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Impact of calcium-to-magnesium ratio on the performance of submerged membrane bioreactors

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ABSTRACT

The performances of membrane bioreactors (MBRs) were compared in bench-scale submerged MBRs treating synthetic wastewater at three Ca:Mg ratios of 3:1, 1:1, and 1:3 with constant divalent cations of 200 mg/L. The dosages of Ca^{2+} were 150, 100, and 50 mg/L, corresponding to Mg^{2+} of 50, 100, and 150 mg/L for Ca:Mg ratios of 3:1, 1:1, and 1:3, respectively. The results showed that Ca:Mg ratio had a slight impact on effluent quality, while membrane fouling was effectively mitigated by enlarging Ca:Mg ratio due to the decline of fouling layer resistance. When Ca:Mg ratio declined from 3:1 to 1:3, the concentration of soluble microbial products (SMP) significantly increased, while the particle size of flocs decreased. SMP and particle size correlated well with the variation of membrane fouling. The carbohydrate in SMP and bound extracellular polymeric substances (BEPS) changed significantly with the decline of Ca:Mg ratio, while the protein in SMP and BEPS varied slightly, suggesting that the divalent cations were more prone to bridging carbohydrate compared with protein. In comparison with Mg^{2+}, Ca^{2+} had a more significant impact on mitigating membrane fouling, because Ca^{2+} was more beneficial to binding and bridging negatively charged functional groups in extracellular polymeric substances such as carbohydrate.

Keywords: Membrane bioreactor; Calcium ion; Magnesium ion; Membrane fouling; Soluble microbial products (SMP)

1. Introduction

Membrane bioreactors (MBRs) combining membrane separation and activated sludge process have been increasingly applied for municipal and industrial wastewater treatments due to their potential advantages over conventional activated sludge process such as better effluent quality, smaller footprint, less sludge production, etc. [1–3]. However, membrane fouling leads to performance reduction, rapid pressure increase, or severe flux decline and frequent membrane cleaning, which directly result in the increase of maintaining and operating costs. Therefore, membrane fouling has become a major obstacle for widespread applications of MBRs.

According to the divalent cation bridging (DCB) theory, calcium and magnesium cations play a major role in bridging negatively charged functional groups within extracellular polymeric substances (EPS), which helps to aggregate and stabilize the matrix of biopolymer and microbes, and then promotes bioflocculation [4,5]. The enhanced bioflocculation by divalent cations could be closely linked with membrane fouling through the EPS characteristics. It has been reported that the presence of optimum quantity of Ca^{2+} could

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be beneficial in reducing the steady state fouling rate in the long-term operation due to its positive effect on sludge flocculation ability [5–9]. Furthermore, the impacts of Ca^{2+} to Mg^{2+} ratio on sludge properties were investigated. It was found that Ca:Mg ratio had a significant effect on floc strength, settling, and dewatering of activated sludge [10,11]. Unfortunately, these reports only focused on the Ca:Mg ratio's role in bioflocculation of sludge flocs, but did not at all address membrane fouling. Given that membrane fouling was not only impacted by sludge properties, the conclusions on the influence of Ca:Mg ratios on membrane fouling could not be directly drawn from these papers.

Arabi and Nakhla [12] have compared the characteristics of membrane fouling in submerged MBRs at three Mg:Ca molar ratios (1:5, 1:1, and 5:1), and found that the higher Mg^{2+} at larger Mg:Ca ratio is beneficial in reducing membrane fouling due to changes in EPS and soluble microbial products (SMP) characteristics as well as molecular weights. However, in their research, the total concentration of divalent cations changed in the exception of Mg:Ca ratio due to maintaining Ca^{2+} constantly at about 1.7 mM. Considering the fact that the ionic strength has a significant impact on membrane fouling due to double layer compression and charge screening [13], it is in doubt whether the variations of membrane fouling are only due to the increase of Mg:Ca ratio. Therefore, more works are needed to investigate the effect of Ca:Mg ratio on membrane fouling.

Thus, the objective of this research was to investigate the impact of Ca:Mg ratio on sludge characteristics and membrane fouling in MBRs with the constant sum of Ca^{2+} and Mg^{2+}. The bench-scale submerged MBRs were operated at three Ca:Mg ratios of 3:1, 1:1, and 1:3 with total Ca^{2+} and Mg^{2+} concentrations of 200 mg/L. The transmembrane pressure (TMP) and sludge properties such as mixed liquor suspended solids (MLSS), BEPS, SMP, and particle size distribution were investigated. The results obtained in this research are expected to provide a sound understanding of membrane fouling mechanisms in the process of adding Mg^{2+} and Ca^{2+}.

2. Materials and methods

2.1. Experimental setup and operation

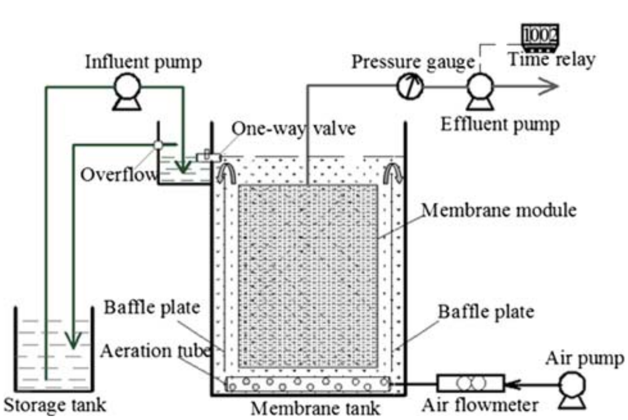

Fig. 1. Schematic diagram of the bench-scale submerged MBR.

As shown in Fig. 1, the bench-scale submerged MBR with an effective volume of 30 L was divided into a riser zone and two down-comer zones by two baffle sheets. A flat sheet membrane module (supplied by Zizheng Environment Incorporated, Shanghai, China) installed in the riser zone was made of polyvinylidene fluoride (PVDF) with nominal pore size of 0.20 μm, and its effective filtration area was 0.175 m^2. Continuous aeration was provided through an axial perforated tube below the membrane module in order to supply oxygen for micro-organisms and induce a cross-flow velocity (CFV) along the membrane surface for reducing membrane fouling. The effluent flow rate and TMP were monitored by a water flow meter and a pressure sensor, respectively. A one-way valve and an overflow tube were used for maintaining a constant water level in the reactor over the whole experiment.

Three MBRs with different Ca:Mg ratios and a control MBR were operated to investigate the impact of Ca:Mg ratio on sludge characteristics and membrane fouling. As for the three MBRs, $MgCl_2$ and $CaCl_2$ were added to influent water to maintain Ca: Mg ratios of 3:1, 1:1, and 1:3 and the total divalent cations of 200 mg/L, respectively. The dosages of Ca^{2+} were 150, 100, and 50 mg/L, corresponding to Mg^{2+} of 50, 100, and 150 mg/L, and the ionic concentrations of the divalent cations were 5.8, 6.6, and 7.4 mM for Ca: Mg ratios of 3:1, 1:1, and 1:3, respectively. Both Ca^{2+} and Mg^{2+} were not added to influent water of the control reactor. The operating conditions of the four MBRs were the same except for the dosage of Ca^{2+} and Mg^{2+}. The MBRs were operated under constant flux mode of 18 L/(m^2 h), and the intermittent filtration mode, i.e. 10 min suction followed by 2 min relaxation (nonsuction) was adopted. It should be noted that in order to maintain the hydraulic retention time (HRT) at about 5 h, another flat sheet membrane was immersed in the reactor to increase the effluent flow. The flux of the membrane used for balancing flow rate was maintained at 12 L/(m^2 h) in the whole operation;

thus, the total membrane flux of the reactor was 30 L/(m^2 h), which ensured that the HRT of the MBRs was about 5.7 h. A target sludge retention time (SRT) of 20 d was maintained through direct removal of sludge from the bioreactor on daily basis. The aeration rate was 1.0 m^3/h, and a temperature controller was used in the MBRs to maintain the temperature in the range of 25 ± 2℃. Chemical cleaning procedure would be carried out if the TMP reached about 25 kPa during the operation. The membrane module was taken out and soaked in 0.5% (v/v) NaClO solution for about 2 h to recover its permeability.

2.2. Composition of synthetic wastewater

The influent to the MBRs was synthetic wastewater made by adding 230.0 mg/L glucose, 40.0 mg/L sodium acetate anhydrous, 60.0 mg/L peptone form fish, 20.0 mg/L beef extract, 170.0 mg/L NH_4HCO_3, 12.0 mg/L KH_2PO_4, 1.2 mg/L $CaCl_2$, 2.4 mg/L $MgCl_2 \cdot 6H_2O$, 1.0 mg/L $FeCl_3 \cdot 6H_2O$, and 0.05 mg/L $CoCl_2 \cdot 6H_2O$ to deionized water. The influent concentrations of chemical oxygen demand (COD), ammonium nitrogen (NH_3–N), total nitrogen (TN), and total phosphorus (TP) were 373.3 ± 18.5 mg/L, 33.1 ± 1.5 mg/L, 46.8 ± 3.8 mg/L, and 3.18 ± 0.25 mg/L, respectively. Additionally, $NaHCO_3$ with the concentration of 198.0 mg/L was used to maintain the pH at 6.8–7.2.

2.3. Analytical methods

Measurements of COD, NH_3–N, nitrate nitrogen (NO_3–N), TN, TP, and pH in the influent and membrane effluent, MLSS, and mixed liquor volatile suspended solids (MLVSS) in the system were performed according to the Chinese NEPA standard methods [14]. The dissolved oxygen (DO) concentration in the reactor was measured by a dissolved oxygen meter (Model YSI 58, YSI Research Incorporated, OH, USA). Particle size distribution was determined by BT-2003 Laser Particle Size Analyzer (Bettersize Instruments Ltd., Dan Dong, China) with a particle size range of 0.04–1,000 μm.

Mixed liquor with the volume of 25 mL was centrifuged in a 50 mL tube at 8,000*g* for 10 min, and then the supernatant was filtered through a membrane with mean pore size of 0.45 μm. Organic substances in the filtrate were termed SEPS or SMP [15]. The sludge pellet was re-suspended in 25 mL of 0.9% NaCl solution and heated at 80℃ in a water bath for 30 min. After that, it was centrifuged at 12,000 × *g* for 15 min to obtain the supernatant, which was regarded as the BEPS of the sludge. Both the SMP and BEPS extractions were normalized as the sum of carbohydrate and protein. Carbohydrate was measured by the phenol-sulfuric acid method with glucose as a standard [16]. Protein was measured by a modified Lowry method [17], and bovine serum albumin (BSA, Sigma fraction V, 96%) was used as protein standard. All the above analyses were conducted in duplicate, and their average values were reported. An analysis of variance (ANOVA) was used to test the significance of results, and $p < 0.05$ was considered to be significant.

2.4. Resistance analysis

According to the resistance-in-series model on the relationship between permeate flux and TMP, the hydraulic resistance at each Ca:Mg ratio was calculated using the following equation:

$$R_t = R_m + R_c + R_f = \frac{\Delta P}{\mu J} \qquad (1)$$

where R_t is the total membrane resistance (m^{-1}), R_m is the intrinsic membrane resistance (m^{-1}), R_c is the fouling layer resistance (m^{-1}), R_f is the fouling resistance due to irreversible adsorption and pore plugging (m^{-1}), J is the flux (L/(m^2 h)), ΔP is the TMP (Pa), and μ is the dynamic viscosity of permeate water (Pa s). R_m is obtained by filtrating pure water through the new membrane before operation, and R_t could be calculated from the final flux and TMP values at the end of the operation. $R_m + R_f$ is measured by filtration of pure water through the membrane that removed the fouling layer by washing the membrane with a sponge. From these values, each of R_t, R_m, R_c, and R_f can be obtained using Eq. (1) [18].

3. Results and discussion

3.1. Process performance

The characteristics of influent and effluent water in the MBRs are summarized in Table 1. It is evident from Table 1 that the quality of effluent water was similar at all reactors, i.e. the COD and NH_3–N concentrations in effluent were lower than 40 mg/L and 1 mg/L, respectively. This observation is consistent with Arabi and Nakhla [12]. In order to further analyze the impacts of Ca:Mg ratio on the performance of MBRs, the ANOVA was used to test statistical differences from the control. As shown in Table 1, the Ca:Mg ratio did not give any significant impact on effluent water of the MBR ($p > 0.05$).

Table 1
Characteristics of the influent and effluent water in the MBRs (mg/L)[a]

	Control	Ca:Mg = 3:1	Ca:Mg = 1:1	Ca:Mg = 1:3
Influent COD	366 ± 15	367 ± 17	374 ± 12	372 ± 7
Effluent COD	26 ± 6	30 ± 8	24 ± 6	22 ± 5
$P_{(0.05)}$		0.970	0.970	0.817
Influent NH_3–N	29 ± 2	30 ± 2	33 ± 2	33 ± 1
Effluent NH_3–N	0.33 ± 0.20	0.28 ± 0.23	0.12 ± 0.10	0.14 ± 0.04
$P_{(0.05)}$		0.981	0.447	0.447
Influent NO_3–N	1.37 ± 0.21	3.28 ± 0.12	2.31 ± 0.10	2.21 ± 0.05
Effluent NO_3–N	29 ± 0.5	31.3 ± 3.4	34.3 ± 2.1	31.6 ± 1.6
$P_{(0.05)}$		0.588	0.067	0.496
Influent TN	42.2 ± 2.3	40.5 ± 2.0	40.0 ± 2.9	43.7 ± 0.7
Effluent TN	36 ± 4.5	34.7 ± 4.3	37.9 ± 2.1	34.5 ± 1.9
$P_{(0.05)}$		0.967	0.903	0.948
Influent TP	3.6 ± 0.26	3.34 ± 0.18	3.49 ± 0.21	3.47 ± 0.17
Effluent TP	2.5 ± 0.4	2.0 ± 0.4	1.8 ± 0.2	1.9 ± 0.2
$P_{(0.05)}$		0.286	0.100	0.171

Notes: [a]Values are given as mean values ± standard deviation (number of measurements: $n = 6$ for all reactors). $P_{(0.05)} < 0.05$ represents statistical differences from control.

The variations of TMP with operation time under different ratios of Ca:Mg are demonstrated in Fig. 2. It could be observed from Fig. 2 that the TMP increasing rate was lower in the three reactors with addition of Ca^{2+} and Mg^{2+} than that in the control reactor, indicating that membrane fouling was mitigated by adding divalent cations. This observation is consistent with other reports [5–9,12]. Furthermore, it could also been seen that the fouling rate increased with the reduction of Ca:Mg ratio, i.e. the chemical cleaning process during the same operating time was carried out 1 time, 2 times, and 4 times for Ca:Mg ratios of 3:1, 1:1, and 1:3, respectively. It might be suggested that Ca^{2+} had a more significant impact on mitigating membrane fouling compared with Mg^{2+}. However, Arabi and Nakhla [12] have reported that higher Mg^{2+} concentration is more beneficial in reducing membrane fouling than Ca^{2+} through comparing membrane fouling rate in submerged MBRs at three Mg:Ca molar ratios. The contradiction might be attributed to the different adding modes of Ca^{2+} and Mg^{2+}. In their research, the ionic concentrations of the divalent ions were 2.0, 3.4, and 10.2 mM for Mg:Ca molar ratios of 1:5, 1:1, and 5:1, respectively, while they were 5.8, 6.6, and 7.4 mM for Ca:Mg ratios of 3:1, 1:1, and 1:3 in this study, respectively. It is true that the total ionic concentration slightly changed in this study, but its impact was rather small. Given that the ionic strength has a significant effect on membrane fouling due to double layer compression and charge screening [13], the variations of membrane fouling could be attributed to combining effects of Mg:Ca ratio and ionic strength in their study, while mainly due to the impacts of Mg:Ca ratio in our study.

3.2. Sludge properties

3.2.1. MLSS and MLVSS

The concentrations of MLSS and MLVSS and the MLVSS/MLSS ratio in the MBRs under different Ca:Mg ratios are listed in Fig. 3. As shown in Fig. 3, the dosage of Ca^{2+} and Mg^{2+} did not give any significant impact on MLSS and MLVSS ($p > 0.05$). However, the ratio of MLVSS to MLSS tended to obviously decline when Ca^{2+} and Mg^{2+} were added to the MBRs. It might be due to the accumulation of divalent cations in the MBRs with the addition of Ca^{2+} and Mg^{2+} [9]. Based on the above results, it could be concluded that the lower fouling rate observed at higher Ca:Mg ratio in this study was not related to MLSS and MLVSS.

3.2.2. EPS

It is well recognized that EPS play an important role in membrane fouling in the MBRs [19,20]. Based

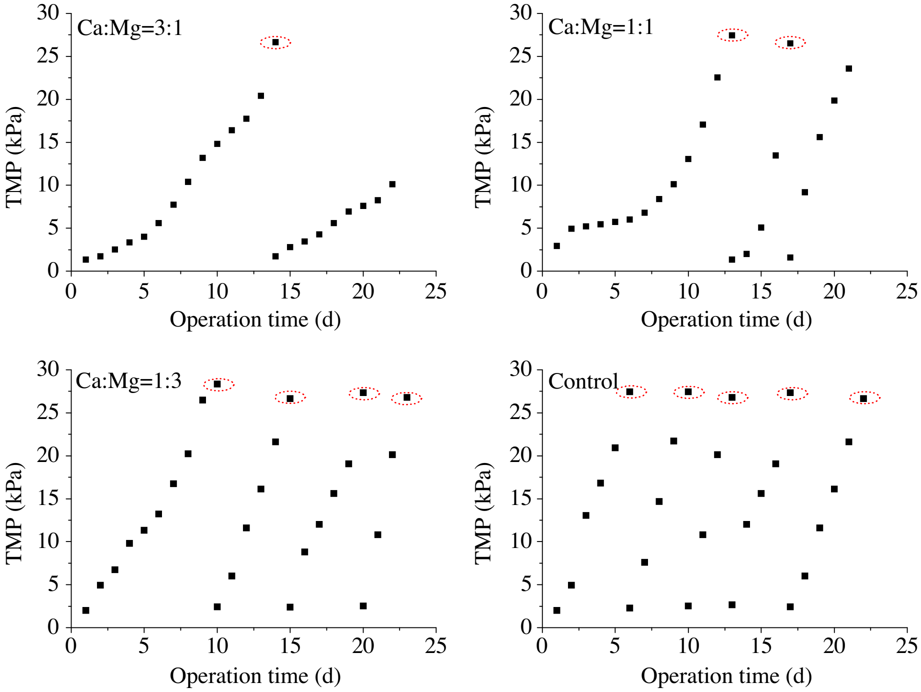

Fig. 2. TMP variations in the MBRs at different Ca:Mg ratios. Dashed circle represents the chemical cleaning process.

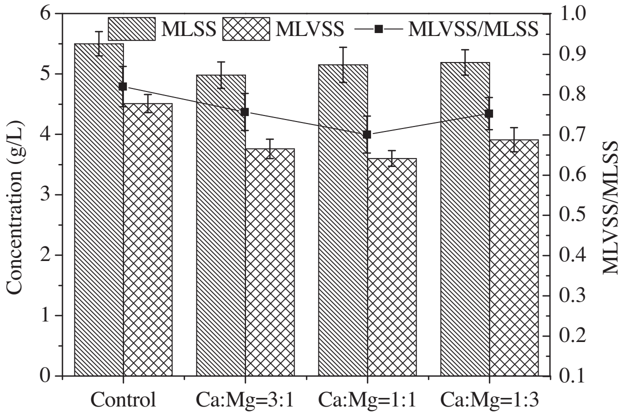

Fig. 3. MLSS, MLVSS, and MLVSS/MLSS ratio in the MBR at different ratios of Ca to Mg. Error bars represent standard deviation of 10 times tests.

on the distribution position feature of EPS on cell, EPS can be classified as BEPS and SEPS [21]. BEPS are located at or outside the cell surface, and the SEPS also called SMP is in sludge supernatant. The concentrations of SMP and BEPS in the MBRs at different Ca:Mg ratios are shown in Fig. 4. It could be seen that the dosage of divalent cations was prone to decrease the SMP and increase the BEPS in the MBRs compared to the control reactor. This observation is consistent with other researchers [6,7,9]. The variations of SMP and BEPS have been mainly attributed to the fact that Ca^{2+} and Mg^{2+} could adsorb the biopolymers in SMP to BEPS with the help of bioflocculation [6,7,9]. It could also be observed from Fig. 4 that as the Ca:Mg ratio declined from 3:1 to 1:3, SMP tended to increase while the average concentration of BEPS decreased from 37.75 ± 1.73 to 25.42 ± 0.76 mg/g VSS. The variations of SMP and BEPS at different Ca:Mg ratio were significant compared with the control reactor ($p < 0.05$). The increase of SMP correlated well with the variations of membrane fouling in the MBRs (as shown in Fig. 2). It has been well recognized that the higher SMP concentration results in the poorer filterability and more severe membrane fouling [22,23]. Based on the fact that the dosage of divalent cations was constant at 200 mg/L, the increase of SMP and decrease of BEPS with the decline of Ca:Mg ratio might be indicated that Ca^{2+} was more beneficial to binding and bridging EPS compared with Mg^{2+}.

In order to better understand the behaviors of SMP and BEPS in the MBRs, the concentrations of protein and carbohydrate in SMP (SMP_p and SMP_c) and BEPS (EPS_p and EPS_c) were measured and compared at different Ca:Mg ratios, as shown in Fig. 5. It could be seen from Fig. 5 that the carbohydrate concentration was higher than protein concentration in both BEPS and SMP. As Ca:Mg ratio decreased from 3:1 to 1:3, SMP_c increased approximately 1.80 times,

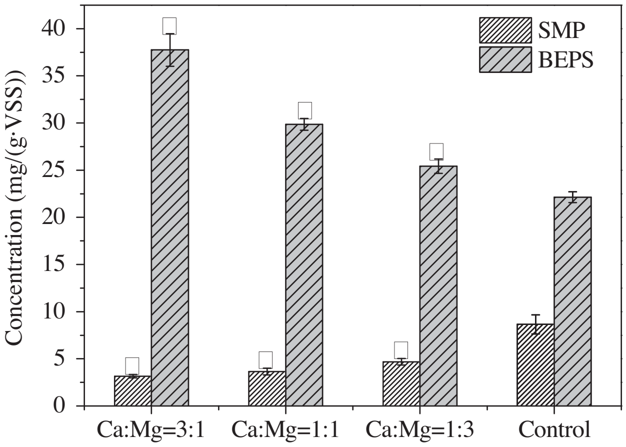

Fig. 4. Concentrations of SMP and BEPS in the MBRs at different Ca:Mg ratios. Asterisks (∗) represent treatments in which Ca:Mg ratio had a significant effect compared to the control reactor (ANOVA, $p < 0.05$). Error bars represent standard deviation of 4 times tests.

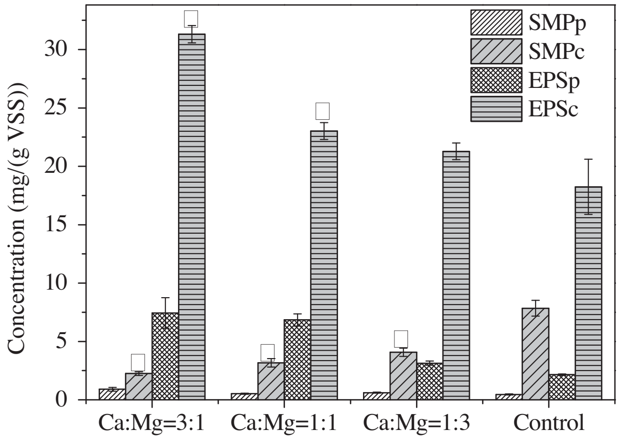

Fig. 5. Protein and carbohydrate concentrations in SMP and BEPS under different conditions of Ca:Mg ratios. Asterisks (∗) represent treatments in which Ca:Mg ratio had a significant effect compared to the control reactor (ANOVA, $p < 0.05$). Error bars represent standard deviation of 4 times tests.

being consistent with the variations of SMP (as shown in Fig. 4), while the SMP_p changed slightly. The changing trends of protein and carbohydrate in BEPS were similar as those in SMP. The EPS_c changed significantly while the EPS_p varied slightly with the decline of Ca:Mg ratio. Considering the fact that the bioflocculation of divalent cations was more prone to bridging carbohydrate compared with protein [9], it might be indicated that the bridging trend of Ca^{2+} was better than that of Mg^{2+} in this study.

3.2.3. Particle size distribution

The particle size distributions of sludge in the MBRs at different Ca:Mg ratios were measured, and the results are shown in Fig. 6. It could be seen that the Ca:Mg ratio had a significant effect on particle size in the MBRs, and the dosage of Ca^{2+} and Mg^{2+} was beneficial to increasing the particle size. It has been demonstrated that the the dosage of Ca^{2+} and Mg^{2+} could enlarge particle size [5–9]. According to the DCB theory, Ca^{2+} and Mg^{2+} could bridge negatively charged functional groups within the EPS of sludge, thus helping bioflocculation and formation of large flocs [4,5]. Furthermore, Bruss et al. [10] found that the extracted divalent cations from sludge lead to the increase of small particles. Therefore, the increase of particle size with the dosage of divalent cations was due to the bioflocculation of divalent cations.

As shown in Fig. 6, it could also been observed that the particle size of sludge decreased steadily with the reduction of Ca:Mg ratio, and their average volume size were 158.77 ± 5.59, 143.86 ± 4.32, and 121.39 ± 7.99 μm at the Ca:Mg ratio of 3:1, 1:1, and 1:3, respectively. It might be indicated that Ca^{2+} was better than Mg^{2+} in the process of enhancing bioflocculation, which could help form large flocs. The larger floc size was in favor of the reduction of membrane fouling, which agreed with the variations of TMP at different Ca:Mg ratios.

3.3. Resistance distribution

The R_m is approximately $0.31 \times 10^{12}\,m^{-1}$ in this study. Resistance analysis was performed at the end

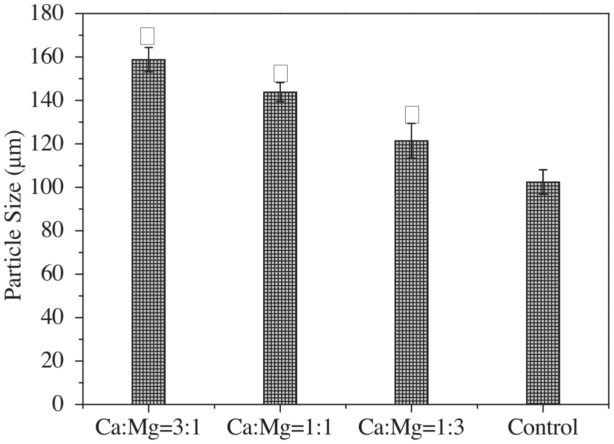

Fig. 6. Particle size distributions of sludge at different Ca: Mg ratios. Asterisks (∗) represent treatments in which Ca: Mg ratio had a significant effect on particle size compared to the control reactor (ANOVA, $p < 0.05$). Error bars represent standard deviation of triplicate tests.

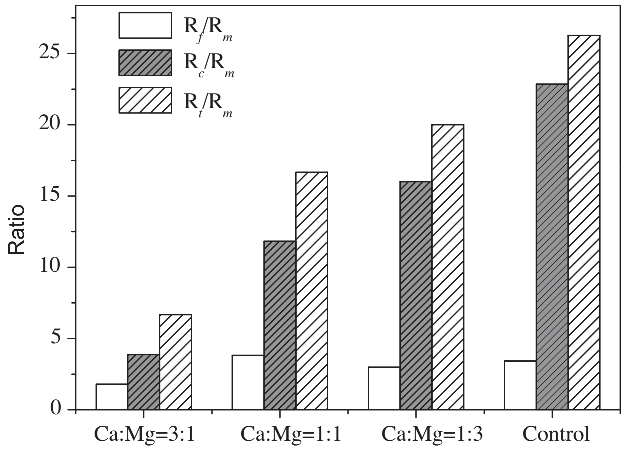

Fig. 7. Resistance distributions at different Ca:Mg ratios.

of each reactor, and R_f, R_c and the total resistance (R_t) are plotted in percentage relative to R_m, as shown in Fig. 7. It could be observed that the R_t increased with the reduction of Ca:Mg ratio from 3:1 to 1:3, which correlated well with the variations of TMP. It could also be seen that the R_c was the main component of the R_t in all reactors. When the Ca:Mg ratio declined from 3:1 to 1:3, the R_c declined, while the R_f changed slightly, suggesting that the reduction of total resistance at higher Ca:Mg ratio was due to the decline of R_c. It was reported that SMP (as shown in Fig. 4) played an important role in formation of the fouling layer [22,23], and larger floc size was in favor of the reduction of fouling layer. Thus, the decline of R_c at higher Ca:Mg ratio was mainly due to the reduction of SMP and increase of floc size by enhancing bioflocculation of Ca^{2+}.

4. Conclusions

Impact of Ca:Mg ratio on the performance of MBRs was investigated in bench-scale submerged MBRs treating synthetic wastewater at three Ca:Mg ratios of 3:1, 1:1, and 1:3 with the total Ca^{2+} and Mg^{2+} concentrations of 200 mg/L. Based on this study, the following specific conclusions could be drawn:

(1) The Ca:Mg ratio had a slight impact on effluent quality; however, its effect on membrane fouling was significant. Membrane fouling was effectively mitigated by enlarging Ca:Mg ratio under constant divalent cations of 200 mg/L, and the best permeability achieved at Ca:Mg ratio of 3:1. It might be suggested that Ca^{2+} had a more significant impact on mitigating membrane fouling compared with Mg^{2+}. Based on the analysis of resistance distribution, the reduction of membrane fouling was mainly due to the decrease of fouling layer resistance.

(2) When the Ca:Mg ratio declined from 3:1 to 1:3, the concentration of SMP significantly increased, while the concentration of BEPS decreased. The variations of SMP and BEPS with the decline of Ca:Mg ratio indicated that Ca^{2+} was more beneficial to binding and bridging negatively charged functional groups in EPS compared with Mg^{2+}.

(3) The carbohydrate concentration was higher than protein concentration in both BEPS and SMP. The SMP_c and EPS_c changed significantly while the SMP_p and EPS_p varied slightly with the variations of Ca:Mg ratio, suggesting that the bioflocculation of divalent cations was mainly by bridging not protein but polysaccharide. It could be further indicated that the bridging trend of Ca^{2+} was better than that of Mg^{2+} in this study.

Acknowledgments

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