Chemically Enhanced Backwash as the Only Ultrafiltration Fouling Control Approach in Seawater Applications

by:

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Abstract

The use of ultrafiltration (UF) membranes as a pre-treatment technology for seawater applications, such as desalination and water production for deep sea oil extraction, has expanded in recent years. Controlling fouling during filtration remains a crucial operational challenge, particularly in applications with equipment footprint constraints. The present study sought to investigate a simplified fouling control approach where chemically enhanced backwash (CEB) was the only technique utilized.

Short-term benchmarking trials were performed to evaluate a wide range of operating conditions. These included: CEB duration, CEB frequency, CEB make up solution, and sodium hypochlorite (NaClO) concentration. Long-term trials were then completed to determine the viability of this approach, and provide operational insight for future applications.

NaClO concentrations as low as 8 ppm were effective in achieving sustainable fouling rates in low temperature UF operation with CEB as the only fouling control approach. Extended CEBs using 150 ppm NaClO solutions were effective at restoring lost permeability, though may not be required for periods of 6 months or more. Both outside-in, and inside-out membrane configurations were evaluated, with outside-in observed to have a lower fouling rate. No difference was observed when comparing UF permeate and nanofiltration concentrate as CEB make up solutions.

A new measure, termed ‘cleaning effort’ (i.e. the product of CEB duration and NaClO concentration divided by CEB frequency), was proposed to compare fouling control efficiency for different CEB operating conditions. Fouling rate followed an exponential decay relationship with respect to cleaning effort. Accumulation of active biomass on membrane fibers was not observed after long-term trials. Residual chlorine in the CEB reject stream was observed to be above regulatory limits, and decayed slowly.
Lay Summary

Membranes are increasingly being used for the filtration of seawater to address water shortages. Membranes which are designed to remove dissolved materials, such as nanofiltration and reverse osmosis, are capable of producing ultra-high quality water. These membranes are most efficiently operated when feedwater is pre-treated using ultrafiltration membranes to remove suspended materials, but accumulation of foulants on the UF membrane surface remains an operational challenge. The present study evaluated a simplified approach to remove foulants from ultrafiltration membranes using chemically enhanced backwash only. Operating conditions capable of maintaining stable operation of the membranes were identified, as well as practical insights for future applications of the approach.
Preface

This statement confirms that the author is the primary person responsible for the research contained in this thesis. All experimental design and procedures were conceived of by the author with input from the supervisory committee, namely Professor Pierre Bérubé, and our industry partners at GE Water. The specific names of those who assisted in conducting experiments are recognized in the acknowledgements section of this thesis.
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<th>Description</th>
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<tr>
<td>ATP</td>
<td>Adenosine triphosphate</td>
</tr>
<tr>
<td>BW</td>
<td>Backwash</td>
</tr>
<tr>
<td>CEB</td>
<td>Chemically enhanced backwash</td>
</tr>
<tr>
<td>CIP</td>
<td>Clean-in-place</td>
</tr>
<tr>
<td>Cleaning effort</td>
<td>CEB effort quantified as: CEB duration* NaClO conc. / CEB frequency (min–ppm/hr)</td>
</tr>
<tr>
<td>DI</td>
<td>Deionized water</td>
</tr>
<tr>
<td>ECEB</td>
<td>Extended chemically enhanced backwash</td>
</tr>
<tr>
<td>Fouling rate</td>
<td>Long-term (i.e. multiple filtration and cleaning cycles) change in TMP over time (PSI/hr)</td>
</tr>
<tr>
<td>MF</td>
<td>Microfiltration</td>
</tr>
<tr>
<td>NaClO</td>
<td>Sodium hypochlorite</td>
</tr>
<tr>
<td>NF</td>
<td>Nanofiltration</td>
</tr>
<tr>
<td>NF Conc</td>
<td>Nanofiltration concentrate</td>
</tr>
<tr>
<td>TMP</td>
<td>Transmembrane pressure (PSI)</td>
</tr>
<tr>
<td>TOC</td>
<td>Total organic carbon (mg/L)</td>
</tr>
<tr>
<td>TSS</td>
<td>Total suspended solids (mg/L)</td>
</tr>
<tr>
<td>UF</td>
<td>Ultrafiltration</td>
</tr>
<tr>
<td>UF Perm</td>
<td>Ultrafiltration permeate</td>
</tr>
<tr>
<td>UV254</td>
<td>Ultraviolet absorption at 254 nm</td>
</tr>
<tr>
<td>ZW1500</td>
<td>GE ZeeWeed® 1500 membrane (outside-in filtration)</td>
</tr>
<tr>
<td>ZW700B</td>
<td>GE ZeeWeed® 700B membrane (inside-out filtration)</td>
</tr>
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</table>
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1 Introduction

Ultrafiltration (UF) membranes are widely used in seawater filtration applications as a preferred pre-treatment step before nanofiltration (NF) or reverse osmosis (RO) processes (Ghaffour et al., 2013). UF pre-treatment systems have demonstrated their ability to consistently provide high quality effluent and accommodate variable source water conditions. However, a remaining central operational challenge is controlling the fouling that accumulates during seawater filtration (Suárez et al., 2015).

Present practice for fouling control typically includes hydraulic cleaning techniques, such as backwashing (BW) and air-sparging, as well as chemical cleaning techniques, such as chemically enhanced backwash (CEB) and chemical soaks (Shi et al., 2014).

The efficacy of these techniques to control UF fouling has been demonstrated at many full scale UF-RO desalination plants over the years, where UF modules have maintained stable trans membrane pressures (TMP) over long operating periods (Kamp et al., 2000). However, though the viability of UF-RO seawater filtration plants has been established, there are emerging seawater applications (i.e. deep sea oil extraction) where conventional fouling control approaches may not be technically feasible, or are prohibitively expensive (Boczkowski et al., 2014).

1.1 Aims and Objectives

The present study evaluated a simplified fouling control approach that could be used to control UF fouling during seawater filtration, without the use of air scouring.

The key objective was to establish operating conditions capable of sustaining a stable permeate flux during simulated long-term (i.e. greater than 6 months of continuous operation) deep-sea filtration operations with frequent CEBs and ‘extended’ CEBs (ECEB), using sodium hypochlorite (NaClO), as the only fouling control technique. The impact of CEB duration, CEB frequency, CEB make up solution characteristics, and NaClO concentrations on the permeate flux that could be sustained, for outside-in and inside-out hollow fiber membranes, was assessed. A new approach, defined as ‘cleaning effort’, was developed to identify optimal operating conditions. The composition and nature of the fouling layer were also of interest, with specific
attention given to evaluate the extent of biological fouling that occurred during long-term operation.

2 Literature Review and Background

2.1 Membrane Filtration of Seawater

The demand for potable water in coastal communities continues to rise around the world, leaving utilities with desalination of seawater as one of the only options to address shortages. Currently, over 300 million people rely on the desalinated water produced at over 18,000 plants operated worldwide (International Desalination Association, 2015). High pressure seawater reverse-osmosis (SWRO) membranes currently dominate the market in areas outside the Middle East (where thermal evaporation is widely used) due to a rapid increase in cost competitiveness over the past decades (Voutchkov, 2016).

To ensure effective operation of the SWRO process, significant pre-treatment of source waters is typically necessary to remove suspended material and colloids. Historically, this was first accomplished using traditional flocculation/sedimentation techniques and dual media filters (Alawadhi, 1997). Operational experience at full scale has since revealed shortcomings to this treatment approach: most notably, variable feedwater quality leading to high turbidity effluent with high fouling potential for vulnerable SWRO membranes (Ebrahim, et al., 2001). Head to head trials confirmed that microfiltration membranes outperformed conventional approaches, and offered lower costs for large plants.

UF pilot studies were first completed in the Netherlands, prompted by a conventional pre-treatment system failing to provide effluent of sufficient quality to maintain sustainable SWRO operation. Positive pilot results lead to a full scale demonstration project, which operated reliably for its first 200 days (Kamp et al., 2000).

Hollow fiber UF membranes operated in inside-out, dead-end configurations performed well during pilot studies at a Trinidad SWRO plant (van Hoof, et al., 2001). UF pre-treatment provided high quality filtrate for SWRO with a low Silt Density Index despite highly variable feedwater quality.
A review by Knops et al. (2007) of various operating plants (pilot and full-scale) confirmed that UF as a pre-treatment to SWRO processes was cost competitive compared to conventional treatment options, largely due to savings on chemical costs.

Continued research and development has resulted in substantial decreases in the cost of UF, as well as dramatically reduced system footprints (Suarez et al., 2015). The sum of these developments has led to UF becoming a preferred pre-treatment solution for SWRO applications.

Another emerging application of membranes in the seawater filtration space has been in the production of sulfate-free seawater for injection into deep-sea oil wells. As these wells descend deeper into the ocean the business case to limit risk associated with barium and strontium scale on injection lines, and to prevent reservoir souring, has substantially improved (Boczkowski et al., 2014). NF membranes have been proven to effectively remove sulfates at lower operating pressures than SWRO membranes (Eriksson et al., 2005), and have therefore gained considerable interest for this application. A recent pilot study established the suitability of NF for improving process water quality, as well as confirmed UF pre-treatment as an effective operating strategy (Alam et al., 2017).
2.2 Membrane Fouling and Transmembrane Pressure

The primary challenge of all water filters is the accumulation of particles on the filtration surface. As these particles accumulate they increase resistance to fluid flow, requiring additional energy for filtration. This phenomenon has been termed ‘fouling’ (Choo & Lee, 1996). A simplified illustration of the concept has been presented below as Figure 1.

![Figure 1 – Illustration of Fouling During Membrane Permeation](image)

As fouling layers accumulate, and the resistance increases, the TMP required to draw the same volume of water through the filter increases (under constant flux conditions). This relationship is presented in Equation 1. Alternatively, for constant pressure systems, the TMP increases as fouling progresses (Equation 2) (Field et al., 1995).

\[
\text{Equation 1}
\]

\[
\text{Equation 2}
\]

where, \(\text{TMP} = \text{transmembrane pressure (Pa)}, \ J = \text{permeate flux (m}^3/\text{m}^2 \cdot \text{s}^{-1}), \ \mu = \text{dynamic viscosity (Pa} \cdot \text{s}), \ R_m = \text{clean membrane hydraulic resistance (1/m)}, \ R_f = \text{fouling resistance (1/m)}.\]
The acute impact of biological fouling (biofouling) on membrane performance has been identified since the early days of membrane water filtration research. It was quickly discovered that biological cells preferentially grow on filtration surfaces due to permeation flow forces bringing suspended micro-organisms and their energy sources towards the membrane surface (Choo & Lee, 1996). Subsequent reviews have confirmed the risk biofouling can present in operating membrane plants, with severe cases of unmitigated biofouling resulting in the necessary replacement of membrane modules (Baker & Dudley, 1998). Biological fouling in SWRO membranes has been highlighted as a major challenge during desalination operations, particularly in warmer climates (Matin et al., 2011).
2.3 Sustainable Flux and a Sustainable Fouling Rate

During filtration, convective flow transports foulants towards the membrane, while fouling control measures transport foulants away from the membrane. When these two transport operations are in balance there is theoretically no accumulation of foulants on the membrane. The permeate flux that generates these conditions is termed the “critical flux” (Field et al., 1995). When membranes operate above the critical flux fouling is expected, while below it theoretically no fouling occurs.

However, experience has demonstrated that fouling still occurs when operating at sub critical flux, and therefore a concept of a “sustainable flux” has been introduced. This definition is more practical in nature and describes an operating flux that minimizes the rate of fouling to the extent that long-term membrane operation is possible (Bachin et al., 2006 and Field & Pearce, 2011).

Previous trials have demonstrated that operating flux and backwash frequency have a significant effect on UF fouling rates (Kim & DiGiano, 2006). Further studies have confirmed this, with the additional insight that failure to remove reversible fouling during backwash cycles (i.e. operation at high fouling rates) promotes the transformation of reversible fouling to irreversible (Raffin et al., 2012). These findings suggest that fouling control approaches that do not adequately remove foulants between filtration cycles fail to produce sustainable fouling rates. This key concept was essential when designing a fouling control approach that is sustainable in the long term in the present study.
2.4 Popular Approaches to Membrane Fouling Control

As membrane science has evolved, high definition direct observation studies have provided insight into the development of fouling layers (Ye et al., 2011). Specifically, the formation of an expandable “cake layer” via the accumulation of particles on the membrane. This is presented in Figure 2, which depicts the evolution of the cake layer over the course of successive filtration and backwash cycles.

![Figure 2 – Fouling cake at the end of filtration cycles, and after 30s backwash without air scouring. Filtration flux 50 l/m²-h, filtration duration 3570s, backwash flux 50 l/m²-h, backwash duration 30s. Feed solution: 50 mg/L bentonite and 50 mg/L alginate. (Ye et al., 2011)](image)

Many innovative approaches have been developed to remove the fouling layer from UF membranes. These can be broadly categorized as either scouring methods utilizing shear stress at the membrane surface to transport particles (i.e. hydraulic backwash, air scouring), or chemical cleaning methods (i.e. clean in place or CEB) (Gao et al., 2011). The specific fouling control techniques relevant to this study are further discussed in the following sections.
2.4.1 Hydraulic Backwash

Backwashing is achieved by reversing the flow direction of water through a membrane. In doing so a shear force is created, transporting foulants away from the membrane. This concept was previously depicted in Figure 2, but has been further illustrated in Figure 3.

Hydraulic BW releases and transport portions of the fouling layer away from the UF membrane surfaces and into the bulk fluid. However, a portion of the fouling layer (termed ‘hydraulically irreversible fouling’) remains and typically requires chemical cleaning to remove (Katsoufidou et al., 2005 and Katsoufidou et al., 2008).

Backwashing UF membranes with SWRO permeate has been used as a fouling control approach in seawater applications (Li, et al., 2012). The trials conducted by Li et al. (2012) produced relatively high fouling rates (0.28 PSI/hr) that required frequent clean-in-place (CIP) operations (approximately daily).
2.4.2 Air Scouring

Air scouring is a fouling control technique that uses rising air bubbles to induce shear forces at a membrane surface. These forces transport particles away from the membrane surface and into the bulk fluid. This concept is illustrated in Figure 4.

![Diagram of Air Scouring a Fouled Membrane](image)

Figure 4 – Air Scouring a Fouled Membrane

Air scouring is a proven fouling control technique used extensively in the UF water treatment space, and is commonly used in full-scale installations, particularly in the membrane bioreactor (MBR) industry (Gao et al., 2011).

Air scouring has been employed in previous SWRO UF fouling control studies with positive effect (Zeng et al., 2009 and Profio et al., 2011).
2.4.3 Chemical Cleaning

Chemical cleaning of UF membranes is used to remove foulants adsorbed to the membrane that cannot be removed by scouring alone (hydraulically irreversible fouling). The mechanism of action has been described as a six step process (Shorrock & Bird, 1998) whereby:

1) bulk reaction of cleaning reagents occurs,
2) cleaning agent is transported to the membrane surface,
3) cleaning agent transmits through the fouling layer to the membrane surface,
4) chemical reactions solubilize and detach foulants,
5) waste cleaning agent is transported to the membrane interface, and
6) transport of waste matter away from the membrane surface and into the bulk solution.

This concept is illustrated in Figure 5.
Chemical cleaning methods are widely used in membranes operations, with the most common cleaning reagents being:

1) oxidizers such as NaClO and hydrogen peroxide (H₂O₂),
2) caustics such as sodium hydroxide (NaOH), and
3) acids such as hydrochloric (HCl) and sulphuric (H₂SO₄).

NaClO is by far the most commonly used cleaning agent of the above (Porcelli & Judd, 2010).

Chemical cleaning is typically performed as a CIP operation, or as a chemically enhanced backwash (CEB). CIP operations are generally infrequent (median frequency of 4 per year) and use high chemical concentrations, long soak times, and possibly even elevated temperatures. CEBs generally happen more frequently (median frequency of 32 per day), but use lower concentrations of cleaning agents than CIP. They also use shorter (or no) soak times, and no temperature adjustment is applied (Porcelli & Judd, 2010). CEB and its application in seawater UF is discussed in greater detail in the following section.

One important consideration regarding chemical cleaning is the tendency for membranes to age as they are exposed to cleaning agents, resulting in a decrease in performance factors such as fouling rate and breach frequency (Robinson et al., 2016). This makes the minimizing of chemical dose an operational goal beyond the obvious economic considerations of reducing chemical use.
2.4.4 Chemically Enhanced Backwash

CEB, also commonly called a “maintenance clean”, combines the hydraulic scouring action of a BW with the benefit of cleaning agents capable of detaching and solubilizing foulants.

The CEB solution contains chemical cleaning agents that are sent in reverse of the membrane permeation direction and in the process liberate and transport adsorbed foulants away from the membrane and into the bulk fluid. This is illustrated in Figure 6.

![Figure 6 – Illustration of a Chemically Enhanced Backwash](image)

Wei et al. (2011) completed filtration trials comparing NaClO, NaOH, and HCl under a variety of operating fluxes in a pilot scale MBR. They demonstrated that NaClO (500 – 3000 ppm) was the most effective cleaning reagent for use during CEB. The pilot MBR operated under sub-critical flux operation for 750 days and was able to effectively control fouling. Scanning electron microscope analysis of the membranes concluded CEB detached the biofouling layer. Zsiriai et al. (2012) operated a pilot scale MBR to further investigate the role of flux and BW duration on fouling rate when using 500 ppm NaClO CEB as a fouling control strategy. They observed that an increased BW flux was of greater benefit than increased BW duration. The pilot successfully operated without requiring CIP for 48 days using this strategy. Irreversible fouling rates were still sufficiently high to require a manual cleaning procedure involving mechanical agitation and water spray to remove accumulated foulants. Wang et al. (2014) evaluated low concentration
(0.05 – 1.5 ppm) NaClO CEB in MBR applications utilizing a lab-scale set-up. They observed that concentrations as low as 0.2 ppm could achieve effective fouling control in membrane bioreactor applications. Biopolymer detachment was quantified using polysaccharide and protein analysis confirming enhanced detachment compared to pure water (particularly above 0.2 ppm). Note that air scouring was used during CEB in the studies by Wei et al., Zsiriai et al., and Wang et al.

Limited work has been completed to assess CEB in seawater filtration applications. Schurer et al. (2012) completed a demonstration project at a Netherlands SWRO drinking water plant, which assessed a routine CEB (every 12 to 168 hours, depending on feedwater quality) utilizing 100 ppm NaClO, 125 ppm NaOH, and 225 ppm HCl. The pilot operated successfully for 14 months with no requirement for additional CIP. No air scouring was used during the CEBs. Li et al. (2012) conducted lab-scale seawater UF trials assessing 1000 ppm NaClO CEBs to remove hydraulically irreversible fouling. The technique was evaluated three times during a 55 hour lab-scale trial and achieved near complete removal of foulants in each case.
3 Knowledge Gap

Previous seawater UF studies have evaluated CEB in the context of a wide range NaClO concentrations (0.05 to 1000 ppm), and though they have proven the efficacy of the technique, these studies are not comprehensive and provide limited insight for operational facilities. This study sought to close these knowledge gaps by addressing the following objectives:

1. Determine if NaClO CEB alone could effectively control fouling in seawater applications, enabling sustainable, long term operation for operating periods longer than 6 months, without requiring more aggressive chemical cleans (i.e. ECEB).

2. Complete a comparison of outside-in vs. inside-out hollow fiber UF configurations.

3. Evaluate the performance differences between UF permeate and NF concentrate as CEB solutions.

4. Investigate the relationship between fouling rate and a newly devised approach to quantify CEB ‘cleaning effort’ to identify optimal operating conditions.

5. Assess the extent of biofouling following long-term operation of the simplified fouling control approach.

6. Quantify the concentration and decay rate of chlorine in CEB reject wastewater.
4 Materials and Methods

4.1 Experimental Program

A series of short-term benchmarking trials were performed to rapidly evaluate the performance of a wide range of possible operating conditions using the CEB technique. These operating conditions included: CEB duration, CEB frequency, CEB make up solution, and NaClO concentration.

The results of these short-term trials formed the basis for the operating conditions evaluated throughout three subsequent longer term trials. During these longer term trials the ability of the ECEB technique to remove foulants and restore lost membrane permeability was also evaluated.

All trials were completed under constant flux conditions using membrane modules with a total surface area of 0.010 m². Feedwater temperature was maintained at 4 °C, and all CEBs and ECEBs were completed at a flux of 34 l/m²-h. A permeation flux of 17 l/m²-h was used for all trials, excepting a portion of Trial 5, which operated at 10 l/m²-h. Following recommendations from GE, maximum TMP for ZW1500 and ZW700B modules was set at 8 PSI (nominal operating range of 6 PSI).

The operating conditions used in each of the trials are summarized in Table 1. Each trial had 1 to 4 operating trains (denoted as Trains A1/A2/B1/B2), with the specific operating conditions for each train noted in the respective row. Note that Trains B1 and B2 were initially used for ZW700B trials, but were later adapted for additional ZW1500 trials following the completion of the comparison trials.

Further details of the trials and the different operating conditions evaluated are presented in Tables 12 and 13.
Table 1 – Experimental Trials Operating Conditions Summary Table

<table>
<thead>
<tr>
<th>Trial</th>
<th>Trains</th>
<th>CEB Duration (min)</th>
<th>CEB Freq (hr)</th>
<th>CEB Solution</th>
<th>Flux (l/m²-h)</th>
<th>NaClO Conc (ppm)</th>
<th>ECEB</th>
<th>Fiber</th>
<th>Duration (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Short term trials</strong></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>A1</td>
<td>2</td>
<td>2</td>
<td>UF Perm</td>
<td>17</td>
<td>10</td>
<td>No</td>
<td>ZW1500</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>A1/A2</td>
<td>2</td>
<td>12</td>
<td>UF Perm</td>
<td>17</td>
<td>10</td>
<td>No</td>
<td>ZW1500</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>A1</td>
<td>5</td>
<td>12</td>
<td>UF Perm</td>
<td>17</td>
<td>10</td>
<td>No</td>
<td>ZW1500</td>
<td>9</td>
</tr>
<tr>
<td><strong>Long term trials</strong></td>
<td></td>
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<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>A1/A2</td>
<td>5/10</td>
<td>6</td>
<td>UF Perm</td>
<td>17</td>
<td>10/50/150</td>
<td>No</td>
<td>ZW1500</td>
<td>33</td>
</tr>
<tr>
<td></td>
<td>B1/B2</td>
<td>5</td>
<td>3, 6</td>
<td>UF Perm</td>
<td>17</td>
<td>10/50/100/150</td>
<td>No</td>
<td>ZW700B</td>
<td>33</td>
</tr>
<tr>
<td>5</td>
<td>A1/A2</td>
<td>5</td>
<td>2</td>
<td>UF Perm</td>
<td>17/10</td>
<td>10/15/25/30</td>
<td>Yes</td>
<td>ZW1500</td>
<td>71</td>
</tr>
<tr>
<td></td>
<td>B1/B2</td>
<td>5</td>
<td>2</td>
<td>UF Perm</td>
<td>17</td>
<td>30/40/50/60</td>
<td>Yes</td>
<td>ZW700B</td>
<td>38</td>
</tr>
<tr>
<td>6</td>
<td>A1/A2</td>
<td>5</td>
<td>2</td>
<td>UF Perm/NF Conc</td>
<td>17</td>
<td>8</td>
<td>Yes</td>
<td>ZW1500</td>
<td>102</td>
</tr>
<tr>
<td></td>
<td>B1/B2</td>
<td>5</td>
<td>2/3/4</td>
<td>UF Perm/NF Conc</td>
<td>17</td>
<td>8</td>
<td>Yes</td>
<td>ZW1500</td>
<td>71</td>
</tr>
</tbody>
</table>
4.1.1 Quality Control

All UF membranes modules were constructed using virgin membrane fibers provided by GE Water (GE). A complete NF membrane module was also provided by GE. Prior to use, modules were conditioned in accordance with the protocols provided in Appendix A.

Before the start of each experiment a clean water flux test was completed to determine the steady state clean membrane resistance.

UF membrane modules were subjected to an integrity test prior to use. Fibers were pressurized to 8 PSI using compressed air and submerged under DI water. If air bubbles were observed to escape from the membrane fibers, the module was considered breached and discarded.

The system flux was regularly monitored (using graduated cylinder and stopwatch) and adjusted as needed to ensure that constant flux conditions were maintained.

TMP was monitored using Omega Engineering Inc. pressure transducers (Model PX243A-15BG5V) and recorded using a HOBOware U12 data logger at an frequency of 5 minutes. Pressure transducers were regularly calibrated using a digital pressure gauge, with calibration details available in Appendix B.

CEB solutions were routinely tested to ensure the prescribed NaClO concentration was maintained.
4.1.2 Fouling Rate Data Analysis

The performance of the fouling control approach was assessed using one primary metric: fouling rate. The fouling rate was quantified by fitting a linear regression to the TMP data over successive filtration and CEBs cycles. This approach is illustrated in Figure 7.

![Figure 7 – Depiction of Fouling Rate Quantification Methodology](image)

The fouling rate is the primary parameter of concern, since fouling rates with large positive values require more frequent ECEB operations. Therefore, it was a central goal of this study to achieve low fouling rates to minimize the number of required ECEBs.

In some instances, a negative fouling rate was observed. This behaviour has been termed ‘cleaning’ in the discussions that follow.
4.2 Experimental Setups

Two filtration systems were used to complete this study. UF filtration systems were used to generate the data presented in Section 5. An NF filtration system was used to generate NF concentrate used as CEB solution in some of the trials.

4.2.1 Ultrafiltration System

Two systems (identified as A and B), each with two parallel trains (i.e. A1/A2 and B1/B2) were used for all trials. The systems were composed of the following process elements:

- Custom potted membrane modules (fiber details provided in Table 2)
  - Casing: clear PVC, diameter = 21 mm, length = 300 mm
- Feedwater tank: 40 L square tank with stirrer
- Permeate and CEB pumps: Masterflex L/S drive with no. 16 heads
- Pressure transducers and gauges: Model PX243A-15BG5V transducer and Cole-Parmer RK digital gauge
- CEB solution tanks: 2 L glass amber bottles
- CEB and drain/fill control valves: Burkert 0124 solenoid valves for CEB flow control and tank drain/fill
- Programmable logic controller: custom 120V cyclic timer box with pump outputs
- Datalogger: HOBOware U12
Table 2 – UF Membrane Modules

<table>
<thead>
<tr>
<th>Fiber type</th>
<th>GE ZW1500</th>
<th>GE ZW700B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow path</td>
<td>Outside-in</td>
<td>Inside-out</td>
</tr>
<tr>
<td>Material</td>
<td>PVDF</td>
<td>PES</td>
</tr>
<tr>
<td>Fiber diameter</td>
<td>1.1 mm OD, 0.66 mm ID</td>
<td>Multi-channel - 3.6mm OD, 0.8 mm ID x 7</td>
</tr>
<tr>
<td>Fiber length</td>
<td>15 fibers x 20 cm length</td>
<td>2 multi-channel fibers x 26 cm length</td>
</tr>
<tr>
<td>Nominal pore size (mm)</td>
<td>0.02</td>
<td>0.04</td>
</tr>
</tbody>
</table>

Schematics of the ZW1500 and ZW700B systems are provided as Figures 8 and 9, respectively. Note that ‘NO’ and ‘NC’ refer to normally-open and normally-closed valves (i.e. unpowered state).

![Figure 8 – ZW1500 Bench Scale Process Diagram](image-url)
The operating sequence for the ZW1500 system is as follows:

1. Filtration cycle: pumps in forward direction, no valves powered
2. CEB cycle: pumps reverse direction (2x speed), valves 1, 2, 4, 5 powered
3. Drain cycle: pumps stop, valve 3 powered
4. Fill cycle: valve 4 powered
5. Repeat 1

Figure 9 – ZW700B Bench Scale Process Diagram
The operating sequence for the ZW700B system is as follows:

1. Filtration cycle: pumps in forward direction, no valves powered
2. CEB cycle: pumps in forward direction (2x speed), valves 1, 2, 3, 4, 5, 6 powered
3. Flush cycle: pumps in forward direction (2x speed), valves 1, 2, 3, 6 powered
4. Repeat 1

An annotated photograph of the ZW1500 and ZW700B systems following construction is presented in Figure 10.

Figure 10 – ZW1500 and ZW700B UF Filtration Systems
4.2.2 Nanofiltration System

The NF system was used to generate the NF concentrate used as one of the evaluated CEB solutions. The system was composed of the following process elements:

- Membrane module: GE DK1812 spiral bound (MWCO: 200 – 400 Daltons)
  - Casing: Synder 1812 (stainless steel)
- Feedwater tank: 20L square polyethylene carboy
- Pump: Masterflex L/S drive with no. 16 head
- Pressure gauge: analog gauge (stainless steel)

UF permeate was collected from the CEB system and concentrated using the NF system. The spiral bound module was pressurized (20 – 30 PSI) with the cross-flow line fed back to the feedwater tank. The system was operated until the 100% feedwater tank level was reduced to 65% (35% of the initial volume permeated and discarded). Concentrate was then sealed and stored at 4 °C until use.

A process diagram and photograph of the NF system are presented as Figures 11 and 12.
Figure 11 – NF System Process Diagram

Figure 12 – Photograph of NF System
4.3 Solutions Used During Trials

Two types of solutions were used for the trials, feedwater and CEB solution. Seawater was used as the feedwater for all trials completed within this study, while two different CEB solutions were used. Relevant information and analyses for these solutions are detailed in Sections 4.3.1 and 4.3.2, respectively.

4.3.1 Seawater (Feedwater)

Seawater used for the study was collected from the Burrard Inlet (Vancouver, BC). The Vancouver Convention Centre operations department provided access to their building heating and cooling system, where water was collected using the system feed pumps. The pumps’ intakes were located approximately 6 meters below the collection area. This location is illustrated in Figure 13.

Seawater for the trials was collected in 20 L plastic containers and transported to the laboratory at UBC where the trials were conducted. Seawater was collected every one to two weeks and stored at 4 °C in the dark. Before being used in the trials, seawater was filtered using a 75μm cartridge filter. Approximately 4 m³ of seawater was collected over a 14 month span for use in these trials (from March 2016 to May 2017).

Figure 13 – Feedwater Collection Location
Filtered (75 µm) seawater was characterized in terms of TOC, UVA, TSS, conductivity, and metals content. As presented in Tables 3 to 7, the characteristics of the raw seawater remained relatively stable during the monitoring period.

Table 3 – Feedwater TOC (n=3)

<table>
<thead>
<tr>
<th>Sample Date</th>
<th>TOC (mg/L)</th>
<th>Standard Error (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apr-11-2017</td>
<td>2.1</td>
<td>0.247</td>
</tr>
<tr>
<td>May-10-2017</td>
<td>1.5</td>
<td>0.013</td>
</tr>
</tbody>
</table>

Table 4 – Feedwater UVA254

<table>
<thead>
<tr>
<th>Sample Date</th>
<th>UVA254 (cm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jun-30-2016</td>
<td>0.056</td>
</tr>
<tr>
<td>Jul-07-2016</td>
<td>0.048</td>
</tr>
<tr>
<td>Jul-15-2016</td>
<td>0.054</td>
</tr>
<tr>
<td>Jul-27-2016</td>
<td>0.052</td>
</tr>
<tr>
<td>Aug-05-2016</td>
<td>0.054</td>
</tr>
<tr>
<td>Aug-05-2016</td>
<td>0.054</td>
</tr>
<tr>
<td>Aug-05-2016</td>
<td>0.053</td>
</tr>
<tr>
<td>Sept-07-2016</td>
<td>0.048</td>
</tr>
<tr>
<td>Sept-07-2016</td>
<td>0.044</td>
</tr>
</tbody>
</table>

Table 5 – Feedwater Conductivity (corrected to 20 ºC)

<table>
<thead>
<tr>
<th>Sample Date</th>
<th>Conductivity (Microseimens/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apr-12-2017</td>
<td>32500</td>
</tr>
<tr>
<td>May-10-2017</td>
<td>32500</td>
</tr>
</tbody>
</table>

Table 6 – Feedwater TSS

<table>
<thead>
<tr>
<th>Sample Date</th>
<th>TSS (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jun-30-2016</td>
<td>100</td>
</tr>
<tr>
<td>Jul-07-2016</td>
<td>90</td>
</tr>
<tr>
<td>Jul-15-2016</td>
<td>92</td>
</tr>
<tr>
<td>Jul-27-2016</td>
<td>95</td>
</tr>
<tr>
<td>Aug-05-2016</td>
<td>105</td>
</tr>
<tr>
<td>Aug-05-2016</td>
<td>102</td>
</tr>
<tr>
<td>Aug-05-2016</td>
<td>98</td>
</tr>
<tr>
<td>May-10-2017</td>
<td>72</td>
</tr>
<tr>
<td>May-10-2017</td>
<td>82</td>
</tr>
<tr>
<td>May-10-2017</td>
<td>110</td>
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</table>
Table 7 – Feedwater Metals Analyses (n = 3)

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average Conc. (mg/L)</td>
<td>Standard Error (mg/L)</td>
<td>Average Conc. (mg/L)</td>
<td>Standard Error (mg/L)</td>
</tr>
<tr>
<td><strong>B</strong></td>
<td>2.80</td>
<td>0.0113</td>
<td>2.39</td>
<td>0.0462</td>
</tr>
<tr>
<td><strong>Ca</strong></td>
<td>207</td>
<td>14.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>K</strong></td>
<td>17.6</td>
<td>0.0722</td>
<td>32.2</td>
<td>0.415</td>
</tr>
<tr>
<td><strong>Li</strong></td>
<td>0.216</td>
<td>0.00176</td>
<td>0.225</td>
<td>0.00265</td>
</tr>
<tr>
<td><strong>Mg</strong></td>
<td>228</td>
<td>0.346</td>
<td>222</td>
<td>1.54</td>
</tr>
<tr>
<td><strong>P</strong></td>
<td>0.355</td>
<td>0.00649</td>
<td>0.245</td>
<td>0.0393</td>
</tr>
<tr>
<td><strong>Si</strong></td>
<td>1.63</td>
<td>0.0103</td>
<td>1.26</td>
<td>0.0290</td>
</tr>
<tr>
<td><strong>Sr</strong></td>
<td>Sat. peak</td>
<td>4.13</td>
<td>0.0648</td>
<td></td>
</tr>
<tr>
<td><strong>V</strong></td>
<td>0.0440</td>
<td>4.90 E-18</td>
<td>0.0803</td>
<td>0.000882</td>
</tr>
<tr>
<td><strong>Zn</strong></td>
<td>0.0247</td>
<td>0.00120</td>
<td>0.242</td>
<td>0.159</td>
</tr>
</tbody>
</table>

Ag, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Sb, Se, Sr, and Ti were scanned but not observed in reportable concentrations. The Burrard Inlet surface water temperature is monitored by NOAA, and ranges from 6.8 °C to 11.2 °C (World Sea Temperature, 2017). Salinity at our sampling location was comprehensively evaluated during a previous UBC study, averaging 26 kg/m³ (Davidson, 1973).
4.3.2 CEB Solutions

All CEB make up solutions were stored in sealed containers at 4°C and used within 4 weeks.

Two types of CEB make up solutions were used: UF permeate and NF concentrate.

UF permeate was collected directly from the UF system described in Section 4.2.1. The characteristics of the UF permeate are summarized in Tables 8 and 9.

Table 8 – UF Permeate TOC (n=3)

<table>
<thead>
<tr>
<th>Sample Date</th>
<th>Average TOC (mg/L)</th>
<th>Standard Error (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apr-11-2017</td>
<td>1.9</td>
<td>0.14</td>
</tr>
<tr>
<td>May-10-2017</td>
<td>1.5</td>
<td>0.048</td>
</tr>
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</table>

Table 9 – UF Permeate Metals Analyses (n=3)

<table>
<thead>
<tr>
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</thead>
<tbody>
<tr>
<td></td>
<td>Average Conc. (mg/L)</td>
<td>Standard Error (mg/L)</td>
</tr>
<tr>
<td>B</td>
<td>2.81</td>
<td>0.0279</td>
</tr>
<tr>
<td>Ca</td>
<td>201</td>
<td>6.16</td>
</tr>
<tr>
<td>K</td>
<td>18.2</td>
<td>0.280</td>
</tr>
<tr>
<td>Li</td>
<td>0.220</td>
<td>0.00145</td>
</tr>
<tr>
<td>Mg</td>
<td>228</td>
<td>0.504</td>
</tr>
<tr>
<td>P</td>
<td>0.417</td>
<td>0.0358</td>
</tr>
<tr>
<td>Si</td>
<td>1.73</td>
<td>0.0261</td>
</tr>
<tr>
<td>Sr</td>
<td>Saturated peak</td>
<td></td>
</tr>
<tr>
<td>V</td>
<td>0.0437</td>
<td>0.00186</td>
</tr>
<tr>
<td>Zn</td>
<td>0.0457</td>
<td>0.0167</td>
</tr>
</tbody>
</table>
NF concentrate was generated using the method described in Section 4.2.2. The characteristics of the NF concentrate are summarized in Tables 10 and 11.

Table 10 – NF Concentrate TOC (n=3)

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<tr>
<th>Sample Date</th>
<th>Average TOC (mg/L)</th>
<th>Standard Error (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apr-11-2017</td>
<td>2.5</td>
<td>0.088</td>
</tr>
<tr>
<td>May-10-2017</td>
<td>1.7</td>
<td>0.064</td>
</tr>
</tbody>
</table>

Table 11 – NF Concentrate Metals Analyses (n=3)

<table>
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<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average Conc. (mg/L)</td>
<td>Standard Error (mg/L)</td>
</tr>
<tr>
<td>B</td>
<td>3.18</td>
<td>0.0364</td>
</tr>
<tr>
<td>Ca</td>
<td>310</td>
<td>14.3</td>
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<tr>
<td>K</td>
<td>19.6</td>
<td>0.227</td>
</tr>
<tr>
<td>Li</td>
<td>0.240</td>
<td>0.00265</td>
</tr>
<tr>
<td>Mg</td>
<td>260</td>
<td>0.987</td>
</tr>
<tr>
<td>P</td>
<td>0.391</td>
<td>0.000333</td>
</tr>
<tr>
<td>Si</td>
<td>2.951</td>
<td>0.028</td>
</tr>
<tr>
<td>Sr</td>
<td>Saturated peak</td>
<td>6.47</td>
</tr>
<tr>
<td>V</td>
<td>0.0483</td>
<td>0.00176</td>
</tr>
<tr>
<td>Zn</td>
<td>0.0213</td>
<td>0.00491</td>
</tr>
</tbody>
</table>
4.5 Analytical Methods

4.5.1 Chlorine (Free)

Chlorine analysis was completed using a Hach Chlorine Kit, Model CN-70 with Cat. 1407099 DPD Free Chlorine Reagent per manufacturers instructions.

4.5.2 Ultraviolet Absorbance (254 mm)

UVA analysis was completed using a Unicam Spectronic UV300 UV-Visible Spectrometer per Standard Methods 5910B.

4.5.3 Total Suspended Solids

Total suspended solids analysis was completed within the UBC laboratory per Standard Methods 2540D.

4.5.4 Adenosine Triphosphate (ATP)

ATP testing was completed using two Luminultra ATP test kits:

- Luminultra Quench Gone Aqueous QGA-25 for seawater samples
- Luminultra Deposit & Surface Analysis DSA-25 for membrane fibers

Samples were collected from the filtration system and analyzed immediately without storage. For aqueous samples a 50 mL sample was used, except in the case of the CEB residual solution, which used a 30 mL sample due to low available sample volume. Membrane fiber samples were harvested immediately following the completion of a trial. A 6 cm membrane length was used for each sample. All samples were analyzed in triplicate. Triplicate standards were completed during each day of testing. Further details of the protocols for ATP analysis are presented in Appendix C.
4.5.5 Total Organic Carbon

TOC analysis was completed using a Shimadzu TOC-L Total Organic Carbon Analyzer in conjunction with a Shimadzu ASI-L Auto Sampler. The non-purgeable organic carbon (NPOC) method was used.

Feedwater and CEB make up solution samples were collected in 40 mL amber vials and preserved using two drops of 10% hydrochloric acid solution before analysis.

A 4 point calibration was completed before each series of analyses. Blanks of the DI water used to complete membrane fiber analysis were analyzed. All samples were analyzed in triplicate.

4.5.6 Metals

29 metals were analyzed using inductively coupled plasma optical emission spectroscopy (ICP-OES). A Perkin Elmer Optima 7300 DV in conjunction with a CETAC ASX-520 Auto Sampler were used to complete the analyses.

Seawater samples were collected in 14 mL plastic vials and preserved using one drop of trace metal grade 70% nitric acid solution.

All samples were analyzed in triplicate.
5 Results and Discussion

The results and discussions are divided into 4 sections:

1. Review of the filtration trials, including (1) short-term benchmarking trials, (2) long-term trials, (3) comparison of UF fiber configurations, (4) comparison of different CEB solutions, and (5) summary of all trials, presented in Tables 12 and 13.

2. Analysis of the impact of cleaning effort on fouling rates.

3. Assessment of the biological activity on fouled membranes.

4. Insights into residual chlorine concentrations and degradation rates.

All figures are edited to include TMP values during the permeation cycle only.

Statistical analysis was completed to determine the 95% confidence interval for both the linear regression’s slope and y-intercept, as well as the $R^2$ for the overall regression.
5.1 Filtration Trials

5.1.1 Short-Term Benchmarking Trials with ZW1500 Modules

The base case condition considered in Trial 1 was operation with a permeate flux of 17 l/m²-h, CEB flux of 34 l/m²-h, CEB duration of 2 minutes, CEB frequency of 2 hours, and NaClO concentration of 10 ppm. These conditions were selected based on discussions with GE and considered as the most conservative conditions that would remain economically viable. As presented in Figure 14, a relatively low fouling rate (0.0039 ± 0.00025 PSI/hr, where ± corresponds to the 95% confidence interval) could be maintained for the base case conditions investigated. At this fouling rate, the system could operate for approximately 2 months before reaching the maximum allowable TMP of 8 PSI, and therefore requiring more extensive cleaning. Here, and in the discussion that follows, the estimated time before reaching the maximum allowable TMP was estimated based on extrapolating the observed fouling rate until a TMP of 8 PSI was reached.

![Figure 14](image)

Figure 14 – Trial 1, ZW1500, CEB duration: 2 min, CEB frequency: 2 hr, NaClO conc: 10 ppm

For Trial 2 the operating conditions were similar to those for Trial 1, with the exception that the CEB frequency was decreased from 2 hours to 12 hours. Results were collected from 2 parallel trains, A1 and A2, operated with identical conditions. As presented in Figure 15, the fouling rate (0.011 ± 0.0005 and 0.00750 ± 0.0004 PSI/hr for Trains A1 and A2, respectively) was greater than that observed in Trial 1. At this fouling rate, the system could only operate for
approximately 1 month before reaching the maximum allowable TMP. For this reason, the conditions investigated in Trial 2 were not considered feasible.

Figure 15 – Trial 2, ZW1500, CEB duration: 2 min, CEB frequency: 12 hr, NaClO conc: 10 ppm

For Trial 3 the conditions were similar to those of Trial 2, with the exception that the duration of CEB was increased to 5 minutes. As presented in Figure 16, a fouling rate (0.013 ± 0.0001 PSI/hr) similar to Trial 2 was observed. At this fouling rate the system could operate for approximately 1 month before reaching the maximum allowable TMP.

Figure 16 – Trial 3, ZW1500, CEB duration: 5 min, CEB frequency: 12 hr, NaClO conc: 10 ppm
The primary outcomes from the short-term benchmarking trials were as follows.

- For the base case operating conditions (permeate flux of 17 l/m²-h, CEB flux of 34 l/m²-h, CEB duration of 2 minutes, CEB frequency of 2 hours, and NaClO concentration of 10 ppm) the fouling rate was low. Under these conditions the system could operate for approximately 2 months before reaching the maximum allowable TMP.

- Increasing the CEB frequency from 2 to 12 hours substantially increased the fouling rate. Under these conditions the system could operate for approximately 1 month before reaching the maximum allowable TMP.

- Increasing the CEB duration from 2 to 5 minutes did not decrease the higher rate of fouling observed when operating with a CEB frequency of 12 hours. Suggesting CEB frequency has a greater impact on fouling rate than CEB duration.
5.1.2 Long-Term Trials with ZW1500 Modules

In contrast to the short-term benchmarking trials, which lasted from 5 to 9 days, the long-term trials were performed over periods ranging from 33 to 102 days. These longer term trials enabled the fouling rates to be more accurately determined, and provided more realistic insight into the fouling that would be expected in field conditions.

The initial operating conditions for Trial 4 were similar to those of Trial 3, with the exception that the CEB frequency was reduced to 6 hours. As presented in Figure 17a, the fouling rate (0.0036 ± 0.00002 and 0.0039 ± 0.00002 PSI/hr for Trains A1 and A2, respectively) was similar to Trial 1. At this fouling rate, the system could operate for approximately 2 months before reaching the maximum allowable TMP.

In an effort to further reduce the fouling rate, after approximately 300 hours of operation the CEB duration was increased from 5 minutes to 10 minutes. As presented in Figure 17b, the fouling rate (0.011 ± 0.0002 and 0.010 ± 0.0002 PSI/hr for Trains A1 and A2, respectively) was higher than the initial conditions despite the higher level of cleaning effort. The fouling rate was initially similar to the first operating period, but, after approximately 380 hours of operation, the fouling rate abruptly increased. This was likely due to changes in the raw seawater characteristics, though additional characterization of the seawater before and after this operating period would have been required to confirm this hypothesis. This result also provided further evidence of the lesser efficacy of increased CEB duration versus CEB frequency.

As the maximum allowable TMP of 8 PSI neared (i.e. 650 operating hours), all operating conditions were kept constant, except that the NaClO concentration was increased from 10 to 50 ppm. As presented in Figure 17c, the previously observed fouling trend reversed, resulting in ‘cleaning’ of the membrane during the 50 hour operating period (fouling rates of -0.012 ± 0.001 and -0.011 ± 0.0009 PSI/hr for Trains A1 and A2, respectively). These results suggested that higher concentration CEB cleanings could reduce system TMP using our existing simplified fouling control approach.

The NaClO concentration was again further increased to 100 ppm for an additional 50 hours of operation. As presented in Figure 17d, despite the higher NaClO concentration, fouling was observed (0.023 ± 0.0008 and 0.016 ± 0.0007 PSI/hr for Trains A1 and A2, respectively). The
greater fouling rate observed was likely due to changes in the raw seawater characteristics at that time. Again, additional characterization of the seawater, before and after 700 hours, would have been required to confirm this hypothesis.

NaClO concentration was again further increased to 150 ppm for the final 50 hours of operation for Trial 4. As presented in Figure 17e, the fouling trend immediately reversed, resulting in cleaning of the membrane at rates 5 to 6 times faster than those observed during the 50 ppm operating period (-0.069 and -0.051 PSI/hr for Trains A1 and A2, respectively).

The results from the latter portion of Trial 4 indicate that the permeability of an extensively fouled membrane can be recovered using CEB with a high concentration of NaClO (i.e. 150 ppm). In trials that follow, CEB with a high concentration of NaClO (i.e. 150 ppm), defined as extended CEB (ECEB), was used to recover permeability of extensively fouled membranes.

The results also suggest that slight changes in raw water characteristics may greatly impact membrane fouling and cleaning, but, additional characterization of the seawater throughout the trial period would is required to confirm this hypothesis.
Figure 17 – Trial 4, ZW1500,
a: CEB dur: 5 min, CEB freq: 6 hr, NaClO conc: 10 ppm, b: CEB dur: 10 min, CEB freq: 6 hr, NaClO conc: 10 ppm,
c: CEB dur: 10 min, CEB freq: 6 hr, NaClO conc: 50 ppm, d: CEB dur: 10 min, CEB freq: 6 hr, NaClO conc: 100 ppm,
e: CEB duration: 10 min, CEB frequency: 6 hr, NaClO conc: 150 ppm
The initial operating conditions for Trial 5 were similar to those of Trial 1, with the exception that the CEB duration was increased to 5 minutes, and the NaClO concentration was increased to 30 ppm. As presented in Figure 18a, the fouling rate (0.0018 ± 0.00001 and 0.0015 ± 0.00001 PSI/hr for Trains A2 and A2, respectively) was lower than observed in Trial 1. At this fouling rate, the system could operate for approximately 5 months before reaching the maximum allowable TMP. Note that during the period of 480 to 650 hours mechanical and datalogging malfunctions took place. This period was omitted from Figure 18a.

After approximately 650 hours of operation the NaClO concentration was reduced to 25 ppm. As presented in Figure 18b, the fouling rate (0.0030 ± 0.00002 and 0.0039 ± 0.00002 PSI/hr for Trains A1 and A2, respectively) was similar to Trial 1. At this fouling rate, the system could operate for approximately 2 months before reaching the maximum allowable TMP.

After approximately 900 total hours of operation, an ECEB was performed on Train A2 with a CEB flux of 34 l/m²-h, CEB duration of 15 minutes, and NaClO concentration of 150 ppm. The ECEB resulted in a complete recovery of permeability. Following the ECEB, Train A2 resumed operation under the previous operation conditions. Train A1 (which did not receive an ECEB) resumed operation under the previous operating conditions, but at a lower flux of 10 l/m²-h (vs. 17 l/m²-h for Train A2). As presented in Figure 18c, the fouling rate (-0.00024 ± 0.00001 and 0.00026 ± 0.00001 PSI/hr for Trains A1 and A2, respectively) was markedly lower than anything observed in previous trials. Train A1 cleaned the membrane, while Train A2 could operate for 2.5 years before reaching the maximum allowable TMP.

After approximately 1245 total hours of operation, the NaClO concentration was reduced to 15 ppm. As presented in Figure 18d, the fouling rate (-0.000090 ± 0.00003 and 0.000052 ± 0.00003 PSI/hr for Trains A1 and A2, respectively) remained extremely low. Train A1 cleaned the membrane, while Train A2 could operate for approximately 13 years before reaching the maximum allowable TMP.

After approximately 1375 total hours of operation, the NaClO concentration was reduced to 10 ppm. As presented in Figure 18e, the fouling rate (0.000059 ± 0.00001 and 0.00091 ± 0.00002 PSI/hr for Trains A1 and A2, respectively) remained low. Train A1 could operate for approximately 140 months before reaching the maximum allowable TMP, while Train A2 could operate for 9 months.
Figure 18 – Trial 5, ZW1500,
a: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 30 ppm, b: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 25 ppm,
c: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 25 ppm, d: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 15 ppm),
e: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 10 ppm.
The primary objective of Trial 6 was to validate the results from Trial 5 using both Trains A1 and A2 and Trains B1 and B2. Note that Train A1 was operated with UF permeate as the CEB solution throughout the trial. However, Train A2 was operated with NF concentrate for the period ranging from 1318 to 2444 hours. Train B1 was also operated with UF permeate throughout the trials, while Train B2 was operated with NF concentrate for the operating period ranging from 68 to 1530 hours. As discussed in Section 5.1.4, similar results were observed when using either UF permeate or NF concentrate. For this reason, in the analysis that follows, the results for CEB with UF permeate or NF concentrate are considered as equivalent.

The initial operating conditions for Trial 6 were similar to those of the final phase of Trial 5, except that the NaClO concentration was further reduced to 8 ppm. As presented in Figures 19a, the fouling rate ($0.0018 \pm 0.000003$ and $0.0011 \pm 0.000002$ PSI/hr for Trains A1 and A2, respectively) was similar to that observed in the first period of Trial 5. At this fouling rate, the system could operate for approximately 6 months before reaching the maximum allowable TMP.

After approximately 1100 total hours of operation, an ECEB was performed on both Trains A1 and A2 with a CEB flux of 34 l/m²-h, CEB duration of 15 minutes, and NaClO concentration of 150 ppm. The ECEB recovered most of the permeability. Following ECEB, operation was resumed under conditions identical to those prior to the ECEB. As presented in Figure 19b, the fouling rate observed after the ECEB ($-0.00012 \pm 0.000002$ and $-0.00024 \pm 0.000002$ PSI/hr for Trains A1 and A2, respectively) was lower than observed prior to the ECEB. This was consistent with the previous result in Trial 5, where superior performance was observed following the ECEB.

As presented in Figure 19c, the fouling rate for Trains B1 and B2, was lower than observed for Trains A1 and A2. The fouling rate ($0.00025 \pm 0.000004$ and $0.00031 \pm 0.000002$ PSI/hr for Trains B1 and B2, respectively) was the lowest observed in the first phase of any of the trials. At this fouling rate, the system could operate for approximately 30 months before reaching the maximum allowable TMP. It is not clear why the fouling rate for Trains B1 and B2 were lower than for Trains A1 and A2. Because Train B1 and B2 started 50 hours after Train A, it is possible that differences in raw water characteristics may have contributed to the observed differences. Additional characterization of the seawater during this 50 hour period would have been required to confirm this hypothesis.
After approximately 850 total hours of operation the CEB frequency was reduced to 3 hours. As presented in Figure 19d, the fouling rate (0.00074 ± 0.00001 and 0.00077 ± 0.000004 PSI/hr for Trains B1 and B2, respectively) increased slightly. At this fouling rate, the system could operate for approximately 11 months before reaching the maximum allowable TMP.

After approximately 1350 total hours of operation the CEB frequency was further reduced to 4 hours. As presented in Figure 19e, the fouling rates (0.0010 ± 0.00001 and 0.0013 ± 0.00001 PSI/hr for Trains B1 and B2, respectively) again increased slightly. At this fouling rate, the system could operate for approximately 7 months before reaching the maximum allowable TMP.

Following the completion of Trial 6, Trains A1 and A2’s fibers were harvested and visually inspected. As presented in Figure 20, a foulant layer was clearly visible on the membranes, with more solids accumulating on fibers near the module bulkhead. Over extended periods, extensive solids accumulation could lead to a sludging (i.e. extensive accumulation of solids within a membrane module), resulting in reduced performance. Means of minimizing solids accumulation should be further investigated.

The primary outcomes from the long-term trials were as follows.

- A low fouling rate could be sustained over an extended period by operating with a permeate flux of 17 l/m²-h, CEB flux of 34 l/m²-h, CEB duration of 5 minutes, CEB frequency of 2 hours, and NaClO concentration of 8-10 ppm. At this low fouling rate, the system could operate for approximately 4 to 30 months before reaching the maximum allowable TMP of 8 PSI, therefore requiring more extensive cleaning.

- Operation with a permeate flux of 10 l/m²-h further reduced the fouling rate. Under these conditions, the system could operate for approximately 140 months before reaching the maximum allowable TMP of 8 PSI, therefore requiring more extensive cleaning.

- ECEB could recover lost permeability due to fouling.

- ECEB not only proved effective at removing accumulated foulants, but the fouling rate in the operating periods following the ECEB was observed to be lower than prior to the ECEB.

- Accumulation of solids on the membrane fibers (sludging) may be a concern during long term operation and should be further investigated.
Figure 19 – Trial 6, ZW1500,

a: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 8 ppm, b: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 8 ppm,
c: CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 8 ppm, d: CEB dur: 5 min, CEB freq: 3 hr, NaClO conc: 8 ppm,
e: CEB dur: 5 min, CEB freq: 4 hr, NaClO conc: 8 ppm
Figure 20 – ZW1500 modules following Trial 6
a: Train A1, b: Train A1 (close up), c: Train A2, d: Train A2 (close up)
5.1.3 Comparison of Outside-In (ZW1500) and Inside-Out (ZW700B) Hollow Fiber UF Configurations

In addition to the filtration trials previously discussed in Sections 5.1.1 and 5.1.2, additional side by side comparison trials were performed using inside-out membrane configurations (i.e. ZW700B fibers) during Trials 4 and 5. Note that only the first operating periods of Trials 4 and 5 are considered in the discussion that follows. This was due to substantial fouling accumulating during the initial trial phase, which may have influenced subsequent fouling rates.

The initial operating conditions for the ZW700B Trains (B1 and B2) during Trial 4 were identical to those of the ZW1500 Trains (A1 and A2). As presented in Figure 21, the fouling rate (0.027 ± 0.0001 and 0.028 ± 0.0002 PSI/hr for Trains B1 and B2, respectively) was higher than observed for the outside-in configuration, previously presented as Figure 17a. At this fouling rate, the system could operate for approximately 9 days before reaching the maximum allowable TMP.

The initial operating conditions for the ZW700B Trains (B1 and B2) during Trial 5 were, again, identical to those of the ZW1500 Trains (A1 and A2). As presented in Figure 22, the fouling rate (0.0053 ± 0.00005 and 0.0036 ± 0.00004 PSI/hr for Trains B1 and B2, respectively) was lower than Trial 4. However, the fouling rate was still considerably higher than observed for the outside-in configuration, previously presented as Figure 18a. At this fouling rate, the system could operate for approximately 2 months before reaching the maximum allowable TMP.

The greater fouling observed for the inside-out configuration was likely due to differences in system geometries. For the ZW700B modules, the CEB fluid was substantially diluted by the liquid present in the module casing at the start of a CEB. Minimal dilution occurred in the ZW1500 system.

Photographs of the cross section and tip of ZW700B fibers harvested at the end of Trial 5 are presented in Figure 23. Substantial deposits were visible at the filtration surface for some fibers, but not for others; this suggests that some channels may have become clogged, reducing the effective filtration area and therefore increasing the permeate flux in remaining areas. The higher than expected permeate flux likely contributed to the higher fouling rate.

The primary outcomes from the comparison of different UF configurations were as follows.
• A low fouling rate could not be sustained with inside-out module configurations (i.e. (ZW700B).

• The ZW700B (outside-in) membrane fiber configuration produced fouling rates far higher than the ZW1500 (inside-out).

• Channel clogging for inside-out membrane fiber configurations may have decreased the effective filtration area, and therefore, increased the effective permeate flux, resulting in greater fouling.

Figure 21 – Trial 4, ZW700B, CEB dur: 5 min, CEB freq: 6 hr, NaClO conc: 10 ppm
Figure 22 – Trial 5, ZW700B, CEB dur: 5 min, CEB freq: 2 hr, NaClO conc: 30 ppm

Figure 23 – Autopsy of Trial 6 ZW700B Fiber
5.1.4 Comparison of UF Permeate and NF Concentrate as CEB Make Up Solutions with ZW1500 Modules

Previous trials by others demonstrated significantly different fouling rates, depending on the characteristics of the CEB make up solution (Li et al., 2012). However, these previous trials were performed using significantly higher operating fluxes than those considered in the present study. To assess the impact of differing CEB make up solution characteristics on fouling rates, for the conditions considered in the present study, a side by side comparison was performed using UF permeate and NF concentrate. The characteristics of each of these solutions is presented in Section 4.3.2.

**UF Permeate vs. NF Concentrate**

UF permeate and NF concentrate were compared as CEB make up solutions during Trial 6. Train A1 was operated with UF permeate throughout the trial, while Train A2 operated with NF concentrate for the period ranging from 1318 to 2444 hours. Train B1 was also operated with UF permeate throughout the trial, while Train B2 operated with NF concentrate for the period ranging from 68 to 1530 hours.

The average fouling rate of trains using UF permeate were similar to those using NF concentrate (0.00013 PSI/hr for Trains A1 and B1 compared to 0.00011 PSI/hr for Trains A2 and B2). Considering that UF permeate is a valuable product and NF concentrate is a waste, the use of the latter is recommended as a solution for CEB.

The primary outcomes from the comparison of UF permeate and NF concentrate as CEB make up solutions were as follows.

- The use of UF permeate as a CEB make up solution did not decrease the fouling rate compared to the use of NF concentrate. The use of NF concentrate is therefore recommended, as it is a waste product of the treatment process.
5.1.5 Summary of all Trials

Tables 12 and 13 summarize the results of the trials completed over the course of this study. Statistical analyses were completed to determine the 95% confidence intervals for both the slope and y-intercept of the linear regressions used to quantify the fouling rate.

$R^2$ values are also included, acknowledging that they are low for fouling rates approaching 0.
### Table 12 – ZW1500 Trials

<table>
<thead>
<tr>
<th>Train</th>
<th>Operating Hours</th>
<th>Fouling rate (PSI/hr)</th>
<th>95% CI +</th>
<th>95% CI -</th>
<th>Y-int 95% CI+</th>
<th>95% CI-</th>
<th>R²</th>
<th>Cleaning Effort**</th>
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<tr>
<td>2 hr perm / 2 min bw / 10 ppm</td>
<td>A1</td>
<td>0 - 137</td>
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<td>0.0041</td>
<td>0.0036</td>
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<td>1.97</td>
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<td>0.0080</td>
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<td>0.013</td>
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<td>6 hr perm / 5 min bw / 10 ppm</td>
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<td>-0.012</td>
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<td>0.022</td>
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<td>95% CI -</td>
<td>Y-int</td>
<td>95% CI+</td>
<td>95% CI-</td>
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<td>0.00091</td>
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</tr>
<tr>
<td>2 hr perm / 5 min bw / 8 ppm</td>
<td>A1</td>
<td>0 - 1100</td>
<td>0.0018</td>
<td>0.0018</td>
<td>0.0018</td>
<td>1.38</td>
<td>1.39</td>
<td>1.38</td>
</tr>
<tr>
<td></td>
<td>A2</td>
<td></td>
<td>0.0011</td>
<td>0.0011</td>
<td>0.0011</td>
<td>1.11</td>
<td>1.12</td>
<td>1.11</td>
</tr>
<tr>
<td>2 hr perm / 5 min bw / 8 ppm (Post ECEB)</td>
<td>A1*</td>
<td>1100 - 2444</td>
<td>-0.00012</td>
<td>-0.00012</td>
<td>-0.00012</td>
<td>2.28</td>
<td>2.28</td>
<td>2.28</td>
</tr>
<tr>
<td></td>
<td>A2*</td>
<td></td>
<td>-0.00024</td>
<td>-0.00023</td>
<td>-0.00024</td>
<td>1.87</td>
<td>1.88</td>
<td>1.87</td>
</tr>
<tr>
<td>2 hr perm / 5 min bw / 8 ppm</td>
<td>B1</td>
<td>0 - 859</td>
<td>0.00025</td>
<td>0.00026</td>
<td>0.00025</td>
<td>1.14</td>
<td>1.14</td>
<td>1.14</td>
</tr>
<tr>
<td></td>
<td>B2</td>
<td></td>
<td>0.00031</td>
<td>0.00032</td>
<td>0.00031</td>
<td>1.46</td>
<td>1.46</td>
<td>1.46</td>
</tr>
<tr>
<td>3 hr perm / 5 min bw / 8 ppm</td>
<td>B1</td>
<td>859 - 1363</td>
<td>0.00074</td>
<td>0.00075</td>
<td>0.00073</td>
<td>0.61</td>
<td>0.62</td>
<td>0.60</td>
</tr>
<tr>
<td></td>
<td>B2</td>
<td></td>
<td>0.00077</td>
<td>0.00078</td>
<td>0.00077</td>
<td>0.99</td>
<td>1.00</td>
<td>0.99</td>
</tr>
<tr>
<td>4 hr perm / 5 min bw / 8 ppm</td>
<td>B1</td>
<td>1363 - 1700</td>
<td>0.0010</td>
<td>0.0010</td>
<td>0.00099</td>
<td>0.32</td>
<td>0.34</td>
<td>0.31</td>
</tr>
<tr>
<td></td>
<td>B2</td>
<td></td>
<td>0.0012</td>
<td>0.0013</td>
<td>0.0012</td>
<td>0.40</td>
<td>0.41</td>
<td>0.39</td>
</tr>
</tbody>
</table>
Table 13 – ZW700B Trials

<table>
<thead>
<tr>
<th>Trial</th>
<th>Train</th>
<th>Operating Hours</th>
<th>Fouling rate (PSI/hr)</th>
<th>95% CI +</th>
<th>95% CI -</th>
<th>Y-int 95% CI+</th>
<th>95% CI-</th>
<th>R²</th>
<th>Cleaning Effort**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial 4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6 hr perm / 5 min bw / 10 ppm</td>
<td>B1*</td>
<td>0 - 450</td>
<td>0.027</td>
<td>0.027</td>
<td>0.027</td>
<td>-1.80</td>
<td>-1.76</td>
<td>-1.83</td>
<td>0.96</td>
</tr>
<tr>
<td></td>
<td>B2*</td>
<td></td>
<td>0.028</td>
<td>0.028</td>
<td>0.028</td>
<td>-2.29</td>
<td>-2.24</td>
<td>-2.33</td>
<td>0.95</td>
</tr>
<tr>
<td>3 hr perm / 5 min bw / 10/50/100/150 ppm</td>
<td>B1*</td>
<td>450 - 800</td>
<td>Not considered</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>B2*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial 5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 hr perm / 5 min bw / 30 ppm</td>
<td>B1*</td>
<td>0 - 380</td>
<td>0.0053</td>
<td>0.0054</td>
<td>0.0053</td>
<td>0.82</td>
<td>0.83</td>
<td>0.81</td>
<td>0.88</td>
</tr>
<tr>
<td></td>
<td>B2*</td>
<td></td>
<td>0.0036</td>
<td>0.0036</td>
<td>0.0035</td>
<td>1.32</td>
<td>1.33</td>
<td>1.31</td>
<td>0.82</td>
</tr>
<tr>
<td>2 hr perm / 5 min bw / 40/50/60 ppm</td>
<td>B1*</td>
<td>380 - 910</td>
<td>Not considered</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>B2*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Trial phase not used in analyses completed in Section 5.2

**Cleaning effort: see Section 5.2
5.2 Modelling Fouling Rate as a Function of Cleaning Effort

A substantial amount of data on long term fouling rates was collected over the course of the present study. When reviewing the results presented in Figures 14 to 19, and Table 12, the filtration trials could be classified as having one of two outcomes:

1. ‘Fouling’: characterized by a fouling rate greater than 0, or
2. ‘Cleaning’: characterized by a negative fouling rate.

In the analysis that follows, only results for the trials operated at a permeate flux of 17 l/m²-h using the ZW1500 modules, for which fouling was observed, were considered. Also, only results from trials for which the NaClO concentration was ≤ 15 ppm were considered. Continuous operation, using higher NaClO concentrations, was not considered to be feasible as there were concerns over high residual levels in the CEB reject. In addition, because operation with the ZW700B modules was not considered to be feasible due to substantial fouling, results from these trials were not considered. The results considered in the analysis are identified in Table 12 as those without an asterisk.

To compare the results from the different trials, a new measure was considered: cleaning effort. Cleaning effort is defined as the ratio of the cleaning dose (i.e. product of the CEB duration and NaClO concentration) to CEB frequency, as presented in Equation 3.

\[
\text{Cleaning Effort (min – ppm/hr)} = \frac{\text{CEB Duration (min) x NaClO Conc. (ppm)}}{\text{CEB frequency (hrs)}} \quad \text{Equation 3}
\]

As illustrated in Figure 24, an exponential decay relationship as presented in Equation 4 was observed between fouling rate and cleaning effort. Additional details and validation of the modelling approach are presented as Appendix D.

\[
y = a \cdot e^{-kx} \quad \text{Equation 4}
\]
The fouling rate rapidly decreased as cleaning effort was increased from 1.7 to 10 min-ppm/hr. In this range significant improvements were possible with marginal increases in cleaning effort. Cleaning effort beyond 20 min-ppm/hr did not provide additional benefits. In this range improvements were only possible with significant increases in cleaning effort. An optimal range was observed from 10 to 20 min-ppm/hr, where most of the benefits of increasing cleaning effort were realized. All trials utilized CEB durations of 2 to 5 minutes, excepting the trial period utilizing a 10 minute CEB (Trial 4, period 2), which has been highlighted in Figure 24. The comparatively high fouling rate for the cleaning effort in this period suggests that CEB frequency has a greater positive effect on fouling rate than CEB duration.

These results were somewhat expected considering that once a surface has been cleaned, further efforts to remove foulants will not prove productive. Therefore, it is logical that when attempting to control fouling in operating systems there is a point of diminishing returns. For the conditions
investigated, the ideal operating range is between 10 and 20 min-ppm/hr, using a CEB duration of 2 to 5 minutes.

The primary outcomes from the analysis of fouling rates and cleaning effort were as follows.

- The relationship between fouling rate and cleaning effort is exponential in nature, with fouling rates initially rapidly decreasing as cleaning effort is increased to an optimal point, beyond which no further benefit was observed.
- The optimal operating range for the proposed fouling control approach was between 10 and 20 min-ppm/hr, using an NaClO concentration between 8 and 15 ppm.
- CEB frequency appears to have a larger impact on fouling rate than CEB duration, with the optimal CEB duration determined to be between 2 and 5 minutes, beyond which additional benefits were not observed.
5.3 Assessment of Biological Activity on the Membranes

Fouling associated with the presence of attached biological growth (i.e. biomass) and associated extra polymeric substances (EPS) has been identified as a key challenge for UF and RO seawater filtration operations, particularly in areas prone to algal bloom (Tabatabai et al., 2014). Previous work demonstrated that hydraulic backwash alone is not capable of maintaining a sustainable fouling rate in seawater applications, due to irrecoverable fouling attributed to organic material derived from biomass (Resosudarmo et al., 2013). It was hypothesized that the presence of NaClO in our trials would address this issue, as previous trials have confirmed NaClO CEBs help detach biopolymers from fouled membranes (Wang et al., 2014).

A biomass balance analysis was performed to determine if biomass accumulated on the membrane over successive cycles (i.e. not effectively removed by CEB). ATP was used as a surrogate measurement for biomass using the techniques described in Section 4.5.4. The biomass balance analysis was completed following the completion of Trial 6 (Train A2).

The biomass balance quantified, within one filtration cycle:

i. ATP added via influent seawater (A).

ii. ATP leaving via permeate (B).

iii. ATP released from the membrane fibers during CEB (CEB residual) (C).

iv. ATP present on membrane fibers following CEB (D).

v. ATP degraded (calculated) (E).

The above 5 processes can be summarized as presented in Equation 5 and Figure 25.

\[
\text{Influent Seawater (A)}_{\text{ATP}} = \text{Permeate (B)}_{\text{ATP}} + \text{CEB Residual (C)}_{\text{ATP}} + \text{Equation 5} \\
\text{Membrane Fibers (D)}_{\text{ATP}} + \text{Degradation (E)}_{\text{ATP}}
\]
As presented in Figure 26, the mass of ATP leaving the system, and present on the membrane fibers, was substantially less than that in the influent seawater. If CEB was not effective at removing or inactivating biomass that can accumulate and grow on the membrane during filtration, then it would be expected to accumulate over successive cycles. The biomass balance determined that there was no accumulation of ATP within the control volume, but instead, that the majority of ATP in the influent seawater could not be accounted for. The amount that could not be accounted for was assumed to have been degraded by NaClO oxidation, as previously observed by others (Nescerecka et al., 2016).
The primary outcomes from the assessment of biological activity on the membranes were as follows.

- There was no evidence of accumulation of active biomass on the membrane fibers following successive filtration cycles.
- A significant fraction of the influent biomass could not be accounted for, suggesting that this fraction was oxidized during CEB. This suggests that frequent NaClO backwashes increase the biological stability of fluids within the overall process train.
5.4 Residual Chlorine and Degradation Rates

The disposal of chlorinated water into sensitive receiving bodies such as streams, lakes, and oceans, is an environmental concern due to negative effect on aquatic life occurring even at relatively low concentrations. As a consequence of this, Environment Canada has set the water quality guideline value for reactive chlorine discharge into marine waters at a relatively low level of 0.5 μg/L (Canadian Council of Ministers, 1999).

With the knowledge that chlorinated wastewater would be generated during our CEB operations, residual chlorine concentrations were continuously monitored throughout the later trials, in order to determine the potential effort that may be required to dechlorinate wastewater in a full-scale system. This information is summarized below in Table 14. Note that the discrepancy between ZW1500 and ZW700B trials is attributed to dilution within the casing prior to sample retrieval.

<table>
<thead>
<tr>
<th></th>
<th>CEB NaClO Conc. (ppm)</th>
<th>CEB Residual Average (ppm)</th>
<th>CEB Residual Range (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>ZW1500</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial 5 – Trains A1/A2</td>
<td>30</td>
<td>10.8</td>
<td>5.4 – 17.0</td>
</tr>
<tr>
<td>Trial 5 – Trains A1/A2</td>
<td>25</td>
<td>9.8</td>
<td>5.2 – 12.0</td>
</tr>
<tr>
<td>Trial 5 – Trains A1/A2</td>
<td>15</td>
<td>6.8</td>
<td>5.1 – 9.0</td>
</tr>
<tr>
<td>Trial 5 – Trains A1/A2</td>
<td>10</td>
<td>2.7</td>
<td>1.5 – 4.2</td>
</tr>
<tr>
<td>Trial 6 – Trains A1/A2</td>
<td>8</td>
<td>1.0</td>
<td>0.4 – 1.6</td>
</tr>
<tr>
<td>Trial 6 – Trains B1/B2</td>
<td>8</td>
<td>1.4</td>
<td>0.5 – 2.4</td>
</tr>
<tr>
<td><strong>ZW700B</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial 5 – Trains B1/B2</td>
<td>30</td>
<td>1.8</td>
<td>0.8 – 2.5</td>
</tr>
<tr>
<td>Trial 5 – Trains B1/B2</td>
<td>60</td>
<td>2.7</td>
<td>0.9 – 4.6</td>
</tr>
</tbody>
</table>

To assess how rapidly chlorine would decay once released, batch degradation tests were performed.
Chlorine decay in water is typically described by the first order relationship presented below in Equation 6 (Hua et al., 1999):

\[ C = C_o \times e^{-k_c \times t} \]  

Equation 6

where, \( C \) = Chlorine concentration (ppm), at time \( t \) (h), \( C_o \) = Initial chlorine concentration (ppm), \( k_c \) = Chlorine decay rate (h\(^{-1}\)).

Decay trials (at 4 °C) were completed for a variety of different solutions used throughout these experiments. A summary table with this information has been included below as Table 15.

### Table 15 – Chlorine Decay Rate Constants

<table>
<thead>
<tr>
<th>Solution</th>
<th>Chlorine Decay Rate, ( k_c ) (h(^{-1}))</th>
<th>( R^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>UF Permeate</td>
<td>0.0007</td>
<td>0.99</td>
</tr>
<tr>
<td>CEB residual</td>
<td>0.0009</td>
<td>0.99</td>
</tr>
<tr>
<td>Raw Seawater</td>
<td>0.0006</td>
<td>0.93</td>
</tr>
<tr>
<td>NF Concentrate</td>
<td>0.0005</td>
<td>N/A (3 data points only)</td>
</tr>
</tbody>
</table>

As presented in Table 15, the decay rate was low for all solutions considered. If a residual NaClO concentration of 1.0 ppm is assumed, it would take approximately 350 to 630 days for the NaClO concentration to naturally decrease to a concentration lower than 0.5 \( \mu \)g/L (for the range of decay rates observed). Therefore, alternative means of removing the NaClO from the CEB reject will likely be required prior to release into the environment.

The primary outcomes from the residual chlorine analyses were as follows.

- Residual chlorine in CEB reject streams can be expected above regulatory limits.
- The rate of chlorine decay was low. Therefore, de-chlorination prior to release into the environment is required.
6 Conclusions and Recommendations for Further Research

6.1 Conclusions

The feasibility of CEB as the only fouling control approach to achieve sustainable fouling rates in low temperature ultrafiltration of seawater was confirmed via multiple long-term filtration trials. Insights gained during these trials are summarized below.

1. CEB with NaClO concentrations as low as 8 ppm were effective at achieving sustainable fouling rates in low temperature UF filtration of seawater. ECEB using 150 ppm NaClO solutions was effective at restoring lost permeability, though may not be required for periods of 6 months or more.

2. Hollow fiber, outside-in membranes (ZW1500) outperformed multi-channel inside-out membranes (ZW700B). The lower effective NaClO concentration in the CEB solution for the ZW700B system may have contributed to the poorer performance.

3. NF concentrate proved to be an effective CEB make up solution despite containing more impurities than higher quality UF permeate.

4. A new measure, cleaning effort, was proposed to compare fouling control efficiency for different CEB operating conditions. Based on cleaning effort, an optimal operating range was identified.

5. CEB with NaClO was effective at preventing the accumulation of active biomass on the membrane fibers.

6. Residual chlorine in the CEB reject stream was observed above regulatory limits. The rate of chlorine decay in solution was low, suggesting de-chlorination will be required prior to release to the environment.
6.2 Recommendations for Further Research

The research presented herein validated a simplified means to control fouling in low temperature seawater filtration operations. Recommendations for further research are summarized below.

1. Results from the bench-scale trials indicated solids accumulation (sludging) may require supplementary cleaning efforts following long term operation. A pilot study using commercial modules is therefore recommended to explore the extent of solids accumulation that would be expected in full-scale operations.

2. Changing water quality was suspected of influencing the fouling rate throughout longer term trials. It is therefore recommended that the impact of feedwater quality on fouling rate be further investigated in subsequent trials.

3. The comparison of outside-in versus inside-out membranes could be enhanced by further trials wherein module design limits the dilution of CEB solution prior to reaching the membrane surface for inside-out geometries.
References


Appendix A – Membrane Conditioning Protocols

Virgin UF Membrane Conditioning Protocol

Each newly constructed UF membrane module was thoroughly cleaned to remove any preservatives before use in trials. The procedure used is as follows:

1) Newly potted membranes are soaked in a 750 ppm NaClO and DI water solution for 18 hours.
2) 750 ppm solution is then filtered through the module at 17 LMH for 20 minutes.
3) Container is drained of NaClO solution and replaced with DI water. DI water is then filtered through the module at 17 LMH for 20 minutes.
4) Cleaned modules are then stored in DI water until use.

Prior to seawater trials, a clean water flux test was completed with DI water, providing a final opportunity to remove any remaining preservatives.

Source: GE

Virgin NF Membrane Conditioning Protocol

The virgin NF module was conditioned via multiple rounds of DI water filtration (approximately 72 hours) to ensure storage chemicals were completely removed. Filtrate was continuously tested for conductivity until the filtrate matched the DI feedwater.
Appendix B - Pressure Transducer Calibration Curves

Typical calibration curves for the pressures transducers used during this study are presented in Figures B1 and B2. Calibrations occurred on the following dates:

March 4th, 2016 – for Trains A1 / A2


Figure B1 - Train A1 Pressure Transducer Calibration

Figure B2 - Train A2 Pressure Transducer Calibration
Appendix C – ATP Testing Protocols

Quick Reference Guide
Deposit and Surface Analysis Test Kit
Product #: DSA-25 / DSA-100

Step 1 - UltraCheck™ 1 Calibration
Perform one UltraCheck 1 calibration per day or per each set of samples analyzed.

Step 2 - Sample Preparation → Select one of the following options:
Option A - SURFACE SWAB
Option B: MEASURED DEPOSIT
Option C: BIOFILM COLLECTOR

Step 3 - Total ATP (TATP™) Analysis → Then perform the following steps:
3.1 – INCUBATION
Allow time for complete extraction.

3.2 – DILUTION
Dilute out interferences.

3.3 – ASSAY
Measure ATP concentration.

Calculations → Carry out calculations that correspond to the selected preparation method:

A - Surface Swab (Default $A_{sample} = 25cm^2$):

\[
\begin{align*}
\text{uATP (pg ATP/cm²)} &= \frac{RU_{ATP}}{RU_{ATP_{ref}}} \times 50,000 \text{ (pg ATP/cm²)}
\end{align*}
\]

OR

\[
\begin{align*}
\text{uATP (pg ATP/g)} &= \frac{RU_{ATP}}{RU_{ATP_{ref}}} \times 50,000 \text{ (pg ATP/g)}
\end{align*}
\]

B - Measured Deposit (Default $m_{sample} = 1g$):

\[
\begin{align*}
\text{uATP (pg ATP/cm²)} &= \frac{RU_{ATP}}{RU_{ATP_{ref}}} \times \frac{1 \text{ ME}}{0.003 \text{ pg ATP}}
\end{align*}
\]

OR

\[
\begin{align*}
\text{uATP (pg ATP/g)} &= \frac{RU_{ATP}}{RU_{ATP_{ref}}} \times \frac{1 \text{ ME}}{0.003 \text{ pg ATP}}
\end{align*}
\]

C - Biofilm Collector:

\[
\begin{align*}
\text{uATP (ME/device)} &= \frac{RU_{ATP}}{RU_{ATP_{ref}}} \times \frac{50,000 \text{ (pg ATP)}}{1 \text{ device}}
\end{align*}
\]

OR

\[
\begin{align*}
\text{uATP (ME/device)} &= \frac{RU_{ATP}}{RU_{ATP_{ref}}} \times 1 \text{ ME} \times \frac{50,000 \text{ (pg ATP)}}{0.003 \text{ pg ATP}}
\end{align*}
\]

Date Modified: 5-Apr-13  © LuminUltra 2013

NOTE: If $RU_{ATP} \leq 5000$ using a PhotonMaster or LuminMaster C-110, rehydrate a new bottle of Luminase for maximum sensitivity.

NOTE: If $RU_{ATP} \leq 50$ using a PhotonMaster or LuminMaster C-110, consider accounting for background (RU_{bkgd}). See Test Kit Instructions for guidance.

NOTE: 1 ME [Microbial Equivalent] assumes 0.001 pg [1 µg] ATP per cell.
Quick Reference Guide
Quench-Gone™ Aqueous Test Kit
Product #: QGA-25 / QGA-100

Step 1 - UltraCheck™ 1 Calibration
Perform one UltraCheck 1 calibration per day or per each set of samples analyzed.

NOTE: If RLU < 5,000 using a PhotonMaster or Lumitester C-110, rehydrate a new bottle of Luminase for maximum sensitivity.

Step 2 - Cellular ATP (eATP™) Analysis

2.1 - MEASURE SAMPLE VOLUME
Determine volume and filter sample.

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Volume (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cooling or Process Water</td>
<td>10 to 25</td>
</tr>
<tr>
<td>Fresh Brackish &amp; Salt Water</td>
<td>25 to 50</td>
</tr>
<tr>
<td>Reclaimed Water, Effluents</td>
<td>25 to 50</td>
</tr>
<tr>
<td>Drinking and Sanitary Water</td>
<td>50 to 100</td>
</tr>
<tr>
<td>High Purity Water</td>
<td>&gt; 100</td>
</tr>
</tbody>
</table>

2.2 - FILTRATION
Filter sample.

2.3 - EXTRACTION
Extract ATP from filter & dilute.

2.4 - ASSAY
Measure ATP concentration.

Calculations

Cellular ATP (eATP) Calculation:

\[ eATP (\mu g \text{ ATP}/mL) = \frac{RLU_{total}}{RLU_{blank}} \times \frac{10,000 (\mu g \text{ ATP})}{RLU_{total} (mL)} \]

Microbial Equivalent (ME/mL):

\[ eATP (ME/mL) = eATP (\mu g \text{ ATP}/mL) \times \frac{1 \text{ ME}}{0.001 \mu g \text{ ATP}} \]

NOTE: 1 ME (Microbial Equivalent) assumes 0.001 pg (1 fg) ATP per cell.

Date Modified: 5-Apr-13 © LuminUltra 2013
Appendix D – Modelling Fouling Rate as a Function of Cleaning Effort (Additional Details)

An exponential decay relationship was observed among the data, described in Equation D1:

\[ y = a \cdot e^{-kx} \]  
\[ \text{Equation D1} \]

Linearized, this becomes Equation D2:

\[ \ln(y) = \ln(a) - k \cdot (x) \]  
\[ \text{Equation D2} \]

Where in the present case: \( y = \) fouling rate (PSI/hr), and \( x = \) cleaning effort (min-ppm/hr)/

In the analysis that follows, only results for the trials operated at a permeate flux of 17 l/m²-hr using the ZW1500 modules for which fouling was observed were considered. Also, only results from trials for which the NaClO concentration was \( \leq 15 \) ppm were considered. Continuous operation using higher NaClO concentrations was not considered to be feasible as there were concerns over high residual levels in the CEB reject. In addition, because operation with the ZW700B modules was not considered to be feasible due to substantial fouling, results from these trials were not considered. The results considered in the analysis are identified in Table 12 as those without an asterisk. The linearized data has been presented in Figure D1. Yielding coefficients of: \( a = 0.0117 \) and \( k = -0.131 \).

![Figure D1 - Linearized Fouling Rate Data vs Cleaning Effort with 95% Confidence Intervals](image)
Note the two trials found outside the 95% confidence intervals were trials in which the CEB duration was taken from 5 minutes to 10 minutes following a sustained fouling period (Trial 4, period 2). As this operating period used operating conditions that are not comparable with the other trials (which had a CEB duration of 2 to 5 minutes), an alternate figure is presented below as Figure D2 with these outliers removed. This yielded a superior $R^2$ of 0.79, though similar exponential decay coefficients of $a = 0.0104$ and $k = -0.140$.

![Figure D2](image.png)

**Figure D2 – Linearized Fouling Rate Data vs. Cleaning Effort (Outliers Removed)**

As illustrated in Figure D3, an exponential decay relationship (as presented in Equation D1) accurately depicts the relationship between fouling rate and cleaning effort (coefficients $a = 0.0104$ and $k = 0.140$).
Figure D3 – Fouling Rate vs. Cleaning Effort (ZW1500 Trials 1 to 6) with exponential decay curve \((a = 0.0104, \ k = 0.140)\). Error bars correspond to 95% confidence interval boundaries.