Pollutant removal and membrane fouling in an anaerobic submerged membrane bioreactor for real sewage treatment

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ABSTRACT

Real sewage was continuously treated by a laboratory-scale anaerobic submerged membrane bioreactor (AnSMBR) for over 160 days. Results showed that around 90% of chemical oxygen demand, and 99% of turbidity and total suspended solids in the sewage could be removed by the AnSMBR system. Membrane flux sustained at 11 L/(m^2 h) was realized with biogas sparging. Small flocs from sludge deflocculation in the early operational period caused a high membrane fouling rate, and the high specific filtration resistance of the cake layer appeared mostly attributable to the osmotic pressure effect. The performance results were also compared with those in the literature for upflow anaerobic sludge blanket reactors and aerobic membrane bioreactors for sewage treatment, demonstrating that AnSMBR could provide a desirable alternative for sewage treatment.

Key words | anaerobic submerged membrane bioreactor, membrane fouling, osmotic pressure, sewage

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INTRODUCTION

Sewage has been regarded as the major worldwide point-source pollutant due to its large amount of discharge, although it is always characterized to be low organic concentration. Its treatment deserves ample research. Anaerobic wastewater treatment has been considered as a desirable option as bioenergy is recovered, sludge production is low and nutrients are preserved for reuse (Seghezzo *et al.* 1998). However, anaerobic processes have been historically regarded to be much more suitable for treatment of high concentration wastewater or sludge digestion as compared with low concentration wastewater such as sewage (Harada *et al.* 1994). There are several challenges limiting such treatments. The first one is that, for low concentration wastewater treatment with shorter hydraulic retention time (HRT), it is difficult to efficiently retain slow-growth anaerobic microorganisms in a conventional anaerobic reactor. Although biofilm and granule formation is the most common strategy for retention of biomass in high-rate anaerobic reactors, it is a time-consuming and complex process that involves complicated physical, chemical and biological interactions, and has been proven to be very problematic for low concentration wastewater treatment (Willetts *et al.* 2000). Another problem is that anaerobic effluents hardly conform to the standards for discharge or reuse of effluent because anaerobic metabolites contain higher content of organic matters (Herrera-Robledo *et al.* 2010). The membrane is an absolute barrier to biomass, and thus submerging membrane modules in an anaerobic reactor (namely, anaerobic submerged membrane bioreactor (AnSMBR)) is expected to resolve these problems (Lin *et al.* 2013). Evidence has extensively shown the benefits of complete biomass retention and enhanced effluent quality by the introduction of membrane (Lee *et al.* 2003; Lin *et al.* 2009; Yang *et al.* 2009).

Although the AnSMBR process shows great potential in sewage treatment, as compared with numerous studies regarding aerobic membrane bioreactors (MBRs) treating sewage, investigation exploring the conception of an AnSMBR, especially for real sewage treatment is very limited. Meanwhile, membrane fouling is still the major barrier limiting the development of AnSMBRs. A lot of studies have extensively investigated the membrane fouling issue by focusing on the various possible factors influencing it. It has been frequently reported that biomass content (Meng *et al.* 2006;

doi: 10.2166/wst.2014.080

Li et al. 2008), distribution of particle size (Meng et al. 2006), fractal dimension (Li et al. 2008; Lin et al. 2010) and microbial products such as extracellular polymeric substances (EPS) (Lee et al. 2003; Meng et al. 2006; Li et al. 2008) and soluble microbial products (SMP) (Meng et al. 2006; Lin et al. 2009) are key factors governing membrane permeability. In contrast, some other researchers found that biomass content (Lin et al. 2010), SMP (Lee et al. 2003) and total amount of EPS (Lee et al. 2003) have no significant correlation with membrane fouling. It can be seen from the above analysis that no definite conclusions on the influence of sludge characteristics on membrane fouling have been drawn, especially for an AnSMBR for real sewage treatment. Meanwhile, based on the contribution of foulant components to the total membrane fouling, several membrane fouling mechanisms, including pore plugging/clogging, adsorption of soluble compounds, formation of gel or cake layer, cake layer consolidation, and long-term spatial and temporal changes of the foulants, have been proposed (Lin et al. 2013). However, recent studies have shown that all these mechanisms cannot satisfactorily explain some fouling phenomena in MBRs (Wang & Li 2008; Chen et al. 2012).

Bearing in mind the information above, the present study aims to investigate the pollutant removal performance of an AnSMBR system for sewage treatment. Moreover, membrane filtration performance and the underlying causes are presented and proposed. To put this study in perspective, a performance comparison between this study and literature studies regarding aerobic MBRs and upflow anaerobic sludge blanket (UASB) reactors for sewage treatment was conducted.

MATERIAL AND METHODS

Experimental setup and operation

The schematic diagram of the AnSMBR treating sewage is shown in Figure 1, which has 80 L total volume ($0.35 \times 0.35 \times 0.65$ m length × width × height) and 60 L effective volume, respectively. A flat sheet polyvinylidene fluoride membrane module (pore size: 0.30 μm; dimension: 28 cm (L) × 18 cm (H) × 0.5 cm (W)) was submerged in the reactor. The reactor headspace biogas was recycled to scour the membrane surface through a tube diffuser located underneath the membrane module. The reactor water level was controlled by a level controller connected to the pump. The effluent was extracted by a peristaltic pump at fixed flow rates and under an intermittent operation mode (9 min on/1 min off) for membrane relaxation. The temperature was maintained at 25 ± 3 °C. A pH control unit consisting of a pH electrode and a pH regulation pump was applied to control the reactor pH at 7.0 by using NaOH solution. A programmable logic controller was used to control all the electric devices. Real sewage obtained from a local town was used as feed. Seed

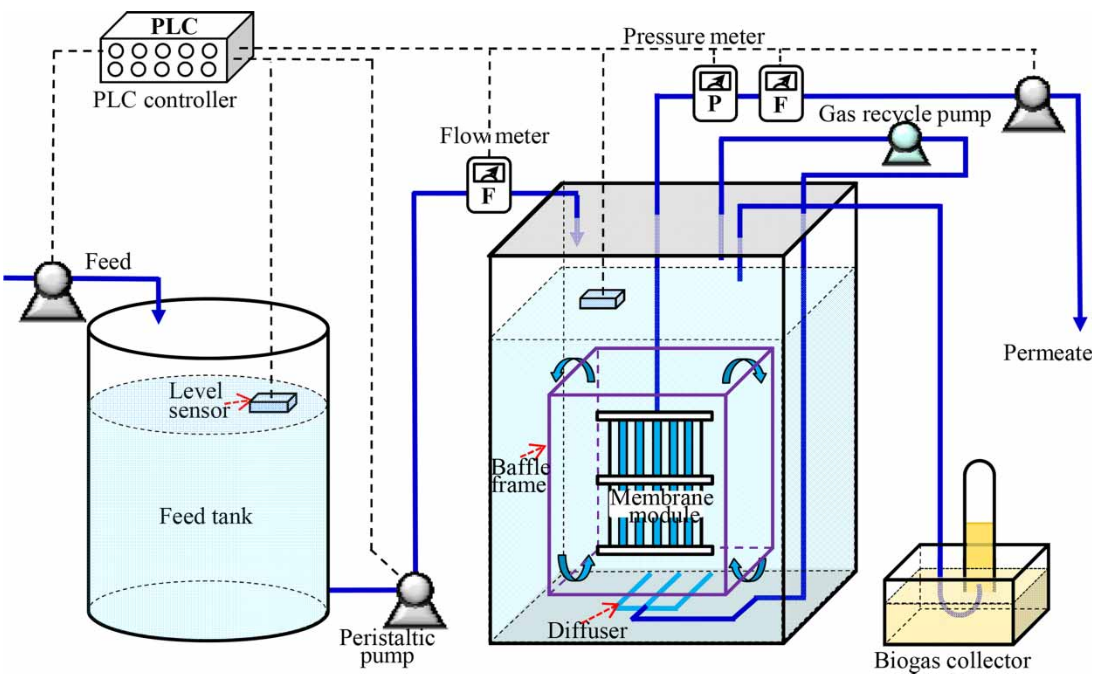

Figure 1 | Schematic diagram of the experimental AnSMBR setup.

sludge originally taken from a local conventional sewage sludge digester was screened through a 100 μm sieve, and then added to the reactor in order to shorten the start-up period. The initial sludge concentration, in terms of mixed liquor suspended solids (MLSS), in the reactor was about 6.4 g/L. The AnSMBR was continuously operated for over 160 days which primarily comprised a start-up period (day 1–12, HRT = 28 h), phase 1 (day 13–100, HRT = 10 h) and phase 2 (day 101–160, HRT = 6 h). During the whole period, there was no sludge discharge except sludge sampling, corresponding to a sludge retention time of over 260 days.

Analysis methods

The mixed sludge liquor taken from the reactor was subjected to a 9000 × g centrifugation for 5 min. A 0.45 μm membrane was then used to filter the centrifuged supernatant. The filtrate was regarded as containing SMP. Bound EPS was extracted according to cation exchange resin (Dowex Na^+ form, Sigma-Aldrich) method. Both SMP and bound EPS are normalized as the sum of polysaccharides (PS) and proteins (PN). PS and PN were colorimetrically measured by using phenol/sulphuric acid method and Folin method, respectively. Experimental details can be found in a previous study (Lin *et al.* 2009).

A laser particle size analyzer (Malvern Mastersizer 2000) was used to measure particle size distribution (PSD) of the mixed sludge liquor samples. Each sample was measured in triplicate. The mean floc size of the sample was denoted by the parameter d_{50}, a value that 50% of the measured particle volumes were equal to.

Cake layer characteristics including porosity, density and charge density were determined. The details of the determination process are given in a previous study (Chen *et al.* 2012).

The influent and effluent samples were obtained from the reactor periodically. Parameters including chemical oxygen demand (COD), MLSS and total suspended solids (TSS) were measured according to *Standard Methods* (APHA 2005).

The total filtration resistance (R_{tol}) was obtained according to Darcy's law (Equation (1))

$$R_{tol} = R_m + R_c = \frac{\Delta P}{\mu J} \tag{1}$$

where ΔP is the trans-membrane pressure (TMP); R_m is the membrane resistance; R_c is the cake layer resistance; J is the membrane flux; μ is the dynamic viscosity. R_m is usually quite low compared to R_c; therefore, R_c can approximate to R_{tol} when an apparent cake layer is formed. The specific filtration resistance (SFR) of cake layer is then calculated as Equation (2), where m_c is cake layer mass

$$\mathrm{SFR} = \frac{\Delta P}{\mu J m_c} \tag{2}$$

The Carman–Kozeny equation provides another way to calculate the hydrodynamic SFR of a porous medium by assuming that there are no extra interactions between the flow and the porous medium

$$\mathrm{SFR} = \frac{180(1-\varepsilon)}{\rho d_p^2 \varepsilon^3} \tag{3}$$

where d_p is the particle diameter; ρ is the cake layer density; ε is the porosity of cake layer.

A recent study reported that a significant osmotic pressure must to be overcome in order to draw the permeate through the cake layer (Chen *et al.* 2012). The osmotic pressure ($\Delta\Pi_c$) during the cake layer filtration could be estimated as follows (Chen *et al.* 2012):

$$\Delta\Pi_c = K_a \sigma \rho RT(1-\varepsilon)/\varepsilon(K_a + 10^{-\mathrm{pH}}) - C_p RT \tag{4}$$

where K_a is the average dissociation constant of the functional groups; σ is the charge density; C_p is the ion concentration of the permeate; R is the universal gas constant; T is absolute temperature.

RESULTS AND DISCUSSION

Pollutant removal performance

Real sewage treatment was continuously treated for over 160 days by the AnSMBR setup. The initial membrane flux was about 4.0 L/(m^2 h) (LMH) with a HRT of approximate 28 h. The variation of the influent and effluent COD with elapsed time is shown in Figure 2. According to the records, the permeate COD gradually decreased from about 180 mg/L to below 50 mg/L during the first 12 days' operation. This period can be considered as the start-up period.

After the start-up period, HRT was decreased to approximately 10 h by increasing membrane flux from 4 LMH to 11 LMH. As a reaction, permeate COD increased to 168 mg/L, which, however, decreased gradually to about 46 mg/L in

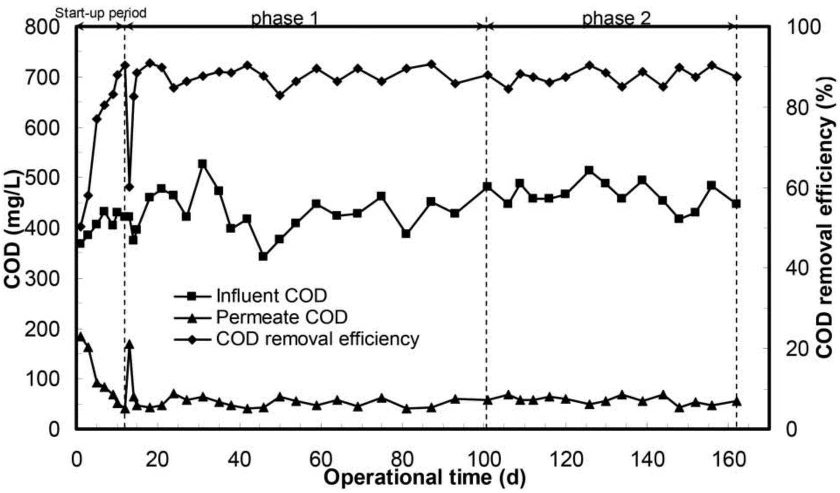

Figure 2 | Variations of influent COD, permeate COD and COD removal efficiency with operational time.

the following 3 days. HRT of 10 h was kept constant in phase 1, which lasted for about 90 days (until day 100). Although certain fluctuation of the feed COD and sludge content occurred in phase 1, the permeate COD showed relative stability with a COD removal of approximately 90%.

At day 100, membrane surface area was increased from 0.6 to 1.0 m^2 by addition of membrane elements in the membrane module. This operation resulted in a HRT of approximately 6 h. Although HRT was significantly reduced, the effluent quality in terms of COD stabilized below approximately 60 mg/L throughout phase 2. This result indicated that HRT of 6 h might be enough to obtain a significant COD removal in an AnSMBR treating sewage, and above that, only minor increase was achieved. With respect to turbidity removal, the permeate had a turbidity lower than 0.2 nephelometric turbidity unit with a removal over 99%. TSS of the raw wastewater was 187 ± 26 mg/L during the whole operational period; however, TSS was not detectable in permeate. This is not surprising, considering that the ability to produce TSS-free permeate by membrane treatment has been widely reported. At such low level of COD and turbidity, the treated water is suitable to reuse for some industrial purposes.

In Table 1, the results in this study were compared with some other studies using an UASB reactor or aerobic MBR to treat sewage. As shown in Table 1, in spite of significant removal of COD and TSS, and well proven advantages, UASB sewage treatment is still counted as a challenge because UASB effluent barely complies with the discharge standards of most countries. Therefore, a post-treatment process is usually necessary to polish the UASB effluent. In

Table 1 | Comparison of pollutant removal in AnSMBR system, UASB reactor and aerobic MBR for sewage treatment

	Operational condition			Influent concentration (mg/L)		Removal efficiencies (%)			
Reactor type	Reactor volume (m^3)	Temp (°C)	HRT (h)	COD	TSS	COD	TSS	Start-up (months)	Reference
AnSMBR	0.060	25 ± 2	6.1	464 ± 26	187 ± 26	88.4 ± 1.9	100	<0.5	This study
UASB	120	18–28	5–15	188–459	67–236	60	70	>2	Vieira & Garcia (1992)
UASB	0.059	~20	6–24	782	393	57.8	76.9	~4	Tang *et al.* (1995)
UASB	0.140	15	6	721 ± 171	398 ± 167	44 ± 9	73 ± 14	>1	Mahmoud *et al.* (2004)
Aerobic MBR	0.006	–	~8	380–400	140–200	86–95	>99	<1	Pollice *et al.* (2008)
Aerobic MBR	0.190	19–31	~5	~230–360	–	94	>99	–	Yang *et al.* (2009)
Aerobic MBR	0.036	20–30	5	230–580	–	90	>99	~0.25	Jin *et al.* (2005)

contrast, the effluent from AnSMBR possessed comparable quality in terms of COD and TSS to that of the effluent from the aerobic MBRs, and might be used for irrigation in agriculture. Meanwhile, AnSMBR sewage treatment features several advantages, including reduced sludge yields, less operation energy consumption, and energy recovery, over aerobic MBR sewage treatment. The comparison demonstrated that AnSMBR would be a promising solution for sewage treatment.

Membrane fouling

In this study, membrane fouling can be directly reflected by the variation of TMP with time as the bioreactor was operated under constant flux mode. The typical evolution cycles of TMP with operational time showed a three-stage profile which was characterized as a short-term rapid TMP rise (stage 1) followed by a relatively long, slow TMP rise (stage 2) and a transition to a rapid TMP rise (stage 3) (Figure 3). Similar TMP profiles have been frequently observed in other MBR research (Zhang *et al.* 2006). It can be seen from Figure 3 that the applied flux of 11 LMH could be sustained over a relatively long period (180–370 h). This flux was therefore considered as a sustainable flux. Moreover, although the applied flux and gas sparging strength were the same, fouling behaviour indicated by TMP during two operational periods was significantly different. If the sum of the first two stages (stages 1 and 2) in the TMP profile is regarded as a sustainable operational time without membrane cleaning, this result demonstrated that the sustainable operational time in the later period was over two times longer than that in the early period. The causes regarding the difference of TMP rise behaviour in the two periods deserved further investigation.

When TMP exceeded approximately 45 kPa, membrane cleaning described in a previous study (Lin *et al.* 2010) was conducted for membrane flux recovery. When the membrane module was taken from the reactor for cleaning, it was observed that an apparent cake layer was formed on the membrane surface. The total filtration resistance was estimated to be $1.6\times10^{-13}\,m^{-1}$ according to Darcy's law when final TMP was 48 kPa. Given the determined cake thickness of 0.61 mm and the estimated water content of cake sludge of 90%, the SFR of the sludge cake was calculated to be around $2.5\times10^{14}\,m^{-1}\,kg^{-1}$. At same time, the Carman–Kozeny equation provided another way to calculate the SFR of a porous medium. Characterization of the cake layer showed that cake layer porosity was in the range of 0.21–0.49, and cake layer density was about 1.20 g/mL. By assuming the average cake layer pore size of 3 μm, the SFR of cake layer was estimated to be 7.2×10^{10} to $1.4\times10^{12}\,m^{-1}\,kg^{-1}$ according to Equation (3), which was over two orders of magnitude lower than $2.5\times10^{14}\,m^{-1}\,kg^{-1}$ obtained from experimental filtration tests. It should be noted that the assumptions made in the calculations were conservative and reasonable. Real situations would make the above difference more significant.

To explore the underlying causes of membrane fouling behaviour in the AnSMBR treating sewage, some parameters of sludge properties were recorded during the

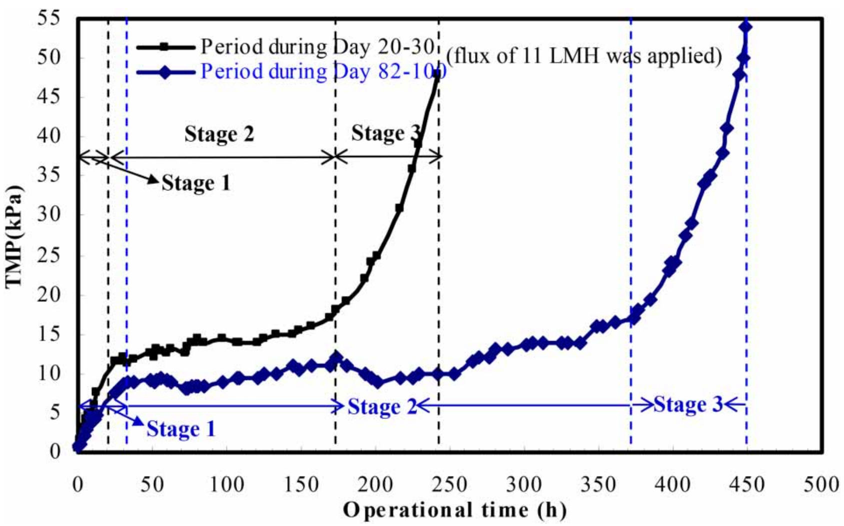

Figure 3 | Typical profile of TMP variation with operational time.

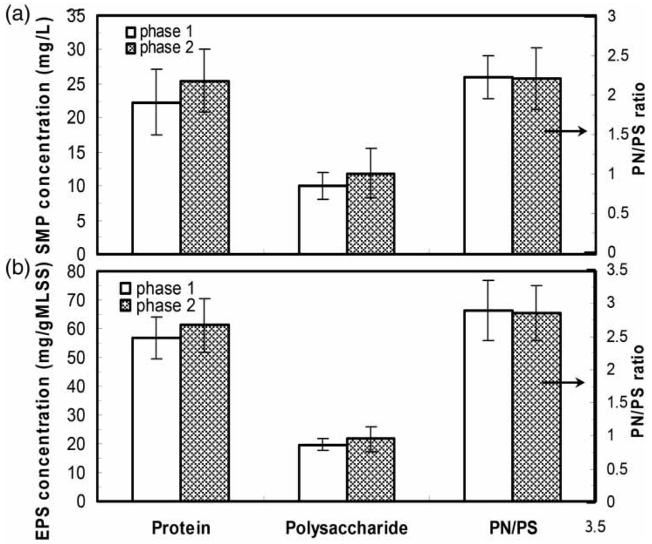

Figure 4 | (a) SMP and (b) bound EPS compositions at phase 1 and phase 2 (sample number = 12).

experiment. Figure 4 shows the contents of supernatant SMP and bound EPS of sludge flocs in the operational period. One-way analysis of variance showed that there was no significant difference in the compositions of SMP and EPS between phase 1 and phase 2 ($p < 0.05$), indicating that SMP and EPS compositions were not significantly affected by the change of HRT. This result suggested that under similar SMP and EPS level, the different membrane fouling behaviour in the two phases was caused by other factors.

Figure 5 shows the PSD profiles which were determined at day 24, 93 and 126, respectively. It is apparent from Figure 5 that PSD at day 24 showed a bimodal distribution, while PSD at day 93 and 126 showed a unimodal distribution. They corresponded to d_{50} values of 31, 26 and 27 μm, respectively. A bimodal distribution at day 24

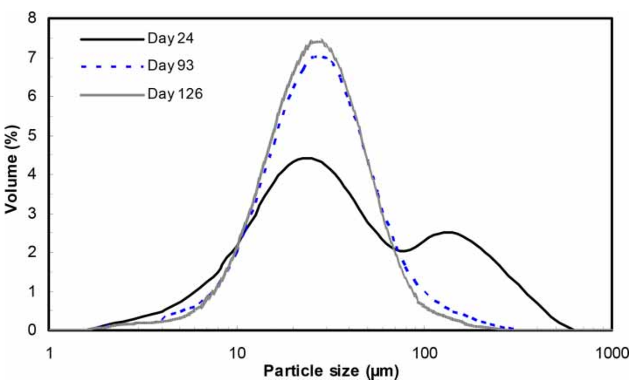

Figure 5 | Particle size distribution of the sludge suspension at days 24, 93 and 126, respectively.

indicated that deflocculation occurred in the early operation period. This could be attributed to the turbulent condition in the AnSMBR, which would break up the seeded sludge flocs and induce the release of small particles. Many studies also reported that deflocculated flocs, especially small particles or colloids, deposited on the membrane surface more easily, and then caused more severe membrane fouling (Meng et al. 2007; Lin et al. 2011). Our recent study showed that the interaction energy per unit mass between small flocs and membrane surface was much higher than that between large flocs and membrane surface (Hong et al. 2013). Therefore, particle size could partly explain the difference of the fouling rate at two operational periods in this study.

The current known membrane fouling mechanisms appeared unable to explain the great difference of SFR between the experimental result and calculated value in this study. Our recent studies have revealed a new fouling mechanism: osmotic pressure effect in cake layer filtration in MBRs (Chen et al. 2012; Zhang et al. 2013). EPS in the cake layer typically carry negatively charged functional groups (such as carboxyl and phenolic groups), causing the presence of large counter-ions within the EPS matrix for maintaining the electro-neutrality. The counter-ions present in one side of the separation medium would induce an osmotic pressure difference as compared to permeate. Due to the distinct EPS structure and cake layer filtration process, the existence of an osmotic pressure mechanism cannot be denied (Chen et al. 2012; Zhang et al. 2013). Therefore, the total filtration resistance of the cake layer should comprise two parts: hydrodynamic filtration resistance and the resistance originating from the osmotic pressure effect. The latter was generally the major contributor to the total filtration resistance (Chen et al. 2012; Zhang et al. 2013). According to the previous study (Chen et al. 2012), the significance of osmotic pressure was controlled by many factors including EPS level, charge density, dissociation constant of functional groups, pH condition, cake layer density and porosity. Table 2 shows the values of these parameters used for osmotic pressure calculation in this study. According to Equation (4), the calculated osmotic pressure ranged from 531 to 1920 kPa, which was much higher than the operational TMP (<50 kPa). The result showed that the osmotic pressure was much more overestimated in the calculation. The overestimation can be attributed to some factors not included in Equation (4). For example, the main force to retain the counter-ions in the EPS matrix is the electrostatic interaction which takes effect in the range of a Debye length. The Debye length is much lower than

Table 2 | Parameter values used for osmotic pressure calculation during cake layer filtration

Parameters	Value
EPS content	76 g/kg MLSS
Charge density	0.28 meq/kg MLSS
Dissociation constant (pK_a)	6.8
pH	7.0
Cake layer porosity	0.21–0.49
Cake sludge density	1.20 g/mL
Ion concentration of the permeate	0.005 mol/L

the size of the pores in the cake layer. This means that the efficiency of retaining counter-ions in the EPS matrix is quite low, and may be only a low percentage. However, even at this level, most of the TMP should be still used to resist the osmotic pressure, as hydrodynamic filtration resistance was minor and cannot fill the big gap of filtration resistance between experimental and calculated value. This mechanism thus provided a plausible explanation for the high SFR of the cake layer and the big SFR gap between experiment and calculation in this study. It should be noted that establishment of a primary mathematical framework regarding the osmotic mechanism is of great research interest to membrane fouling, and this will be the focus of our future work.

CONCLUSIONS

Around 90% of COD and 99% of turbidity and TSS in the sewage were stably removed by AnSMBR, and the permeate was suitable for urban or agricultural reuse. A sustainable flux of 11 LMH could be realized with biogas sparging. Small flocs in the sludge suspension could cause a high membrane fouling rate, and the high SFR of the cake layer was mostly attributed to the osmotic pressure mechanism. The results showed that the AnSMBR could be a promising alternative for sewage treatment.

ACKNOWLEDGEMENTS

Financial support of the National Natural Science Foundation of China (No. 51108424), Zhejiang Provincial Natural Science Foundation of China (No. LY13E080011), and Qianjiang Talent Project of Zhejiang Province (No. 2012R10061) is gratefully acknowledged.

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First received 29 October 2013; accepted in revised form 3 February 2014. Available online 18 February 2014