



SUSTAINABLE ANAEROBIC MEMBRANE BIOREACTOR (ANMBR) TREATMENT OF INDUSTRIAL WASTEWATERS: FOULING CONTROL

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Abstract: An external anaerobic membrane bioreactor (AnMBR) equipped with a nanofiltration tubular membrane module was operated for 170 days treating a synthetic wastewater simulating a high strength industrial wastewater. The organic loading rate supplied to the AnMBR was in the range of 0.8 – 8.2 kg/m³/d, with a mixed-liquor suspended solids (MLSS) concentration in the range of 13.6 – 33.9 g/L. The membrane module was operated for 170 days of continuous data collection, where the loading limitations of the reactor were gauged. Following operation, the membrane module underwent various chemical cleaning processes to alleviate membrane fouling and recover its flux. These successive chemical cleanings applied 5 different methodologies in an effort to analyze their efficiencies in membrane fouling removal. General results from the chemical cleanings concluded that a 1% sodium hydroxide and a 1% sodium hypochlorite solution, without backwashing processes, offered the greatest clean water flux recovery over all other chemical cleaning methodologies. The 10% citric acid cleans were found to decrease recoverable clean water flux. A better understanding of chemical cleaning protocols is required to reduce the quantities of chemical agents used and quantify the relationship between sequential chemical cleanings and reversible fouling removal efficiency. As membrane 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 their success.

1 INTRODUCTION

Sustainable wastewater treatment is important to protect environment and public health. Anaerobic membrane bioreactor (AnMBR) technology offers a promising, sustainable wastewater treatment alternative when it may be applied effectively. 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 AnMBRs' generally lower operation and maintenance costs (due to zero oxygen requirements, 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 have limited AnMBRs' widespread applications is 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). 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 within them may remove both surface and internal pore blockages effectively (Dvorak et al. 2015, Liao et al. 2007, Vera et al. 2014). There have been few comparative studies on different cleaning methods and conditions presented within literature (Ramos et al. 2014 and Zhou 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. This research project compared the efficiency of 5 different chemical cleaning methodologies applied to a tubular nanofiltration membrane module following operation in a novel AnMBR system. The different chemical cleanings had their efficiencies analyzed with regards to their abilities to recover clean water flux and clean water operational permeability, two important operational parameters for AnMBR systems.

2 MATERIALS & METHODS

2.1 Reactor Configuration

The pilot-scale AnMBR 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 ($\pm 0.7^\circ\text{C}$), standard mesophilic conditions, for the duration of all data collection periods by a heat wrap surrounding the reactor. A simplified process flow diagram (PFD) of the experimental setup may be viewed in Figure 1.

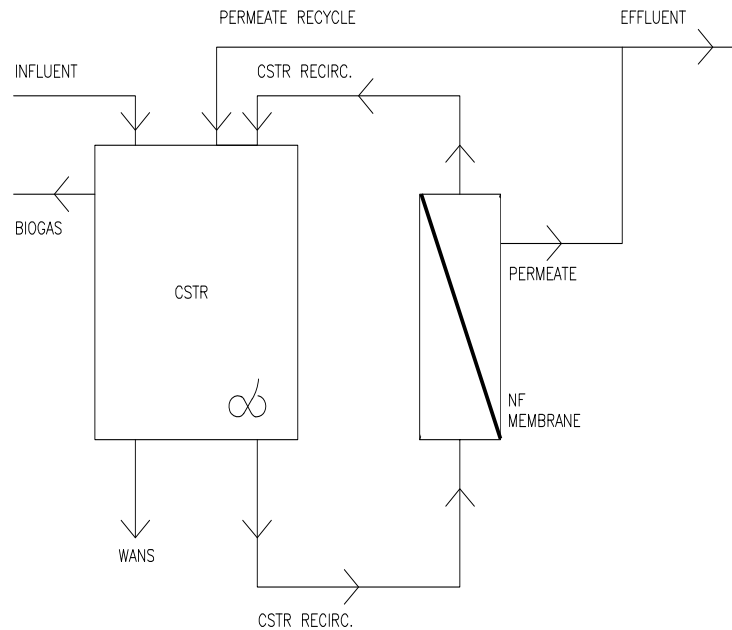


Figure 1: Simplified PFD of reactor configuration.

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 (Masterflex, Ontario) was set to automatically feed the system whenever the tank level fell below a pre-set threshold level. The tank level decreased through effluent flows and waste anaerobic sludge (WANS) flows. WANS was wasted from the system using an automatic peristaltic pump (Masterflex, Ontario) located at the base of the CSTR (set on a 2h 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 (Nemo, New Brunswick), which acted 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. The membrane module was cylindrical in shape, composed of polyvinylidene fluoride (PVDF), with an outer diameter of 50 mm and a total length of 1.436 m. The module contained 13 tubular membrane channels, each 8 mm in diameter, which resulted in a total membrane area of 0.42 m². The membrane surfaces had a mean pore size of 30 nm. The membrane module had backwash functionality present. Backwash functionality being present allowed the membrane module to switch from forward filtration flow to backward flow. This created a backwash that rinsed out the pores of the membrane using filtrate, or permeate.

Before passing into the membrane module, the CSTR mixed-liquor was forced through a stainless steel screen (having a pore size diameter of 0.8 mm, and being present 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 (Masterflex, Ontario) 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 known as permeate, was recycled back into the CSTR through the permeate recycle line or collected for backwashing and effluent flows. Mixed-liquor that was not filtered through the membrane module was recycled back into the CSTR via the CSTR recirculation line.

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 a massflow meter (AALBORG, Orangeburg), where its total mass was measured and logged, before exiting the system. The massflow meter was calibrated with a headspace gas having a composition of 70% methane gas and 30% carbon dioxide gas. There was also a secondary gas trap attached to the system that wasted directly into the environment, in case of emergencies.

2.2 Wastewater Characterization

A potato starch-based wastewater, simulating a high-strength food industry wastewater, was supplied to the reactor through two feed tanks and two respective peristaltic pumps. One feed tank contained sucrose only, accounting for approximately 15% of the total feed volume and approximately 80% of the total feed chemical oxygen demand (COD) (approximately 59 g/L as COD). The second feed tank was continuously mixed and contained waste potato starch obtained from a potato processing plant, macronutrients, and a source of alkalinity. Macronutrient (nitrogen and phosphorous) requirements were calculated using the typical composition of a bacterial cell, $C_5H_7O_2N$, the rate of sludge wastage, and knowing the phosphorous requirement is approximately 1/7th 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 15 g/L as COD). A thorough overview of the wastewater composition may be viewed in Table 1. 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 1: Characteristics of the synthetic industrial wastewater.

Parameter	Unit	Value	
		(Avg.)	(S.D.)
Chemical Oxygen Demand (COD)	g/L	74.3	± 9.7
Biochemical Oxygen Demand (BOD)	g/L	21.5	± 2.8
Total Suspended Solids (TSS)	g/L	6.2	± 2.3
Total Kjeldahl Nitrogen (TKN)	mg/L	630	± 420
Ammonia Nitrogen (NH ₃ -N)	mg/L	256	± 189
Total Phosphorous (TP)	mg/L	210	± 56
Phosphate Phosphorous (PO ₄ -P)	mg/L	222	± 55
Total Alkalinity	g-CaCO ₃ /L	5.1	± 1.7
pH	-	8.75	± 0.35

2.3 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, total suspended solids (TSS) of 21.4 g/L, volatile suspended solids (VSS) of 19.5 g/L, and a specific methanogenic activity (SMA) of 0.18 g-COD_{CH₄}/g-VSS/day.}

2.4 Analytical Methods

COD, TSS, VSS, biochemical oxygen demand (BOD), and total alkalinity were conducted following Standard Methods (APHA 2005). Total phosphorous (TP), total kjeldahl nitrogen (TKN), phosphate-

phosphorous, and ammonia nitrogen were conducted according to HACH Water Analysis Handbook (Hach Company 2002). 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 maintained at a constant temperature of 180°C. 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. Biogas injections were done in triplicates. SMA tests were conducted in duplicates using batch studies operated at 36°C. The tests were conducted in 500 mL serum bottles with a working volume of 450 mL. The bottles were filled with reactor sludge, acetic acid as a substrate, pH buffer, and mineral bases. Young and Cowan outlined the procedures and mixed solutions used (Young and Cowan 2004). All flow and pressure readings were measured once daily at similar times using inline flow meters and pressure sensors.

2.5 Experimental Design

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, first 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 operating the system in forward filtration mode 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 2.

Table 2: Chemical cleaning methodologies for membrane module 1.

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

Clean 4	1	45	N/A	3 and 2 min	10% Citric Acid
	2	N/A	30	N/A	
	3	45	N/A	3 and 2 min	
Clean 5	1	60	N/A	N/A	1% Sodium Hypochlorite
	2	N/A	45	N/A	
	3	60	N/A	N/A	

In Table 2 the recirculation time is the time that the membrane module was operated in forward filtration mode, where the water-chemical mixtures were filtered through the membrane module in the same forward direction as permeate. 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 and TMPs achieved at each of these recirculation flow rates were recorded, and compared back to the control. This allowed the clean water flux and clean water operational permeability recovery of each chemical cleaning methodology to be quantified to identify which chemical cleaning methodology was the most efficient in recovering clean water flux and operational permeability. 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 the ability of each clean to recover a favourably low TMP could be factored into the flux analysis.

3 RESULTS & DISCUSSION

3.1 Recovery of Clean Water Flux

The average clean water flux recovery for each chemical cleaning methodology applied to the membrane module may be viewed in Figure 2.

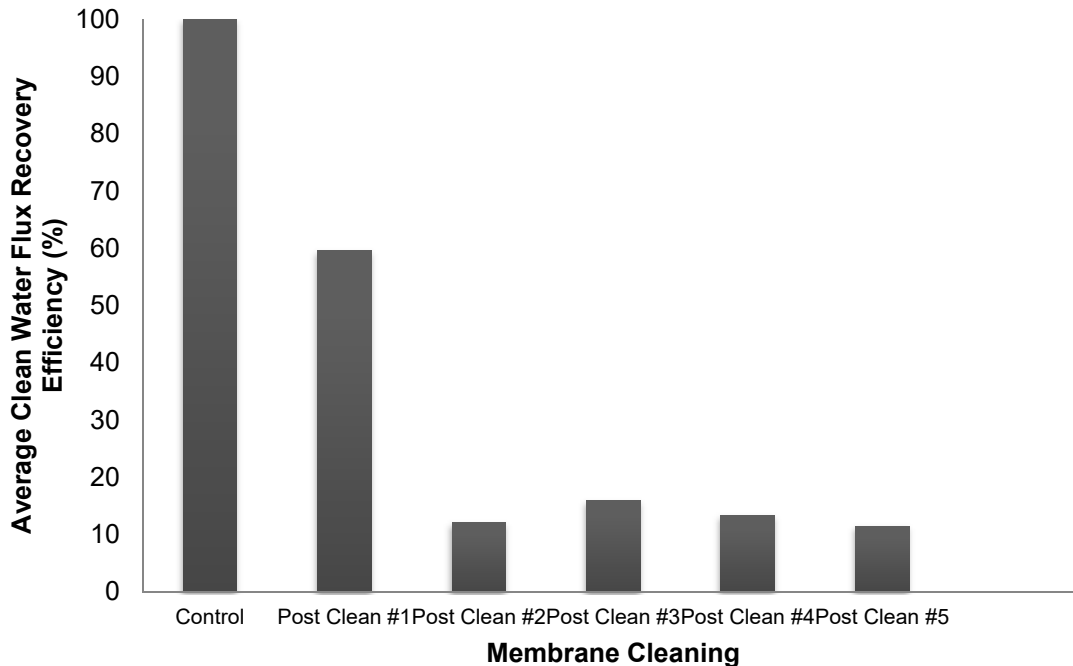


Figure 2: Average clean water flux recovery following each clean for the membrane.

The first chemical cleaning applied to the membrane module offered the only substantial recovery in clean water flux, with a recovery of 60%. Chemical cleaning 1 consisted of a 30 min recirculation phase, a 1h soak, and a second 30 min recirculation phase using a mixed 1% sodium hydroxide and 1% sodium hypochlorite solution. It is likely that the caustic solutions were successfully able to weaken the bonds between foulant materials and the membrane surface. Since the majority of membrane foulants would have most likely been organic in nature (due to a high mixed-liquor volatility of 91% and a high feed wastewater volatility of 99%), sodium hydroxide and sodium hypochlorite would have been highly effective in removing them (Liao et al. 2007). Rinsing with tap water following the completion of the chemical cleaning was then able to remove the loosened foulants from the membrane module effectively.

The second chemical cleaning methodology applied consisted of a 30 min recirculation phase, a 1h soak, and a second 30 min recirculation phase using a 10% citric acid solution. Applying this acidic clean on the day following the first clean lead to a decline in membrane flux recovery, decreasing to 12% of the membranes' initial clean water flux. Citric acid is generally used to remove inorganic fouling, and therefore was not anticipated to provide much more flux recovery than the prior caustic clean, however such a drastic decline in flux recovery was not anticipated. Vera et al. 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 (Vera et al. 2014). Considering that cleans 1 and 2 were identical in methodology other than the chemical solutions used, the concentration of citric acid may have been sufficiently high that it damaged the membrane surface, and resulted in the decline in chemical cleaning efficiency. A surface investigation was unable to be performed following clean 2 as it would have required the membrane module be destroyed, which would not have allowed cleans 3 – 5 to be applied.

Following the citric acid cleaning, in an effort to improve the membrane module's clean water flux recovery cleans 3 – 5 were applied, and offered clean water flux recoveries of 16%, 13%, and 12%, respectively. Clean 3, which incorporated sodium hypochlorite, sodium hydroxide, and citric acid cleaning phases as well as backwashing processes recovered only 4% more clean water flux following clean 2. However, if clean 2 permanently damaged the membrane surface then the ability of this cleaning methodology to offer an improvement in clean water flux, overall, lead the authors to believe it could be a

viable option for future cleaning studies. Nonetheless, comparing the recovery of clean 3 to that of clean 1, it is apparent that clean 1 outperformed clean 3 significantly. Both of these cleans used similar chemical solutions, in similar concentrations, however clean 1 offered longer soak times, and no backwashing processes, making it a more static cleaning procedure, whereas clean 3 incorporated backwashing and had longer recirculation times. Clean 1 offering a significantly greater clean water flux recovery suggests that a static clean is superior to a more dynamic clean under the given operating conditions and membrane characteristics. Recent 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).

Clean 4, incorporating a 10% citric acid solution once again, resulted in a decline in clean water flux recovery from clean 3. This result again suggests that the citric acid solution may have been sufficiently high in concentration that it had a detrimental impact on the membranes' surfaces. Furthermore, clean 4 utilized the same chemical solution as clean 2, however incorporated a longer recirculation phase and backwashing processes. Since clean 4 offered only a 1% increase in clean water flux recovery over clean 2, it may be concluded that shifting the cleaning methodology from a static clean to a dynamic clean offered no worthwhile improvement in clean water flux recovery. It appears that the actual cleaning solution, with preference being given to caustic solutions in this project, holds a greater influence on cleaning performance than the type of cleaning methodology applied.

Finally, clean 5 applied a 1% sodium hypochlorite cleaning solution with a longer recirculation time and shorter soak time compared to clean 1. Additionally, no backwashing processes were incorporated. Following clean 5 the clean water flux recovery was only 12%, making it one of the lowest performing chemical cleans. Assuming the membrane was not permanently damaged following clean 2, clean 5 performing much worse than clean 1 suggests that the 1% sodium hydroxide solution used in clean 1 may have been the cleaning solution that was the most effective in removing membrane foulants, which is why it had the best performance in terms of clean water flux recovery. Clean 3 also utilized 1% sodium hydroxide, and was found to have the second highest clean water flux recovery. Additionally, these results suggest that an increased soak time is superior to an increased recirculation time. The bonds created between the foulants and the membrane surface may require a longer exposure to the cleaning solutions for them to be broken and removed from the membrane surfaces effectively.

3.2 Recovery of Clean Water Operational Permeability

The average clean water operational permeability recoveries for each chemical cleaning methodology applied to the membrane module may be viewed in Figure 3.

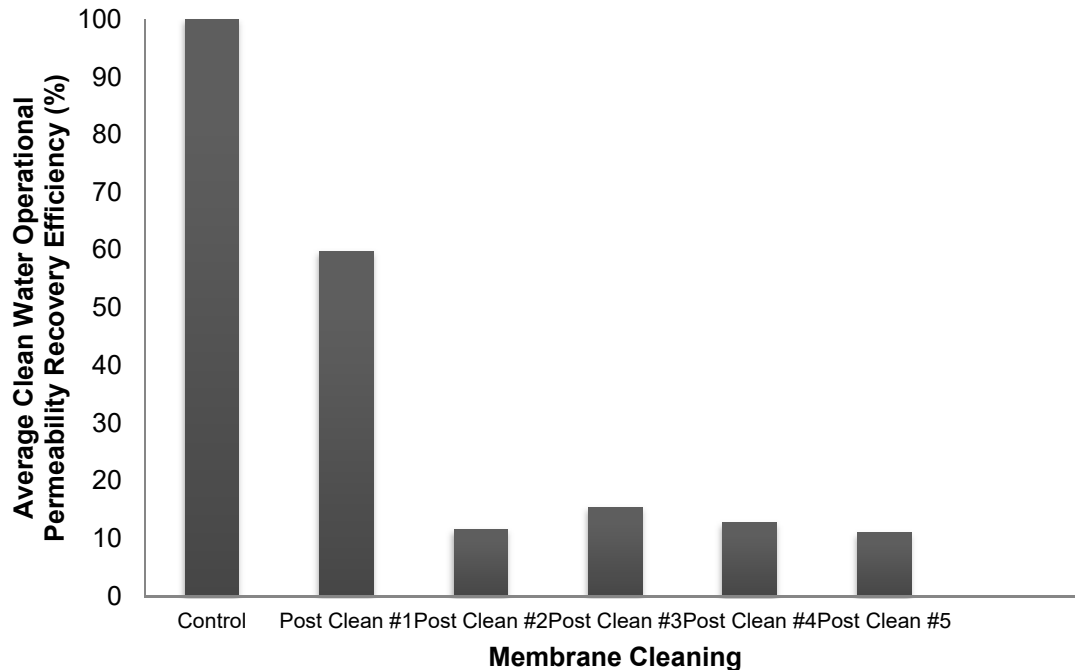


Figure 3: Average clean water operational permeability recovery following each clean for membrane 1.

A similar trend to that of the clean water flux recoveries occurred when examining the operational permeability recovery results whereby clean 1 was the most effective in average clean water permeability recovery (average recovery of 60%), clean 2 resulted in a drastic decline in clean water operational permeability recovery (average recovery of 12%), and clean 3 recovered a small portion of operational permeability (average recovery of 15%). From these results it was concluded that the clean 1 methodology was the most promising, since clean 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 membranes' clean water flux recovery following the 10% citric acid clean. Overall, since all operational permeability recoveries were just as effective as their clean water flux recovery counterparts, it may be concluded that all chemical cleanings were effective in recovering TMP as well.

3.3 Reactor Performance

The AnMBR system operated for 170 days prior to the membrane being taken offline treating organic loading rates (OLRs) in the range of 0.8 – 8.2 kg/m³/d. The OLRs fluctuated greatly during this portion of data collection as the loading capacity of the reactor was being gauged. The mixed-liquor suspended solids fluctuated from a lowest value of 13.6 g/L to a highest value of 33.9 g/L. Even through these large fluctuations in OLR the AnMBR system was able to achieve an average COD removal efficiency of 99.5% (\pm 0.6%), indicating very good COD removal rates. The TSS removal efficiency remained above 99% throughout the entirety of operation, indicating near 100% solid-liquid separation achieved by the membrane module. The average methane yield remained close to the theoretical value of 0.395 m³-CH₄/kg-COD throughout this period of operation as well, which signified that the majority of COD removed through biological processes was converted to biogas effectively.

4 CONCLUSIONS

Through this study an AnMBR system integrated with an external nanofiltration tubular membrane module was able to effectively treat a synthetic wastewater simulating a high-strength food processing

wastewater. Several chemical cleaning methodologies were applied to the fouled membrane module following 170 days of continuous operation. A summary of the major conclusions is provided:

- A primarily static chemical cleaning using a mixed 1% sodium hydroxide and 1% sodium hypochlorite solution was able to achieve the highest clean water flux and clean water operational permeability recovery efficiencies.
- A chemical cleaning using a 10% citric acid cleaning solution may cause permanent damage to the membranes' surfaces. Even if permanent damage was not done, the membrane module's clean water flux recovery efficiency dropped by 48% between cleans after being exposed to the citric acid cleaning solution, and was not able to recover more than 16% clean water flux after exposure.
- For chemical cleanings, in general, it appears that a higher soak time is more favourable than a higher recirculation time or the incorporation of backwashing processes.
- Overall, it appears that the 1% sodium hydroxide cleaning solution was the most effective cleaning solution for removing membrane foulants.

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