

# **Project Report**

# **Demonstration of Low Maintenance Chemical Free Recycling**

of Secondary Treated Effluent by Ceramic Membranes

A report of a study funded by the Australian Water Recycling Centre of Excellence

### Victoria University September 2014



# Demonstration of Low Maintenance Chemical Free Recycling of Secondary Treated Effluent by Ceramic Membranes

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# About the Australian Water Recycling Centre of Excellence

The mission of the Australian Water Recycling Centre of Excellence is to enhance management and use of water recycling through industry partnerships, build capacity and capability within the recycled water industry, and promote water recycling as a socially, environmentally and economically sustainable option for future water security.

The Australian Government has provided \$20 million to the Centre through its National Urban Water and Desalination Plan to support applied research and development projects which meet water recycling challenges for Australia's irrigation, urban development, food processing, heavy industry and water utility sectors. This funding has levered an additional \$40 million investment from more than 80 private and public organisations, in Australia and overseas.

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# Demonstration of Low Maintenance Chemical Free Recycling of Secondary Treated Effluent by Ceramic Membranes

**Project Final Report** 

Institute for Sustainability and Innovation

September 2014



# Demonstration of Low Maintenance Chemical Free Recycling of Secondary Treated Effluent by Ceramic Membranes

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# **Executive summary**

#### Project purpose

This project was primarily focussed on the technical and economic viability of water recycling using ceramic membrane technology. Water treatment using membrane filtration for potable and recycling purposes is now a common yet still growing treatment technology option. However, the issue of membrane fouling is an ongoing limitation to a membrane process' productivity and profitability, particularly in the context of wastewater reclamation. Meanwhile water reclamation as a strategy in finding alternative water sources, or for compliance with more stringent discharge requirements, is gaining increased interest and importance. Therefore, addressing the membrane fouling issue is critical to progressing low cost future water recycling schemes.

The issues of membrane fouling is normally resolved by using cleaning strategies, but the current generation of polymeric materials impose constraints on the cleaning strategies that can be used as they are more prone to material degradation from chemicals used in water treatment including chlorine and ozone. One approach to relax cleaning constraints is to employ more resilient membrane materials that can withstand a wider variety of cleaning techniques. The membrane can then continue to reliably carry out its function as a barrier to suspended solids and pathogens. Membranes made of ceramic materials are physically robust and largely chemically inert. Ceramic membrane installations dating from 1998 are still in operation today, and have reported no replacement, no membrane breakage, and no loss of flux. Ceramic membranes, however, are more expensive on a per m<sup>2</sup> filtration area basis than polymer membranes. But it is the combination of the ceramic membrane material with an upstream ozonisation step that results in an integrated hybrid process that may control membrane fouling and achieve the gains of lower production costs. The combination of ozone and ceramic membranes is an innovative step for water recycling as ceramics do not degrade in the presence of ozone, but instead may actually assist in the ozone oxidation reactions. Therefore, ozone not only degrades foulants in the incoming water but also acts to constantly clean the membrane surface.

#### Ceramic membrane trial setup

This project featured a trial on a 25 m<sup>2</sup> ceramic microfiltration membrane pilot plant based on the CeraMac process from PWN Technologies. The plant was coupled to an ozone injection system and operated the combined process on municipal secondary effluent from Melbourne Water's Eastern Treatment Plant (ETP). This project seeks to explore the potential for secondary effluent water recycling in terms of cost, reliability and treated water quality when using a hybrid ceramic membrane / ozonisation process. The degree of performance enhancement from combining these two distinct processes forms the main goal of this work. Also of key importance is the relative cost of the new process compared to traditional polymeric membrane plants. Furthermore, an additional benefit of this hybrid process is the potential enhanced deactivation of pathogens found in these wastewaters though much longer contact times. The ozone is the disinfectant and the ceramic membrane holds the viable organisms in the ozone stream for much longer than a typical ozonisation process. This in theory would enhance disinfection of both permeate and reject streams, which potentially expands the options for where reject water can be disposed. The potential enhanced pathogen inactivation was explored as part of this study.

Secondary treated effluent from the ETP is relatively coloured, containing high concentrations of non-biodegradable coloured compounds and with dissolved organic carbon content typically between 10 - 16 mg/L. The effluent for the project had passed through secondary clarifiers and the turbidity was commonly 3 - 5 NTU. This relatively high fouling wastewater represents a good water source to explore the advantages and limitations of this hybrid process. The trial experimental plan was based on performance when ozonisation was used as pre-treatment to the ceramic microfiltration step, with and without in-line coagulation. Filtration performance was evaluated by determining the maximum flux (operating in constant flux mode) that could be sustained over the long term by monitoring transmembrane pressure (TMP) and deducing the rate of fouling. Determination of fouling was based on TMP rise and the estimation of the frequency of membrane clean-in-place routines (CIPs) needed where TMP hits the maximum that can be delivered by the feed pump. Additional tests were carried out to further optimise the conditions and to explore the features of the novel hybrid process.

#### Ceramic membrane sustainable performance findings

The maximum sustainable flux achievable without ozonisation or coagulation prior to the ceramic membrane filtration was 50 L/m<sup>2</sup>.h. When higher flux was attempted, membrane fouling was estimated to require very frequent CIP routines that would be uneconomical to the operator. The addition of in-line coagulation (ILC) with polyaluminium chloride (PACI dosed at 3mg/L as AI) led to enhancement of sustainable flux between 100 L/m<sup>2</sup> and 150 L/m<sup>2</sup>.h. Employing ozonisation before the membrane process resulted in a modest increase in sustainable flux compared to no ozonisation. It was when ozonisation and coagulation were applied together that significant increases in flux were possible. Injection of ozone at the maximum rate of the available equipment saw sustainable fluxes reach 182 L/m<sup>2</sup>.h. Higher fluxes appeared to be very likely but the ozone injection equipment prevented experimentation of this. It was assumed for the costing model that 200 L/m<sup>2</sup>.h was achievable due to the stable operation observed at 182 L/m<sup>2</sup>.h.

#### Pathogen barrier and backwash disinfection

During the trial, the potential of the ceramic membrane as a barrier to pathogens in water recycling schemes was studied. A challenge test program was established to demonstrate the ability of the membrane to reject protozoa, bacteria and virus. Protozoan and bacterial log reduction values (LRV) were estimated based on the challenge particle *E.coli* which was present in the incoming secondary effluent. The virus LRV was estimated using the surrogate MS2 coliphage which was spiked into clean water at high membrane flux. The choice of conditions was aimed at obtaining conservative estimates of the LRVs across the ceramic membrane for protozoa, bacteria and virus. The LRV for bacteria and protozoa, which was inferred from the reduction of *E.coli*, was found to be >3.2, with the ceramic membrane blocking all *E.coli* present in the incoming feed water from reaching the permeate stream (*E.coli* not detected in permeate). The LRV for virus, based on MS2, was conservatively determined to be 4. The concept of backwash disinfection, due to constantly flowing ozone over the ceramic membrane filter cake was confirmed. The study found fewer *E.coli* present in the backwash water than the feed water, despite an theoretical 14-fold concentration increase due to a water recovery efficiency of 93%. The viability of ceramic membranes as a pathogen barrier was therefore demonstrated, together with the unique backwash disinfection feature of the hybrid process.

#### Exploration of high flux mechanisms

This project also sought to explore the mechanisms of the unique hybrid process as there is little fundamental explanation is available the scientific literature. Overlaying TMP profiles of the four pretreatment combinations revealed a clearer illustration of the benefits (displayed below). These graphs show the regular TMP rise of each filtration cycle as the cake accumulates, then a sudden drop as a backwash occurs. The difference between the profiles highlights the incremental effect of each pre-treatment. Compared to no pre-treatment (black line), ozonisation prevented TMP rise between backwash cycles (red line), indicating ozone reduces the fouling nature of the influent. On the other hand, the TMP profile of coagulant pre-treated effluent (green line) displays a parallel TMP rise to no pre-treatment but offset to a lower TMP, indicating more permeable filter cake structure. However, it is the combination of coagulation and ozonisation that show dramatic effects. The TMP profile (blue line) shows no rise per filtration cycle nor does it display a rising trend of long term fouling. The combined pre-treatment will reduce the interaction of both high and low molecular weight compounds that interact with the membrane, allowing for a less dense filter cake at the membrane surface and lower TMP as a result. It must be noted these results were generated at low flux (50 L/m<sup>2</sup>.h) to illustrate the mechanism, and the flat profile of the blue line is not seen at higher flux operation.



TMP profile overlay of no pre-treatment, ozone only, coagulant only and combined ozone and coagulant

#### Cost comparison of ceramic membranes and polymeric membranes

The cost comparison between ceramic and polymeric membrane plants has been completed in the context of water recycling in Australia on a 22 MLD plant basis. It showed that high flux and long life of ceramic membranes are the key features that reduce their CAPEX and OPEX. When coagulant and ozone is used to achieve a ceramic membrane flux of 200 L/m<sup>2</sup>.h the economics of the equivalent process using polymer membranes became comparable. There are slight CAPEX benefits for polymer membranes over ceramic membranes (10.5% cost favourable to polymers), but OPEX showed lower annual operating costs for ceramic membranes as they are replaced less frequently. With these findings it is important to assess the lifetime cost of the two plant options to measure the overall cost differences. Based on the Net Present Value analysis for the 25 year plant life, we see that the lower OPEX of ceramic membranes, with membrane replacement after either 15 years, or lasting the full life of the plant (25 years), is cheaper over the life of the plant. The differences between the two analyses were minor, with the ceramic membrane plant at least 2.3% cheaper compared to the polymer membrane plant. Therefore, the other benefits of ceramic membrane such as high integrity, longevity and chemical robustness can be realised in water recycling schemes while being economically comparable to the equivalent process using polymer membranes. As competitively built ceramic membrane installations are still emerging, the cost of ceramic membrane systems are decreasing. Polymer membrane systems may also experience cost reductions or improved pricing efficiencies over time. Therefore an updated cost model will be required in future due to the cost saving developments in the technologies. However, the cost comparison does indicate that whole of life (NPV) costs for ceramic membrane and polymer membrane systems are comparable, so consideration of both ceramic and polymeric membrane options for projects appears economically justified.

#### Conclusions

This work demonstrated the performance benefits proposed by ceramic membranes in conjunction with ozone and coagulation. High flux operation for challenging waters such as secondary effluent and enhanced total disinfection are unique features which have significant benefits for wastewater recycling and would allow some operational features such as high turn down to be exploited, particularly in smaller plants. While there is potential for reduced chemical cleaning, this would come at the cost of the principle benefit of high flux operation. Regardless which of the above options is chosen, the ceramic membrane's other benefits of robustness and longevity give greater operational freedom and make it a strong option for treatment of challenging waters.

# **Table of Contents**

| 1  |    | Introduction7 |  |      |  |  |  |
|----|----|---------------|--|------|--|--|--|
| 2  |    | Objective7    |  |      |  |  |  |
| 3  |    | Background    |  |      |  |  |  |
| 4  |    | Plant Trial1  |  |      |  |  |  |
|    | 4. | 1             | The Trial Site   | .11  |  |  |  |
|    | 4. | 2             | Pilot Plant  | .13  |  |  |  |
|    |    | 4.2.1         | Ceramic Membrane   | .14  |  |  |  |
|    |    | 4.2.2         | The Installation   | .15  |  |  |  |
|    | 4. | 3             | Experimental Matrix Development  | .18  |  |  |  |
|    | 4. | 4             | Analysis Methods   | .22  |  |  |  |
|    |    | 4.4.1         | Plant Performance Testing  | .22  |  |  |  |
|    |    | 4.4.2         | Chemical Testing   | .22  |  |  |  |
|    |    | 4.4.3         | Microbiological Testing  | .24  |  |  |  |
|    |    | 4.4.4         | Direct Integrity Testing   | .25  |  |  |  |
|    | 4. | 5             | Plant Performance Results  | .27  |  |  |  |
|    |    | 4.5.1         | Membrane Performance with No Pre-treatment (Test Number 1, Table 3)            | .28  |  |  |  |
|    |    | 4.5.2         | Membrane Performance with Coagulant (Test Number 2, Table 3)                   | .29  |  |  |  |
|    |    | 4.5.3         | Membrane Performance with Ozone (Test Number 4, Table 3)                       | . 30 |  |  |  |
|    |    | 4.5.4         | Membrane Performance with Coagulation and Ozonisation (Test Number 5, Table 3) | .31  |  |  |  |
|    |    | 4.5.5         | Effect of Residual Ozone Quenching (Test Number 8, Table 3)                    | . 32 |  |  |  |
|    |    | 4.5.6         | Low Frequency Chemical Cleaning (Test Number 7, Table 3)                       | .34  |  |  |  |
|    |    | 4.5.7         | Organic Matter Degradation   | .34  |  |  |  |
|    |    | 4.5.8         | Pathogen Inactivation (Test Number 9, Table 3)                                 | . 37 |  |  |  |
|    |    | 4.5.9         | Pressure Decay Test Results  | . 39 |  |  |  |
|    | 4. | 6             | Discussion on findings   | .40  |  |  |  |
| 5  |    | Plant         | Costing  | .42  |  |  |  |
| 6  |    | Bene          | fits of the Technology   | .43  |  |  |  |
| 7  |    | Conc          | lusions  | .43  |  |  |  |
| 8  |    | Ackn          | owledgements   | .44  |  |  |  |
| 9  |    | Refe          | rences   | .45  |  |  |  |
| A  | эр | endix         | Α  | .46  |  |  |  |
| At | ta | chme          | nt 1. Cost Comparison Report   | .48  |  |  |  |

# 1 Introduction

Water treatment using membrane filtration for potable and recycling uses are now a common yet still growing treatment option. However, the issue of membrane fouling is an ever present limitation to a membrane process' productivity and profitability. This is particularly the case for wastewaters where fouling can potentially render treatment impractical or very expensive. Yet it is the area of wastewater reclamation that is finding higher demand to meet ever increasing need for alternative water sources or for compliance with more stringent discharge requirements.

Strategies and processes that combat membrane fouling are a major focus of plant designers and operators. With mitigation of fouling comes higher sustainable production rates, therefore smaller sized plants can reach the target capacity at lower capital expenditure. However, fouling control is often achieved through chemical additions which incur on-going costs of materials, facilities and logistics. Therefore, a plant configuration that marries a suitable membrane material with a tailored pre-treatment method may allow minimisation of these chemical costs. While most membrane materials are made from polymers, ceramic materials are attracting interest due to the robustness of the material compared to the conventional polymer types. The versatility of the more robust material gives added flexibility to how they are operated with other chemicals used in the treatment process.

The combination of the ceramic membrane material with an ozonisation treatment step results in an integrated hybrid process that may achieve the gains of higher production or lower cleaning chemical consumption. To investigate the potential of this hybrid process, this work utilized an existing ceramic membrane pilot plant coupled to an ozone injection system and operated the combined process on municipal secondary effluent.

This report is the final report for the project titled "*Demonstration of low maintenance chemical free recycling of secondary treated effluent by ceramic membranes*" which was approved for funding from Australian Water Recycling Centre of Excellence (AWRCoE) in 2011 under its Goal 1 – Demonstrating and Enhancing the Social, Economical and Environmental Value of Water Recycling. The project is a continuation of preliminary proof-of-concept laboratory tests carried out by a Water Research Australia Summer Scholarship student in 2009/2010. The student worked on a project having a collaboration between Eastern Treatment Plant's operator Melbourne Water and Victoria University, which investigated the combination of ceramic membranes and ozone in the laboratory. As a result of its success the decision was made to progress the project to a pilot scale. Consequently the project team expanded to include the developer of the innovative CeraMac ceramic membrane system, PWN Technologies (The Netherlands). Engineering expertise was provided by Black and Veatch and water recycling expertise from water retailer South East Water. Together with the AWRCoE, the team was instrumental in ensuring a successful trial outcome and economic assessment demonstrating the virtues of robust ceramic membrane technology for water recycling in Australia.

# 2 Objective

This project seeks to explore the potential for secondary effluent water recycling in terms of cost, reliability and treated water quality when treatment is provided by ceramic membrane microfiltration combined with an ozonisation advanced oxidation process. The degree of performance enhancement from combining these two distinct processes forms the main goal of this work. Also of key importance is the relative cost of the new process compared to traditional polymeric membrane plants.

Project objectives

- Operate a ceramic membrane system for at least 1 year on secondary effluent;
- Investigate the effect of ozone to increase efficiency with catalytically active ceramic membranes;
- Explore pathogen inactivation and rejection, colour removal, and general TOC reduction by combined ozone and filtration;
- Demonstrate reduced maintenance and cleaning chemical compared to polymer membranes;
- Estimate the economics of the combined treatment system on a \$/m<sup>3</sup> water processed basis and produce an impartial capital and operating cost estimate; and
- Dissemination of outcomes to the Australian water industry.

# 3 Background

Membrane technologies are now widely adopted in water treatment, with most installed membranes using polymeric materials. Virtually all surface waters contain materials that foul membranes, being typically derived from vegetative debris, algal growth and other sources. All water treatment processes using membrane filtration therefore suffer loss of filtration throughput over time due to the membrane becoming fouled. The cost of fouling manifests itself as low production rates and shorter run-lengths between cleaning cycles, along with the direct costs of cleaning chemicals. This leads to larger plant and equipment being required to achieve design capacity, resulting in higher capital costs through larger membrane modules, pumps, equipment and materials and generates larger waste streams. Increased chemical use and membrane replacement frequency contribute towards increased operating costs.

Many low pressure membrane filtration processes for water treatment operate in dead-end mode where periodic reversal of the flowing stream dislodges accumulated sediment with a "backwashing" event. As pressure is controlled to ensure constant flux, fouling is described as a rise in the transmembrane pressure (TMP) required to 'push' water through the fouling layer compared to that of a clean membrane having no fouling layer. However, flux decline due to solution chemistry, feedwater hydraulic effects, or a loose accumulation of solid material are not considered serious fouling if the original flux is recovered when a backwash occurs and clean water is re-applied. This is termed reversible fouling. The condition that results in the more serious irreversible fouling has been defined as the process resulting in the loss of performance of a membrane due to deposition of suspended or dissolved substances on its external surfaces, at its pore openings, or within its pores (Schafer et al., 2001). Irreversible fouling can often only be removed with some form of chemical cleaning of the membranes.

A commonly used method to clean membranes fouled with organic material is to apply a low concentration of oxidising compound, such as sodium hypochlorite at regular frequencies during a backwash event. This is known as the chemically enhanced backwash (CEB). Yet this is generally insufficient to maintain full production rates and more intensive chemical routines are used to remove more strongly bound fouling compounds. This intensive clean is the Clean-in-Place (CIP) where the operating unit is removed from service and undergoes a process of filling and soaking with a moderately high concentration of hypochlorite, or other compounds. Heating of the cleaning solution can also be used to facilitate cleaning. The unit is then rinsed and returned to service. This process has costs associated with chemical materials and loss of plant availability, yet is applied frequently when more 'challenging' waters are being filtered. Furthermore, an additional cost to the operation is chemical damage to the membrane from these cleaning routines.

Degradation of polymeric membrane from chemical attack as a direct consequence of cleaning weakens their structure over time. Eventually membrane elements fail, compromising water quality and ultimately the complete replacement is needed after 5 to 10 years (Gijsbertsen-Abrahamse et al 2006). The cost associated with membrane degradation can be significant for secondary effluent filtration as cleaning and membrane replacement costs are responsible for about 60% of the total operating costs (Bartels et al. 2004). Therefore exploration of more robust membrane materials is gaining interest, especially in the areas of challenging waters. One group of materials that resists cleaning chemical attack is ceramics, which have recently emerged as a viable water treatment technology (Karnik et al. 2005, Lehman and Liu 2009). Ceramic membranes are largely chemically inert and according to ceramic membrane manufacturer Metawater, installations dating from 1998 are still in operation and have reported no membrane replacement, no membrane breakage, and no loss of flux. The application of ceramic membranes is attracting increased interest, with Japan installing many plants, mostly for decentralised treatment (Clement et al. 2009). Metawater have placed ceramic membranes in 117 plants operating in Japan. Metawater claims the current total operating capacity is 547 ML/day. In the Netherlands, PWN Technologies recently commissioned their 120 ML/day Andijk III plant containing the CeraMac system which reduces the capital cost of the ceramic membrane plant that contains Metawater's ceramic membranes. Based on these experiences from surface water treatment, ceramic membranes offer longer life, more robust operation and lower failure As there are very few plants operating on wastewater, rates than polymeric membranes. opportunities for water recycling are still emerging.

Ceramic membranes would also be better suited for pairing with ozone addition due to its greater stability. Ozonisation is one of the Advanced Oxidation Processes and is a chemical treatment

designed to attack organic matter for removal or deactivation by oxidation using highly reactive hydroxyl radicals. The combination of ozone and ceramic membranes is an innovative step for water recycling for a number of reasons. Ceramic membranes do not degrade in the presence of ozone, but instead assist in the oxidation reactions. Breakdown of ozone is rapid in the presence of aluminium oxide (Qi *et al.* 2010), the key material of the ceramic membrane. Dissolved ozone in contact with the ceramic surface accelerates the formation of these reactive free radicals that break down organic matter and disinfect the water (see Figure 1 for the principle involved). This can improve ozonisation efficiency over conventional ozone treatment and may potentially reduce ozone demand. This leads to the second innovation where the ozone works as a continuous membrane cleaner (Clement *et al.* 2009, Karnik *et al.* 2005, Lehman and Liu 2009, Zhu *et al.* 2011) and creates the opportunity of a potential "low cleaning chemical" operation.



Figure 1. Ozonisation / Ceramic membrane principle

Although ceramic membrane costs are higher per square metre of filtration area than conventional polymeric technology, ceramic prices are reducing as markets grow. Due to higher flux, chemical robustness, and longer life, there are many applications where the ceramic membrane material costs are offset and can therefore be economically justified. The use of ozone has been found to lead to major flux improvement of ceramic membranes which leads to cheaper operation on a cost per volume of water treated basis (Karnik *et al.* 2005, Lehman and Liu 2009). Further, in the context of recycling of wastewater where feedwater quality and membrane fouling present considerable challenges for economic treatment by polymeric processes, the ceramic membrane / ozonisation hybrid process may see an even greater benefit. This project seeks to explore these cleaning, performance and economic virtues of ceramic membrane combined with ozonisation in the context of recycling of secondary effluent.

#### Disinfection performance

All water treatment processes for potable and recycling applications require approval from health authorities before any plant is allowed to supply the community. The approval process involves demonstrating disinfection performance to a standard that protects the health of all water users. One such performance indicator is the microbiological removal efficiency, measured with respect to the main classes of water-borne pathogens; viruses, bacteria, protozoa and parasites. Removal efficiency is enumerated as the log reduction value (LRV) obtained from laboratory measurements of treated water samples compared to untreated water. This project seeks to investigate the disinfection performance of the ozone/membrane system, yet did not attempt to perform a full process validation trial (the scale of such an undertaking was outside the scope of this research project). Nevertheless, the disinfection testing followed the primary reference document for this work, the US-EPA Membrane

Filtration Guidance Manual (US-EPA 2005). The purpose of this was to estimate the pathogen rejection and inactivation capability of the new hybrid process.

One area of interest was the effect of the ozone/ceramic membrane process on the pathogen load in the reject water. All disinfection processes using a membrane barrier result in a reject stream with higher concentration of organisms than the feed water, by reason of simple mass balance. However, the fate of these potentially pathogenic organisms can pose a problem for treatment operators. The basin or estuarine system receiving reject streams need to be sufficiently large and / or carefully managed to avoid hazardous human exposure. Alternatively, treatment process reject streams are sent to the head of the plant to pass through the whole process again. Some organisms will be rendered inactive by multiple passes of conventional treatment, yet some organisms will remain active and pathogenic and over time a build-up of undesirable concentration of pathogens may develop.

Therefore, an additional benefit of the combined ozone / ceramic membrane process is the potential for enhanced deactivation of reject pathogen concentrations. This enhancement is hypothesized as follows: organisms in the feedwater undergo ozonisation with some fixed combination of time-weighted and dose-weighted average contact time resulting in a proportion of deactivation. The surviving active organisms then encounter the membrane and are held on the membrane surface or within the filter-cake for the entire duration of the filtration cycle. As the viable organisms are in a flowing stream of ozonised water, their individual contact times are substantially longer. This in theory would enhance disinfection of both permeate and reject streams, which potentially expands the options where reject water can be disposed. The potential for enhanced pathogen inactivation was explored as part of this study.

### 4 Plant Trial

The plant trial phase of the project involved installing a self-contained, demountable pilot plant at a wastewater treatment plant where treated effluent representative of a typical wastewater could be processed over a one year period. The plant was loaned to the project by the technology provider, PWN Technologies, the Netherlands, and needed to be configured for the specific task at the manufacturer before shipping to Melbourne. The pilot plant's treatment objectives were broadly defined and planned to be typical of those commonly faced by recycled water producers for reuse applications. The equipment under test was a microfiltration membrane incorporated into an automated plant and was to be experimented with two pre-treatment processes; that of ozonisation and in-line coagulation.

The pilot plant successfully cleared Customs and Quarantine services and was delivered to the trial site, Melbourne Water's Eastern Treatment Plant on 15th May 2012. The plant was first operated on 18th September and commissioning trials were completed on 25th September, 2012. The plant operated continuously performing a series experiments until July 2013 when the trial was concluded.

Of primary focus of the trial was the improvement in microfiltration performance when ozonisation was applied as a pre-treatment compared to no pre-treatment. However, application of inorganic coagulants are widely reported as a very cost effective method to improve filtration performance generally. Previous work in this area has shown high fluxes are possible with ceramic membranes where both ozonisation and coagulation are used as a pre-treatment (Lehman and Liu 2009). While this work was able to demonstrate a reduction in the required coagulant dose when ozone was applied, it did not determine the need for coagulant addition in membrane performance where ozone is also used. Therefore, to explore the full implications of ozonisation pre-treatment before a ceramic filter, the experimental matrix of membrane performance was expanded to include the benefit of inline coagulation pre-treatment with and without ozonisation. Figure 2 shows a schematic representation of the plant configuration along with key process control points and measurement samples points.



Figure 2. Schematic of process

Ozonisation of wastewater injects ozone gas that was produced "at-line" by an ozone generator from the reaction of oxygen within an electrical discharge. Ozone injection into the water stream uses a mixing device, such as side-stream, venturi or static mixer, to ensure rapid and efficient dissolution. Typically downstream of the mixer is an ozone contactor of carefully determined volume to detain the ozonised water for a fixed contact time. This is to ensure sufficient exposure of the reactants to free ozone, and to provide adequate time for the ozone to decay prior to downstream processes that cannot tolerate ozone. As polymer membranes and their seals/housings are irreparably damaged by ozone a large contactor is used to guarantee complete decomposition, via both mechanisms of reaction with organic material and self-decay. However, the present project calls for free dissolved ozone to be at the ceramic membrane surface, therefore the contact volume must be designed to ensure a residual amount of ozone is present on the membrane surface.

# 4.1 The Trial Site



Figure 3. Melbourne Water's Eastern Treatment Plant, Carrum, Victoria (Source: Melbourne Water web site)

Melbourne Water operates two sewage treatment plants in the greater Melbourne area. The Eastern Treatment Plant (ETP), 40 km south east of the city treats 330 ML/day of domestic and industrial effluent. The plant uses a conventional activated sludge treatment process before extensive tertiary treatment and disinfection to Class A recycled water standard for supply to customers for reuse or discharge to an ocean outfall. See Figure 4 for an overview of the ETP process. This work used as its feed the secondary effluent obtained from the holding lagoon prior to any tertiary treatment.

The water quality used to supply a membrane process has a significant influence on the plant's performance. Any water can be theoretically treated to meet some criteria of use by applying multiple treatment technologies. However, secondary treated effluent sourced from municipal sewage is generally regarded as a difficult water to further treat, particularly when the sewage catchment contains heavy and light industry. The combination of organic matter that is not easily biodegradable, along with extracellular polymeric substances from the activated sludge process, among others, represents significant fouling potential to membrane processes. Therefore, treatment of secondary effluent for reuse applications is never a trivial undertaking, and one that requires careful plant design and sound understanding of the cost implications. Yet it is precisely this type of challenging water that this project seeks to treat using the more robust ceramic membrane combined with ozonisation to investigate if cost effective exploitation of the low value resource is possible.



Figure 4. Overview of ETP's primary and secondary processes

Secondary treated effluent from the ETP is highly coloured containing high concentrations of non-biodegradable coloured compounds. Further, ultraviolet absorbance at 254 nm (UV<sub>254</sub>) is also relatively high, signifying high concentrations of olefins and aromatic species, and the dissolved organic carbon (DOC) content was typically between 10 - 16 mg/L during the course of the trial. The organic content of the effluent was considerably variable, principally due to a large number of industries following variable weekly discharge practices. The effluent for the project has passed through secondary clarifiers and the turbidity was commonly 3 - 5 NTU, but high turbidity events did occur during high rainfall periods or during agitation of the effluent holding basins from windy conditions.

| AWRCoE Project Final Report – Cerami | ic Membrane Demonstration |
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Table 1. Typical ETP secondary effluent water quality data collected prior to and during the earlystages of operation.

| Parameter  | Value  |
|--|--------|
| COD (mg/L) <sup>1</sup>                              | 58.7   |
| pH <sup>1</sup>                                      | 7      |
| Suspended Solids (mg/L) <sup>1</sup>                 | 16.1   |
| E Coli (counts/100mL) <sup>1</sup>                   | 50,500 |
| Alkalinity (mg/L as CaCO <sub>3</sub> ) <sup>1</sup> | 97.8   |
| TDS (mg/L) <sup>1</sup>                              | 486    |
| TDS – Inorganic (mg/L) <sup>1</sup>                  | 397    |
| TKN (mg/L) <sup>1</sup>                              | 5.5    |
| True Colour (PtCo) <sup>1</sup>                      | 80     |
| Turbidity (NTU) <sup>1</sup>                         | 7      |
| UV Transmittance 254nm <sup>1</sup>                  | 49     |
| Aluminium (mg/L) <sup>2</sup>                        | 0.72   |
| Ammonia (mg/L as N) <sup>1</sup>                     | 1.75   |
| Calcium (mg/L) <sup>2</sup>                          | 20.7   |
| Iron (mg/L) <sup>2</sup>                             | 0.26   |
| Magnesium (mg/L) <sup>2</sup>                        | 12.25  |
| Manganese (mg/L) <sup>2</sup>                        | 0.058  |
| Nitrate (mg/L as N) <sup>1</sup>                     | 7.9    |
| Nitrite (mg/L as N) <sup>1</sup>                     | 0.46   |
| Potassium (mg/L) <sup>2</sup>                        | 23     |

1. Collected over the period from January 2012 to March 2013 2. Collected over the period from May to August 2012.

### 4.2 Pilot Plant

The pilot plant was provided by PWN Technologies, constructed by RWB Water Services (the Netherlands) and was fully containerised, easily demountable and able to be installed at the test site rapidly. The plant was built into two shipping containers design to sit together with an interconnecting hatchway as a conduit for cables and hoses. The plant was equipped with a single ceramic Metawater element of 25 m<sup>2</sup> membrane area. At a nominal permeate flux of 100 L/m<sup>2</sup>.h the production capacity was 2.5 m<sup>3</sup>/h. The plant was configured in dead-end filtration mode and programmed for constant flux control. Clearing of the filtrated material was affected by programmed hydraulic backwash with permeate driven by compressed air, and facilities for chemical enhanced backwashing were provided. See section 4.2.2 for details of the plant operation.

The plant's process control computer enabled automatic, unattended operation with logging of all process sensors. The installed communications equipment allowed remote access for monitoring and data collection. Also technical support and programming modifications were provided over the remote link by the owners in The Netherlands.



Figure 5. Process flow diagram

### 4.2.1 Ceramic Membrane



Figure 6. The Metawater 25 m<sup>2</sup> ceramic element side view (left) and along the monolith (right)

The ceramic membrane incorporated into the pilot plant was a single Metawater, monolith element. This element consists of  $\alpha$ -alumina material formed into one piece 1.5 m long x 0.18 m in diameter. Through the element are 2,000 lumen channels of active layer with a pore size of 0.1  $\mu$ m. The total area of membrane is 25 m<sup>2</sup>.



Figure 7. Photographs of containers delivery (left) and placement (right)

#### 4.2.2 The Installation

Installation activities were carried out to connect all services necessary to operate the plant on Melbourne Water's facilities. Site health and safety requirements were met through the execution of a HAZOP process led by Black and Veatch, carried out on 25th July, 2012 and approved on 12th September, 2012. Following this process all requisite site operation documentation such as Standard Operating Procedures, Task Risk Assessments and Safe Work Method Statements were prepared and reviewed by Melbourne Water personnel. The final approval for operation was obtained after a HAZID hazard identification activity was performed and completed on 17th September, 2012.

The larger (20 foot container) held the main plant equipment of feedwater pump, membrane housing, pressurised backwash tank, product water tank and reject tank. The smaller (10 foot container) held the chemical dosing facilities and the air compressor. See Figure 8 for photographs of inside the two containers.

The plant was operated in constant flux mode under VFD feed pump control with transmembrane pressure (TMP) continuously monitored. Hydraulic backwashes (BW) were performed under PLC control according to a predetermine frequency (See section 4.3 for details of the experimental matrix). During a BW sequence the backwash tank is filled with 55 L of permeate using a separate pump, pressurised to 5 bar, then about  $2/3^{rd}$  of the volume rapidly passed through the membrane to a reject receiver in about 10 seconds. The remaining  $1/3^{rd}$  of the tank volume continued to backwash but is accompanied with an air scour across the feed side (or lumen) for 5 seconds, followed by compressed air only for a further 5 seconds. The last of these is used to drive water from the feed side, effectively leaving the membrane dry after backwash.

As hydraulic BWs do not completely clear the membrane of all filtered material, some foulant material remains on the membrane surface and is dealt with by chemical enhanced backwashing (CEB). These were programmed to occur after a fixed number of hydraulic BWs, and used either sodium hypochlorite (100 mg/L as free Cl<sub>2</sub>) or hydrochloric acid (at pH 2). The sequence of a CEB differed from hydraulic BW in a few aspects. While the BW tank is filling with permeate a dosing pump in the small container starts and doses the required amount of neat chemical to achieve the correct CEB concentration. The CEB solution is then driven through the membrane at a reduced pressure (2 bar). The program then pauses for fixed soak time (15 min for hypochlorite, 20 min for acid). A hydraulic BW is then carried out at the conclusion of the CEB soak time to remove all traces of chemical before the next filtration cycle starts.



Figure 8. Inside the containers – membrane filtration (left) and chemical dosing (right)

#### Process Monitoring

The pilot plant was equipped with the following sensors that were logged every 30 seconds.

- Membrane inlet pressure, bar
  - Membrane outlet pressure, bar
- Calculated transmembrane pressure (TMP), bar
- Feed flow, m<sup>3</sup>/h
- Turbidity of feed, NTU (was found to be unreliable)
- Turbidity of permeate, NTU
- Temperature of feed, °C
- pH of feed
- pH of permeate

#### Ozonisation

The ozonisation facilities were provided by Melbourne Water as part of their Tertiary Treatment Trials Plant and consisted of a Wedeco ozone generator with integrated ozone concentration measurement and dosing flow control. An air separation unit was also available to provide purified oxygen to the ozone generator sourced from the air. Ozone gas was injected into a stream of feed water at a Statiflo static mixer of 25mm diameter to aid dissolution. One of Trial Plant's supply pumps provided constant supply of secondary effluent to the ozone injector and pilot plant. The supply pump, ozone generator and ozone injector were monitored by the Trials Plant's process control system that included various safety interlocks to provide warning alarms and/or shutdown the ozonisation in the event of an unsafe condition.

The ozonised water flowed to a height of 3.5m before falling to a 500 L detention tank before overflowing to the sewer. The detention tank ensured greater than 5 min of detention to allow free ozone to decay before entering the sewer. A tee in the ozonised stream pipe was connected to the pilot plant's feed pump and the 3m of height provided sufficient head to ensure flooded suction. After modifications in The Netherlands, the pilot plant was able to accept ozonised feedwater with minimal hold-up time between this tee and the ceramic membrane. This was important due to the fast decay rate of ozone and the requirement to achieve a target concentration of residual ozone on the membrane surface of approximately 0.5 to 1.0 mg/L. The total residence volume between ozone injection point and membrane was 34.63 L. This was the sum of two sections operating at slightly different flow rates that must be taken into account when determining residence time. The residence time determined by the flow rate of the supply pump. The residence volume of the second section between the supply pump. The residence time determined by the pilot

plant's feed pump. Under the different production rates used in the project the residence time varied from a minimum of 25 s to a maximum of 84.7 s.

Ozone was injected into the feed water at a rate of approximately 22 - 25 mg  $O_3/L$  (from the metered gas flow rate and  $O_3$  concentration obtained from the generator's internal meter). However, the static mixer efficiency proved to be sub-optimal and significant loss of ozone through undissolved gas resulted. Therefore, the ozone injection rate was controlled based on residual ozone measurements made directly at the sample point before the membrane (sample point 3) using the indigo wet chemical method (see section 4.4.2 for details of the test method).

Ozone is a very hazardous substance. Human 8 hour exposure limits are mandated at 0.1 ppmv in a working environment and the gas can be fatal above 5 ppmv. Therefore, much of the installation effort was focused on safe operation of the ozonisation facilities. All closed vessels that could contain ozone were required to have vapour vent lines attached and directed to an ozone destruction system provided by Melbourne Water at the trials facility. These vessels were the effluent detention tank and the product water tank, and were maintained under a slight negative pressure. In addition to an active ozone destruction system, ozone monitors were used in the pilot plant containers. In the main plant an integrated ozone sensor incorporated into the plant's control system to alarm if ozone concentrations inside the container reached 0.3 ppmv and interlocked to shutdown the ozone generator if 0.5 ppmv was reached.

#### Coagulation

The pilot plant was equipped with in-line coagulant dosing facilities. Coagulant could be applied at various doses at a point after the feed pump inside the container: just before a static mixer and before the membrane. The coagulant used in this trial was poly aluminium chloride, 23% (as  $AI_2O_3$ ) obtained from Orica Chemicals.

A jar test was performed to determine the minimal coagulant dose for the effluent. Results indicated a 3 mg (as  $AI^{3+})/L$  produced adequate floc formation. To confirm validity of this dose when translated to the fast dynamics for the in-line dosing, an operational test was performed at the plant. Samples were collected just before the membrane while a 3 mg/L addition rate was applied. Observation of the samples indicated equivalent presence of pin floc as the jar tests. The dose of 3 mg (as  $AI^{3+})/L$  was therefore applied throughout the trial unless otherwise stated.



Figure 9. Photographs of ozone generator (left) and injector (right)

### 4.3 Experimental Matrix Development

For most membrane systems, for a given water quality, permeate flux is largely influenced by fouling and the frequency of CEBs employed to control it. Thus plant capacity must account for expected, or anticipated fouling. For ceramic membrane systems, due to the wide latitude of tolerated CEBs, significant membrane fouling can be effectively managed with frequent CEBs, provided the cost of chemical consumption can be justified. Therefore, for an objective assessment of the operational capacity of these systems, measurements need to be made at a realistic CEB frequency. Further, backwash frequency, design flux and the duration until clean-in-place routines, are examples of parameters that need to be declared in the context of a given CEB regime. One measurement that is particularly informative for membrane processes is critical flux, described when operating in constant flux mode as the flux above which irreversible fouling appears. But as the use of chemical backwashing can control a degree of fouling, then the determination of maximum sustainable flux over the longer term at a fixed and stated CEB frequency is an operationally more useful measure than critical flux. Maximum sustainable flux was determined by carrying out a series of filtration runs with an increasing increment of flux and the degree of fouling was measured for each flux step. The maximum flux was said to be the flux step that resulted in acceptable fouling determined by criteria outline below.

Therefore, the primary measurement of filtration performance for this trial was centred on the determination of maximum sustainable flux under a set of pre-treatment conditions. To carry out these tests, the controlled conditions were first established from information obtained from the technology supplier and from initial experimental testing.

The establishment of the controlled conditions were:

- **Backwash Frequency**: due to the effluent's suspended solid content, the membrane required a backwash frequency of 50 L/m<sup>2</sup> of membrane area. Thus the 25 m<sup>2</sup> membrane processed 1250 L of water between backwashes. At a nominal flux of 100 L/m<sup>2</sup>.h this was a BW frequency of 30 min. However this frequency will vary for each flux to maintain the 50 L/m<sup>2</sup> condition.
- **CEB Frequency**: Preliminary tests of the plant's operation under the environment of secondary effluent with no pre-treatment were performed to establish initial CEB conditions. Upon the first introduction of effluent rapid fouling occurred indicated by a rise in TMP to 3 bar within four days at the lowest useful flux (50 L/m<sup>2</sup>.h). Acid CEBs did not improve TMP suggesting minor inorganic fouling. This testing led to the initial operating parameters of a hypochlorite backwash event occurring after every five backwash events and an acid CEB occurring after every seven hypochlorite CEB cycles. However, when an acid CEB was programmed the organic fouling was able to accumulate during that period, making the subsequent hypochlorite CEB less effective. Consequently, acid CEB were modified to include a mixed hydrochloric acid and hydrogen peroxide solution, 100 mg/L as H<sub>2</sub>O<sub>2</sub> at pH 2.
- **Run Length**: The run length for each flux step required to treat the same volume of water to expose the membrane to identical fouling loads. At the lowest flux of 50 L/m<sup>2</sup>.h over 12 days filtered 360 m<sup>3</sup>, which was considered a reasonable duration for the number of tests needed to be performed. At higher flux steps, the run length reduces.

Then the criterion for determining the highest flux that could be operated over the long term was established. Membrane fouling is ultimately controlled by performing a Clean-in-Place (CIP) routine. However, the frequency of CIP routines has a large influence of plant profitability and operability. Too frequent CIPs results in unacceptably high chemical costs and excessive plant downtime. An acceptable frequency for CIPs was assumed to be every 90 days. Therefore with all other plant conditions controlled, a flux that results in CIPs being required at greater than 90 day was considered to be sustainable in the long term.

The final aspect of the maximum sustainable flux test structure was the trigger point to initiate a CIP routine. The technology providers suggest economical operation when feed pumps are sized for efficient supply pressure at a maximum of 1.5 bar above initial, clean membrane TMP. Pumps can commonly operate above this point, and the membrane can withstand much higher pressures, but 1.5 bar above initial serves as a good trigger point for a CIP.

A summary of controlled parameters and the determining factor were:

| Variable Parameter    | Increasing flux in steps  |
|-----------------------|---|
| Controlled Parameters | Hypochlorite CEB frequency = after 5 filtration cycles.<br>Acid CEB frequency = after 7 Hypochlorite CEB events.<br>Backwash frequency = every 1.25 m <sup>3</sup> .<br>Total length of each step = after filtered 360 m <sup>3</sup> or 7 days<br>(whichever is the longer time)<br>Ozone dose (if used) = $0.5 - 1.0$ mg/L at membrane<br>Coagulant dose (if used) = $3$ mg/L as Al <sup>3+</sup> |
| Determination         | Maximum flux step when CIP frequency is > 90 days   |

#### Experimental Matrix

With plant operating conditions ascertained and a measure of plant performance established the experimental framework was designed to explore various pre-treatment options. Of primary concern was the degree of performance gain, or loss when ozonisation was used as pre-treatment, with and without in-line coagulation. Additional to these experiments were tests to further optimise the conditions and to explore the mechanism of this process. Therefore, the experimental matrix included the following tests as summarised in Table 2 and detailed in Table 3.

| Test<br>Number | Condition                       | Details  |
|----------------|---------------------------------|--|
| 1              | Baseline conditions             | Secondary effluent fed directly to the ceramic membrane  |
| 2              | Coagulant pre-treatment only    | Addition of in-line poly aluminium chloride<br>coagulant at 3 mg/L. Dosing point: between feed<br>pump and membrane.   |
| 4              | Ozonisation pre-treatment only  | Injection of ozone gas to deliver an excess<br>dissolved ozone conc. 0.5-1.0 mg/L on the<br>membrane measured at sample point 3. Injection<br>point was 34.6 L of residence volume before<br>membrane. |
| 5              | Combined ozone and<br>coagulant | Application of both pre-treatments together  |
| 7              | Low CEB operation               | Operating with ozone and coag. at high flux with reduced CEB frequency.  |
| 8              | Effect of ozone quenching       | Comparison of performance when excess ozone is quenched with a reducing agent.   |
| 9              | Pathogen removal efficiency     | Challenge test for:<br>Virus with MS2 coliphage<br>Bacteria with naturally occurring <i>E.coli</i> .   |

Table 2. Summary of main experiments carried out

### Table 3. Experimental Matrix

| Mode<br>(aim of test)                              | Baseline                | Improvement<br>over baseline<br>with<br>coagulant | Improvement<br>over baseline<br>with ozone | Improvement<br>over baseline<br>with ozone &<br>coagulant | Low CEB<br>frequency   | Quenched<br>ozone                 | Pathoger                       | n testing                      |
|--|-------------------------|---|--|---|------------------------|-----------------------------------|--------------------------------|--------------------------------|
| Test number  | Test 1                  | Test 2  | Test 4                                     | Test 5  | Test 7                 | Test 8                            | Tes                            | it 9                           |
| Description  | no $O_3$ , no coagulant | no O₃<br>coagulant                                | O <sub>3</sub><br>no coagulant             | O₃<br>coagulant   | O₃<br>coagulant        | O₃<br>no coagulant<br>SMBS dosing | Virus<br>No O₃<br>no coagulant | Bacteria<br>O₃<br>no coagulant |
| Ozone concentration<br>on membrane<br>(mg/L)       | none                    | none  | 0.5  | 0.5   | 0.5                    | 0.5                               | None                           | 0.5                            |
| Coagulant<br>(mg/L as Al)                          | none                    | 3   | none                                       | 3   | minimum                | none                              | None                           | None                           |
| BW frequency (L/m <sup>2</sup> )                   | 50                      | 50  | 50   | 50  | 50                     | 50                                | 1 cycle                        | 1 cycle                        |
| CIP frequency/end<br>point                         | measure kPa<br>rise/hr  | measure kPa<br>rise/hr                            | measure kPa<br>rise/hr                     | measure kPa<br>rise/hr                                    | measure kPa<br>rise/hr | measure kPa<br>rise/hr            | None                           | None                           |
| Number of regular<br>BW before<br>hypochlorite CEB | 5                       | 5   | 5  | 5   | Variable               | 5                                 | None                           | None                           |
| Number of<br>hypochlorite CEB<br>before acid CEB   | 3                       | 7   | 7  | 7   | None                   | 7                                 | None                           | None                           |
| Flux steps   | 50, 75, 100             | 50, 100, 150,<br>200                              | 50, 100                                    | 147, 169, 182   | 182                    | 100                               | 200                            | 150                            |
| Volume of step (m <sup>3</sup> )                   | 360                     | 360   | 360  | 360   | 360                    | 360                               | 1                              | 1                              |

#### CIP Routine

The structure of this trial involved a series of distinct experiments to measure membrane fouling. At the conclusion of each experiment the membrane would be left with some degree of fouling. This needed to be completely removed for subsequent experiments to be valid. The procedure followed to "reset" the membrane was a conventional CIP process.

The procedure was as follows:

- Step 1 Filtration was started with tap water feed with 30 minute cycle. 2 successive Hypochlorite CEBs were performed to remove the relatively easily dislodged material.
- Step 2 Backwashed the membrane with 1000 mg/L hypochlorite and soaked for 1 hour to soften the remaining foulant material.
- Step 3 Circulated 1000 mg/L hypochlorite solution across the membrane surface (the lumen) at high velocity (approx. 2.5 m<sup>3</sup>/h) for 30 min to dissolve and scour the membrane surface.
- Step 4 Rinsed the membrane with tap water.
- Step 5 Performed one acid CEB event (hydrochloric acid at pH between 1.9 2.0) to dissolve inorganic material.

#### Clean Water Flux Test

Before each run the membrane submitted to a baseline test to create a reference point for evaluation. The baseline test consisted of running the plant on tap water feed for a minimum of 30 min, usually 1 hour, allowing at least one BW event to occur. Flux was set at 100  $L/m^2$ .h (equalling 2.5 m<sup>3</sup>/h) and BW frequency was set to 30 min. Typically the TMP for this flux was between 0.2 - 0.3 bar

Before each run the membrane submitted to a baseline test to create a reference point for evaluation. The baseline test consisted of running the plant on tap water feed for a minimum of 30 min, usually 1 hour, allowing at least one BW event to occur. Flux was set at 100  $L/m^2$ .h (equalling 2.5 m<sup>3</sup>/h) and BW frequency was set to 30 min. Typically the TMP for this flux was between 0.2 - 0.6 bar

| Date       | TMP (bar) | Date       | TMP (bar) |
|------------|-----------|------------|-----------|
| 26/09/2013 | 0.60      | 16/02/2014 | 0.20      |
| 12/10/2013 | 0.60      | 22/02/2014 | 0.21      |
| 17/10/2013 | 0.50      | 27/02/2014 | 0.24      |
| 19/10/2013 | 0.39      | 1/03/2014  | 0.30      |
| 2/11/2013  | 0.34      | 8/03/2014  | 0.20      |
| 13/11/2013 | 0.25      | 18/03/2014 | 0.21      |
| 23/11/2013 | 0.24      | 22/03/2014 | 0.27      |
| 26/11/2013 | 0.32      | 3/04/2014  | 0.32      |
| 7/12/2013  | 0.34      | 10/04/2014 | 0.53      |
| 11/12/2013 | 0.43      | 18/04/2014 | 0.30      |
| 14/01/2014 | 0.25      | 29/04/2014 | 0.28      |
| 5/02/2014  | 0.16      | 24/06/2014 | 0.62      |
| 15/02/2014 | 0.22      | 28/06/2014 | 0.50      |

Table 4. TMP of clean water test at flux 100 L/m<sup>2</sup>.h performed before each run.

### 4.4 Analysis Methods

#### 4.4.1 Plant Performance Testing

The process performance was assessed by monitoring TMP over time for the entire run length until either the target was reached or gross fouling terminated the run. Gross fouling was said to have occurred when the feed pump at maximum output could no longer maintain the flux setpoint due to high membrane resistance. The TMP at which this occurred depended on the flux; approximately 2.8 bar was found to be the maximum at a flux of 50 L/m<sup>3</sup>.h, but 2.5 bar would be the maximum at 150 L/m<sup>3</sup>.h

Plotting a TMP profile on a time series gave an indication of the filtration performance, as showed in blue on Figure 10. However, the important data for each run was the estimate of the frequency of CIP obtained from a prediction of fouling. This was calculated be plotting the TMP at the start of each filtration cycle (red points of Figure 10) and taking the linear regression of those points (black line). The slope of this relationship reveals the degree of fouling expressed in TMP rise per day which was used to determine 'days to CIP' as described in Section 4.3.



Figure 10. Example of TMP profile and fouling trend

In the structure of these tests, the maximum sustainable flux for a given experiment was the flux step that displayed an estimated CIP frequency of > 90 days. A flux that results in a CIP frequency less than 90 days would be deemed to be too frequent for economical operation. A flux choice that results in minimal fouling as to require infrequent CIPs would be considered over-sized, so operating as close to the minimum CIP frequency gives the most practical performance.

The plant performance results in Section 4.5 were obtained as described in the paragraph above. However, to display all the flux steps for each experiment on a single graph, TMP as a function of volume filtered was used.

#### 4.4.2 Chemical Testing

Water samples collected from; feed water (sample point 1), post ozone injection (sample point 2), and just before the membrane (sample point 3) were vacuum filtered through a 0.45  $\mu$ m cellulose acetate membrane before analysis. Post membrane samples (sample point 4) were analysed as collected. The test frequency program is listed in Table 5.

| Purpose   | Purpose Parameter  |  | Frequency           |
|---|--|--|---------------------|
| Determine O₃ dose for<br>process control                  | Residual O <sub>3</sub>  | O <sub>3</sub> injector - SP2<br>membrane inlet - SP3<br>membrane outlet SP4                 | 3 per week          |
| Determine colour reduction                                | Colour (PtCo)  | Feed - SP1<br>O <sub>3</sub> injector - SP2<br>membrane inlet - SP3<br>membrane outlet - SP4 | 3 per week          |
| Determine organics<br>reduction                           | UV abs at 254 nm (UV $_{\rm 254})$   | Feed - SP1<br>O₃ injector - SP2<br>membrane inlet - SP3<br>membrane outlet - SP4             | 3 per week          |
| Determine DOC<br>reduction/change                         | DOC  | Feed - SP1<br>O₃ injector - SP2<br>membrane inlet - SP3<br>membrane outlet - SP4             | 3 per week          |
| Determine metal ion for<br>changed across the<br>membrane | Metals: Aluminium, Calcium,<br>Copper, Iron, Potassium,<br>Magnesium, Manganese<br>Nickel, Silica and Zinc | Feed - SP1<br>O₃ injector - SP2<br>membrane inlet - SP3<br>membrane outlet - SP4             | 3 per week          |
| Organic characterisation                                  |  | Feed - SP1<br>O₃ injector - SP2<br>membrane inlet - SP3<br>membrane outlet - SP4             | One set of analyses |

#### Table 5. Chemical testing routine

#### Residual Ozone in Water

The ozone concentration in effluent sample needed to be measured accurately and reliably to form robust conclusions from plant data, but was also used to control ozone addition rate. The method used was the indigo method (Standard Methods, 4500-B, APHA, 2005). This test is the method of choice for low level ozone concentration in water due to its accuracy, precision and ability to produce the analytical colour used for measurement at sample collection. This eliminates the rapid decay rate of ozone as a source of error in the test.

#### Dissolved Organic Carbon

Dissolved Organic Carbon (DOC) was measured on all samples using a Shimadzu TOC-V thermal decomposition analyser, calibrated against Potassium Hydrogen Phthalate standards.

#### UV Absorbance and Colour

Organic content in the effluent was also measured by UV absorbance. Filtered samples were tested according to method 5910-B (Standard Methods, APHA, 2005) on a HACH DR5000 spectrophotometer. True colour was measured using method HACH Method 8025 (Platinum-Cobalt technique based on Standard Method 2120-B (APHA 2005)) on the same instrument.

#### Organic Characterisation

A series of water samples were collected and analysed by Size Exclusion Liquid Chromatography with Organic Carbon Detection (LC-OCD) at the Water Supply Engineering group at Technical University of Dresden (Germany). This instrument is able to separate organic compounds by molecular size and broad chemical class, and then determine the relative amounts of each class. This was used to deduce the type of organic matter that:

- Was reduced by pre-treatment processes
- Was rejected by the membrane
- Passed unaffected through the treatment process.

#### Trace Metal Analysis

Trace metal analysis was performed by acidifying all samples with 1% nitric acid and analysing with a Shimadzu Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) measured against standards prepared from purchased, certified metal standard solutions. Initial method development found that digesting the filtered water samples before analysis did not recover measureable quantities

of analyte. The trace metals analysed for were: Aluminium, Calcium, Copper, Iron, Potassium, Magnesium, Manganese Nickel, Silica and Zinc.

#### 4.4.3 Microbiological Testing

On 27th September 2012 a special meeting of the project team was held to explore the value and challenges faced by incorporating a pathogen validation test into the project. The meeting was attended by project partners from Victoria University, South East Water, Water Research Australia and Black and Veatch. Additional advisors, Australian Water Quality Centre (Paul Monis) and Victorian Department of Health (Luc Richard and Renwick Chan) also attended. Discussions focused on the cost of any validation study compared to the value to the project. It was identified the limited resources precluded a full membrane validation study, yet a carefully targeted experimental plan could lead to interesting and valuable outcomes for:

- Academic and research communities (outputs measured by publications in scientific journals and science conferences);
- Industry (outputs measured by industry conference presentations and future uptake of the technology);
- Regulators (outputs measured by industry conference presentations and acceptance of the technology as a pathogen reducing/deactivating process);

Therefore, the experimental plan to determine disinfection performance of the trial treatment process was developed around a series of challenge tests. Of the three pathogen classes only viruses and bacteria were investigated in this trial and an abridged challenge testing regime was adopted, although the work still followed the US EPA Membrane Filtration Guidance Manual for experimental design as mentioned in Section 3.

#### Viruses

Ozone processes are generally considered to be inappropriate for virus removal due to their poor inactivation of these organisms. Therefore, the virus LRV was determined for the ceramic microfiltration membrane alone using the virus surrogate, MS2 coliphage as the challenge particle. As these challenge tests must be performed conservatively, the worst case conditions were chosen. The highest practical flux for this plant was 200 L/m<sup>2</sup>.h and clean water was use to minimise cake layer effects.

A concentrate of MS2 coliphage was purchased form a commercial laboratory at  $10^{12}$  pfu/100ml (plaque-forming units). This was used to prepare the feed challenge solution in a 1350 L stirred feed tank with tap water at a target concentration of  $10^7$  pfu/100ml. MS2 coliphage is very susceptible to inactivation with chlorination, so prior to preparing the feed solution the tap water was dechlorinated with sodium thiosulphate. Testing for free chlorine after dechlorination found < 0.02 mg/L. After addition of MS2 concentrate the tank was stirred for 15 min.

Water samples were collected aseptically by flame sterilising the sample points before collection. Samples were collected in microbiological bottles provided by the commercial laboratory, and contained thiosulphate preservative. Field blanks were collected at the same time as samples and were stored, transported and delivered to the laboratory together with challenge test samples to ensure the risk of cross contamination occurring during sampling or transport was detected.

The water samples were delivered to the commercial laboratory (ALS Environmental - 22 Dalmore Drive, Scoresby Victoria) in cooled boxes on the same day as collection and analysis commenced immediately. Coliphage were enumerated using the fRNA double agar layer method for premembrane samples and the single agar layer method for post membrane samples and blanks.

#### Bacteria

For the bacterial test particle, naturally occurring *E.coli* was chosen to assess the bacteria LRV over all process operation modes (including coagulation and ozone). *E.coli* in the secondary effluent was found to be  $10^{3.3} - 10^4$  cfu/100mL

Sampling was performed aseptically as outline in the virus section above, and collected into similar bottles with field blanks also collected. The *E.coli* was measured by the Collilert method by the same commercial laboratory.

In addition to the specific sample points, samples were also collected from the backwash water. This was accomplished by placing a sterilised plastic container inside the reject tank oriented directly under the membrane backwash outlet. When the BW event occurred the plastic container would fill and overflow with backwash water. The water was scooped out with a sterilised glass beaker, transferred immediately to a microbiological bottle and stored in cooled boxes. Due to the likely non-homogeneous nature of the backwash water, duplicate samples were collected and analysed.

#### 4.4.4 Direct Integrity Testing

The purpose of direct integrity testing was to provide an easily performed quality check of the membrane which can be related to the primary measure of membrane disinfection performance, which is the LRV determined during a full challenge test. Health authorities will only give a disinfection credit to a membrane process determined by the challenge test, the  $LRV_{C-test}$ , if it is supported by a direct integrity test,  $LRV_{DIT}$ . This is because direct integrity tests are performed frequently and therefore are the first signs of a breach in the membrane. For example, drinking water treatment operations must perform a direct integrity test at least once per day and the sensitivity of the test dictates the credit awarded to the process.

This project's plant trial did not produce classified water nor did it supply any customers so the requirement for direct integrity testing did not exist. However, the pilot plant had the capability of performing a direct integrity test in the form of the pressure decay test (PDT). Normally PDTs are automated and programmed to be carried out daily unattended, but the present pilot plant did not have that level of automation. Thus, PDTs were performed irregularly over the trial period.

Over the course of the trial, PDTs were carried out as outlined in the guidance manual (US-EPA 2005). The PDT is the most common direct integrity test currently in use and is generally associated with low pressure membrane process such as MF and UF. In a pressure decay test, compressed air is applied to one side of a fully wet membrane until a fixed set pressure (the test pressure) is reached. If all pores and defects in the membrane are below a known diameter (the test resolution, calculated in  $\mu$ m), surface tension will hold water in the pores and not allow the air to escape. The compressed air is then isolated and monitored for 10 min. An integral membrane unit will maintain the initial test pressure or exhibit a very slow rate of decay. However, if the membrane has developed a breach of size greater than the resolution diameter, then the defect will leak air and the pressure will decay at a faster rate than prescribed.

#### The Test Pressure

The US-EPA guidelines considers a membrane is integral if it is capable of safely removing the protozoa *Cryptosporidium*. The size of this organism is greater than 3  $\mu$ m, therefore a PDT that can detect a defect at the resolution of 3  $\mu$ m is required. The calculation of the minimum PDT pressure needed for a resolution diameter of 3  $\mu$ m is:

$$P_{min} = \frac{0.58 \text{ x } k \text{ x } \sigma \text{ x } \cos \theta}{d_{res}} + BP_{max} \quad \text{Equation B.4/B.6 (EPA Guideline)}$$

Where:

 $P_{min}$  = minimum test pressure (psi) k = pore shape correction factor  $\sigma$  = surface tension at the air-liquid interface (dynes/cm)  $\theta$  = liquid-membrane contact angle (degrees)  $d_{res}$  =direct integrity test resolution requirement (µm)  $BP_{max}$  = maximum backpressure on the system during the test (psi)

• The parameters k and  $\sigma$  are intrinsic properties of the membrane. Generally conservative values of k = 1 and  $\theta = 0$  are used.

- The surface tension ( $\sigma$ ) at the coldest anticipated water temperature is suggested to be used; the surface tension of water at 5 °C is 74.9 dynes/cm.
- *BP<sub>max</sub>* is the backpressure on the vented side of the membrane. In this configuration the membrane element is oriented vertically and the lumen in drained therefore a BP of zero is assumed.

Solving the equation for a defect resolution of 3  $\mu$ m (i.e. removal of protozoa) sees a minimum test pressure of:

$$P_{min} = 14.5 \text{ psi} = 1.0 \text{ bar}$$

To ensure an adequate margin to exceed the critical resolution of 3  $\mu$ m, a test pressure between 1.2 and 1.3 bar was used for regular testing.

#### PDT Method

A PDT must be performed on clean membrane as filter cake on the membrane surface will be a resistance to the applied air pressure and potentially mask a defect. Therefore, PDTs were performed after a CIP. The PDT method was as follows:

- 1. At the conclusion of the CIP, the plant was configured to run with tap water feed for 1 hour with no backwashing. This tested the effectiveness of the CIP and ensured the membrane was fully wet. A fully wet membrane is a critical requirement for a precise PDT.
- 2. Filtration was stopped and the membrane housing's feed side top and bottom valves were opened. The lumen allowed to drain.
- 3. Compressed air at ~ 1.2 bar was applied to the permeate side of the housing. This displaced all the water in the permeate side through the membrane out to drain. This was allowed to equilibrate until no more water was observed to be draining from the membrane and no flow of air was observed through the compressed air line.
- 4. The compressed air supply was isolated and the test started. The plant's process control system recorded the pressure of the permeate side of the membrane every 30 seconds.
- 5. After 10 min the membrane was depressurised and the plant returned to service.
- 6. The recorded data was processed to obtain pressure decay over 10 min and expressed at kPa/min.

#### Test Sensitivity

Sensitivity is defined as the maximum log removal value that can be reliably verified by the direct integrity test  $(LRV_{DIT})$ . The sensitivity of the direct integrity test establishes the maximum log removal credit that a membrane process is eligible to receive if it is less than or equal to that demonstrated during challenge testing  $(LRV_{C-Test})$ .

The sensitivity of a PDT can be calculated by converting the response from the test that measures the flow of air to an equivalent flow of water through an integrity breach during normal operation from:

$$RV_{DIT} = \log\left(\frac{Q_p \times ALRC \times P_{atm}}{\Delta P_{test} \times V_{sys} \times VCF}\right)$$

Equation 4.9 (EPA Guideline)

Where:

 $Q_p$ 

= maximum design flux, 42 L/min (from 100 L/ $m^2$ .h).

*ALRC* = air-liquid conversion ratio, assumed to be 490 for a test pressure of 1.2 bar.

$$P_{atm}$$
 = atmospheric pressure (psia).

- $\Delta P_{test}$  = smallest rate of pressure decay that can be reliably measured of known integrity breach (converted to psi/min). This is unknown for this installation. However, for this exercise was assumed to be 0.1 kPa/min
- $V_{\text{sys}}$  = volume of pressurized air in the system, 200 L in this setup.

Solving the equation, the  $LRV_{DIT}$  that can be credited to this PDT, assuming a detectable decay rate of 0.1 kPa/min is:

$$LRV_{DIT} = 5.0 \log$$

Or, by rearrangement of equation 4.9, a PDT upper control limit can be calculated to act as an alarm point when the credited LRV is not being met.

$$P_{(UCL)} = \left(\frac{Q_p \times ALRC \times P_{atm}}{10^{LRV} \times V_{sys} \times VCF}\right)$$
 Equation 4.17 (EPA Guideline)

For example, if the process is awarded a 4 log credit then the upper control limit for the PDT could conceivably be:

$$\Delta P_{(UCL)} = 1.0 \text{ kPa/min}$$

But a more conservative upper control limit would be more practical.

### 4.5 Plant Performance Results

As outlined in the experimental matrix (Table 3 in Section 4.3), the plant was operated under differing pre-treatment conditions; effluent without pre-treatment, coagulant only pre-treatment, ozone only, and finally ozone with coagulant pre-treatments. The following sections display the plant performance data under each of these pre-treatment environments in the form of initial TMP (i.e. the TMP after backwash, at the start of each filtration cycle) vs. volume filtered, along with tabulated inference of the fouling.

An example of the full TMP profile over the entire test period for the nominal flux of 100  $L/m^2$ .h under 3 conditions; no pre-treatment, coagulation only, and ozone plus coagulant runs at 162  $L/m^2$ .h are supplied in Appendix A.

#### 4.5.1 Membrane Performance with No Pre-treatment (Test Number 1, Table 3)

Figure 11 shows TMP rise per m<sup>3</sup> volume filtered using the controlled parameters outlined in section 4.4.1 with baseline conditions of no pre-treatment. The maximum sustainable flux was determined over three flux steps; 50, 75 and 100 L/m<sup>2</sup>.h. At a flux of 50 L/m<sup>2</sup>.h for over 300 m<sup>3</sup> of water was filtered with negligible rise in initial TMP. However, when flux was increased to 75 L/m<sup>2</sup>.h, TMP rise was rapid, and the run terminated when the TMP maximum was reached. Therefore, the maximum sustainable flux of none pre-treated effluent was between 50 and 75 L/m<sup>2</sup>.h



*Figure 11. Fouling rate as a function of volume filtered, non pre-treated effluent. The slopes were determined in units of bar/m<sup>3</sup>.* 

| Flux step<br>(L/m <sup>2</sup> .h)               | Calculated fouling<br>rate (TMP/day) | Correlation<br>coefficient | Estimated CIP<br>frequency (days) |  |  |  |
|--|--------------------------------------|----------------------------|-----------------------------------|--|--|--|
| 50   | negative                             | -                          | no fouling observed               |  |  |  |
| 75   | insufficient data                    | insufficient data          | CIP needed every day              |  |  |  |
| 100  | 1.37                                 | 0.67                       | CIP needed every day              |  |  |  |
| Maximum sustainable flux = $50 \text{ L/m}^2$ .h |                                      |                            |                                   |  |  |  |

Victoria University

#### 4.5.2 Membrane Performance with Coagulant (Test Number 2, Table 3)

Polyaluminium chloride coagulant dosing was initiated with otherwise identical conditions as baseline operation. It was observed that filtration characteristics were altered when in-line coagulation was employed. Figure 12 reveals a gradual TMP rise at 100  $L/m^2$ .h over entire test period when coagulant was used compared to gross fouling with no coagulant. At 150  $L/m^2$ .h, after an initial rapid rise, the TMP rise stabilised. However at 200  $L/m^2$ .h, the TMP rise resulted in reaching the upper TMP limit and the run was terminated.



Figure 12. Fouling rate as a function of volume filtered, coagulant pre-treated effluent. The slopes were determined with units of bar/m<sup>3</sup>.

| Flux step<br>(L/m <sup>2</sup> .h) | Calculated fouling<br>rate (TMP/day) | Correlation<br>coefficient | Estimated CIP<br>frequency (days) |
|------------------------------------|--------------------------------------|----------------------------|-----------------------------------|
| 50                                 | negative                             | -                          | no fouling observed               |
| 100                                | 0.01                                 | 0.64                       | 172                               |
| 150                                | 0.13                                 | 0.55                       | 10                                |
| 200                                | 2.79                                 | 0.95                       | CIP needed every day              |

Maximum sustainable flux = between  $100 - 150 \text{ L/m}^2$ .h

#### 4.5.3 Membrane Performance with Ozone (Test Number 4, Table 3)

Similar to previous sections, the fouling rate with ozonised pre-treatment at stepped fluxes or 50, 75 and 100 L/m<sup>2</sup>.h are show in Figure 13. The effect of ozone provided more stability to the TMP as compared to no pre-treatment at 50 L/m<sup>2</sup>.h. At the increased flux of 75 L/m<sup>2</sup>.h, stable TMP was also observed, which provides evidence that ozone on its own can assist with fouling control. However, this improvement was not achieved at 100 L/m<sup>2</sup>.h.



Figure 13. Fouling rate as a function of volume filtered, ozonised pre-treated effluent. Slopes were determined with units of bar/m<sup>3</sup>.

| Flux step<br>(L/m².h) | Calculated fouling<br>rate (TMP/day) | Correlation coefficient | Estimated CIP<br>frequency (days) |
|-----------------------|--------------------------------------|-------------------------|-----------------------------------|
| 50                    | 0.004                                | 0.21                    | 360                               |
| 75                    | negative                             | -                       | Not estimated                     |
| 100                   | 5.3                                  | 0.88                    | CIP needed every day              |

Maximum sustainable flux = between 75 - 100 L/m<sup>2</sup>.h

#### 4.5.4 Membrane Performance with Coagulation and Ozonisation (Test Number 5, Table 3)

Figure 14 shows the results of the maximum flux test under the same conditions, but with simultaneous in-line coagulation and ozone injection applied. Due to anticipated fouling reduction in this combined configuration the first flux step was increased to 147 L/m<sup>2</sup>.h. Further flux steps of 167 and 182 L/m<sup>2</sup>.h saw similar fouling pattern. The plot of initial TMP vs filtered volume displayed a variable trend with poor correlation co-efficient indicating longer filtration period would be needed to better estimate the fouling rate. Nevertheless a the flux of 182 L/m<sup>2</sup>.h showed only moderate fouling so a higher flux step was needed to determine maximum sustainable flux. However, the ozone injection equipment was unable to supply the target ozone dose at feed rates above 182 L/m<sup>2</sup>.h (or 4.6 m<sup>3</sup>/h). Diagnosis of this limitation found the high flow rate effluent over the static mixer restrictor was causing higher pressure than the ozone generator could overcome. As the ozone dose could not be supplied at the required level, higher flux steps would not be under equivalent condition and therefore were abandoned. But the significant potential of higher flux when ozone and coagulant were used together was noted.



Figure 14. Fouling rate as a function of volume filtered, ozonised and coagulated pre-treated effluent. Slopes were determined with units of bar/m<sup>3</sup>.

| Flux step<br>(L/m².h) | Calculated fouling<br>rate (TMP/day) | Correlation coefficient | Estimated CIP<br>frequency (days) |
|-----------------------|--------------------------------------|-------------------------|-----------------------------------|
| 147                   | 0.03                                 | 0.53                    | 38                                |
| 169                   | 0.02                                 | 0.09                    | 71*                               |
| 182                   | 0.27                                 | 0.42                    | 20                                |

Note: Poor correlation co-efficient of these trend lines render the estimation of CIP frequency unreliable. Longer filtration run would be required to resolve the trend

Maximum sustainable flux = between >  $182 \text{ L/m}^2$ .h

#### 4.5.5 Effect of Residual Ozone Quenching (Test Number 8, Table 3)

In an attempt to explore the mechanism when ozone is in the presence of the alumina ceramic membrane a controlled experiment was design to compare the filtration performance with and without ozone on the membrane surface. The hypothesis under test was that an excess of dissolved ozone in ozonised effluent on the membrane leads to lower fouling than the same ozonised effluent but without excess ozone. To test this, comparison of filtration performance was made using identical ozonised effluent with one stream having its ozone completely consumed. This was achieved by dosing a solution of chemical reducing agent into the ozonised effluent before the membrane.

Sodium metabisulphite (SMBS) is a reagent used to inhibit oxidation reactions and a known ozone quenching agent. The pilot plant possessed facilities to dose a SMBS solution into the reject water tank to neutralise hypochlorite CEB wastes before discharge. Further, the dosing point was easily relocated to the pipe between plant's feed water pump and membrane. References in the literature declare the stoichiometric relationship for the reaction of ozone with SMBS is 2.2 times more SMBS than ozone on a weight basis is required (Rakness 2005).



Figure 15. Flow diagram of SMBS dosing

Calculations to obtain SMBS dose rate were:

| Plant feed rate                     | 1.2 m <sup>3</sup> /h (ie flux of 50 L/m <sup>2</sup> .h) |
|-------------------------------------|---|
| Residual ozone conc.                | 1.5 mg/L (estimated upper level)                          |
| Ozone mass flow                     | 1.8 g/h   |
| SMBS demand (2.2 x O <sub>3</sub> ) | 4.0 g/h   |
| SMBS conc. in reservoir             | 6 g/L   |
| SMBS dose rate                      | 0.66 L/h  |

Initial testing confirmed that this dose rate quenched excess ozone, after some optimisation, determined by measurements of residual ozone content in sample point 3, just before the membrane. The residual ozone test provided a limit of detection of 0.001 mg/L, and therefore had sufficient sensitivity to confidently measure the absence of dissolved ozone.

The filtration performance profile of the quenching test is displayed in Figure 16. The structure of the experiment was to insert two quenching events into continuous operation and observe the fouling behaviour. The test progressed as follows:

- Plant started and normal operations were established with ozonisation resulting in excess ozone at approximately 1.0 mg/L at the membrane (individual test results display in green on Figure 16). Relatively stable TMP profile confirmed fouling was being controlled by combination of ozone and CEB regime.
- 2. Initiated SMBS dosing and confirmed efficient quenching with residual ozone analysis.
- Observed TMP rise from fouling. Shut-off SMBS dosing. But fouling was out of control and resumption of ozone could not lower TMP. Switched on coagulant for 7 hours to reduce TMP.
- 4. When TMP had reduced to approx. 0.7 bar coagulant was removed and stable TMP re-established. SMBS was again turned on. Residual ozone analysis again confirmed complete quenching.
- 5. TMP rise was observed. This time SMBS was used for only 8.5 hours and stopped before excessive fouling resulted.
- 6. After ozone quenching was stopped, fouling appeared to come under control with the TMP rise changing immediately to a decline.



Figure 16. TMP profile of quenching experiment

The performance gain with ozonisation pre-treatment before a ceramic membrane could be attributed to ozone reacting with the organics to prevent their build-up on the membrane, either because they are physically smaller, or because of their altered chemistry. This quenching study has now confirmed that the ozone effect is more than just size and altered chemistry, but dissolved ozone on the alumina membrane aids filtration performance. However, this work could not ascertain if the mechanism was enhanced hydroxyl radical formation or ongoing oxidation of the filter cake.

Further trials were planned to repeat the quenching test with combined ozonisation and coagulation to explore the effects of different filter cake structure but the test failed due to an inability to fully quench the ozone. It appeared that the addition of SMBS in the same region as coagulant dosing point caused the quenching agent to be bound to the coagulant or coagulated matter and was unavailable to react with ozone. A very high dose rate of SMBS was attempted yet residual ozone concentration at the membrane remained at 0.1 mg/L. This level of ozone would have some effect on the membrane surface, therefore rendering any result ambiguous so the test was abandoned.

#### 4.5.6 Low Frequency Chemical Cleaning (Test Number 7, Table 3)

A test was conducted to observe if the hypochlorite consumption used during CEBs could be reduced. At the highest flux achieved, 182 L/m<sup>2</sup>/h, the rate of hypochlorite CEB was every 2 hours for the program of a CEB after five regular backwashes. The TMP profiles for this typical operation were shown earlier (Figure 14). This was compared with the reduced frequency hypochlorite CEB to every 22 hours, and when no CEB was used at all (Figure 17). The standard run matched the target line for clean in place (CIP) of every 90 days. Applying hypochlorite CEB less often, to around once per day (every 22 hours), a major continuous rise in TMP was still not observed. This result showed that the coagulant and ozone acted to clean the membrane. However, TMP started to rise more significantly prior to the next scheduled CEB, after which the rise was out of control and the run was terminated. The TMP rise without CEB showed gradually increasing TMP rise leading to run termination at very high TMP.



Figure 17. TMP profiles of standard CEB run (every 2 hours), at reduced CEB frequency (every 22 hours) or no CEBs. Ozone and coagulation pre-treatment, Flux = 182 L/m2/h. Target line = CIP every 90 days.

The opportunity for reduced hypochlorite chemical use appears likely. While frequent hypochlorite CEBs were effective at maintaining a stable TMP rise, reducing their frequency leads to potential instability and subsequent rapid fouling. If chemical consumption was a key issue, then the major cleaning chemical, hypochlorite, can be significantly reduced if it was programmed to occur on feedback from a TMP trigger point (i.e. upon detection of rapidly rising TMP). This is suitable for highly variable feed waters such as secondary treated effluent, where higher fouling events occur from time to time. Therefore, the ceramic membrane system can maintain high fluxes and low chemical use, but engage more frequent hypochlorite CEBs only when needed. Flux can also be reduced to achieve more stable performance, but the compromise is less water productivity from the membranes.

#### 4.5.7 Organic Matter Degradation

Water quality indicators (true colour,  $UV_{254}$  absorbance and DOC) were routinely measured throughout the trial. An indicative representation of the fate of organic matter across the various process options are shown in Figure 18 (left) in terms of % reduction with respect to influent concentration at a flux of 50 L/m<sup>2</sup>.h. The results show little reduction of organic matter when no pre-treatment options were employed. This was to be expected as the MF membrane had little rejection of dissolved organic compounds. When coagulation was used approximately 20% reduction in colour and dissolved organic carbon was experienced, indicating this proportion of the influent existed in colloidal-like form or was absorbed onto the coagulant floc. The addition of ozone showed an expected reduction in colour measurements and some reduction in UV absorbance, but resulted in little DOC removal. This indicates the ozone reacts with chromophore-containing compounds but the organic matter was not mineralised.



Figure 18. Water quality indicator overall percent reduction (left) and percent reduction over the membrane only (right)

Results of organics reduction over the membrane alone are shown in Figure 18 (right). Colour was the only monitored parameter that reduced over the membrane. These indicate colour forming compounds measured by true colour undergo additional oxidation across the membrane whereas UV active compounds are not further treated by the membrane. This is likely to be due, in part to the slow kinetics of oxidation of coloured compounds by ozone. In the case of some coloured compounds for example, ozonisation occurs by slower molecular oxidation reactions (Ozbelge and Erol 2009). This would mean that a longer exposure time is required for oxidation and the oxidation would still be occurring through and after the membrane. Of greater interest, however, is the potential catalysis of this reaction that occurs at ceramic surfaces. Free hydroxyl groups on the membrane surface are known to act as adsorption sites for organic molecules that may then form complexes with ozone more readily and catalyses their oxidation (Timerghazin et al 1997). This too would result in the loss of colour seen after the ceramic membrane. Finally, the effect of the filtration itself must be taken into account. There is the possibility that some of the contribution to colour comes from particles larger than 0.1  $\mu$ m in size, but less than 0.45  $\mu$ m. This would be detected in the true colour measurements prior to the membrane, but not after due to the ceramic membrane's smaller pore size.

#### Organic Characterisation

Figure 19 shows organic characterisation results obtained from an LC-OCD for different pretreatments. This instrument separates the organic matter into broad classes of compounds and presents the relative proportions of each class contained in the water sample. The compound classes are listed below;

| Compound class                         | labelled as |
|--|-------------|
| Biopolymers                            | BP          |
| Humic substances                       | HUMIC       |
| Humic substance building blocks        | BB          |
| Low molecular weight neutral compounds | LMN         |
| Low molecular weight acid compounds    | LMA         |

As would be expected with no pre-treatment, only biopolymer was removed efficiently by the membrane. This implies that most of the TMP rise that occurs between backwashes is a result of accumulation of biopolymer, but also with input from smaller molecular weight substances by adsorption effects. The substances removed during backwashing are seen in the characterization of the backwash waters shown in Figure 20. Regular backwashes removed primarily biopolymer with a lesser amount of other materials. As evident from Figure 19, the addition of coagulant leads to aggregation primarily of humic substances and biopolymer that are then easily captured by the membrane. Ozone, on the other hand, was ineffective at improving removal efficiency across the membrane, but did result in a shift of the higher molecular weight compounds to lower molecular weight. This has led to an increase in the concentration of organics in the permeate and decreased

fouling as a result. Furthermore, the formation of low molecular weight acid compounds is also indicative of the functionalization of organic compounds by the ozone. The combination of the two has the most marked effect. The shift to lower molecular compounds is still apparent, as is the effect of functionalization. As with the ozone treatment, the combination would result in an overall increase in the organic content of the permeate.

The characterization of the backwash shows an interesting effect to help explain the fouling chemistry due to the combinations of coagulation, ozone and the membrane. These results are shown in Figure 20. As was noted earlier, regular backwash is largely effective at removing biopolymer and this would be the primary cause of TMP rise between backwashes. CEB with hypochlorite on the other hand removes significant quantities of humic substances. This indicates that TMP recovery as a result of hypochlorite backwashes is due to the oxidation and removal of the larger organics by hypochlorite. In comparing the organic content of hypochlorite backwashes under different pre-treatment regimes (Figure 20 right) we can see the important effect of coagulation. While ozone is effective in converting compounds to lower molecular weight materials, coagulation is effective in ensuring there is less organic matter to be removed during a hypochlorite backwash. Where coagulation is used, significantly lower concentrations of biopolymer and humic substances are seen in the hypochlorite backwash. While there are no results for regular backwashes the lack of an increase in TMP after backwashing where coagulation is used would be indicative of more efficient removal of the fouling layer. This implies that the adherence of these larger compounds is reduced by the use of coagulation. This could be the result of aggregation of the compounds and a more open filter cake. A more likely theory, however, is that the positively charged aluminium polymers in the coagulant are bonding preferentially with sites on the negatively charged organics that would also interact with the positively charged aluminium oxide/hydroxide at the membrane surface. Ozone is considered to increase the negative charge of the organic molecules. Consequently, organics that have been exposed to coagulant are less likely to be chemically bonded to the surface of the membrane allowing for easier removal during regular backwashes.



Figure 19. LC-OCD characterisations for the raw water, after pre-treatment and after filtration for different pre-treatment options.



Figure 20. Left; LC-OCD characterization of normal backwash and CEB for directly filtered (unpretreated) water. Right; characterization of hypochlorite CEB when different pre-treatments are utilized.

#### 4.5.8 Pathogen Inactivation (Test Number 9, Table 3)

#### MS2 challenge test (virus removal potential)

Table 6 lists the results of MS2 challenge testing carried out at 200 L/m<sup>2</sup>.h. The challenge test was carried out in duplicate with microbiological samples collected before and after the membrane at 5 min and 10 min into the filtration cycles. The size of the feed tank used to make up the challenge solutions limited the filtration time to 12 min. The overall LRV was found to be 4.0, determined as the more conservative of the two individual measurements. Microfiltration is not used at as virus barrier, but some studies on polymeric membranes have shown LRVs of 3 (DeCarolis and Adham 2007) and 2 to 3 (Huang et al 2012). In the first study, tests were performed in a membrane bioreactor, meaning a significant cake layer was likely to be present that would enhance removal. The second value for a PVDF membrane is likely to be more representative of polymer membranes. The difference between the polymer membrane and the ceramic membrane used here is likely to be due to different mechanisms of virus removal. In particular, the greater charge of the ceramic surface is likely to reduce the permeation of viruses through the membrane in the case of ceramics. This surface charge is not considered to be as strong in the case of polymeric membranes. This trial represents new data for virus removal using a ceramic membrane.

#### E.coli Removal (Bacteria and Protozoa Removal Potential)

To assess the LRV potential for bacteria, *E.coli* present in the secondary effluent was utilised as the challenge particle. The results displayed in Table 7 show the bacteria LRV under each pre-treatment condition. Microbiological samples were collected from feedwater, after the pre-treatment step and from the permeate. All permeate samples were found to contain no *E.coli* indicating the naturally occurring population was insufficient to estimate an absolute LRV. Further, consistent with the US EPA guidelines (US EPA 2005) conservative data treatment of LRVs dictates the lower result for each set is the final value. Therefore an estimate of overall LRV, or LRV<sub>C-test</sub> was 3.2

| MS2 Coliphage           |                       |                       |                        |                       |                 |  |
|-------------------------|-----------------------|-----------------------|------------------------|-----------------------|-----------------|--|
| Test                    | 1 2                   |                       |                        |                       | Achieved<br>LRV |  |
| Sample Time (min)       | 5                     | 10                    | 5                      | 10                    |                 |  |
| Feed<br>(pfu/100mL)     | 6.7 x 10 <sup>6</sup> | 6.9 x 10 <sup>6</sup> | 11.4 x 10 <sup>6</sup> | 7.6 x 10 <sup>6</sup> |                 |  |
| Permeate<br>(pfu/100mL) | 200                   | 700                   | 400                    | <100                  |                 |  |
| LRV overall*            | 4                     | .0                    | 4.3                    | 3                     | 4.0             |  |

Table 6 LRV results of MS2 coliphage, flux 200 L/m<sup>2</sup>.h, clean water (no ozone or coagulant)

\*Note: LRV = Log[lowest feed] - [highest permeate]

| NO PRE-TREATMENT  |   |  |   |  |   |   |   |  |  |   |
|---|---|--|---|--|---|---|---|--|--|---|
| Test  |   | 1  |   |  | 2   |   |   |  |  | Achieved<br>LRV   |
| Sample Time (min)   | 5   | 10   | 18  | 5  | 10  | 18  |   |  |  |   |
| Feed<br>(cfu/100mL)   | 2700  | 2700   | 2600  | 3300   | 2800  | 2900  |   |  |  |   |
| Permeate<br>(cfu/100mL)   | 1   | 1  | 1   | 1  | 1   | 1   |   |  |  |   |
| LRV overall*  |   | 3.4  | 1   |  | 3.4   | 1   |   |  |  | 3.4   |
|   |   |  | COAG  | ULATIO   |   | 1   |   |  |  |   |
| Test  |   | 1  |   |  | 2   |   |   |  |  | Achieved<br>LRV   |
| Sample Time (min)   | 5   | 10   | 18  | 5  | 10  | 18  |   |  |  |   |
| Feed<br>(cfu/100mL)   | 3100  | 28000  | 5200  | 2200   | 3900  | 3400  |   |  |  |   |
| Post Pre-treatment<br>(cfu/100mL)   | 3500  | 2400   | 5000  | 3000   | 6500  | 4400  |   |  |  |   |
| LRV Pre-treatment   |   | 0  |   |  | 0   |   |   |  |  | 0   |
| Permeate<br>(cfu/100mL)   | 1   | 1  | 1   | 1  | 1   | 1   |   |  |  |   |
| LRV Overall*  |   | 3.4  |   |  | 3.3   |   |   |  |  | 3.3   |
| OZONISATION ONLY  |   |  |   |  |   |   |   |  |  |   |
|   |   |  | OZON  | IISATIO  |   |   |   |  |  |   |
| Test  |   | 1  | OZON  | ISATIO   | N ONLY  |   |   | 3  |  | Achieved<br>LRV   |
| Test<br>Sample Time (min)   | 5   | 1<br>10  | 18  | 5  | 2<br>10   | 18  | 5   | 3<br>10  | 18   | Achieved<br>LRV   |
| Test<br>Sample Time (min)<br>Feed<br>(cfu/100mL)  | 5<br>3700   | 1<br>10<br>2800  | 18<br>3900  | 5<br>2500  | 2<br>10<br>3100   | 18<br>2900  | 5<br>3700   | 3<br>10<br>2900  | 18<br>3000                                     | Achieved<br>LRV   |
| Test<br>Sample Time (min)<br>Feed<br>(cfu/100mL)<br>Post Pre-treatment<br>(cfu/100mL)   | 5<br>3700<br>84   | 1<br>10<br>2800<br>89  | 18<br>3900<br>89  | 5<br>2500<br>91                                    | 2<br>10<br>3100<br>64   | 18<br>2900<br>120                                   | 5<br>3700<br>62                                       | 3<br>10<br>2900<br>39  | 18<br>3000<br>71                               | Achieved<br>LRV   |
| Test<br>Sample Time (min)<br>Feed<br>(cfu/100mL)<br>Post Pre-treatment<br>(cfu/100mL)<br>LRV Pre-treatment  | 5<br>3700<br>84   | 1<br>10<br>2800<br>89<br>1.5   | 18<br>3900<br>89  | 5<br>2500<br>91                                    | 2<br>10<br>3100<br>64<br>1.3  | 18<br>2900<br>120                                   | 5<br>3700<br>62                                       | 3<br>10<br>2900<br>39<br>1.6   | 18<br>3000<br>71                               | Achieved<br>LRV<br>1.3                                  |
| Test<br>Sample Time (min)<br>Feed<br>(cfu/100mL)<br>Post Pre-treatment<br>(cfu/100mL)<br>LRV Pre-treatment<br>Permeate<br>(cfu/100mL)   | 5<br>3700<br>84<br>1                                      | 1<br>10<br>2800<br>89<br>1.5<br>1  | 18<br>3900<br>89  | 5<br>2500<br>91<br>1                               | 2<br>10<br>3100<br>64<br>1.3<br>1   | 18<br>2900<br>120<br>1                              | 5<br>3700<br>62<br>1                                  | 3<br>10<br>2900<br>39<br>1.6<br>1  | 18<br>3000<br>71<br>1                          | Achieved<br>LRV<br>1.3                                  |
| Test<br>Sample Time (min)<br>Feed<br>(cfu/100mL)<br>Post Pre-treatment<br>(cfu/100mL)<br>LRV Pre-treatment<br>Permeate<br>(cfu/100mL)<br>LRV Overall*   | 5<br>3700<br>84<br>1                                      | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4   | 18<br>3900<br>89<br>1                                       | 5<br>2500<br>91<br>1                               | 2<br>10<br>3100<br>64<br>1.3<br>1<br>3.4  | 18<br>2900<br>120<br>1                              | 5<br>3700<br>62<br>1                                  | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5                                       | 18<br>3000<br>71<br>1                          | Achieved<br>LRV<br>1.3<br>3.4                           |
| Test<br>Sample Time (min)<br>Feed<br>(cfu/100mL)<br>Post Pre-treatment<br>(cfu/100mL)<br>LRV Pre-treatment<br>Permeate<br>(cfu/100mL)<br>LRV Overall*   | 5<br>3700<br>84<br>1<br><b>CO</b>                         | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4<br>MBINED   | 18<br>3900<br>89<br>1<br>OZONIS                             | 5<br>2500<br>91<br>1<br>SATION                     | 2<br>10<br>3100<br>64<br>1.3<br>1<br>3.4<br>AND C   | 18<br>2900<br>120<br>1                              | 5<br>3700<br>62<br>1<br>ATION                         | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5                                       | 18<br>3000<br>71<br>1                          | Achieved<br>LRV<br>1.3<br>3.4                           |
| Test Sample Time (min) Feed (cfu/100mL) Post Pre-treatment (cfu/100mL) LRV Pre-treatment Permeate (cfu/100mL) LRV Overall* Test   | 5<br>3700<br>84<br>1<br><b>CO</b>                         | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4<br>MBINED<br>1  | 18<br>3900<br>89<br>1<br>OZONIS                             | 5<br>2500<br>91<br>1<br>SATION                     | 2<br>10<br>3100<br>64<br>1.3<br>1<br>3.4<br><b>AND C</b><br>2   | 18<br>2900<br>120<br>1<br>OAGUL                     | 5<br>3700<br>62<br>1<br>ATION                         | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5<br>3                                  | 18<br>3000<br>71<br>1                          | Achieved<br>LRV<br>1.3<br>3.4<br>Achieved<br>LRV        |
| Test Sample Time (min) Feed (cfu/100mL) Post Pre-treatment (cfu/100mL) LRV Pre-treatment Permeate (cfu/100mL) LRV Overall* Test Sample Time (min)   | 5<br>3700<br>84<br>1<br><b>CO</b><br>5                    | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4<br>MBINED<br>1<br>10                                  | 020N<br>18<br>3900<br>89<br>1<br>020NI<br>18                | 5<br>2500<br>91<br>1<br>SATION<br>5                | N ONLY           2           10           3100           64           1.3           1           3.4           AND C           2           10  | 18<br>2900<br>120<br>1<br>OAGUL                     | 5<br>3700<br>62<br>1<br>ATION<br>5                    | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5<br>3<br>10                            | 18<br>3000<br>71<br>1<br>18                    | Achieved<br>LRV<br>1.3<br>3.4<br>Achieved<br>LRV        |
| Test Sample Time (min) Feed (cfu/100mL) Post Pre-treatment (cfu/100mL) LRV Pre-treatment Permeate (cfu/100mL) LRV Overall* Test Sample Time (min) Feed (cfu/100mL)  | 5<br>3700<br>84<br>1<br><b>CO</b><br>5<br>3400            | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4<br>MBINED<br>1<br>10<br>2600                          | 18<br>3900<br>89<br>1<br>0ZONIS<br>18<br>3100               | 5<br>2500<br>91<br>1<br>SATION<br>5<br>2400        | N ONLY           2           10           3100           64           1.3           1           3.4           AND C           2           10           2.700  | 18<br>2900<br>120<br>1<br>OAGUL<br>18<br>3300       | 5<br>3700<br>62<br>1<br>ATION<br>5<br>2400            | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5<br>3<br>3<br>10<br>2000               | 18<br>3000<br>71<br>1<br>1<br>18<br>3100       | Achieved<br>LRV<br>1.3<br>3.4<br>Achieved<br>LRV        |
| Test Sample Time (min) Feed (cfu/100mL) Post Pre-treatment (cfu/100mL) LRV Pre-treatment Permeate (cfu/100mL) LRV Overall* Test Sample Time (min) Feed (cfu/100mL) Post Pre-treatment (cfu/100mL)   | 5<br>3700<br>84<br>1<br>1<br><b>CO</b><br>5<br>3400<br>76 | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4<br><b>MBINED</b><br>1<br>10<br>2600<br>76             | 18<br>3900<br>89<br>1<br>0ZONIE<br>18<br>3100<br>120        | 5<br>2500<br>91<br>1<br>SATION<br>5<br>2400<br>180 | N ONLY           2           10           3100           64           1.3           1           3.4           AND C           2           10           2700           150                           | 18<br>2900<br>120<br>1<br>OAGUL<br>18<br>3300<br>64 | 5<br>3700<br>62<br>1<br>1<br>ATION<br>5<br>2400<br>35 | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5<br>3<br>10<br>2000<br>27              | 18<br>3000<br>71<br>1<br>1<br>18<br>3100<br>74 | Achieved<br>LRV<br>1.3<br>3.4<br>Achieved<br>LRV        |
| Test         Sample Time (min)         Feed<br>(cfu/100mL)         Post Pre-treatment<br>(cfu/100mL)         LRV Pre-treatment         Permeate<br>(cfu/100mL)         LRV Overall*         Test         Sample Time (min)         Feed<br>(cfu/100mL)         Post Pre-treatment<br>(cfu/100mL)         LRV Pre-treatment<br>(cfu/100mL) | 5<br>3700<br>84<br>1<br>1<br><b>CC</b><br>5<br>3400<br>76 | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4<br>MBINED<br>1<br>10<br>2600<br>76<br>1.5             | 020N<br>18<br>3900<br>89<br>1<br>020NI<br>18<br>3100<br>120 | 5<br>2500<br>91<br>1<br>SATION<br>5<br>2400<br>180 | N ONLY           2           10           3100           64           1.3           1           3.4           AND C           2           10           2700           150           1.1             | 18<br>2900<br>120<br>1<br>0AGUL<br>18<br>3300<br>64 | 5<br>3700<br>62<br>1<br>1<br>ATION<br>5<br>2400<br>35 | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5<br>3<br>10<br>2000<br>27<br>1.4       | 18<br>3000<br>71<br>1<br>1<br>18<br>3100<br>74 | Achieved<br>LRV<br>1.3<br>3.4<br>Achieved<br>LRV<br>1.1 |
| Test Sample Time (min) Feed (cfu/100mL) Post Pre-treatment (cfu/100mL) LRV Pre-treatment Permeate (cfu/100mL) LRV Overall* Test Sample Time (min) Feed (cfu/100mL) Post Pre-treatment (cfu/100mL) LRV Pre-treatment (cfu/100mL)   | 5<br>3700<br>84<br>1<br>1<br>5<br>3400<br>76<br>1         | 1<br>10<br>2800<br>89<br>1.5<br>1<br>3.4<br><b>MBINED</b><br>1<br>10<br>2600<br>76<br>1.5<br>1 | 18<br>3900<br>89<br>1<br>0ZONI<br>18<br>3100<br>120         | 5<br>2500<br>91<br>1<br>SATION<br>5<br>2400<br>180 | N ONLY           2           10           3100           64           1.3           1           3.4           AND C           2           10           2700           150           1.1           1 | 18<br>2900<br>120<br>1<br>0AGUL<br>18<br>3300<br>64 | 5<br>3700<br>62<br>1<br>1<br>5<br>2400<br>35<br>1     | 3<br>10<br>2900<br>39<br>1.6<br>1<br>3.5<br>3<br>10<br>2000<br>277<br>1.4<br>1 | 18<br>3000<br>71<br>1<br>1<br>18<br>3100<br>74 | Achieved<br>LRV<br>1.3<br>3.4<br>Achieved<br>LRV<br>1.1 |

Table 7. LRVs for E.coli through the ceramic membrane and under various pre-treatments

\*LRV = log[lowest feed] – log[highest permeate]

| No Ozone                      |      |           |     |      |      |      |      |       |
|-------------------------------|------|-----------|-----|------|------|------|------|-------|
| Pre-Treatment                 |      | No        | ne  |      | C    | coag | ular | nt    |
| Test                          | 1    |           |     | 2    | 1    |      |      | 2     |
| Feed<br>(cfu/100mL)           | 2667 | 2667 3000 |     | 3000 | 3700 |      | ÷    | 3167  |
| Backwash Water<br>(cfu/100mL) | 2350 | 0         | 4   | 3500 | 6900 | 0    | 6    | 6000  |
| LRV absolute                  | -0.9 |           |     | -1.2 | -1.3 |      |      | -1.3  |
| LRV average                   |      |           |     | -1.  | 2    |      |      |       |
|                               | Wi   | ith C     | zon | е    |      |      |      |       |
| Pre-Treatment                 |      | Ozo       | one |      | Ozor | ie+C | oag  | ulant |
| Test                          | 1    | 1 2 3     |     |      | 1    | 2    | 2    | 3     |
| Feed<br>(cfu/100mL)           | 3467 | 28        | 33  | 3200 | 3033 | 28   | 00   | 2500  |
| Backwash Water<br>(cfu/100mL) | 1600 | 27        | 00  | 565  | 495  | 68   | 5    | 510   |
| LRV absolute                  | 0.3  | 0         | .0  | 0.8  | 0.8  | 0.   | 6    | 0.7   |
| LRV average                   |      |           |     | 0.5  | 5    |      |      |       |

Table 8. LRV of E.coli in Backwash water, 150 L/m<sup>2</sup>.h

Table 8 show the results of viable *E.coli* measured in the backwash water. Negative values indicate a concentrating of bacteria, which is to be expected considering the membrane is an absolute barrier to bacteria sized organisms and all filterable material leave the process via the backwash. The pilot plant was configured for a water recovery of 93% which represents a 14-fold concentration of filtered solids in the backwash water. This concentration factor concurs with the determined LRV of -1.2 found between feed and backwash waters. However, when ozonisation was applied disinfection of the backwash water was observed. Measurements of LRV ranged between 0 and 0.8 log but needs to be referenced to the -1.2 log of un-ozonised pre-treatment. Therefore, when conservatism is applied to the test LRVs this results in a determined disinfection enhancement of 0.4 to 0.6 log when ozone is used. This is evidence of inactivation of *E.coli* on the membrane surface from a continuous stream of ozonised water that flows for the entire filtration cycle.

Therefore, the difference in pathogen concentration in backwash water when ozonisation is employed compared to no ozonisation can be expressed as a log reduction value as:

But ozonisation alone confers some disinfection (found to be 1.3 log alone, and 1.1 log with coag) so the log reduction attributed to using excess ozone on the membrane is:

#### 4.5.9 Pressure Decay Test Results

The PDT was carried out on 8 occasions during the trial period. On one occasion the test was performed without a CIP routine first, and one another occasion a limited CIP process was followed. The tests performed on un-cleaned membrane are indicated. As the membrane was received in a used state no baseline PDT could be performed.

These results confirm the membrane was installed correctly and the separation layer was intact. Also, over the course of the trial no undue damage resulted from chemical attack or pressure induced displacement of the sealing mechanisms. From the EPA Guideline manual (Ref) a system with 4 log credit would need to demonstrate a PDT of better (lower) than 1 kPa/min.

| Data of PDT | Test pressure<br>(bar) | Result<br>(kPa/min) | Comment                         |
|-------------|------------------------|---------------------|---------------------------------|
| 27/07/2012  | 1.32                   | 0.313               |                                 |
| 21/11/2012  | 1.22                   | 0.318               |                                 |
| 16/01/2013  | 1.24                   | 0.046               | PDT done before a CIP           |
| 1/3/2013    | 1.44                   | 0.438               |                                 |
| 18/3/2013   | 1.22                   | 0.185               | Only partial CIP performed      |
| 3/4/2013    | 1.25                   | 0.375               |                                 |
| 12/6/2013   | 1.30                   | 0.237               |                                 |
| 17/06/2013  | 1.34                   | 0.185               | CIP done but had little effect. |

#### Table 9. Pressure decay test results

To explore the effect of different test pressures an experiment was performed at 3 pressures. The graph below show the pressure decay profile over 10 min at 1.0, 1.3 and 1.8 bar. As expected, the pressure decay rate was proportional to applied pressure. See Section 4.4.4 for details of the criteria for PDT results.



Figure 21. Pressure decay curves at different pressures

### 4.6 Discussion on findings

This project's goal was to explore the benefits of the ceramic membrane / ozonisation process for water recycling of secondary effluent under typical operating conditions. Therefore a primary focus was the operability and cost of such a process, thus control of membrane fouling and the determination of a maximum practical permeate flux were key objectives. Summarising the plant trial results finds the enhanced filtration performance when ozonisation and coagulation were employed together as the central finding. Closely examining the filtration performance of each pre-treatment option helps to interpret the possible mechanism at play.

Overlaying TMP profiles of the four pre-treatment combinations reveals a clearer illustration of the benefits (Figure 22). These graphs show the regular TMP rise of each filtration cycle as the cake accumulates then a sudden drop as a BW occurs. The period of these data were selected between CEB events for clarity. The amount of TMP rise per filtration cycle and the degree to which TMP returns to initial TMP are key performance indicators. The difference between the profiles highlights the incremental effect of each pre-treatment. Compared to no pre-treatment (black line), ozonisation prevented TMP rise between backwash cycles (red line), indicating ozone reduces the fouling nature of the influent. This could be due to ozone reacting with the organics which prevents their build-up on the membrane, either because they are physically smaller, or because of their altered chemistry. This was observed in previous studies of this group (Zhu et al. 2011). On the other hand, the TMP profile of coagulant pre-treated effluent (green line) displays a similar but lower TMP rise to no pre-treatment, indicating a more permeable filter cake structure (as described earlier is Section 4.5.7.

However, it is the combination of coagulation and ozonisation that shows a dramatic effect. The TMP profile (blue line) shows no rise per filtration cycle nor does it display a rising trend of long term fouling. There are a number of mechanisms for this change in filtration behaviour. As was noted in the organic characterization (Section 4.5.7), the use of coagulant is vital in reducing fouling that

requires the use of hypochlorite for removal. It seems likely that this is due to the interaction of the biopolymers and humic substances with the aluminium polymers in solution, and would reduce their potential for interaction with the aluminium oxide surface. This effect means that the TMP almost fully recovers after a regular backwash. Where coagulant is not used, a gradually increasing TMP would be expected until a CEB is performed. Ozonisation results in a decrease in the TMP rise between backwashes due to the decrease in large molecular weight compounds that otherwise foul the membrane. Potential changes in the charge on organic compounds in the water would also have the effect of decreasing fouling potential. Finally the combination produces a synergistic effect with no TMP rise. This was also the pre-treatment option that chemical analysis showed the most significant increase in low molecular weight acids that are likely to associate with the Al<sup>3+</sup> coagulant prior to the membrane surface, reducing the likelihood of interaction with the membrane surface. The combined pre-treatment will reduce the interaction of both high and low molecular weight compounds that interact with the membrane, allowing for a less dense filter cake at the membrane surface and lower TMP as a result.



Figure 22. TMP profile overlay of all pre-treatment options

One of the project's goals was to explore the possibility using the membrane cleaning effect of ozonisation to offset regular chemical cleaning, and the results in Figure 22 offers encouragement to this suggestion. The filtration profile of combined ozonisation and coagulation, at a flux of 50 L/m<sup>2</sup>.h, would indicate scope for significant reduction in CEB frequency, and even reduced BW frequency, leading to high water recovery operation. However, in all membrane processes the degree of fouling is proportional to flux. When operating at high flux, such as 150 L/m<sup>2</sup>.h or higher, fouling can be controlled by ozone and coagulant pre-treatments, and indeed allows this flux to be maintained. However, it was found that high flux operation cannot be combined with reduced chemical cleaning, at least with these types of challenging feed waters. A number of tests were attempted (section 4.5.6) but a frequency of hypochlorite CEBs below the 'after 5 hydraulic BWs' when operating at a flux of 182 L/m<sup>2</sup>.h resulted in significant fouling.

# 5 Plant Costing

A costing assessment was carried out comparing a plant based on the ceramic membrane technology used in this trial to that of a typical polymer based membrane plant, both being use for water recycling in Australia. To ensure an impartial assessment, the costing was led by Victoria University and Black & Veatch only. However project partners contributed information to conduct the cost assessment. See Attachment 1 for the full cost report.

Data was obtained from testing carried out on both membrane types at Eastern Treatment Plant (ETP), Melbourne, to measure performance needed to determine both capital costs (CAPEX) and operating costs (OPEX). Ceramic membrane plant cost data was provided by PWN Technologies, and polymer membrane plant cost data was sourced from actual construction costs of two plants built within the last five years and operating in Australia. Net present value (NPV) analysis was also carried out for both ceramic and polymeric membrane trains over both 25 and 20 year plant lifetimes. Both ceramic and polymeric membrane process trains included coagulation and ozone operation. This was done because the additional operations led to high ceramic membrane flux which is one key aspect in offsetting their higher material costs. The basis of the costing was for a plant capacity of 22 million litres per day (MLD). This was based on a medium scale water recycling plant that had the potential to provide water to a number of industrial, agricultural and municipal users.

The cost results are shown in the Table 10 below. Option 1 and Option 2 present the case where the ceramic membranes are replaced after 15 years, or not replaced at all, respectively. Ceramic membrane warranties of 20 years are now available. This could possibly be increased up to 25 years, suggesting Option 2 represents a realistic scenario. Option 3 is the polymer membrane plant cost, which includes replacement after every 7 years. It is apparent that the ceramic membrane plant was higher in CAPEX by 10.5%, but lower in OPEX. In terms of NPV, the ceramic membrane plant was cheaper than the polymer membrane plant in its 25 year life in both Options 1 and 2, which was due to the lower OPEX.

Therefore, the other benefits of ceramic membrane such as high integrity, long life and chemical robustness can be realised in water recycling schemes as being economically comparable to polymer membranes.

| Option   | CAPEX        | OPEX (per annum) | NPV (25 years)                                |
|--|--------------|------------------|---|
| Option 1 – Ceramic membrane<br>system with module design life 15<br>years  | \$11,792,000 | \$1,316,900      | \$32,514,000<br>(\$0.179 per m <sup>3</sup> ) |
| Option 2 – Ceramic membrane<br>system without module<br>replacement        | \$11,792,000 | \$1,085,000      | \$30,684,000<br>(\$0.169 per m <sup>3</sup> ) |
| Option 3 – Polymeric membrane<br>system with module design life 7<br>years | \$10,668,000 | \$1,381,800      | \$33,292,000<br>(\$0.183 per m <sup>3</sup> ) |

| Table 10. Costing results for ceramic or polymer membrane plants. 7 | The cost includes |
|---|-------------------|
| coagulation and ozone. Plant capacity is 22 MLD.                    |                   |

This cost assessment assumed a flux of 200 L/m<sup>2</sup>/h for the ceramic membrane system, which is achieved from the contact of ozone on the ceramic membrane when treating recycled water. However, not all plant operators require ozone treatment to the water, so the highest ceramic membrane fluxes would not be realised. Therefore the effect of reduced flux to cost is of interest for these circumstances not involving ozone. A flux of 130 L/m<sup>2</sup>/h could be considered a sustainable operation flux for a coagulant only plant based on the results shown in Figure 12. As CAPEX shown in Table 10 was higher for ceramic membranes than polymer membranes when operating at 200 L/m<sup>2</sup>/h with ozone (and coagulant), operating at a lower flux will increase the CAPEX in proportion to polymer membranes.

However, the cost comparison does indicate that whole of life (NPV) costs for ceramic membrane and polymer membrane systems are comparable, so consideration of both ceramic and polymeric membrane options for projects appears economically justified.

# 6 Benefits of the Technology

In addition to the previously stated benefits of ceramic membranes in combination with ozonisation, 10 months of operation have highlighted three main benefits of this approach. This first is the ability of the combined ozonisation/ceramic membrane filtration to deal with challenging waters (in this case a municipal secondary effluent) has been proven at a pilot scale. This implies that there is a robustness to the technology that would potentially allow it to deal with some variability in the water source. This leads to the second point – experience in operating the plant at the ETP site has shown that optimizing and tuning the plant can be performed rapidly even when the water is not fully understood or undergone initial trialling. Importantly, the robustness of the ceramic membrane means that less care is needed in ensuring sufficient holding time to reduce the ozone residual in water. Ozone was detected in trace amounts in the membrane permeate. Managing this would be particularly important in recycled water operations, for example where desalination via reverse osmosis would follow the microfiltration step i.e. the ozone must be completely removed prior to feeding to the downstream reverse osmosis membranes. It may be beneficial to install a biological media filter, which is typically applied after ozone processes, to remove ozone prior to downstream membrane processing.

The configuration of the technology means that changes in water quality could be quickly met by changes in chemical addition or CEB frequency. This robustness would be significant in challenging waters where variations due to seasonal effects, wet weather events or other phenomena require rapid and robust response. Importantly, the size of the plant is dictated purely by flow and does not require precise holding times for settling or to decrease residuals. This has the added benefits of reducing the footprint of the treatment process and permits easy production rate changes. The simplicity associated with this also makes the technology suitable for operation in remote locations. Finally, the higher fluxes achievable in this study using ceramic membranes have meant that a high turn down ratio can be used in practice. This allows for greater scope to deal with large peaks flows such as during wet weather events.

When operated at lower flux, this trial found the potential to greatly reduce chemical cleaning frequency and associated operating costs. However, the cost of this was not assessed as the installed membrane area would need to be significantly higher. Nevertheless, a remote location, where transport costs are high or not readily available, may find economic benefits by operating a low flux with greatly reduced chemical cleaning requirement.

# 7 Conclusions

These results report the performance outcomes of the hybrid ceramic membrane/ozonisation process for secondary effluent. Plant trial conditions over 10 months, while controlling many operational parameters, found a demonstrable increase in plant performance when the hybrid process was employed. The structured experiments undertaken in this study analysed the relative filtration performance under four distinct pre-treatment options.

Secondary effluent without any pre-treatment applied to the ceramic membrane process alone achieved a permeate flux of 50 L/m<sup>2</sup>.h, determined to be sustainable over the long term. The addition of in-line polyaluminium chloride coagulant led to enhancement of sustainable flux, which reached and exceeded 100 L/m<sup>2</sup>.h. Employing ozonisation before the membrane process resulted in a modest increase in sustainable flux compared to no ozonisation. It was when ozonisation and coagulation were applied together that significant increases in flux were possible. Injection of ozone at the maximum rate allowable for the equipment saw an estimated long term sustainable fluxes of at 182 L/m<sup>2</sup>.h. Higher fluxes appeared to be very likely but the ozone injection equipment prevented experimentation of this. The function of coagulant and ozone appeared to offer synergistic improvements in membrane performance. Coagulant assisted TMP rise reversibility during backwash, while ozone reduced the TMP rise between backwashes. Together, these effects yielded the higher fluxes achieved.

The significant performance improvement when ozonisation and coagulation was employed allowed for the potential of reduction in chemical cleaning (reduced CEB frequency), but only when operating at low permeate flux. However, the performance advantage of combined pre-treatment would most likely be better exploited by high design flux and correspondingly smaller plant size. This does not preclude a viable low flux low chemical operation, indeed this configuration may find a useful application in remote areas where chemical availability is poor and delivery costs are very high or prohibitive.

MS2 challenge testing estimated an LRV(virus) of 4.0 attributed to the membrane process alone. The absolute bacterial LRV was not determined as no *E.coli* was detected in the permeate. Therefore, the removal calculation could not be made, but an LRV of >3.2 was estimated. The concept of enhanced pathogen inactivation in the reject stream was confirmed with an LRV of 0.5 between feed and reject waters. This contrasts with a 1.2 log increase in the reject stream when ozone was not employed, which leads to the reject water disinfection LRV of 1.7. While the challenge testing indicated expected performance across microfiltration membranes, it is the enhanced backwash pathogen disinfection that is unique to this ozone/ceramic membrane hybrid process. This has benefits for operations where disinfected reject water would be useful with respect to disposal strategies.

This work, therefore, supports the performance benefits proposed by ceramic membranes in conjunction with ozone. High flux operation for challenging waters such as secondary effluent and enhanced total disinfection are unique features which have significant benefits for wastewater recycling. The potential for reduced chemical cleaning exists but the ceramic membrane features of robustness and longevity can be most likely better exploited through smaller plant size and greater operational freedom.

The cost comparison between ceramic and polymeric membrane plants has been completed in the context of water recycling in Australia. It showed that high flux and long life of ceramic membranes are the key features that reduce their CAPEX and OPEX amounts. When the flux of 200 L/m<sup>2</sup>/h is reached on ceramic membranes with the use of coagulant and ozone, the economics of the equivalent process using polymer membranes become comparable. There are slight benefits to CAPEX for polymer membranes (10.5% cost favourable to polymers), but OPEX showed lower annual operating costs for ceramic membranes because they are replaced less often. Therefore, the other benefits of ceramic membrane such as high integrity, long life and chemical robustness can be realised in water recycling schemes as being economically comparable to polymer membranes.

# 8 Acknowledgements

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Appendix A

Figure 23. TMP profile of entire run, no pre-treatment, 100LMH



Figure 24. TMP profile of entire run with coagulation pre-treatment, 100LMH



Figure 25. TMP profile of entire run with ozonisation, 100LMH



Figure 26. TMP profile of entire run, combined ozonisation and coagulation, 169LMH

**Attachment 1. Cost Comparison Report** 



# **Cost comparison of ceramic and polymeric membranes for water recycling**

Project title "Demonstration of low maintenance chemical free recycling of secondary treated effluent by ceramic membranes"

May 2014 (revised August 2014)



Report and data sourcing by Mikel Duke and Noel Dow of Victoria University



Report, cost assessment and data sourcing by Kieran Barriscale and Hazel Ho of Black & Veatch

# **Executive summary**

This report summarises the costing assessment and outcomes comparing ceramic and polymer membranes for water recycling in Australia. Testing was carried out on both membrane types at Eastern Treatment Plant (ETP), Melbourne, to measure performance needed to determine both capital costs (CAPEX) and operating costs (OPEX). Ceramic membrane plant cost data was provided by PWN Technologies, and polymer membrane plant cost data was sourced from actual construction costs of two plants built within the last five years and operating in Australia. Net present value (NPV) analysis was also carried out for both ceramic and polymeric membrane process trains included coagulation and ozone operation. This was done because the additional operations led to high ceramic membrane flux which is one key aspect in offsetting their higher material costs. The basis of the costing was for a plant capacity of 22 million litres per day (MLD).

The cost results are shown in the table below. Option 1 and Option 2 present the case where the ceramic membranes are replaced after 15 years, or not replaced at all, respectively. Ceramic membrane warranties of 20 years are now available. This could possibly be increased up to 25 years, suggesting Option 2 represents a realistic scenario. Option 3 is the polymer membrane plant cost, which includes replacement after every 7 years. We see that the ceramic membrane plant was higher in CAPEX by 10.5%, but lower in OPEX. In terms of NPV, the ceramic membrane plant was cheaper than the polymer membrane plant in the 25 year life case in both Options 1 and 2, which was due to the lower OPEX.

| Option  | САРЕХ        | OPEX<br>(per annum) | NPV (25 years)                                |
|---|--------------|---------------------|---|
| Option 1 – Ceramic<br>membrane system with<br>module design life 15<br>years  | \$11,792,000 | \$1,316,900         | \$32,514,000<br>(\$0.179 per m <sup>3</sup> ) |
| Option 2 – Ceramic<br>membrane system<br>without module<br>replacement        | \$11,792,000 | \$1,085,000         | \$30,684,000<br>(\$0.169 per m <sup>3</sup> ) |
| Option 3 – Polymeric<br>membrane system with<br>module design life 7<br>years | \$10,668,000 | \$1,381,800         | \$33,292,000<br>(\$0.183 per m <sup>3</sup> ) |

Costing results for ceramic or polymer membrane plants. The cost includes coagulation and ozone. Plant capacity is 22 MLD.

The high fluxes that lead to these favourable costs for ceramic membranes are tied to the use of ozone. However the ozone will also offer a function in the water recycling scheme, where it is typically used to reduce colour, taste and odour features of water, as well as to provide disinfection. Coagulation is also commonly used in water treatment schemes for organic removal. Therefore ozone and coagulation have also been included in the cost assessment of the polymeric membrane system (Option 3), having similar effect on the recycled water. However ozone may not be required in the water treatment scheme being considered, so an understanding of the effect to relative cost to polymer membranes when not using ozone would be of interest. Operating without ozone (using only coagulant) will reduce flux. A flux of  $130 \text{ L/m}^2$ /h has been considered as a sustainable operation flux for a coagulant only plant based on the results from this project. Since CAPEX was higher for ceramic membranes than

polymer membranes when operating at 200  $L/m^2/h$  with ozone (and coagulant), operating at a lower flux will increase the CAPEX in proportion to polymer membranes.

This assessment has shown that ceramic membranes operating at high flux have better economics than polymer membranes over the life of the plant. For water recycling, this means the other benefits of ceramic membranes, such as their high integrity and chemical robustness can be realised without additional cost when compared to polymer membranes.

As competitively built ceramic membrane installations are still emerging, the cost of ceramic membrane systems are decreasing. Polymer membrane systems may also experience cost reductions or improved pricing efficiencies over time. Therefore an updated cost model will be required in future due to the cost saving developments in the technologies.

# Introduction

This report summarises a cost comparison made between ceramic and polymeric membranes in a non-site specific water treatment plant, but based on performance results from trials on a specific secondary treated effluent. The costing is one of the key objectives for a research project led by Victoria University titled "Demonstration of low maintenance chemical free recycling of secondary treated effluent by ceramic membranes" which received funding from the Australian Water Recycling Centre of Excellence, Water Research Australia Ltd, Melbourne Water Ltd and South East Water Ltd. PWN Technologies (The Netherlands) partnered directly on the project provided the ceramic membrane pilot plant and expertise in its operation.

The project team and collaborators includes:

- *Victoria University:* Mikel Duke (project lead), Noel Dow, Julia Roehr, Stephen Gray, Nicholas Milne, Bo Zhu
- Melbourne Water: Judy Blackbeard, Danny Murphy, John Mieog, Lisa Solomon
- South East Water: Pam Kerry, Ann Gooding
- Water Research Australia: David Halliwell, Gareth Roeszler
- Black and Veatch: James Currie, Kieran Barriscale, Hazel Ho
- PWN Technologies: Jonathan Clement, Gilbert Galjaard

The costing was carried out by Hazel Ho and Kieren Barriscale with assistance from Noel Dow and Mikel Duke. Costs of the polymer membrane plants were sourced from two water recycling plants built in Australia. There are referred to as Polymer Plant A and Polymer Plant B.

The purpose of this cost assessment was to compare the cost differences between two recycling treatment plant trains, one containing ceramic membranes and the other containing polymer membranes. Polymer membranes have succeeded globally, and are regarded as a highly efficient and low cost technology in water treatment. However, ceramic membranes are emerging to resolve integrity and robustness limitations of polymer membranes, mostly in drinking water and industrial water treatment. Ceramic membranes have been commercial for decades, being primarily used in food applications where their higher costs have been justified by the value of the components they separate (e.g. dairy proteins). For water treatment however, the cost of the membranes and plants designed for food applications are prohibitive in water treatment. Cost reduction would therefore expand the application of ceramic membranes. With the introduction of large compact elements such as Metawater's 25 m<sup>2</sup> monolith, together with PWN Technologies' compact CeraMac design, ceramic membranes and their equipment are now becoming cost effective for water treatment. While progress is being made in drinking water installations worldwide (e.g. in Japan and the Netherlands), there is only one plant in operation for water recycling globally that uses ceramic membranes, and this is located in Japan. However, water recycling could greatly benefit from ceramic membranes through their inherent robustness making them suitable for 'challenging' wastewaters, but also they can offer excellent integrity over their long life (>15 years) as a pathogen and turbidity barrier. Yet their higher price per  $m^2$  still suggests that ceramic membranes are too expensive to be successful in water recycling. Therefore a key question to be answered in this assessment for water recycling is 'does the higher flux performance and longer life of ceramic membranes offset their higher material costs?"

The performance data utilised to make the cost estimation came from the ceramic membrane pilot plant trial at Melbourne Water's Eastern Treatment Plant (ETP). Polymer membrane performance data for estimating operating costs was taken from previous trials also undertaken at ETP. The capital cost of polymer membrane plants was sourced from Polymer Plant A and Polymer Plant B.

### **Process arrangement**

The ETP ceramic membrane pilot plant trial revealed that ozone in combination with coagulation was key to the high flux performance which in turn leads to reduced number of membranes used for the same treatment capacity. Therefore, the process arrangement used for this cost exercise was ozone treatment, followed by in-line coagulation, then ceramic membrane filtration. This arrangement is shown in Figure 1. The equivalent polymer membrane process was proposed as having the same treatment train, but the polymer membrane is likely to be placed prior to the ozone process following the current practice in industry. Cases where ozone is applied prior to the polymer membranes typically include a biological filter between the ozone and membrane to digest organics. While performance data is available for this arrangement, the effect of the biological filter on the organics alters membrane performance and is thus not similar to ozone followed directly by membranes. Further, ozone would not be applied immediately prior to polymer membranes due to its effect to oxidise the polymer material. Therefore the two process trains selected for comparison are as shown in Figure 1.



*Figure 1:* equivalent process trains using ceramic and polymeric membranes used for the cost comparison

As increasing flux is critical to reducing plant costs, it has been argued that placing the ozone prior to the polymer membrane will have a similar flux increasing effect. The only evidence of the flux potential found in this assessment on secondary treated effluent was from the polymer membrane trials at ETP. In this case, a biological filter was applied between the ozone and the UF. While this too should assist in removing organic load to the membranes and lead to increased flux, the experience from this trial showed fluxes reaching up to 70  $L/m^2/h$ . This is still lower than the ceramic membrane fluxes achieved in the project of 182  $L/m^2/h$ . Therefore, the similar process for polymer membranes to substantially increase flux

with ozone pre-treatment has not been witnessed and therefore could not be considered in this assessment.

# **Costing setup**

The two membrane trains were costed based on a design capacity of 22 million litres per day (MLD). The assumptions made to perform the cost model are provided in this section.

# **Cost model assumptions**

- 1. Costing was performed using Australian cost data and all costs are presented in AUD.
- 2. Annual inflation is 3%;
- 3. Equipment delivery cost is 10% of equipment cost;
- 4. Equipment installation cost is 25% of equipment cost;
- 5. Electrical and Instrumentation (E&I) cost is 17.5% of total civil and mechanical costs based on previous project estimate;
- 6. Testing and Commissioning cost is 2% of total civil, mechanical and E&I costs based on previous project estimate;
- 7. Indirect costs (Design, Project / Construction Management, Margin and Risk) is 60% of the total direct cost;
- 8. Power unit rate is \$0.15/kWh;
- 9. Chemical dosing system power consumption is negligible;
- 10. Operation cost is 1.5% of CAPEX and maintenance cost is 1% of CAPEX; and
- 11. Assume the plant operates 330 days per year (approx. 90% availability).

# Net present value (NPV) analysis

- 1. Conducted for 20 years and 25 years asset life respectively; and
- 2. Assume discount factor 3%.

### **Building/civil assumptions**

- 1. Footprint for UF membranes taken from plant located in Australia;
- 2. Chemical storage area for ceramic membrane system taken from plant located in Western Australia; and
- 3. Chemical storage area for polymer membrane taken from Polymer Plant B.

### **Operating assumptions**

- 1. Chemical costs provided by Australian vendor;
- 2. Power consumption for polymer plants taken from average power consumption of Polymer Plant A and Polymer Plant B;

- 3. Ozone power consumption provided by vendor determined for 9,000 g/h O<sub>3</sub>. This correlates to an ozone dose rate of approximately 10 mg/L;
- 4. Replacement cost provided from Polymer Plant B; and
- 5. The number of operators is the same for both polymer and ceramic membrane plants

# **Chemical consumption**

The chemicals required for sustainable flux operation determined from trials of both polymer membranes and ceramic membranes at Eastern Treatment Plant as shown in Table 1. These chemicals are used for chemically enhanced backwashes (CEB) and clean in place (CIP). The CEB is performed more frequently and does not lead to long plant downtime. However as this clean is typically insufficient over the longer term, a more rigorous clean is needed where the operation is stopped for a longer time. This is known as the CIP. Chemicals used for both CEB and CIP vary according to the type of water treated, the type of membrane and the membrane's performance (i.e. flux and water recovery). Based on trials at Eastern Treatment Plant, the time between CIP was determined as 90 days for ceramic membranes, and 30 days for polymer membranes.

| Chemicals         | Functions               | Chemical consumption                              |        |     |  |  |  |  |
|-------------------|-------------------------|---|--------|-----|--|--|--|--|
|                   |                         | (L of as delivered chemical per ML water treated) |        |     |  |  |  |  |
|                   |                         | Dosing  | CIP    | CEB |  |  |  |  |
| Ceramic membrane  | Ceramic membrane system |   |        |     |  |  |  |  |
| Coagulant (PACL   | Dosing                  | 22  | N/A    | N/A |  |  |  |  |
| solution, 23%     | prior to membrane       |   |        |     |  |  |  |  |
| concentration)    | module at 3 mg(as       |   |        |     |  |  |  |  |
|                   | $Al^{3+})/L$            |   |        |     |  |  |  |  |
| Acid (HCl, 33%    | Membrane cleaning       | N/A   | 0.0093 | 0.6 |  |  |  |  |
| concentration)    |                         |   |        |     |  |  |  |  |
| Hypochlorite (13% | Membrane cleaning       | N/A   | 0.29   | 8   |  |  |  |  |
| concentration)    |                         |   |        |     |  |  |  |  |
| Polymer membrane  | system                  |   |        |     |  |  |  |  |
| Coagulant (PACL   | Dosing                  | 22  | N/A    | N/A |  |  |  |  |
| solution, 23%     | prior to membrane       |   |        |     |  |  |  |  |
| concentration)    | module at 3 mg(as       |   |        |     |  |  |  |  |
|                   | $Al^{3+})/L$            |   |        |     |  |  |  |  |
| Acid (HCl, 33%    | Membrane cleaning       | N/A   | 0.057  | N/A |  |  |  |  |
| concentration)    |                         |   |        |     |  |  |  |  |
| Hypochlorite (13% | Membrane cleaning       | N/A   | 3.2    | 6   |  |  |  |  |
| concentration)    |                         |   |        |     |  |  |  |  |
| Citric Acid (50%  | Membrane cleaning       | N/A   | 0.58   | N/A |  |  |  |  |
| concentration)    |                         |   |        |     |  |  |  |  |

**Table 1**: Chemical use for ceramic and polymeric membrane plants used in costing.Consumption in L of as delivered chemical for every ML water treated

# **Common equipment assumptions**

 Coagulant dosing system CAPEX and consumption OPEX are same for both trains. Coagulant storage is a 10m<sup>3</sup> GRP tank for 15 days storage (7260L of 23% PACL solution). Coagulant (aluminium chlorohydrate - PACL) dosing system and storage tank cost provided from vendor quotes;

- 2. Ozone system CAPEX and consumption OPEX for the polymer system train are the same as ceramic membrane system. Ozone system costs were taken from vendor quotes current at the time of costing;
- 3. Ozone contactor is a concrete tank with size 10m x 1m x 2.8m (Length x Width x Depth) to allow for 2.5m water depth and 300mm freeboard. Assumed 500mm wall and slab thickness, 200mm roof thickness. Concrete rates are \$1,000/m<sup>3</sup> for slab, \$1,800/m<sup>3</sup> for wall and \$2,700/m<sup>3</sup> for roof based on previous project estimates;
- 4. Ceramic Membrane Building Footprint is same as Polymeric Membrane Building; and
- 5. Building unit rate is  $1,500/m^2$  based on previous project estimates.

# **Ceramic membrane system assumptions**

- 1. The cost of the ceramic membrane process was provided by PWN Technologies. The scale of this plant was 22 MLD, with a water recovery prescribed at 97.5% and membranes operating at 200 L/m<sup>2</sup>/h. This correlates to 184 membrane elements based on the area of a single monolith of 25 m<sup>2</sup>;
- 2. While only 182  $L/m^2/h$  was reached on the ETP pilot trial due to the upper limit of the ozone supply system, the results indicated that achieving the 200  $L/m^2/h$  proposed in this costing is realistic. Fluxes higher than 200  $L/m^2/h$  may be achieved, but was not considered in this cost assessment;
- 3. The CIP chemicals and CEB chemicals will be from the same dosing system. PWN Technologies provided a quote that includes CIP/CEB equipment. No additional CAPEX for CIP/CEB equipment was determined;
- 4. Civil works unit rate for chemical area bund and car-port structure is \$750/m<sup>2</sup> (50% of building unit rate);
- 5. Ceramic membrane modules have 15 years design life and the module unit rate is 4 times of polymeric membrane module. Ceramic membrane warranties of 20 years are now available. Plants might also be offered a 25 warranty. Therefore these longer design life values will also be considered;
- 6. CIP and CEB consumption rates determined from project trial at Eastern Treatment Plant which was operated from September 2012 to July 2013; and
- Time between CIP was targeted at 90 days based on advice from PWN Technologies. ETP ceramic membrane trial confirmed chemical cleaning requirement to achieve 90 day CIP target.

# Polymer membrane train assumption

 Costs for polymer membrane plants were provided from two plants located in Australia, known as Polymer Membrane Plant A and Polymer Membrane Plant B. Both plants featured UF membranes. After scaling both plants to 22 MLD and indexing their costs to 2014, the average of the two prices was used for the cost model;

- 2. Polymeric membrane modules have 7 years design life and the module unit rate is estimated using cost data for Polymer Membrane Plant B;
- 3. CIP and CEB consumption rates determined from previous trial conducted by Melbourne Water at Eastern Treatment Plant (February 2008 to March 2009);
- 4. Polymer membrane fluxes at the Eastern Treatment Plant trials ranged from 25 to 65  $L/m^2/h$ . A nominal flux of 43  $L/m^2/h$  was chosen which is the same flux at Polymer Membrane Plant B. In this plant there is  $40m^2$  of membrane area in each element; and
- 5. Time between CIP targeted at 30 days based on ETP trial results.

# **Assessment limitations**

The costing assessment was carried out within limitations. These are as follows:

- The cost model is high level, generic and non-site specific. However membrane cleaning routines and sustainable fluxes are site specific so cleaning requirements and performance used in the costing have been based on clarified secondary treated effluent from Eastern Treatment Plant, Melbourne;
- The two treatment processes considered in the cost model are not a complete water recycling process train. Upstream storage, pre-treatment and downstream treatment, storage and pumping are not included. The intent of the cost model is to highlight the costs specific to the core technologies;
- Scope and total cost for a 22 MLD ceramic membrane system were provided by PWN Technologies but no breakdown of prices was included. Not enough CeraMac plants have been built to provide sufficient information to carry out any benchmarking of costs provided by PWNT. Further verification is required from CeraMac plants built in the future; and.
- Design life of ceramic membrane module beyond 16 years is yet to be proven. However warranties of 20 years are now available.

# Results

# **CAPEX** analysis

The capital cost of the ceramic and polymer membranes plants is shown in Figure 2. The total CAPEX of the ceramic plant was determined to be \$11,792,000 and the total CAPEX of the polymer membrane plant was \$10,668,000. The ceramic membrane plant CAPEX was therefore estimated to be 10.5% more than the equivalent polymer membrane plant. The largest contributor to this cost difference was the ceramic membrane plant itself, followed by the indirect costs (which are calculated at 60% of the direct costs which includes the membrane plant). In terms of the overall cost, the ceramic membrane system contributed to 35% of the CAPEX while the polymer membrane system contributed to 30% of the CAPEX.

The coagulation system was included into both membrane systems in Figure 2 due to its minor contribution to CAPEX, i.e. in both cases the coagulation system cost was \$70,157.



*Figure 2: CAPEX assessment results for ceramic membrane and polymer membrane process trains proposed in Figure 1.* 

### **OPEX analysis**

The annual OPEX for the ceramic and polymer membrane plants is shown in Figure 3. The ceramic membrane plant annual OPEX was \$1,316,900 while for the polymer membrane plant, it was \$1,381,800. Therefore, in terms of OPEX, the ceramic membrane plant was cheaper than the polymer membrane plant. This was principally due to the lower cost of replacement due to their 15 year lifetime compared to the 7 year lifetime of polymer membranes. The annualised membrane replacement was \$231,900 and \$318,800 for the ceramic membrane and polymeric membrane plants respectively. This also comes about from the relative number of membrane elements, where 184 ceramic monoliths are replaced vs 528 polymer membrane elements. Further, in the 25 year life of the plant, the ceramic membranes will be replaced once, while the polymer membrane three times. If the ceramic membranes lasted for the entire plant life, then this cost in the OPEX would be removed completely, bringing the OPEX to \$1,085,000 per annum. Ceramic membrane warranties are now available for 20 years, so the lower OPEX due to reduced replacement costs can be realised in such cases.



*Figure 3: Annual OPEX assessment results for ceramic membrane and polymer membrane process trains proposed in Figure 1.* 

### **NPV** analysis

With the finding that the ceramic membrane plant is more expensive in CAPEX but cheaper in OPEX, it is important to assess the lifetime cost of the two plant options to measure the overall cost differences. Table 2 shows the NPV assessment results for three options, one being for the polymer membrane plant and the remaining two for the ceramic membrane plant (with and without replacement). The NPV for the standard 25 year plant life is presented, together with the shorter 20 year plant life for comparison.

Table 2: NPV analysis results for 25 year plant life including water cost per  $m^3$ . NPV for 20 year plant life also calculated for comparison.

| Option   | NPV (25 years)                                | NPV (20 years)                                |
|--|---|---|
| Option 1 – Ceramic membrane system with module design life 15 years  | \$32,514,000<br>(\$0.179 per m <sup>3</sup> ) | \$29,763,000<br>(\$0.205 per m <sup>3</sup> ) |
| Option 2 – Ceramic membrane system<br>without module replacement     | \$30,684,000<br>(\$0.169 per m <sup>3</sup> ) | \$27,933,000<br>(\$0.192 per m <sup>3</sup> ) |
| Option 3 – Polymeric membrane system with module design life 7 years | \$33,292,000<br>(\$0.183 per m <sup>3</sup> ) | \$29,498,000<br>(\$0.203 per m <sup>3</sup> ) |

Based on the NPV analysis for the 25 year plant life, we see that the lower OPEX of ceramic membranes, both with and without membrane replacement, is cheaper over the life of the plant. The difference between the two key Options (1 and 3) is however, very minor, where the ceramic membrane plant was 2.3% cheaper (Option 1) compared to the polymer membrane plant (Option 3). However, the difference is much stronger (7.8% cheaper) if the ceramic membranes are found to last for the life of the plant (Option 2).

If the NPV is calculated for 20 years instead of 25 years, we see that the polymer membrane Option 3 becomes slightly favourable, but these differences are negligible. In this analysis, CAPEX was considered the same as for 25 years. In 20 years, the polymer membranes will be replaced twice, while the ceramic membranes would still be replaced once (Option 1).

Replacing them when the plant only has five years of operation remaining works against the economics of long life ceramic membranes. Instead, running them for the life of the plant (Option 2) is a more realistic in this case. The lifetimes are based around three drinking water plants in Japan using Metawater membranes have been operating since no later than 1998, and still in operation today using the same membranes. Therefore it seems likely that ceramic membranes will last 20 to 25 years. Recently, ceramic membrane warranties have been extended to 20 years. Therefore the lower costs of ceramic membrane plants that don't have membrane replacement costs are a realistic option.

# **Discussion**

This cost assessment aimed to resolve the uncertainty of the true cost differences between ceramic membrane and polymer membrane systems. In order to do this, recent plant cost data was needed and a comparable process needed to be proposed. In achieving this, it was found that the higher fluxes and longer lifetime of ceramic membranes do indeed substantially impact both their CAPEX and OPEX to yield comparable prices. For this to be achieved, ceramic membrane fluxes must be high enough such that their higher price on a per m<sup>2</sup> basis is offset by using less monolith units. The ETP trials showed that coagulant and ozone must be used together to achieve these high fluxes. Therefore, treatment systems that would benefit from the application of ozone to the water as well as a membrane stage would be well placed for economical ceramic membrane use. The other benefits of ceramic membranes which include low failure risk (high integrity) and chemical robustness that are important in water recycling schemes would therefore be realised at very similar economics to polymer membranes. Recent research in other projects is showing that upstream oxidation, including ozone, reduces organic fouling on downstream reverse osmosis membranes if they are to be included in the water recycling scheme.

### Ceramic membrane plant cost without ozone

The assessment included the use of ozone due to its effect of enhancing flux and reducing the cost of the ceramic membrane plant. However ozone may not be required in the water treatment scheme being considered, so the effect to cost of ceramic membranes in these situations would be of interest. The effect can be observed simply by the removal of the ozone stages shown in Figure 1. Ozone is added to the end of the process for the polymer membrane option and would not alter membrane performance (and costing) when removed. As ozone is added prior to the ceramic membrane, removing ozone from the ceramic membrane train will lead to a decrease in the flux as demonstrated in the trial. However chemical use remains the same as the plant operated on the same cleaning routine in both cases. In both polymer and ceramic membrane cases, removing ozone would lower CAPEX and OPEX due to the removal of the ozone equipment and energy costs, which were considered to be similar for both trains. Plant footprint however will increase for the ceramic membrane system due to the decreased flux, and would require altering the costing slightly to accommodate this change. However this is not expected to impact cost as significantly as the reduction in flux performance.

A flux of 130  $L/m^2/h$  can be reasonably considered as a sustainable operation flux for a coagulant only plant based on the results from the trial. Therefore, since CAPEX was higher for ceramic membranes than polymer membranes when operating at 200  $L/m^2/h$  with ozone

(and coagulant), operating at a lower flux will increase the CAPEX in proportion to polymer membranes.

### **Polymer UF vs ceramic MF**

The polymer membranes considered in this assessment were UF types, while the ceramic membrane is regarded as MF. The average pore size of the ceramic membrane is 0.1µm, which is approximately double that of polymer UF membranes (average pore size 0.05µm). The pore size of the ceramic membrane is, however, at the lower boundary of the MF pore range, where polymer MF membrane pore sizes are normally around 0.5µm. Regardless ceramic and polymer membrane pore sizes must also be compared in terms of structure. Ceramic membrane pores have a narrower size distribution in comparison to polymer membrane pores, which implies there may actually be less large pores in a ceramic membrane than a polymer membrane. Whether this can be realised in water recycling schemes (i.e. pathogen barrier) is outside the scope of this project. Challenge testing of *E.coli* and coliphage virus surrogate (MS2) during the trial in this project showed that the ceramic membrane is an effective barrier against protozoa, bacteria and viruses, behaving as expected for a small pore MF membrane, able to achieve 4 LRV for each pathogen class. MS2 removal is known to be higher than 4 LRV for polymer UF membranes as their average pore size is smaller. However regulators may not give credit greater than 4 LRV for any single membrane stage, even if the virus removal integrity of the membrane can be monitored.

### Differences between ceramic and polymer membrane trains

The two process trains in Figure 1 were selected to propose equivalent trains for the purposes of comparable costing, and based on real test data that could be obtained (i.e. trials on ozone prior to polymer UF normally include a biological filter which is not comparable to ozone feeding directly to ceramic membranes). However one difference that may negatively alter the performance of the polymer membrane train is the use of coagulant before ozone. Ozone is known to assist coagulation and improves organic carbon removal. This benefit can be realised in the ceramic membrane train, but not by the polymer membrane train due to the arrangement. On the other hand, applying ozone after membrane filtration may improve efficiency of the ozone process. How much the effect will influence performance is uncertain because the MF/UF membranes typically do not reduce organic carbon. However, they do greatly reduce turbidity.

# Timing of polymer trials and ceramic trials

It should also be acknowledged that the testing of the ceramic membrane trial unit was carried out approximately three years after the polymer membrane trials. Upgrades to the ETP's biological treatment occurred between the polymer and ceramic trials which could alter the feed water characteristics Ideally, polymer membrane testing should have been carried out in parallel, but the project did not have the resources to undertake this. The impact of changes in the water on membrane plant performance could affect the cleaning regimes used. Despite this uncertainty, the water was regarded as similar for the purposes of this cost process.

Flux could have also changed between the two periods, but the fluxes for polymer membranes found from the ETP trials and two other plants in Australia found fluxes to be around the 43-44  $L/m^2/h$  value and has been considered as typical in UF plants fed with secondary treated effluent.

# Cost reductions of membrane technology

As competitively built ceramic membrane installations are still emerging, the cost of ceramic membrane systems are decreasing. Polymer membrane systems may also experience cost reductions or improved pricing efficiencies over time. Therefore an updated cost model will be required in future due to the cost saving developments in the technologies.

# Conclusion

The cost comparison between ceramic and polymeric membrane plants has been completed in the context of water recycling in Australia. It showed that high flux and long life of ceramic membranes are the key features that reduce their CAPEX and OPEX amounts. When the flux of  $200 \text{ L/m}^2$ /h is reached on ceramic membranes with the use of coagulant and ozone, the economics of the equivalent process using polymer membranes become comparable. There are slight benefits to CAPEX for polymer membranes (10.5% cost favourable to polymers), but OPEX showed lower annual operating costs for ceramic membranes because they are replaced less often. Therefore, the other benefits of ceramic membrane such as high integrity, long life and chemical robustness can be realised in water recycling schemes as being economically comparable to polymer membranes.

