



BREWERY WASTEWATER TREATMENT BY ANAEROBIC MEMBRANE BIOREACTOR

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Abstract: The micro-brewery business has been subjected to a rapid growth within the last decade. Many brewers are seeking reliable on-site treatment methods because of expensive wastewater compliance bills. Anaerobic membrane bioreactors (AnMBR) can achieve a high quality effluent that is free of suspended solids and exhibit a great performance stability under loading shocks. To date, there have been limited studies on the performance of AnMBRs for brewery wastewater. In this study, a state-of-the-art AnMBR lab-scale system (supplied by GE Water and Process Technologies) was used to investigate the performance and capacity of AnMBR for the treatment of brewery wastewater. The system was subjected to a 230 days of continuous operation with both synthetic and real brewery wastewater at organic loading rates (OLRs), ranging from 3.5 to 11.5 kg COD/m³/day. During all tested OLRs, the AnMBR system demonstrated an excellent COD removal efficiency (>98%) and produced an effluent with a less than 300 mg/L COD concentration and a stable methane yield at 0.31 m³ CH₄/kg COD (35 °C). While the system demonstrated an excellent biological performance, it was found that the efficiency of the AnMBR was limited by membrane fouling, which reduced the membrane permeability and caused an unstable membrane filtration performance. The results showed that AnMBRs should be operated at an OLR below 6.5 kg COD/m³/day and a filtration flux of 7 L/m²/hour (LMH) to achieve a long-term stable membrane filtration for the treatment of brewery wastewater.

1 Introduction

Over the last decade, the structure of the brewery industry has changed in Canada. Large operation plants have reduced in number while the number of micro-breweries has increased considerably (Bureau, 2009). There are 47 micro-breweries in Ontario, which have approximately \$190 million sales in 2010, equivalent to a 5% share of the Ontario beer market (MEDEI, 2012). Beer brewing is a highly water intensive process. An average of about 7 litres of water is used to produce one litre of beer and about 70% of the intake water of a micro-brewery is discharged to the sewer (Brewers Association, n.d.). With the large growth in business, discharging brewery wastewater into the sewer will become very expensive since most of municipalities in Canada have set up the maximum permissible limits for the main contaminant contents in the wastewater discharged in to the sanitary sewer system and discharging wastewater over these limits will bring about a surcharge (Natural Resources Canada, 2012). The high surcharge caused by discharging high strength brewery wastewater has limited the growth of the micro-brewery industry.

Wastewater characteristics vary significantly among different breweries, as well as from day to day operation. According to Driessen and Verejken (2003), a typical brewery wastewater consists of 200-1000 mg/L of total suspended solids (TSS), 2000-6000 mg/L of COD, 25-80 mg/L of nitrogen and 10-50 mg/L of phosphorous with a BOD to COD ratio of 0.6 to 0.7. The organic contents of brewery wastewater are mainly easily biodegradable sugar, starch, ethanol and volatile fatty acids (VFAs). Instead of relying on the municipalities treating brewery wastewater aerobically, the anaerobic on-site treatment has become a more attractive option for micro-brewery. The advantages of the anaerobic treatment include a lower sludge production, smaller footprint, and, more importantly, the production of biogas for the energy recovery. In fact, the anaerobic treatment is already a proven technology for brewery wastewater treatment with the upflow anaerobic sludge blanket (UASB) being widely accepted by the industry (Brito et al., 2007). Anaerobic membrane bioreactors (AnMBRs), which is a combination of the anaerobic reactor and membrane filtration, are an advanced high rate anaerobic wastewater treatment technology. With the integration of ultrafiltration (UF) membrane filtration with the anaerobic digestion, the AnMBRs can entirely retain the biomass and solids in the system, thus offering a complete separation between the solid retention time (SRT) and hydraulic retention time (HRT) as well as a high tolerance to pH and organic loading shocks (Dereli et al., 2012).

Although the AnMBR technology is more industrialized in some European countries, it is still in the developing stage in other parts of the world. Recently, General Electric has announced its first full-scale AnMBR installation at Dogfish Head Craft Brewery in Milton, DE to reuse the brewery wastewater, where the system is expected to reduce the wastewater volume by 80% (GE Water & Process Technologies, 2014).

In our previous study, the performance of a state-of-the-art AnMBR system was evaluated earlier using synthetic brewery wastewater adopted from Scampini (2010), with a COD:N:P ratio of approximately 260:4:1. The system had gone through an operation over various organic loading rates (OLRs) ranging from 2 to 10 kg COD/m³/day and sustained a long term stable operation over 10 kg/m³/day at 11 ± 1 g/L MLSS with observed SRT of 31 days. The AnMBR had a quick start-up and demonstrated a high tolerance to the changes in wastewater organic strength. Overall, the system achieved over a 99% COD removal efficiency and excessive methane yield at 0.38 m³ CH₄/kg COD with minor membrane fouling observed. In this study, the efficacy of AnMBR technology was further investigated using high strength brewery wastewater sampled from a local craft brewery. The system was subjected to an operation with the higher organic loading fluctuation and more complex wastewater composition. In addition, this study examined the membrane fouling mechanism under a low biomass concentration, to evaluate whether if the low biomass operation is more practical for AnMBRs.

2 Materials and Methods

2.1 AnMBR System

As shown in Figure 1, the lab-scale AnMBR used in this study consisted of a complete mix anaerobic digester and a membrane tank. Continuous mixing was performed in the anaerobic reactor using a mechanical mixer (Eurostar, IKA). The mixed liquor was recirculated from the anaerobic reactor to the bottom of the membrane tank and overflowed from the top portion of the membrane tank back to the reactor. A submerged hollow fibre membrane module with a total surface area of 0.047 m² (GE Water and Process Technologies, Oakville ON) was used for liquid-solid separation. A peristaltic pump (Miniplus 3, Gilson) was installed to extract permeate at a constant flux and the biogas was recirculated to the membrane tank for membrane scouring. Pressure sensors (Cerabar T, Endress Hauser) were installed on the digester tank and permeate line to monitor the system pressure and transmembrane pressure (TMP), respectively. Level sensors were used to control the feed pump to maintain a work volume of 15 L in the system. The bioreactor temperature was maintained by recirculating hot water in the water jacket. The system pH was controlled by dosing sodium bicarbonate with a diaphragm metering pump (Stepdos 08, KNF). Biogas production was measured on-line by a mass flow meter (Burkert 8700). A PLC system was used to control the AnMBR system and for the collection of operation data.

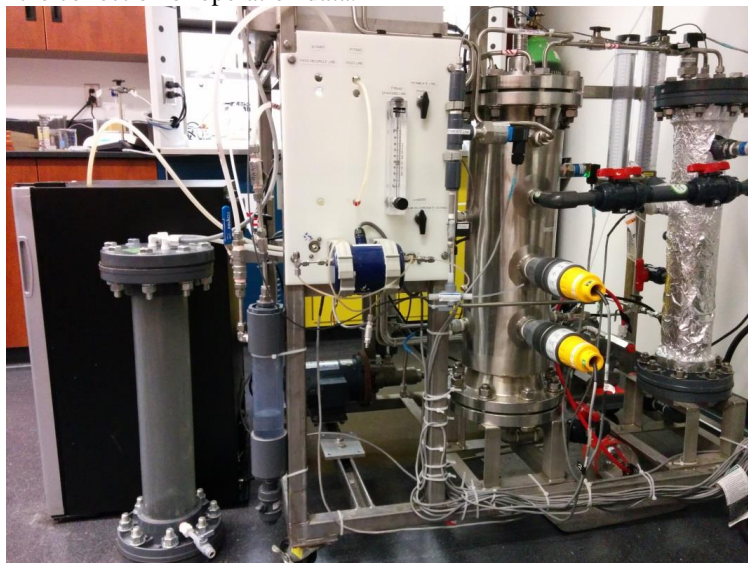


Figure 1 AnMBR system showing digester (centre) and membrane tank (right)



2.2 Wastewater Sampling

This study used high strength brewery wastewater from a local craft brewery. Weekly sampling of 4 x 20 litre wastewater was conducted. The 20 L container was placed in a bar refrigerator (6 °C) and was directly connected to an AnMBR feed pump. Excess wastewater was stored in a cooler room (4 °C) prior to use during the week. Characteristics of each batch were tested and will be further discussed in the results section.

2.3 AnMBR Operation

The reactor was started in an active state inherited from the previous synthetic brewery wastewater study, with the initial mixed liquor suspended solids (MLSS) and mixed liquor volatile suspended solids (MLVSS) concentrations of 11800 mg/L and 10200 mg/L, respectively. The reactor had gone through 90 days of continuous operation at a constant HRT of 44 hours. The OLR varied from 3.5 to 11.5 kg/m³/day and a target active biomass concentration of 7000 mg/L MLVSS was maintained by changing the daily sludge wasting volume. An average SRT was then calculated for each of the OLR conditions, shown in Table 1.

Table 1 Operation conditions during the study

Days	OLR (g/L/d)	SRT (d)
0-7	6.5	30
8-12	11.5	17
13-29	6.5	30
30-50	3.5	46
51-90	5.0	50

The AnMBR was operated at pH 7.02 ± 0.01 and temperature of 35.5 ± 0.1 °C. An online alkalinity dosing system was used to maintain the desired pH condition and total alkalinity of the mixed liquor during the operation period. A constant filtration flux was set at 8 LMH and the operation cycle involved 10 minute permeation and 1 minute relaxation throughout the entire study. Weekly online maintenance cleaning was conducted using 2000 mg/L citric acid and 2000 mg/L sodium hypochlorite. The maintenance cleaning operation involved a 15 minute backwash cycle at the permeation flux with each of the cleaning reagents.

2.4 Analytical Methods

Samples were regularly taken from the digester, membrane tank and membrane permeate for analysis. The supernatant of the mixed liquor was obtained using a centrifugation/filtration procedure with centrifugation at 8000 rpm for 15 min at 4 °C followed by filtration through a 1.5 µm filter paper, and the soluble samples were further filtered through 0.45µm disposable filters.

The Standard Methods (APHA-AWWA-WEF, 2005) were used for the measurements of total suspended solid (TSS), MLSS and MLVSS. Wastewater parameters including COD, total nitrogen (TN), total phosphorus (TP), ammonia nitrogen (NH₄⁺-N), nitrate nitrogen (NO₃-N), nitrite nitrogen (NO₂-N), phosphate phosphorus (PO₄⁻³-P), and volatile fatty acids (VFA) were measured using the Hach methods (HACH, 2008). Protein and polysaccharide concentrations of EPS and EPS were determined using the Bradford method (Bradford, 1976) and phenol-sulphuric acid method (DuBois et al., 1956), respectively. Bovine serum albumin (BSA) and glucose were used as standard references and the absorbance was taken by a spectrophotometer (DR5000, Hach). Total alkalinity was measured using an automatic titrator (TitraLab 870, Radiometer analytical). Biogas composition was analyzed using gas chromatograph service from a commercial lab (ALS Environmental).

3 Results and Discussion

3.1 Brewery Wastewater Characteristics

Wastewater quality indication parameters including pH, Total Alkalinity, TSS, VSS, BOD, tCOD, sCOD, TN, TP, NH₃-N and PO₄-P were analyzed for each batch of the wastewater and are shown in Table 2. The wastewater strength and characteristics fluctuated significantly from batch to batch due to the batch brewing operation., Based

on the average analysis readings, the craft brewery wastewater had a C:N:P ratio of 200:3.2:1 with an average COD of 11000 ± 2800 mg/L and over 90% was soluble COD. The BOD/COD ratio of the brewery wastewater was in the range from 0.5 to 0.77, indicating a good biodegradability of the wastewater (Metcalf & Eddy, 2002). The wastewater contained suspended solids at 950 ± 450 mg/L, primarily from mash tun and filter cleaning, with a VSS/TSS ratio ranged from 48 to 97%. Due to the high protein content in the wastewater, over 95% nitrogen and over 50% phosphorus was organic. The wastewater was slightly acidic, with a pH of 5.7 ± 0.5 and has a total alkalinity of 270 ± 80 mg/L as CaCO_3 .

Table 2 Brewery wastewater characteristic

Batch	TSS	VSS	tCOD	sCOD	TN	TP	NH ₃ -N	PO ₄ -P	pH	Alk
Unit	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	-	meq/L
Avg	950	770	11080	10190	180	55	8	26	5.7	13.5
	± 450	± 390	± 2760	±2320	±100	± 35	± 3	± 19	± 0.5	± 10.8
Min	270	230	5950	5420	70	15	2	8	4.9	2.3
Max	2610	2130	19140	17000	560	186	16	107	7.2	49.1

3.2 COD Removal Efficiency

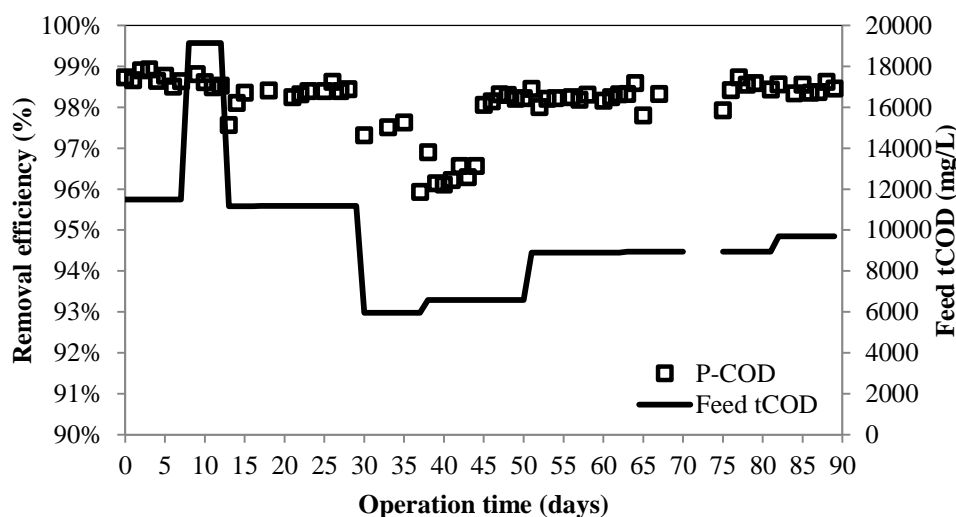


Figure 2 Influent COD and removal efficiency

The AnMBR system continuously operated for 90 days on 10 batches of brewery wastewater, excluding day 71 to 74, during which the recirculation pump failed, but a new pump was installed on day 75 and the operation was resumed. Figure 2 shows the influent COD concentration in black lines and the overall system COD removal efficiency. Figure 3 shows the effluent COD and VFA concentration. The influent COD varied from 6000 mg/L to 19100 mg/L which corresponded to OLRs from 3.5 to 11.5 $\text{kg/m}^3/\text{day}$. The effluent COD fluctuated with OLR, but the maximum effluent COD observed during the operation was below 300 mg/L at all times. An average effluent COD of 149 ± 7 mg/L and 172 ± 15 mg/L was observed under the OLR of 5 and 6.5 $\text{kg/m}^3/\text{day}$ operation, respectively, with VFA concentration under detection limit (50 mg/L). A high effluent COD concentration at 268 ± 21 mg/L with a detectable VFA concentration at 81 ± 5 mg/L was observed during OLR 11.5 $\text{kg/m}^3/\text{day}$ operation. The VFA residual in the mixed liquor at OLR 11.5 $\text{kg/m}^3/\text{day}$ indicated an incomplete biodegradation. The fluctuation in the effluent COD was significantly smaller than the changes in the influent COD and operation OLR, indicating that the AnMBR had a high tolerance to organic loading changes from the batch brewing operations, while it maintained good quality effluent free of solid. This was likely due to the retention of the suspended solids and colloidal substances by the membrane, in contrast to the conventional high rate anaerobic reactors.

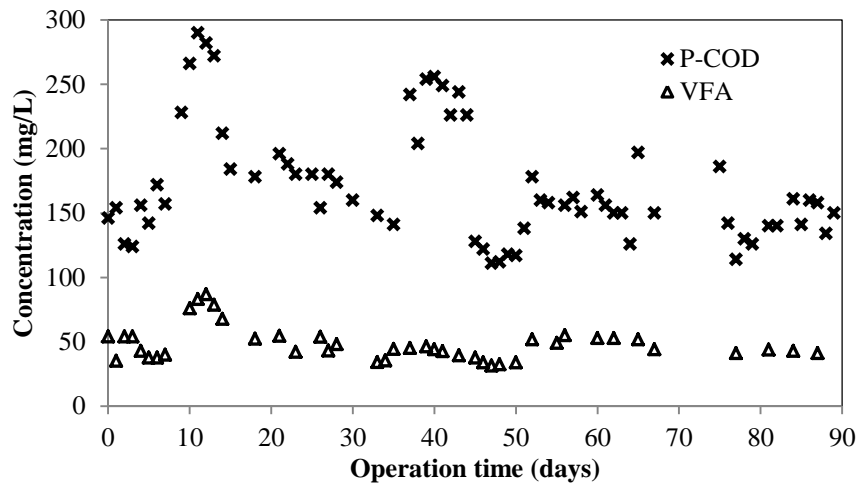


Figure 3 Effluent COD and VFA concentration

In all of the above OLR operations, the AnMBR system has achieved over 98% COD removal efficiencies, with the exception from day 30 to day 44, where the removal efficiency dropped below 98%. During day 30 to 35, the removal efficiency dropped to 97%, which was caused by the decrease of influent COD concentration thus resulting in a higher effluent/influent COD ratio in the removal efficiency calculation. During day 37 to day 44, the effluent COD concentration increased to 250 mg/L, dropped the removal efficiency down to 96%. The cause was argued to be the abnormal wastewater being fed into AnMBR during day 30 to day 37. The TSS and COD concentration of that batch of wastewater was comparably lower; however, the influent wastewater potentially contained cleaning detergent or other compounds, which could be toxic to the biomass. The toxic effect on the biomass had a delay of approximately 6 days and the bioreactor recovered in 7 days after the feed batch was switched on day 38. On day 45, the effluent COD concentration dropped below 150 mg/L and maintained overall removal efficiency above 98% for the rest of the study. With no acidification observed, the CSTR design of AnMBR established a high tolerance to toxic load and demonstrated a quick recovery, while suspended biomass was believed to be more susceptible for toxicants than granular sludge (Dereli et al., 2012).

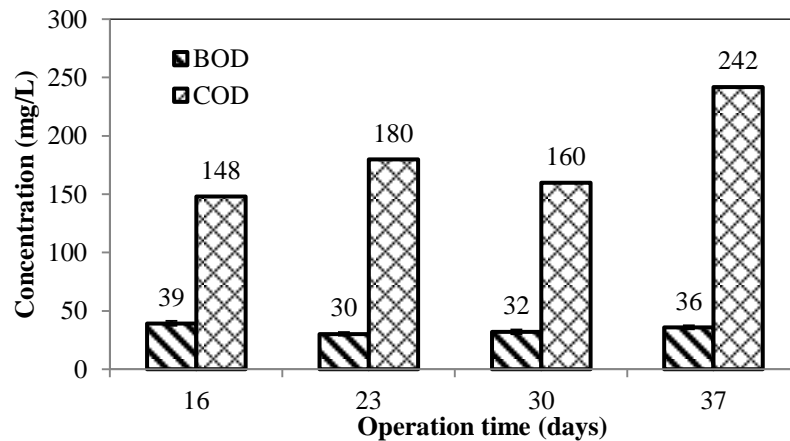


Figure 4 Effluent BOD and COD concentration comparison

Figure 4 shows the BOD and COD concentration in the effluent. The BOD concentration remained in the range of 30 to 40 mg/L, while the effluent COD fluctuated with the operation OLR. The low BOD/COD ratio (0.20 ± 0.05) suggest that the AnMBR effluent mostly contains non-biodegradable soluble substance, likely EPSs, which will be further discussed in the later section.

3.3 COD in the Mixed Liquor

The results demonstrated that the membrane filtration played an important role in improving the treatment performance of anaerobic digestion and the effluent quality. As shown in Figure 5, a significant difference was



observed between the COD concentration in 1.5 μm sludge filtrate, 0.45 μm soluble filtrate and 0.04 μm ultrafiltration permeate. The permeate COD was found to be significantly lower ($170 \pm 36 \text{ mg/L}$), with the ratios to 1.5 μm and 0.45 μm sludge filtrate calculated to be 0.06 ± 0.01 and 0.16 ± 0.03 , respectively, based on the data shown in Figure 5. These results indicated that the mixed liquor in the AnMBR reactor contained a significant portion of particulate and colloidal COD which were retained by the 0.04 μm hollow fiber membrane, implying that the importance of the membrane filtration in improving the effluent quality of the AnMBR.

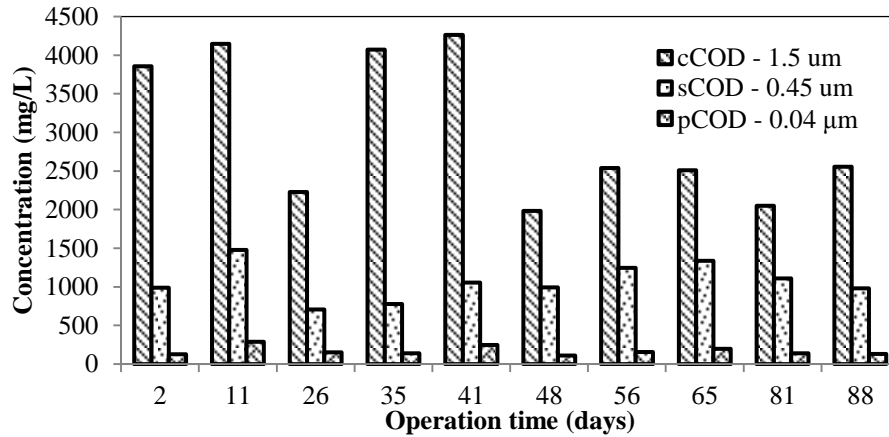


Figure 5 COD concentration in the 1.5 μm , 0.45 μm filtrate and membrane permeate

3.4 Biogas Production

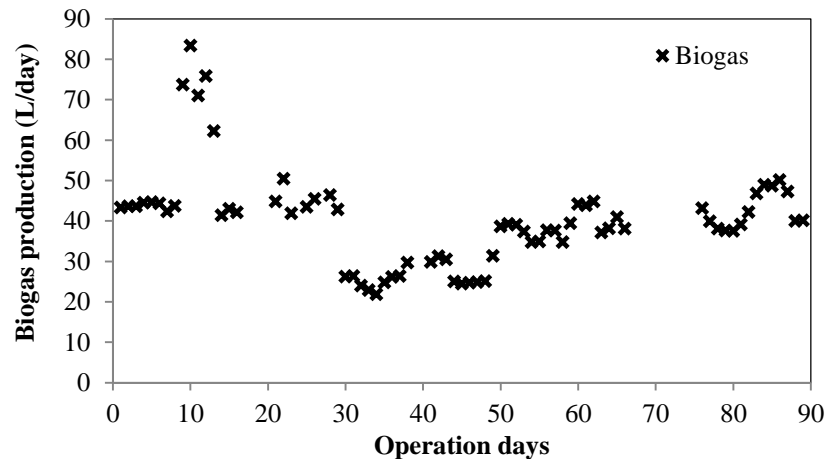


Figure 6 Biogas production under various OLR operation

The biogas production rate was very stable under each organic loading rate (Figure 6). Daily biogas production for OLR 3.5, 5.0 and 6.5 $\text{kg COD/m}^3/\text{day}$ was measured at 27.1 ± 1.8 , 40.7 ± 1.6 and $44.0 \pm 0.9 \text{ L/day}$, respectively. Gas chromatography analysis reported a biogas composition of 59% CH_4 , 31% CO_2 , 10% N_2 with no significant detectable CO and O_2 . The results demonstrated a feasible energy recovery, having an overall methane yield of $0.31 \pm 0.01 \text{ m}^3 \text{ CH}_4/\text{kg COD}_{\text{removed}} (35^\circ\text{C})$.



3.5 Bicarbonate Dosage

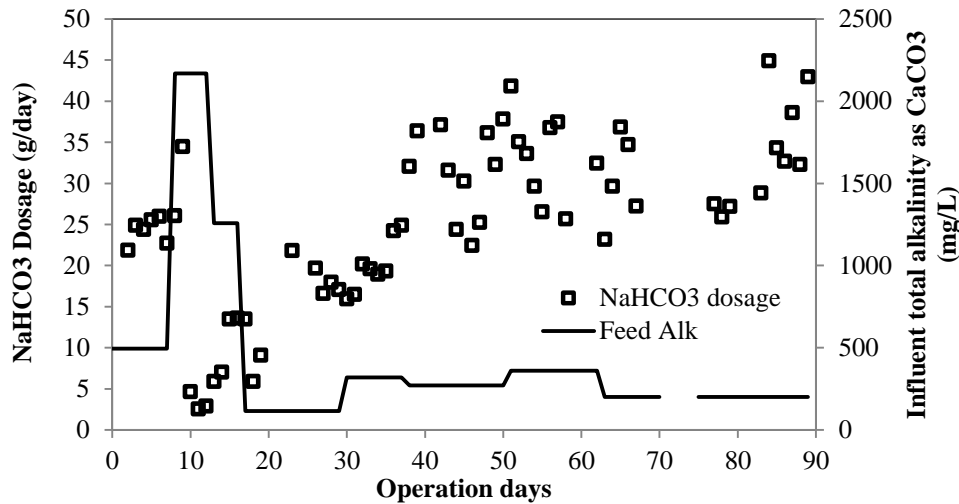


Figure 7 Influent total alkalinity and sodium bicarbonate dosage over the study

The pH and total alkalinity of the mixed liquor in the reactor were controlled to have favourable conditions for methanogenesis. Figure 7 shows the influent wastewater total alkalinity in black lines and the sodium bicarbonate dosage over the study. Excluding two batches from an earlier stage of the study, the influent wastewater was slightly acidic in general, with a low alkalinity of 270 ± 80 mg/L as CaCO_3 . With the intention of maintaining a favourable pH and total alkalinity in the mixed liquor at 7.02 ± 0.01 and 2800 ± 150 mg/L as CaCO_3 , respectively, sodium bicarbonate was added to the digester. An average of 30 ± 2 g NaHCO_3 was dosed per day, corresponding to a bicarbonate dosing rate of 3.7 ± 0.3 kg $\text{NaHCO}_3/\text{m}^3$ wastewater or 0.47 ± 0.03 kg NaHCO_3/kg COD treated.

3.6 Biomass Characteristics

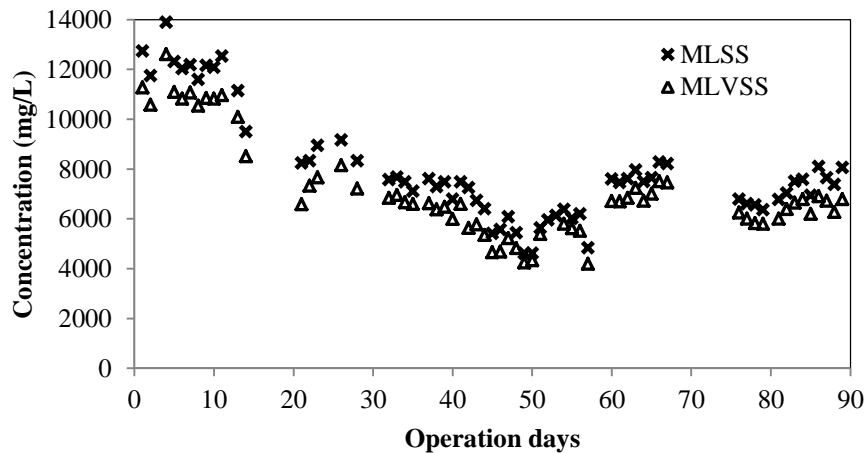


Figure 8 MLSS and MLVSS profile

Figure 8 shows the MLSS and MLVSS concentration during the different operation periods. The study started by using active anaerobic sludge inherited from the previous synthetic brewery wastewater study, with an initial MLSS and MLVSS concentration of 11800 mg/L and 10200 mg/L, respectively. One of the research scopes was to investigate the system and membrane performance under low biomass concentrations. The target was to stabilize the MLVSS around 7000 mg/L by adjusting the daily sludge wasting. During the earlier stage, the biomass concentration was gradually decreased by having high sludge wasting operation. The average sludge wasting for OLR 6.5 and 11.5 kg/m³/day operation period were 500 ml/day and 900 mL/day. The set target was achieved after the OLR 6.5 kg/m³/day operation on day 29; however, the stabilization failed. The biomass further decreased in the next 20 days, due to a combination of toxic influent wastewater on day 30 to 37 and low organic loading of 3.5



kg/m³/day. Excessive wasting of 325 ml/day on average was observed during day 30 to 50, corresponding to an average SRT of 46 days. On day 32, an immediate decline in MLVSS was observed followed by MLSS reduction in the next few days. The MLSS was found to be less sensitive to condition changes. The lowest MLSS and MLVSS observed were 4825 mg/L and 4250 mg/L before increased OLR to 5.0 kg COD/m³/day.

During the final OLR of 5 kg/m³/day stage, sludge was wasted at 300 ± 50 mL/day, corresponding to SRT of 50 days. A stable biomass concentration was achieved at 7600 ± 200 mg/L MLSS and 6700 ± 200 mg/L MLVSS. The observed yield and specific growth were calculated to be 0.029 ± 0.001 gVSS /gCOD and 0.022 ± 0.001 gVSS/gVSS/day. Both biomass yield and specific growth values are comparably lower than the typical values on conventional anaerobic processes (Metcalf & Eddy, 2002). Comparing to the earlier AnMBR study on synthetic wastewater, the biomass yield was identical but specific growth was found to be 25% lower. Over the entire study, a very high and stable VSS/TSS ratio of 0.86 ± 0.04 was observed, indicating very active biomass.

3.7 Membrane Filtration Performance

Constant flux filtration at 8 LMH with continuous biogas recirculation rate of 12 LPM was operated over the entire study, corresponding to a scouring rate of approximately 15.3 m³/m²/hr, similar to the operation scouring at 17.6 m³/m²/hr in an earlier lab scale hollow fibre AnMBR operation study (Diez et al., 2012). Over the 90 day study period, the TMP was monitored, and average daily cycle TMP, as well as the determined critical fluxes are reported in Figure 9. Critical flux tests were performed weekly using the flux stepping method (Cho and Fane, 2002), by increasing the flux in a 15 min step-wise fashion while analysing the corresponding TMP. According to Le Clech et al. (2003), critical flux was determined as the corresponding flux where in a 10 minute duration, the observed TMP had increased by 0.1 kPa or greater.

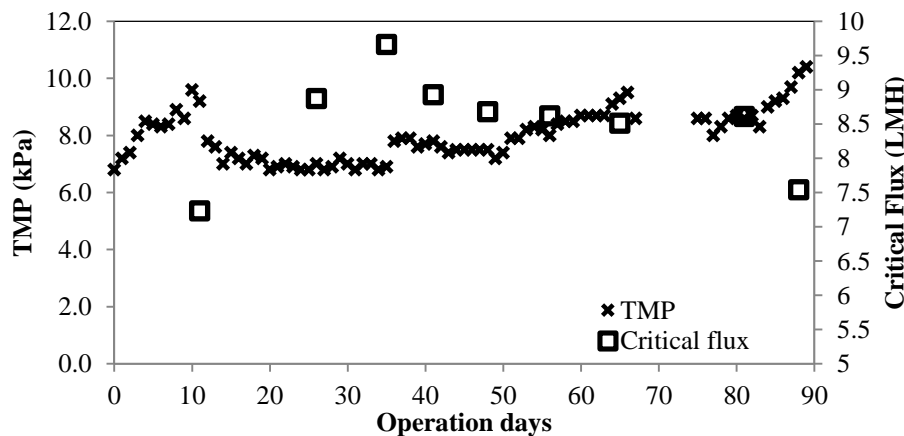


Figure 9 TMP profile and measured critical flux

A rapid increase in TMP at a daily rate of 0.28 kPa was observed in the first 11 days, when operating at OLR of 11.5 kg COD/m³/day. The critical flux test conducted on day 11 showed that the operation flux was higher than the critical flux that was found at 7.2 LMH. The EPS measurement conducted on the same day showed that the protein and polysaccharide concentrations were 2550 ± 35 and 600 ± 7 mg/L in 1.5 µm sludge filtrate and 550 ± 21 and 210 ± 2 mg/L in 0.45 µm membrane filtrate, respectively.

Organic loading rate was then immediately adjusted to 6.5 kg/m³/day by diluting the wastewater, with the operation flux still maintained at 8 LMH. A sudden drop of 1.4 kPa in TMP was observed on day 12, followed by a gradual decrease over the next week, which stabilized at 6.9 ± 0.1 kPa from day 20 to 35. A comparatively high critical flux was observed on day 35, due to lower organic loading (3.5 kg/m³/day) and lower observed EPS concentration in the mixed liquor. It was noted that the weekly chemical cleaning-in-place (CIP) was skipped on day 35 after the critical flux test, which resulted in a TMP jump of 0.9 kPa shown on day 36. A gradual decline in TMP was then witnessed after weekly CIP was back to schedule and it was maintained at 7.5 ± 0.1 kPa for the rest of 3.5 kg/m³/day operation.

After switching to organic loading of 5.0 kg/m³/day, the operation TMP gradually increased and a slight drop was observed after each weekly CIP. With the weekly cleaning protocol, an operation TMP of 8.7 ± 0.1 kPa was



perceived during the organic loading. The critical flux measured on day 88 was below the operation flux. Thus, a gradual increase in TMP was observed at the end of the study period, suggesting that a recovery cleaning of the membrane was necessary.

Over the 90 day study period, a TMP increase of 3.6 kPa was observed without performing any recovery cleaning. A sustainable operation of 8 LMH was achieved at organic loading of 3.5, 5.0 and 6.5 kg/m³/day, with detected critical flux at 8.8 ± 0.3 LMH. Lower critical flux below 8 LMH was identified during the high organic loading 11.5 kg/m³/day operation, which caused a significant increase on operation TMP over a short time frame, thus a sustainable operation at 8 LMH could not be maintained. The study suggests the AnMBR should be running at an organic loading less than 6.5 kg/m³/day and more importantly, a lower operation flux at 7 LMH is recommended to achieve long term sustainable operation and extend membrane lifetime. Aside from the design flux and OLR, a weekly CIP protocol is essential to control the long term membrane fouling.

4 Conclusions

In this study, the efficacy of the submerged AnMBR technology was demonstrated in the treatment of the high organic strength effluent from a local craft brewery. Due to the variation of the influent wastewater strength over the batch brewing processes, the AnMBR unit was subjected to an operation with a highly variable organic loading ranged from 3.5 to 11.5 kg COD/m³/day. Overall, the system achieved over 98% COD, 54% TN and 28% TP removal efficiency. Effluent COD was slightly increased with the organic loading, where the observed permeate COD was at 149 ± 7 , 172 ± 15 and 268 ± 21 mg/L for OLR of 5.0, 6.5 and 11.5 kg/m³/day, respectively. The permeate contained mostly non-biodegradable EPS, mainly soluble protein. The ultrafiltration membrane rejected 85% soluble polysaccharide but no significant rejection on soluble protein was observed. A favourable pH and total alkalinity condition was maintained with an average dosage of 0.37 kg NaHCO₃/m³. The methane yield was found to be very stable at 0.31 m³ CH₄/kg COD (35 °C). In comparison to the earlier synthetic wastewater study, a significantly higher concentration of EPS was found in the mixed liquor. Moreover, a moderate correlation was found between the critical flux and soluble protein concentration. An overall TMP increase of 3.6 kPa was observed over the 90 days period without any recovery cleaning. The study recommends running high strength brewery wastewater AnMBR on organic loading under 6.5 kg/m³/day with operation flux at 7 LMH, coupled with a weekly CIP protocol to maintain long term sustainable operation and extend membrane lifetime.

5 Acknowledgements

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