Short communication

Bisphenol A removal by a membrane bioreactor

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Abstract

Endocrine disrupting chemicals (EDCs) are potentially harmful chemicals during wastewater reclamation. Bisphenol A (BPA) is a typical EDC, and its removal using a submerged membrane bioreactor (MBR) was investigated. For comparison, a conventional activated sludge reactor (CASR) was simultaneously tested using the same BPA sludge loadings as the MBR. The results showed that MBR could remove BPA a little more effectively than CASR, despite changes in sludge loadings ranging from 0.046 to 10.2 g kg^{-1} d^{-1}. However, MBR could bear much higher volume loadings than CASR and still achieve the same BPA removal efficiencies. In MBR, HRT did not obviously influence the removal of BPA. The results also showed that the contributions of sludge adsorption to BPA removal were quite low in both reactors. In addition, one metabolite of BPA biodegradation, 4-hydroxy-acetophenone, was detected. These results suggested that biodegradation dominated the BPA removal process.
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Keywords: BPA; Membrane bioreactor; Removal; Adsorption; Biodegradation

1. Introduction

Wastewater reclamation has been attracting attention as a potential countermeasure for alleviating water shortage problems. To ensure the safety of reclaimed water, enhanced treatment for toxic trace chemical pollutants, such as endocrine disrupting chemicals (EDCs), is required. The membrane bioreactor (MBR) is an effective wastewater treatment process developed in recent decades, and is a potential technique for wastewater reclamation. Although the effluent quality of MBR is generally better than that of conventional treatment processes, it is unknown whether it is effective enough to remove EDCs. Thus, it is necessary to investigate the performance of the MBR in removal of EDCs.

Bisphenol A (BPA) is a typical EDC. It is used to synthesize polycarbonates and epoxy resins, flame retardants, and other specialty products [1,2]. Because of its wide use, BPA has been detected in many places in the world. The maximum concentrations of BPA detected were 17.2 mg l^{-1} in waste landfill leachates [3]. In China, the investigation in a typical municipal wastewater treatment plant found that there was 825 µg l^{-1} BPA in influent [4]. BPA was also adsorbed by the activated sludge, within a concentration range of 0.033–36.7 µg g^{-1} in Canada [5], 30–330 µg g^{-1} in Germany [6] and 28.3 µg g^{-1} in China [7]. The potential risks of BPA included reproduction and development effects, neurochemical effects and behavioral effects [8]. However, the low dose effect should not be neglected [9,10]. Some enhanced removal methods for BPA have been investigated [11–17], and the BPA-degradable bacteria have been isolated from activated sludge [18] and river water [19]. The degradation pathway has also been studied [20].

Some studies have shown that MBRs could remove more than 80% of nonylphenol and BPA from dumpsite leachate [21,22]. Other researchers found that both MBR and conventional activated sludge plants could remove some pharmaceutical substances [23]. All of these studies indicated that the MBR had the potential to remove EDCs, but the fate of EDCs in the treatment process was not yet clear. Moreover, because of the hydrophobicity of many EDCs, the excess sludge would also be a potential source of risks.

Thus, this study focused on BPA removal in a submerged MBR without active sludge discharge in order to reduce the secondary pollution. A conventional activated sludge reactor (CASR) was simultaneously tested, and the removal efficiencies of BPA at different BPA sludge loadings were compared. In addition, the contributions of adsorption and biodegradation to

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BPA removal were investigated. Preliminary investigation of BPA metabolites was also conducted to better understand the biodegradation behavior of BPA in MBR.

2. Materials and methods

2.1. Experimental equipment and operational conditions

A submerged membrane bioreactor and a conventional activated sludge reactor were used in the continuous experiments (Fig. 1(a) and (b)).

The volume of MBR was 10 l. A polyethylene hollow fiber membrane module (Korea Membrane Separation Co. Ltd., Korea), with an effective area of 0.2 m^2 and a nominal pore size of 0.4 μm, was used in the MBR. The CASR, with a total volume of 24.8 l, consisted of an aeration tank (20.4 l) and a settling tank (4.4 l).

Both reactors were seeded with activated sludge taken from Qinghe WWTP of Beijing, China. Before addition of BPA (Sigma–Aldrich, purity ≥97%), the sludge was first incubated in reactors for 3 weeks with the synthetic influent containing COD of 400 mg l^{-1}, NH_4^+-N of 35 mg l^{-1} and TP of 9 mg l^{-1}. Except for some necessary usages and the release with the effluent, sludge was not discharged from either reactor. In the MBR, aeration was applied with an air flow rate of 0.4–0.6 $m^3 h^{-1}$ to create turbulence for retarding membrane fouling, and the dissolved oxygen (DO) was within the range of 4–6 mg l^{-1}. For comparison, the DO in the CASR was controlled to approach the ranges in the MBR.

In the first stage, Run-1 to Run-3, the comparison of the BPA removal between MBR and CASR was conducted using the same BPA sludge loadings. Since the sludge was inactively discharged, the SRT of MBR achieved 350 d, but that of CASR was only 40 d because of the extra effluent SS. The HRTs of MBR and CASR were 8 and 11 h respectively, according to the normal state of these two types of processes. Different BPA sludge loadings were achieved by changing the influent concentrations of BPA from 0.1 to 5 mg l^{-1} and then to 20 mg l^{-1} corresponding to Run-1, Run-2 and Run-3, respectively. Moreover, in Run-3, the HRT of CASR was prolonged to slow the sludge decrease caused by the increased effluent SS.

In the second stage, Run-4 to Run-6, in order to investigate whether the HRT would influence the BPA removal in MBR, HRTs of MBR were shortened from 8 to 6 h and then to 4 h, while influent concentrations of BPA were kept constant.
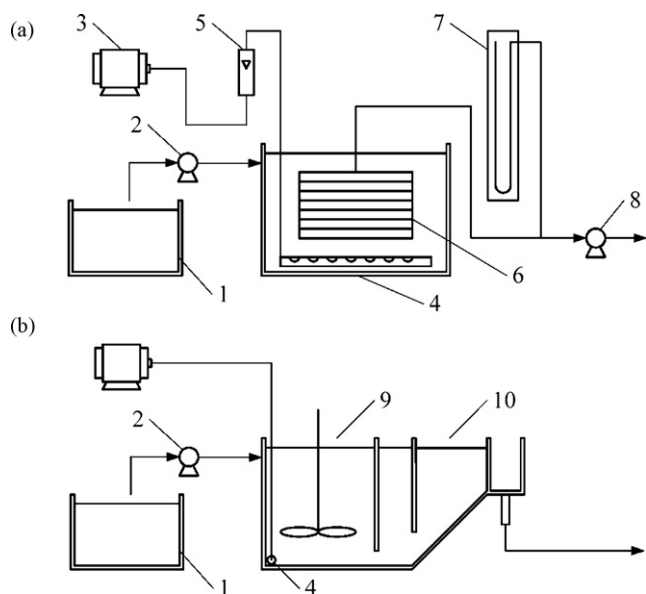

Fig. 1. Diagrams of the submerged MBR (a) and CASR (b). (1) Influent reservoir, (2) influent pump, (3) air compressor, (4) perforated diffuser, (5) air flow meter, (6) membrane module, (7) manometer, (8) suction pump, (9) aeration tank and (10) settling tank.

2.2. Batch experiments on sludge adsorption of BPA

Sludge adsorption tests of BPA were performed to study the fate of BPA in MBR and CASR. The adsorption isothermal curve was investigated, and the contribution of adsorption to BPA removal was calculated. The equilibrium time was previously determined to be 30 min.

The different equilibrium concentrations of BPA were achieved by spiking different concentrations of BPA (2.5, 5.0, 10.0, 20.0, and 40.0 mg l^{-1}) into the same masses of sludge. The sludge, taken from MBR and CASR before or after BPA addition, was centrifuged at 8000 rpm at 4 °C for 10 min, and was then softly washed. After adding sodium azide to sterilize the sludge, the mixture was placed in conical flasks and shaken at 150 rpm at 20 °C for 30 min, and then centrifuged at 8000 rpm at 4 °C for 5 min. Finally, the supernatant was filtered by a 0.45 μm membrane and the BPA concentrations were determined by HPLC. The recoveries were 81.7 ± 6.8% to BPA of 2.0 mg l^{-1}, 99.7 ± 5.7% to 5.0 mg l^{-1} and 99.0 ± 0.4% to 20 mg l^{-1}. The experiments were performed twice for each concentration level. The control samples without sludge were simultaneously processed under the same conditions. The results showed that BPA was steady during the entire process without sludge.

The specific adsorption coefficient K_D (l kg^{-1}) was calculated as the equation in Ref. [24].

2.3. Analytical methods

2.3.1. BPA

BPA was analyzed by HPLC (hp-1050, USA) with a reverse-phase C-18 column (Aichrombond-AQ C18, 4.6 mm × 250 mm, 5 μm, USA), a mobile-phase of water and acetonitrile (30:70), a flow of 1 ml min^{-1}, and a UV detector (hp-1040M, USA) at 278 nm at room temperature.

An additional solid-phase extraction (SPE) process was conducted for the lower concentration of BPA in Run-1. The SPE cartridge (Superlclean ENVI-18, Supelco, USA) was conditioned with 7.5 ml methanol and 7.5 ml purified water at a flow rate of 1 ml min^{-1}, then the sample was loaded at a rate of 5 ml/min. After being dried through overnight freezing-drying, the cartridge was eluted with 5 ml methanol at a rate of 1 ml min^{-1}. The eluate was dried with nitrogen at 40 °C, and redissolved in 0.5 ml methanol for HPLC analysis. The recovery of SPE was 100.6 ± 6.9% (n = 3).

2.3.2. Metabolites of BPA

To investigate the biodegradation of BPA, 100 ml of BPA (20 mg l^{-1}) was mixed with sludge, then the mixture was centrifuged and the supernatant was obtained for the measurement of BPA metabolites.

After acidification with 1 mol l^{-1} sulphuric acid to pH 2.5, the supernatant was loaded onto a SPE cartridge (Superlclean ENVI-18, Supelco, USA), which was conditioned with 5 ml methanol, 5 ml acetone and 5 ml purified water at a flow rate of 1 ml min^{-1}. Then the cartridge was dried with a soft nitrogen stream and eluted with 10 ml acetone at a rate of 1 ml min^{-1}. The eluate was dried with nitrogen at 40 °C, redissolved in 1 ml acetone, and analyzed by GC–MS. The GC–MS (DSQ, Thermo, USA) was operated in electron impact (EI) and full scan mode, with a DB-5MS capillary column (30 m × 0.25 mm i.d., 0.25 μm film, J&W, USA). The carrier gas is helium. The temperature program is from 70 °C (initial time, 2 min) to 300 °C (hold time, 30 min) at a rate of 10 °C min^{-1}.

3. Results and discussion

3.1. Comparison of BPA removal between MBR and CASR

In the first stage, comparison of BPA removal between MBR and CASR was conducted based on similar sludge loadings in both reactors.

In Run-1, the effluent concentrations of BPA decreased with time in both reactors. Before the 21st day, the effluent concentrations of MBR were almost all lower than those of CASR, but they tended to remain the same during the following
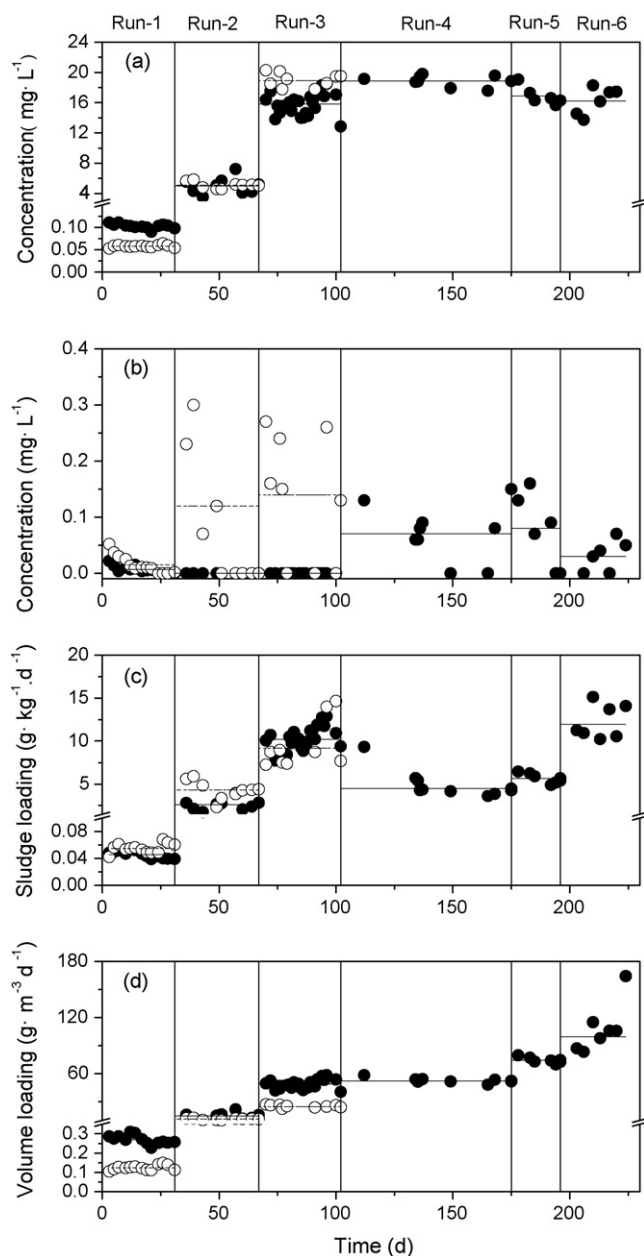
Fig. 2. Comparison of BPA removal between MBR (●) and CASR (○) with the average values of MBR (–) and CASR (· · ·). The HRTs were set as 8.0 ± 0.2 h for MBR from Run-1 to Run-3, and 8.0, 5.3 and 3.9 in Run-4, Run-5 and Run-6, respectively. The HRT was set as 11.0 ± 0.2 h for CASR from Run-1 to Run-2, but in Run-3, it was prolonged to 17.9 h to decrease the sludge release. The SRT was 350 d in MBR from Run-1 to Run-6, and 40 d in CASR from Run-1 to Run-3. (a) Influent concentrations of BPA. (b) Effluent concentrations of BPA. (c) BPA sludge loadings. (d) BPA volume loadings.

days (Fig. 2(b)). In Run-2 and Run-3, there were some fluctuations in the BPA concentrations of the influent because of the difficulty in dissolution of BPA. Because the higher concentrations after extraction may harm the HPLC column, the pretreatment of samples was not used in these two run periods. The concentrations of BPA in effluents of MBR were under the minimum limit of HPLC, but those of CASR were in the range of 0.07–0.27 mg l^{-1} on most sampling days (Fig. 2(b)). These concentrations were all higher than those

of MBR. The removal rates of MBR were on average higher than 93.7%, but those of CASR averaged 73.2, 97.8 and 99.3%, respectively.

The sludge loadings of BPA in MBR are also shown in Fig. 2(c). The average sludge loadings of BPA in MBR were 0.046, 2.6 and 10.2 g kg^{-1} d^{-1}, and those in CASR were 0.055, 4.3 and 9.2 g kg^{-1} d^{-1}. The sludge loadings of BPA in both reactors were at approximately equal levels. This showed that both MBR and CASR could effectively remove BPA despite the changes in sludge loadings. The MBR could remove BPA faster than CASR, but as time passed, the efficiencies of these two reactors tended to equalize.

The volume loadings of BPA in MBR were 0.3, 14.7 and 48.8 g m^{-3} d^{-1}, and those in CASR were 0.1, 11.4 and 24.8 g m^{-3} d^{-1}, corresponding to Run-1, Run-2 and Run-3, respectively (Fig. 2(d)). This showed that MBR could bear higher BPA volume loadings than CASR to achieve similar removal efficiencies because of its higher MLSS.

After adding BPA in higher concentrations (Run-2 and Run-3), the removal of BPA by both reactors was easier than expected. The variation of BPA effluent concentrations of MBR could not be shown directly because they were lower than the limitation of the analytical method on almost all of the sampling days, but the effluent concentrations of BPA of CASR showed a similar trend as the lower concentration run (Run-1). This showed that the acclimating procedure of sludge to BPA in MBR required less time than in CASR. This could be caused by the higher diversity of microorganisms in MBR.

To decrease the probable secondary pollution of BPA released from the excess sludge, we took a mode with inactive sludge discharge. The SRT in this mode reflects the characteristics of the reactors, that is, MBR could keep a very long SRT through the membrane rejection of the sludge, but CASR could only achieve a SRT of 40 d resulted by the effluent SS. According to the Monod kinetic model and experiments, a critical SRT for removal of BPA at 10 °C of 10 d was identified by Clara et al. [25]. He states that a complete removal of BPA would be expected with SRTs higher than this value. According to his equation, the critical SRT at 20 °C was calculated to be 5 d. In our experiments, the SRTs in MBR and CASR were 350 and 40 d, respectively, all much higher than this value. Therefore, the influence of the different SRTs between MBR and CASR should not be obvious.

3.2. Influence of HRT on BPA removal in MBR

In the second stage, to investigate the influence of HRT on BPA removal in MBR, the influent concentrations of BPA were kept at constant level, and HRTs were shortened from 8.0 to 5.3 h and then to 3.9 h. The results are shown in Fig. 2. The volumetric loadings of BPA increased from 52.2 to 74.4, and then gradually to 99.2 g m^{-3} d^{-1} (Fig. 2(c)), but the effluent concentrations of BPA were not obviously changed. The results showed that HRT did not obviously influence the BPA removal in MBR.

The BPA sludge loadings were also calculated and plotted in Fig. 2(d), which showed that the sludge loadings were almost in
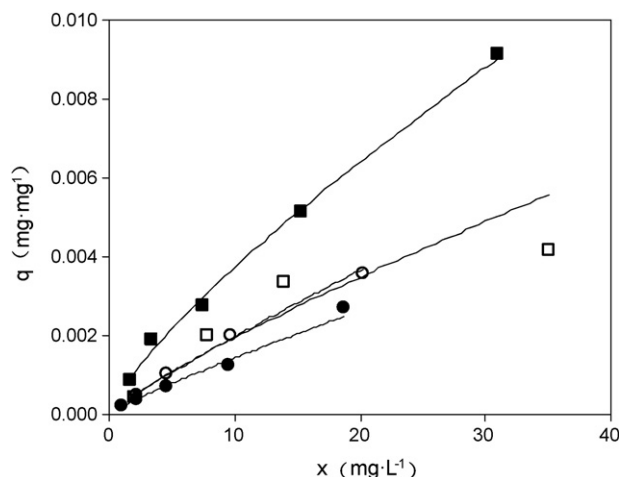
Fig. 3. The adsorption isothermal curves of BPA adsorbed by sludge from MBR before ($\bigcirc$) and after ($\bullet$) BPA was added and from CASR before ($\square$) and after ($\blacksquare$) BPA was added. The x represents the equilibrium concentration of BPA in liquor. The q represents the adsorbed BPA in sludge when in equilibrium. The fitting equations for MBR were $q = 0.0003x^{0.8897}$ ($R^2 = 0.9985$) and $q = 0.0002x^{0.8721}$ ($R^2 = 0.9947$) before and after BPA was added, respectively. Those for CASR were $q = 0.0003x^{0.8405}$ ($R^2 = 0.9221$) and $q = 0.0006x^{0.7810}$ ($R^2 = 0.9863$) before and after BPA was added, respectively.

the same ranges as in the first stage. Although the effluent concentrations of BPA were frequently higher than the detection limit of HPLC, they were still lower than those of CASR in the first stage.

3.3. Contribution of sludge adsorption to BPA removal

The adsorption isothermal curves are shown in Fig. 3. The fitting results showed that the sludge adsorption of BPA followed the Freundlich adsorption. This could match the experimental results reported by Clara et al. [24]. Before adding BPA into the reactors, the adsorbability of sludge in MBR was almost same as that of CASR. After BPA had been in the reactors for a certain time, the adsorbability changed slightly. However, the range of log K_D was from 2.00 to 2.75, which was similar to the reported value between 1.89 and 2.41 [24]. This result suggests that the differences of adsorption isothermal curves between the sludge of MBR and CASR might not be caused by the differences in sludge characteristics, but by the differences between experiments.

The masses of adsorbed BPA were calculated based on the experimental adsorption isothermal equations (Table 1). The

results showed that the percentages of adsorbed BPA to total removed BPA were quite low. Therefore, the sludge adsorption did not dominate the BPA removal in both MBR and CASR.

3.4. The metabolite of BPA biodegradation in MBR

Further investigation of BPA biodegradation was performed by preliminary detection of BPA metabolites. One of the metabolites of BPA biodegradation by bacterium [20], 4-hydroxy-acetophenone (Fig. 4(b)), was detected in the sample. It proved that BPA could be biodegraded by bacteria in activated sludge of MBR.

3.5. Further discussion

Firstly, BPA losses due to evaporation could be ignored because BPA is low-volatility. The vapor pressure of BPA is 5.3 μPa, and the calculated Henry's constant is 10 μPa m^3 mol^{-1} [2]. Chemicals with Henry's constant lower than 10 mPa m^3 mol^{-1} are considered low-volatility, as the chemical is less volatile than water [2]. Secondly, as was our initial assumption, the adsorption of BPA should be an important contribution to BPA removal. If such was the case, the effluent concentrations of BPA should increase with the continuous injection of the influent. However, the effluent concentrations of BPA either continuously decreased or stayed below detection limit in experiments. This result suggests that biodegradation occurred. Furthermore, adsorption contributed very little to the removal, according to the calculation of the adsorbed BPA, so biodegradation dominated the removal. What should be emphasized is that sludge adsorption might play an important role in the process of BPA removal. In both activated and inactivated sludge, there was immediate adsorption of BPA in the initial period; subsequently BPA concentration decreased gradually in activated sludge but remained steady in inactivated sludge (data not shown). This suggests that microorganisms might need contact with the BPA through adsorption in order to achieve biodegradation. Similar phenomena were also reported by Urase and Kikuta [26]. Thirdly, the detection of intermediate products of BPA biodegradation could also help demonstrate the importance of biodegradation.

Many studies have reported that there are bacteria capable of degrading BPA [27]. Because of the rejection by membranes, the amount of microorganisms in MBR was higher than that in CASR (Table 1), and the diversity of microorganisms in MBR

Table 1
The contributions of BPA adsorption to BPA removal in MBR and CASR

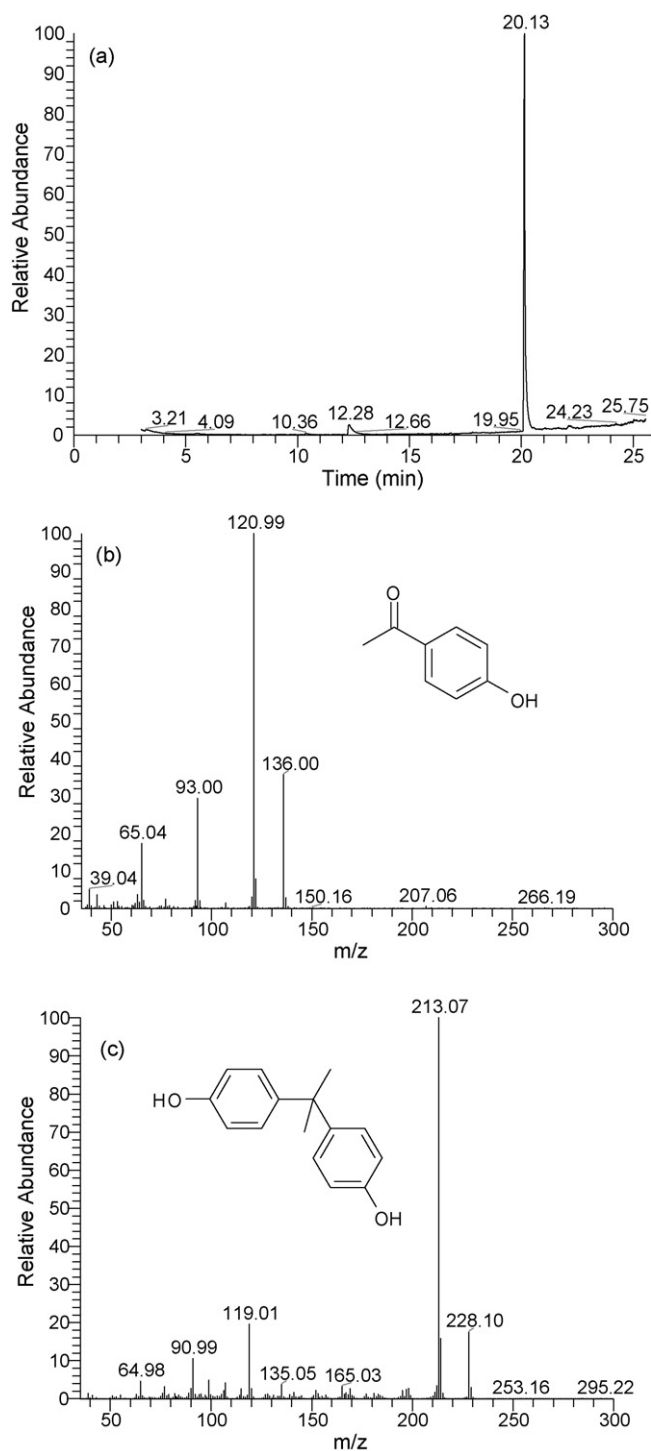|	MBR						CASR		
	Run-1	Run-2	Run-3	Run-4	Run-5	Run-6	Run-1	Run-2	Run-3
Concentration of BPA in mixed liquor (mg l^{-1})	0.007	ND	ND	0.07	0.08	0.03	0.016	0.12	0.14
MLSS (mg l^{-1})	5200	5550	5700	12,130	13,670	9060	2300	3230	2800
Mass of adsorbed BPA (mg)	0.1	–	–	2.4	3.0	0.8	1.1	7.6	7.4
Mass of added BPA (10^3 mg)	0.07	5.1	21.9	36.1	14.5	20.7	0.0725	8.1	25.9
Percentage of adsorbed to added (%)	0.19	–	–	0.01	0.02	0.004	1.35	0.09	0.03

"ND" means not detected.

Fig. 4. (a) Chromatograph of BPA and one of its intermediates, 4-hydroxy-acetophenone, by GC–MS. The retention time of BPA was 20.13 min, and that of 4-hydroxy-acetophenone was 12.28 min. (b) Spectrum of 4-hydroxy-acetophenone. The molecular structure is also shown. (c) Spectrum of BPA. The molecular structure is also shown.

was different with that of CASR [28]. Thus, the BPA removal in MBR was faster than in CASR. Nevertheless, as time passed without discharge of sludge, the microorganisms in CASR could also remove BPA effectively, probably because of the increase of the BPA-degradable bacteria. Thus, the efficiencies

of BPA removal in MBR and CASR tended to be same over longer periods.

Finally, because of the hydrophobicity of BPA, the adsorption of BPA into suspended solid and organic matter [29] should also be considered [30]. In this case, the rejection of the membrane might provide a safer method for decreasing the risk of released EDCs with effluent. There should be more investigations into this area.

4. Conclusions

The presented results concluded that the MBR could remove BPA slightly more effectively than CASR under conditions of equal sludge loadings ranging from 0.046 to 10.2 g kg^{-1} d^{-1}. However, MBR could bear much higher volume loadings than CASR and still achieve the same BPA removal efficiencies. In MBR, HRT did not obviously influence the removal of BPA. Although BPA is hydrophobic, the contribution of sludge adsorption to removal was quite low, and biodegradation dominated the BPA removal process. The BPA biodegradation by sludge in MBR was supported by the detection of one metabolite, 4-hydroxy-acetophenone.

Acknowledgement

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