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Enhanced removal of organics and phosphorus in a hybrid coagulation/membrane bioreactor (HCMBR) for real textile dyeing wastewater treatment

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ABSTRACT

In this work the performance of a hybrid coagulation/membrane bioreactor (HCMBR) was assessed for real textile dyeing wastewater advanced treatment. Poly-aluminum chloride (PACl) was selected and used in the experiments. Batch experiment results showed that the optimal dosage was 40 mg Al/L. During the pilot-scale experiment, the suitable dosage and dosing interval were once per day at the dosage of 40 mg Al per L of amount of mixed liquor into the HCMBR system. It was found that membrane fouling was effectively retarded by PACl addition in the running MBR system. Additionally, removal of pollutants according to influent and effluent quality was monitored. HCMBR achieved much higher removal efficiencies than membrane bioreactor (MBR) in COD_{cr}, UV_{254}, and total phosphate, due to PACl coagulation in the HCMBR bioreactor. And the application of PACl was effective in decreasing the content of extracellular polymeric substances and the membrane fouling resistances. The transmembrane pressure of HCMBR developed much slower than that of MBR, which indicated that coagulation with PACl in the MBR could alleviate membrane fouling. PACl addition was practically feasible for real textile dyeing wastewater advanced treatment without large excess sludge production.

Keywords: Hybrid coagulation/membrane bioreactor (HCMBR); Textile dyeing wastewater; Coagulation; Organics; Phosphate; Membrane fouling

1. Introduction

The textile industry is one of the major contributions to many Asian economies and one of the main revenue generating sectors. However, the textile dyeing process is a water- and energy-consuming process [1]. Large quantities of toxic, low biodegradable, highly colored wastewater are produced, which could destroy the natural water environment if it was untreated. Many attempts have been made to treat textile wastewater using conventional wastewater treatment methods such as physical treatments, chemical treatments, and biological treatments [2–5]. Since wastewater treatment for dyeing and finishing with membrane technology in China in 1975 was investigated for the first time, this technology has been gradually applied to other wastewater treatments in the textile industry such as wool scouring wastewater, polyvinyl alcohol (PVA) desizing effluent, chemical

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fiber finishes, etc., and the obvious advantages have led to more and more attention [6]. However, the membrane process has a problem of membrane fouling. The fouling limits the water flux through the membrane, sometimes to the point that the process is economically unattractive [7]. Besides, due to membrane bioreactor (MBR) maintaining long sludge retention time (SRT), the system has a problem with phosphorous removal. Previous investigations show such fouling can be mitigated by adding metal coagulants to MBRs [8,9]. And the effluent total phosphate (TP) concentration can be maintained at acceptable level by supplemental additions of chemicals such as Al or Fe (III) salts, poly-aluminum chloride (PACl), and/or lime. Although coagulants such as alum and $FeCl_3$ are used to control fouling in MBR [10], very limited information on coagulant addition, especially optimization of coagulants addition and feasibility of their application, has been obtained so far. And previously these investigations were major in the drinking water, micro-polluted source water, and synthetic wastewater treatment. The real textile dyeing wastewater advanced treatment was not involved and the influence of coagulants PACl on performance of MBR for real textile dyeing wastewater treatment discussed was very limited. On the basis of these, this study tries to solve these problems for feasibility of PACl application in real textile dyeing wastewater treatment.

The aim of this work is to confirm the effect of PACl in a long-term running MBR, clarify the behavior of PACl in an MBR, and examine the coming problems by PACl addition which might be negative for the MBR performance. Two MBRs, with PACl and without PACl addition, were operated to investigate the effect of coagulant addition on organics removal and membrane fouling. In addition, the performance of hybrid coagulation/membrane bioreactor (HCMBR) for the real textile dyeing wastewater treatment is also discussed.

2. Materials and methods

2.1. Experimental devices and operations

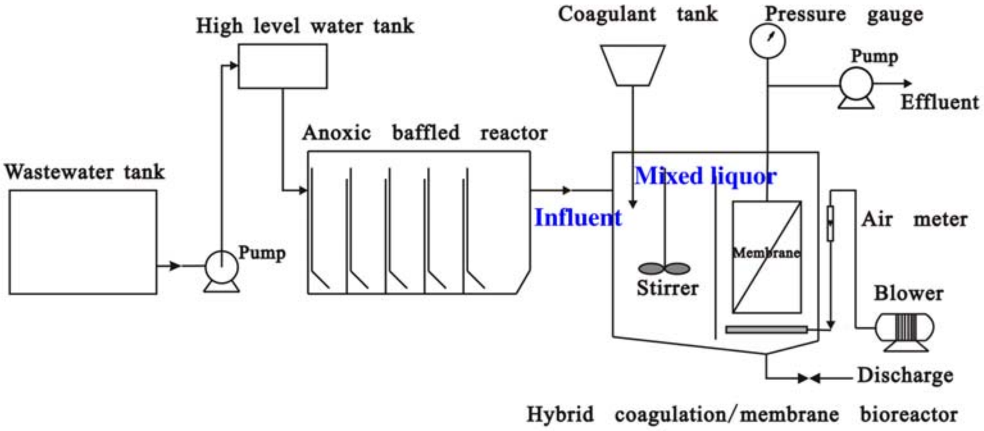
A pilot-scale submerged MBR combined with coagulation (HCMBR) and the same scale MBR without coagulant (MBR) were engineered and investigated in this study. A schematic diagram of HCMBR and MBR is shown in Fig. 1(a) and (b). Hollow fiber membrane module made from polyvinylidene fluoride (PVDF) membrane (Tianjin Motian Membrane Engineering and Technology Co. Ltd., Tianjin, P.R. China) was submerged in a reactor. An effective volume of each membrane bioreactor (HCMBR or MBR) was 1.67 m^3. A nominal pore size was 0.2 μm and effective filtration area was 25 m^2. Mixed liquor suspended solids (MLSS) concentration in the reactor was maintained at the desirable value by discharge of excess sludge. Each effluent flow rate was kept at the design value using a valve. Transmembrane pressure (TMP) was measured each day. Membrane modules were cleaning in 0.01 M NaOH and 0.5% NaClO for 24 h to obtain a permeability recovery more than 96%.

The study was carried out in the Meili wastewater treatment plant, Changshu, P.R. China. The capacity of the treatment plant was 10,000 m^3/d of highly colored textile dyeing wastewater. Wastewater was composed of 93% textile dyeing wastewater, 6% chemical fiber industrial wastewater, and 1% domestic sewage. The main component of wastewater originated from the production processes of dyeing, printing and terylene fiber printing, and dyeing. The characteristics of wastewater (Table 1) were high pH, chemical oxygen demand (COD), TP, color, and low biochemical oxygen demand (BOD)/COD ratio, which were different from traditional printing and dyeing wastewater. In this study, both the influent wastewater and seed sludge were taken from the Meili wastewater treatment plant. To enhance the biodegradability of organic matter in wastewater, an anoxic baffled reactor (ABR) as a hydrolysis process pretreatment was also adopted (Fig. 1). Therefore, water for HCMBR use was from effluent of ABR. The main characteristics of wastewater for HCMBR influent are shown in Table 1.

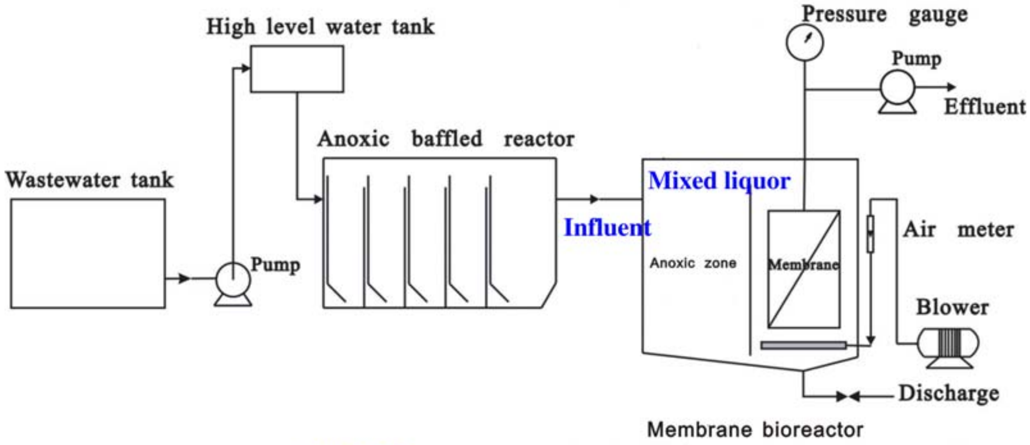
Coagulants used in a developing country such as China are frequently alum based and iron-based ones [8,10]. However, the chemical instability of iron-based coagulants such as ferric chloride limits its widespread applications. PACl has been widely adopted as the most common coagulant in water treatment. As a result, PACl was selected and used in the experiments.

2.2. Batch test

A jar tester was used for the coagulation of mixed liquor from the MBR. The concentration of PACl (as Al) was varied from 0 to 200 mg Al/L. The batch test was performed to determine the optimum coagulant and coagulation conditions. The procedure of batch test was as follows [9]: 1 L of mixed liquor was poured into a jar and predetermined amount of coagulant stock solution was added. The mixed liquor was then stirred for 5 min at a speed of 150 rpm. Stirring was further performed for another 5 min at a speed of 30 rpm. The mixed liquor was settled for 15 min and



(a) HCMBR



(b) MBR

Fig. 1. Schematic diagram of the pilot-scale HCMBR (a) and MBR (b).

Table 1
The main characteristics of wastewater for HCMBR influent

Temperature (℃)	pH	COD_{cr} (mg/L)	BOD_5/COD_{cr}	UV_{254} (cm^{-1})	TP (mg/L)	Color
23.1–31.8	8.12–9.23	383.1–534.8	0.42–0.54	2.015–2.634	5.45–11.63	60–100

clean supernatant from the jar was taken for further analysis.

2.3. Membrane filtration test

Based on the above batch test results, to evaluate the effect of PACl addition on MBR system performance in terms of organics removal as well as membrane fouling, an MBR system with PACl (HCMBR) was operated and compared with an MBR system without PACl (MBR). The MLSS concentration in the reactor was 6,050 mg/L prior to membrane filtration. In order to reduce membrane fouling, the MBR flux was set at a relatively low value of 7 L/(m^2 h). The effluent suction pump was controlled by a timer based on a time sequence of 8 min on and 2 min off in each cycle. The mixed liquor was aerated through the diffusers located beneath the membrane module to provide oxygen for biomass growth as well as shear to reduce cake formation on the membrane surface. The flow rate was set at 200 L/(m^2 h) when the MBR systems were stably running to increase dissolved oxygen concentration for sufficient removal of NH_4^+–N from MBR system.

2.4. Analytical methods

Water quality analysis was conducted following the standard methods [11]. COD_{cr} was analyzed by the potassium dichromate oxidation methods. Once oxidation is complete, the excess potassium dichromate is titrated with ferrous ammonium sulfate. TP concentration was determined by the potassium persulfate oxidation and colorimetric methods with wavelength 700 nm using a spectrometer (WFZ UV-2800AH, Unic); TN was also determined by the potassium persulfate oxidation and ultraviolet spectrophotometer methods with wavelength 220 nm and 275 nm; UV_{254} was also determined by using the spectrometer with wavelength 254 nm. Color concentration was determined dilution times methods. The MLSS were measured by filtering and drying the mixed liquor sample at 103°C for 1 h in a desiccator. The pH was measured with a pH meter (pHS-3C).

Membrane resistance was evaluated by the resistance-in-series models as follows:

$$J = \frac{\Delta p}{\mu R_t} \tag{1}$$

$$R_t = R_m + R_c + R_f \tag{2}$$

where J is the permeate flux, Δp is TMP, μ is the viscosity of the permeate, and R_t is total membrane resistance, R_m is the intrinsic membrane resistance, R_c is the cake resistance, and R_f is the fouling resistance due to pore blocking. The experimental procedure to get each resistance value was as follows [12]: R_t was evaluated by the final flux of wastewater microfiltration. The membrane surface was then flushed with tap water to remove the fouling cake layer. After that, clean water flux was measured again to get the resistance of $R_m + R_f$. Filtration of clean water with new membrane before operation gave R_m. From these values each of R_t, R_m, R_c, and R_f could be obtained using Eq. (2).

The extraction of extracellular polymeric substances (EPS) from the suspended microbial flocs was carried out using sulphuric acid extraction method [13]. Polysaccharide content was measured by the anthrone method using glucose as the standard. About 1 mL assay samples were added to 2 mL of anthrone-H_2SO_4 reagent. After vortexing, samples were incubated at 100°C for 10 min and then cooled in cold water. Standards and samples were measured spectrophotometrically at 625 nm. For protein determination, ultraviolet spectrophotometer method was applied [14]. Ultraviolet (UV) absorbance was measured at 260 nm ($A_{260\,\text{nm}}$) and 280 nm ($A_{280\,\text{nm}}$), and the protein concentration (C_{protein}) was determined using Eq. (3):

$$C_{\text{protein}} = (1.55 \times A_{280\,\text{nm}} - 0.76 \times A_{260\,\text{nm}}) \times 1,000 \tag{3}$$

The relative hydrophobicity was analyzed according to [7]. The relative hydrophobicity was determined using Eq. (4):

$$\text{Relative hydrophobicity } (\%) = \left(1 - \frac{\text{MLSS}_a}{\text{MLSS}_b}\right) \times 100 \tag{4}$$

where MLSS_a is the concentration of MLSS in the aqueous phase after emulsification and MLSS_b is the concentration of MLSS in the aqueous phase before emulsification.

3. Results and discussion

3.1. Effect of PACl addition on water quality of mixed liquor

Table 2 shows the changes of water quality of the same supernatant of mixed liquor treated with different dosages of PACl. With the dosage of PACl increasing, the mixed liquor became more acidic. From Table 2, it can be observed that pH decreased little by little, which might adversely affect not only the microorganism activity [9], but also the hydrolysis product of coagulants and coagulation mechanisms [15]. However, it could recover to the initial pH level soon due to the pH level of HCMBR influent maintaining 8.12–9.23. Thus, there was no apparent difference of pH between the two MBR systems. COD_{cr} removal at some content could be improved with PACl addition from 0 to 40 mg Al/L. When Al concentration of the mixed liquor was over 60 mg Al/L, excess addition of PACl led to COD_{cr} back up.

UV_{254} represents the aromatic or unsaturated compounds content in supernatant of mixed liquor. As shown in Table 2, UV_{254} initial value was 2.189 cm^{-1}, which indicated that organic matters content was very high in the mixed liquor. The supernatant organic fraction could be removed with coagulant addition. In textile wastewater, approximately 80% of the acid and reactive dyes are azo compounds, which were converted into other intermediate organic forms, such as aromatic amines in ABR according to our previous study [5]. A proportion of these aromatic amines could be aerobically degraded in MBR. The remaining proportion of the aromatic amines would autoxidize to products, which were easily removed by polymerization to flocs that could then be easily separated [16].

Table 2
Changes of water quality of mixed liquor after PACl addition

PACl, as Al (mg Al/L)	pH	COD_{cr} (mg/L)	UV_{254} (cm^{-1})	TP (mg/L)	TN (mg/L)
0	7.65	415.3	2.189	8.12	40.25
5	7.41	402.3	2.102	6.39	35.24
15	7.39	352.2	1.853	3.28	33.21
20	7.36	268.3	1.324	1.56	30.38
40	7.26	213.1	0.786	0.43	27.24
60	7.23	215.2	0.515	0.26	26.34
80	6.50	220.1	0.216	0.09	28.41
100	6.06	236.3	0.102	0.05	20.78
120	5.42	222.1	0.096	0.02	25.34
200	5.09	219.3	0.086	0.00	29.76

TP removal efficiency was very well with addition of PACl to the mixed liquor. A PACl dose of 40 mg Al/L (molar ratio of Al:$p = 5.69$:1) decreased the TP concentration from 8.12 to 0.43 mg/L, and the TP concentration decreased even more with increasing PACl addition. TP removal mechanism is discussed in the next section. TN removal at some extent was somewhat irregular. The excess PACl could have a bad effect on the nitrifying bacteria and would affect the nitrification and denitrification [9].

Based on the above jar test results, the use of PACl in concentration range from 40 to 60 mg Al/L seemed to be effective. However, beyond 40 mg Al/L, further decrease in the COD_{cr}, TP, UV_{254} absorbance was not noticeable, and 40 mg Al/L addition quantity in practice was more economical. Thus, the optimal dosage should be 40 mg Al/L, and the optimal pH was 7.26.

3.2. Effect of PACl on MBR system performance

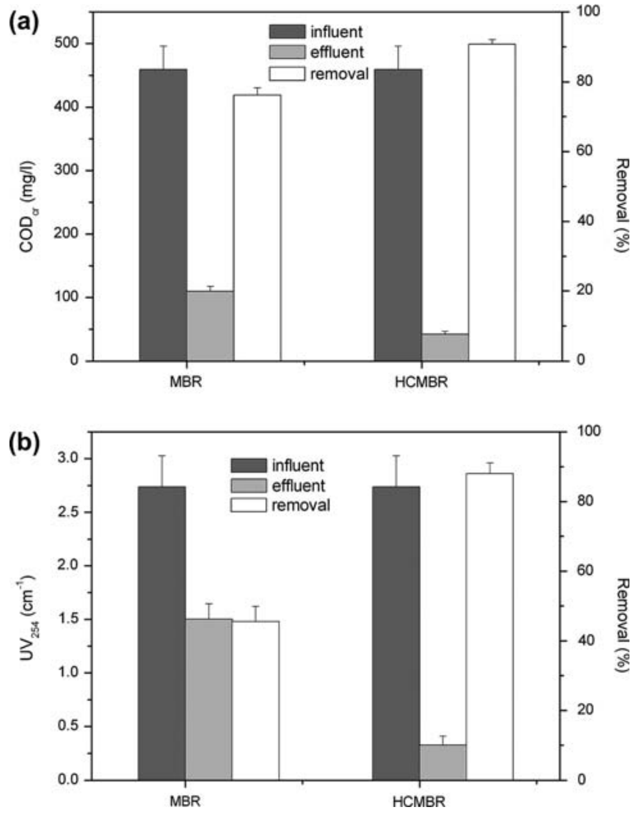


Fig. 2. Comparison of (a) COD_{cr}, (b) UV_{254} removals by HCMBR and MBR.

PACl was injected once per day into the HCMBR at a dosage of 40 mg Al/L. HCMBR was operated and compared with an MBR system without PACl (MBR). In this experiment, COD_{cr} concentration and UV_{254} absorbance of influent, effluent, and mixed liquor of two MBR systems were measured to show organic matters removal in wastewater. As shown in Fig. 2(a), HCMBR decreased COD_{cr} from 459.4 ± 36.6 mg/L to 42.3 ± 4.5 mg/L at steady state, corresponding to a removal efficiency of 90.72%, which was much higher than the 76.16% removal by MBR. It was obvious that 14.56% increase of nutrient removal in the HCMBR should be attributed to coagulation by PACl. Fig. 2(b) shos the dissolved organic matter removal efficiencies in terms of UV_{254}. The results indicate that HCMBR reduced influent UV_{254} by 88.04 ± 3.07%; while the removal efficiency by MBR was only 45.58 ± 4.34%. Obviously, 42.46% higher of UV_{254} removal was achieved by addition of PACl. According to the batch test, zeta potential in supernatant increased toward positive charge from −9.2 to 0.3 mV after PACl addition (0–200 mg Al/L). It was believed that polymeric coagulants could supply more positive charges for organic particles and sludge flocs, and enhance the functions of charge neutralization, which resulted in higher removal of supernatant organic matter and enlargement of sludge floc size [15]. Fig. 3(a) and (b) shows that COD_{cr} and UV_{254} in the mixed liquor of HCMBR was 41.3 and 65.4% lower than that of MBR, indicating that PACl in the bioreactor could decrease the organic content of the mixed liquor, and thus alleviate membrane fouling. Besides, COD_{cr} in the mixed liquor rejected by the membranes in HCMBR and MBR amounted to 80.80 ± 2.65 and $70.62 \pm 4.17\%$, while UV_{254} rejected by the membranes were 58.26 ± 6.41 and $32.58 \pm 4.18\%$; respectively, indicating that coagulation of PACl in the MBR was able to enhance the organic matter rejection efficiency. PACl coagulation flocs themselves might have constituted a part of the membrane fouling layer and helped to reject organic matter in the reactor [8].

The TP removal performances of HCMBR and MBR were illustrated in Fig. 4. The TP concentration of influent of HCMBR was 8.92 ± 1.34 mg/L HCMBR achieved $97.63 \pm 1.59\%$ of TP removal with 0.20 ± 0.13 mg/L as the effluent concentration, which was much higher than the $28.61 \pm 10.94\%$ removal obtained by MBR. As Al^{3+} salts react rapidly not only with phosphate forming stable precipitates, but also with hydroxyl ions, a portion of the metal coagulants is first precipitates as hydroxides, and the phosphate ions then adsorb onto those solids [17].

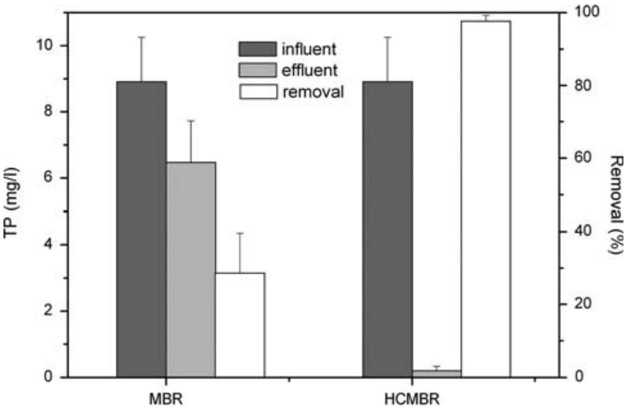


Fig. 4. TP removal in HCMBR and MBR systems.

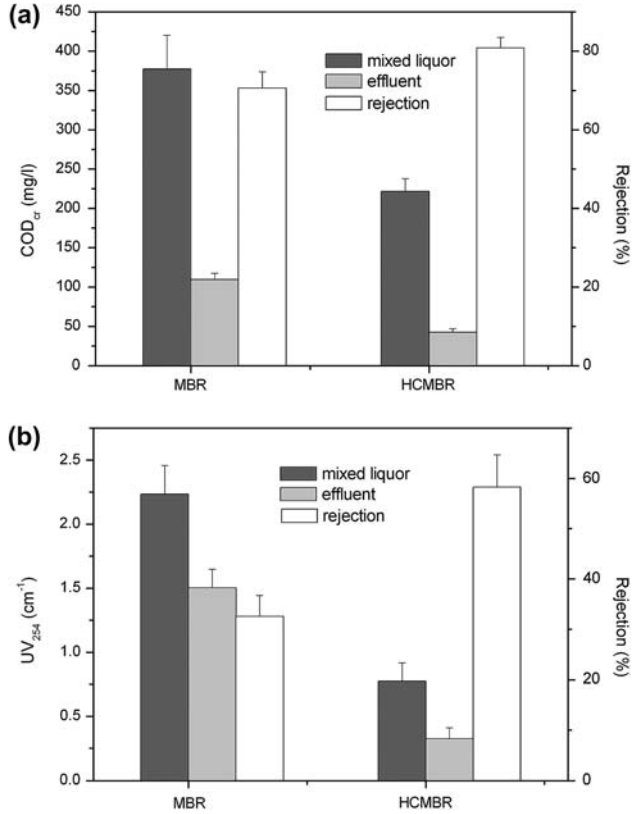


Fig. 3. Comparison of (a) COD_{cr}, (b) UV_{254} in the mixed liquor and effluent of both HCMBR and MBR systems.

3.3. Membrane filtration resistance analysis

To investigate the fouling tendency quantitatively, various resistances, the ratios of resistances were calculated and summarized in Table 3. TMP was monitored under constant flux condition (7 L/(m^2 h)), and the operation was stopped when the TMP reached 20 kPa. In this study, the cake layer resistance (R_c) was a major contributor to the total resistance (R_t). Of the total resistance in HCMBR system, about 58.47% came from the cake layer resistance, which was a little higher than the 57.66% obtained in the MBR system. However, the pore resistance (R_f) in HCMBR system was $0.26 \times 10^{12}\,m^{-1}$, almost half as much as the pore resistance in MBR system. These results revealed that coagulation with PACl in the bioreactor mainly decreased the pore resistance, and also could decrease the total fouling resistance effectively. In general, membrane pore blocking mainly results from the adsorption of colloids, macromolecules, biologically active substances, various ions, and so on [7]. Due to Al salts' bridging ability during coagulation, these matters can be captured. Thus, there were few colloids and macromolecules in the supernatant of mixed liquor of the HCMBR system. At the same time, the formation of a cake layer on the membrane surface was the major factor that affects membrane fouling behavior.

The TMP variation in HCMBR and MBR is shown in Fig. 5. It can be seen that the TMP increased with the operation time in MBR system from the initial 6.58 kPa to final 7.68 kPa. However, the TMP of HCMBR developed much slower than that of MBR. The final TMP was 0.5 kPa lower than that of MBR.

Table 3
A series of resistances of the membrane

Resistances	MBR		HCMBR	
	Value ($\times 10^{12}$ m^{-1})	Resistance ratio (%)	Value ($\times 10^{12}$ m^{-1})	Resistance ratio (%)
R_m	1.21	28.95	1.21	34.18
R_c	2.41	57.66	2.07	58.47
R_f	0.56	13.40	0.26	7.34
R_t	4.18	100	3.54	100

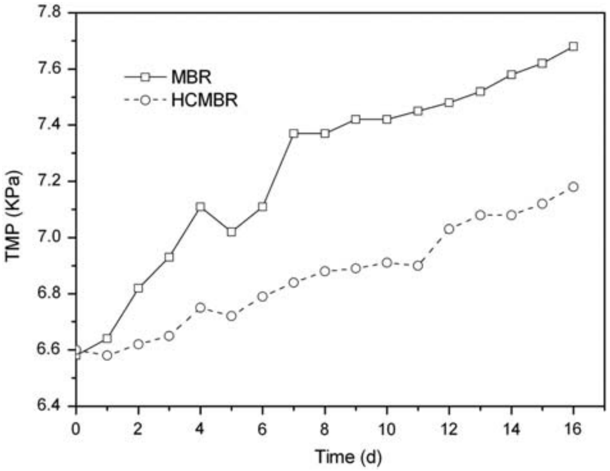


Fig. 5. Change of TMP in HCMBR and MBR systems.

The results showed that coagulation with PACl could alleviate or retard membrane fouling in the MBR.

3.4. Comparison of EPS and Relative hydrophobicity

As shown in Table 4, the changing tendency of the polysaccharide, protein, and total EPS between MBR and HCMBR was similar to that of membrane fouling resistance (Table 3). This result revealed that EPS might be a significant factor affecting membrane fouling in MBR [7]. From Table 4, it can also be seen that the protein appeared to be the major component of EPS. The predominance of protein in EPS could be due to the presence of large quantities of exoenzymes from bacterial excretions, such as lysis products and extracellular products in the flocs [18]. The protein had a strong positive influence on the hydrophobicity of microbial floc, while polysaccharide had no remarkable influence [19]. Hydrophobicity of microbial floc was the tendency of flocs to repel water. It could be seen that the relative hydrophobicity of sludge flocs in HCMBR system was lower than that of sludge flocs in MBR system (Table 4). Higher hydrophobicity of sludge flocs could render a stronger adherence of floc particles onto the membrane surface, thereby augmenting the membrane fouling [7]. Thus, the lower membrane fouling in HCMBR system might be mostly attributed to the decrease in EPS.

Apart from the importance of EPS and bioflocculation on membrane fouling in MBRs, also low temperature due to seasonal variations and soluble microbial products has been reported to increase membrane fouling [20,21]. Thus, further research is needed to decouple their respective influence on membrane fouling.

3.5. Amount of excess sludge

To investigate the increased amount of excess sludge, the MBR and the HCMBR systems were run for 16 days, respectively. The difference between the MLSS concentrations in HCMBR and MBR systems was less than 1.2 g/L (Fig. 6). The initial MLSS concentration in both systems was adjusted to 6.05 g/L

Table 4
Comparison of EPS and relative hydrophobicity in HCMBR and MBR systems

Parameter[a]	MBR	HCMBR
Polysaccharide (mg/L)	15.6 ± 2.1	8.6 ± 1.5
Protein (mg/L)	40.6 ± 6.3	18.5 ± 4.2
EPS (mg/L)	56.2 ± 4.5	27.1 ± 2.6
Relative hydrophobicity (%)	58.2 ± 3.6	31.4 ± 2.8

[a] Data are means ± standard deviation.

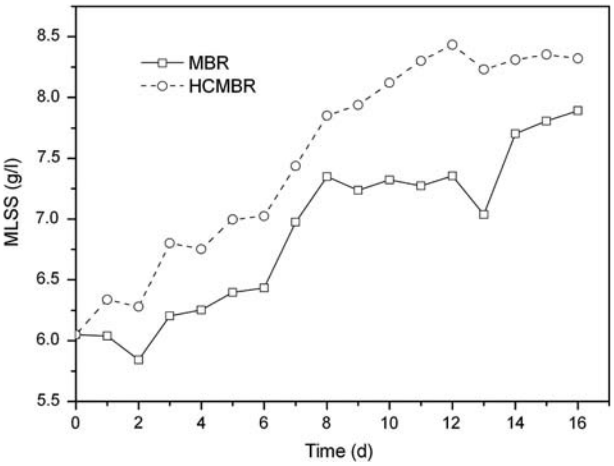


Fig. 6. Change of MLSS in HCMBR and MBR systems.

by discharge of excess sludge. At the end of this experiment, the MLSS concentration of MBR was 7.89 g/L, while that of HCMBR only increased to 8.32 g/L, unexpectedly. Because of HCMBR system operated at long SRT, the PACl was expected to accumulate and hold for a long time in the bioreactor. The components of insoluble phosphate could flow out AlO_3, which dissolved into AlO_2 and it could react over again [9]. During the experiment, PACl was intermittent addition, which was once per day at the dosage of 40 mg Al per L of amount of mixed liquor into the HCMBR system. This addition approach contributed to the textile dyeing wastewater advanced treatment requirement without large excess sludge production. Thus, it was believed that PACl addition was practically feasible for the textile dyeing wastewater advanced treatment.

4. Conclusions

This article investigated the HCMBR for the textile dyeing wastewater advanced treatment. According to batch test results, the optimal coagulation dosage should be 40 mg Al/L. HCMBR achieved much higher organic matter removal than MBR in terms of COD_{cr} and UV_{254}. Also, the HCMBR achieved 97.63% of TP removal efficiency, which was obviously higher than 28.61% removal obtained by MBR. Coagulation with PACl in the MBR mainly decreased the pore resistance; it also could decrease the total fouling resistance effectively. EPS was a significant factor affecting membrane fouling. PACl addition was practically feasible for the textile dyeing wastewater advanced treatment without large excess sludge production. Thus, coagulation followed by microfiltration membrane technology was practically feasible for the textile dyeing wastewater advanced treatment.

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