


Effect of powder-activated carbon pre-coating membrane on the performance of the UF system for wastewater reclamation: a pilot-scale study

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ABSTRACT

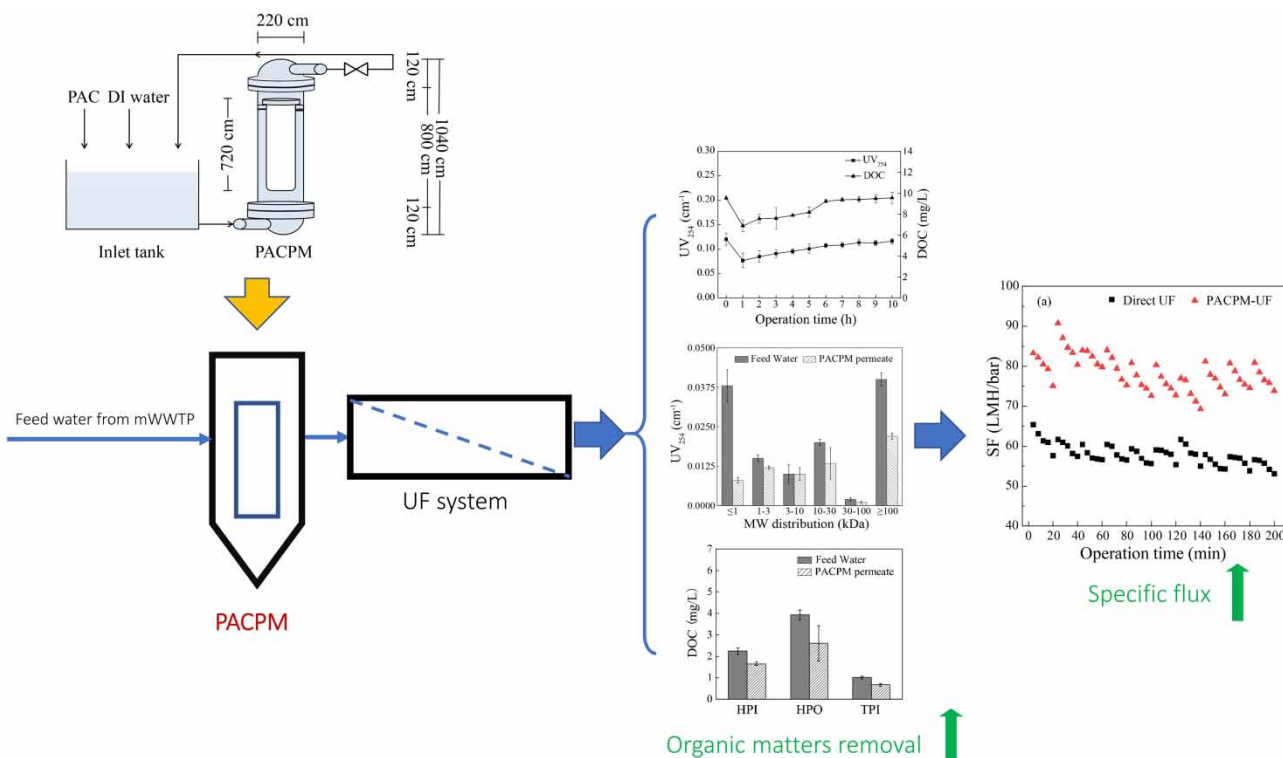
Pretreatment is an indispensable means to alleviate membrane fouling and improve ultrafiltration (UF) performance. In this study, we designed and established a powder-activated carbon pre-coating membrane (PACPM) unit as a pretreatment for the UF system. The effective filtration area of PACPM was 0.5 m². 300 g/m² PAC was selected as the optimal dosage in the pilot-scale apparatus according to the laboratory-scale trial. The pre-coating membrane could be formed within 30 min. PACPM could remove different kinds of organic compounds with different molecular weights and hydrophobicity during a certain period of time. During 10 operation cycles, the specific flux decrease rate of PACPM-UF was only 2.8%, which was much lower than that of direct UF (13.4%). PACPM could improve the performance of the UF system, not only for the increase of the initial SF value but also for the increase of the flux recovery thorough backwash. Nevertheless, a regular replace-regeneration process is necessary to maintain PACPM performance.

Key words: membrane fouling, pilot-scale system, powder-activated carbon pre-coating membrane (PACPM), UF membrane

HIGHLIGHTS

- The pilot-scale powder-activated carbon pre-coating membrane (PACPM) unit was designed and established.
- The pre-coating membrane could be formed in a short time.
- PACPM-UF had a better efficiency of contaminant removal than direct UF.
- PACPM could improve the performance of the UF system treating the municipal wastewater treatment plant effluent.

GRAPHICAL ABSTRACT



INTRODUCTION

Wastewater reclamation is an attractive approach for overcoming the global water crisis (Nahrstedt *et al.* 2020). Ultrafiltration (UF) technology has become increasingly applied for wastewater reclamation and leachate treatment due to the stable permeate quality and automatic operation (Snyder *et al.* 2007; Qu *et al.* 2015; Qu *et al.* 2019; Abuabdou *et al.* 2021; Yu *et al.* 2021). However, organic fouling of UF membranes is still a major impediment to the effective operation of UF systems. Organic fouling causes multiple adverse effects on the performance of UF systems (e.g., decreased production, water quality deterioration and decreased module lifetime) (Zhao *et al.* 2018; Ahmad *et al.* 2020).

Pretreatment can increase the permeate quality and reduce UF membrane fouling (Huang *et al.* 2009; Wang *et al.* 2020). Powder-activated carbon (PAC) adsorption is one of the most popular pretreatment techniques because of its contaminant adsorption ability and commercial availability (Gao *et al.* 2011). Although much work has been done to study the effect of PAC on UF membrane fouling, the results are somehow contradictory: some researchers found that PAC could not only remove organic matter but also alleviate UF membrane fouling (Campinas & Rosa 2010; Sun *et al.* 2017; Cheng *et al.* 2021), while other researchers reported that PAC could not alleviate UF membrane fouling (Lee *et al.* 2000; Zhang *et al.* 2003; Shao *et al.* 2016; Zhang *et al.* 2018).

Pre-coating membrane is a filter cake layer formed by pre-coated agent on the surface of base membrane, which could improve the filtration accuracy and reduce the fouling of the base membrane (Ma *et al.* 2013; Yang *et al.* 2015). However, it was reported that PAC directly coating on the UF membrane was liable to cause UF membrane damage and increase backwash difficulty (Malczewska *et al.* 2015). Compared with the direct coating on the UF membrane, PAC pre-coating on the base membrane could avoid the damage to the UF membrane. The PAC cake layer and the base membrane could not only adsorb organic matter but also remove colloids and particles in the influent, which could protect the UF membrane from damage. However, to date there are few reports on the powder-activated carbon pre-coating membrane (PACPM) as the UF system pretreatment for wastewater reclamation, especially in pilot-scale studies.

In this study, a pretreatment technique using PAC pre-coating on the base membrane was investigated to improve the performance of the UF system. The dosage and organic matter removal efficiency of PAC were determined via laboratory-scale

experiments. The pilot-scale PACPM-UF system was set up to investigate the effects of PACPM on contaminant removal and the performance of the UF system treating the effluent of a municipal wastewater treatment plant (mWWTP).

METHODS

Water samples

PACPM feed water, lab-scale and pilot-scale PACPM permeates were collected and stored at 4 °C prior to analyses. The feed water was collected from the effluent of a coastal municipal wastewater treatment plant (cmWWTP), which was located in Qingdao, Shandong Province, China. The cmWWTP adopted anaerobic–anoxic–oxic with moving-bed biofilm reactor as a secondary treatment process for the biological removal of nitrogen and phosphorus, with additional coagulation, filtration and UV disinfection as advanced treatment. Feed water characteristics are listed in Table 1. The conductivity was 1,810–9,790 $\mu\text{S}/\text{cm}$, which was significantly higher than other common mWWTPs (Xu *et al.* 2010; Chon *et al.* 2012; Ayache *et al.* 2013; Wang *et al.* 2019; Tong *et al.* 2020), which could result from the periodical seawater intrusion of this cmWWTP.

Laboratory-scale experimental setup

A vacuum filter holder was applied for the lab-scale experiment to determine the dosage and organic matter removal efficiency (Supplementary Figure S1). The qualitative filter paper was put on the core of the filter as the base membrane. The filtering area was $1.67 \times 10^{-3} \text{ m}^2$. The filtration pressure was maintained at 0.07 MPa via a vacuum pump. PAC with an average particle diameter of 32 μm and an average pore size of 1 nm was used to form the pre-coating dynamic membrane (purchased from Sinopharm Chemical Reagent Co., Ltd). To fabricate lab-scale PACPM, different loads of PAC (0, 150, 300, 600, 900, 1,200 and 1,500 g/m^2) were suspended by deionized (DI) water and filtered by vacuum filtration onto a qualitative filter paper. The feed water sample (500 mL) was pumped through PACPM at 0.07 MPa using a vacuum pump. The flux of the feed water sample was calculated by time recording. Then, PACPM permeate was pumped through the 0.2- μm nylon membrane (Whatman, England) at 0.07 MPa. The flux of PACPM permeate was calculated by time recording.

Pilot-scale experimental setup

The pilot-scale PACPM apparatus was designed and fabricated based on the bag filter, schematically shown in Figure 1. The effective filtration area of the base membrane was 0.5 m^2 . To fabricate PACPM, PAC particles were suspended with DI water into the PACPM feed tank. Then, the PAC suspension was pumped into the bag filter and circulated in the PACPM apparatus. The PACPM-UF system, as shown in Figure 2, has a capacity of $72 \text{ m}^3/\text{d}$. The characteristics of the UF membrane (Inge dizzer[®] 5000 plus 0.9MB50) used in the pilot system are shown in Table 2. The water backwash of the UF membrane was performed with a flux of $230 \text{ L}/(\text{m}^2 \cdot \text{h})$ and a duration of 40 s. The chemical enhanced backwash (CEB) process was performed on a daily basis. The sequence of the CEB process was as follows: alkaline cleaning (NaOH, pH 12, 40 s), alkaline immersion (10–60 min), water backwash (40 s), acid cleaning (H_2SO_4 , pH 2, 40 s), acid immersion (40 min), and then water backwash (40 s). The UF permeate was used for the water backwash and CEB process.

Table 1 | Feed water characteristics

Characteristics	Concentration range	Average value
pH	7.05–7.87	7.46
SS (mg/L)	1–9	4
Conductivity ($\mu\text{S}/\text{cm}$)	1,810–9,790	5,500
Turbidity (NTU)	0.53–2.93	1.13
DOC (mg/L)	7.68–11.35	9.62
UV_{254} (cm^{-1})	0.10–0.13	0.12
TN (mg/L)	7.94–14.30	11.12

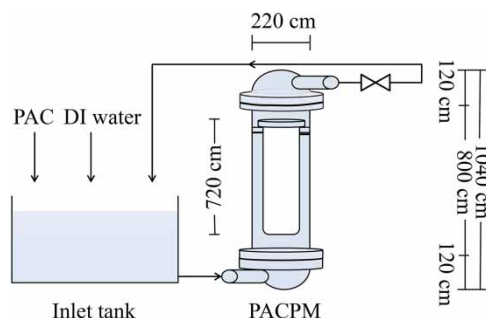


Figure 1 | Schematic diagram of the pilot-scale PACPM apparatus.

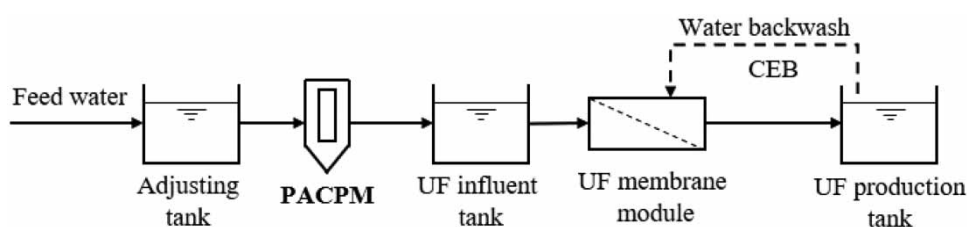


Figure 2 | Schematic diagram of the major process in the pilot PACPM-UF system.

Table 2 | Characteristics of the UF membrane applied in the process

Items	UF membrane
Manufacturer	inge dizzer®
Type	5000 plus 0.9MB50
Membrane material	PESM
Pore size (µm)	0.02
Effective surface area (m ²)	50
Filtration mode	Dead-end
Membrane flux (L/(m ² ·h))	60
maximum pressure (MPa)	0.5
Recovery rate (%)	90

Analytical methods

Transmembrane pressure (TMP) and membrane flux (J) of the UF membrane were measured by the online monitor of the PACPM-UF system. Specific flux (SF) was defined as membrane flux (J) divided by TMP. pH and conductivity were measured by a multi-parameter controller (LEICI, China). Suspended solids (SS) was measured by an ultraviolet-visible spectrophotometer (HACH, USA). Turbidity was measured by an SGZ-20B portable turbidimeter (Mingbolm, China). Dissolved organic carbon (DOC) and total nitrogen (TN) were measured by a Multi N/C 2100 analyzer (Analytik Jena, Germany). UV absorbance at 254 nm (UV₂₅₄) was measured by a Uvmini-1240 spectrophotometer (Shimadzu, Japan). The molecular weight (MW) distribution of the organic matter (UV₂₅₄) in the water sample was measured using cellulose ultrafiltration membranes with MW cut-offs of 1, 3, 10, 30 and 100 kDa (Millipore, USA) as described by Zhao *et al.* (2014). Resin fractionations of the dissolved organic matter (DOM) in the water samples were performed using DAX-8 and XAD-4 ion exchange resins with a method described by Zularisam *et al.* (2007).

RESULTS AND DISCUSSION

Optimal PAC dosage of PACPM

To determine the optimal PAC dosage of PACPM, a lab-scale apparatus was applied using different loads of PAC. With the increase of PAC dosage, the flux of the feed water through PACPM decreased rapidly from 5.4 to 3.1 m/h at the beginning and then decreased steadily (Figure 3(a)). Correspondingly, the flux of PACPM permeate through the 0.2- μm membrane increased from 3.8 to 7.1 m/h (Figure 3(b)). Regarding the removal of the organic matter, with the increase of PAC dosage from 0 to 300 g/m², the DOC and UV₂₅₄ removal rates increased to 41.7 and 89.5%, respectively. Subsequently, the DOC and UV₂₅₄ removal rates kept stable with the increase of PAC dosage from 300 to 1,500 g/m² (Figure 3(c) and 3(d)). Considering the effects of the PAC dosage on both flux and organics removal, 300 g/m² was selected as the PAC dosage in the pilot-scale study.

Effects of PACPM on contaminant removal

As the effective filtration area of the base membrane was 0.5 m², 150 g PAC (300 g/m²) particles were suspended with DI water and pumped from the bottom of the PACPM apparatus. The PAC particles gradually and evenly adhered to the base membrane with the circulation of the PAC suspension in the apparatus (Figure 4). Meanwhile, PACPM permeate became more and more clear (Supplementary Figure S2). After 30 min circulation of the PAC suspension, the SS of the permeate decreased to less than 5 mg/L and remained stable, which indicated that the pre-coating membrane has been successfully developed (Figure 5).

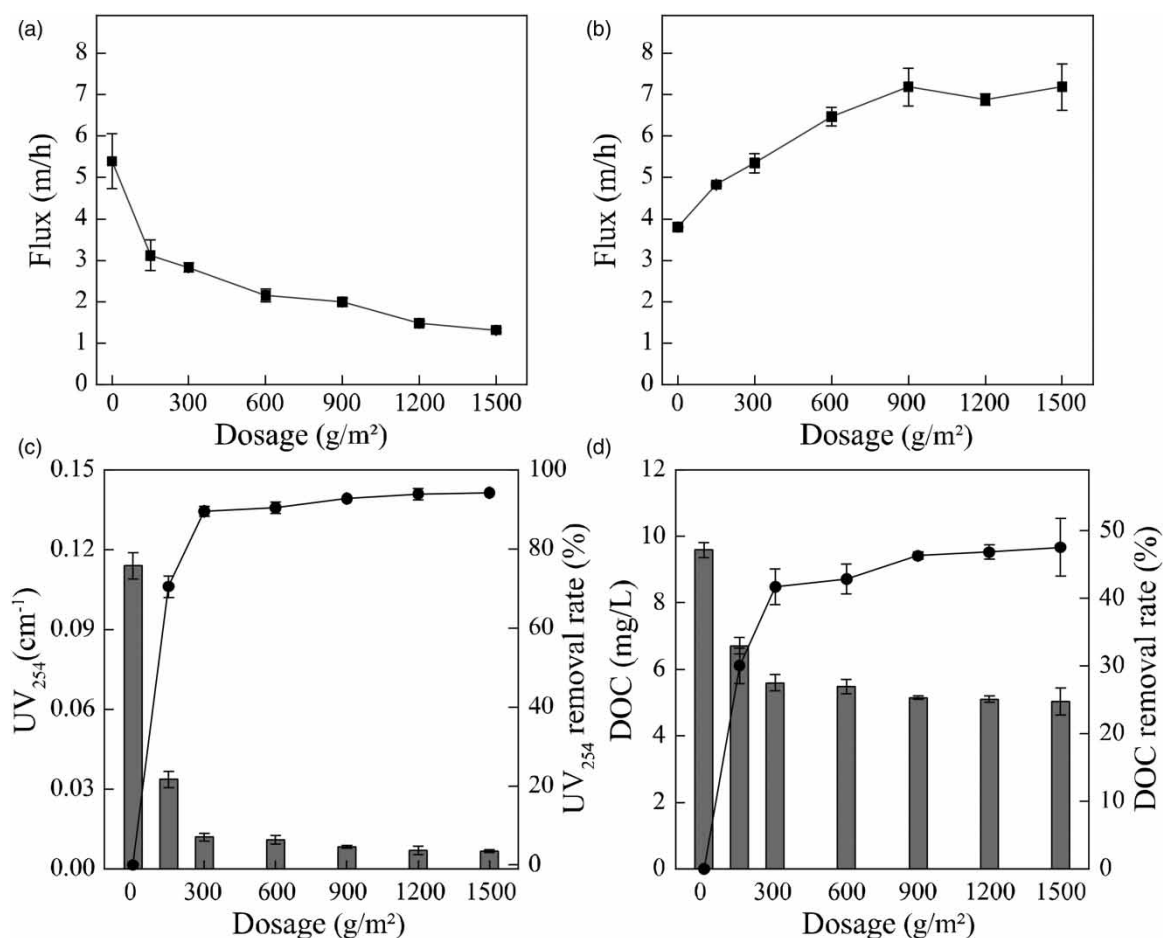


Figure 3 | Effect of PACPM dosages on the flux and organics removal. (a) Flux of the feed water sample through the PACPM. (b) Flux of the PACPM permeate through the 0.2- μm membrane (c) UV₂₅₄ removal and (d) DOC removal.

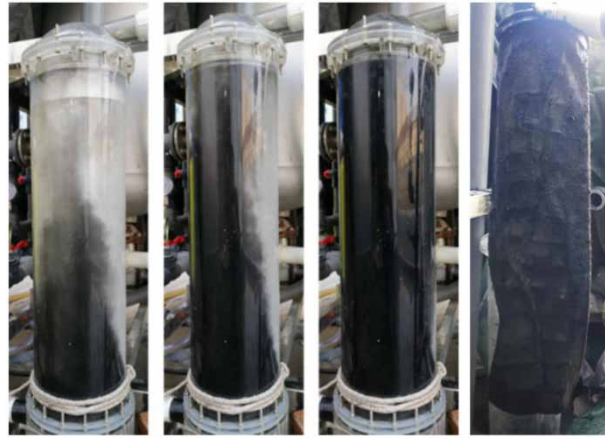


Figure 4 | Coating process of the pilot-scale PACPM.

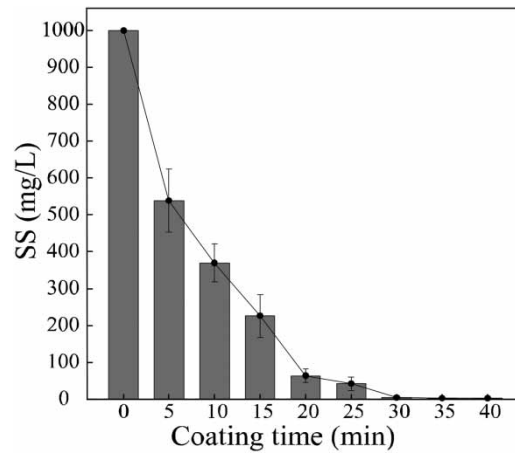


Figure 5 | Changes of SS of PACPM permeate during the coating process.

With the accomplishment of the coating progress, the feed water from the cmWWTP was treated continuously by PACPM at 0.02 MPa (Figure 2). The DOC and UV_{254} of PACPM permeate were measured hourly to evaluate the contaminant removal capacity of PACPM. As shown in Figure 6, the concentrations of the DOC and UV_{254} decreased sharply from 9.55 to 6.91 mg/L and from 0.120 to 0.077 cm^{-1} in the first hour, respectively. The removal rates of DOC and UV_{254} in the first

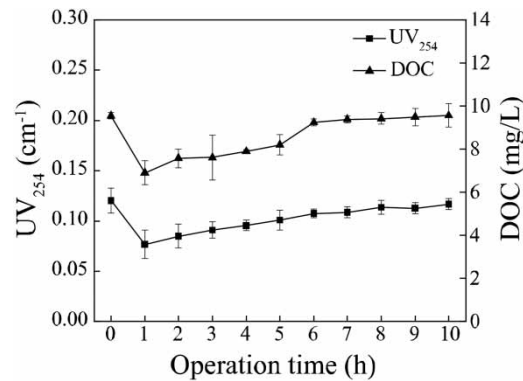


Figure 6 | Removal of DOC and UV_{254} by PACPM in different operation times.

hour were 27.6 and 35.8%. However, with the increase of operation time, the contaminant accumulated in the PAC particles, which led to a gradual decrease in the removal efficiency of organic matter by PAC. After 8 h of operation, the removal rate of UV_{254} by PACPM was reduced to about 5%, while the removal rate of DOC by PACPM was nearly zero. These results indicated that PACPM could effectively remove organic matter within a certain range, but it would gradually lose its removal capacity, which was consistent with the absorption characteristics of PAC.

The MW distributions of the organic matter of feed water and PACPM permeate in the first hour were measured to investigate the capability of PACPM to remove contaminants with different MWs. The result indicated that most of the organics with different MWs could be removed by PACPM. Among them, PACPM showed better removal ability for organic matter less than 1 kDa and more than 100 kDa, which could be explained by a combined effect of adsorption and filtration (Figure 7).

Similarly, the DOM fractions of feed water and PACPM permeate in the first hour were measured to investigate the capability of PACPM to remove different DOM fractional components. The water samples were fractionated into several components which were hydrophilic (HPI), hydrophobic (HPO) and transphilic (TPI) fractions using DAX-8 and XAD-4 ion exchange resins. The concentrations of HPI, HPO and TPI in the feed water were 2.24, 3.93 and 1.01 mg/L, respectively. After PACPM treatment, the concentrations of HPI, HPO and TPI decreased to 1.6, 2.61 and 0.67 mg/L, respectively. The removal rates of HPI, HPO and TPI were 26.34, 33.59 and 33.66%, respectively (Figure 8). The result indicated that both hydrophilic and hydrophobic fractions could be removed by PACPM, and the removal capacity of hydrophobic fraction by PACPM was slightly higher than that of hydrophilic fraction. The result was consistent with another study evaluating the removal of organic fraction by PAC (Wang *et al.* 2016).

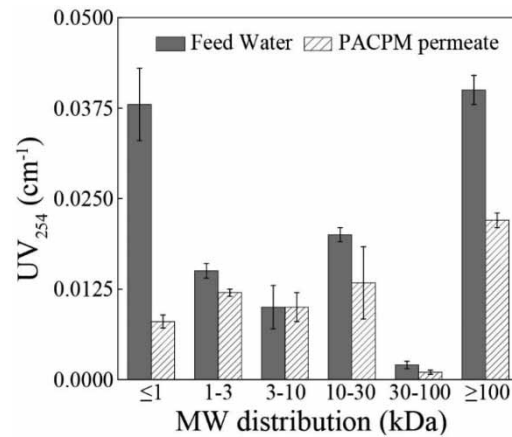


Figure 7 | MW distributions of feed water and PACPM permeate.

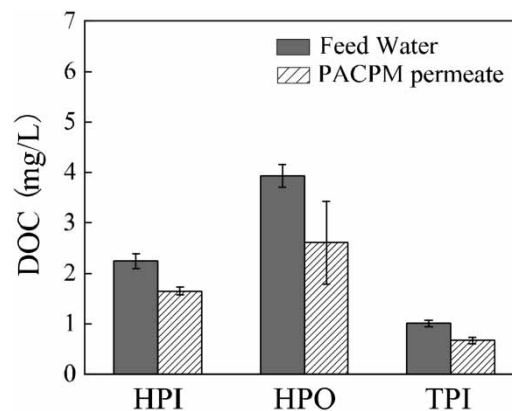


Figure 8 | DOM fractions of feed water and PACPM permeate.

Effects of PACPM on the performance of the UF system

To evaluate the effects of PACPM on the performance of the UF system, the SF variations between direct UF and PACPM-UF were compared (Figure 9). One operation cycle lasted for 20 min. The water backwash of the UF membrane was performed every 20 min operation with a duration of 40 s. The initial SF value of PACPM-UF was 83.26 LMH/bar, which was much higher than that of direct UF (65.37 LMH/bar). As shown in Figure 9(a), during 10 operation cycles, the SF values of PACPM-UF were always significantly higher than those of direct UF. Within each cycle, the decrease of SF for PACPM-UF was higher than direct UF. However, the recovery by water backwash of PACPM-UF was much better than that of direct UF (Figure 9(a)). After 10 operation cycles, the SF value of direct UF decreased from 65.37 to 56.64 LMH/bar, and the decrease rate was 13.4%. However, the SF value of PACPM-UF only decreased from 83.26 to 80.89 LMH/bar, and the decrease rate was only 2.8% (Figure 9(b)). These results indicated that PACPM could improve the performance of the UF system in a certain period of operation time, not only for the increase of the initial SF value but also for the increase of the recovery by water backwash.

The 24-h continuous operation experiments were conducted to assess the durability of PACPM for improving the performance of the UF system. As shown in Figure 10, the SF values of PACPM-UF were significantly higher than those of direct UF

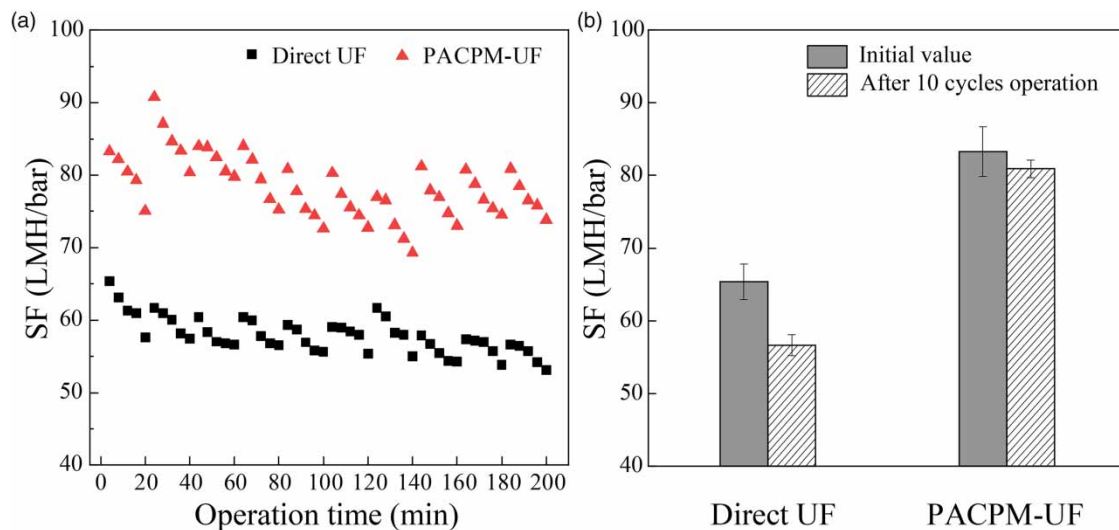


Figure 9 | Comparison of SF variations between direct UF and PACPM-UF: (a) SF variations in the period of 10 operation cycles and (b) SF of initial value and the value after 10 operation cycles.

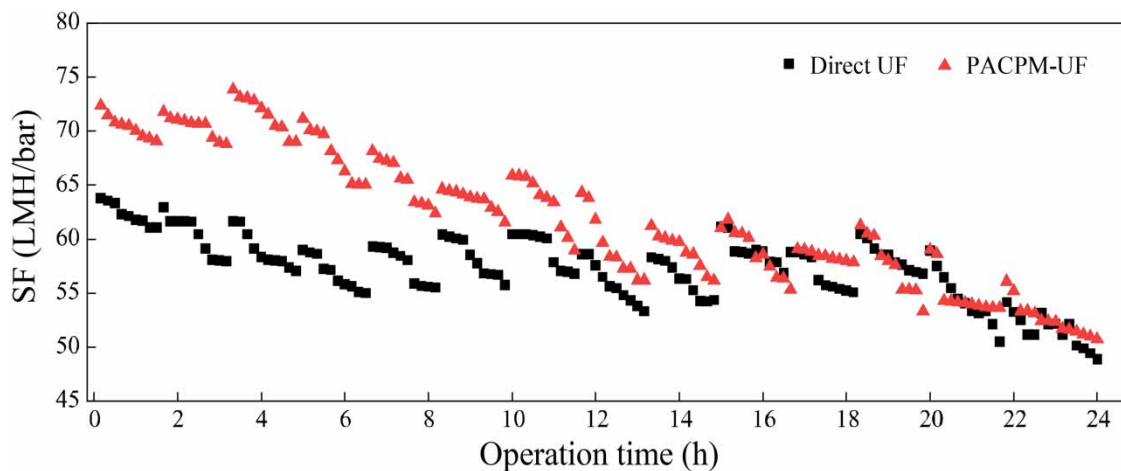


Figure 10 | Comparison of SF variations between direct UF and PACPM-UF in 24 h.

Table 3 | Water qualities of water backwash effluents

	Direct UF	PACPM-UF
UV ₂₅₄ (cm ⁻¹)	0.167 ± 0.015	0.114 ± 0.017
DOC (mg/L)	8.41 ± 0.33	7.31 ± 0.58
Turbidity (NTU)	21.39 ± 10.61	6.16 ± 0.96
SS (mg/L)	19.67 ± 15.80	13.12 ± 5.61

Table 4 | Water qualities of alkaline cleaning effluents of the CEB process

	Direct UF	PACPM-UF
UV ₂₅₄ (cm ⁻¹)	1.57 ± 0.69	0.79 ± 0.11
DOC (mg/L)	76.72 ± 8.82	44.18 ± 3.61
Turbidity (NTU)	7.48 ± 6.45	5.83 ± 3.28
SS (mg/L)	5.83 ± 2.20	6.01 ± 2.83

during the first 12 h. Even though PACPM was almost unable to remove organic matter after 8-h operation (Figure 6), the improvement of UF operation was maintained up to more than 12 h (Figure 10). This may be due to the formed pre-coating membrane which could still remove some particles and colloids. After 12 h, the performance of PACPM deteriorated further and the membrane itself became clogged, resulting in the SF value of PACPM-UF gradually approaching that of direct UF. The result suggested that PACPM could only improve the performance of the UF system for a period of time. Therefore, in order to improve the performance of the UF system steadily, other means may need to be implemented, such as increasing the dosage of PAC, increasing the filtration area, parallel operation of multiple groups of PACPM, regular replacement and regeneration of PAC.

To investigate the effect of PACPM on the UF membrane fouling, the water qualities of effluents for water backwash and the CEB process were analyzed. As shown in Table 3, the organic matter and particles in the backwash water for PACPM-UF were lower than those in the backwash water for direct UF. Similarly, the organic matter in the CEB alkaline cleaning effluent of PACPM-UF was also significantly lower than that of direct UF (Table 4). The water quality of backwash and CEB alkaline cleaning effluents showed that PACPM could effectively reduce the contaminant accumulation on the UF membrane. The recovery by the water backwash indicated that PACPM could remove the contaminant which was more difficult to rinse out from the UF membrane surface so as to improve the effect of water backwash (Figure 9).

CONCLUSIONS

In this work, we designed and established the PACPM with a bag filter as the base membrane. Based on the results of lab-scale experiments, the optimal PAC dosage of PACPM was determined as 300 g/m². The results of pilot-scale experiments indicated that PACPM could remove different kinds of organic compounds with different MWs and hydrophobicity during a certain period of time. Furthermore, PACPM could improve the performance of the UF system during a certain period of operation time, not only for the increase of the initial SF value but also for the increase of the recovery by water backwash. After PACPM lost the ability of organic matter removal, it could still maintain the optimization effect on the UF system for a certain period of time. Nevertheless, a regular replace-regeneration process is necessary to maintain PACPM performance.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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