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Effects of salinity on the removal of trace organic contaminants by membrane bioreactor treatment for water reuse

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ABSTRACT

This study investigated the effects of salinity on the performance of a membrane bioreactor (MBR) system with a specific focus on the removal of trace organic contaminants. Eight trace organic contaminants were selected for this investigation. The obtained results indicated that changes in salinity in the range of 1–12 g/L have small impact on the removal of carbonaceous organic matter and total nitrogen (TN) by the MBR. The permeate water quality in terms of total organic carbon and TN slightly decreased when the system was exposed to higher salt concentration. A decrease in sludge production in saline mixed liquor was observed at salt concentration of 4 g/L, and then, microbial could adapt to the saline condition as evidenced in a gradual increase in biomass throughout this study. At a low salinity level, removal efficiencies of the selected trace organics are consistent with values previously reported in the literature. There was no significant impact of salinity on removal of the eight selected trace organic contaminants with bisphenol A being the only exception. However, severe membrane fouling was observed, when the salinity of the mixed liquor increased beyond 4 g/L. This could be explained by the increase in protein concentration in the supernatant which was probably released by the microbial population in response to the increase in salinity.

Keywords: Membrane bioreactor (MBR); Trace organics; Saline wastewater; Water reuse

1. Introduction

The many problems worldwide associated with inadequate sanitation are well known [1,2]. The introduction of contaminants to water supply sources and the environment is often associated with economic growth and can occur not only in developing nations but also in industrialized ones. These contaminants range from traditional pollutants such as heavy metals, pesticides, viruses and bacteria to emerging trace organics such as pharmaceutically active compounds and endocrine disrupting chemicals [2]. As a result, there has been a substantial increase in the scientific effort to develop and improve wastewater treatment technologies for human health and

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environmental protection. A notable example of such technologies is membrane bioreactor (MBR) which essentially combines the biological activated sludge treatment process with membrane filtration [3,4]. Although only being developed over the last three decades, MBR has established itself as preferable alternative over the conventional activated sludge (CAS) treatment technology. In comparison with CAS, MBR is more robust with a much smaller physical footprint and improved effluent quality [5]. Evidence has also emerged that MBR technology can offer an enhanced removal efficiency for moderately biodegradable and hydrophobic trace organics in comparison with CAS treatment [6,7]. The global market penetration of MBR technology has been reported to grow at an average of 11.6–12.7% per year since the turn of the millennium [3]. The most significant growth of MBR is in water recycling or the treatment-specific industrial wastewater where a high effluent quality is required. For example, in Australia, most recent MBR installations are for water recycling applications in coastal towns and small cities and are driven largely by stringent environmental regulations and freshwater scarcity.

High concentration or fluctuation in salinity is a challenge to biological treatment process in general when coastal sewers are subjected to infiltration by sea water or when industrial effluent discharged from high-salinity process such as the seafood process, cheese and canning. Recent attempt to integrate MBR with the forward osmosis process (which is commonly known as osmotic—MBR) [8,9] is another example where the issue of salinity build-up in the activated sludge reactor is of significant concern. There have been some studies on the effect of salinity on the MBR performance focusing on the effect of salinity on sludge characteristics, membrane permeability and effluent water quality. However, there remains considerable inconsistency regarding the effects of high salinity on the MBR performance [10,11]. Sun et al. [10] reported that high salinity could lead to severe membrane fouling and some decrease in the chemical oxygen demand (COD) removal. According to Sun et al. [10] the fouling mechanisms could quite complex and could be attributed to the response of the microbial population to high and/or salinity variation. Indeed, it has been shown that moderate to high salinities can cause toxic effects on groups of microorganisms that cannot adapted to a saline condition resulting in plasmolysis and/or loss of cell activity [12–14]. Reid et al. [15] found high salinity (up to 5 g/L) could significantly affect the physical and biochemical properties of activated sludge, increasing SMP and EPS concentrations as well as decreasing membrane permeability. In contrast, Sridang et al. [16] evaluated the MBR performance and structure of microbial community in the reactor for the seafood processing wastewater treatment and reuse. High and stable COD removal from a high strength and high-salinity seafood processing wastewater was reported [16]. The authors concluded that MBR was capable to tolerate organic-loading variation in a wide range [16]. Similarly, it has been reported that salt concentration at below 15 g/L had no effect on nitrification in a biofilm-suspended biomass MBR [17]. It is also noteworthy that little is known about the effect of salinity on the removal of trace organic contaminants by MBR treatment.

This study investigated the effects of salinity on the performance of an MBR system with a specific focus on the removal of trace organic contaminants. The effects of salinity increase on basic performance of the MBR were also examined.

2. Materials and methods

2.1. MBR set-up

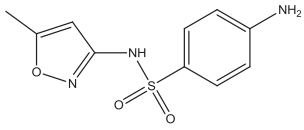
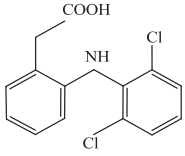
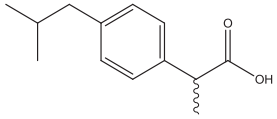
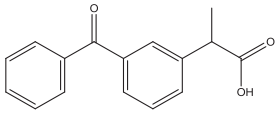
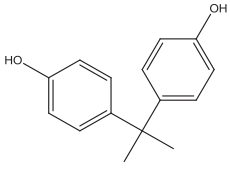
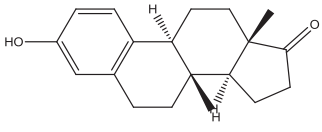
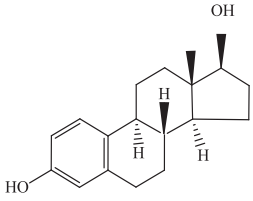
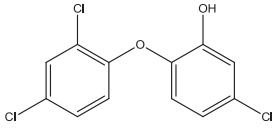
A laboratory-scale MBR set-up was used in this study. The MBR system consisted of a glass reactor with active volume of 9 L, a continuous mixer, two air pumps, a pressure sensor, and influent and effluent pumps. Two ZeeWeed-1 (ZW-1) submerged hollow fibre ultrafiltration membrane modules supplied by Zenon Environmental (Ontario, Canada) were used in this set-up. The membrane has a nominal pore size of 0.04 μm. Each module has an effective membrane surface area of 0.047 m^2. An electrical magnetic air pump (Heilea, model ACO 012) with a maximum airflow rate of 150 L/min was used to aerate the MBR set-up via a diffuser located at the bottom of the reactor. Dissolved oxygen concentration in the reactor was monitored daily and kept constant at 2 ± 1 mg/L by controlling the aeration flow rate. Another small air pump was also used to provide a constant airflow rate through the membrane module to reduce fouling and cake formation. Transmembrane pressure (TMP) was continuously monitored using a high-resolution pressure sensor (±0.1 kPa) that was connected to a personal computer for data recording. A Neslab RTE 7 equipped with a stainless steel heat-exchanging coil was used to maintain a constant temperature in the MBR reactor. The personal computer was also used to control the permeate peristaltic pump to operate on a 14 min suction and 1 min off cycle to provide relaxation time to the membrane modules. Flow rate of the influent pump was matched with that of the permeate pump to maintain a constant reactor volume. The

continuous mixer was used to ensure homogeneous conditions of the mixed liquor and to prevent the settling of biomass.

2.2. Model micropollutants

Eight compounds were selected for this study to represent three major trace organic groups of concern in water reuse applications—namely pharmaceuticals, steroid hormones and industrial compounds. The selection of these model trace organic compounds was also based on their widespread occurrence in domestic sewage and their diverse physicochemical properties (e.g. ionizable vs non-ionizable and hydrophobicity). Molecular structures and physicochemical properties of these trace organics are shown in (Table 1). In addition, the intrinsic hydrophobicity of these compounds varies significantly as reflected by

Table 1
Physicochemical properties of the selected trace organic contaminants

Trace organics	Molecular structure	Log D (at pH 8)[a]	Dissociation constant (pK_a)[a]
Sulphamethoxazole		−0.96	5.81
Diclofenac		1.06	4.18
Ibuprofen		0.14	4.41
Ketoprofen		−0.64	4.23
Bisphenol-A		3.43	9.73
Estrone		3.62	10.25
Estradiol		4.15	10.27
Triclosan		4.76	7.8

[a]Values obtained from the SciFinder Scholar (ACS) database.

their log D values. The most hydrophilic compound is sulphamethoxazole with log D at pH 8 of −0.96 and the most hydrophobic compound is triclosan with log D at pH 8 of 4.76. All selected trace organic compounds were of analytical grade. A cocktail of stock solution was prepared in pure methanol. The trace organic stock solution was kept in at −18°C in the dark and used within less than a month.

2.3. Micropollutant analysis

The analysis of the model trace organics was based on a method previously reported elsewhere [18]. Analytes were extracted using 5 mL, 500 mg hydrophilic/lipophilic balance cartridges (Waters, Millford, MA, USA). Cartridges were pre-conditioned with 5 mL of tert-butyl methyl ether (MTBE), 5 mL of methanol and 5 mL of reagent water. Samples were spiked with a solution containing 50 ng of an isotopically labelled version of each analyte. The sample was then loaded onto the cartridges at 15 mL/min, after which the cartridges were rinsed with 5 mL of reagent water and dried with a stream of nitrogen for 30 min. Loaded cartridges were stored at 4°C in sealed bags under nitrogen until elution and analysis. Analytes were eluted from the cartridges with 5 mL of methanol followed by 5 mL of 1/9 (v/v) methanol/MTBE into centrifuge tubes. The resulting extract was concentrated using vacuum assisted evaporation to approximately 100 μL. The extract was brought to a final volume of 1 mL with methanol.

Analytes were separated using an Agilent (Palo Alto, CA, USA) 1,200 series high-performance liquid chromatography (HPLC) system equipped with a 150 × 4.6 mm, 5 μm particle size, Luna C18 (2) column (Phenomenex, Torrence CA, USA). A binary gradient consisting of 5 mM ammonium acetate in water (A) and 100% methanol (B) at a flow rate of 800 μL/min was used. For ESI-positive analyses, the gradient was as follows: 10% B held for 0.50 min, stepped to 50% B at 0.51 min and increased linearly to 100% B at 8 min, then held at 100% B for 2 min. For ESI-negative analyses, the gradient was as follows: 10% B held for 0.50 min, stepped to 60% B at 0.51 min and increased linearly to 100% B at 8 min, then held at 100% B for 3 min. A 5 min equilibration step at 10% B was used at the beginning of each run. An injection volume of 10 μL was used.

Mass spectrometry was performed using an API 4,000 triple quadrupole mass spectrometer (Applied Biosystems, Foster City, CA, USA) equipped with a turbo-V ion source employed in both positive and negative electro-spray modes. For each analyte and internal standard, a precursor ion and two product ions were monitored for reliable confirmation. Relative retention times of the analyte and isotopically labelled internal standard were also monitored to ensure correct identification.

2.4. Analysis of basic water parameters

Conductivity and pH were measured using an Orion 4-Star Plus pH/conductivity meter. Total organic carbon (TOC) and total nitrogen (TN) were analysed using a Shimadzu TOC/TN-V_{CSH} analyser (Kyoto, Japan). TOC analysis was conducted in non-purgeable organic carbon mode. Samples were kept at 4°C until analysed, and calibrations were performed in the range between 0 and 1,000 mg/L and 0–100 mg/L for TOC and TN, respectively. Mixed liquor suspended solid (MLSS) and mixed liquor volatile suspended solids (MLVSS) contents in the MBR reactor were measured in accordance with the Standard Methods for the Examination of Water and Wastewater [19]. The concentrations of soluble microbial products (SMP) were determined by a previously described method [20].

2.5. MBR experimental protocol

Prior to the addition of NaCl to the biological reactor, the MBR system was operated for a month to establish a steady-state condition. The performance of the MBR under-steady state condition was evaluated in terms of effluent quality (pH, conductivity, TOC and TN) and sludge characteristics (ratio of MLVSS to MLSS). Effluent samples were collected and analysed every two days and sludge characteristics were carried out once a week. The aeration was provided in the reactor at DO of 2 ± 1 mg/L. The initial MLSS level was approximately 10 g/L. The average TOC and TN influent concentrations were 526 and 26.6 mg/L, respectively. The hydraulic retention time was set at 24 h, corresponding to permeate flux of 4.3 L/m^2 h (or 6.7 mL/min). The MBR reactor temperature was kept constant at 20.0 ± 0.1°C.

Five different NaCl concentration loads of 1, 2, 4, 8 and 12 g/L were operated in this experiment. Each load was investigated by measuring conductivity of feed solution and effluent to confirm the steady state of salt condition in the system. The reactor was maintained at the steady stated for a period of 1–5 days with trial of salt concentration of 1 and 2 g/L and for a period of 10–14 days with salt concentration ranging from 4 to 12 g/L.

Synthetic wastewater was used in this study to simulate high-strength municipal sewage. The concentrated synthetic wastewater was prepared and stored in a refrigerator at 4°C. It was then diluted with

MilliQ water on a daily basis to make up a feed solution containing glucose (800 mg/L), peptone (150 mg/L), KH_2PO_4 (35 mg/L), $MgSO_4$ (35 mg/L), $FeSO_4$ (20 mg/L), and sodium acetate (450 mg/L). This composition was based on a previous study by Nghiem et al. [21].

Once stable operation has been achieved, trace organic contaminants were spiked into the feed solution each day to make up a concentration of approximately 2,000 ng/L of each selected compound. The feed solution was kept in a stainless steel reservoir at room temperature (20 ± 2°C). The collected effluent was kept at 4°C in the dark and analysed within less than 48 h. Removal efficiency was calculated as $R = 100 \times \left(1 - \frac{C_{Eff}}{C_{Inf}}\right)$, where C_{Inf} and C_{Eff} are influent and effluent concentrations (ng/L) of the trace organic compound, respectively. It is noteworthy that the term removal here does not necessarily indicate complete mineralization of the trace organics to carbon dioxide and water.

3. Results and discussion

3.1. Removal of TOC and TN

Fig. 1 shows the variation of TOC and TN removal by the MBR system at different NaCl loading. During initial stabilization time of one month, the TOC and TN removal efficiencies were high and stable at approximately 99 and 97%, respectively (data not shown). At 1 g/L of NaCl, TOC removal efficiency was still in the range of 98–99% (Fig. 1) corresponding to the effluent TOC of less than 5 mg/L. The TOC removal efficiency deteriorated at 2 g/L NaCl to 88.6%. The lowest TOC removal of 87.4% was observed at the NaCl loading of 4 g/L. The decrease in organic removal efficiency with the increase in salt concentration might be due to inhibitory effect of salt on microbial resulting in loss of metabolic activity and plasmolysis causing releasing of the intracellular constituents and SMP [13]. This result is in good agreement with a previous study by Artiga et al. [17]. They found that lower COD removal efficiency was affected by high salinity. Similarly, Colunga and Martinez [22] reported only 37% COD removal by a sequencing biological reactor. However, as can be seen in Fig. 1, TOC removal was stable in the range of 4–12 g/L. This can be attributed to the adaptation of the microbial population in the sludge to hyper-saline condition [17].

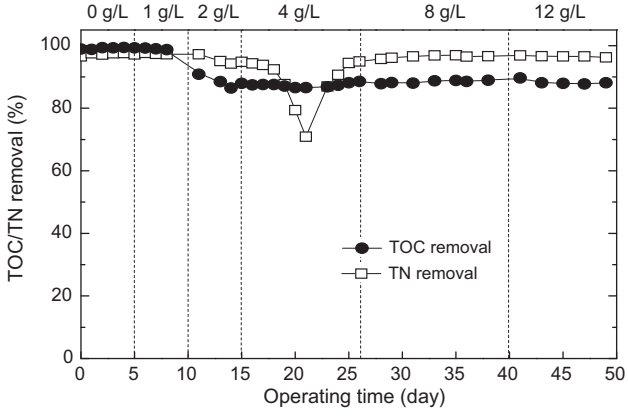


Fig. 1. Variation of percent removals of TOC.

TN removal efficiency was also observed to decrease when the NaCl loading increased from 2 to 4 g/L. The lowest TN removal of 70.9% was observed at the NaCl loading of 4 g/L, and then, the removal efficiency of TN recovered to the normal condition (approximately 97%) despite the high NaCl loading of up to 12 g/L. The decrease in TN removal efficiency with increasing NaCl concentration could be due to the wash out of dead biomass and lysed cell constituents [23] and inhibition of nitrification process [24]. Diverging conclusions have been reported regarding the impact of high salinity on the denitrification process. Yang et al. [25] reported that high salinity can adversely affect nitrifying bacteria and reduce the nitrification process. In contrast, Sakairi et al. [26] showed almost 100% nitrogen removal under seawater condition provided that sufficient phosphate was also available for adenosine tri-phosphate generation. Results reported in Fig. 1 suggest that high salinity could exert some impact on the removal of nitrogen and that the system can recover as the microbial community adapt to the more saline condition.

3.2. Sludge production

The MLSS and MLVSS at different NaCl loading were compared in Fig. 2. When the NaCl concentration in the reactor increased to 4 g/L, there was a notable decrease in sludge production and both the MLSS and MLVSS concentrations decreased slightly. However, as the MBR was operated at 4 g/L of NaCl in the reactor, the MLSS concentration gradually increased again. Sludge production continued to occur as the NaCl loading was increased to 12 g/L. The ratio of MLVSS/MLSS was ranged from 0.88 to 0.98 throughout the experiment indicating that most of the MLSS was active biomass. The decrease in MLSS concentration at 4 g/L of NaCl could be attributed to the death of biomass due to salinity shock load [23]. On the other hand, at higher concentration of NaCl ranged from 8 to 12 g/L, there was no apparent effect of salt on the biomass concentration (Fig. 2). Our results

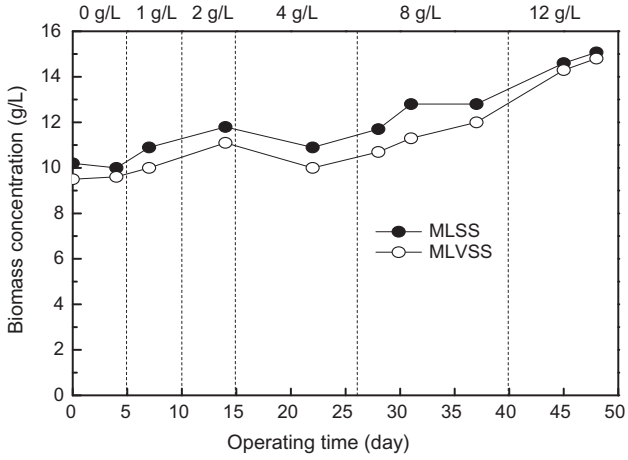


Fig. 2. Variation of biomass concentration in the bioreactor as a function of salinity (in NaCl concentration).

are consistent with that reported by Hamoda and Al-Attar [11] who found that salinity in the range of 10 and 30 g/L had no impact on biomass growth inhibition and biomass production during activated sludge treatment. Results reported here suggest that the impact of salinity on the performance of the activated sludge reactor is due to shock loading; however, it is possible for the microbial community to adapt with a hyper-saline condition.

3.3. Removal of trace organics

The performance of a laboratory-scale MBR-treating synthetic wastewater with eight model compounds was investigated at different NaCl load. Fig. 3 shows the removal efficiency in compounds throughout the salinity experiment. Salinity had no effect on the removal efficiency of the five pharmaceuticals investigated in this study (Fig. 3(a)). High removal efficiencies of over 94% were observed for ibuprofen and sulphamethoxazole whereas ketoprofen was moderately removed at the rate of 68%. Ibuprofen and sulphamethoxazole were removed by the MBR by up to 95%. Diclofenac was partially degraded, presenting a removal efficiency of approximately 40%. Similarly, the removal efficiencies of estrone, 17 β-estradiol and triclosan were high and stable regardless of NaCl concentrations in the reactor (Fig. 4(b)). The only trace organic contaminant that showed some response to the variation in NaCl concentration in the reactor is bisphenol A. The removal efficiency of bisphenol A decreased from approximately 90% to well below 80% as NaCl concentration increased from 8 to 12 g/L. Bisphenol A is a moderately hydrophobic compound, and its removal is governed by both adsorption to the biomass and biological degradation [21]. It is probable that high salinity could deteriorate microbial flocs in saline mixed liquor, thus reducing the adsorption of bisphenol A to the activated sludge. This is also consistent with the slight decrease in TOC removal at high salinity as observed in Fig. 1.

3.4. Membrane fouling

Apart from the variation in the salinity level in the reactor, all other operating parameters were kept constant during this study. The MBR system was operated with a constant flux and no salt addition in

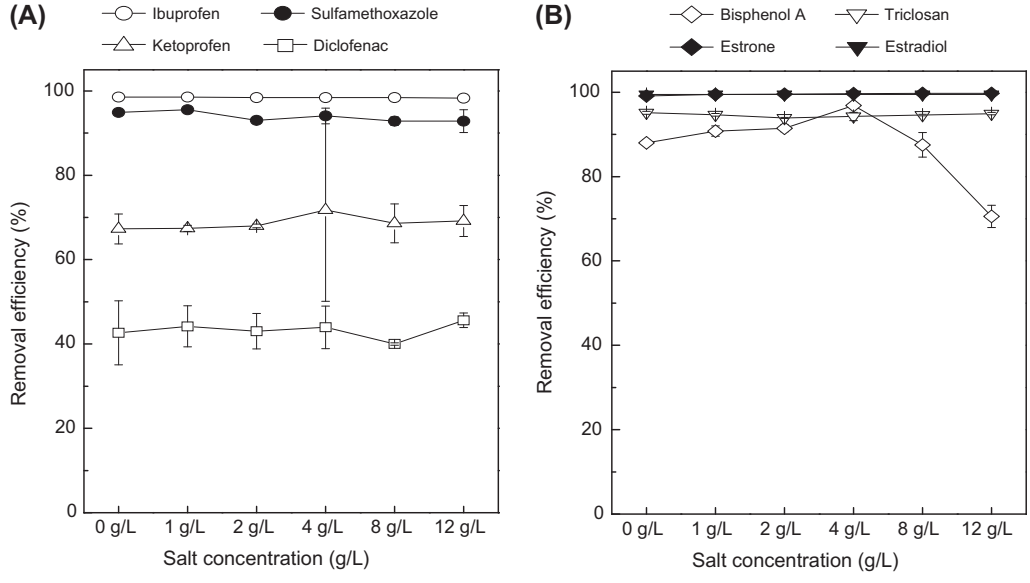


Fig. 3. Variation of model compound removals with salinity (A) pharmaceutical compounds (B) endocrine disrupting compounds. Error bars represent the standard deviation of four repetitive measurements.

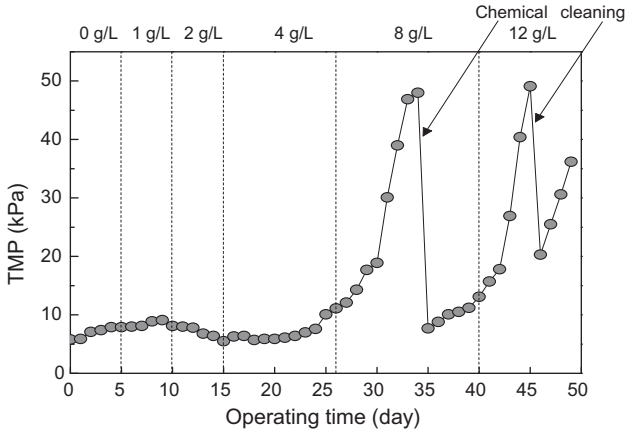


Fig. 4. The overall TMP profile during the experiment.

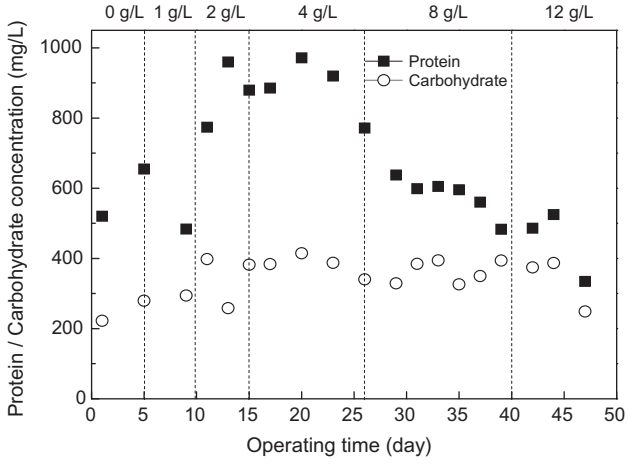


Fig. 5. SMP (protein and carbohydrate) concentration of the supernatant in the reactor.

the first 14 days, resulting in a stable TMP profile (Fig. 4). When the salinity increased to 4 g/L, a gentle increase in the TMP could be observed. This was followed by severe membrane fouling as the NaCl concentration increased to 8 and 12 g/L.

Fig. 5 shows the concentrations of protein and carbohydrate in SMP at various NaCl concentrations. As the NaCl concentration increased to 1 g/L, there was a small in both protein and carbohydrate concentrations in the supernatant of the mixed liquor. As the salinity level increased further, there was a sharp increase in the protein content of the SMP. At NaCl concentration of 4 g/L, the highest concentration of protein for SMP was observed. It then decreased gradually as the salinity in the reactor increased to 12 g/L. On the other hand, the concentration of carbohydrate fluctuated slightly throughout this study. The changes in protein concentration appeared to be more sensitive to salinity than carbohydrate and could be attributed to the release of extracellular constituents, accumulation of unmetabolized and intermediate products of incomplete degradation of organic substances when the microbial population was under stress [27] and polymer production of some microbial for self-protection again adverse environmental condition [24,27]. It is noteworthy that the peak of protein (or SMP) concentration in the supernatant (Fig. 5) coincided with the lowest removal of TN (Fig. 1). The increase in protein concentration may partially explain for the severe membrane-fouling condition at high-salinity level in the reactor. However, the severe fouling condition observed in Fig. 4 could also be attributed to other reasons that are not known at this stage. Indeed, as the salinity increased further, the release of protein into the supernatant decreased to a normal level, indicating that the microbial population has adapted to the hyper-saline condition.

4. Conclusion

This study investigated the effect of salinity on the removal of compounds by the MBR system. Results indicated that changes in salinity ranging of 1–12 g/L have small impact on the MBR performance for removing organic matter and nitrogen. A decrease in biomass production in saline mixed liquor was observed at salt concentration of 4 g/L, and then, microbial could adapt to saline condition as a result of gradual increase in biomass throughout the rest of the study. The increase in SMP (mostly as protein) concentration resulted from disturbance in microorganism behaviour and response to salinity variation. There was no discernible impact of salinity on the removal of the trace organic contaminants selected in this study with bisphenol A being the only exception. The removal of bisphenol A decreased slightly as the salinity level increased to 12 g/L (as NaCl).

Acknowledgements

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