Short communication

Micro-polluted surface water treatment and trace-organics removal pathway in a PAC-MBR system

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\textbf{A B S T R A C T}

Trace-organic contaminants elimination effectiveness and their removal pathways in a powder-activated carbon-membrane bioreactor (PAC-MBR) used for micro-polluted surface water treatment were investigated in this study. The results showed that PAC-MBR was capable of consolidating DOM elimination, enhancing trace-organic contaminants (2,4,6-trichlorophenol, nitrobenzene and trichloroethylene) removal efficiency, improving permeate biostability, and decreasing bulk organic content and specific fractional components, which was beneficial for membrane fouling mitigation. By series of well-designed batch-tests and estimation, trace-organics removal pathways were analyzed. The primary way of TCE removal was aeration volatilization, while that of NB and 2,4,6-TCP elimination was biodegradation which was greatly affected by biomass content within bulk. PAC addition was a positive way for effective trace-organics removal by increasing mixed solid adsorption capability, especially in start-up phase, and offering beneficial circumstances for biomass enrichment and functional bacteria accumulation. Thus, PAC addition into MBR is a simple and effective strategy to treat micro-polluted water resources.

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1. Introduction

Water resources contamination caused by irregular overflow and/or discharge of industrial wastewater that are not subjected to effective treatment around or inside urban agglomeration affects drinking water quality significantly [1]. It has been reported that a great number of trace-organic contaminants, such as trichloroethylene (TCE), nitrobenzene (NB) and 2,4,6-trichlorophenol (2,4,6-TCP), were found in some water resources in China [2]. Owing mainly to their potential adverse effects on human health, a reliable technique with stable removal effectiveness is highly expected. Membrane bioreactor (MBR) is an appealing technology that combines membrane filtration with biological degradation process for wastewater and drinking water treatment [3]. Considering its potential for the development of a novel, compact and high-rate organic chemicals removal/rejection system, MBR thus may lead to a fundamental advance in micro-polluted water resources treatment [4].

Different from the MBRs for wastewater treatment, where high biochemical oxygen demand (BOD) and ammonia removal efficiencies could be obtained as a result of sufficient activated sludge retained by membrane filtration [3], treatment efficiency of the MBR for water resources that has to confront non-degradable organic matters and co-existed refractory organics, is always unstable. In addition, some experimental results displayed that a potential risk such as refractory organics breakthrough would be occurred in MBR effluent [5]. Hence, it is of great significance to figure out the removal pathway of trace-organics in MBR, and then to look for a reliable enhancement method to guarantee effluent quality. However, many researchers mainly focused on the MBR effectiveness for individual trace organic removal, other than for a feeding mixture containing dissolved organic matters (DOM) and several of the refractory organics that may have possible inhibition or competition [4].

It has been noted that the treatment effectiveness of MBR relates closely with the pollutants and biomass properties, and can be enhanced by addition of powdered activated carbon (PAC) [6]. PAC has a large specific surface area for organic adsorption and a coarse and irregular surface providing favorable micro-environment for bacteria growth and accumulation, making it quite beneficial to elevate MBR capability in organics removal [7]. However, PAC improvement for MBR was a complex process as it would affect activated sludge and membrane filtration [8], and most of the studies on PAC-MBR hybrid technology aimed to evaluate...
2. Materials and methods

2.1. Laboratory-scale plat-sheet MBRs set-up and operation

Two identical submerged MBRs with working volume of 4.0 L were operated side by side. Plat-sheet polyvinylidene fluoride (PVDF) ultrafiltration membrane (Peier, China) with an average pore size of 0.08 μm was immersed for liquid-solid separation. The operational conditions were kept the same except that one reactor was added with a predetermined amount of PAC (~1 g/L) to form PAC-MBR. The PAC particles were profusely washed with double-distilled water to remove colloidal fractions, and then oven-dried at 105 °C for 24 h before used. A suction pump (BT100-2, Longer, China) was used to withdraw effluent through the membrane at a filtration-to-idle cleaning ratio of 8:2 min. Aeration was provided at the reactor bottom for DO supplement and continuous membrane cleaning. Both of MBR and PAC-MBR were operated in a constant flux mode, and trans-membrane pressure (TMP) was monitored with a manometer in mmHg to indicate membrane fouling. Three chemicals, TCE, 2,4,6-TCP and NB were selected as model trace-organics. Real raw water collected from a local reservoir (Xili Reservoir, Shenzhen, China) was used to prepare micro-polluted water resources containing DOM and trace-organics. On average, the concentration of DOM, NH₃–N, TCE, 2,4,6-TCP and NB was 5.0 mg TOC/L, 1.3 mg/L, 80 μg/L, 300 μg/L and 100 μg/L, respectively.

2.2. Trace-organics removal pathway analysis and estimation

Four major ways to remove trace organic substances were recognized, i.e. (i) volatilization by continuously aeration (Cvol), (ii) adsorption by suspended biomass (Cads), (iii) biodegradation by activated sludge biomass (Cbio), and (iv) rejection by membrane filtration (Cfil) [9] in MBR. Thus, the total trace-organics removed by the control MBR (C_T) could be subdivided by using Eq. (1).

\[ C_T = C_{vol} + C_{ads} + C_{bio} + C_{fil} \]  

(1)

With continuous permeate production by membrane filtration, membrane pore clogging and cake layer formation would occur, and then Cfil will become a combination of three parts, including intrinsic membrane rejection (Cfil-m), the rejection by membrane pore narrowing and clogging (Cfil-p), and adsorption and interception by formed cake sludge (Cfil-c). Therefore, Eq. (1) was modified to Eq. (2). Accordingly, series of well-designed batch-scale tests assays were carried out to estimate the specific amount of trace-organics removed by every removal pathways (SI-1).

\[ C_T = C_{vol} + C_{ads} + C_{bio} + C_{fil-m} + C_{fil-p} + C_{fil-cs} \]  

(2)

2.3. Biomass growth estimation and its community structure analysis

MLSS concentration in MBR was measured in accordance with the Standard Methods. The concentration of active microorganism was estimated in the term of lipid-P content in MBR bulk and membrane surface [10]. The modified phosphor lipid analysis method was carried out according to the reports of Findlay et al. [11]. Microbial community analysis in MBR and PAC-MBR was conducted following the procedure described by Lv et al. [12] (SI-2).

2.4. Analytical methods

Comparative treatment performance of MBRs was evaluated in terms of DOM, NH₃–N and trace-organics removal efficiencies. TOC was measured by a TOC analyzer (TOC-L CPN, Shimadzu, Japan). NH₃–N and UV absorbance at 254 nm (UV254) were determined by a spectrometer (UV2600, Shimadzu, Japan). TCE, NB and 2,4,6-TCP were determined following US EPA Method 502.2 and 609 by gas chromatography (7890A, Agilent, USA). Assimilable organic carbon (AOC) concentration was calculated following the method reported by Liu et al. [13]. The florescence characteristics of organic compounds in MBRs influent, bulk solution and effluent were examined by a fluoresence excitation–emission matrix (F-EEM) [14].

3. Results and discussion

3.1. Variations of treatment performance and biomass accumulation

As illustrated in Fig. 1a, with a feeding raw water containing DOM of about 5.0 mg TOC/L, DOM removal efficiency in MBR increased gradually, and stably kept at 60% after a week operation. In comparison, the DOM concentration in PAC-MBR effluent stably averaged at 1.81 ± 0.35 mg TOC/L throughout the experimental phase (Fig. 1a). It was reasonable to claim that PAC adsorption of organic substances would take major responsibility to the higher TOC removal efficiency in PAC-MBR initial phase. The removal performance of UV254 in MBR and PAC-MBR showed similar trend with those of DOM. In experimental start-up, UV254 removal efficiency by MBR was about 30%, whilst those of PAC-MBR were stably kept around 65% (Fig. 1b). After more than two weeks operation, both of MBR and PAC-MBR achieved up to 65% UV254 removal efficiency. The phenomenon was consistent with the results of Tian and his coworkers, where they found that better organics removal performances in the membrane filtration-involved system [15]. NH₃–N is mainly removed through biological way in drinking water treatment process, where sufficient nitrifying bacteria are necessary [16]. Herein, with the progression of reactor operation and biomass enrichment for more than 10 days, the NH₃–N concentrations in MBR and PAC-MBR effluent were stably below 0.5 mg/L (Fig. 1c). By using the MBR-involved biological treatment, the biostability of the finishing water was improved considerably, reflected by that the AOC decreasing from 67.4 to 48.2 and 28.7 μg/L in MBR and PAC-MBR, respectively (SI-3, Fig. S1). This improvement of biostability caused by PAC addition into MBR could eventually minimize bacterial re-growth and biofilm formation in water distribution systems.

Lipid-P contents in the bulk suspension and membrane surface were measured to estimate the profile of active biomass growth and accumulation. As shown in Fig. 1d, obvious accumulation of active biomass was observed during the initial two weeks in both of MBR and PAC-MBR bulk. Afterwards, lipid-P concentration reached to a relatively stable level of 11.2 nmol/ml at day 16 in MBR, and of 12.2 nmol/ml at day 12 in PAC-MBR. A higher lipid-P...
increasing rate in PAC-MBR may attribute to the fact that dosed PAC offered beneficial micro-environment for active biomass growth, and effective retention of biomass by membrane filtration. Similar phenomena were also found in the MBR and PAC-MBR membrane surface, where its lipid-P increased firstly and then reached to a relative stable level.

The microbial community variation and changes in raw water, MBR and PAC-MBR bulk suspension were analyzed. As shown in Fig. 2, similar to the community structure of typical drinking water treatment process involving biological organic degradation [17], Proteobacteria were the most abundant phylum in all of the samples, which accounted for more than 24.9–51.9% of the total bacterial sequences. After four days operation, the community structure of PAC-MBR bulk sludge changed significantly, while that in MBR was kept relative comparable with the raw water. A great decreasing of Firmicutes from 9.4% to 1.6%, and an obviously accumulation of Actinobacteria from 5.7% to 13.5% and Cyanobacteria from 9.7% to 18.2%, respectively, in PAC-MBR, might be resulted from PAC addition and its selective function for specific microbial species. As that most of the phylum Actinobacteria were heterotrophs, its abundance augment evidenced that PAC addition favored biomass accumulation in the system start-up phase. However, with the continuous operation of MBR and PAC-MBR, their microbial community and bacterial abundance on day 24 were quite similar. Although most of the functional bacteria were commonly found in water treatment process, some bacterial sequences were worthy of special attention. An average abundance of Nitrosopina in MBR and PAC-MBR bulk of 0.28% and 0.20%, respectively, was quite higher than that in raw water of 0.11%, which seem as a reliable explanation for their high NH₃–N removal efficiencies. Especially, it was to be remarked that Deinococcus, one specific microbe that was found in mature landfill leachate for refractory organic degradation [18], was observed in both of MBR and PAC-MBR after a long-term system operation, which may take major response for the trace organics biodegradation in a high removal efficiency (see detail in Sections 3.3 and 3.4).

3.2. DOM fractionation and its removal enhancement by PAC addition

The specific components of DOM that were removed distinctively by PAC were identified when the PAC-MBR system reached a steady-state. Five components, i.e. tyrosine, tryptophan, fulvic-like, soluble biological metabolites (SBM) and humic-like substances were categorized (SI-4, Fig. 52). The DOM and its fractionized components removed in MBR and PAC-MBR systems were estimated by F-EEM observation (Table 1). It was noted that humic-like substances were the major fraction in raw water, followed by SBM, fulvic-like, tryptophan, and tyrosine. The DOM content in MBR bulk accumulated to more than 1.6 times higher than that in its influent. In contrast, PAC-MBR bulk contained rather low tryptophan content, and its SBM and humic-like substances content also showed a slight decreasing trend. However, tyrosine component increased dramatically more than 44.4%, which may due to the PAC-assisted enhancement to membrane interception [19]. Both of MBR and PAC-MBR bulk contained consistently higher organic content than that of permeate, which agreed well with the results found by Sun et al. [20]. Tryptophan was preferentially intercepted by membrane filtration compared with others, as there were 57.9% and 67% removal efficiency in MBR and PAC-MBR, respectively. Tyrosine and fulvic-like substances were easily to pass through membrane, resulting in its rather high component fraction in MBR.
permeate. PAC addition could enhance all of the DOM components removal capability, by which an excellent effluent quality could be maintained. The membrane retention efficiency of tyrosine and fulvic-like substances improved greatly from 22% in MBR to 55.6% in PAC-MBR, and from 29.1% in MBR to 45.7% in PAC-MBR, respectively. Meanwhile, there were a certain increasing amount of tryptophan, SBM and humic-like substances rejection efficiency in PAC-MBR. Therefore, it could be recognized that there was an obvious organic substances selective removal in MBR that may affected effluent quality, especially for the situation where contained insufficient biomass.

3.3. Trace-organic contaminants removal performance

The removal efficiency of TCE by PAC-MBR was consistently higher than that of MBR, as the average TCE removal efficiency in MBR and PAC-MBR was 65% and 79%, respectively (Fig. 3a). This discrepancy related closely with the PAC addition, by which the TCE would be effectively absorbed. This result was in accordance with the findings of Erto and his coworkers [21], where they revealed that activated carbon was able to remove TCE from pure water, and more than 20 mg TCE/gPAC adsorption rate could be obtained. Notwithstanding, the rather low adsorption rate herein (no more than 1.5 mg TCE/gPAC) may be due to low influent TCE content and co-existed pollutants in bulk solution that would compete PAC reactive point [8]. The humic-like substances in bulk solution showed limited influence onto the TCE removal in PAC-MBR (Fig. 3a), which has been well demonstrated in previous study [22]. Biomass enrichment in these two systems displayed insignificant improvement on TCE removal, implying that the TCE was rarely removed by biomass degradation. As one refractory organic contaminant, NB is difficult to be biodegraded in conventional activated sludge process [23]. Similarly, NB removal efficiency in MBR was about 20% at the experimental beginning, and increased gradually to 90% on day 15 (Fig. 3b). This improvement of NB removal performance indicated that the membrane interception of NB was rather limited, and would mainly attributed to biomass enrichment in MBR. In comparison, PAC-MBR had a higher NB removal efficiency about 81% at the experimental beginning (the first 5 days), which may relate well with the PAC adsorption, and was then improved further to 92% on day 14 (Fig. 3b). For 2,4,6-TCP removal, PAC-MBR displayed a quit better removal efficiency than those of MBR throughout the experimental study. More than 86% 2,4,6-TCP could be stably removed in PAC-MBR, whilst only 23% 2,4,6-TCP eliminated in MBR, at the experimental beginning. Nevertheless, after 10 days operation, the 2,4,6-TCP effluent of MBR and PAC-MBR decreased below 30 and 20 μg/L, respectively (Fig. 3c).

3.4. Pathway analysis of trace-organics removal in MBR and PAC-MBR

Upon the operational steady-state reached, TCE could be effectively removed in MBR. With an influent containing 80 μg/L TCE, \( C_{\text{in}} \) in MBR and PAC-MBR averaged at 49.1 and 46.3 μg/L, respectively (Fig. 4a). In contrast, the average \( C_{\text{out}} \), \( C_{\text{bio}} \), and \( C_{\text{fil}} \) in MBR were 0.5, 4.6, and 6.1 μg/L, respectively. It was thus found that aeration was the dominant pathway for TCE removal in MBR, other than membrane filtration and biodegradation. This finding agreed well with the results of Tadkaew who found that the membrane rejection capability of moderately hydrophobic organic compounds in MBR was relative low [24]. The high effectiveness of aeration in MBR resulted in a quite low TCE concentration in its bulk throughout the operational phase, which might be an important reason for the low \( C_{\text{out}} \) in MBR. Through up to 25 days acclimation, \( C_{\text{bio}} \) displayed a slightly decline trend, while \( C_{\text{fil}} \) was improved in a certain level, which resulted in a stable \( C_{\text{fil}} \) of 62%, whilst both of \( C_{\text{fil-m}} \) and

![Fig. 2. Abundances comparison of different phyla in raw water, MBR and PAC-MBR bulk solution, and their changes with operational time.](image-url)

Table 1

Comparison of removed DOM and its components by MBR and PAC-MBR.

<table>
<thead>
<tr>
<th>The ratio in raw water (%)</th>
<th>Removal (%) of mixed liquor⁴</th>
<th>Removal (%) of filtration⁵</th>
<th>Total removal (%)⁶</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MBR</td>
<td>PAC-MBR</td>
<td>MBR</td>
</tr>
<tr>
<td>Tyrosine</td>
<td>7.0</td>
<td>-21.8</td>
<td>-44.4</td>
</tr>
<tr>
<td>Tryptophan</td>
<td>12.7</td>
<td>-28.6</td>
<td>20.6</td>
</tr>
<tr>
<td>Fulvic-like</td>
<td>13.0</td>
<td>-43.7</td>
<td>-3.0</td>
</tr>
<tr>
<td>SBM</td>
<td>22.4</td>
<td>-19.4</td>
<td>8.5</td>
</tr>
<tr>
<td>Humic-like</td>
<td>44.8</td>
<td>-46.5</td>
<td>0.8</td>
</tr>
</tbody>
</table>

⁴ Removal (%) of mixed liquor = (concentration in raw water − in mixed liquor)/(concentration in raw water) × 100.

⁵ Removal (%) of filtration = (concentration in mixed liquor − in the effluent)/(concentration in mixed liquor) × 100.

⁶ Total removal (%) = (concentration in raw water − in the effluent)/(concentration in raw water) × 100.
C\textsubscript{fil-p} kept consistently low. Continuous increasing of C\textsubscript{fil} was mainly attributed to the formation of cake layer on the membrane surface, which would enhance TCE removal by active area enlargement and biomass adsorption [25]. Different from MBR, C\textsubscript{ad} in PAC-MBR was caused mainly by PAC addition, which had to decrease after more than 15 days due mainly to the PAC saturation. In contrast, C\textsubscript{bio} in PAC-MBR displayed a continuous increasing trend with operational time. This improvement of C\textsubscript{bio} in PAC-MBR related closely with the lipid-P increasing profile, which evidenced that biomass accumulation by membrane retention was beneficial for trace organics biodegradation.

Aeration volatilization had negligible contribution to NB removal in MBR and PAC-MBR, which was indicated by the constantly small C\textsubscript{vol} as low as 2.4 and 2.8 $\mu$g/L, respectively (Fig. 4b). Whilst, NB has small molecular weight and high solubility, making it rather limited interception capacity by UF membrane. Cake layer formation on the membrane surface improved C\textsubscript{fil-eff} from 4 to 20 $\mu$g/L in MBR, and from 8 to 18 $\mu$g/L in PAC-MBR, and their continuous elevation could be expected. C\textsubscript{bio} in MBR displayed directly correlation with the biomass accumulation profile, and it increased dramatically up to 85 $\mu$g/L in day 20 that was equivalent to 64% NB removal efficiency with lipid-P content increased from 2.0 to 11.2 mmol P/mL. In addition, C\textsubscript{ad} was kept stably lower than 0.5 $\mu$g/L that was independent with biomass content, which concurred with the fact that NB was difficult to be adsorbed onto activated sludge due to its strong hydrophobicity properties [26]. In contrast, a faster increase rate of NB biodegradation capability than that in MBR was observed in PAC-MBR, whose C\textsubscript{bio} increased to 54 $\mu$g/L only after 8 days operation. It was reasonable to deem that PAC addition provided microorganism carriers and changed the bulk solution conductivity to a favorable circumstance for microorganism accumulation. Afterwards, biodegradation began to be the major pathway for NB removal during the steady-state phase of PAC-MBR. However, PAC adsorption process played a dominant role in NB removal in the initial 5 days, since that, its C\textsubscript{ad} was much greater than C\textsubscript{bio} due to the severe lack of biomass.

As for 2,4,6-TCP, at the MBR operational beginning, where had very limited bulk biomass content, C\textsubscript{vol} was the dominant removal pathway, which averaged at 35 $\mu$g/L (Fig. 4c). After more than 12 days, MBR obtained up to 97.5% 2,4,6-TCP removal efficiency. The major reason was that the biodegradation activity, mainly indicated as C\textsubscript{bio}, increased from below 20 $\mu$g/L to a level as high as 200 $\mu$g/L in day 14, which was positively related with the biomass content that increased from 2.0 to 8.2 mmol P/mL. Meanwhile, C\textsubscript{ad} in MBR was also correspondingly improved to about 0.53 $\mu$g/L. In comparison, in PAC-MBR, C\textsubscript{ad} was kept above 240 $\mu$g/L owing to the high 2,4,6-TCP adsorption capability of PAC. Thanks to the initial PAC adsorption, the total 2,4,6-TCP removal efficiency was as high as 96.7% at the beginning time. C\textsubscript{bio} in PAC-MBR had a much faster increasing rate compared with that in MBR, since that it increased to up to 136 $\mu$g/L at day 4, which could compensate the loss of PAC adsorption. Membrane filtration displayed a rather low retention capability for 2,4,6-TCP in both MBR and PAC-MBR, and the sum of C\textsubscript{fil-m}, C\textsubscript{fil-p} and C\textsubscript{fil-c} was kept lower than 56 and 44 $\mu$g/L in these two systems, respectively, which major attributed to the cake layer formed on the membrane surface.

Therefore, the biodegradation was evidenced to take major responsible for NB and 2,4,6-TCP removals in MBR process, implying that a stably high removal efficiency depended on its biomass abundance. It was observed that PAC could effectively improve trace-organics removal efficiencies after PAC dosage into MBR, and PAC would also assemble with activated sludge flocs to produce more active surface for trace-organics adsorption, other than

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Fig. 3. Comparison of trace-organic contaminants removal in MBR and PAC-MBR: (a) TCE, (b) NB and (c) 2,4,6-TCP.
improving the biodegradation capability in this short duration. PAC also had a positive influence on the total membrane rejection efficiencies in these three trace-organics by membrane coverage and pore clogging. However, with the continuous permeate production and therewith biomass accumulation, the capability of PAC declined gradually, resulting in that MBR and PAC-MBR had rather comparable effectiveness in trace organics removal efficiencies.

3.5. Membrane fouling and its alleviation by PAC addition

PAC-MBR showed a rather moderate membrane fouling compared with MBR. TMP in MBR increased rapidly from 0 to 41 kPa in the first 48 h (SI-5, Fig. S3). In comparison, the TMP in the PAC-MBR increased from 0 to 35 kPa took more than 100 h. It was reviewed that PAC addition into MBR could mitigate membrane fouling in a certain level, since that PAC could absorb humic-like and organic
substances to result in a low organic content in PAC-MBR bulk. Moreover, the high humic substance in the MBR would also affect the fouling recovery ratio, where the irreversible flux reduction was obviously occurred after chemical cleaning. The fouled membrane surface from MBR was closely covered by a thin sticky fouling layer, and physical cleaning could hardly remove these small size particles that induced irreversible fouling layers and caused quick initial membrane flux decline. In contrast, the surface of fouled membrane from PAC-MBR had an even and compact cake layer (SI-5, Fig. S4), which could be easily removed by physical cleaning to achieve obvious and clean pore distribution surface profiles.

4. Conclusions

PAC-MBR was an effective process to treat micro-polluted water resources containing TCE, NB, and 2,4,6-TCP. Comparative experimental results showed that PAC-MBR has consistently higher removal efficiency in DOM, ammonia and AOC, compared with conventional MBR. PAC could decrease DOM content and its fractionized components concentration in MBR bulk, which was beneficial to permeate quality and membrane flux. After trace-organics removal pathways specification, it was revealed that the primary pathway of TCE was aeration volatilization, while that of NB and 2,4,6-TCP was biodegradation. Notwithstanding, the importance of different removal pathways related closely with the activate biomass accumulation and PAC addition profiles. Thus, it is deduced that PAC addition is important for trace-organic contaminants removal in MBR, especially in the start-up phase where has insufficient biomass content for organic removal and biodegradation.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jprocbio.2015.05.021

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