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Ultrafiltration of distinct natural waters: correlation of fouling resistances with water constituents

Junxia Liu[a,b,*], Zhihong Wang[a], Bangqing Cao[c], Bingzhi Dong[b,*], Dongsheng Zhao[b], Huaqiang Chu[b]

[a]*Faculty of Civil and Transportation Engineering, Guangdong University of Technology, Guangzhou 510006, China, Tel./Fax +86 20 39322515, email: whjunxia@163.com (J. Liu), dbz77@tongji.edu.cn (B. Dong)*
[b]*State Key Laboratory of Pollution Control and Resource Reuse, School of Environmental Science and Engineering, Tongji University,Shanghai200092, China*
[c]*Academy of Civil Engineering & Architecture, Nanyang Normal University, Nanyang 473061, China*

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ABSTRACT

This paper was to explore the correlation of fouling reversibility with raw water qualities by investigating the ultra filtration (UF) fouling resistance of distinct surface waters (lake water, campus water, river water and reservoir water) and by analyzing these water characteristics. Ultra filtration performance revealed that the order of different natural waters was lake >> campus > river > reservoir according to their impact on trans-membrane pressure (TMP) increase. Hydraulically reversible fouling resistance (R_{re})was largely responsible for the sharply elevated TMP in lake water, while hydraulically irreversible fouling resistance (R_{ir}) contributed much to the increased TMP of the other three waters. High performance size exclusion chromatography (HPSEC) coupled with peak-fitting indicated that biopolymers (BP) fractions were mainly retained by UF membrane, while a small quantity of low molecular weight (LMW) and humic substances (HS) were held back. Principal component analysis (PCA) method was also used to identify fouling reversibility associated with water constituents. Results manifested that BP as well as fulvic-acid-like substances were the main components of hydraulically reversible foulants, while LMW contributed greatly to the hydraulically irreversible foulants. The proposed PCA method has the potential for diagnosing and controlling the predominant foulants present in natural waters.

Keywords: Ultra filtration; Fouling resistances; HPSEC; Peak-fitting; Principal component analysis

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1. Introduction

The main research issue of low-pressure membrane (LPM) systems is membrane fouling, for which natural organic matters (NOM) was responsible during natural water treatment [1]. Characterization of NOM in the feed water can provide useful information about their contribution to membrane fouling. The molecular weight (MW) distribution of organics has great influence on membrane filtration performance. Fan et al. found that high MW fractions of NOM (> 30 kDa) contributed to flux decline greatly [2], while Filloux and Ayache suggested Low MW compounds had a high fouling potential [3,4]. However, some other studies disapproved these points. For instance, Arabi et al. suggested that MW with the range of 10–100 kDa, rather than >100 kDa and 1–10 kDa fractions, was responsible for membrane fouling [5]. Yamamura et al. revealed that small MW components narrowed the membrane pores firstly and high MW components consequently plugged the membrane pores and deposited on the membrane surface

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*Corresponding authors.

[6]. No consistent conclusions have been achieved about this issue till now, which may be explained by (1) NOM is complex, various and unstable, thus difficult to control and predict; (2) some results for observing membrane fouling, obtained based on one water only, were lack of universal meaning. For better elucidate the water characteristics on membrane fouling, investigation on UF of distinct natural waters were necessary.

High performance size exclusion chromatography (HPSEC) and fluorescence excitation-emission matrix (EEM) spectroscopy proved useful and promising tools to characterize natural water constituents and these constituents' characteristics impact on membrane fouling [7–10]. Using HPSEC, NOM could be fractionated into different components [11]. Recently, peak-fitting was testified to be helpful in separating chromatographic peaks into isolated fractions and provide quantitative information to identify the removable and non-removable components of dissolved organic carbon (DOC) by coagulation [12–14]. In addition to HPSEC, fluorescence EEM also proved to be of use to comprehend membrane fouling behavior in drinking water treatment process [15,16]. With F-EEM, humic-like, fulvic-like and protein-like substances in NOM could be differentiated by high sensitivity F-EEM [7,17,18]. Sutzkover-Gutmanetal [19] suggested that the accumulation of humic-like substances caused severe membrane fouling, while Peldszusetal [20] found that protein-like matters were responsible for the reversible and irreversible fouling of LMP membrane. Additionally, Peirisetal [7] reported that both humic- and protein-like substances contributed to the hydraulically irreversible fouling. There were obvious contradictory results on the fouling tendency of different fractions detected by F-EEM. It indicated that which constituents of F-EEM contributing to fouling were not well elucidated. In addition, in previous studies, the components of NOM were generally classified by HPSEC or F-EEM, however, few literature was available for isolating NOM into different components using a combination of these methods, i.e., EEM and HPSEC coupled with peak-fitting.

LPM fouling is generally classified into hydraulically reversible and irreversible fouling. Although LPM was periodically backwashed to control fouling, some fouling matters accumulated in/on the membrane during long-time running. These depositing foulants, which could only be eliminated by chemical cleaning, resulted in irreversible fouling with the sustaining loss of membrane permeability [21]. Fouling reversibility is of great practical significance and its analysis will help to identify fouling mechanisms [22,23]. Yet, limited studies were available to analyze the variations of reversible and irreversible fouling against water constituents to determine predominant fouling parameters.

Principal component analysis (PCA), a multivariate statistical analysis method, has applications for assessing the contributions of major fouling components present in natural water and for understanding the fouling mechanisms of these components from a fundamental perspective [4,7,9]. Nevertheless, there are limited studies on the comparison of fouling resistances with different natural waters to determine predominant fouling parameters using PCA method.

The main objective of this paper was (1) to identify which constituents in raw water were responsible for hydraulically reversible and irreversible fouling; (2) to expound the behavior changes of fouling resistances during the whole filtration process; (3) to propose a proper method to diagnose the predominant foulants present in natural waters. The main novelty of this work was as follows: (1) a combination of EEM and HPSEC with peak-fitting was applied to analyze the characteristics of foulants in natural waters; (2) PCA method was used to identify fouling reversibility associated with water constituents; (3) behavior changes of fouling resistances during the whole filtration process were explored. The results were expected to provide useful information for understanding the mechanism of membrane fouling during surface water treatment.

2. Material and methods

2.1. Source water

Four natural waters were selected for this study. They are the Sanhaowu river in Tongji University (campus water), which represented the surface water polluted by allochthonous NOM; the Huangpu river water in Shanghai City (river water), which represented the river polluted by industrial and human waste; the Qingcaosha reservoir in Shanghai City (reservoir water), which presented the slightly polluted reservoir water; and the Taihu Lake in Jiangsu Province in June (lake water), where algae bloom outbreaks (i.e., algae cell concentrations was greater than 1.0 × 10^5 cells/L). The elementary water qualities are summarized in Table 1.

2.2. UF experimental

A schematic exhibition of the UF process is shown in Fig. 1. Hollow-fiber UF polyvinylidene fluoride membrane, supplied by Toray, was used in this study. The surface area and nominal pore size were 0.16 m^2 and 0.02 μm respectively.

Raw water was firstly pre-treated by filtration membrane (10 μm) to remove large suspended matters, and then pumped into the feed water tank. The filtration process in each cycle lasted 90 min: 2 min for filling water, 86 min for filtration, and 1 min for forward wash by feed water and 1 min for backwash by DI water in succession, maintaining a fixed flux of 70 L/m^2/h. The whole process was controlled by a programmable logic controller (PLC). At the termination of the filtration, the fouled membrane was successively cleaned by DI water, acid agent, alkali agent and oxidant agent.

Table 1
Elementary water qualities of sourcewaters[a]

	Campus	Reservoir	River	Lake
Turbidity (NTU)	6.2	17.6	40.9	44.6
UV_{254}(cm^{-1})	0.073	0.037	0.105	0.100
DOC(mg/L)	3.356	1.919	4.468	5.936
SUVA(L/mg m)	2.209	1.928	2.350	1.685

[a] The data in Table 1 are average value of samples.

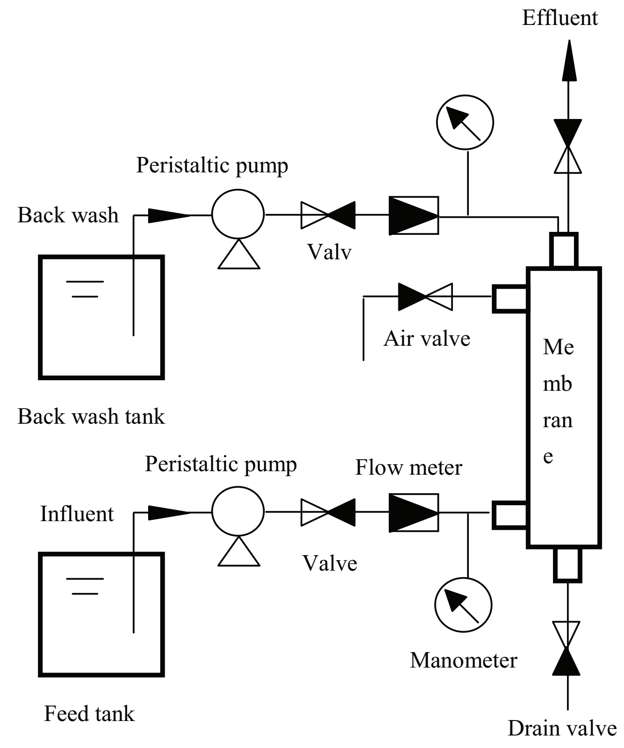

Fig. 1. Schematic diagram of the UF set-up.

2.3. Fouling reversibility

Fouling behavior was expressed by the resistance-in-series model [24,25], as expressed in Eq. (1).

$$R = \frac{TMP}{\mu J} \tag{1}$$

where TMP was the trans-membrane pressure (bar), μ was the dynamic viscosity (Pa·s), J was the permeate flux (LMH). The values of different resistances were calculated via Eq. (2)–(5) according to the previous report [25].

$$R_{\mathrm{m}} = \frac{TMP}{\mu J_0} \tag{2}$$

$$R_{\mathrm{t}} = \frac{TMP}{\mu J_{\mathrm{e}}} \tag{3}$$

$$R_{re} = R_t - \frac{TMP}{\mu J_1} \tag{4}$$

$$R_{ir} = R_t - R_{re} - R_m \tag{5}$$

where R_t, R_m, R_{re} and R_{ir} represented total resistance (m^{-1}), membrane intrinsic resistance (m^{-1}), hydraulic reversible resistance (m^{-1}) and hydraulic irreversible resistance (m^{-1}) respectively. In this paper, R_{re} and R_{ir} were differentiated by hydraulic backwash. J_0 was the virgin membrane flux (LMH) at initial stage, J_e was the fouled membrane flux (LMH) at the end of each cycle, and J_1 was the membrane flux (LMH) after hydraulic backwash.

2.4. Analytical methods

Turbidity was measured using a Turbidimeter (2100N, Hach, USA). Dissolved organic carbon (DOC) was determined by a total organic carbon (TOC) analyzer (Shimadzu TOC-L, Japan), UV254 by a UV-spectrophotometer (DR5000, Hach, USA), and MW distribution by HPSEC (Waters e2695, USA; UV254 detector, Waters 2489, USA; TOC analyzer, Sievers 900 Turbo, USA). Peak Fit (Version 4.12, Systat Software Inc., USA, CA) was applied in this analysis, using the method explained by Lai et al. [14]. The fluorescence EEM of samples was recorded on a fluorescence spectrometer (Cary Eclipse, Varian, USA) following the method described by Chen et al. [26]. PCA was adopted via the XLSTAT2015 software to expound the relationship of fouling resistances with water characteristics.

3. Results and discussion

3.1. UF performance

Fig. 2 presents the TMP changes of UF of the four natural waters. It should firstly be noted that in the primary design, there were six operating cycles in total. However, the TMP sharply increased during UF of lake water (algae rich water), thus only four cycles were conducted for this water. UF of the other three waters were conducted for 6 cycles (i.e. campus, river and reservoir waters).Different water exhibited distinct TMP in an order of lake >> campus > river > reservoir. The changes of total resistances are shown in Fig. 3. At the end of the 1st filtration cycle, R_t of lake increased to 6.81 × 10^{12} 1/m, far surpassing the other three waters. The total resistance of Lake consequently elevated to 9.11 × 10^{12} 1/m, 9.41 × 10^{12} 1/m and 10.26 × 10^{12} 1/m, respectively at the end of the 2nd, 3rd and 4th cycles, indicating severe membrane fouling. UF

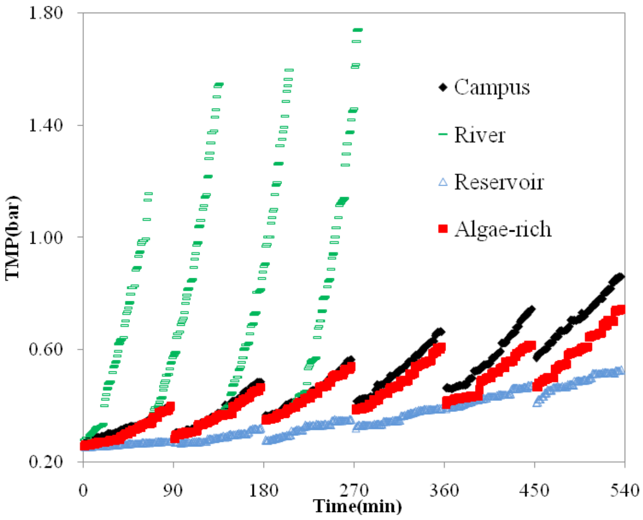

Fig. 2. TMP variations under UF of different waters.

of river and campus waters showed a similar R_t during the first cycle. Yet, as the system proceeded, R_t of campus water was slightly higher than the river water. The values of R_t for campus and river water were 5.07 × 10^{12} and 4.37 ×10^{12} 1/m respectively, at the completion of the experiments. Reservoir water exhibited a low-to-moderate flux decrease, i.e., the value of R_t was 3.11 × 10^{12} 1/m, implying the lowest fouling potential.

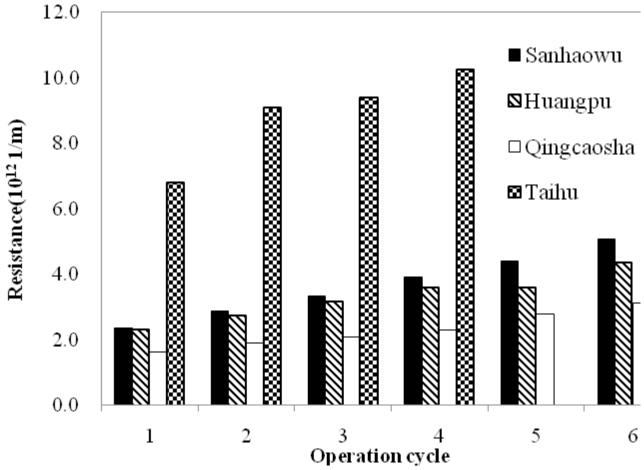

Fig. 3. Total resistance variations under UF of different waters.

In order to further investigate the changes of fouling resistances, R_{re} and R_{ir} in each cycle were calculated (Fig. 4). R_{ir} presented continuous increase as the operation prolonged, probably because their reversible foulants were not removed by the hydraulic backwash and continually accumulated on the membrane surface or plugged in the membrane pores. It could be calculated that the values of R_{ir}/R_t were 65.8%, 68.6% and 64.1% for campus water, river water and reservoir water, respectively,suggesting that R_{ir} was the main contributor to total fouling for these three waters. Nevertheless, a different phenomenon was also observed for lake water. R_{ir} in lake water was much smaller compared with R_{re}, and the proportion of R_{ir} in R_t from 1st to 4th cycle was 11.09%, 8.91%, 10.01% and 11.68% respectively. It appeared that R_{re} was the critical factor for the increase of total fouling, rather than R_{ir}.

3.2. Water characteristics

Fig. 5 shows the MW distribution of the four natural waters, using HPSEC coupled with UV_{254} and TOC detection. Three peaks were observed in the HPSEC. Peak 1 (MW < 2 kDa) had an obvious TOC response but a weak UV254 response, probably related to the low molecular

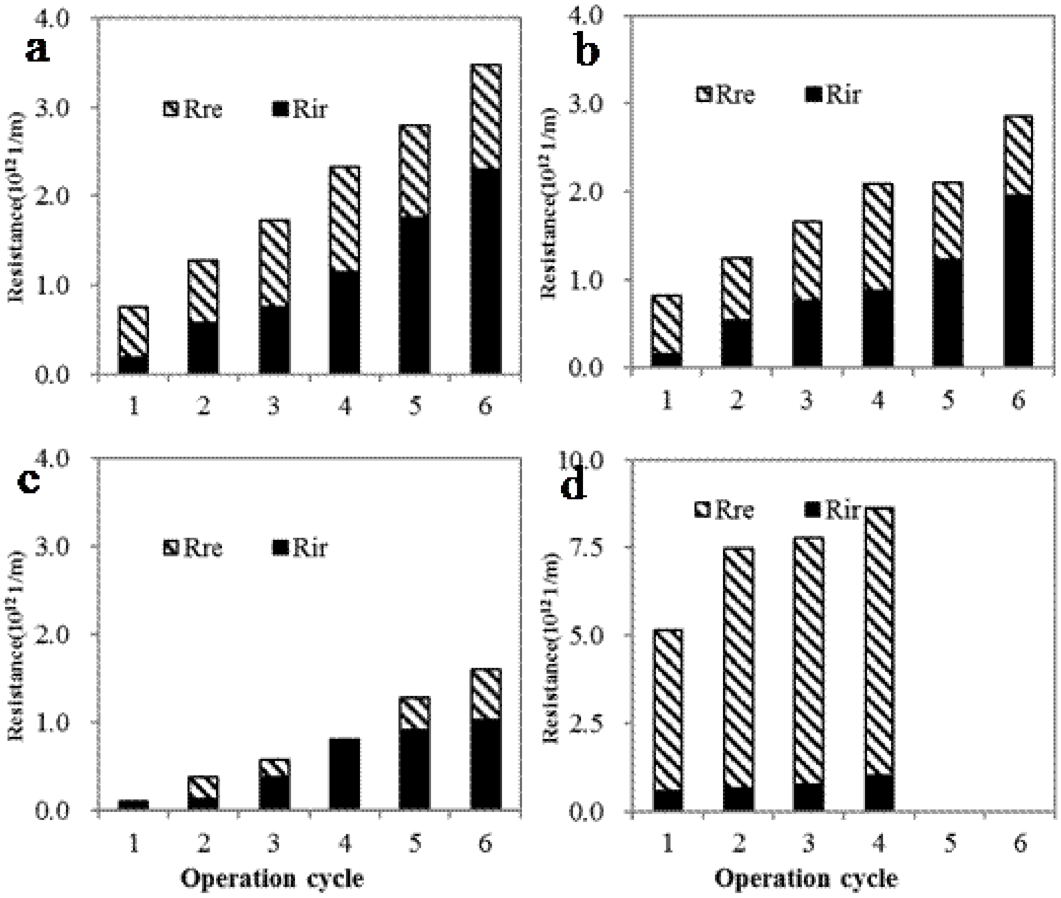

Fig. 4. R_{re} and R_{ir} variations under UF of campus water (a), river water (b), reservoir water(c) and lake water (d).

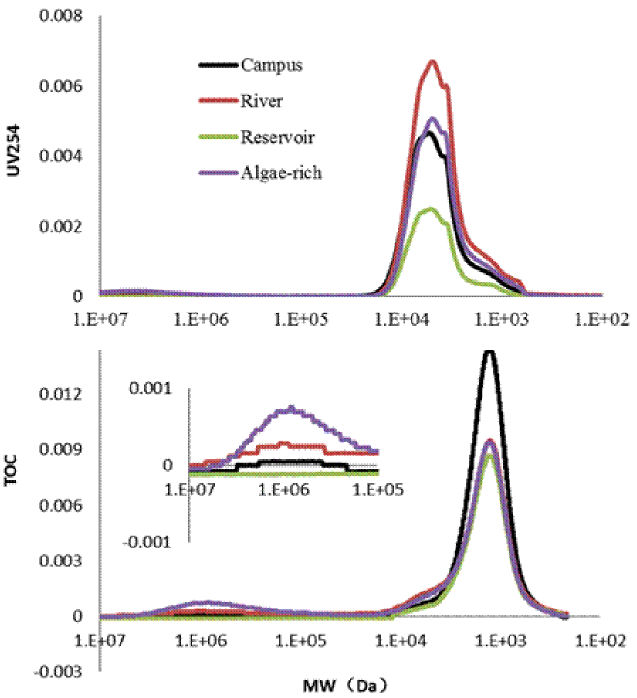

Fig. 5. MW distribution of raw waters.

weight matters (LMW) such as LMW neutrals, LMW acids and building blocks [4,11,13,14]. On the contrary, the 2nd peak (2 kDa < MW < 25 kDa) had a weak TOC response but a strong UV254 response value, identified as humic substances (HS), mainly very hydrophobic organic matters such as humic acids and unsaturated aromatic compounds [27]. The last peak ($10^5 < MW < 10^7$ Da) had a weak TOC response but no UV 254 response, regarded as biopolymers (BP), such as colloid, polysaccharide (PS), proteins (PN), etc. [3,8]. Peak-fitting was used to separate MW distribution of raw water into three isolated peaks (Fig. 6). The peak values were 1,420 Da, 5,250 Da and 10^6 Da for LMW, HS and BP, respectively. Fractions and removal efficiency of UF for different waters are shown in Fig. 7. It could be observed that in all the four waters, LMW fraction was the main component, followed by HS, while BP only accounted for a small part. A comparison of the fractions in different waters revealed that LMW content in campus was much higher than that in other three waters. The differences of HS fraction in the four waters were not obvious, while BP fractions exhibited remarkable differences in these waters: lake water has an obvious BP response while limited BP fraction was obtained in reservoir water. As expected, UF alone could only remove BP fraction effectively [14,28] and more than half of the BP fractions were retained by UF of campus, river and lake waters. This could be explained by the main mechanism of UF via size exclusion [7]. The normal membrane pore size in our study was 0.02 μm, equivalent to the MW distribution between 1×10^5 Da and 2×10^5 Da, as calculated by the previous method [29]. From a comparison of the NOM fraction size and membrane pore, it could be drawn that a majority of BP was retained by UF, while LMW and HS could pass through the pores due to the size exclusion.

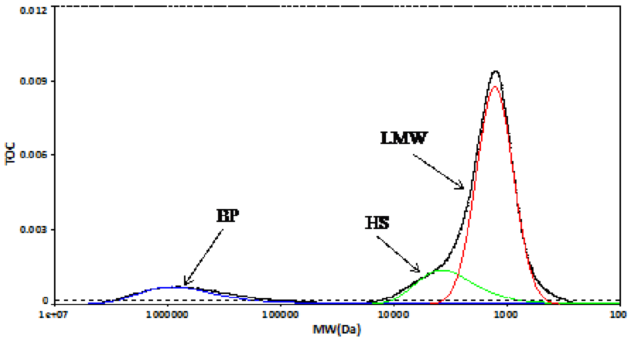

Fig. 6. LC-OCD analysis with peak-fitting of raw water.

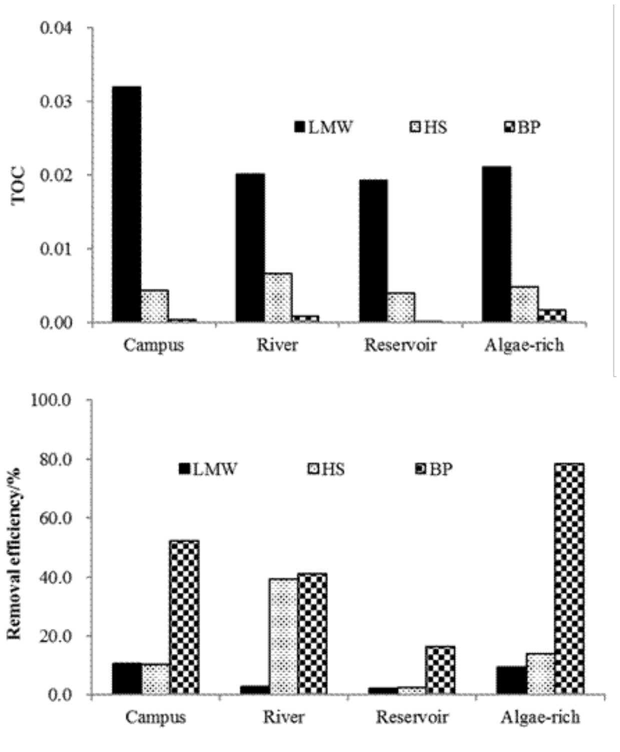

Fig. 7. Fractions (a) and removal efficiency (b) under UF of different waters determined by LC-OCD with peak-fitting.

In addition to HPSEC, EEM fluorescence was also used to determine the differences of the natural waters (Fig. 8). Generally, peaks at excitation (E_x) <250 nm and emission (E_m) (280–380 nm) were related to simple aromatic proteins (EEM-B); peaks at intermediate wave (250 nm < E_x < 300 nm, 280 < E_x < 380 nm) were assigned to soluble microbial-by-product-like materials (EEM-T); peaks located at wave lengths of 200 nm < E_x < 250 nm and E_x > 380 nm represented fulvic-acid-like substances (EEM-A); peaks at longer wave lengths (280 nm < E_x < 350 nm, E_x > 380 nm) corresponded to humic-acid-like organics (EEM-C) [30]. EEM-A and EEM-C were attributed to humic-like substances while EEM-T and EEM-B were associated with protein-like substances [31]. As shown in Fig. 8, the dissolved organic matter in different water sources exhibited

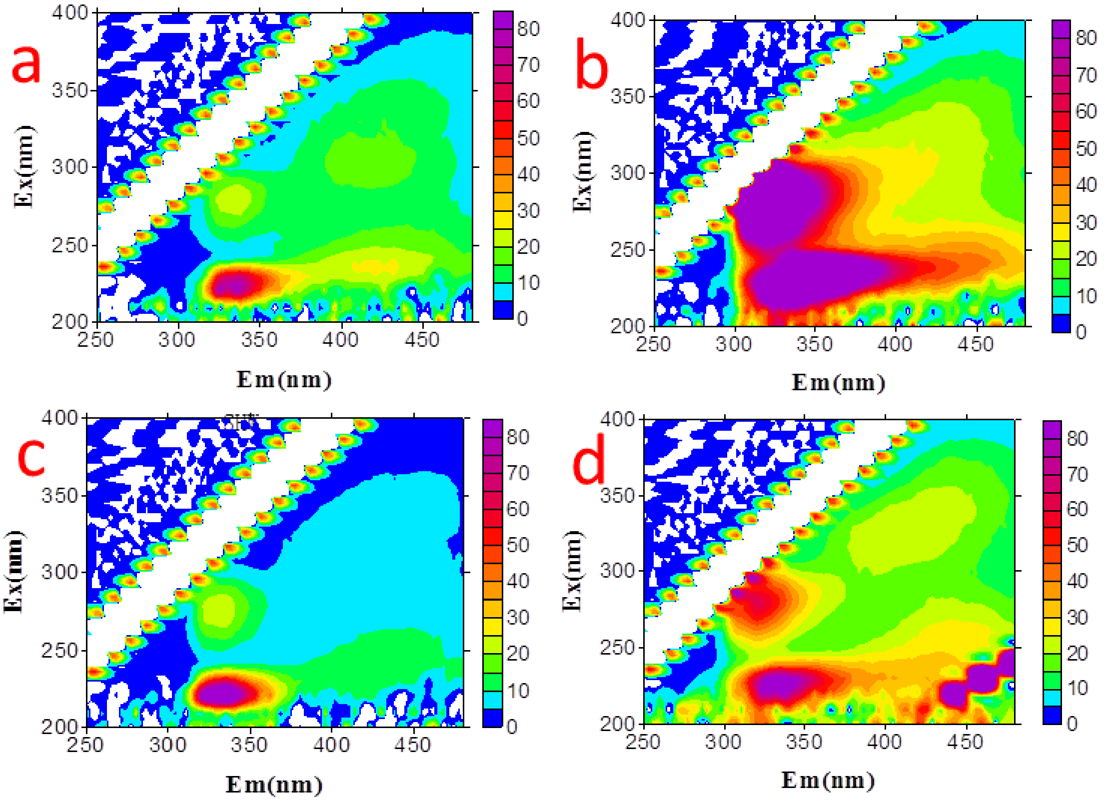

Fig. 8. EEM fluorescence spectra of campus water (a), river water (b), reservoir water(c) and lake water (d).

different fluorescence responses. From the distribution of fluorescence regions, campus water contained two obvious peaks, EEM-B and EEM-A, signifying high contents of aromatic proteins and fulvic-acid-like substances. As for the river, reservoir and lake waters, the peaks of EEM-B and EEM-T exceeded these of EEM-A and EEM-C, suggesting an abundance of protein-like substances in the three samples, possibly due to the metabolism products of organisms and wastewater [27,31]. It is interesting that protein-like substances (EEM-B and EEM-T) in river water were significantly higher than those in campus water, yet river water did not deteriorate the performance of UF compared with campus water. Expectedly, reservoir water contained low intensities of both protein-like and humic-like substances compared with others.

3.3. Correlation of fouling resistances with water constituents

PCA was applied to correlate fouling resistances with water constituents (Turbidity, UV254, TOC, SUVA, LC-LMW, LC-HS, LC-BP, EEM-B, EEM-T, EEM-A and EEM-C). As illustrated in Fig. 9, an inclusion of 83.46% variability was captured in the first two principal components, indicating the effectiveness of this method. Lake water's approaching location to the R_{re} showed its high reversible fouling, while campus and river waters were close to R_{ir}, indicating their severe irreversible fouling. Reservoir water was discriminated by PC A and located at the opposite direction, highlighting the low fouling potential for both reversible and irreversible fouling.

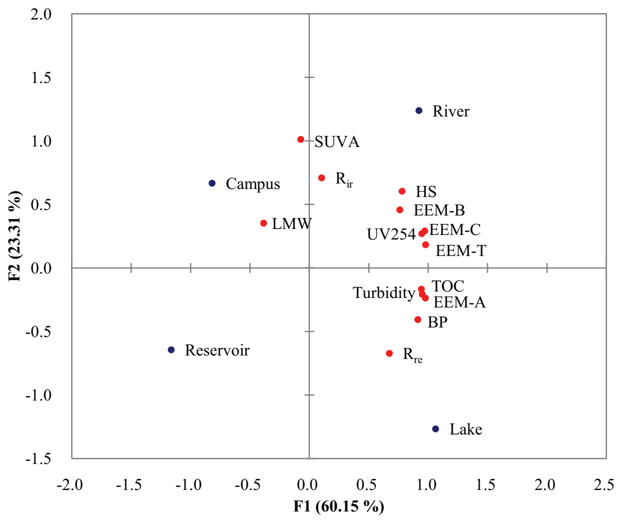

Fig. 9. PCA of fouling potentials with natural water constituents.

The weak correlation of turbidity with R_{re} ($r^2 = 0.396$) and R_{ir} ($r^2 < 0.01$) (Table 2 listed the correlation (r^2) of water qualities with R_{re} and R_{ir}) was observed. TOC had an obvious correlation with R_{re} ($r^2 = 0.710$), suggesting its role in reversible fouling; its weak linear relation with R_{ir} ($r^2 = 0.052$) revealed that the quality rather than quantity in organics was responsible for the irreversible fouling [7]. Previous studies highlighted the contribution of BP to the LMP foul-

Table 2
Correlation (R^2) between fouling resistances and water characteristics

	R_{re}	R_{ir}
Turbidity	0.396	0.071
UV_{254}	0.284	0.219
TOC	0.710	0.052
SUVA	0.550	0.357
LMW	0.025	0.662
HS	0.000	0.092
BP	0.860	0.001
EEM-B	0.000	0.001
EEM-T	0.183	0.003
EEM-C	0.235	0.151
EEM-A	0.722	0.005

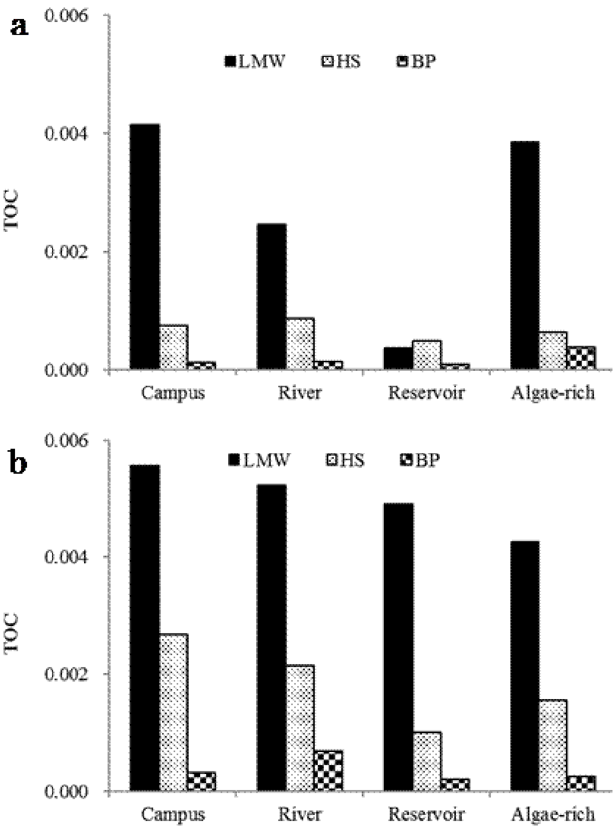

Fig. 10. LC-OCD analysis of the extracted foulants by acid cleaning (a) and base cleaning (b).

ing [32–34].There was remarkable correlation of BP with R_{re} (r^2 = 0.860) but nearly no correlation with R_{ir}, indicating BP was mainly responsible for reversible fouling. That is to say, the accumulation of BP on the membrane surface led to the increase of R_{re}. Although most of the NOM had low and medium MW (Fig. 7), reversible membrane fouling was highly related to the high MW.

In the past years, much work has been done to assess the effect of HS on LPM fouling [19,35–38]. However, little correlation of HS with R_{re} and R_{ir} (r^2 < 0.1) was observed in our study. The parameters of UV254 and EEM-C were largely related to HS. Nevertheless, there were no clear correlation between UV254 and R_{re}, UV254 and R_{ir}, EEM-C and R_{re}, and EEM-C and R_{re}(Fig. 9). It could be implied that HS, specifically humic acids, were not a key factor in the evolution of reversible and irreversible fouling. However, a strong linear relationship of R_{re} with EEM-A (r^2 = 0.722) suggested that fulvic-acid-like substances played an important role in the evolution of reversible fouling. LMW clustered with R_{ir} (r^2 = 0.662),confirming that LMW compounds were highly responsible for their reversible fouling.

In order to further make clear the relationship between the constituents and irreversible fouling, hydraulically irreversible foulants were extracted by chemical agents and shown in Fig. 10. LMW fractions were observed as the main components irrespective of water sources, which was in favor of the point that LMW fractions were contributed to irreversible fouling greatly.

According to the above analysis, BP and fulvic-acid-like substances were identified as the main components of hydraulically reversible foulants, while LMW contributed to hydraulically irreversible fouling. Lake water sharply increased the TMP, followed by campus, river, and reservoir waters (Fig. 2). The increased TMP was due to the foulants' blocking the membrane pores [29], or macromolecules' depositing on the membrane surface [39]. The abundance of BP in lake was a key factor to accelerate the formation of cake layer on membrane surface, with the increase of hydraulically reversible fouling (Fig. 4(d)). The high content of LMW in campus water (Fig. 7) contributed to irreversible fouling greatly compared with river water. This could also explain the phenomenon that TOC concentration of river water was higher than that of campus water, and TMP of UF of river water was slightly lower than that of campus. With respect to reservoir water, limited BP and a small quantity of LMW fractions were responsible for the lower TMP and low fouling resistance compared with the other waters (Figs. 2 and 3).

4. Conclusion

Based on the correlation between fouling resistances and water constituents during UF of four surface waters, the following conclusion could be drawn.

1. The order of different natural waters based on their impact on TMP increase was as follows: lake >> campus > river > reservoir.
2. R_{re} in Lake water was largely responsible for the sharp TMP increase, while R_{ir} in the other three waters contributed to TMP increase greatly.
3. Analysis of water characteristics indicated BP fractions could be mainly retained by UF, while LMW and HS were not removed much.
4. PCA of water characteristics with R_{re} and R_{ir} suggested BP andfulvic-acid-like substances were identified as the main constituents to hydraulically reversible foulants, while LMW fractions contributed to hydraulically irreversible fouling greatly.

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