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Effects of EPS on membrane fouling in a hybrid membrane bioreactor for municipal wastewater treatment

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Abstract. A pilot-scale hybrid membrane bioreactor (HMBR) for real municipal wastewater treatment was developed by adding biofilm carriers into a conventional membrane bioreactor, distribution and dynamic changes of the extracellular polymeric substances (EPS) and their roles in membrane fouling were investigated. The results showed that the concentrations of loosely bond EPS (LB-EPS) and tightly bond EPS (TB-EPS) in activated sludge, carrier biofilm and sludge cake layer have been increased significantly with the running time of HMBR, during operation of the HMBR, EPS demonstrated positive correlations with membrane fouling. Compared to TB-EPS, LB-EPS showed more significant correlations with sludge physical properties and specific resistance to filtration (SRF) in HMBR, and thus demonstrated that LP-EPS have a stronger potential of fouling than TB-EPS. It was also found that a lower organic loading in HMBR could result a significant increase in EPS concentration, which would in turn influence membrane fouling in HMBR. This critical investigation would contribute towards a better understanding of the behavior, composition and fouling potential of EPS in HMBR operation.

Keywords: extracellular polymeric substances (EPS); membrane fouling; hybrid membrane bioreactor (HMBR); wastewater treatment

1. Introduction

As an efficient technology for municipal and industrial wastewater treatment, membrane bioreactors (MBRs) have gained increasing popularity in recent years. MBRs, in which solid/liquid separation is performed by membranes, offer several prominent advantages over conventional activatedsludge (CAS) system, including a higher biomass concentration, reduced footprint, less sludge production, and highly-improved effluent quality (Kimura *et al*. 2005, Miura *et al*. 2007, Wang *et al*. 2006). However, membrane fouling is a major obstacle for wide-spread applications of MBRs (Wang *et al*. 2009, Nagaoka and Akoh 2008). Membrane fouling results in reduced performance, severe flux decline or rapid trans-membrane pressure (TMP) increase, high energy consumption, and frequent membrane cleaning or replacement, which directly leads to the increase in maintenance and operating costs.

In order to control membrane fouling, many studies have been conducted. Among them, one

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attracting wide attention is a hybrid membrane bioreactor (HMBR) using suspended carriers as supporting media for biofilm development in the aeration tank. Compared with a conventional membrane bioreactor (CMBR) with only suspended biomass in the reactor, the HMBR has additional attached biomass and therefore the total biomass increases (Artiga *et al*. 2005). This not only improves the efficiency of biodegradation, but also enhances the nitrification process (Mtinch *et al*. 2000). Another advantage of the HMBR over the conventional membrane bioreactor (CMBR) was that it slowed down the increase of transmembrane pressure (TMP) so that a higher flux could be maintained without frequent chemical cleaning (Liu *et al*. 2012). However, a topic still remains on the mechanisms of membrane fouling control by the HMBR.

Extracellular polymeric substances (EPS), which are excreted by bacteria and composed of a variety of organic substances (Comte *et al*. 2006, FrØlund *et al*. 1996, Liu *et al*. 2011), are usually reported as a controlling factor of membrane fouling in MBRs (Chang *et al*. 2001, Drews *et al*. 2006, Nuengjamnong *et al*. 2005, Wang *et al*. 2012, Zhang *et al*. 2008). Although those intensive efforts mentioned above are very helpful to understand EPS characteristics and their impacts on membrane fouling in HMBR, the accumulation of knowledge to establish a general understanding of the role of EPS in membrane fouling in pilot-scale HMBR is insufficient. More detailed research on the EPS behaviours, their effects on fouling and EPS concentration control are needed in pilot-scale and full-scale HMBRs for real wastewater treatment in order to better understand the role of EPS in real HMBR applications.

In this study, a pilot-scale HMBR at an existing wastewater treatment plant (WWTP) was operated in order to investigate the properties of EPS and their role in membrane fouling. The correlations of bound EPS, loosely bound EPS (LB-EPS) and tightly bound EPS (TB-EPS) with membrane fouling in this pilot-scale MBR were also studied. The systematic investigation on EPS in the pilot-scale HMBR for real municipal wastewater treatment would provide valuable new insights into the characteristics of EPS and the effects on membrane fouling, and would consequently further advance our knowledge on the behavious of EPS in full-scale HMBR operation for real wastewater treatment.

2. Materials and methods

2.1 Pilot plant

The experimental work was conducted at Xi'an Beishiqiao Wastewater Treatment Plant (WWTP) in Xi'an, China. A pilot-scale HMBR system with a capacity of 4.8 m^3/day was installed. As shown in Fig. 1, the pilot system consisted of an aerobic reactor equipped with a submerged hollow-fiber microfiltration (MF) membrane module. The effective volume of the aerobic reactor was 2 m^3. By a perforated wall, the tank was partitioned into two compartments: one as the aeration tank with biofilm carriers, and another as the membrane tank holding the submerged MF module. Aeration was provided continuously by two fine bubble air diffusers to each compartment, one for aerating the suspension and driving the carriers to circulate and another for providing shearing force on the hollow-fiber membrane for fouling control.

2.2 Raw water characteristics

Table 1 shows the characteristics of the raw water that was fed to the HMBR from the inlet of the biological treatment unit of the WWTP.

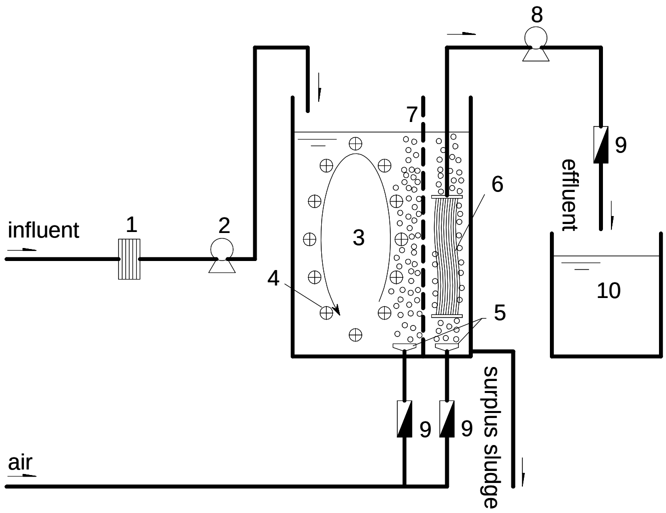

Fig. 1 Schematic diagram of the HMBR system. 1: screen, 2: feed pump, 3: aeration tank, 4: suspended carrier, 5: air diffuser, 6: hollow-fiber membrane module, 7: perforated wall, 8: suction pump, 9: flowmeter, and 10 permeate tank

Table 1 Characteristics of the influent to the pilot HMBR

COD (mg/L)	BOD_5 (mg/L)	NH_4^+-N (mg/L)	TN (mg/L)	TP (mg/L)	TSS (mg/L)	pH	Tempreture (°C)
240-896	185-423	20.3-46.8	22.5-54.1	4.3-13.2	200-1210	7.0-7.5	13.1-25.7

2.3 Operational conditions

At the start of the pilot operation, activated sludge from the oxidation ditch of the WWTP were seeded to the bioreactor to provide a mixed liquor suspended solids (MLSS) concentration of 3500 mg/L with a VSS/SS ratio of 0.58. Under stable operational condition, the sludge retention time was controlled through direct removal of sludge from the bioreactor (1/10 of the tank volume) on a daily basis for maintaining an SRT of 10 d. The operational conditions in the whole experimental period are given in Table 2.

The MF module operated under intermittent operation of the suction pump at an 8 min “on” and 2 min “off” cycle. Membrane cleaning was conducted as the transmembrane pressure (TMP) reached 20 kPa (Huang *et al*. 2001).

Table 2 Operation conditions of the pilot-scale HMBR system

Membrane flux ($L/m^2 \cdot h$)	DO (mg/L)	HRT (h)	SRT (d)	MLSS (mg/L)	Biofilm (mg/L)	Total biomass (mg/L)
10	0.4-3.5	10	10	3810-4350	1605-1750	5415-6100

This pilot experiment was continuously conducted for approximately one year. To better investigate the performance of HMBR, the experiment was conducted in two conditions, namely, Condition 1 (0-100 d) with the volume loading is average 0.565 kgCOD/m^{3}d, Condition 2 (101-200 d) with the volume loading is average 0.352 kgCOD/m^{3}d.

2.4 EPS extraction and analysis

The sludge samples for EPS extraction were the mixed liquor collected from the aeration room of the HMBR with MLSS concentration shown in Table 2. The suspended biomass concentrations were determined as MLSS. To measure the attached biomass on the carriers, 25 carriers which represented about 0.3% of the total number, were carefully collected from the reactor, and the attached biomass concentration was finally expressed as mg/L by considering the total number of carriers and the total volume of the reactor. EPS extraction were conducted by the following ways to obtain different microbial products (Domínguez *et al*. 2010a, b).

(1) Sludge samples were simply separated by two membrane to remove microbial cells (0.2 μm membrane) and low molecular weight metabolites (dialysis membrane of 3500 Da), the liquid obtained was centrifugated by high speed (20000 g), the microbial product loosely bound to the cells could be extracted as well as the soluble one, so the liquid obtained was considered to contain both the soluble and physically extractable microbial products which might roughly represent the S-EPS and loosely bound EPS (LB-EPS).

(2) The residues from the above-mentioned centrifugation and membrane filtration were resuspended using a buffer solution (2 mM Na_3PO_4, 4 mM NaH_2PO_4, 9 mM NaCl, 1 mM KCl, pH 7). The suspension was heated at 60°C for 30 min and then centrifuged and filtered with 0.2 μm and 3500 Da membranes. The liquid finally obtained contained mainly the heat extractable microbial product which might roughly represent the tightly bound EPS (TB-EPS).

The concentration of each part of the EPS extracted was measured as the total organic compound (TOC) using a TOC analyzer (1030 Aurora Sin, OI Analytical).

2.5 Characterization of the physical properties of the activated sludge

In this study, the supernatant turbidity of the mixed liquor from the aeration room was used as a parameter to characterize the flocculablity of the activated sludge. The sludge volume index (SVI) was used as a parameter to characterize the settleablity of the activated sludge. The suspended biomass was characterized by mixed liquor suspended solids (MLSS) in the aeration room, and the attached biomass was analyzed by direct measurement of the attached solid weight following Luostarinen *et al*. (2006).

2.6 Characterization of membrane fouling

In order to characterize the membrane resistance, attention was mainly paid to the specific resistance of the cake layer which was analyzed by collecting sludge from the cake layer on the outer surface of the hollow fiber, measuring its filtering property using a filtration device (Model 8200, Amicon, USA), and calculating its specific resistance to filtration. The transmembrane pressure (TMP) value was monitored using a vacuum gauge at different stages of membrane filtration (Lee *et al.* 2001), and the TMP value was used to represent the degree of membrane fouling.

3. Results and discussion

3.1 Variation of biomass and EPS concentration in a pilot-scale HMBR

Fig. 2 shows the variation of biomass in the bioreactor. During the start-up period (0-7d), no surplus sludge withdrawal was conducted and MLSS increased quickly. On day 12, as MLSS reached approximately 4300 mg/L, surplus sludge was removed on a daily basis and MLSS was almost kept at a constant level. At the same time, suspended carriers were added to the aeration tank and the attached biomass started to accumulate on the surface of the carriers. After about 45 d from addition of the suspended carriers, the attached biomass in the biofilm reached a level about 2000 mg/L and almost kept constant afterwards. As a result, the total biomass in the aeration tank became more than 6000 mg/L.

The experimental results related to the production of LB-EPS, TB-EPS and total EPS in mixed liquid (MLSS), biofilm, and sludge cake layer are shown in Figs. 3(a)-(c) for the whole duration of the system operation (100 days). It is clear that by increasing operation time from 1 to 100 days the value of total EPS gradually increases from 56.7 mg/g VSS to 123.0 mg/g VSS, 3.1 mg/g VSS to 81.5 mg/g VSS and 59.8 mg/g VSS to 204.5 mg/g VSS in mixed liquid (MLSS), biofilm, and sludge cake layer, respectively, with a increasing rate of 1.2 times, 25.1 times, and 2.6 times, respectively. TB-EPS behaves in a similar fashion with a increasing rate of 1.1 times, 21.7 times, and 2.3 times in mixed liquid (MLSS), biofilm, and sludge cake layer, respectively. Regarding TB-EPS, there is a increasing rate of 1.4 times, 13.5 times, and 2.9 times, in mixed liquid, biofilm, and sludge cake layer, respectively. The results indicate that the biomass in HMBR has a significant impact on the levels of LB-EPS and TB-EPS for operation time lower than 60 days. Operation time higher than 60 days have only a marginal impact on the concentration of TB-EPS, and there is almost no variation for LB-EPS concentration, this is attributed to the steady-state conditions, total biomass in the aeration tank became more than 6000 mg/L and almost kept constant afterwards, which result in the minimization of variations in sludge characteristics.

3.2 Relationship between EPS and activated sludge physical properties

During the pilot experiment of HMBR, sludge samples were continuously collected for EPS analysis, and the supernatant turbidity and SVI of the sludge were measured for an investigation of

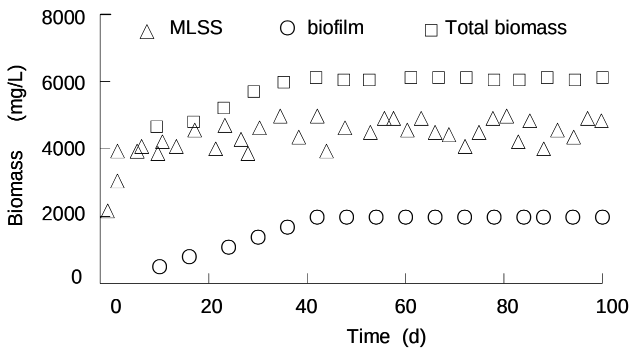

Fig. 2 Variation of biomass in the bioreactor

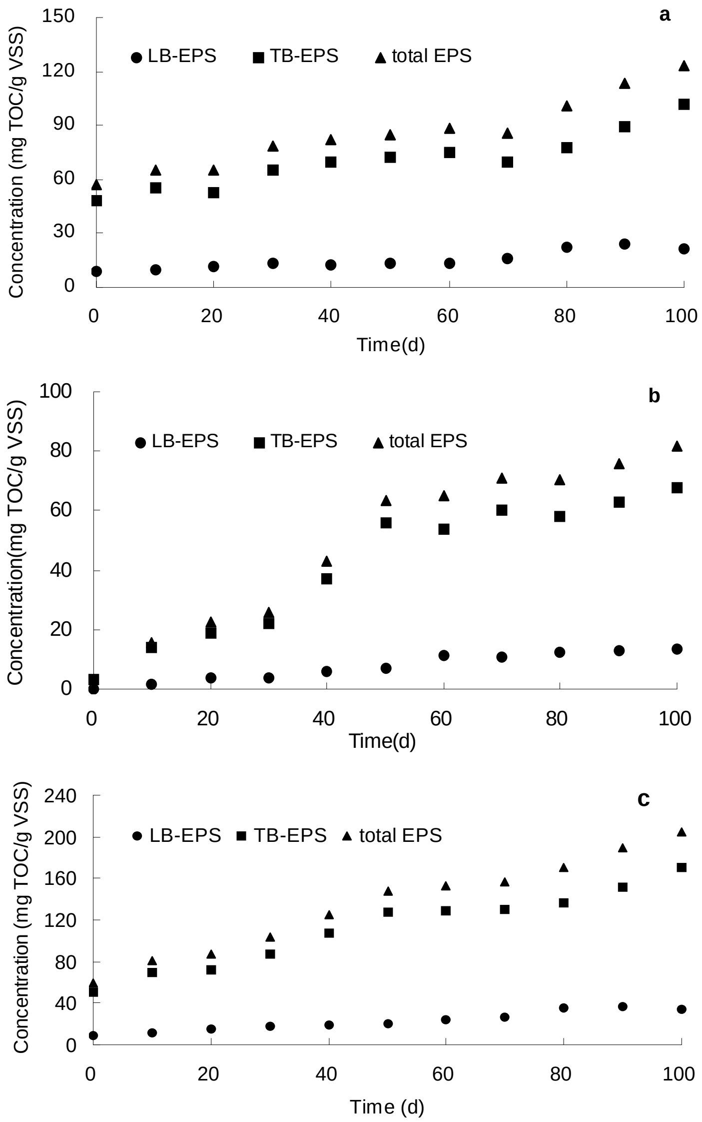

Fig. 3 Distribution and dynamic changes of EPS in HMBR: (a) EPS in mixed liquid (MLSS); (b) EPS in biofilm; and (c) EPS in sludge cake layer

the flocculability and settleability of the sludge particles. Figs. 4(a)-(d) showed that LB-EPS concentration closely related with the supernatant turbidity ($R^2 = 0.85$) and SVI ($R^2 = 0.83$), but the influence of TB-EPS concentration on supernatant turbidity ($R^2 = 0.20$) and SVI ($R^2 = 0.22$) were relatively small. It was apparent that as the LB-EPS concentration became higher, higher turbidity was measured from the supernatant, indicating a poorer flocculability of the activated sludge with fine particles. Meanwhile, at higher LB-EPS concentration, SVI also became higher, indicating a poorer settleability of the sludge floc. Ramesh *et al.* (2006) proved that the fouling resistance were primarily caused by the loosely bound EPS, but not by the tightly bound EPS. The loosely bound

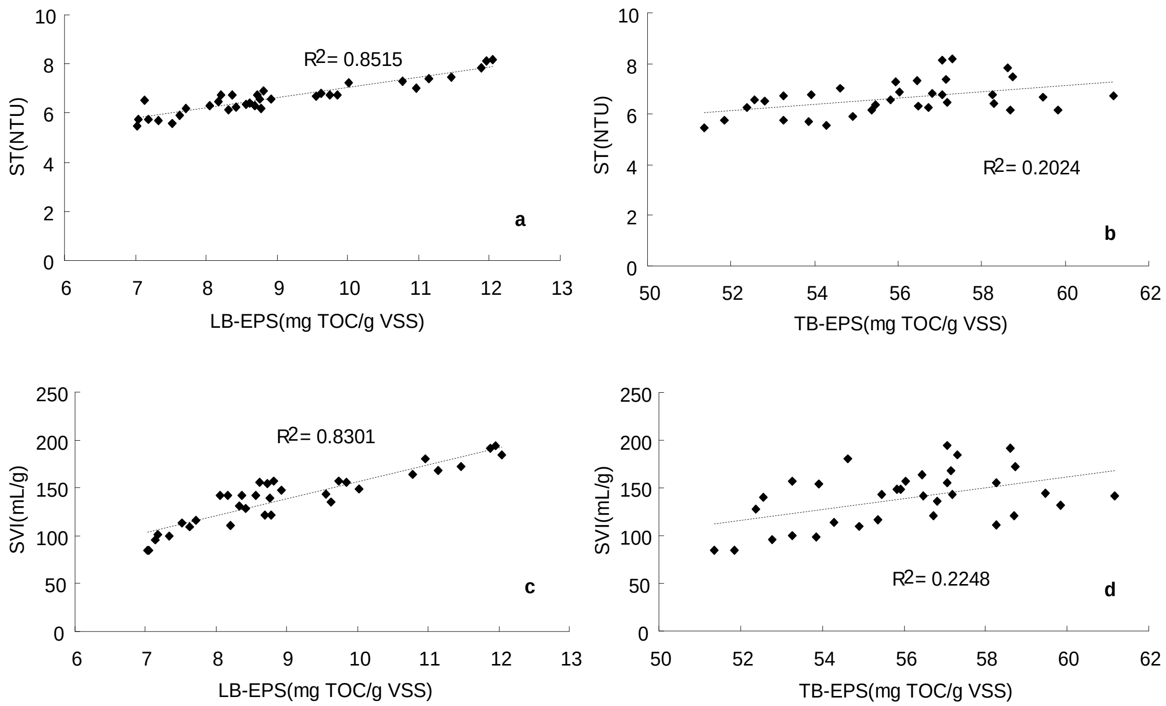

Fig. 4 Relationship between concentrations of EPS and activated sludge physical properties: (a), (b) Relationship between LB-EPS and TB-EPS and supernatant turbidity (ST) of the activated sludge, respectively; (c), (d) Relationship between LB-EPS and TB-EPS and sludge volume index (SVI), respectively

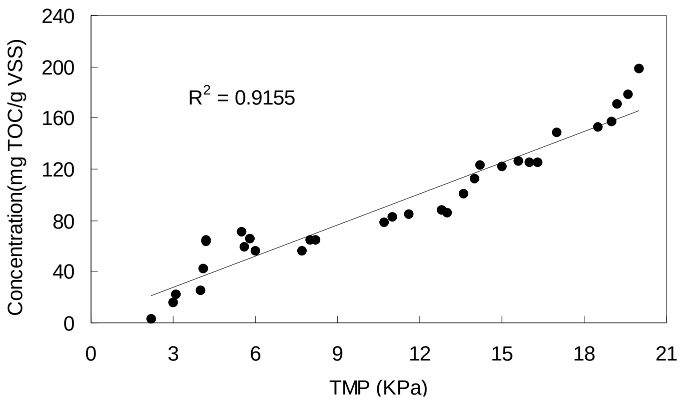

Fig. 5 Relationship between the concentrations of EPS and TMP in HMBR

EPS correlates with the performance of flocculation and sedimentation processes (Li and Yang 2007).

Several studies, however, reported that bound EPS had little correlation with membrane fouling. Rosenberger and Kraume (2003) found that contrary to some literature, no impact of bound EPS

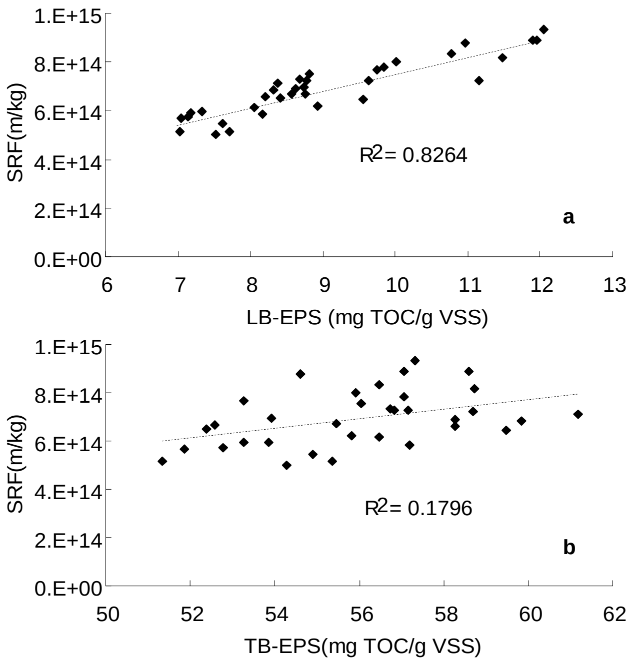

Fig. 6 Relationship between the concentration of EPS and specific resistance to filtration (SRF) in HMBR. a: Relationship between LB-EPS and SRF; b: Relationship between TB-EPS and SRF

on the filterability could be observed. Instead, the soluble EPS or SMP was found to have great impact on the filterability of sludge (Yamato *et al.* 2006). In order to have a better understanding of sludge characteristics and their effects on membrane fouling, several investigations have been carried out (Germain *et al.* 2005, Fan *et al.* 2006, Amer *et al.* 2011, Zuriaga-Agustí *et al.* 2013). These investigations showed that activated sludge has very complex impacts on membrane fouling process. Bound EPS cannot be considered as the sole cause for membrane fouling, even though it has great effects on sludge characteristics and membrane fouling.

Despite the fact that the research results on bound EPS are different from each other, it must be addressed that bound EPS concentrations are closely connected to sludge characteristics such as sludge volume index, flocculation ability, hydrophobicity, surface charge, sludge viscosity. Therefore, considering the important roles of bound EPS in sludge characteristics and membrane fouling, bound EPS should be controlled in order to mitigate membrane fouling more efficiently.

3.3 The roles of EPS on membrane fouling

Several research works have shown that EPS and SMP have a significant impact on membrane fouling based on batch tests or labscale studies (Ye *et al.* 2005a, b, Di *et al.* 2011, Amer *et al.* 2011a, b), in our pilot-scale study, showed as Fig. 5, EPS have been verified to be well correlated to membrane fouling (R^2 = 0.9155), and the increase of EPS concentrations could deteriorate membrane fouling. Fig. 6(a)-(b) shows the relationship between the concentration of EPS and specific resistance to filtration (SRF) in HMBR. It could be observed that influence degree of

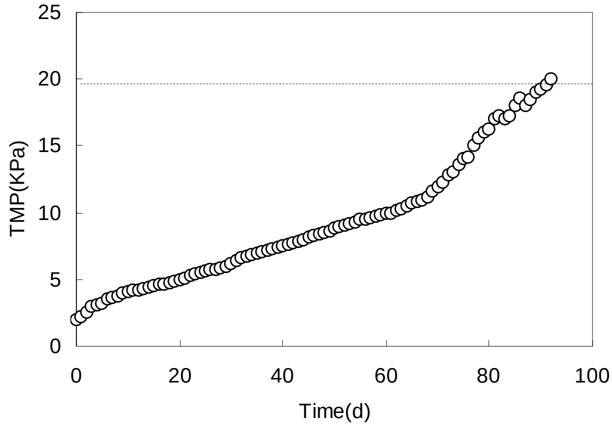

Fig. 7 Variation of TMP in the HMBR

LB-EPS on SRF (R^2 = 0.8264) was far more than that of TB-EPS on SRF (R^2 = 0.1796), indicating that TB-EPS have no obvious relations to membrane fouling rate while LB-EPS demonstrate positive correlations.

Fig. 7 shows the variation of TMP in the whole experimental period. When the TMP reached the prescribed maximum value of 20 kPa, the experiment underwent about 100 days. From the beginning of experiment, as the suspended carriers were added and the bioreactor operated as an HMBR, TMP accumulation was apparently slowed down compared with the bioreactor operated as a conventional membrane bioreactor (CMBR) (Wang *et al*. 2010), it was understood that the formation and maturation of the biofilm on the suspended carriers in HMBR resulted in much less accumulation of foulants on the membrane surface. From these results, it may be reasonable to draw a linkage between EPS concentration, sludge property, and cake layer formation on the membrane.

Current research are focused on the effect of EPS on MBR performance and on the effect of operating conditions (i.e., SRT, HRT) on sludge properties and on membrane fouling (Huang *et al.* 2000, Rosenberger *et al*. 2006, Zhang *et al*. 2006, Dvořák *et al*. 2011, Al-Halbouni *et al*. 2008). It has been reported that enhanced fouling occurs at low SRT due to increased levels of EPS (Ahmed *et al.* 2007, Tazi-Pain *et al*. 2002). However, some researchers have found that there is no correlation between EPS and fouling. Others found that EPS impact on membrane fouling only when found in specific ranges (Drews *et al*. 2008, Lee *et al*. 2003). In this study, we found that compared to TB-EPS, LB-EPS showed more significant correlations with membrane fouling, and thus we think how to reduce the concentration of LB-EPS is one of the important factor for membrane fouling control in MBR.

3.4 Effects of organic loading on EPS concentration in HMBR

To better investigate the performance of HMBR, the experiment was conducted in two conditions, namely, Condition 1 (0-100 d) with average volume loading about 0.566 kgCOD/m^{3}d and Condition 2 (101-200 d) with average volume loading about 0.352 kgCOD/m^{3}d. Table 3 shows the changes of biomass in HMBR under different operating conditions. Results indicated

Table 3 Variation of biomass in HMBR under different operating conditions

HMBR	Volume loading ($kgCOD/m^3d$)	MLSS (mg/L)	Biofilm (mg/L)	Total biomass (mg/L)
Condition 1	0.565	4279	2101	6380
Condition 2	0.352	2352	2350	4702
Increase or decrease	- 37.4%	- 45.0%	11.8%	- 26.3%

*Note. Value as the average

Table 4 Variation of EPS in HMBR under different operating conditions (mg TOC/g VSS)

HMBR	In MLSS		In biofilm		In HMBR			TMP (KPa)
	LB-EPS	TB-EPS	LB-EPS	TB-EPS	LB-EPS	TB-EPS	Total EPS	
Condition 1	13.9	86.8	19.2	56.5	33.1	143.3	176.4	9.6
Condition 2	19.1	124.5	27.7	101.6	46.8	226.1	272.9	11.8
Increase	37.4%	43.4%	44.2%	80.3%	41.3%	57.8%	54.7%	22.9%

*Note. Measured on day 40~60 for both conditions, value as the average.

that volume loading decreased 37.4% under Condition 2, compared with Condition 1, total biomass decreased 26.3% from 6380 mg/L to 4702 mg/L, but biofilm increased about 11.8% from 2101 mg/L to 2350 mg/L.

Table 4 shows the variation of EPS in HMBR under different operating conditions. Results showed that LB-EPS, TB-EPS and total EPS in mixed liquid (MLSS), biofilm significantly increased when the volume loading decreased from 0.566 $kgCOD/m^3d$ to 0.352 $kgCOD/m^3d$, and the TMP increased 22.9% under Condition 2, compared with Condition 1, indicating that a lower organic loading in HMBR could result a significant increase in EPS concentration, which would in turn influence membrane fouling in HMBR.

According to literature, there is no efficient approach to control the EPS directly since the MBRs include living microorganisms and their metabolites. Therefore, most of the recent reported literature is focused on finding suitable operating parameters in order to modify the sludge suspension. Sludge loading rate and correspondingly HRT and organic loading rate (OLR) are main operating parameters affecting the production of EPS since they govern biomass growth and decay. In addition, HRT can govern both the F/M of the bioreactor and the MLSS concentration. Meng *et al*. (2007) reported that there were high EPS concentrations and high sludge viscosity as F/M ratio increased. The formation of EPS is growth-related and is produced in direct proportion to substrate utilisation (Laspidou and Rittmann 2002). Thus, the increase of organic loading rate or F/M ratio will induce the generation of more EPS. On the contrary, in our pilot-scale experiment, it was found that a lower organic loading in HMBR could result a significant increase in EPS concentration.

In addition, SRT is one of the very important operating parameters affecting MBR performance, in particular membrane fouling (Grelier *et al*. 2006, Al-Halbouni *et al*. 2008). Cho *et al*. (2005) reported that as SRT decreased, the amount of EPS in sludge flocs increased at MLSS condition of 5000 mg/L. A recent investigation reported by Ng *et al*. (2006) showed that a longer SRT may improve membrane permeation (10 day and 20 day SRTs were better than 3 day and 5 day SRTs). They also observed that membrane fouling rate increased with rising EPS concentrations, both of which increased with decreasing SRT. Masse *et al*. (2006) found that EPS content decreased from 45-70 to 20-40 mg/gVSS when SRT increased from 10 to 53 d. The above-mentioned results suggest that too short SRT might do harm to membrane performance.

4. Conclusions

A pilot-scale HMBR process which employed suspended and attached biomass simultaneously in the aeration tank was operated for municipal wastewater treatment. The performance of the HMBR regarding the distribution of EPS and their roles in membrane fouling was studied. The following conclusions can be drawn from the experimental results.

- Concentrations of LB-EPS and TB-EPS in activated sludge, carrier biofilm and sludge cake layer have been increased significantly with the running time of HMBR, and the biomass in HMBR has a significant impact on the concentrations of LB-EPS and TB-EPS.
- Compared with TB-EPS, LB-EPS showed more significant correlations with sludge physical properties. It was apparent that as the LB-EPS concentration became higher, sludge turbidity and SVI also became higher, indicating a poorer flocculability and settleability of the sludge floc.
- EPS have been verified to be well correlated to membrane fouling (R^2=0.9155), and the increase of EPS concentrations could deteriorate membrane fouling. Inadditon, TB-EPS have no obvious relations to membrane fouling rate while LB-EPS demonstrate positive correlations, indicating that LP-EPS have a stronger potential of fouling than that TB-EPS.
- Lower organic loading in HMBR could result a significant decrease of total biomass, however the concentration of EPS increased significantly in HMBR, which would in turn influence membrane fouling in HMBR.

Acknowledgments

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