

The MD performance at the same  $\Delta T$  but lower overall temperatures (input feed and permeate cycle were 45°C and 5°C respectively) was found to be appreciably different. Figure 18 shows initial flux was 16 L/m<sup>2</sup>hr and was sustained between 15 - 17 L/m<sup>2</sup>hr for 43 hours or until the feed concentration had increased to 38,000 mg/L. Flux then declined to approximately 2 L/m<sup>2</sup>hr, when at 67 hours a sudden rise saw flux reach 8-9 L/m<sup>2</sup>hr until the run was terminated at 74 hours due to insufficient feed volume. At this time the feed concentration was 75,000 mg/L, representing a recovery of 67% from the initial 20 L. The reason for the sudden recovery of flux was attributed to a large amount of scale material peeling off the membrane in one piece, exposing a relatively clean area. This theory was supported by recording an increase in feed pressure after the flux improved, consistent with an agglomeration of scale material suddenly blocking the outlet ports of the module. When the experiment was complete, the presence of scale material blocking the outlet ports was confirmed.



**Figure 18** Fouling associated with BIP RO concentrate (low temp).  
Flux vs run length. Flow = 400 mL/min. Rejection > 99.7%

## MEMBRANE DISTILLATION OF BRINE WASTES

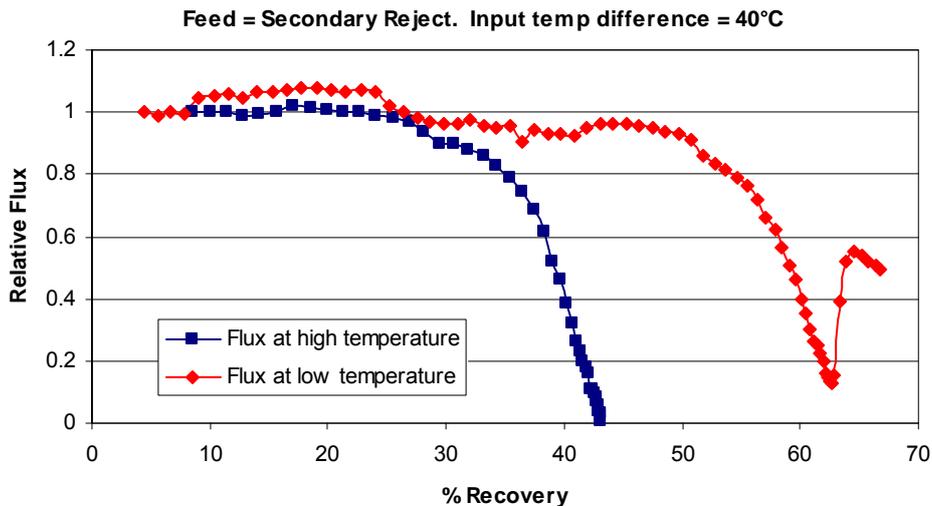
Examination of the membrane after the low temperature experiment showed significantly more brown scale material than was present after the high temperature run. Additionally the brown deposit formed during the low temperature run was loosely packed and was washed from the membrane readily. This contrasts significantly with the sticky material formed at high temperature. Upon re-installation on the membrane after the low temperature experiment, initial performance was fully restored indicating that the membrane itself had not been irreversibly fouled.

Figure 19 displays both sets of data as Relative Flux on a common axis of % Recovery, which demonstrates benefits of operating at lower overall temperatures. Nevertheless, these experiments show a clear fouling response over moderate run lengths due to precipitation. Filtration of the feed before it enters the membrane module may be an effective strategy to minimise this type of fouling.

For the purposes of this experiment, Recovery was calculated according to equation (3)

$$\% \text{ Recovery} = \frac{\text{permeate produced (L)} \times 100}{\text{initial feed volume (L)}} \quad (3)$$

$$\text{Relative Flux} = \frac{\text{Flux (L/m}^2\text{hr)}}{\text{Initial Flux (L/m}^2\text{hr)}} \quad (4)$$



**Figure 19** Relative Flux vs Recovery from BIP RO concentrate

In contrast to the experiments with seawater feeds where only minor differences were observed in membrane fouling due to scale build up at high and low temperatures, there appears to be a significant difference in fouling from groundwater. The results show that the initial high flux was rapidly lost under high temperature conditions (without using any filtration schemes) until the membrane was totally covered with an apparently irreversible foulant. In contrast a low temperature regime, with its initial lower flux, appeared to form larger amounts of precipitate but was able to distil the RO secondary reject at a reasonable flux to greater than 67% recovery.

## 4 SUMMARY AND CONCLUSIONS

The aim of this research project was to examine the feasibility of membrane distillation as a treatment technique to increase water recovery from RO processes. The study compared a selection of newly available membranes against typical performance data reported in the scientific literature. Assessment of membrane performance was in the form of desalted water production rate generated from brackish or saline water, with respect to potential water recovery options for RO concentrate streams. Membranes of 0.014 m<sup>2</sup> dimensions, along with a 0.35 m<sup>2</sup> hollow fibre element, were evaluated for overall water production rate and fouling tendency on a laboratory scale apparatus equipped with electrically heated and cooled process streams.

Performance testing found that at brine feed temperature of 60°C and permeate cycle temperature of 20°C, water flux of 21 to 24 L/m<sup>2</sup>hr and salt rejections greater than 99.9% were achieved with flat sheet PTFE membranes of 0.45 - 1.0 µm pore size. This level of flux appears to be similar to that reported in the scientific literature in the early to mid 2000s. However, more recent studies [11,12,15,30] suggest that with newer generation membranes, flux levels of 50 L/m<sup>2</sup>hr are possible. For a 0.35 m<sup>2</sup> hollow fibre module a flux of 1.0 L/m<sup>2</sup>hr was experienced. Throughout the course of these experiments it became apparent that flow dynamics within the membrane module have a significant bearing on water flux, and this may be the reason for lower than expected flux. The parameter with the most effect on flux was the input temperature difference, but membrane support material and thickness were also important. However, of significant influence to water flux was the degree of turbulence along the membrane surface which greatly affects the temperature difference on each side of the membrane (the temperature polarisation), and therefore the rate that water vapour will pass.

The degree of hydrophobicity a membrane possesses will largely determine its suitability for an MD application. Highly hydrophobic membranes will resist wetting and oppose liquid water from entering the membrane pores. However, hydraulic pressure of the bulk water on either side of the membrane can overcome the inherent surface tension and allow feed water to pass through the membrane. As the membrane does not offer any resistance to salt passage, any feed water entering the pores will result in saline permeate and be seen as poor salt rejection. These results indicate the newly available PTFE membranes are able to maintain salt rejections of greater than 99.9% at the higher pressures (15 kPa) associated with desirable flow dynamics.

Within an MD process, water flux is known to be proportional to the vapour pressure difference on either side of the membrane, which is imposed by a temperature difference. While maintaining all other parameters constant, and keeping permeate cycle temperature to 20°C, feed temperatures of 45, 60 and 70°C saw water flux of 9.6, 22.3 and 26.1 L/m<sup>2</sup>hr respectively. However, successful MD operation requires an efficient method of moving the hot feed from the heating device to one face of the membrane, and cold permeate to the other. The method of choice is to provide highly turbulent flow across both membrane faces by driving feed and permeate streams at high volumetric flow rates through membrane cavity spacers. Therefore, for a given spacer material, membrane barrier layer turbulence and temperature polarisation is related to linear velocity across the membrane. The equipment used in this study was able to produce flow rates of 250, 400 and 550 mL/min, which represent velocities of 0.17, 0.25 and 0.35 m/s across the membrane face. These relatively low velocities, together with suspected poor spacer design are thought to contribute to the generally low flux levels observed.

The very thin film flat sheet membranes used in the study were obtained bonded to various support materials, but these can impede flow along the membrane face and increase temperature polarisation on one side of the system. Evidence of this effect was displayed by the substantially lower flux (13.7 L/m<sup>2</sup>hr) of a 0.45 µm membrane with a fibre backing compared to that obtained from a similar membrane with a scrim backing (23.4 L/m<sup>2</sup>hr). To eliminate all possible effect of backing material an unsupported membrane was tested, but this membrane deformed under the conditions inside the module and was forced into the mesh spacers causing a restriction that led to low flow and high pressure within the module, resulting in low flux. In addition to support material, membrane thickness (gauge) also provides a physical resistance to flux. Hydrophobic membranes that offer the shortest possible path length for vapour to pass generate highest possible water flux, for a given set of thermal conditions. It was found that PTFE membrane gauges in the range 0.010 mm were appropriate for MD applications, whereas thicknesses much above that may limit water flux. Membranes gauges in the

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range of 0.005 mm may produce slightly higher flux but are potentially too delicate for extended use, and those much below that appeared to allow salt passage in DCMD mode.

The conditions examined in this study that produce the highest flux were:

Membrane material	PTFE
Pore size:	1.0 $\mu\text{m}$
Gauge	0.01 mm
Membrane backing	Thin layer of open mesh-like material operated at the highest practical turbulent flow across the membrane surface while maintaining a safe margin below the LEP, and at the greatest temperature difference that economy allows.

Similar to other membrane filtration applications MD is sensitive to fouling, which is most likely exhibited as a lowering of flux or low salt rejection, or both. In MD applications that concentrate feed solutions to high salt concentrations, fouling could potentially be from precipitation of sparingly soluble salts onto the membrane that cause pore blockage or reduce the membrane's effective area. Furthermore, organic material may accumulate on the membrane causing areas of hydrophilicity that reduces salt rejection. In this study, fouling influenced flux, but salt rejection always remained >99%.

Fouling experiments were conducted using synthetic seawater feed and operated continuously for approximately three days. It was concluded that operating the MD process in a low temperature mode, with a sub-ambient permeate cycle stream (inlets were 45°C and 5°C), may counter potential scaling problems associated with MD's elevated temperature operation. However, scaling that occurred from seawater at moderate concentrations appears to be readily removed from the membrane surface and benefits from operating at low temperature may be marginal. RO concentrate from a groundwater source obtained from Edenhope WTP containing approximately 3,300 mg/L TDS and 6.9 mg/L TOC operated for 3 days with only minor flux decline. An initial flux of 23 L/m<sup>2</sup>hr reduced to 18.5 L/m<sup>2</sup>hr over 70 hours with no sign of scale on the membrane at the conclusion of the experiment.

Further fouling experiments were performed on groundwater obtained from the Botany Industrial Park RO plant. The sample was from the secondary reject stream and had a TDS concentration of 19,000 mg/L and a TOC of 64 mg/L. In contrast to the experiments with seawater feeds where only minor differences were observed in membrane fouling due to scale build up at high and low temperatures, there appeared to be a significant difference in fouling from the BIP groundwater. The results show that the initial flux of 21 L/m<sup>2</sup>hr was rapidly lost under high temperature conditions (without using any filtration schemes) until the membrane was totally covered with a recalcitrant foulant. Whereas a low temperature regime, with its initial lower flux of 16 L/m<sup>2</sup>hr, appeared to form larger amounts of loosely packed precipitate but was able to distil the RO secondary reject to greater than 67% recovery.

## 5 RECOMMENDATIONS

This work demonstrates the potential benefits of MD processes for the minimisation of brine wastes. To further develop the technology the following areas require investigation:

- Optimisation of the membrane module and spacer design to maximise water flux for a given input temperature difference. This was identified as a likely source of lower than expected flux.
- Identify the main foulants observed on the membrane and investigate strategies to combat fouling. Removal of precipitate using in-line filtration may significantly improve long term performance. Simple pH control also may show fouling improvements.
- Determine what influence antiscalant chemicals used in conventional RO systems have on the operation of MD membranes with respect to scaling, fouling and flux.
- Scale up to semi-pilot dimensions to investigate limitations of larger membrane areas and include heat recovery equipment to determine economic feasibility of potential commercial installations
- Undertake research into the extent of liquid water protrusion into membrane pores to predict optimal membrane thickness.
- Identify obstacles to further scale-up to a demonstration size MD plant

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## APPENDIX I

### Calculations

$$\text{average temperature difference (}^{\circ}\text{C)} = \left( \frac{T_{\text{feed in}} + T_{\text{feed out}}}{2} \right) - \left( \frac{T_{\text{perm. in}} + T_{\text{perm out}}}{2} \right) \quad (5)$$

where  $T$  = feed or permeate stream temperature ( $^{\circ}\text{C}$ )

$$\text{average vapour pressure difference (Pa)} = \left( \frac{P_{\text{feed in}} + P_{\text{feed out}}}{2} \right) - \left( \frac{P_{\text{perm. in}} + P_{\text{perm. out}}}{2} \right) \quad (6)$$

where  $P$  = water vapour pressure calculated from equation (7).

$$\text{water vapour pressure (Pa)} = 6.1121 \frac{T \left( 18.678 - \left( \frac{T}{234.5} \right) \right)}{257.14 + T} \quad (7)$$

$$\text{Flux (L/m}^2\text{.hr)} = \frac{\text{permeate produced per hr (L/hr)}}{\text{surface area (m}^2\text{)}} \quad (8)$$

$$\text{mass transfer (L/m}^2\text{.hr.Pa)} = \frac{\text{permeate produced per hour (L/hr)}}{\text{surface area (m}^2\text{)} \times \text{vapour pressure difference}} \quad (9)$$

$$\% \text{ salt rejection} = 100 \left( 1 - \frac{\text{permeate conc. (g/L)}}{\text{feed conc (g/L)}} \right) \quad (10)$$

where permeate concentration is calculated from equation (11).

$$\text{permeate conc (g/L)} = \text{conc. in cold cycle (g/L)} \times \left( \frac{\text{total cold cycle vol. (L)}}{\text{permeate produced (L)}} \right) \quad (11)$$

$$\text{linear velocity (m/s)} = \frac{\text{flow rate (ml/sec)}}{\left( \frac{\text{cavity vol. (ml)} - \text{spacer vol. (ml)}}{\text{cavity length (m)}} \right)} \quad (12)$$

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## APPENDIX II

### Water Quality Data

Sea Salts Analysis of the prepared solution.  
(based on manufacturer's salt breakdown data)

Component	Concentration (mg/L)
Total Dissolved Solids (as prepared)	35,000
Sodium	9930
Chloride	17770
Calcium	368
Magnesium	1216
Potassium	390
Sulphate	2450
Carbonate / Bicarbonate	184
Bromide	52
Strontium	8.1
Boron	5.2
Fluoride	0.92
Iodide	0.22
Lithium	0.28
pH	7.8
Total Organic Carbon	trace

RO Concentrate Analysis.  
(analysis performed by MGT Environmental  
Consulting on actual feed solutions)

Component	Edenhope WTP (mg/L)	BIP RO Secondary Reject (mg/L)
Total Dissolved Solids (summed)	3,230	18,700
Sodium	850	6800
Chloride	1500	9900
Calcium	200	450
Magnesium	100	340
Potassium	27	130
Sulphate	198	500
Carbonate / Bicarbonate (as CO <sub>3</sub> )	354	600
Silica	47	59
Phosphate (as P)	0.49	2.1
Aluminium	0.24	0.24
Barium	0.08	0.96
Iron	0.21	0.63
pH	7.4	7.2
Total Organic Carbon	6.9	64

## APPENDIX III

## Literature Flux Data for MD

Direct Contact Membrane Distillation										
Mem type	pore (um)	Feed	Velocity - hot (m/s)	Velocity - cold (m/s)	Feed temp (°C)	Cold temp (°C)	Delta T°C	Initial Flux (L/m <sup>2</sup> hr)	Final flux after x hrs	Reference
Flat sheet, PP	0.2	DI water	n/a	n/a	59	14	45	25	n/a	Criscuoli 08 [15]
HF, PP	0.2	Brine, 400g/L	n/a	n/a	33	17	16	0.3	0.15 in 8 hr	Mariah 06 [26]
HF, PP	0.22	Brine, 200g/L	n/a	n/a	60 - 85	20	40-65	10.8	10.2 in 45 hr	Gryta 06 [19]
Flat sheet, PVDF	0.22	NaCl, 260g/L	0.53	0.53	60	20	40	21	0 on 7 hr	Tun 05 [31]
Flat sheet, PVDF	0.22	NaCl, 260g/L	0.53	0.53	60	30	30	14	0 in 9 hr	Tun 05 [31]
Flat sheet, PVDF	0.22	NaCl, 260g/L	0.53	0.53	50	30	20	7	0 in 38 hr	Tun 05 [31]
Flat sheet, PTFE	0.2	NaCl, 96g/L	n/a	n/a	45	35	10	9	n/a	Martinez-Diez 01 [29]
Flat sheet, PTFE	0.2	NaCl, 96g/L	n/a	n/a	25	15	10	3.6	n/a	Martinez-Diez 01 [29]
Flat sheet, PTFE	0.2	NaCl, 30g/L	n/a	n/a	45	20	25	40	n/a	Hsu 02 [21]
Flat sheet, PTFE	0.2	NaCl, 30g/L	n/a	n/a	45	20	25	18	18 in 160 hr	Hsu 02 [21]
Flat sheet, PTFE	0.2	Raw seawater	n/a	n/a	45	20	25	10	8 in 160 hr	Hsu 02 [21]
Flat sheet, PTFE	0.2	Filtered SW	n/a	n/a	45	20	25	15	11 in 160 hr	Hsu 02 [21]
Flat sheet, PTFE	0.5	Filtered SW	n/a	n/a	40	30	10	7	5 in 160 hr	Hsu 02 [21]
Flat sheet, PVDF	0.45	Water	n/a	n/a	48	14	34	22	n/a	Martinez 01 [27]
Flat sheet, PVDF	0.22	Water	n/a	n/a	48	14	34	11	n/a	Martinez 01 [27]
Flat sheet, PVDF	0.22	NaCl 247g/L	0.145	0.145	79	20.5	58.5	27	7 in 5 hr	Yun 06 [34]
Flat sheet, PTFE	0.2	NaCl 120g/L	0.35	0.35	?	?	13	10.1	n/a	Martinez 06
Flat sheet, PTFE	0.2	NaCl 240g/L	0.35	0.35	?	?	13	7.6	n/a	Martinez 06 [28]
Flat sheet, PVDF	0.11	DI water	1.85	2.92	70	20	50	58	n/a	Srisurichan 06 [30]
Flat sheet, PVDF	0.11	NaCl/CaCl <sub>2</sub> 1g/L	0.23	0.23	70	20	50	34	23 in 9 hr	Srisurichan 06 [30]
HF, PP	0.22	RO permeate	1.35	0.25	80	20	60	29	27 in 700 hr	Gryta 05 [17]
HF, coated PP	0.2	NaCl 10g/L	0.045	0.12	85	17	68	36	54 in 120 hr	Li B. 04 [24]
HF dual layer PVDF	0.41	NaCl 35g/L	n/a	n/a	90	27	63	55.2	n/a	Bonyadi 07 [12]
HF dual layer PVDF	0.41	NaCl 35g/L	n/a	n/a	63	22	41	22	n/a	Bonyadi 07 [12]

HF = hollow fibre

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Air Gap Membrane Distillation									
Mem type	pore (um)	Feed	Velocity - hot (m/s)	Velocity - cold (m/s)	Feed temp (°C)	Cold temp (°C)	Delta T°C	Initial Flux (L/m2hr)	Reference
Flat sheet, PTFE	0.2	NaCl, 30g/L	n/a	n/a	45	20	25	5	Hsu 02 [21]
proprietary		NaCl 35g/L	n/a	n/a	60-70	20-30	40	5.4	Walton 04 [37]
proprietary		NaCl 125g/L	n/a	n/a	50-60	10-20	40	3.2	Walton 04 [37]
proprietary		NaCl 270g/L	n/a	n/a	50-60	10-20	40	2.5	Walton 04 [37]

Sweep Gas Membrane Distillation									
Mem type	pore (um)	Feed	Velocity - feed (m/s)	Velocity - sweep gas (m/s)	Feed temp (°C)	Sweep gas temp (°C)	Delta T°C	Initial Flux (L/m2hr)	Reference
Flat sheet, PTFE	0.2	DI water	0.15	1.5	65	10	55	17	Khayet 00 [22]
Flat sheet, PTFE	0.45	DI water	0.15	1.5	65	10	55	21	Khayet 00 [22]

Vacuum Membrane Distillation							
Mem type	pore (um)	Feed	Velocity - hot (m/s)	Feed temp (°C)	Vacuum (mbar)	Initial Flux (L/m2hr)	Reference
Flat sheet, PP	0.2	DI water	n/a	59	60	50	Criscuoli 08 [15]
HF, PVDF	0.2	NaCl, 120g/L	n/a	25	10	0.23	Wirth 02 [32]
HF, PE	0.1	NaCl, 120g/L	n/a	20	10	0.1	Wirth 02 [32]
HF, PE (prepared in-house)	0.09	NaCl, 35g/L	n/a	60	80	4	Li J. 03 [25]
HF, PE (prepared in-house)	0.07	NaCl, 35g/L	n/a	60	80	2.2	Li J. 03 [25]
HF PP	n/a	seawater	n/a	45	900	3	Xu 06 [33]
HF PP	n/a	seawater	n/a	55	900	5.4	Xu 06 [33]
HF, coated PP (prepared in-house)	0.2	NaCl 10g/L	270	85	800	36	Li B. 04 [24]

HF = hollow fibre



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CRC for Water Quality  
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The CRC for Water Quality and Treatment officially ended in October 2008, and has been succeeded by Water Quality Research Australia Limited (WQRA), a company funded by the Australian water industry. WQRA will undertake collaborative research of national application on drinking water quality, recycled water and relevant areas of wastewater management.

The research in this document was conducted during the term of the CRC for Water Quality and Treatment and the final report completed under the auspices of WQRA.



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