ANAEROBIC MEMBRANE BIOREACTORS: ENHANCING PERFORMANCE OF AN EXTERNAL TUBULAR NANOFILTRATION MEMBRANE MODULE

by

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ABSTRACT

An anaerobic membrane bioreactor (AnMBR) integrated with an external nanofiltration tubular membrane module was operated for approximately 577 days in a cross flow mode to treat a potato-starch based high-strength (highly concentrated in chemical oxygen demand (COD)) wastewater. Research objectives were to enhance the physical and chemical aspects of membrane fouling prevention, without hindering the reactor performance. First, the rate of membrane fouling was reduced through optimizing the physical cleaning processes applied to the membrane module. Secondly, different chemical cleaning methods were applied to two membrane modules with a goal of selecting an optimal cleaning methodology to apply to fouled membrane modules.

Investigations conducted alongside the chemical and physical cleaning optimizations analyzed a decline in the methanogenic activity of the biomass, the foaming propensity of the reactor, and a micronutrient deficiency affecting the performance of the bioreactor. Conclusions found that incorporating a single chemically enhanced backwash (CEB) cycle offered a larger increase in sustainable membrane flux (therefore lower rate of membrane fouling) than varying the permeate backwash duration, frequency, or flow rate. Throughout this period of data collection the reactor was able to maintain a COD removal efficiency greater than 97%, a total suspended solids (TSS) removal efficiency greater than 99%, and methane yields within 71% – 89% of theoretical. A caustic clean without backwashing cycles and with a longer soak-time using a mixed 1% NaOH and 1% Sodium Hypochlorite solution was the best chemical cleaning methodology. Organic loading rate (OLR) had the largest influence on the foaming propensity of the reactor, and biomass activity declined by approximately 65% over the course of this research project.
DEDICATION

This thesis is dedicated to the author’s father and mother, Johnny and Nadine Snowdon, as well as his younger brother Zachary Snowdon for their help, support, and belief in the author throughout the length of the project.
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<tr>
<td>AnMBR</td>
<td>Anaerobic Membrane Bioreactor</td>
</tr>
<tr>
<td>BMP</td>
<td>Biochemical Methane Potential</td>
</tr>
<tr>
<td>BOD</td>
<td>Biochemical Oxygen Demand</td>
</tr>
<tr>
<td>CEB</td>
<td>Chemically Enhanced Backwash</td>
</tr>
<tr>
<td>COD</td>
<td>Chemical Oxygen Demand</td>
</tr>
<tr>
<td>CSTR</td>
<td>Continuous Stirred Tank Reactor</td>
</tr>
<tr>
<td>EPS</td>
<td>Extracellular Polymeric Substances</td>
</tr>
<tr>
<td>F/M</td>
<td>Food-to-Microorganisms Ratio</td>
</tr>
<tr>
<td>HRT</td>
<td>Hydraulic Retention Time</td>
</tr>
<tr>
<td>MLSS</td>
<td>Mixed-Liquor Suspended Solids</td>
</tr>
<tr>
<td>MLVSS</td>
<td>Mixed-Liquor Volatile Suspended Solids</td>
</tr>
<tr>
<td>OLR</td>
<td>Organic Loading Rate</td>
</tr>
<tr>
<td>PFD</td>
<td>Process Flow Diagram</td>
</tr>
<tr>
<td>PVDF</td>
<td>Polyvinylidene Fluoride</td>
</tr>
<tr>
<td>SMA</td>
<td>Specific Methanogenic Activity</td>
</tr>
<tr>
<td>SMP</td>
<td>Soluble Microbial Products</td>
</tr>
<tr>
<td>SMP(_M)</td>
<td>Specific Methane Production</td>
</tr>
<tr>
<td>SRT</td>
<td>Sludge Retention Time</td>
</tr>
<tr>
<td>TKN</td>
<td>Total Kjeldahl Nitrogen</td>
</tr>
<tr>
<td>TMP</td>
<td>Transmembrane Pressure</td>
</tr>
<tr>
<td>TP</td>
<td>Total Phosphorous</td>
</tr>
<tr>
<td>TS</td>
<td>Total Solids</td>
</tr>
</tbody>
</table>
**TSS**: Total Suspended Solids

**TVS**: Total Volatile Solids

**WANS**: Waste Anaerobic Sludge

**VSS**: Volatile Suspended Solids
1.0 INTRODUCTION

1.1 Background and Problem Statements

Sustainable wastewater treatment is important to protect environment and public health. Anaerobic membrane bioreactor (AnMBR) technology, being relatively recent, offers a promising, sustainable wastewater treatment alternative when it may be applied effectively. The concept of AnMBRs first appeared in the 1970s where they were used for the treatment of septic tank effluent (achieving high biomass retention, high biochemical oxygen demand (BOD) removal efficiency, and a high nitrate removal efficiency). Over the last 3 decades, AnMBRs have seen a large increase in their applications and success, proving their advantages over aerobic and non-membrane integrated anaerobic systems. In the 2000s studies on AnMBR technology focused on system performance, filtration characteristics, and membrane fouling control. Success of submerged aerobic membrane bioreactors in the early 2000s caused the idea of submerged AnMBRs to be explored. ADI Systems Inc. developed a successful submerged AnMBR for food industry wastewater treatment. The largest AnMBR application, as of 2013, was completed by ADI Systems Inc., producing an effluent free of suspended solids and with a chemical oxygen demand (COD) removal efficiency of 99.4%. In the 2010s the focus of AnMBR technology research shifted to trying to improve the energy generation efficiency, extend the product application scope, and solve membrane fouling issues (Lin et al. 2013).

Combining anaerobic processes for the degradation of organic pollutants with the physical separation capabilities of membrane filtration provides a number of benefits
over more traditional anaerobic and aerobic wastewater treatment technologies, and non-membrane integrated systems currently being used. These benefits are allocated in AnMBR’s generally lower operation and maintenance costs (due to zero oxygen requirement, and much lower excess sludge handling costs), very high quality effluent with the potential for recycle or reuse, and the production of value-added products such as biogas and select biochemicals such as volatile fatty acids, among other benefits (Dvorak et al. 2015, Evren Ersahin et al. 2016, Herrera-Robledo and Noyola 2015, Liao et al. 2007).

What had originally limited the widespread application of AnMBRs were their disadvantages with regards to membrane fouling and membrane costs. Membrane costs have decreased significantly in recent years due to advances in new membrane or module materials and manufacturing technologies, however their costing will always remain an important consideration in large-scale wastewater treatment plant applications (Skouteris et al. 2012). Continuing to remain the most significant factor limiting membranes’ cost-effectiveness, and ultimately their widespread application, is the issue of membrane fouling (Ramos et al. 2014 and Skouteris et al. 2012). Membrane fouling increases transmembrane pressure (TMP) and decreases flux, while the control of fouling increases manpower and energy requirements, demands costly chemical cleaning (which further requires waste handling), and may result in increased deterioration of the membrane material, affecting its lifespan (Ramos et al. 2014).

Fouling mitigation for a membrane module is a two-pronged approach: first, the membrane-fouling rate should be minimized and second, technologies to restore the flux of a fouled membrane must be implemented (Liao et al. 2007). Minimizing the fouling rate of a membrane module involves active, continuous management of the three major
factors known to influence its rate: the nature of the feed (wastewater to be treated) and mixed-liquor (biomass and wastewater mixture in the bioreactor), the membrane module properties, and the hydrodynamic environment experienced by the membrane module. For a given membrane module its actual membrane properties would remain consistent for the duration of its usage, however the hydrodynamic environment experienced by the membrane module could be subject to change. The hydrodynamic environment, in this study, was defined as the hydraulic processes that occur surrounding the membrane module. Operational membrane flux (subcritical, or supercritical), relaxation of forward filtration processes, backwashing of a membrane module, and module design are all factors that change the hydrodynamic conditions a membrane module experiences (Zhang et al. 2006). If enhancing the hydrodynamic environment of the membrane module, through manipulating its physical cleaning protocols, could offer a worthwhile decrease in the rate of fouling, and an increased sustained flux, then it could help justify AnMBR technology for further applications. To fully isolate and understand the impact of various physical cleaning protocols on the hydrodynamic conditions of the membrane module, reactor mixed-liquor properties and the characteristics of the influent wastewater being fed to the reactor would need to be held constant. Furthermore, the impact these cleaning protocols hold on reactor performance (such as COD removal efficiency, total suspended solids (TSS) removal efficiency, and biogas yields) would also need to be considered.

After a membrane has undergone sufficient fouling to reach its TMP or flux thresholds, further physical and chemical cleaning alternatives may be applied to remove reversible fouling from the membrane module. Chemical cleaning alternatives are generally preferred; as the strong chemical agents used in them may remove both surface
and internal pore blockages effectively (Dvorak et al. 2015, Liao et al. 2007, Vera et al. 2014). If chemical membrane cleaning processes could be enhanced and better understood they could help further justify AnMBR applications for wastewater processing. A better understanding of chemical cleaning protocols is required to reduce the quantities of chemical agents used (which incur additional operation and maintenance costs) and quantify the relationship between sequential chemical cleanings and reversible fouling removal efficiency. As membrane and fouling prevention costs will always be an important constraint in AnMBR applications, prolonging the longevity of a given membrane module through efficient chemical cleaning processes is critical to the overall success and sustainability of AnMBRs, treating either industrial or municipal wastewaters.

### 1.2 Research Objectives

The research project sought to enhance both the physical and chemical cleaning portions of membrane fouling management. First, varied physical cleaning protocols were applied to an external tubular nanofiltration membrane module, attached to an anaerobic continuous stirred tank reactor (CSTR), in an attempt to enhance the membrane module’s ability at maintaining higher fluxes and minimizing the rate at which the membrane module fouled. Secondly, a number of chemical cleaning processes were applied to two fouled membrane modules in an effort to enhance the flux recovery. To meet these goals, the following research objectives were developed:

- To evaluate the cause and effect relationship between physical cleaning variables and membrane performance parameters under pseudo steady-state conditions. In this study, variables that have an influence over the performance
of the physical cleaning protocol are the permeate backwash duration, frequency, and intensity, and the incorporation of CEBs. Ideal membrane performance would have a high flux value and a low TMP value, resulting in an increased volume of permeate (or filtrate) generation when mixed-liquor is filtered through the membrane module. Membrane performance was monitored in terms of TMP, flux, net flux, and operational permeability.

- To evaluate the cause and effect relationship between physical cleaning variables and reactor performance parameters under pseudo steady-state conditions. Reactor performance, or the ability of the overall reactor to effectively remove pollutants from the wastewater while producing good biogas generation, was monitored in terms of COD/BOD removal efficiencies, TSS removal efficiency, biogas production rate, and methane yield.

- To evaluate the cause and effect relationship between chemical cleaning variables and reversible fouling removal efficiency. Variables that have an influence over the performance of chemical cleaning protocols are the types of chemical cleaning solutions used, the duration of exposure of membrane foulants to the chemical cleaning solutions, and the incorporation of intermittent backwashing processes within the chemical cleaning process. Reversible fouling removal efficiency, or the effectiveness of the chemical cleaning to remove reversible membrane foulants, was monitored in terms of clean water and actual flux recoveries, membrane resistance coefficients, and TMP reduction.
1.3 Scope of the Research Project

The scope of the research project was broken down into 4 tasks. An overview of the scope, and a brief description of each of these tasks may be viewed in Table 1.

<table>
<thead>
<tr>
<th>Task</th>
<th>Part of Project</th>
<th>Description of Task</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Performance Enhancement Study</td>
<td>1 membrane module underwent various physical cleaning protocols to evaluate the best performing alternative.</td>
</tr>
<tr>
<td>2</td>
<td>Performance Enhancement Study</td>
<td>Over the duration of Task 1, reactor performance was simultaneously monitored. 2 membrane modules underwent various chemical cleaning protocols to evaluate the best performing alternative.</td>
</tr>
<tr>
<td>3</td>
<td>Chemical Cleaning Study</td>
<td>Methanogenic activity, foaming propensity, and micronutrient activity issues were investigated.</td>
</tr>
<tr>
<td>4</td>
<td>Complimentary Studies</td>
<td></td>
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</table>

Task 1 consisted of reducing the membrane fouling rate of an external membrane module through applying various physical cleaning protocols to it and then observing the impact of the variances. Control conditions were established, and then permeate backwash duration, frequency, and intensity were varied sequentially. Following the investigation concerning permeate backwashing, conditions were returned to those of the control and a chemically enhanced backwash (CEB) cycle was incorporated. The CEB had its frequency varied as well creating a total of 6 runs of experimental data collection, totalling 164 days of operation. It should be mentioned that both Task 1 and Task 3 are complimentary studies, as each task sought to enhance a different portion of membrane fouling mitigation. However, these tasks will be distinguished as different studies within this document for simplicity of reading. This portion of the research project will henceforth be referred to as Study 1.
Membrane performance throughout the duration Study 1 was monitored through TMP and flux data. Furthermore, operational permeability (daily flux divided by its associated daily TMP value) and net flux (the flux value factoring in the duration of backwashing processes, and the associated volume of permeate lost to backwashing processes daily) were also considered. Higher flux, net flux, and operational permeability values as well as lower TMP values were indicators of enhanced fouling-mitigation.

Task 2 was to monitor the reactor performance throughout the duration of Study 1 to ensure high biological process removal efficiencies were maintained. This was to verify that the different physical cleaning methodologies did not influence these removal efficiencies. COD removal efficiency, TSS removal efficiency, biogas production rate, and methane yield were all actively monitored and used as indicators of biological process efficiencies.

Task 3 applied different chemical cleaning processes to two fouled membrane modules, evaluating their performance. This portion of the research project will henceforth be referred to as Study 2. The first membrane module was operated for 170 days and underwent 5 chemical cleanings in total, and the data collected from these cleanings was used to select the optimum chemical cleaning protocols for the second membrane module. The second membrane module was operated for 406 days, 164 days consisting of the data collected during the membrane performance enhancement study. In total, the second membrane module underwent 6 runs of chemical cleanings. The effectiveness in the chemical cleanings was quantified through comparing both their clean water flux recovery, and their actual flux recovery.
Task 4 was to determine causations for three reactor issues that occurred over the course of the chemical and physical fouling mitigation studies. These three issues had small-scale investigations conducted around them to investigate their causations. During reactor start-up foaming issues were encountered, and therefore an investigation was conducted to evaluate the foaming propensity of the reactor. Second, it was determined within Study 1 that the pre-prepared micronutrient solution that was being dosed to the AnMBR was deficient in certain trace metals (calcium, iron, magnesium, and zinc). To gauge the impact of these deficiencies, a micronutrient study was conducted in an attempt to meet all micronutrient requirements. Additionally, over the course of Study 1 it was noticed that the methanogenic activity of the biomass decreased. Specific methane production tests were conducted to compare the methanogenic activity of the biomass on day 1 of operation, day 271 of operation (just before the commencement of Study 1), and on day 491 of operation (after Study 1 had ceased). The conclusions of these complimentary studies are found later within this document.

1.4 Organization of the Thesis
This thesis is organized into 5 chapters, the first of which consists of the background of the project, the problem statement, and the research objectives established to answer the problem statement.

Chapter 2 contains a review of pertinent literature for the research project. The basic mechanisms and benefits of both anaerobic digestion processes and membrane filtration are first outlined. Mechanisms of membrane fouling and the specific parameters known to influence the fouling rate are then considered. Finally, membrane
fouling rate reduction and membrane module chemical cleaning procedures are examined, and relevant research gaps have been identified.

The experimental methodology of the study is presented in Chapter 3 of the document. An overview of the experimental setup such as reactor configuration, membrane specifications, influent wastewater characteristics, and seed sludge characteristics are outlined. The experimental design and techniques used to collect, measure, and monitor all data pertinent to the research project are also outlined. Finally, the specific statistical approaches used to analyze the results of the two studies are mentioned.

Chapter 4 consists of the results and discussion. For Study 1, the specific physical cleaning protocols for each experimental run are detailed, along with their respective operating conditions, membrane performance results, and reactor performance results. The results from Study 1 are currently available online and awaiting publication in June 2018 within the American Society of Civil Engineers’ Journal of Environmental Engineering, and were presented at the 15th International Water Association (IWA) World Congress on Anaerobic Digestion in Beijing, China. For Study 2, the cleaning techniques used, as well as the methodology that was followed for conducting the clean water tests are explained. All chemical cleaning performance results are outlined and discussed. A portion of these results have a goal of being published in the Journal of Membrane Science, with the other portion having a goal of being presented and published in the conference proceedings at the 2018 conference of the Canadian Society of Civil Engineering in Fredericton, Canada. Finally, the results from the three complimentary studies, consisting of a micronutrient deficiency assay, an analysis on the foaming propensity of the reactor when foaming issues were exhibited,
and an observed decline in specific methanogenic activity (SMA) and specific methane production ($SMP_M$) of the mixed-liquor's biomass, are presented. The results from the foaming investigation were presented and published in the conference proceedings at the 2017 conference of the Canadian Society for Civil Engineering in Vancouver, Canada.

Chapter 5 includes the conclusions drawn from the research project, as well as potential recommendations for future studies. Future research could be conducted to further refine the conclusions found from this research project, and they are listed in this chapter. Additionally, the applicability of the research project’s results to practice are discussed.

Finally, Appendix A contains sample calculations for the COD balance that was conducted for the duration of Study 1. Appendix B contains a sample calculation for the membrane resistance coefficients determined in Study 2. Full raw data plots for flux, TMP, COD, mixed-liquor suspended solids, pH, and biogas production rate over the duration of the study are shown in Appendix C.
2.0 Literature Review

2.1 Anaerobic Digestion

Anaerobic digestion is a biological microbial enzyme mediated process consisting of four major stages, accomplished through four respective groups of microorganisms, which may be used for the degradation of organic and certain inorganic wastes in a given wastewater. The first stage of anaerobic digestion is hydrolysis, where complex polymers (such as fats, cellulose, and proteins) are converted to long-chain fatty acids, monosaccharides, and amino acids. Following hydrolysis is fermentation, an oxidation-reduction process whereby soluble organic chemicals are converted to intermediates (butyric acid, propionic acid, alcohols, and soluble sugars) by fermentative microorganisms. Acetogenic bacteria then convert the soluble fermentation by-products to acetic acid and hydrogen. Finally, two groups of methane-forming bacteria convert the acetic acid (acetoclastic methanogens) and the hydrogen produced during both fermentation and acetogenesis (through hydrogenotrophic methanogenesis) to carbon dioxide and methane gas, respectively (Young and Cowan 2004). An overview of the anaerobic digestion pathway may be viewed in Figure 1.
The inhibition of one or more of the aforementioned stages can result in a stoppage of the entire biological process. For this reason, it is critical that optimum operational conditions be supplied to the microorganisms. The optimum temperature range for anaerobic digestion is 30 – 38°C, standard mesophilic operating conditions. The optimum pH ranges from 6.6 – 7.6, being crucial that pH remains above 6.2, as methanogens cannot function below this level (Metcalf and Eddy 1991). To maintain pH above 6.2, sufficient alkalinity must be maintained in the reactor. The alkalinity is often supplied in the form of sodium bicarbonate. In addition to a source of alkalinity, macronutrients (nitrogen, phosphorous, and sulphur) as well as select micronutrients (calcium, cobalt, iron, magnesium, nickel, and zinc) must also be supplied to the reactor to insure good microbial activity and system performance (Speece 2008).
The anaerobic digestion process is generally preferred over aerobic processes for the removal of highly concentrated biodegradable organic pollutants (measured through COD and BOD) in a given wastewater due to the benefits it provides, especially in the case of highly concentrated biodegradable industrial wastewaters. Anaerobic digestion provides high removal efficiencies of COD and BOD, the potential for energy (biogas) generation, and decreased operational costs due to the absence of aeration requirements and lower waste sludge yields (decreasing sludge handling costs) (Dvorak et al. 2015). Anaerobic digestion becomes especially favourable compared to aerobic technologies when treating highly concentrated wastewaters because of more potential for value-addition (waste-to-energy and recovery of valuable biochemicals) and better cost-savings (due to no oxygen supply requirements and much lower net waste sludge production rates).

2.2 Anaerobic Membrane Bioreactors
Anaerobic membrane bioreactors are a combination of anaerobic digestion processes with membrane filtration technologies, coalescing their benefits. There are many types of membrane modules that may be used such as flat-sheet, tubular, and hollow-fibre. Hollow-fibre membrane modules have higher packing densities and cost efficiency when compared to the other types of membrane modules, making them generally a superior choice. Additionally, there are a number of membrane materials currently being used and studied such as polymer, ceramic, and metallic-based membrane modules (Dvorak et al. 2015).

There are two primary operating configurations for AnMBRs: side-stream and submerged. In side-stream configurations, also known as external membrane
configurations, the membrane module is placed outside of the reactor, and therefore a recirculation pump insures sufficient TMP in the membrane chamber. The cross-flow velocity (typically in the range of 2 – 4 m/s) of the mixed-liquor disrupts cake layer formation. Benefits associated with this mode of operation are decreased membrane fouling rate, and ease of access to the membrane module for cleaning or replacement (due to it being isolated in a loop and not located directly within the reactor). A primary issue with this configuration is increased pore clogging in the membrane module due to the shearing forces generated from the mixed-liquor’s velocity breaking down sludge flocs (Dvorak et al. 2015).

Submerged membrane modules are placed directly within the anaerobic reactor. This mode of operation generally has a decreased energy demand due to there being no requirement for a recirculation pump. Since there is no cross-flow velocity provided in this configuration shearing forces are non-existent, leading to less stress on the anaerobic microorganisms. Its disadvantages come in the form of an increased fouling rate, more down time and difficulties with membrane cleanings, and a larger membrane surface area required to achieve the same hydraulic performance as side-stream configurations (Dvorak et al. 2015).

AnMBRs provide benefits over non-membrane integrated anaerobic digestion bioreactors such as high quality effluent (with the potential for reuse of wastewaters for agricultural purposes), efficient treatment for high suspended solids wastewaters or poorly granulated mixed-liquors, and decoupling of the hydraulic retention time (HRT) and solids retention time (SRT) (Dvorak et al. 2015 and Zhang et al. 2007). Providing a high quality effluent offers a stepping-stone in the direction of closing the water loop in wastewater treatment leading to zero-liquid discharge from the industries and water
conservation, something that will be crucial to developing stronger, sustainable wastewater treatment alternatives. Decoupling of the SRT and HRT allows for greater biomass retainment within the bioreactor, leading to enhanced treatment efficiencies.

2.3 Membrane Fouling

Membrane fouling is a major disadvantage of AnMBR technology as it leads to decreased flux, and available membrane flux is one of the most important economic considerations for AnMBR applications (Liao et al. 2007). Membrane fouling can be defined as the sum of processes leading to flux deterioration and TMP increase due to surface or internal pore blockage of the membrane (Lousada-Ferreira et al. 2014). This surface or internal pore blockage is generated through the contact and deposition of suspended particles, colloids, and solutes located in the mixed liquor suspended solids (MLSS) on the membrane surface or in its pores (Iorhemen et al. 2016). For constant flux operation, the development of membrane fouling is thought to be a three-stage mechanism consisting of an initial short-term rise in TMP, a long-term gradual increase in TMP, and finally a rapid increase in TMP known as the TMP jump (Zhang et al. 2006).

Membrane fouling in AnMBRs is a composite fouling contributed from biofouling, organic, and inorganic fouling. Biofouling mechanisms are contributed from pore clogging (sources consisting of cell debris and colloidal particles), sludge cake formation (dependent on the solids concentration of the mixed-liquor in contact with the membrane module), and extracellular polymeric substances (EPS) adsorption (due to the accumulation of both EPS and soluble microbial products (SMP) on at the membrane module surface). Organic fouling is supplied through the accumulation and adsorption of
organic constituents on the membrane module surface (being largely a factor of supplied organic loading rate (OLR) to the reactor). Inorganic fouling occurs when inorganic colloids or crystals accumulate on the membrane surface and pores (Liao et al. 2007). As a result of many membrane-fouling mechanisms being present, there are a number of variables that contribute towards the overall membrane-fouling rate. These variables may be divided into three primary categories: the nature of the feed wastewater and reactor mixed-liquor, the membrane module properties, and the hydrodynamic environment encompassing the membrane module (Zhang et al. 2006).

2.3.1 Nature of the Feed Wastewater and Reactor Mixed-Liquor
The feed wastewater characteristics and the food-to-microorganism ratio (F/M) influence the nature of the reactor’s mixed-liquor, and thereby the membrane fouling rate. Mixed-liquor characteristics such as EPS concentration, SMP concentration, and colloid concentration have all been linked to the rate of membrane fouling. Certain other operational parameters such as SRT and OLR also contribute towards the relative concentrations of each of these constituents in the anaerobic reactor’s mixed-liquor (Zhang et al. 2006).

2.3.2 Membrane Properties
Membrane properties such as pore size, hydrophilicity (or the membrane’s affinity for water), type of membrane, and surface charge are all important contributors towards the membrane-fouling rate. The hydrophilicity of a membrane module can affect the permeability and affinity for initial attachment of biofouling (Zhang et al. 2006). The optimal membrane pore size is greatly dependent on the nature of the wastewater being
treated. A wastewater with a small average particle size should have a sufficient level of membrane filtration (microfiltration, ultrafiltration, or nanofiltration) selected so that its particles do not accumulate in the pores of the membrane module quickly (leading to a high membrane fouling rate). Polymer-based membrane modules are generally preferred due to their lower costs, with metallic and ceramic membrane modules being used for specialty applications. Ceramic membrane modules are generally applied where more intensive cleaning procedures are required, whereas metallic membrane modules are generally applied when temperatures much higher than typical mesophilic ranges are utilized (Dvorak et al. 2015).

2.3.3 Hydrodynamic Environment

The hydrodynamic environment experienced by a membrane module, in this study, is defined as the hydraulic processes (backwashing, cross-flow operation, etc.) that occur surrounding the membrane module, and therefore influence its performance. It is a function of both the module’s characteristics (configuration (submerged or external), and geometry (hollow-fibre, tubular, or flat-sheet)) and its mode of operation (bubbling or backwashing processes). The contributions of these two factors influences the mode of flux operation as well as the magnitude and distribution of shearing forces provided to the membrane surface and membrane pores (Zhang et al. 2006). Increased shearing forces generally lead to a decreased membrane-fouling rate due to the prevention of cake-layer formation or pore blockages, but at the cost of increased biological floc disruption (Dvorak et al. 2015 and Vera et al. 2014).
2.4 Membrane Fouling Control

Membrane fouling control is, essentially, a two-pronged approach. First, the membrane fouling rate is controlled through applying fouling-mitigation strategies such as cross-flow mode of operation, permeate backwashing, and gas scouring with biogas (Liao et al. 2007). Secondly, further physical and chemical cleaning methods are applied to previously fouled membrane modules to recover flux and decrease TMP. Chemical cleaning methods are generally preferred due to their increased flux recovery efficiencies (Dvorak et al. 2015, Liao et al. 2007, Vera et al. 2014).

Where the benefits of physical and chemical cleaning methods may be coalesced is through a relatively new process known as chemically enhanced backwashes (CEBs). A CEB combines conventional physical backwashing with chemical cleaning in one process, which could be of high importance for the design and operation of membrane-based wastewater treatment systems. Results so far with a NaOH CEB have demonstrated that it is an effective strategy in lowering the rates of total membrane fouling and irreversible membrane fouling accumulation, at the cost of a potentially enlarged average membrane pore size. CEBs using caustic cleaning solutions were also found to be more effective in lowering the fouling rate of a membrane module compared to the use of clean water as a backwashing solution, due to their caustic nature providing effective removal of organic membrane foulants. However, as of current literature, CEBs have only been investigated at weekly or monthly intervals (Zhou et al. 2014).

2.4.1 Fouling Rate Reduction

External AnMBRs use pumps to recirculate mixed-liquor through the membrane module creating a cross-flow velocity (cross-flow mode of operation), and as a result, shearing
forces on the membrane module’s surface. These shearing forces permanently disrupt cake layer formation on the membrane module’s surface, resulting in a decreased rate of fouling (Dvorak et al. 2015). Backwashing with permeate may be applied in both external and submerged AnMBRs, removing pore clogging by reversing the direction of flow through the membrane module. Parameters that influence the efficiency of permeate backwashing are backwashing frequency, backwashing duration, and the backwash flux. Typical backwash durations are 30 – 60s, occurring once every 5.8 – 15 minutes (Vera et al. 2014). Scouring of the membrane surface may be accomplished with biogas recycling, which reduces the rate of formation of cake layer on the membrane module’s surface (Dvorak et al. 2015). However, it has been remarked that biogas scouring loses efficiency with high biomass concentrations (Ruigomez et al. 2017).

2.4.2 Chemical Cleaning
Chemical cleaning processes are generally applied to a membrane module that has already been fouled (through its flux decreasing to a pre-set threshold, or its TMP increasing to a pre-set threshold) in an effort to recover membrane flux and decrease membrane TMP. Commonly used chemicals such as sodium hydroxide, sodium hypochlorite, and citric acid have been found to reduce biofouling, dissolved organics fouling, and inorganic fouling, respectively (Liao et al. 2007). Caustic, or alkaline, cleaning agents remove membrane fouling through breaking the bonds between the membrane surface and the fouling material. Sodium hypochlorite is used as one of the most common caustic cleaning reagents due to its advantages of chemical stability, high cleaning efficiency against organic foulants, and easy combination with other cleaning
agents (Cai et al. 2016 and Wang et al. 2017). Results have demonstrated that the soak time, or contact time, between a sodium hypochlorite solution and the membrane surface has a larger impact on clean water flux recovery than adjusting the actual concentration of the cleaning solution used (Wang et al. 2017). Acidic cleaning agents remove membrane fouling through dissociating salts and dissolving the fouling matrix (Zhang et al. 2006).
3.0 Materials and Methods

3.1 Reactor Configuration and Operation

The pilot-scale AnMBR reactor system used for the experiment consisted of a 1000L stainless steel anaerobic continuous stirred tank reactor (CSTR), integrated with an external tubular nanofiltration membrane module. The reactor was maintained, on average, at 36.3°C (± 0.7°C), mesophilic conditions, for the duration of all data collection periods by a heating coil surrounding the reactor. A simplified process flow diagram (PFD) of the experimental setup may be viewed in Figure 2.

Influent wastewater was supplied to the reactor through two feed tanks, one containing a sugar-based solution, and the other containing a potato-starch, macronutrient, and alkalinity mixture. The influent wastewater peristaltic pump (pump P_3 in the PFD, Masterflex, Model No. 77410-10) was set to automatically feed the system whenever the tank level fell below a pre-set threshold level, due to effluent flows and waste anaerobic sludge (WANS) flows. WANS was wasted from the system using an automatic peristaltic pump (pump P_4 in the PFD, Masterflex, Model No. 77410-10) located at the base of the CSTR (set on a 2 hour loop, with the duration of waste flow being dependent on the desired mixed-liquor suspended solids concentration).

CSTR mixed-liquor was recirculated through the external nanofiltration membrane module by a progressive cavity pump (pump P_1 in the PFD, Nemo, Model Type NMO53BYO2S14V), acting as a continuous loop. The progressive cavity pump recirculated the mixed-liquor at a flow rate of 6000 L/h. This flow rate provided a reactor turnover time of 10 minutes, and a cross-flow velocity on the membrane surface of 2.5 m/s.
Biogas Flow

Feed Tank 1

Feed Tank 2

Influent Flow

Waste Sludge Flow

P_1

P_3

GM_1

Anaerobic CSTR

Nanofiltration Membrane Module

Permeate Recycle Flow

CSTR Recycle Flow

SV_2

SV_1

Effluent & Backwash Tank Flow

P_2

Where:
P = pump,
V = solenoid valve.
GM = gas meter.

Figure 2: Schematic diagram of the experimental setup.
Before passing into the membrane module, the CSTR mixed-liquor was forced through a 0.8 mm diameter stainless steel screen (to reduce contact between large particulate materials and the membrane surface, especially during initial seeding of the reactor). As the mixed-liquor moved through the membrane module an instantaneous pressure gradient, supplied through a peristaltic pump (pump P\textsubscript{2} in the PFD, Masterflex, Model No. 7017-21) at the beginning of the permeate flow line, forced a portion of the mixed-liquor to be filtered through the membrane module. This filtrate, henceforth referred to as permeate, was recycled back into the CSTR by passing through an open solenoid valve (solenoid valve SV\textsubscript{2} in the PFD, FIP, Model No. S22). Mixed-liquor that was not filtered through the membrane module was recycled back into the CSTR via the CSTR recirculation line.

Every 15 minutes, a secondary peristaltic pump (Masterflex, Model No. 7553-12) would automatically re-direct a portion of the permeate recycle flow to a holding tank. This holding tank supplied both the permeate required for permeate backwashing of the membrane and the permeate wasted as effluent. Effluent was wasted automatically based on the level in the holding tank.

Biogas exited the CSTR through a gas-trap at the top of the reactor and passed into a plastic gas collection bag, where the flow of biogas was regulated. The regulated outflow of biogas moved through an AALBORG Mass Flow Meter (gas meter GM\textsubscript{1} in the PFD, GFM37, USA), where its total mass was measured and logged, before exiting the system. The AALBORG Mass Flow Meter was calibrated with a headspace gas having a composition of 70% CH\textsubscript{4} and 30% CO\textsubscript{2}. There was also a secondary gas trap attached to the system that wasted directly into the environment, in case of emergencies.
Two similar membrane modules were used throughout the duration of this study. The first membrane module was used for some preliminary data collection prior to the beginning of Study 1. Both membranes had the same characteristics, a schematic of the membrane modules may be viewed in Figure 3, and a cross-sectional view of the membrane modules is shown in Figure 4.

CSTR mixed-liquor recirculation flow entered the membrane module through one of its thirteen 8 mm diameter tubular channels. As the flow moved through the membrane module, a portion of it was filtered instantaneously through its 30 nm pores and moved out of the membrane module as permeate. The membrane module was composed of polyvinylidene fluoride (PVDF) membranes, and had backwash functionality present. Backwash functionality being present allowed the membrane module to be switched from forward filtration flow to backward flow. This created a backwash that rinsed out the pores of the membrane using filtered permeate.
Where:

- \( Q_{\text{Recirc}} \) = Recirculation flow path,
- \( Q_{\text{Perm}} \) = Permeate flow path,
- \( d_{\text{out}} \) = Outer diameter of membrane module channel,
- \( d_{\text{in}} \) = Inner diameter of membrane channels,
- \( L \) = Membrane module length,
- \( A_{\text{Tot}} \) = Total area of the membrane surface.

Figure 3: Schematic of the membrane module.
Figure 4: Pictorial view of cross-section of the tubular nanofiltration membrane module.
3.2 Influent Wastewater Characterization

A potato starch-based wastewater, simulating a high-strength (containing a high COD concentration) food industry wastewater, was supplied to the reactor through two tanks and two respective peristaltic pumps. One feed tank contained sucrose only (supplied through granulated sugar), accounting for approximately 15% of the total feed volume and approximately 80% of the total feed COD (approximately 68 g/L). A 20 kg bag of granulated sugar was mixed with 23 gallons of water to provide this portion of the synthetic industrial wastewater.

The second feed tank was continuously mixed and contained waste potato starch obtained from a potato processing plant (as a source of particulate organic matter), alkalinity, and macronutrients. The sources of alkalinity were sodium bicarbonate (0.36 g-HCO$_3$-/g-NaHCO$_3$) and urea (2.04 g-HCO$_3$-/g-urea). The sources of nitrogen were urea (0.46 g-N/g-urea) and solutec (0.1 g-N/g-solutec). The source of phosphorous was solutec (0.23 g-P/g-solutec). Solutec is a commercial soluble fertilizer rich in nitrogen and phosphorous. Additionally, the source of sulphur was sodium sulphate.

Macronutrient (nitrogen and phosphorous) requirements were calculated using the typical composition of a bacterial cell, C$_5$H$_7$O$_2$N, the rate of sludge wastage, and knowing the phosphorous requirement is approximately 1/7$^{th}$ of the nitrogen requirement, as mentioned in pertinent literature (Speece, 2008). Alkalinity dosages were adjusted to maintain reactor pH around a neutral pH. The potato starch mixture accounted for approximately 85% of the total feed volume and approximately 20% of the total feed COD (approximately 17 g/L).
Throughout the duration of the study nitrogen, phosphorous, and alkalinity quantities supplied in the feed varied based on system requirements. A thorough overview of the wastewater composition, with values averaged over the duration of Study 1, may be viewed in Table 2. Micronutrients were bulk-dosed to the reactor once weekly on a per-gram COD supplied basis to meet minimum requirements for a high acetic acid utilization rate (Speece 2008).

Table 2: Characteristics of the synthetic industrial wastewater.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Oxygen Demand (COD)</td>
<td>g/L</td>
<td>84.8 ± 8.9</td>
</tr>
<tr>
<td>Sucrose COD</td>
<td>g/L</td>
<td>80.2 ± 2.8</td>
</tr>
<tr>
<td>Potato Starch COD</td>
<td>g/L</td>
<td>5.2 ± 1.2</td>
</tr>
<tr>
<td>Biochemical Oxygen Demand (BOD)</td>
<td>g/L</td>
<td>29.2 ± 1.5</td>
</tr>
<tr>
<td>Total Suspended Solids (TSS)</td>
<td>g/L</td>
<td>5.4 ± 1.6</td>
</tr>
<tr>
<td>Total Kjeldahl Nitrogen (TKN)</td>
<td>mg/L</td>
<td>598 ± 125</td>
</tr>
<tr>
<td>Ammonia Nitrogen (NH₃-N)</td>
<td>mg/L</td>
<td>370 ± 181</td>
</tr>
<tr>
<td>Total Phosphorous (TP)</td>
<td>mg/L</td>
<td>176 ± 44</td>
</tr>
<tr>
<td>Phosphate Phosphorous (PO₄-P)</td>
<td>mg/L</td>
<td>176 ± 47</td>
</tr>
<tr>
<td>Total Alkalinity</td>
<td>g-CaCO₃/L</td>
<td>6.5 ± 0.5</td>
</tr>
<tr>
<td>pH</td>
<td>-</td>
<td>8.69 ± 0.40</td>
</tr>
</tbody>
</table>

3.3 Seed Sludge Characteristics

The reactor was seeded with a granular sludge acquired from an anaerobic digester treating a fruit-juice processing plant wastewater in Lassonde, Quebec. The seed sludge had an initial total solids (TS) concentration of 23.2 g/L, total volatile solids (TVS) concentration of 19.8 g/L, TSS of 21.4 g/L, VSS of 19.5 g/L, and a specific methanogenic activity (SMA) of 0.18 g-COD₃H₄/g-VSS•day.
3.4 Analytical Techniques

There were a number of wastewater quality parameters, reactor flow rates, and pressures monitored consistently throughout the duration of the research project. These parameters and their frequency of measurement may be viewed in Table 3. An overview of the processes used to measure each parameter is detailed in the following sections.
### Table 3: Wastewater quality and operating parameters.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Frequency of Measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Oxygen Demand (COD)</td>
<td>5 times per week for influent and effluent</td>
</tr>
<tr>
<td></td>
<td>1 time per week for mixed-liquor supernatant</td>
</tr>
<tr>
<td>Biochemical Oxygen Demand (BOD)</td>
<td>1 time every 2 – 3 weeks for influent and effluent</td>
</tr>
<tr>
<td>Filtered COD (fCOD)</td>
<td>1 time per 2 – 3 months for influent</td>
</tr>
<tr>
<td>Soluble COD (sCOD)</td>
<td>1 time per 2 – 3 months for influent</td>
</tr>
<tr>
<td>Total Suspended Solids (TSS)</td>
<td>1 – 2 times per week for influent and effluent</td>
</tr>
<tr>
<td></td>
<td>5 times per week for mixed-liquor</td>
</tr>
<tr>
<td>Total Volatile Suspended Solids (TVSS)</td>
<td>1 – 2 times per week for influent and effluent</td>
</tr>
<tr>
<td></td>
<td>5 times per week for mixed-liquor</td>
</tr>
<tr>
<td>Total Kjeldahl Nitrogen (TKN)</td>
<td>1 – 2 times per week for influent and effluent</td>
</tr>
<tr>
<td>Ammonia Nitrogen (NH₃-N)</td>
<td>1 – 2 times per week for influent and effluent</td>
</tr>
<tr>
<td>Total Phosphorous (TP)</td>
<td>1 – 2 times per week for influent and effluent</td>
</tr>
<tr>
<td>Phosphate Phosphorous (PO₄-P)</td>
<td>1 – 2 times per week for influent and effluent</td>
</tr>
<tr>
<td>Total Alkalinity</td>
<td>5 times per week for effluent</td>
</tr>
<tr>
<td>pH</td>
<td>5 times per week for influent, effluent, and mixed-liquor</td>
</tr>
<tr>
<td>Viscosity</td>
<td>5 times per week for mixed-liquor</td>
</tr>
<tr>
<td>Biogas Characteristics</td>
<td>1 – 2 times per week for biogas</td>
</tr>
<tr>
<td>Headspace Gas O₂ Content</td>
<td>5 times per week for biogas</td>
</tr>
<tr>
<td>Biogas Production Rate</td>
<td>7 times per week for biogas</td>
</tr>
<tr>
<td>SMA</td>
<td>1 time per 2 – 3 months</td>
</tr>
<tr>
<td>Mixed-Liquor Temperature</td>
<td>7 times per week for mixed-liquor</td>
</tr>
<tr>
<td>Effluent and WANS Flows</td>
<td>7 times per week</td>
</tr>
<tr>
<td>Inflow, Outflow, Permeate Pressures</td>
<td>7 times per week</td>
</tr>
<tr>
<td>Membrane Flux</td>
<td>7 times per week</td>
</tr>
<tr>
<td>Recycle Flow</td>
<td>7 times per week</td>
</tr>
<tr>
<td>Backwash Pressure</td>
<td>7 times per week</td>
</tr>
<tr>
<td>Backwash Flow</td>
<td>7 times per week</td>
</tr>
</tbody>
</table>
3.4.1 COD and BOD

Influent and effluent CODs were measured using the Closed Reflux, Colorimetric Method following Standard Methods (APHA 2005). Samples were digested in a Hach DRB 200 Block Digester (USA) and had their adsorption measured using a Hach DR 3900 (620 nm wavelength, USA). The Hach DR 3900 had its calibration checked using a COD standard once monthly. Influent and effluent filtered and soluble CODs were measured after filtering a select volume of sample through standard 1.5 µm glass-fiber filter papers (Whatman 934-AH, UK) and 0.45 µm standard membrane filters (GN-6 Grid, Pall Corporation, USA), respectively. Mixed-liquor total COD, filtered COD, and soluble COD was measured after centrifuging 50 mL of mixed-liquor in an IEC Clinical Centrifuge (operated at the maximum speed of 3175 rpm, USA) for 20 minutes and using the supernatant for the processes listed above.

Influent and effluent 5-day BOD was measured using the 5-Day BOD Test following Standard Methods (APHA 2005). Final and initial dissolved oxygen (DO) concentrations were measured using an YSI DO Meter (Model No. 58, USA) calibrated at the time of usage for ambient temperature and pressure. The overall procedure used was checked once monthly using a BOD standard.

3.4.2 TSS and VSS

Influent, effluent, and mixed-liquor TSS and VSS were measured using the gravimetric method, following Standard Methods (APHA 2005). Select sample volumes were filtered through standard 1.5 µm glass-fiber filter papers (Whatman 934-AH, UK) prior to drying in the oven at 105°C.
3.4.3 Nutrients

TKN, NH$_3$-N, TP, and PO$_4$-P were all measured according to Hach’s Water Analysis Handbook (Hach Company 2002). TKN followed the Nessler Method (digestion required, range of 0 – 150 mg-TKN/L) for water, wastewater, and sludge. NH$_3$-N was measured using the Nessler Method (EPA approved – distillation required, range of 0 – 2.50 mg-NH$_3$-N/L) for water, wastewater, and seawater. PO$_4$-P was quantified using the PhosVer 3 (Ascorbic Acid) Method (power pillows required, EPA approved, range of 0 – 2.50 mg-PO$_4$$_3$-P/L) for water, wastewater, and seawater. TP measurement followed the Acid Persulfate Digestion Method (EPA approved) for water, wastewater, and seawater.

3.4.4 Alkalinity

Total alkalinity of the reactor was measured using the Titration Method following Standard Methods (APHA 2005). A selected sample size of effluent had a standard solution of sulphuric acid added until reaching a pH of 4.3. The pH of the solution during the titration process was monitored using an Orion pH Meter (Model No. 720A, USA). The probe was calibrated daily before measurement with standard solutions of pH 4 and 7.

3.4.5 pH

The pH of the mixed-liquor, effluent, and influent were measured using an Orion pH Meter (Model No. 720A, USA). The probe was calibrated daily before measurement with standard solutions of pH 4 and 7.
3.4.6 Viscosity

The apparent viscosity of the mixed-liquor was monitored using a Brookfield DV-E Viscometer (LVDVE, USA). The viscometer had a voltage of ~15 V, frequency of 50/60 Hz, power of 20 Watts, and was operated at 100 rpm.

3.4.7 Biogas Characterization and Production Rate

The methane content of the biogas was measured using a Varian CP-3800 gas chromatograph. The chromatograph was fitted with an Alltech CTR 1 concentric packed column and a TCD detector. Helium was used as a carrier gas operating at a flow rate of 30 mL/min. The temperature of the injection port was set to 120°C and the temperature of the oven was set to 180°C. Biogas injections were done in triplicates. Actual biogas flow rate was monitored continuously through an AALBORG Mass Flow Meter (GFM37, USA), where its total mass was measured and logged, before exiting the system. The headspace gas O₂ content was monitored daily using a Bacharach Oxor III Oxygen Meter (USA).

3.4.8 Methanogenic Activity (SMPₘ and SMA)

SMPₘ was determined in duplicates using a batch study at 36°C. The test was conducted in 500 mL serum bottles with a working volume of 450 mL. The bottles were filled with reactor sludge, acetic acid as substrate, pH buffer, and mineral bases. Young and Cowan 2004 outlined the procedures and mixed solutions used. SMA was measured as the highest point on the SMPₘ curves produced through the respirometry study.
3.4.9 Reactor Flow Rates, Flux, and Pressures

Effluent and WANS flows were monitored daily through measuring their actual flow during one cycle of operation, and multiplying it by the number of cycles that occur in one day of operation. CSTR mixed-liquor recirculation flow was measured through an inline flow meter, and noted once daily. Inflow, outflow, and permeate pressures were measured through inline pressure valves, and noted once daily. The membrane flux was measured daily through collecting a sample of flow over a 30 second period and multiplying it by the quantity of time the membrane module was operating in forward mode (non-backwashing time) throughout the period of one day of operation. The backwash pressure and flow was measured once daily through inline pressure and flow meters during a period of backwashing.

3.5 Experimental Design

The experimental design was developed so as to collect the data necessary for Studies 1 and 2 (the membrane performance enhancement and chemical cleaning studies, respectively) and allow a statistical analysis of the data. The three complimentary studies (decline in methanogenic activity study, micronutrient deficiency study, and foaming investigation) were initially operational issues encountered throughout Studies 1 and 2, and therefore did not have an initial specific experimental design developed for them. The complimentary studies sought to use what data was available in an attempt to determine if there were any trends or conclusions surrounding the operational issues encountered. If causations could be determined for these three operational issues, they could potentially be prevented in future pilot and full-scale AnMBR studies.
3.5.1 Experimental Design: Membrane Performance Enhancement Study

The membrane performance enhancement study experimental design was developed using the alternating treatment design methodology (Leedy and Ormrod 2005). This experimental design methodology involved a sequential variation of the physical cleaning processes applied to the membrane module so that the effects each variation had on membrane and reactor performance could be compared against the consequences of no variation (Leedy and Ormrod 2005). An overview of the experimental plan, developed using this design methodology, may be viewed in Table 4.

<table>
<thead>
<tr>
<th>Run</th>
<th>Backwash Duration (s)</th>
<th>Permeate Backwash Frequency (/30 min)</th>
<th>Permeate Backwash Intensity (Flow) (L/h)</th>
<th>Chemical Backwash Cycle</th>
<th>Run Duration (day)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20</td>
<td>1</td>
<td>30 – 50</td>
<td>N/A</td>
<td>15</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>1</td>
<td>30 – 50</td>
<td>N/A</td>
<td>25</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>2</td>
<td>30 – 50</td>
<td>N/A</td>
<td>55</td>
</tr>
<tr>
<td>4</td>
<td>20</td>
<td>1</td>
<td>55 – 70</td>
<td>N/A</td>
<td>27</td>
</tr>
<tr>
<td>5</td>
<td>20</td>
<td>1</td>
<td>30 – 50</td>
<td>1 per 24 h</td>
<td>21</td>
</tr>
<tr>
<td>6</td>
<td>20</td>
<td>1</td>
<td>30 – 50</td>
<td>1 per 8 h</td>
<td>21</td>
</tr>
</tbody>
</table>

Variations in the hydrodynamic environment experienced by the membrane module were created through changes in the permeate backwash duration, frequency, and intensity, and through the incorporation of a chemically enhanced backwash cycle. Run 1 established control conditions with a permeate backwash duration of 20s, a permeate backwash frequency of one backwash cycle per 30 min, a permeate backwash intensity in the range of 30 – 50 L/h, and no chemically enhanced backwash cycle present. After data collection for run 1 was complete, the permeate backwash duration was increased to 40s, with all other parameters influencing the hydrodynamic environment remaining constant. This process was repeated 4 more times following the physical cleaning protocols outlined in Table 4.
Each run was operated for a minimum period of three weeks or until a period of pseudo steady-state data collection had been established. For this study, pseudo steady-state conditions were considered to be a period of one week where COD removal efficiency was greater than 99% and biogas production rate fluctuated by less than 10% on a day-to-day basis. If pseudo steady-state conditions could not be established during a run, a minimum operational period of three weeks was selected so that sufficient data could be collected and verify that potential reactor operational issues did not have a direct impact on membrane performance results.

Study 1 took place during operational days 281 – 445 of overall reactor operation. Reactor operational days 1 – 281 were used to investigate various chemical cleaning alternatives, foaming issues encountered, and the organic loading rate capacity of the reactor. On day 170, the first membrane module was removed from the reactor, and a second membrane module was placed inline. It received chemical cleaning on days 191 and 267 recovering 100% of initial actual flux, and 68% of initial actual flux with each cleaning, respectively. Day 281 was the first day of data collection for Study 1 as a steady organic loading rate, effluent COD, and flux had been established for a week prior to this date.

3.5.2 Experimental Design: Chemical Cleaning Study

There were two membrane modules cleaned and analyzed in the chemical cleaning study. The first membrane module was operated for 170 days treating OLRs ranging from 0.8 – 8.2 kg/m³•d (± 1.6 kg/m³•d) and maintaining a MLSS concentration ranging from 13.6 – 33.9 g/L (± 5 g/L). This period of data collection was used to define the loading limits of the reactor, as well as foul the membrane module so various chemical
cleaning methods could be applied to it. On day 170 of operation, the membrane module was sufficiently fouled that chemical cleaning was applied.

The chemical cleaning study experimental design was created applying the pretest-posttest control group design methodology. This methodology demonstrated that an effect occurred only following the change in a certain variable from control conditions (Leedy and Ormrod 2005). Using the pretest-posttest control group design methodology, an initial clean water flux for the membrane module was established on day one of operation, prior to the membrane coming in contact with the mixed-liquor. This was accomplished through filling the CSTR with water and running the system, with the membrane module in place, noting down the clean water membrane flux exhibited by the membrane module at various recirculation flow rates.

On day 170, the membrane module loop in the system was isolated from the mixed-liquor flow path, allowing a small 50L tank to be filled with clean water and filtered through the membrane module without requiring the CSTR to be emptied. Chemical solutions were added to this clean water, as required for the particular chemical cleaning methodology in place. A total of 5 chemical cleaning methodologies were applied to the membrane module over 5 days, leading to a total of 5 successive membrane module cleanings. The overview of each chemical cleaning methodology may be viewed in Table 5.
Table 5: Chemical cleaning methodologies for membrane module 1.

<table>
<thead>
<tr>
<th>Membrane 1</th>
<th>Cycle</th>
<th>Recirculation Time (min)</th>
<th>Soak Time (min)</th>
<th>No. of Backwashes and Duration (No. and min)</th>
<th>Chemicals Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clean 1</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>60</td>
<td>N/A</td>
<td>10% Citric Acid</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Clean 2</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>60</td>
<td>N/A</td>
<td>10% Citric Acid</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Clean 3</td>
<td>1</td>
<td>90</td>
<td>N/A</td>
<td>6 and 2 min</td>
<td>1% NaOH</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>60</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>1 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>48</td>
<td>N/A</td>
<td>3 and 2 min</td>
<td>2% Citric Acid</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>N/A</td>
<td>30</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>45</td>
<td>N/A</td>
<td>3 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>45</td>
<td>N/A</td>
<td>3 and 2 min</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>N/A</td>
<td>30</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>45</td>
<td>N/A</td>
<td>3 and 2 min</td>
<td></td>
</tr>
<tr>
<td>Clean 4</td>
<td>1</td>
<td>45</td>
<td>N/A</td>
<td>3 and 2 min</td>
<td>10% Citric Acid</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>30</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>45</td>
<td>N/A</td>
<td>3 and 2 min</td>
<td></td>
</tr>
<tr>
<td>Clean 5</td>
<td>1</td>
<td>60</td>
<td>N/A</td>
<td>N/A</td>
<td>1% Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>45</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>60</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
</tbody>
</table>

Within Table 5, the recirculation time is the time that the loop was operated in forward filtration mode, where the water-chemical mixtures were filtered through the membrane module in the same forward direction as permeate would be. The soak time is the time that filtration was ceased, and the membrane module remained in contact with the water-chemical mixtures. The number of backwashes and duration show the frequency and duration at which filtration was switched from forward filtration mode to backwards filtration mode (backwashing) during a cleaning cycle. Finally, the chemicals used column details the exact concentrations of chemicals within the clean water that were used for each cycle of the chemical cleaning.
Following each chemical cleaning, the 50L tank was filled with clean water and operated in forward filtration mode at recirculation flow rates similar to the control conditions. The clean water fluxes achieved at each of these recirculation flow rates were recorded, and compared back to the control. This allowed the clean water flux recovery of each chemical cleaning methodology to be quantified to identify which chemical cleaning methodology was the most efficient in recovering clean water flux.

The second membrane module was operated from days 171 – 577. Days 171 – 280 consisted of establishing pseudo-steady state operating conditions for Study 1 (establishing a consistent OLR, effluent COD, and MLSS concentration), days 281 – 445 consisted of data collection for Study 1, and days 457 – 577 were a period of supplemental data collection for Study 2 (the reactor was maintained at conditions similar to those of operational days 281 – 445). The membrane module had a total of 6 chemical cleanings applied to it over its duration of operation.

Unlike the first membrane module, the second membrane module had its control conditions established using the 50L clean water loop. Its initial clean water fluxes were recorded at various recirculation flow rates, and it also had its initial actual flux measured once coming into contact with the mixed-liquor for the first time. The methodologies of the chemical cleanings that were applied to the second membrane module may be viewed in Table 6.
The chemical cleaning methodologies that were applied to the second membrane module were those that offered the largest recoveries in clean water flux during the testing on the first membrane module. Following each chemical cleaning, the 50L tank was filled with clean water and operated in forward filtration mode at recirculation flow rates similar to the control conditions. The clean water fluxes achieved at each of these recirculation flow rates were recorded, and compared back to the control. This allowed

<table>
<thead>
<tr>
<th>Membrane 2</th>
<th>Cycle</th>
<th>Recirculation Time (min)</th>
<th>Soak Time (min)</th>
<th>No. of Backwashes and Duration (No. and min)</th>
<th>Chemicals Used</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clean 1</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td>1% NaOH</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>20</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>N/A</td>
<td>20</td>
<td>N/A</td>
<td>1% Citric Acid</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>N/A</td>
<td>20</td>
<td>N/A</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td>Clean 2</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>60</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Clean 3</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>60</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Clean 4</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>60</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Clean 5</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>60</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Clean 6</td>
<td>1</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td>1% NaOH</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>N/A</td>
<td>20</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>N/A</td>
<td>20</td>
<td>N/A</td>
<td>1% Citric Acid</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>N/A</td>
<td>20</td>
<td>N/A</td>
<td>1% NaOH and Hypochlorite</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>30</td>
<td>N/A</td>
<td>2 and 2 min</td>
<td></td>
</tr>
</tbody>
</table>
the clean water flux recovery of each chemical cleaning methodology to be quantified and compared to identify which chemical cleaning methodology was the most efficient in recovering clean water flux.

After the clean water flux was recorded for each membrane cleaning, the membrane was placed back inline and operation continued until it fouled sufficiently to warrant another chemical cleaning (fouled sufficiently being when operational permeability decreased close to 10 L/m²•h•bar). This allowed the actual flux to be recorded immediately following each chemical cleaning. Actual fluxes were compared back to the original actual flux of the control, and allowed the recovery efficiency in terms of actual flux to be compared for each chemical cleaning methodology.

3.5.3 Experimental Design: Complimentary Studies

There were three investigative studies that took place throughout the duration of main Studies 1 and 2. The first complimentary study was an investigation into a decline in methanogenic activity of the biomass. Over the course of the research project, it was noticed that methane yields and organic loading rate limitations decreased. Simultaneously, the effluent COD of the reactor trended upwards. This required supplemental sludge addition at different instances throughout the study, in an attempt to keep OLR at the desired level while also maintaining a low effluent COD and high methane yield.

After an investigation into the literature surrounding these trends, it was hypothesized that the shearing forces surrounding the membrane module might disrupt the anaerobic bacterial flocs, resulting in decreased methanogenic activity (Evren Ersahin et al. 2016). To test this hypothesis, specific methanogenic activity test data
from days 1, 271, and 466 of operation were compared. Each test used a food-to-
microorganism ratio (F/M) of 0.6 g-COD/g-VSS, was done in duplicates with a blank,
and used an identical influent feed solution of acetic acid feed (COD of 57 g/L). This
allowed initial control conditions to be established, and then allowed trends in SMA and
SMP_M to be analyzed following the period of operation.

The second complimentary study that took place was an investigation into the
foaming propensity of the anaerobic CSTR, examining the influence specific operational
parameters held over it. This study took place during days 1 – 170 of operation. During
this period, large and small foaming events were noted. Small foaming events were
characterized by only filling the primary gas trap of the anaerobic CSTR with foam,
leading to a quick removal and minimal disruption in operation. Large foaming events
were determined to be those events that caused a period of system shut down and
temporary period of no feeding. During large foaming events, foam poured down the
sides of the reactor through creating small gaps between the tank’s cover and liner due to
the excessive pressure, leading to a carry-over of mixed-liquor, and resultant biomass.
There were 5 large foaming events that took place over the duration of the experiment,
on days 17, 28, 42, 100, and 116.

Trends in the anaerobic CSTR’s OLR, temperature, total alkalinity, and ammonia
nitrogen concentrations in both the influent and effluent were investigated for this study.
These operational parameters and characteristics were examined as they had been found
in previous literature to have an influence over the foaming propensity of an anaerobic
reactor. The trends in each of these parameters were analyzed leading into each foaming
event, allowing percent-changes in each parameter to be quantified, ultimately
determining which factor held the greatest influence over the reactor’s foaming propensity.

The final complimentary study that took place over the duration of the study was a micronutrient deficiency assay. If the proper quantities of micronutrients are not supplied to the biomass, the system will have decreased performance in terms of COD removal efficiency and biogas production rate. A micronutrient solution was added to the anaerobic CSTR for days 1 – 323 of operation. It was determined, however, that this micronutrient solution was deficient in calcium, iron, magnesium, and zinc. To validate this hypothesis, a batch respirometric study was conducted.

A total of three 500 mL respirometer bottles were each filled with 6.7 g-VSS and fed 1.24 g-COD (the COD being supplied through feeding the respirometer bottles a portion of the synthetic wastewater fed to the CSTR). This established an F/M of 0.19 g-COD/g-VSS, the F/M conditions that were occurring in the reactor at the time of testing. One bottle was filled with only 6.7 g-VSS, and acted as a blank. Each respirometer bottle, other than the control, was fed a micronutrient blend alongside their dose of COD, and the concentration of each micronutrient in these blends may be viewed in Table 7.
Table 7: Micronutrient study blends.

<table>
<thead>
<tr>
<th>Micronutrient</th>
<th>Micronutrient Blend</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
</tr>
<tr>
<td>Calcium Concentration (mg/L)</td>
<td>84</td>
</tr>
<tr>
<td>Cobalt Concentration (mg/L)</td>
<td>5 470</td>
</tr>
<tr>
<td>Iron Concentration (mg/L)</td>
<td>91</td>
</tr>
<tr>
<td>Magnesium Concentration (mg/L)</td>
<td>44</td>
</tr>
<tr>
<td>Nickel Concentration (mg/L)</td>
<td>6 200</td>
</tr>
<tr>
<td>Zinc Concentration (mg/L)</td>
<td>8 930</td>
</tr>
</tbody>
</table>

Blend 1 was the typical micronutrient dosage being fed to the CSTR at the time of this study. Blend 2 consisted of blend 1 being doubled when it was fed to the respirometer. Blend 3 consisted of blend 1 with supplemental zinc, magnesium, calcium, iron, and magnesium supplied through the chlorides (salts) of these metals being dissolved within the regular micronutrient solution.

The quantity of each blend used was determined based on the micronutrient requirements necessary to achieve high acetate utilization rates, as outlined by Speece 2008. The regular micronutrient dosage, blend 1, was 667 mg/15 mL-wastewater, blend 2’s dose was 1447 mg/15 mL-wastewater, and blend 3’s dose was 1446 mg/15 mL-wastewater. A biochemical methane potential (BMP) test was conducted on each respirometer bottle through successive doses of COD and respective micronutrient solution to each respirometer bottle every 24 hours.
3.6 Analysis of Results

There were five groups of results that were analyzed following the completion of this research project. Membrane performance results compared average flux, TMP, operational permeability, and net flux maintained throughout each experimental run, while reactor performance results compared COD, BOD, and TSS removal efficiencies, biogas production rate, and methane yields throughout each experimental run. Additionally, nutrient concentrations in the influent and effluent, alkalinity concentration in the effluent, pH, and a COD balance were monitored over time. Chemical cleaning results sought to compare clean water and actual flux, and operational permeability recoveries pre-membrane cleaning and post-membrane cleaning. Finally, the results from the complementary studies sought to analyze an observed decline in biomass methanogenic activity throughout the duration of Study 1, operational parameters influencing the fouling propensity of the reactor, and a potential deficiency in the micronutrient solution being provided to the reactor.

3.6.1 Analysis of Results: Membrane Performance Enhancement Study

Membrane performance was analyzed with respect to average TMP, flux, operational permeability, and net flux maintained throughout the duration of each experimental run. The average values maintained were verified for statistical significance using a paired t-test, with a confidence interval of 95% and $\sigma = 5\%$. Operational permeability is defined as membrane flux divided by instantaneous operational TMP, or in other words, pressure-averaged flux (Hamdan de Andrade et al. 2013). Operational permeability was considered so that each run’s ability to maintain a favourably low TMP could be factored into its analysis.
Net flux is defined as the overall productivity of the membrane, taking into account the permeate losses and filtration cessations during backwashing cycles, and may be evaluated using Equation 1:

\[
J_{net} = \frac{J t_f - J_B t_B}{t_f + t_B}
\]

(1)

where \(J_{net}\) is the net flux (L/m\(^2\)•h), \(J\) is the permeate flux (L/m\(^2\)•h), \(J_B\) is the backwash flux (L/m\(^2\)•h), \(t_f\) is the time of filtration (h), and \(t_B\) is the time of backwash (h) (Ruigomez et al. 2017). Net flux was considered so the impact of the additional permeate backwash volume supplied through longer, higher flow, or more frequent backwashing cycles could be factored into the analysis.

The optimal operational condition was considered to be the physical cleaning protocol that maintained the highest average flux, net flux, and operational permeability and lowest average TMP throughout the duration of its run. With the nature of the feed, mixed-liquor, and membrane properties being consistent between runs, an elevated flux, operational permeability, and net flux over control conditions would signify an improvement in fouling mitigation. Furthermore, COD removal efficiency, TSS removal efficiency, and biogas production rate and yield were examined for each run to verify whether the varied physical cleaning methodologies influences these factors. Nutrient removal efficiencies (nitrogen and phosphorous), reactor total alkalinity level, and pH were all additionally monitored throughout the length of each experimental run. Nutrient removal efficiency was not attempted to be optimized, as it was defined at the beginning of the study to maintain sufficient nitrogen and phosphorous in the reactor for good biomass growth and activity without trying to minimize the effluent concentrations.
of these nutrients. Similarly, alkalinity was maintained at a sufficient level to keep the reactor pH close to neutral.

### 3.6.2 Analysis of Results: Chemical Cleaning Study

Chemical cleaning performance was analyzed according to flux and operational permeability recovery following the chemical cleaning processes. For the first membrane module clean water flux and clean water operational permeability values were collected (clean water meaning only water maintained close to 30°C filtered through the membrane for the duration of data collection). These clean water flux and clean water operational permeability values were divided by the control’s initial clean water flux and clean water operational permeability for each respective recirculation flow rate, and then multiplied by 100 to provide a clean water percent-recovered, or recovery efficiency, value for each parameter. The respective sets of flux and operational permeability recovery efficiencies were then averaged to provide one average clean water flux and operational permeability recovery efficiency value for each cleaning methodology.

For the second membrane module, the above analysis was completed once again to yield average clean water flux and operational permeability recovery efficiency values for each cleaning methodology. During the clean water testing of the second membrane module, however, five recirculation flow rates were used to collect flux and operational permeability data sets. The second membrane module was placed back inline following each chemical cleaning, therefore actual flux and operational permeability values were also calculated. Each time the membrane module was placed back inline it was raised to a recirculation rate of 6000 L/h, and allowed to recirculate for 24 h before the actual flux
and operational permeability data was collected. The actual flux and operational permeability values were again divided by control values (the value of flux and operational permeability recorded 24h after membrane module 2 was first placed in contact with the mixed-liquor) and multiplied by 100 to yield actual flux and operational permeability recovery efficiency values.

The chemical cleaning methodologies that were applied to the second membrane module were those that offered the largest clean water flux and operational permeability recovery efficiencies on the first membrane module. The optimal chemical cleaning methodology was determined to be the cleaning method that offered the largest clean water flux and operational permeability recovery efficiencies, as well as the largest actual flux and operational permeability recovery efficiencies.

3.6.3 Analysis of Results: Decline in Methanogenic Activity Study

The decline in methanogenic activity study was analyzed through comparing the SMA and average specific methane production (SMP$_{M}$) values measured through the batch respirometric study conducted for this small-scale study. A decline in biomass methanogenic activity over the course of the study would result in a decrease in its SMA and a decrease in its average SMP$_{M}$. The highest point of each set of SMP$_{M}$ data was compared for each SMP$_{M}$ test conducted, this value indicating the actual SMA of the biomass at each point of the study. Furthermore, the set of data was averaged to yield a value for average SMP$_{M}$ at each point of the study. These results were all compared to see if a decline in SMA and average SMP$_{M}$ was actually observed over the course of the experiment.
3.6.4 Analysis of Results: Foaming Investigation

The way in which certain operational factors and influent/effluent concentrations influenced the foaming propensity of the reactor was analyzed through plotting their values and concentrations leading into one of the large foaming events. Percent-change values leading into each major foaming event were determined for OLR, temperature, influent/effluent ammonia nitrogen concentration, and total alkalinity concentration. These values were compared for each factor leading into each of the five major foaming events exhibited, in an attempt to determine if there were any trends amongst the five factors occurring consistently prior to one of the major foaming events. Once a thorough analysis was completed, a foaming-mitigation framework was developed to minimize the potential chance of future foaming events occurring to the system.

3.6.5 Analysis of Results: Micronutrient Deficiency Study

The micronutrient deficiency study was analyzed through comparing the BMP test data collected throughout its duration. BMP is a measure of the quantity of methane produced per gram of COD fed to the reactor, a higher BMP indicating a greater potential for methane gas generation. Both the quantities of methane gas generated after each successive feeding and its rate of production were determined for each of the three respirometric vessels. The average rate of methane generation, average daily methane volume produced, and cumulative methane volumes produced were compared for each respirometric vessel. The optimal micronutrient blend was determined to be that which produced the greatest volume of methane at the greatest rate.
4.0 Results and Discussion

4.1 Membrane Performance Enhancement Study\textsuperscript{1,2}

4.1.1 Background
The physical cleaning protocol applied to the tubular nanofiltration membrane module was varied to enhance its ability at maintaining a favourably higher average flux, net flux, and operational permeability, while also maintaining a lower average TMP. The AnMBR system was operated for 164 days of continuous data collection for this study, changing one variable of the physical cleaning protocol at a time. Each set of cleaning conditions was maintained until pseudo steady-state operating conditions were established, or for a minimum period of three weeks of data collection. During each variance in the physical cleaning methodology, all other operational conditions were maintained as steady as possible, so as to minimize the influence other operational parameters may hold over sustained average flux, net flux, operational permeability, and TMP values.

4.1.2 Physical Cleaning Protocols
As mentioned in Section 3.5.1 there were a total of 6 varied physical cleaning protocols applied. The different protocols were used through varying the permeate backwash frequency, duration, and intensity, and by incorporating a chemically enhanced backwash cycle, varying its frequency as well. Run 1 conditions operated as a control,

For reference to publication/presentation:
and used operational conditions recommended by the membrane module manufacturer.

Runs 2, 3, and 4 varied only the permeate backwash duration (raising permeate backwash duration from 20s to 40s), frequency (increasing permeate backwash frequency from one 20s backwash per 30 min to two 20s backwashes per 30 min), and intensity (raising the backwash flow rate from 30 – 50 L/h to a backwash flow rate from 55 – 70 L/h), respectively. Runs 5 and 6 incorporated low-strength chemically enhanced backwash cycles into regular operation. Run 5 used one 1 min chemically enhanced backwash cycle per day, and run 6 incorporated three 20s chemically enhanced backwash cycles per day.

### 4.1.3 Operating Conditions

Average operational parameters for each run, and their standard deviations, may be viewed in Table 8.

**Table 8: Average operational parameters.**

<table>
<thead>
<tr>
<th>Run</th>
<th>OLR (kg-COD/m³·d)</th>
<th>MLSS (g/L)</th>
<th>MLVSS (g/L)</th>
<th>SRT (d)</th>
<th>HRT (d)</th>
<th>F/M (kg-COD/kg-VSS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.07 ± 0.14</td>
<td>21.0 ± 0.8</td>
<td>19.8 ± 0.3</td>
<td>68 ± 2</td>
<td>27 ± 1</td>
<td>0.15 ± 0.01</td>
</tr>
<tr>
<td>2</td>
<td>2.69 ± 0.57</td>
<td>19.5 ± 1.4</td>
<td>18.4 ± 1.2</td>
<td>477 ± 473</td>
<td>22 ± 4</td>
<td>0.14 ± 0.03</td>
</tr>
<tr>
<td>3a</td>
<td>2.60 ± 0.90</td>
<td>18.3 ± 1.0</td>
<td>17.4 ± 1.0</td>
<td>691 ± 470</td>
<td>25 ± 9</td>
<td>0.15 ± 0.05</td>
</tr>
<tr>
<td>3b</td>
<td>2.99 ± 0.28</td>
<td>18.4 ± 0.8</td>
<td>17.6 ± 0.8</td>
<td>536 ± 457</td>
<td>22 ± 2</td>
<td>0.17 ± 0.01</td>
</tr>
<tr>
<td>4</td>
<td>2.03 ± 0.19</td>
<td>19.9 ± 1.3</td>
<td>18.8 ± 1.2</td>
<td>802 ± 378</td>
<td>26 ± 2</td>
<td>0.11 ± 0.01</td>
</tr>
<tr>
<td>5</td>
<td>2.34 ± 0.34</td>
<td>20.2 ± 1.6</td>
<td>19.2 ± 1.4</td>
<td>202 ± 27</td>
<td>35 ± 2</td>
<td>0.13 ± 0.03</td>
</tr>
<tr>
<td>6</td>
<td>2.86 ± 0.24</td>
<td>23.2 ± 1.9</td>
<td>22.0 ± 1.7</td>
<td>180 ± 39</td>
<td>29 ± 2</td>
<td>0.13 ± 0.01</td>
</tr>
</tbody>
</table>

Note:
3a: Run 3 including the data where OLR spiked due to mechanical issues, and as a result there was an extended period of elevated effluent COD.
3b: Run 3 with 20 days of data omitted due to the OLR spike.

For the majority of the operational parameters, average values were quite consistent between runs. There was a period of data collection during run 3 where
mechanical issues caused a rapid increase in OLR (from an OLR of 3.10 kg/m$^3\times$d to an OLR of 4.49 kg/m$^3\times$d over a period of one day of operation). This abrupt increase in OLR resulted in an abrupt increase in effluent COD (from 458 mg/L to 1439 mg/L over 4 days of operation), and as a result OLR was decreased to 0.40 kg/m$^3\times$d and gradually increased back towards 3 kg/m$^3\times$d over 20 days of operation in an effort to decrease the effluent COD. Run 3a and 3b were both average operational parameters for the duration of run 3 conditions, however run 3b omitted the period of 20 days of data collection where this issue occurred so that average operational conditions during run 3 could be compared to all others with and without factoring this OLR fluctuation into the results.

Wastewater feed characteristics (OLR) and mixed-liquor characteristics (MLSS concentration) are factors known to influence the fouling propensity of a membrane module. For this reason, these parameters were maintained in the 2 – 3 kg/m$^3\times$d and 17 – 22 g/L ranges, respectively, to minimize their impacts on flux and TMP results. As the study progressed, it became apparent that the methanogenic activity of the reactor’s biomass decreased, resulting in a requirement for lower average OLR values than the beginning of the study. This phenomenon is further discussed in Section 4.3.2. To boost the methanogenic activity of the anaerobic reactor’s biomass, supplemental sludge was added to the reactor on days 91, 93, 111, 112, and 113 of Study 1. This supplemental sludge was composed of approximately 30% of the original seed sludge and 70% of WANS collected over the duration of operation. After the addition of the supplemental sludge, the reactor was able to operate at OLRs much closer to those of the control conditions (reflected when comparing the average OLR of run 6 of 2.86 kg/m$^3\times$d to that of run 1, 3.07 kg/m$^3\times$d).
SRT fluctuated from a lowest value of 61d to a highest value of 1000d. The large fluctuations in SRT were due to sludge wastage being ceased (aside from 1 L samples taken for testing, daily) for certain portions of the experiment so as to maintain MLSS and MLVSS concentrations in the desirable range. MLSS, MLVSS, and sludge wastage rate over the duration of all 6 experimental runs may be viewed in Figure 5.

![Figure 5: MLSS, MLVSS, and sludge wastage rate vs. time.](image)

Both HRT and F/M were quite consistent throughout the duration of all 6 experimental runs. Average F/M did decrease slightly in runs 4 – 6, again reflective of the decline in biomass activity that was observed. As biomass methanogenic activity declined, effluent COD increased, which resulted in OLR being decreased, and thus F/M decreasing as well.
4.1.4 Membrane Performance Results

Membrane performance was evaluated in terms of average sustained membrane flux, net flux, operational permeability, and TMP. The optimal physical cleaning protocol was the run that maintained the highest average flux, highest average net flux, highest average operational permeability, and lowest average TMP.

4.1.4.1 Flux

Flux and TMP data collected throughout the duration of Study 1 may be viewed in Figure 6.

![Figure 6: Flux and TMP vs. time.](image)

In terms of enhancing average sustained membrane flux, runs 4, 5, and 6 offered the greatest improvements with average percentage improvements of 19%, 30%, and 22%, respectively. The inclusion of CEBs offered the two most significant increases in membrane flux, with a longer, less frequent CEB (run 5) being observed as more
beneficial than a shorter, more frequent CEB (run 6). The incorporation of a CEB within regular backwashing protocol allows contact between potential fouling materials (organic or biological foulants) and chemical solutions known to remove these foulants, in this instance contact with a low-strength caustic solution. Caustic solutions cause the hydrolysis and solubilisation of organic foulants, especially polysaccharides and proteins (Kale and Singh, 2016). Allowing repetitive contact between the caustic backwashing solution and organic foulants on the membrane surface and in the membrane pores would solubilize them and allow regular backwashing to remove them from the membrane module more efficiently. A longer, less frequent CEB was more beneficial than a shorter, more frequent CEB likely due to the longer contact time between chemical solution and foulants supplied by the longer CEB. Ramos et al. 2014 observed that a dynamic chemical cleaning (chemical cleaning with recirculation and backwashing in place) offered an improvement over a static chemical cleaning (chemical cleaning with no recirculation and backwashing in place) in terms of fouling removal efficiency. The improvement was attributed to the dynamic clean promoting more contact and a better distribution of the chemical cleaning solutions and foulants. The same mechanisms are likely in place in this instance, as the longer CEB would allow a better distribution of the chemical solution within the membrane module, and three times the contact time than the shorter CEB.

Without the incorporation of a CEB, maintaining a permeate backwash flow rate in the range of 55 – 70 L/h offered a significant improvement in membrane flux over increasing the permeate backwash duration or frequency, with average improvements in membrane flux of 19%, 4%, and 3%, respectively. During run 3 conditions, increasing the permeate backwash frequency, did not offer a statistically significant improvement
in membrane flux, while the other two conditions did. However, the improvement offered by run 2 conditions was quite minimal (only a 4% increase). This result agrees with Diez et al. 2012, who found that increasing the backwash duration above 20s did not result in a major improvement in sustained membrane flux.

Backwashing removes reversible fouling, however too long of a backwashing interval leads to an increase in irreversible fouling (due to compacting of the cake foulant layer) and too short of a backwashing interval leads to both excessive wasted permeate and foulants removed from the pores of the membrane not being propelled far enough from the membrane surface (which results in them clogging the pores of the membrane module once again when filtration begins) (Smith et al. 2006). Comparing run 2 conditions to run 3 conditions, the average backwash fluxes passing through the membrane module during each backwash cycle were 108.8 L/m²h, and 77.9 L/m²h, respectively. Similarly, comparing run 4 conditions to run 2 conditions, the average backwash fluxes passing through the membrane module during each backwash cycle were 141.2 L/m²h, and 108.8 L/m²h, respectively. Therefore, it appears that a higher average backwash flux results in an increase in the sustained membrane flux during the period of operation. The large backwash flux of run 4 was potentially able to move pore-clogging foulants sufficiently far from the membrane surface that they were unable to quickly block the pores once filtration started back up.

There appears to be a relationship between average membrane flux and average backwashing flux, where an increase in average backwashing flux provides an increase in average membrane flux. However, this relationship should be further tested at higher and lower backwashing flux values than those accomplished in this study. This apparent relationship supports the thought that the increased backwash flux successfully removed
pore-clogging foulants far from the membrane surface, and that potentially increasing the backwash flux further could offer larger improvements in achievable average membrane fluxes. During run 4 the average backwash pump pressure shifted from 1.9 bar (the average for runs 2 and 3) to 2.2 bar, and this increase in pressure is not accounted for in the potential relationship. The increase in pressure likely contributed towards the large increase in average membrane flux noted.

4.1.4.2 TMP

TMP versus time data for all runs may be viewed in Figure 6. In terms of minimizing TMP, all runs aside from run 6 offered decreases in average TMP over control conditions. The larger TMP value of run 6 (1.16 bar for run 6 vs. 1.14 bar for control) may be attributed to gradual irreversible membrane fouling accumulated over the second half of run 3, and runs 4 – 6. Smith et al. 2006 found that a more frequent backwash was more effective in maintaining lower TMP values than a longer duration backwash. The results of this study found that both an increased backwash frequency (run 3) and an increased backwash duration (run 2) offered decreases in average TMP, with average TMP values of 1.13 bar and 1.12 bar, respectively. If a 13-day period of data during run 3 was omitted (due to this period being a period where membrane fouling increase rapidly, leading up to a chemical cleaning), then the average TMP for run 3 improved to 1.12 bar as well. However, unlike Smith et al. 2006, this study found that an increased permeate backwash duration was more effective in maintaining a lower TMP than an increased backwashing frequency. This may, once again, be attributed to the longer backwash duration providing a more thorough rinsing of the membrane module during
its one larger cleaning cycle compared to the more frequent backwashes providing two less thorough rinses.

Run 4 and 5 conditions offered the lowest average TMPs with values of 1.07 bar and 1.10 bar, respectively. It would appear that a large backwash flux is more effective in maintaining a favourably low TMP than the use of CEBs. This may be due to the large backwash flux fully removing pore-clogging foulants from the membrane module, whereas regular CEBs offer a more gradual decomposition of them. Additionally, run 4 was completed following a chemical membrane module cleaning during run 3, therefore the average TMP values may have been lower due to this event as well. Overall, the largest decrease in average TMP was only 6%, which is quite small when compared to the percent-changes in fluxes. This result aligns with Prieto et al. 2013, who found that the incorporation of scheduled backwashing protocols resulted in large increases in permeate flux, but nearly even values of TMP.

A longer CEB appeared to maintain a lower TMP than a more frequent CEB, which may be attributed to the increased contact time offered through the longer duration CEB. Run 6 conditions, overall, did not offer a decrease in TMP over control conditions, however this may have been due to gradual membrane fouling, as the membrane module was chemically cleaned following the termination of run 6.

During run 3 flux decreased and TMP increased sufficiently that a chemical membrane cleaning was required. The results from this cleaning will be further discussed in Section 4.2. Following this chemical cleaning, the system was operated sufficiently long (approximately 20 days) so that the membrane’s flux and TMP levels could return to their average values pre-membrane cleaning.
4.1.4.3 Net Flux

Net flux and operational permeability data collected throughout the duration of each experimental run may be viewed in Figure 7. Net flux factors the quantity of permeate recycled back into the system for permeate backwashes into the flux value, thereby giving a good measure of membrane productivity.

![Operational Permeability and Net Flux vs. Time](image)

**Figure 7: Operational permeability and net flux vs. time.**

Runs 4, 5, and 6 offered the largest improvements in net flux with average percentage improvements of 20%, 36%, and 28%, respectively. Net flux, unlike flux and operational permeability, accounts for the quantity of downtime used for backwashing. When comparing the net flux percent-improvements (20%, 36%, and 28% for runs 4, 5, and 6, respectively) to the flux percent improvements (19%, 30%, and 22% for runs 4, 5, and 6, respectively) there is a 1% increase in net flux over flux for run 4, a 6% increase for run 5, and a 6% increase for run 6. This is where the efficiency of the CEBs may be viewed, as their incorporation requires a very minimal increase in the downtime of the
membrane or volume of permeate used for backwashing, while still offering large increases in membrane flux. The chemical solution used for the CEBs weaken the cohesion forces between foulants and the membrane surface, allowing the regular permeate backwashing forces to dislodge these foulants more effectively (Chen et al. 2003). Runs 2 and 3 provided decreases in net flux, as it appears that the additional volume of permeate pumped back through the membrane under these run conditions did not offer a significant enough increase in membrane flux to warrant the trade-off.

### 4.1.4.4 Operational Permeability

Operational permeability, essentially pressure-averaged flux, factors the TMP maintained by run conditions into their respective flux values. This is important, as good system performance should have both low TMP values and high flux values. Similar to the results found in membrane flux, runs 4, 5, and 6 offered the largest improvements in operational permeability with average percent improvements of 27%, 35%, and 19%, respectively. Operational permeability factors the TMPs maintained throughout the duration of each experimental run into the flux values, offering a more standardized flux comparison. With the highest sustained flux, and the second lowest sustained TMP, run 5 conditions provided the largest increase in operational permeability. This may, once again, be attributed to the synergistic effect of the chemical solution weakening bonds between the foulants and membrane surface and the physical forces provided by the backwashing removing these foulants from the membrane module. Run 4 conditions offered an increase over run 6 conditions, however this discrepancy may be due to the period of data collection for run 4 taking place sooner after the chemical membrane cleaning than the period of data collection for run 6 (where some gradual irreversible
membrane fouling may have begun to build-up). Overall, it would appear that maintaining a forced high backwash flow, and therefore high backwash flux, offers a competitively worthwhile increase in flux compared to a more frequent CEB. The more frequent CEB likely requires a higher concentration chemical solution, as there is less contact time provided than the longer duration CEB.

Runs 2 and 3 offered minimal increases in operational permeability over control conditions, being increases of 7% and 1%, respectively. However, run 2 conditions offered a statistically significant increase in operational permeability, whereas run 3 conditions provided did not. If the 13-day period prior to the chemical cleaning in run 3 were omitted, the percent-increase in operational permeability becomes 5%, however this result provides no statistically significant change either. It appears that an increased permeate backwash duration is more favourable than an increased permeate backwash frequency; however, since both increases are below 10%, their results are not very significant.

4.1.5 Reactor Performance Results

Reactor performance was evaluated in terms of COD and TSS removal efficiencies, as well as biogas and methane gas production rates and yields. Other parameters such as nutrient removal efficiencies (nitrogen and phosphorous), effluent alkalinity, and reactor pH were also monitored throughout the period of study. Nitrogen and phosphorous were monitored, not to minimize their concentrations in the effluent, but to ensure sufficient nitrogen and phosphorous was supplied to the biomass within the bioreactor to promote good microbial health. Ensuring good microbial health, generally, results in increased COD removal efficiencies and biogas production rates. Alkalinity was maintained at a
sufficient level that it could maintain a favourable mixed-liquor pH around neutral. COD balances were also conducted throughout each experimental run to verify that the quantity of COD being supplied to the reactor was equivalent to or close in level to the quantity of COD exiting the reactor, ensuring no COD was being lost from the reactor due to unforeseen circumstances.

### 4.1.5.1 COD Removal Efficiency

The COD removal efficiency and OLR of the system are presented in Figure 8, and the effluent COD concentration and OLR of the system may be viewed in Figure 9.

![Figure 8: COD removal efficiency and OLR vs. time.](image)
All runs experienced COD removal efficiencies greater than 97% throughout their lengths. It was observed over the course of the project that effluent COD was gradually increasing while OLR remained quite steady. This gradual increase was attributed to a decline in methanogenic activity (an approximate decline of 67%) of the biomass over the duration of data collection. This phenomenon is further discussed in Section 4.3.2.

Run 5 had the highest average COD removal efficiency, with a value of 99.7% and the lowest average effluent COD, with a value of 330 mg/L. The reason for these high COD removal efficiencies, and low effluent COD values later into the study is most likely due to the supplemental sludge addition provided during run 3 and towards the end of run 4. This additional sludge would have provided biomass with an increased methanogenic activity, and boosted the system performance.
The OLR had a target value of 3 kg/m$^3$•d however, as mentioned previously, during a few instances pump issues resulted in spike increases in OLR, which in turn resulted in a rapid increase in effluent COD (and therefore a decrease in COD removal efficiency). To counteract the increases in OLR and effluent COD, OLR was decreased substantially for a few day period, and then gradually ramped back up towards the target value of 3kg/m$^3$•d.

4.1.5.2 TSS and VSS Removal Efficiencies

Influent TSS concentration and TSS removal efficiencies may be viewed in Figure 10.

Throughout the length of the study TSS removal efficiencies were greater than 99%, indicating very high removal rates. The maximum effluent TSS had a concentration of 17.9 mg/L, whereas the lowest effluent TSS had a concentration of 0.6 mg/L. The maximum influent TSS was 8340 mg/L and the minimum influent TSS was

Figure 10: Influent TSS and TSS removal efficiency vs. time.

64
1919 mg/L. The nanofiltration membrane was highly effective in removing suspended particles, showing close to 100% liquid-solid separation. These organic particles then accumulated in the CSTR until they were decomposed by the biomass.

**4.1.5.3 Biogas Production Rate and Yield**

Biogas production rate and OLR vs. time, biogas yield, methane yield, and OLR vs. time, and actual and theoretical biogas production rates vs. time are presented in Figures 11, 12, and 13, respectively.
Figure 12: Biogas yield, methane yield, and OLR vs. time. Biogas and methane yields are expressed on a per kg-COD removed basis.

Figure 13: Actual and theoretical biogas production rate vs. time.

It was observed that following an OLR spike increase or decrease biogas production rate increased or decreased simultaneously, as expected. With more soluble
organic matter being provided suddenly to the reactor, the biomass was able to rapidly digest it, and biogas production rate increased. Similarly, when a constant supply of soluble organic matter was briefly interrupted, the biomass was left with minimal organic material to digest, leading to a decrease in biogas production rate.

It may be noticed in Figure 12 that the methane yields and biogas yields gradually decreased over from run 1 to run 4, with a small increase following supplemental sludge addition. Comparing the biogas production rates and biogas yields of run 1 to run 6, average OLR was quite close (a 7% difference), whereas the biogas production rates and biogas yields showed quite a discrepancy with percent-differences of 25% and 19%, respectively. Similarly, actual methane yield declined by approximately 15% throughout the duration of the experiment. The decreases in biogas and methane yields may be attributed to the hypothesized decrease in biomass methanogenic activity, as less methane and biogas would be anticipated per gram VSS due to this decrease. Evren Ersahin et al. 2016 found that an external membrane configuration resulted in less methane production than a submerged membrane configuration operating side-by-side, attributing some of the discrepancy to the detrimental interaction between membrane shearing forces and microorganisms.

Overall, the average methane yields were in the range of 71 – 89% of the theoretical methane yield of 0.395 m³-CH₄/kg-COD, indicating satisfactory yields. Average biogas yields were in the range of 73 – 86% of the theoretical biogas production rate, indicating a satisfactory biogas production rate. Both the largest average methane yield (0.34 m³-CH₄/kg-COD) and largest average biogas production rate (1894 L/d, with a respective biogas yield of 0.62 m³-biogas/kg-COD) were both observed.
during run 1 conditions, again indicating a decline in biomass biogas generation and performance as the project progressed.

4.1.5.4 Nutrient Removals, Alkalinity, and Reactor pH

The influent and effluent TKN and NH$_3$-N values may be viewed in Figures 14 and 15.

![Figure 14: Influent TKN and NH$_3$-N vs. time.](image)
Influent TKN was, for the most part, greater than influent NH$_3$-N. This was anticipated, as TKN encompasses NH$_3$-N within its value, and indicates that there may have been additional organic or inorganic nitrogen within the feed other than NH$_3$-N. Effluent TKN and NH$_3$-N concentrations were close indicating that the majority of effluent nitrogen was in the form of NH$_3$-N. Influent nitrogen concentrations were gradually decreased in an attempt to also decrease the effluent nitrogen concentrations. There was no attempt to optimize either macronutrient concentration, however their concentrations in the feed were reduced to use fewer chemicals. Overall, average TKN removal efficiency and average NH$_3$-N removal efficiency were 77% and 62%, respectively.

Influent TP and PO$_4$-P and effluent TP and PO$_4$-P concentrations versus time may be viewed in Figures 16 and 17.
Both influent and effluent TP and PO$_4$-P values were very close indicating that nearly all of the influent and effluent phosphorous was in the form of PO$_4$-P. Similar to the nitrogen supply, the quantity of phosphorous in the feed wastewater was decreased
slightly over the duration of the project to decrease chemical costs. This may be noticed in Figure 17 when the effluent TP and PO$_4$-P concentrations gradually decrease towards the later half of data collection. On average TP and PO$_4$-P removal efficiencies were 2% and 3%, respectively.

Total alkalinity and MLSS pH vs. time may be viewed in Figure 18.

![Total Alkalinity and MLSS pH vs. Time](image)

**Figure 18: Total alkalinity and MLSS pH vs. time.**

Total alkalinity of the MLSS was initially quite steady during run 1, however it increased gradually as sludge wastage rate was ceased. MLSS pH was maintained between 6.70 – 7.36, with an average value of 7.06. On days 92 and 110 an abrupt decrease in total alkalinity may be noted, and these values coincided with a decrease in MLSS pH. These were two days where effluent COD increased rapidly due to spike loadings. The loading spikes would most likely have resulted in a brief accumulation of VFAs (which was observed in the VA/PA ratio of the effluent, it increased from approximately 0.3 to 0.41 on both of these dates), increasing effluent COD, decreasing
MLSS pH, and decreasing MLSS total alkalinity (as the alkalinity would be consumed to buffer the additional acids).

Influent and effluent pH vs. time may be viewed in Figure 19.

![Figure 19: Influent and effluent pH vs. time.](image)

Effluent pH was very steady throughout the duration of the experiment, with an average value of 7.22. Influent pH appeared to fluctuate as the feed aged with pH increasing the longer a batch of feed was allowed to sit exposed to the atmosphere. To counteract this, feed batch HRT was decreased, so as to minimize the amount of time it was allowed to sit before being fed to the reactor. The average influent pH was 8.69.

### 4.1.6 COD Balances

A COD balance conducted surrounding the system for Study 1 may be viewed in Figure 20. A COD balance is conducted to verify that close to 100% of the COD entering the system is accounted for in the various ways it might exit the system (biogas, effluent,
waste sludge, etc.). It is important that a COD balance account for the majority of the influent COD so that there are no unexpected issues within the system, which would lead to large quantities of unaccounted-for COD.

Figure 20: COD balances over the duration of the experiment.

In Figure 20 the total mass of COD supplied to the reactor (supplied via influent wastewater) for each day of operation is divided by the total mass of COD removed from the reactor each day (removed via sludge wastage, biogas generation, cell reproduction, and effluent wastage). The COD balance with accumulation includes an observed gradual increase in MLSS supernatant COD over the duration of the project, whereas the COD balance without accumulation does not include this observation. Without factoring in the accumulation of supernatant COD within the reactor, an average of 94% of COD was accounted for in the COD balance, with COD\textsubscript{out} being 6% larger than COD\textsubscript{in}. When MLSS supernatant COD accumulation was factored into the
COD balance, providing overall a more accurate COD balance, an average of 98% of the COD was accounted for in the COD balance, with COD_{out} once again being larger than COD_{in} by 2%. With 98% of the COD being accounted for, the COD balance is relatively accurate. Minor data collection errors and data measurement errors are sources of error that may have led to the small discrepancy between COD_{in} and COD_{out}.

On days 36 and 153 COD_{in}/COD_{out} spiked to values of 3.97 and 3.58, respectively. Prior to day 36 the influent pump was accidentally left off, while the system continued to waste effluent and sludge. Due to this, the system’s reactor content height level dropped significantly, and the influent pump fed the reactor more feed than usual to raise the reactor content height level back to its set point. This resulted in a brief period of system overload, where the biomass was not able to keep up with the quantity of COD provided to the reactor, resulting in much more apparent COD_{in} than COD_{out}. A similar event occurred on day 153.

The average COD balance for each run with MLSS COD accumulation factored in may be viewed in Figure 21.
Figure 21: Average COD balance for each run (with MLSS COD accumulation factored in).

During run 1 the ratio of COD\textsubscript{in}/COD\textsubscript{out} was greater than 1, and for all other runs this ratio was less than 1. This further demonstrated the accumulation of MLSS supernatant COD over the duration of the project, as it likely accumulated towards the beginning of the study, primarily during run 1, until a steady-state concentration was established for the given operational conditions. During runs 2 – 6 the COD\textsubscript{out} was greater than the COD\textsubscript{in}, as the biomass was able to consume some of the initially accumulated COD when other food sources may not have been directly present, resulting in apparent higher biogas production values. Sample calculations for the COD balance may be viewed in Appendix A.
4.2 Chemical Cleaning Study

4.2.1 Background

Two membrane modules were tested throughout the chemical cleaning study. The first membrane module was inline for the first 170 days of system operation, where the loading limitations of the system were gauged. The second membrane module was placed inline on day 171 and operated for a further 406 days, a portion of which comprised Study 1. The first membrane module had a variety of chemical cleaning methodologies applied to it, in an effort to gauge those that were the most efficient in removing membrane fouling. The second membrane module had the two cleaning methodologies that offered the largest recoveries in clean water flux and clean water operational permeability for the first membrane module applied to it.

4.2.2 Membrane Resistance Coefficients

The membrane resistance coefficients were calculated using the resistance in series model for each membrane module. The membrane resistance coefficients for each membrane module may be viewed in Table 9 and Table 10, and the resistance in series model equation may be viewed in Equation 2. The membrane resistance coefficients are important to calculate for a particular membrane module as they provide an indicator on which type of fouling resistance the membrane is being exposed to primarily. The membrane resistance coefficient, $K_M$, is the initial, natural resistance of the membrane.
module prior to exposure to mixed-liquor, or filtration processes. This value is found through filtering clean water of a known temperature through the membrane module at monitored TMP levels, and measuring the resultant membrane flux. The reversible fouling coefficient, $K_R$, is resistance to membrane filtration contributed from foulants that may be removed through regular physical and chemical cleaning processes.

Generally, over the course of membrane filtration operation, $K_R$ gradually increases as regular physical cleaning processes are used to remove it. Eventually, these physical cleaning processes alone cannot remove a sufficient quantity of the reversible membrane fouling, and a chemical cleaning is performed to improve the amount of reversible fouling being removed. Thus, $K_R$ may be calculated through comparing the total membrane resistance just prior to a chemical cleaning to the total membrane resistance following a chemical cleaning, where the quantity of membrane resistance removed from the cleaning was contributed from reversible membrane fouling. The irreversible fouling coefficient, $K_{IR}$, is resistance to membrane filtration contributed from foulants that, generally, may not be removed through the regular physical and chemical cleaning processes. Generally, over the course of membrane filtration operation $K_{IR}$ increases gradually as well, but cannot be reduced unless the regular cleaning processes already set in place are improved.

<table>
<thead>
<tr>
<th>Table 9: Membrane ($K_M$) and irreversible fouling ($K_{IR}$) membrane resistance coefficients for membrane 1.</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_M$ ($/m \times 10^{12}$)</td>
</tr>
<tr>
<td>-----------------------------------------------</td>
</tr>
<tr>
<td>2.27</td>
</tr>
</tbody>
</table>
Table 10: Membrane, irreversible fouling, and reversible fouling (K_R) membrane resistance coefficients for membrane 2 for each clean. All expressed in units of m⁻¹ x 10¹².

<table>
<thead>
<tr>
<th>KM</th>
<th>Clean 1</th>
<th>Clean 2</th>
<th>Clean 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>K_R</td>
<td>K_IR</td>
<td>K_R</td>
</tr>
<tr>
<td>0.62</td>
<td>4.66</td>
<td>0.73</td>
<td>9.33</td>
</tr>
<tr>
<td></td>
<td>K_R</td>
<td>K_IR</td>
<td>K_R</td>
</tr>
<tr>
<td>0.62</td>
<td>18.7</td>
<td>1.45</td>
<td>6.27</td>
</tr>
</tbody>
</table>

The resistance in series model may be viewed in Equation 2, and a sample calculation applying this equation to experimental data may be viewed in Appendix B. This model was adopted and developed from other studies (Ruigomez et al. 2017).

\[
J = \frac{\Delta P}{\mu (K_M + K_{IR} + K_R)}
\]  

(2)

where \( J \) is the permeate flux (m³/m²•s), \( \Delta P \) is the transmembrane pressure (Pa), \( \mu \) is the dynamic viscosity of the permeate (Pa•s), \( K_M \) is the membrane resistance coefficient (m⁻¹), \( K_{IR} \) is the irreversible fouling resistance coefficient (m⁻¹), and \( K_R \) is the reversible fouling resistance coefficient (m⁻¹).

Typically for two membrane modules created by the same manufacturer similar membrane resistance coefficients would be anticipated, ensuring consistency. Comparing the \( K_m \) of membranes 1 and 2, a large discrepancy may be noted between the two values. The membrane resistance coefficient of membrane 1 was 3.66 times larger than the membrane resistance coefficient of membrane module 2. There was a difference in how the clean water tests were conducted on membranes 1 and 2 as the first membrane module had the entire CSTR filled with water recirculated through it, whereas the second membrane module had only the isolated cleaning loop filled with
clean water for recirculation. This difference may be a major cause of the observed discrepancy.

The irreversible and reversible fouling membrane coefficients were calculated for membrane 2 following each chemical cleaning. Generally it is expected that the reversible and irreversible membrane resistance coefficients increase over time as a membrane module operates. This trend is anticipated since cake layer compaction on the membrane surface, membrane age, and potential damage to the membrane would all contribute towards these resistance terms as the membrane operated over an extended period of time. The membrane module followed this trend until its fourth chemical cleaning, which took place following 48 days of operation without a chemically enhanced backwash cycle in place, and 72 days of operation with a chemically enhanced backwash in place (42 of the days with CEBs in place being runs 5 and 6). This was the first membrane cleaning following a period of filtration where chemically enhanced backwashes took place, and instead of the $K_{ir}$ value continuing to increase, it decreased. This fourth membrane cleaning followed an identical methodology to the second and third membrane cleanings, yet the membrane module was able to remove 44% of its irreversible fouling, compared to clean 3, through this membrane cleaning. It is possible that the constant contact with a low concentration caustic solution, supplied through the chemically enhanced backwashes, was able to keep the pores and surface of the membrane module free of built-up, irreversible membrane fouling more effectively than not having the chemically enhanced backwash in place, and was thus the reason for this improvement in membrane resistance removal.

This phenomenon is further justified through examining the irreversible and reversible membrane resistance coefficients following the fifth membrane cleaning.
Again, this membrane cleaning had an identical methodology to both membrane cleanings 2 – 4, yet it was able to further reduce the $K_{IR}$ value by 9%, and reduce the $K_R$ value by 67%. In the period of operation leading into this membrane cleaning chemically enhanced backwash cycles were present for the entire length of time. The chemically enhanced backwash cycles potentially remove membrane foulants more efficiently than adjustments in permeate backwash duration, frequency, or flow rate, thus making chemical membrane cleanings that take place during their operation appear much more effective.

The sixth chemical membrane cleaning followed a different methodology than the third, fourth, or fifth. This cleaning applied the intensive cleaning procedure recommended by the membrane manufacturer, which was composed of three parts: an initial caustic clean with 1% NaOH, an acidic clean with 1% citric acid, and a secondary caustic clean with 1% NaOH and 1% hypochlorite, all with backwashing cycles integrated. The $K_R$ values between cleans 5 and 6 were similar, however the $K_{IR}$ value increased following the sixth membrane cleaning. As this intensive cleaning procedure is thought to be superior to a regular caustic cleaning such as cleans 3 – 5 by the manufacturer, this increase in the $K_{IR}$ value was not anticipated.

The irreversible fouling resistance coefficient was calculated for each part of the final chemical cleaning, in an effort to compare the effectiveness of each part of the intensive cleaning process. The results from these calculations may be viewed in Table 11.
Table 11: Membrane, irreversible fouling, and reversible fouling membrane resistance coefficients for each part of chemical clean 6 for membrane 2. All expressed in units of $\text{m}^{-1} \times 10^{12}$.

<table>
<thead>
<tr>
<th>$K_M$</th>
<th>$K_R$</th>
<th>Part 1</th>
<th>Part 2</th>
<th>Part 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$K_{IR}$</td>
<td>$K_{IR}$</td>
<td>$K_{IR}$</td>
</tr>
<tr>
<td>0.62</td>
<td>6.34</td>
<td>6.84</td>
<td>9.09</td>
<td>3.72</td>
</tr>
</tbody>
</table>

Following the acidic portion of the intensive cleaning the $K_{IR}$ value increased by 33%. Acidic cleans are principally used to remove inorganic membrane fouling, which most likely would not have been present in this situation due to the nature of the feed wastewater. Therefore, theoretically, the acidic portion of the clean should have kept the $K_{IR}$ value approximately the same as prior to its use. This was not the case, as the $K_{IR}$ value increased, indicating more irreversible membrane resistance, and meaning the acidic portion of the clean may have damaged the membrane surface or its pores. After the secondary caustic cleaning was applied to the membrane during part three of the intensive clean the $K_{IR}$ value decreased by 59%, indicating that the caustic clean was much more effective in removing irreversible membrane fouling.

4.2.3 Chemical Cleanings Performance Results

For the first membrane module the effectiveness of each chemical cleaning methodology was evaluated according to its ability to recover clean water flux and clean water operational permeability. The second membrane module had the ability to be placed back inline following each chemical cleaning, therefore each chemical cleaning methodology applied to the second membrane module had its performance evaluated in terms of both clean water flux and operational permeability recovery and actual flux and operational permeability recovery.
4.2.3.1 Recovery in Clean Water Flux

The average clean water flux recovery efficiency for each chemical cleaning methodology applied to the first membrane module may be viewed in Figure 22.

![Diagram showing average clean water flux recovery efficiency](image)

**Figure 22: Average clean water flux recovery following each clean for membrane 1.**

The first chemical cleaning applied to membrane module 1 offered the only substantial recovery in clean water flux, with a recovery of 60%. Chemical cleaning 1 consisted of a mixed 1% hypochlorite and 1% sodium hydroxide solution recirculated through the membrane module for 30 min, allowed to soak for 1h, and then recirculated once again for 30 min. It is likely that the caustic solutions were successfully able to weaken the bonds between foulant materials and the membrane surface. Rinsing with tap water following the completion of the chemical cleaning was able to remove the loosened foulants from the membrane module.

The second chemical cleaning methodology applied to this membrane module consisted of a 10% citric acid solution recirculated through the membrane module for 30
min, allowed to soak for 1 h, and then recirculated for 30 min before draining. Applying this acidic cleaning on the day following the first cleaning lead to a decline in membrane flux recovery, decreasing to 12% of the membrane’s initial clean water flux. Citric acid is generally used to remove inorganic fouling, and therefore likely would not have provided much more flux recovery than the prior caustic clean, however such a drastic decline in flux recovery was not anticipated. The concentration of citric acid may have been sufficiently high that it harmed the membrane surface, and resulted in the decline in chemical cleaning efficiency. Vera et al. 2014 used a combined 0.05% sodium hypochlorite, 0.6% citric acid, and 0.05% sodium hypochlorite chemical cleaning on a PVDF hollow-fibre membrane and found that the citric acid portion of the cleaning did remove a portion of the membrane fouling, leading to an increased recovery.

Following the citric acid cleaning, in an effort to recover more clean water flux, an intensive clean, followed by another citric acid clean, and finally a last hypochlorite clean were applied offering clean water flux recoveries of 16%, 13%, and 12%, respectively. The methodologies for all of these chemical cleanings may be viewed in Table 5. The intensive cleaning, which incorporated hypochlorite, sodium hydroxide, and citric acid cleaning phases as well as backwashing processes offered a slight recovery over the citric acid cleaning, which made it a potentially viable cleaning alternative to apply to the second membrane module. The second citric acid clean, this time with backwashing cycles integrated, resulted in a decline in clean water flux recovery once again.

The average clean water operational permeability recoveries for each chemical cleaning methodology applied to the first membrane module may be viewed in Figure 23.
A similar trend occurred when examining the operational permeability recovery results whereby the first clean was the most effective in average clean water permeability recovery (average recovery of 60%), the second clean resulted in a drastic decline in clean water operational permeability (average recovery of 12%), and the third clean did recover a small portion of operational permeability (average recovery of 15%). From these results it was concluded that clean 1 and clean 3 conditions would be applied to the second membrane module when chemical cleaning was required, as run 1 offered a good initial average clean water flux and average clean water operational permeability recovery. Clean 3 showed a promising chemical cleaning alternative, as it was able to increase the membrane’s clean water flux recovery following the 10% citric clean.

The average clean water flux recovery for each chemical cleaning methodology applied to the second membrane module may be viewed in Figure 24.
In terms of clean water flux recovery, cleans 1, 5, and 4 offered the largest recovery efficiencies with 53%, 34%, and 30%, respectively. Clean 1 was the first chemical cleaning applied to the membrane module, therefore it was anticipated to offer the largest efficiency in clean water flux recovery. Clean 1 was a three-cycle clean consisting of an initial 1% sodium hydroxide clean with backwashing and relaxation processes included, a 1% citric acid clean under the same hydraulic conditions, and a 1% sodium hydroxide and 1% hypochlorite clean under the same hydraulic conditions. The caustic solutions would have removed organic membrane fouling, and the citric acid would have removed any inorganic fouling, with the backwashing processes assisting in removing pore-blocking foulants. This first cleaning may have been highly efficient in removing membrane fouling because it was completed within one week of the membrane’s installation. With a small period of contact time allowed between potential...
foulants and the membrane surface, their bonds would have been much weaker than if the same foulants were allowed a contact time 10 – 15 times longer.

Similar to the trends in membrane resistance coefficients, as a membrane module is operated for a longer period of time its recoverable clean water flux is anticipated to either remain at a similar level or decrease under consistent operating conditions. This was the case until membrane cleanings 4 and 5 took place. For 60% of the time leading into the fourth chemical cleaning a chemically enhanced backwash cycle was in place during regular membrane backwashing protocols. Regular contact between caustic chemicals and organic foulants would have likely kept their bonds weak and allowed a reduced level of accumulation on the membrane surface and in its pores. In turn, when a chemical cleaning was deemed necessary these weaker bonds between foulants and membrane surface resulted in the foulants being removed more effectively, resulting in an overall more efficient chemical cleaning. Similarly, Zhang et al. 2007 concluded when operating a tubular polyethersulphone side stream AnMBR for the treatment of swine manure that long term operation without chemical cleaning resulted in residual membrane fouling that was difficult to remove with the chemicals used in their study (HNO₃ and EDTA). They concluded that weekly versus monthly membrane cleanings provided no large benefits, attributing it to the fact that irreversible membrane fouling occurs very rapidly. Perhaps under the conditions of this study, where low-strength daily chemical cleanings took place through the chemically enhanced backwash cycles, this irreversible fouling was unable to accumulate, thus resulting in an increase in clean water flux recovery as the membrane aged.

In the period of time leading into the fifth chemical cleaning a chemically enhanced backwash was in place for 100% of the time. Similar to the above mechanism,
this likely resulted in much weaker bonds and no ability for instantaneous irreversible fouling accumulation (due to regular chemically enhanced backwashes being in place directly following the chemical cleaning), thus a more efficient cleaning overall.

Membrane cleanings 2 – 5 all followed the same cleaning methodology, yet the fourth and fifth successive cleanings were the second most and most efficient in clean water flux recovery efficiency, respectively, other than the first membrane cleaning.

The sixth chemical cleaning consisted of the intensive cleaning procedure, and resulted in a clean water flux recovery of only 14%. Chemically enhanced backwash procedures were in place for 100% of the time leading into this chemical cleaning. Compared to the 34% clean water flux recovery offered by the fifth chemical cleaning with chemically enhanced backwash procedures in place 100% of the time leading into it, it would appear that the intensive cleaning process is not as effective as the chemical cleaning methodology applied in cleans 2 – 5. The larger clean water flux recovery observed after the first intensive clean (clean 1) may have been primarily due to the short duration of operation prior to the cleaning (10 days) and not the actual mechanisms that the cleaning procedure utilizes. The membrane module was operated for 74 days prior to clean 5 and 28 days prior to clean 6, meaning there was a much larger opportunity for irreversible fouling to accumulate prior to clean 5, yet this clean was still 2.4 times more efficient in recovering clean water flux than clean 6.

Since clean 6 consisted of three parts, as mentioned previously, its clean water flux was measured following each part in an effort to gauge where the inefficiencies in clean water flux recovery may be present. The clean water flux recovery following each part of clean 6 may be viewed in Figure 25.
Figure 25: Average clean water flux recovery following each part of clean 6 for membrane 2.

The first part of the chemical cleaning was able to recover 9% of clean water flux, the second part dropped the clean water flux to 6%, and the final part recovered clean water flux to 14% of the control. Overall, it appears that the citric acid portion of the clean may have damaged the membrane surface as this portion of the clean decreased the quantity of recovery. This trend is similar to when a 10% citric acid solution came into contact through chemical cleaning with the first membrane module. The caustic portion of the clean was then able to improve the overall clean water flux recovery, demonstrating once again that a 1% NaOH and 1% hypochlorite chemical cleaning solution is the most effective means of chemical cleaning in terms of clean water flux recovery.

As a secondary means of comparison, the clean water operational permeability recovery efficiency was calculated following each of the chemical cleaning
methodologies applied to the second membrane module, and the results may be viewed in Figure 26.

![Average Clean Water Operational Permeability Recovery Efficiency (%)](image)

Figure 26: Average clean water operational permeability recovery following each clean for membrane 2.

The clean water operational permeability recoveries for each chemical cleaning followed a near identical trend to the clean water flux recoveries. Cleans 1, 5, and 4 offered the largest clean water operational permeability recoveries with recovery efficiencies of 49%, 31%, and 30%, respectively. The only exception was that the clean water operational permeability recoveries following cleans 4 and 5 were much closer, being within 1% of one another, whereas their flux recoveries differed by 4%. Clean water operational permeability recovery factors TMP into the clean water flux recovery calculation, therefore the fact that the overall trend in clean water operational permeability recovery efficiency follows that of the clean water flux recovery indicates that TMP in the clean water tests was not influenced significantly by the cleaning protocol. The variances in clean water flux recovery values would describe the
differences in clean water operational permeability recovery values, and TMP remained fairly consistent.

4.2.3.2 Recovery in Actual Flux

Overall, all of the clean water flux and operational permeability recovery efficiencies were quite low (in the 14% – 53% range). To further compare recovery efficiencies, for the second membrane module, the average actual flux and actual operational permeability recovery efficiencies were calculated. The values for average actual flux recovery efficiency following each membrane cleaning for the second membrane module may be viewed in Figure 27.

![Figure 27: Average actual flux recovery following each clean for membrane 2.](image)

The recovery efficiencies for actual flux were much greater than those of the clean water flux test, with average flux recovery efficiencies in the range of 67% - 100%. The largest actual flux recoveries were, however, following cleans 1, 4, and 5,
with average actual flux recovery efficiencies of 100%, 84%, and 84%, respectively. As mentioned previously, the reason for the first clean achieving 100% removal efficiency may be due to it occurring only 10 days after the membrane module was placed inline. This short time span did not allow irreversible fouling to develop, resulting in an increased cleaning efficiency. Cleans 4 and 5 performed the second best in terms of average actual flux recovery efficiency. This performance may be attributed to them having ongoing chemically enhanced backwashes prior to the cleaning process, which would have resulted in weaker bonds between organic foulants and the membrane’s surface and pores. The sixth clean did, however, pull ahead of the second and third cleans in terms of average actual flux recovery efficiency. It appeared that the intensive cleaning process, while chemically enhanced backwash processes are in place, might outperform the strictly caustic clean when no chemically enhanced backwashes are in place. If chemically enhanced backwashes are in place, however, the strictly caustic cleaning protocol offered on-par, if not better, performance than the intensive cleaning.

Similar to clean water testing, the average actual operational permeability recovery efficiency was calculated following each chemical cleaning. The results of average actual operational permeability recovery efficiency following each chemical cleaning applied to membrane 2 may be viewed in Figure 28.
When factoring TMP into the calculation, clean 1 still offered the greatest recovery, however clean 3 became on par with cleans 4 – 6. Prior to clean 3 the increased backwash flow rate conditions were in place, and the increased backwash flow rate may have been efficient in alleviating TMP build-up in the membrane pores and on its surface. The increased flow would have been effective in keeping foulants that would normally deposit in the membrane pores or on its surface at a further distance from the membrane module, decreasing their rate of accumulation. Therefore, overall, it would appear that the caustic cleaning protocol was more effective in removing membrane fouling than the intensive clean. If the large recovery values following the first membrane cleaning can be attributed primarily to it only occurring 10 days after placing the membrane inline, then the strictly caustic clean (cleans 2 – 5) outperformed the intensive clean (clean 6) in almost all categories. If there is no regular chemically enhanced backwash cycle in place, and the permeate backwash is not optimized.
(backwash flow rate not maintained at a high level), then the intensive clean would likely offer greater actual flux and operational permeability recovery efficiencies than the strictly caustic clean. However, it may be concluded through this study that chemically enhanced backwashes, or a constant elevated permeate backwash flow rate, should always be in place, if possible, due to their ability to both maintain a higher average net flux, operational permeability, and flux while also synergizing with regular chemical cleanings providing higher recovery efficiencies in clean water and actual flux and operational permeability.
4.3 Complimentary Studies

4.3.1 Background
Over the course Studies 1 and 2 there were three reactor issues that took place, and investigations into their causations were completed. The first study was an investigation into the methanogenic activity of the biomass, in an effort to evaluate whether or not the biomass activity was declining with an increased time of exposure to the membrane module. The second study was an analysis concerning the foaming propensity of the reactor, and operational factors that were influencing the tendency of large foaming events to occur. Finally, the third complimentary study sought to evaluate if a deficiency in certain micronutrients may have been present with the pre-prepared micronutrient solution that was being bulk dosed to the reactor once weekly.

4.3.2 Decline in Methanogenic Activity Study
The decline in methanogenic activity of the biomass study was found through conducting an activity test on the biomass on day one of the project, on day 271 (a few weeks prior to Study 1 commencing), and on day 491 of the project (a few weeks after Study 1 was complete). Each activity test consisted of three respirometry bottles filled with reactor biomass, two of which were fed the same substrate for each test (labeled MA 1 and MA 2, for methanogenic activity), and one of which was not fed and acted as a blank (not pictured – the MA 1 and MA 2 values shown had any biogas produced from the blank subtracted from them). The results from these activity tests may be viewed in Figures 29 – 31.
Figure 29: Methanogenic activity on day 1.

Figure 30: Methanogenic activity on day 271.
Figure 31: Methanogenic activity on day 491.

There was a drastic decline in both the SMA and the average SMP_M of the biomass over the duration of the project. In Figure 29, the small rapid spike in activity 3 hours into the study may be attributed to mechanical noise in the system. The SMA on day one of the study was 0.18 mL-CH_4/g-VSS•d, whereas the SMA on day 271 was 0.17 mL-CH_4/g-VSS•d, and on day 491 of the study was 0.06 mL-CH_4/g-VSS•d. This represents an overall 67% decline in SMA from day 1 to day 491, which is quite a large reduction. The average SMP_M of the biomass on day 1 of the study was 0.08 mL-CH_4/g-VSS•d, whereas the average SMP_M of the biomass on day 271 of the study was 0.07 mL-CH_4/g-VSS•d, and on day 491 of the study was 0.03 mL-CH_4/g-VSS•d, again showing a large decline (63% from day 1 to day 491).

These results align with observed trends in the loading capabilities, the effluent COD, and the biogas production rate of the reactor. Prior to the beginning of Study 1,
the loading capacity of the reactor was determined through applying different OLRs to
the reactor and observing how they affected its effluent COD and biogas production rate.
During this period (days 1-170) OLRs in the range of 4.5 – 6 kg/m³•d could be reached
with COD removal efficiencies remaining well above 99%. Prior to the activity test on
day 491 the reactor was only able to achieve removal efficiencies in the range of 98% -
99% at loading rates less than 2 kg/m³•d, approximately 44 – 67% of its original loading
capacity. Similarly, particularly over the duration of Study 1, there was a decline in
biogas yield, methane yield, and biogas production rate observed.

All of these trends are indicators of a net decline in methanogenic activity, which
is reflected in the results of the activity tests. Therefore, it may be concluded that the
methanogenic activity of the biomass did decrease over the duration of the project. The
principle mechanism causing this decline may be debated, however it has been
hypothesized in other studies using external membrane modules that the shearing forces
surrounding the membrane module harm bacterial flocs, leading to the decline. Since
this system recirculates the reactor contents through the membrane module at a recycle
flow rate of 6000 L/h (resulting in a full reactor turnover every 10 minutes, and a cross
flow velocity of 2.5 m/s) and the net flow area decreases by 67% leading into the reactor
module, providing a location of concentrated shearing forces, it is quite likely that
shearing forces applied to the biomass lead to the decline in methanogenic activity that
was observed.

It should be noted that supplemental sludge was also added over the duration of
the study. In total, 32% of the reactor volume was added in supplemental sludge. This
supplemental sludge was composed of seed sludge and waste sludge produced over the
duration of the study. Its methanogenic activity was not tested, but would be similar to
the methanogenic activity of the sludge on day one of the study. Supplemental sludge addition was deemed necessary when the reactor couldn’t maintain COD removal efficiencies above 98% even at very reduced loading rates (< 2 kg/m³•d). In total, 216 L of this supplemental sludge was added through days 134 – 141 of the study, 30 L of it was added through days 373 – 375, and 70 L of it was added through days 393 – 395. Had this supplemental sludge not been added, the resultant activity of the biomass on day 491 might have been lower than it was observed to be.

4.3.3 Foaming Investigation

The goal of the foaming investigation was to identify which operational parameters contributed the greatest towards the foaming propensity of the reactor, specifically focusing on excessive foaming events. Excessive foaming events were defined as foaming events that were sufficiently large that they caused system shutdown and loading reduction in this pilot-scale system. In total, there were 5 days of excessive foaming observed, and they occurred on operational days 17, 28, 42, 100, and 116.

The specific operational parameters that were analyzed leading into each excessive foaming event were OLR, temperature, total alkalinity concentration, and both influent and effluent ammonia nitrogen concentrations. A number of researchers have remarked that a shock loading in the feed supplied to an anaerobic digester may result in foaming due to an excess of organic compounds not being readily degraded by the bacteria, which then causes an abrupt accumulation of surface active by-products.

For reference to publication/presentation:
(surfactants) (Ganidi et al. 2009). Reactor temperature has been found to have both a positive and negative effect on foaming propensity in an anaerobic digester. With a rapid increase in temperature, both the rate of gas bubble formation, and the rate of conversion of complex substrates to volatile fatty acids and biogas increase rapidly, increasing foaming tendency (Subramanian and Pagilla 2015). If a reactor were operated under thermophilic conditions, and compared to another reactor operating at mesophilic conditions, the thermophilic reactor would have a lower foaming tendency due to its lower viscosity providing better foam drainage and sludge breakup (Ganidi et al. 2009). Reactor sludge alkalinity level has been proposed to be inversely proportional to reactor surface tension, therefore making sludge with a higher alkalinity concentration more surface active, and increasing its foaming propensity (van Niekerk et al. 1987). High ammonia nitrogen concentrations may act as surfactants increasing the foaming propensity of a reactor (Kale and Singh 2016).

In order to evaluate which of these operational factors influenced the foaming propensity of the reactor the most significantly, their percent-change values leading into each excessive foaming event were calculated, and may be viewed in Table 12.

**Table 12: Trends in operational parameters leading into large foaming events.**

<table>
<thead>
<tr>
<th>Day of Excessive Foaming Event</th>
<th>%-Change in Organic Loading Rate (%)</th>
<th>%-Change in Temperature (%)</th>
<th>%-Change in Total Alkalinity (%)</th>
<th>%-Change in Influent NH$_3$-N (%)</th>
<th>%-Change in Effluent NH$_3$-N</th>
</tr>
</thead>
<tbody>
<tr>
<td>17</td>
<td>+ 35%</td>
<td>-</td>
<td>+ 5%</td>
<td>- 39%</td>
<td>-</td>
</tr>
<tr>
<td>28</td>
<td>+ 23%</td>
<td>-</td>
<td>+ 3%</td>
<td>+ 136%</td>
<td>-</td>
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<tr>
<td>42</td>
<td>+ 31%</td>
<td>-</td>
<td>+ 1%</td>
<td>+ 234%</td>
<td>+ 6%</td>
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<tr>
<td>100</td>
<td>- 4%</td>
<td>+ 8%</td>
<td>+ 3%</td>
<td>+ 19%</td>
<td>+ 117%</td>
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<tr>
<td>116</td>
<td>+ 24%</td>
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<td>- 3%</td>
<td>+ 28%</td>
<td>- 4%</td>
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It appeared that a shock increase in OLR had the greatest influence on reactor foaming propensity. Every day of excessive foaming, aside from day 100, had a percentage-increase in OLR greater than 23% exhibited. On day 100, there was a quick increase in reactor temperature, an increase in total alkalinity concentration, and an increase in both influent and effluent ammonia nitrogen concentration. All of these factors are known to increase foaming propensity, and happened simultaneously, which is likely the reason an excessive foaming event occurred on this day. The shock increase in temperature, and the additional volatile fatty acids and biogas formation associated with it, most likely initiated the foaming event, along with the increased alkalinity and ammonia nitrogen concentrations accelerated the event.

To prevent future foaming events from occurring, the following framework was developed and applied to minimize the reactor foaming propensity:

1. Ensure OLR increases on a day-to-day basis are below 23%. A lower increase in OLR would be favourable, if possible.
2. Have cooling mechanisms present for the anaerobic digester to prevent shock increases in temperature.
3. Be cautious of shock increases in ammonia nitrogen present in feed wastewaters. This may be accomplished through maintaining a low hydraulic retention time on feed tanks containing a high pH and a nitrogen concentration that may be readily converted to ammonia nitrogen.
4. When the sludge wastage rate is reduced rapidly, ensure nitrogen and total alkalinity concentrations supplied to the reactor are lowered as well to prevent their accumulation in-situ. For full-scale application, influent wastewater could be diluted
or stripped of a portion of its nitrogen and alkalinity concentrations to ensure they do not accumulate in the reactor. After sludge wastage rate increases once again, the previously removed concentrations may be slowly re-added to new feed wastewater for treatment.

4.3.4 Micronutrient Deficiency Study

The micronutrient solution used to dose the reactor during days 1 – 323 was suspected to be deficient in certain micronutrients. A metal scan revealed that this micronutrient solution was potentially deficient in calcium, iron, magnesium, and zinc, therefore a batch BMP study was conducted to evaluate whether or not supplementing the deficient micronutrients would result in better or quicker methane yields from the biomass. Figures 32 – 34 show the results of the daily BMP, the results of the daily methane production rate, and show the results of the cumulative BMP, respectively.
Figure 32: Micronutrient study daily BMP.

Figure 33: Micronutrient study daily methane production rate.
Blend 1 was the original micronutrient solution dosed at its regular rate, blend 2 was double the original micronutrient solution, and blend 3 was the original micronutrient solution supplemented with other micronutrients as deemed necessary. From Figure 32 it may be viewed that blend 3 produced slightly more daily methane, with an average improvement in BMP of 4% over blend 1, and 3% over blend 2. This is quite a small increase in biogas production rate, indicating that there may have been a slight deficit in required micronutrients.

Blend 3 produced methane at a rate 5% greater than blend 1, however at a rate 4% lower than blend 2. Methane was produced at a quick rate in all three cases, likely consuming the easily digestible sugar fraction of the wastewater, and then decreased to a much lower rate for the remainder of the 24-hour feed cycle. This was most likely a lag phase associated with the additional time required to break down the more complex
starch molecules in the feed wastewater, and allow them to be digested and converted into equivalent methane. The additional calcium, cobalt, iron, magnesium, and zinc supplied through blends 2 and 3 appeared to increase the methane production rate of the biomass, however the supplemental micronutrients supplied in blend 3 provided no additional benefit over the original micronutrient solution.

Similar to the trend in daily BMP, in Figure 34 the increase in cumulative BMP due to blend 3 may be observed. Overall, blend 1 produced 4.38 L-CH₄/g-COD, blend 2 produced 4.43 L-CH₄/g-COD, and blend 3 produced 4.57 L-CH₄/g-COD. Blend 3 provided 4% more methane produced per gram of COD supplied than blend 1, and 3% more methane produced per gram of COD supplied than blend 2. The supplemental micronutrients in blend 3 met minor deficiencies in the biomass, thereby increasing their methane yield. The supplemental micronutrients also increased the methane production rate of the biomass over the initial dosage (blend 1), but did not provide an increase over double the initial dosage (blend 2). Due to these conclusions, for the remainder of the study, the supplemented micronutrient solution (blend 3) was dosed to the reactor.
5.0 Conclusions, Recommendations and Applications to Practice

5.1 Conclusions and Summary

There were a number of conclusions that could be drawn from the data collected during this research project. The research objectives could successfully be answered, and it is anticipated that these conclusions will have good applications to practice. There were some shortcomings observed in the research project, however, and therefore recommendations for future studies are also suggested.

5.1.1 Reaching Research Objectives

The primary research objective of the study was to evaluate the cause and effect relationship between physical cleaning protocol variables (such as permeate backwash duration, frequency, intensity, and the incorporation of CEBs) and membrane performance parameters (TMP, flux, net flux, operational permeability). This objective was successfully met through the performance enhancement study accomplished during days 281 – 445. This study identified that there are cause and effect relationships present between these operational and performance parameters, especially in the case of permeate backwash intensity and the incorporation of CEBs.

A secondary objective of the study was to evaluate the cause and effect relationship between physical cleaning protocol variables and reactor performance (COD removal efficiency, TSS removal efficiency, biogas production rate and yield). It was anticipated that varying the physical cleaning protocols should not hold a large influence over reactor performance, and this was observed to be the case. The AnMBR system held high COD removal efficiencies, very high TSS removal efficiencies, and
high biogas production rates and yields (when compared to theoretical values) throughout the duration of each experimental run within Study 1.

The final primary objective of the study was to evaluate the cause and effect relationship between chemical cleaning protocol variables (type of clean, duration of clean, integration of backwashing processes) and cleaning efficiency (clean water and actual flux and operational permeability recovery efficiencies). This objective was successfully met as an optimal chemical cleaning protocol was identified, and certain other chemical cleaning protocols were deemed detrimental to flux recovery efficiencies.

5.1.2 Membrane Performance Enhancement
Overall, run 5 conditions (a CEB with a duration of 60s every 1d) were able to achieve the highest sustained flux, operational permeability, net flux, and second lowest TMP. This makes run 5 the optimal physical cleaning methodology to apply to the membrane module on a regular basis. Run 4 conditions and run 6 conditions were the second and third best performing physical cleaning methodologies, with run 4 performing better at maintaining a lower TMP and higher operational permeability, while run 6 offered a higher flux and net flux. With both runs 5 and 6 being the overall best performing runs, it may be concluded that a low-strength regular chemically enhanced backwash cycle should be incorporated in regular permeate backwashing cycles, and outperforms increases in the frequency, duration, or intensity of the permeate backwash cycle. If CEBs cannot be utilized, then increasing the intensity of the permeate backwash cycle is a much superior alternative to increasing its duration or frequency.

All of the performance enhancement runs were able to maintain COD removal efficiencies greater than 98% throughout the length of operation. TSS removal
efficiencies were maintained above 99% through this period as well, indicating near 100% solid-liquid separation using the nanofiltration membrane module. Biogas yield and production rate decreased over the length of the study, and was attributed primarily to the shearing forces near the membrane module disrupting the bacterial flocs within the reactor’s mixed-liquor. Even with the decline in biogas yield and production rate, the system was able to maintain biogas production rates in the range of 73% - 86% of theoretical, and methane yields in the range of 71% - 89% of theoretical, indicating good gas yields. A COD balance was conducted over the length of the performance enhancement study, and it was concluded that, on average, only 2% of the COD was unaccounted for.

5.1.3 Chemical Cleaning
For the first membrane module, a 1% hypochlorite and 1% sodium hydroxide chemical cleaning without backwashing processes offered the greatest clean water flux recovery (clean 1). The next best performing chemical cleaning methodology was the intensive cleaning procedure that utilized 3 cleaning cycles and incorporated backwashing (clean 3 applied to membrane module 1). When citric acid was used as the only cleaning chemical clean water flux recovery decreased substantially, and was unrecoverable. Citric acid cleaning was also found to be detrimental in the second membrane module. From these observations, it may be concluded that under the given wastewater conditions and membrane characteristics citric acid should be avoided as a cleaning solution.

The second membrane module had two different chemical cleaning methodologies applied to it. Cleans 1, 5, and 4 offered the largest clean water flux
recoveries at 53%, 34%, and 30%, respectively. They also offered the largest actual flux recoveries with 100%, 84%, and 84%, respectively. The first clean took place only 10 days after the second membrane module was placed inline, and therefore its performance may be exaggerated due to there not being much time provided to allow organic and biological foulants to develop bonds between the membrane surface. Cleans 4 and 5 followed the same methodologies as cleans 2 and 3, yet had much greater performance in terms of clean water and actual flux recoveries. This difference is attributed to a noted synergistic effect between CEBs and regular chemical cleaning processes. It is believed that the CEBs continuously weakened the bonds between organic and biological foulants and the membrane’s surface, making the chemical cleanings following a period of operation much more efficient. Overall, the methodology applied in cleans 2–5 appeared to be the best performing chemical cleaning methodology. Removal of the citric acid portion of cleans 1 and 6 could potentially improve its performance.

5.1.4 Complimentary Studies
There was an observed 67% decline in SMA over 490 days of reactor operation. This was accompanied by a decline in average SMP$_M$ of 63%, and was most likely the reason for loading capabilities of the reactor decreasing, effluent COD gradually increasing, and methane yield gradually decreasing. These declines were noted even after adding 32% of supplemental sludge back into the reactor over its course of operation, meaning the decrease could have been greater had this not been done.

The foaming investigation found that four out of the five major foaming events had an OLR increase greater than 23% leading into them. The other major foaming event did not have an OLR increase leading into it, but did have an increase in effluent
and influent ammonia nitrogen, alkalinity, and temperature leading into it, all parameters thought to increase the foaming propensity of a reactor. For this reason it may be concluded that rapid OLR increases are the principle causation of major foaming events, and should therefore be avoided to prevent them.

The micronutrient deficiency study identified that through supplementing certain micronutrients into the pre-prepared solution methane production rate could be increased by 5%, and daily/cumulative methane produced could be increased by 4%.

5.2 Recommendations

Through Study 1 it was identified that permeate backwash intensity and the incorporation of CEBs within regular permeate backwashing hold the largest influence over sustained membrane flux, TMP, net flux, and operational permeability. Further studies could seek to further optimize these processes, varying backwash intensity gradually over time to view if there is a strongly correlated relationship between backwash flux and membrane flux. Additionally, different concentrations of CEB solution could be tried to examine if a higher concentration, more frequent CEB could offer better performance than the low concentration, less frequent CEB. This would help justify whether contact time between the CEB solution and foulants was the principle reasoning for why the longer duration CEB outperformed the more frequent one.

Further studies surrounding the chemical cleaning protocols could look to run two AnMBRs side by side applying the methodology used for cleans 1 and 6 (applied to membrane module 2) to one membrane module, and the methodology used for cleans 2 – 5 (applied to membrane module 2) to the other. Running new membranes side by side under the same loading and wasting conditions without having other chemical cleaning
processes in place, or interference from other variables (such as CEBs), would help
distinguish which of these two chemical cleaning protocols is the superior alternative in
an environment without CEBs in place. This could again be repeated with CEBs in
place.

Finally, it is apparent through this study and within recent literature that there is a
mechanism within external AnMBR systems that decreases their biomass’ methanogenic
activity relative to submerged AnMBR systems. Further studies could seek to run this
AnMBR configuration side by side with a submerged AnMBR system while conducting
weekly activity tests to examine how methanogenic activity decreases over time and
with increased exposure to shearing forces.

5.3 Applications to Practice

The results collected over the duration of this research project present great potential for
the industry. If a membrane module can incorporate a CEB cycle into its regular
physical cleaning protocols it could potentially offer a large increase in sustainable
membrane flux. A larger sustainable membrane flux in one membrane module would
lead to a decrease in the number of overall membrane modules required for a AnMBR
system, thereby decreasing installation costs significantly. The use of this CEB would
likely offer a much more worthwhile increase in flux than adjusting its permeate
backwash duration or frequency alone, and would recycle less permeate back into the
reactor. If CEBs cannot be incorporated, then operators should focus on maximizing
backwash flow rate instead of the backwash’s frequency or duration.

If a system is treating an entirely organic wastewater, under similar operating
conditions to this study, then a citric acid chemical cleaning may not offer a worthwhile
increase in clean water flux recovery. Under conditions like these, the use of a caustic clean should be prioritized and utilized. It would also appear that longer soak times in membrane cleaning processes may contribute more towards clean water flux recoveries than increased recirculation times.

Finally, this study has again demonstrated that biomass activity tends to decrease in external, cross-flow membrane module operation. The loss of biomass SMA and $\text{SMP}_M$ was quite significant, and in practice supplemental sludge addition would not be very practical. This is a major downfall of external, cross-flow systems, and future research should seek to further investigate what the exact mechanism causing this decrease is.
REFERENCES


Appendix A: COD Balance Sample Calculation

Net COD Balance:

\[ \text{COD}_{\text{in}} = \text{COD}_{\text{out}} \]

where:

\[ \text{COD}_{\text{in}} = \text{COD}_{\text{influent}} + \text{COD}_{\text{accumulated}} \]

\[ \text{COD}_{\text{out}} = \text{COD}_{\text{effluent}} + \text{COD}_{\text{biogas}} + \text{COD}_{\text{WANS}} + \text{COD}_{\text{cell}} \]

where:

\[ \text{COD}_{\text{influent}} = \text{Daily influent COD supplied to the reactor, g/d}; \]

\[ \text{COD}_{\text{accumulated}} = \text{Daily COD accumulated in the reactor, g/d}; \]

\[ \text{COD}_{\text{effluent}} = \text{Daily effluent COD removed from the reactor, g/d}; \]

\[ \text{COD}_{\text{biogas}} = \text{Daily COD converted to methane, g/d}; \]

\[ \text{COD}_{\text{WANS}} = \text{Daily COD removed from the reactor as WANS, g/d}; \]

\[ \text{COD}_{\text{cell}} = \text{Daily COD consumed through cell reproduction, g/d}. \]

where:

\[ \text{COD}_{\text{influent}} = Q_{\text{inf}} \times \text{COD}_{\text{inf}} \]

\[ \text{COD}_{\text{accumulated}} = \frac{(\text{COD}_{\text{supernatant,day164}} - \text{COD}_{\text{supernatant,day1}})}{t_{\text{performance enhancement study}}} \]

\[ \text{COD}_{\text{effluent}} = Q_{\text{eff}} \times \text{COD}_{\text{eff}} \]

\[ \text{COD}_{\text{biogas}} = \frac{C_{\text{CH}_4} \times Q_{\text{biogas}}}{Y_{\text{CH}_4}} \]

\[ \text{COD}_{\text{WANS}} = Q_{\text{WANS}} \times \text{COD}_{\text{supernatant}} \]

\[ \text{COD}_{\text{cell}} = (\text{COD}_{\text{inf}} - \text{COD}_{\text{eff}}) \times 1.42 \times Y_{\text{cell}} \]
Example Calculation for Day 3:

\[ \text{COD}_{\text{influent}} = 37.76 \frac{L}{d} \times 79.9 \frac{g}{L} = 3017 \frac{g}{d} \]

\[ \text{COD}_{\text{accumulated}} = \frac{(22.74 \frac{g}{L} - 9.52 \frac{g}{L}) \times 1000 \frac{L}{164 \text{ d}}}{0.14 \frac{m^3}{10^3 \text{ kg - COD}}} = 75 \frac{g}{d} \]

\[ \text{COD}_{\text{effluent}} = 37.44 \frac{L}{d} \times 0.49 \frac{g}{L} = 18 \frac{g}{d} \]

\[ \text{COD}_{\text{biogas}} = \frac{(55\%) \times 1905 \frac{L}{d}}{0.35 \frac{m^3}{10^3 \text{ kg - COD}}} = 3017 \frac{g}{d} \]

\[ \text{COD}_{\text{WANS}} = 14.80 \frac{L}{d} \times 9.52 \frac{g}{L} = 141 \frac{g}{d} \]

\[ \text{COD}_{\text{cell}} = (79.9 \frac{g}{L} - 0.49 \frac{g}{L}) \times 1.42 \times 0.034 \frac{g - VSS}{g} - \text{COD} = 145 \frac{g}{d} \]

**In Total:**

\[ \text{COD}_{\text{in}} = 3017 \frac{g}{d} + 75 \frac{g}{d} = 3092 \frac{g}{d} \]

\[ \text{COD}_{\text{out}} = 18 \frac{g}{d} + 3017 \frac{g}{d} + 141 \frac{g}{d} + 145 \frac{g}{d} = 3321 \frac{g}{d} \]

\[ \text{COD}_{\text{in}} \text{ out} = \frac{3092 \frac{g}{d}}{3321 \frac{g}{d}} = 93.1\% \text{ COD Accounted for, 6.9\% COD Unaccounted for.} \]
Appendix B: Membrane Resistance Coefficients Sample

Calculation

Resistance in Series Model:

\[ J = \frac{\Delta P}{\mu_{\text{permeate}} \cdot (K_M + K_{IR} + K_R)} \]

Membrane 2 First Clean Water Test:

\[ K_M = \frac{\Delta P}{\mu_{\text{permeate}} \cdot J} = \frac{72534 \text{ Pa}}{7.805 \times 10^{-4} \text{ Pa} \cdot s \cdot 1.508 \times 10^{-4} \text{ m/s}} = 6.16 \times 10^{11} \text{ m}^{-1} \]

repeated for all clean water recirculation flow rates, and averaged.

Membrane 2 Clean Water Test After First Chemical Cleaning:

\[ K_{IR} = \frac{\Delta P}{\mu_{\text{permeate}} \cdot J} - K_M = \frac{77034 \text{ Pa}}{7.337 \times 10^{-4} \text{ Pa} \cdot s \cdot 7.937 \times 10^{-5} \text{ m/s}} - 6.21 \times 10^{11} \text{ m}^{-1} \]

\[ = 7.02 \times 10^{11} \text{ m}^{-1} \]

repeated for all clean water recirculation flow rates, and averaged.

Membrane 2 Clean Water Test Before First Chemical Cleaning:

\[ K_R = \frac{\Delta P}{\mu_{\text{permeate}} \cdot J} - K_M - K_{IR} \]

\[ = \frac{84034 \text{ Pa}}{7.805 \times 10^{-4} \text{ Pa} \cdot s \cdot 1.865 \times 10^{-5} \text{ m/s}} - 6.21 \times 10^{11} \text{ m}^{-1} - 7.29 \times 10^{11} \text{ m}^{-1} \]

\[ = 4.42 \times 10^{12} \text{ m}^{-1} \]

repeated for all clean water recirculation flow rates, and averaged.
Appendix C: Raw Data Plots
Figure 35: Flux and TMP vs. time for all days of operation.
Figure 36: Influent and effluent COD and COD removal efficiency for all days of operation.
Figure 37: Influent flow and OLR vs. time for all days of operation.
Figure 38: Mixed-liquor TSS and VSS vs. time for all days of operation.
Figure 39: Influent, effluent, and mixed-liquor pH vs. time for all days of operation.
Figure 40: Biogas and theoretical biogas production rate vs. time for all days of operation.
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