

Fouling control mechanism of SBAC-UF: role of organics and particles

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ABSTRACT

In a laboratory-scale experimental setup, slow biological activated carbon (SBAC) filtration was conducted to control membrane fouling during ultra filtration (UF) of artificial raw water, and the underlying mechanism of fouling control was analysed by various means. The trans-membrane pressure (TMP) during the experimental SBAC-UF was 60.2% lower than that of UF performed without SBAC. The best fit of classic filtration models indicated that SBAC did not change the fouling model, with standard blocking and cake formation as the main fouling mechanisms, but it reduced the extent of the fouling. Organic material was investigated and particle characteristics were determined to analyse their role in fouling control. Protein-like substances were demonstrated to be major components of deposited foulants, and these could be effectively removed by SBAC. Fewer particles were present in SBAC effluent, while their size distribution was narrower. This led to formation of a thinner cake layer on fouled membranes compared to membranes fouled with untreated raw water. In summary, SBAC filtration can control membrane fouling by specific removal of protein-like substances and favourably affect the quantity and size distribution of remaining particles.

Keywords: Membrane fouling; Slow biological activated carbon; Organics; Particles

1. Introduction

Ultra filtration (UF), a low-pressure membrane filtration process, is widely applied to purify surface water and groundwater for preparation of drinking water; the treatment is suitable to reduce turbidity by removal of particles, organics, and microorganisms including pathogens [1,2]. However, the application of UF in drinking water treatment is considerably constrained by membrane fouling, which causes a decrease in membrane permeability due to the accumulation of contaminants on the membrane surface or within its pores [3]. Identification and quantification of the major foulants that are responsible for membrane fouling is thus of crucial importance. Previous studies have suggested that organic particles are important foulants. Various strategies have been employed to control membrane fouling by changing the characteristics of organics and other particles, including ion exchange, peroxidation, media filtration, and integrated processes [4,5].

Biologically activated carbon filtration is widely used for the removal of ammonia nitrogen in micro-polluted water, as this technique combines the benefits of simple operation and low costs [6–8]. In addition, biologically activated carbon filtration was shown to be effective to reduce membrane fouling by particles and biodegradable materials during subsequent UF [9]. During this process, the filtration rate was a significant parameter that influenced the removal of contaminants and the effectiveness of the operating cycle. Fast-cycle biofiltration processes usually work at a filtration rate of 7~10 m/h, resulting in a relatively short contact time but also in rapid blocking. Slower biofiltration is effective

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to reduce foulants in secondary effluents and improves the performance of the membrane. Zheng et al. [10] performed slow sand filtration as a pretreatment of UF and found that this integrated process was an improvement compared to direct secondary effluent filtration. A study of bank filtration applying filtration rates of 0.25–0.5 m/h showed that this slow biofiltration process could decrease the concentration of biopolymers and reduce the trans-membrane pressure (TMP) of the subsequent UF process [11]. To the best of our knowledge, the fouling control mechanism of slow biological activated carbon (SBAC) filtration on UF has not yet been investigated, which was the objective of the work presented here.

We investigated the fouling mechanism of membranes during UF treatment of artificially polluted raw water, and its control by prior SBAC treatment. Analyses of organic compounds and particles were used to assess the fouling control mechanism. Moreover, scanning electron microscopy and functional group detection of the membranes fouled with and without SBAC were used to compare the differences of membrane fouling.

2. Materials and methods

2.1. Raw water

The artificial raw water was prepared similarly to that used by Tian et al. [12]. For this, domestic sewage was added to local (Beijing, China) tap water in a volumetric ratio of 3:50 to simulate a surface water supply slightly polluted by sewage discharge. Furthermore, 2 mg/L of humic acid (Shanghai, China) was added to simulate presence of organic matter. Before use, the artificial raw water was stabilized at room temperature for 24 h.

2.2. UF experimental setup

A schematic of the laboratory-scale device used for biofiltration and UF is shown in Fig. 1. The filter consisted of a 60 mm inner-diameter Plexiglas pipe, with a height of 1.0 m. Coal-based granular active carbon (Ruineng, China) was used in the filter at a bed depth of 0.5 m. The membrane consisted of a hollow-fibre polyvinyl chloride membrane module (Litree, China) with a surface area of 0.017 m^2 , a nominal pore size of 0.01 µm, an internal diameter of 0.8 mm, an outer diameter of 1.3 mm, and a length of 25 cm. Prior to use, the membrane was immersed in deionized (DI) water for at least 24 h at room temperature.

For SBAC filtration, raw water was fed through a peristaltic pump. Continuous aeration was provided at the top of the biologically activated carbon to provide dissolved oxygen to the influent. The subsequent effluent was then fed into a constant-level tank to manipulate the water level for the subsequent unit. The membrane module was submerged in the cylinder, and continuous aeration was provided at the bottom of the cylinder to clean the membrane. The effluent was collected directly from the membrane module by a peristaltic pump. A manometer, connected to a programmable controller, was installed between the membrane module and the peristaltic pump to monitor the TMP periodically. Before the experiment, the SBAC filtration unit fed with raw water had been operated stably for four months, leading to the assumption that the biomass was stable in the filter.

2.3. Operating conditions

The UF process was conducted in a dead-end filtration mode at a constant flux of 30 $L/(m^2 \cdot h)$, corresponding to a



Fig. 1. Schematic diagram of UF system (1 – peristaltic pump; 2 – biological activated carbon layer; 3 – constant level water tank; 4 – membrane pool; 5 – air blower; 6 – air flowmeter).

hydraulic retention time of 0.5 h. No sludge was discharged during the experiment. For SBAC, the filtration rate was set at 1.0 m/h, and the empty bed contact time was 0.5 h. The filter was backwashed once a week. The ratio of air to influent water was kept constant at 20:1 (v:v) during UF, whereas the ratio was approximately 5:1 for SBAC.

2.4. Analytical methods

2.4.1. UV absorbance, dissolved organic carbon, ammonia-N, and nitrite-N

UV absorbance at 254 nm (UV₂₅₄) was evaluated using a UV/Vis spectrophotometer (T6, Puxi, China). The content of dissolved organic carbon (DOC) was obtained by a vario TOC (Elementar, Germany) using the nonpurgeable organic carbon method. Samples for UV₂₅₄ and DOC determination were filtered through a 0.45 µm cellulose acetate membrane before evaluation. Nitrogen content in the form of ammonia and nitrite was determined by Nascar reagent spectrophotometry and N-(1-naphthyl)-ethylenediamine dihydrochloride spectrophotometry respectively [13].

2.4.2. Particle size distribution

The particle size distribution of nanometer to micrometer-range particles was determined. Particles with a size range of 0.6 nm to 7 μ m at 25°C were measured with a particle analyzer (DelsaNano S, Beckman Coulter, USA) based on dynamic light scattering analysis. The analysis was carried out in triplicate, and representative results are presented. The micrometer particles were counted with an online particle counter (PCX 2200, HACH, USA). The counter recorded particles sized between 2 μ m and 750 μ m, and 8 detection ranges were set: 2–3 μ m, 3–4 μ m, 4–6 μ m, 6–8 μ m, 8–11 μ m, 11–14 μ m, 14–17 μ m, and 17–20 μ m, to investigate the distribution within these size ranges.

2.4.3. Excitation-emission matrix (EEM) fluorescence spectroscopy

EEM fluorescence measurements were conducted using a spectrofluorometer (F-4500, Hitachi, Japan) equipped with a 150 W Xenon lamp at 24°C. A 1-cm quartz cuvette with four optical windows was used. Emission scans were performed from 220 to 550 nm at 5 nm steps, with excitation wavelengths from 220 to 450 nm at 5 nm intervals. The scanning speed was maintained at 1200 nm/ min, and the slit widths for excitation and emission were both 5 nm. The fluorescence spectrum of a Milli-Q® water blank was subtracted from all the spectra to eliminate water Raman scattering and reduce background noise from other sources. According to Coble [14] and Baker [15], four typical fluorescence peaks, denoted as A, C, T₁, and T_{γ} are commonly observed in natural water and sewage. Peaks A and C, which usually appear at 320-350 nm and 230–260/400–500 nm excitation and emission wavelengths respectively, were related to humic-like substances derived from degraded plant material. Peaks T₁ and T₂ were associated with protein-like substances, which normally occur at 225-280 nm and 310-340 nm excitation and emission wavelengths respectively.

2.4.4. Attenuated total reflection fourier transform infrared (ATR-FTIR) spectroscopy

ATR-FTIR spectroscopy was carried out with a Nicolet 6700 (Thermo Fisher, USA) to characterize major functional groups and deposited foulants on the membrane surface. All membranes, unfouled and fouled, were gently flushed with DI water, followed by drying for 24 h in a dryer at room temperature. The membranes were scanned from 525–4000 cm⁻¹ at a resolution of 4 cm⁻¹

2.4.5. Scanning electron microscopy (SEM)

A piece of membrane was cut from the membrane module and flushed gently with DI water to remove foulants not firmly attached. It was then dried for 24 h in a dryer at room temperature before it was investigated with a scanning electron microscope (HITACHI S-4300, Japan), using coarse vacuum with an accelerating voltage of 15 kV.

2.5. Modelling of membrane fouling process

To investigate changes in the fouling mechanism after the SBAC process, an unstirred filtration test was employed, with the details of the process described elsewhere [16,17]. The four classic filtration models of complete blocking, standard blocking, intermediate blocking, and cake filtration were used to identify the underlying fouling mechanism. The filtration models' assumptions were as follows. For complete blocking, it was assumed that the particles completely blocked the entrance of the pores with a single layer barring water from passing through the blocked pore. For standard blocking it was assumed that the particles accumulated on the walls of the straight cylindrical pores, resulting in a decrease in pore volume proportional to the permeate volume. Intermediate blocking resembled complete blocking but here it was assumed that some particles were deposited on top of the blocked particles. For cake filtration particles were assumed to be assembled uniformly on the membrane surface and the formed layer would be permeable.

The fouling evolution of the raw water and the SBAC effluent were fitted to four filtration models according to equations with *R*-squared (R^2) values, with higher R^2 values of filtration models indicating a better fit of the model (Table 1).

Table 1

Equation of different fouling mechanism for dead-end filtration

Models	Equations
Complete blocking	$J_0 - J = AV$
Standard blocking	$1/t + B = J_0/V$
Intermediate blocking	$\ln J_0 - \ln J = CV$
Cake filtration	$(1/J) - (1/J_0) = DV$

Note: V, filtrated volume; t, filtration time; A, B, C and D are constants, respectively.

3. Results and discussion

3.1. Effect of SBAC on fouling control

Two filtration systems were operated at a constant permeate flux of 30 L/(m^2 h) without chemical cleaning. TMP development of the raw water and of the SBAC effluent are shown in Fig. 2. Backwashing was conducted twice, with specific volumes of 700 L/ m^2 and 1400 L/ m^2 , respectively. At the beginning of the experiment TMP was around 9.5 kPa. At the end of the experiment, the final TMP of UF without SBAC was 89.8 kPa, whereas that of SBAC-UF was 41.4 kPa. This indicated that SBAC filtering prior to UF could significantly reduce membrane fouling. Without SBAC, fouling, resulted in a 60% reduction of TMP. Further analysis was conducted to identify the difference of these two processes.

3.2. Modelling of SBAC-UF process

To investigate the fouling mechanism of UF membranes with and without SBAC, four classic filtration models were applied. The regression results from this modelling are shown in Fig. 3. The R^2 values of the raw water treated by UF only were 0.827, 0.999, 0.951, and 0.994 for models describing complete blocking, standard blocking, intermediate blocking, and cake filtration, respectively. With a near-to-perfect fit, this indicates that the mechanism of flux decline of UF was primarily caused by standard blocking, with cake formation as second best, allowing for the possibility that intermediate blocking was involved to some extent as well. Complete blocking was considered to be less relevant. Moreover, the R² values of standard blocking and cake filtration for combined SBAC-UF treatment were the same as those for UF (Fig. 3), which indicates that the fouling mechanisms of the two processes were identical. In other words, SBAC did not change the main mechanism responsible for fouling, although it partially mitigated the extent of the fouling. The results were in accordance with reports by Taniguchi et al. [18], who indicated that prefiltra-



Fig. 2. TMP changes during UF and SBAC-UF treatment as a function of the specific water volume.

tion could reduce the rate of membrane resistance during UF, but it did not change the fouling mode. These authors found that standard blocking was mostly due to particles that were smaller than the membrane pores, and cake filtration was attributed to larger particles that deposited on the membrane surface. In other words, although foulants were partially removed by SBAC, a residual remained present that was responsible for a decreased degree of fouling, via conserved mechanisms.

3.3. Organics and particle removal of SBAC

3.3.1. Organics removal

Presence of dissolved organic matter (DOM), which can be quantified by DOC and UV₂₅₄, is a major problem in UF processes [19]. The removal of DOC and UV₂₅₄ from raw water in the experimental setup is summarized in Table 2. The raw water had an average DOC concentration of 2.46 ± 0.17 mg/L and a UV₂₅₄ absorbance of 0.055 ± 0.004 cm⁻¹. The DOC concentration and UV₂₅₄ absorbance of SBAC effluent were reduced to 1.51 ± 0.12 mg/L and 0.024 ± 0.002 cm⁻¹, representing removal rates of 38.6% and 56.3%, respectively. Thus, much fewer organics were present in the effluent of SBAC compared to untreated raw water.

Three-dimensional EEM fluorescence spectroscopy has been successfully utilized to identify the chemical composition of DOM [20,21]. The EEM fluorescence spectra of different samples are presented in Fig. 4. A reduction in intensity of the fluorescence peaks of raw and treated water indicates a reduction in organics content. Identification of the fluorescence spectral peaks is summarized in Table 3. When the raw water was filtered using UF, the intensity of peaks T₁ and T₂, indicative of proteins or protein-like substances being present, decreased with 78.6% and 65.7% compared to raw water, respectively. However, the peak intensity of humic-like substances (peaks A and C) did not decrease as dramatically. This indicates that protein-like substances were more easily removed by the UF membrane than humiclike substances were. This can mostly be attributed to the fact that the humic-like substances in the raw water used here were mostly of low molecular weight, whereas the protein-like substances were most likely hydrophilic, high molecular weight molecules with particle sizes larger than the membrane pore size [22]. Qu et al. [23] observed similar results, where protein-like substances from the extracellular organic matter (EOM) of Microcystis aeruginosa concentrated in the high molecular weight portion; however, the intensities of the humic-like substances were still quite strong even though the EOM was filtered through a 10 kDa membrane.

When the raw water was filtrated by SBAC, the intensity of both humic- and protein-like peaks reduced strongly: peak T_1 reduced by 90.1% and T_2 by 77.2% compared to raw water, and the reduction in peaks A and C was 69.6% and 70.1%, respectively. Thus, SBAC treatment removed both humic- and protein-like substances from the raw water. Here, the reduction of protein-like substances can be mostly attributed to biological degradation during SBAC [24]. Protein-like substances are important membrane foulants, as they can form a dense cake layer and block the membrane pore. For this reason, their removal is an effective measure to control membrane fouling.



Fig. 3. Fitting the flux decline of raw water (1) and SBAC effluent (2) to the four fouling models: complete blocking (a), standard blocking (b), intermediate blocking (c), and cake filtration (d)

Table 2	
Pollutants removal efficiencies of SBAC filtration	

Water quality parameters	Raw water	SABC effluent	SBAC-UF effluent	Paralled UF effluent	
Turbidity (NTU)	1.78 ± 0.29	0.72 ± 0.10	0.10 ± 0.01	0.10 ± 0.01	
UV254 (cm ⁻¹)	0.055 ± 0.004	0.024 ± 0.002	0.023 ± 0.002	0.048 ± 0.003	
DOC (mg/L)	2.46 ± 0.17	1.51 ± 0.12	1.43 ± 0.07	2.11 ± 0.15	
SUVA (L/mg·m)	2.23 ± 0.02	1.58 ± 0.01	1.60 ± 0.02	2.27 ± 0.02	
NH ₃ -N (mg/L)	1.64 ± 0.17	0.78 ± 0.14	0.75 ± 0.08	1.60 ± 0.15	
$NO_2^{-}-N (mg/L)$	0.23 ± 0.06	0.12 ± 0.07	0.13 ± 0.05	0.21 ± 0.04	

Results are shown as average values with standard deviation from 6 measurements.



Fig. 4. EEM spectra of raw water (a), SBAC effluent (b), SBAC-UF effluent (c) and paralled UF effluent (d).

Table 3	
Identification	of fluorescence spectral peaks

	Peak A		Peak C		Peak T1		Peak T2	
	Ex/Em	Int.	Ex/Em	Int.	Ex/Em	Int.	Ex/Em	Int.
Raw water	265/430	103.3	330/415	60.72	280/335	239.8	230/335	124.7
UF effluent	265/430	106.1	330/410	32.13	280/335	51.19	235/340	42.65
SBAC effluent	255/420	31.33	330/410	17.59	275/335	23.78	230/335	28.39
SBAC-UF effluent	260/425	6.70	330/405	3.91	275/335	6.97	235/335	2.10

3.3.2. Particles removal

Particles are critical membrane foulants that can cause cake layer formation on the membrane surface. The particle size, their number and distribution can all affect the flux decline and thus influence the relative resistance to membrane fouling. The removal efficiency of matter responsible for turbidity is a measure for fouling control. As shown in Table 2, SBAC treatment reduced the turbidity from 1.78 ± 0.29 NTU to 0.72 ± 0.10 NTU, corresponding to a removal efficiency of 59.5%. Additionally, particles over 2 µm in size were quantified, with results shown in Fig. 5. The number of these particles was 740 ± 46 per milliliter of raw water, which was reduced to 94 ± 3 following SBAC treatment. These particles were removed by the subsequent UF process, leading to membrane fouling. The number of particles in the SBAC effluent was 12% of that of raw water, however, membrane fouling of SBAC effluent was 39.8% compared to membrane fouling caused by raw water. Therefore, these particles were significantly removed by SBAC filtration, which was able to alleviate the fouling of the subsequent UF process.

The nanometer-size distribution of particles present in raw water and SBAC effluent were also determined. As depicted in Fig. 6, four main peaks occurred in the raw water, at 0.72 nm, 3.6 nm, 82.1 nm, and 1401.1 nm. After SBAC treatment, the effluent produced only one strong peak around 308.6 nm. The change in particle size distribution was probably attributed to the sieving adhesive action of the SBAC and to biodegradation by microorganisms. Large particles were removed by the sieving function of the SBAC, while only particles with sizes smaller than the pores of the filter could pass. The smallest particles were mostly macromolecules, and these could attach to biofilms or be



Fig. 5. Micro-size particles distribution of raw water and SBAC effluent.



Fig. 6. Distribution of nanosized particles in raw water and SBAC effluent.

absorbed by the activated carbon. It should be noted that particle size changes lead to a change in cake permeability, thereby affecting membrane fouling [25]. In summary, there were fewer particles in SBAC effluent but their size distribution was narrower compared to untreated raw water.

3.4. Cake layer analysis

Cake filtration is thought to be the predominant fouling mechanism during the UF process. The properties of a formed cake layer, such as its components and thickness, greatly influence membrane fouling. Therefore, investigations of the cake layer were considered helpful to explain the fouling mechanism of SBAC-UF, for which we used EEM fluorescence spectroscopy, ATR-FTIR spectroscopy and, SEM.

3.4.1. EEM

The results of EEM fluorescence spectroscopy of the foulants deposited on the membrane surface are shown in



Fig. 7. EEM spectra of foulants on the surface of SBAC-UF (a) and UF (b) membranes.

Fig. 7. Protein-like substances (peaks T_1 and T_2) were the major primary component of deposited foulants on the surfaces of the two membranes, with few humic-like substances being present. Therefore, protein-like substances rather than humic-like substances contributed to cake formation and fouling during these two UF processes. This is in accordance with the experiments by Hong et al. [26] and Drews et al. [27], who reported that proteins could induce serious membrane fouling. Moreover, by comparing the EEM fluorescence spectra from the two membranes, it was evident that the intensity of all peaks was weaker on the surface of the SBAC-UF membrane compared to the UF membrane. This indicates that there were fewer protein-like substances on the SBAC-UF membrane surface, which would explain how fouling is controlled by the SBAC process.

3.4.2. ATR-FTIR spectroscopy

ATR-FTIR spectroscopy of unused and fouled membranes was conducted in triplicate, and only representative results are represented in Fig. 8. A comparison of absorption peaks among the unused, UF-treated, and SBAC-UF treated membranes identified stronger spectral



Fig. 8. ATR-FTIR spectra of an unused membrane, and following UF and SBAC-UF treatment.

bands at wavelengths 1040 and 1384 cm⁻¹ for the fouled membranes. This would indicate the presence of polysaccharides on the membrane, based on the C-O-C vibrations that are responsible for a peak at this wavelength [28,29]. Noticeably, a broad region of absorption at 3300 cm⁻¹ that resulted from the stretching of the O-H bonds [30] and two peaks (at 1638 and 1541 cm⁻¹) attributed to protein-like structures [31] only appeared in the spectra of the fouled membranes. These results clearly show that the identified major foulants on both fouled membranes were protein-and polysaccharide-like substances. Moreover, a stronger spectral intensity was obtained for the parallel UF membrane, implying a larger amount of deposited foulants on this membrane.

3.4.3 SEM imaging

SEM images were taken to determine the morphology of the unused and of the fouled membrane surfaces (Fig. 9). The unused membrane exhibited a flat surface with no visible deposits and a uniform marking of the pores. There was a large amount of nonhomogeneously deposited foulants on the surface of the membranes that had been directly fouled with raw water. This resulted in a rough membrane surface, while pores could no longer be observed. However, when the raw water was first filtered by SBAC, the foulants deposited on the membrane surface were significantly reduced, resulting in a visible improvement for the SBAC-UF membrane. A thin cake layer was deposited on the SBAC-UF membrane surface, that still allowed visualizing pores in the supporting layer. In other words, there were much fewer deposited foulants on the SBAC-UF membrane surface than there were on the parallel UF membrane surface, indicating a greatly reduced membrane fouling as a result of the SBAC treatment.

In summary, protein- and polysaccharide-like substances could be largely removed from raw water by SBAC, reducing the amount of major foulants accumulating on the membrane surface. The amount of particles was reduced by 88.0% in the SBAC effluent compared to raw







Fig. 9. SEM images of the unused membrane (a) and following SBAC-UF (b) and UF (c) treatment.

water, and the remaining particles displayed a much narrower size distribution, forming a thinner cake layer onto the membrane. Therefore, the fouling control of SBAC was attributed to the removal of protein-like substances and particles.

4. Conclusion

SBAC-UF was conducted to control membrane fouling, and the fouling mechanism was analysed through determination of removal of organics and particles. Based on the results, the following conclusions could be drawn:

- TMP of the SBAC-UF process was 60.2% lower than that of the parallel UF process. Results from classic filtration models indicated that, although SBAC did not change the fouling model of the polluted raw water, it partially mitigated the extent of the fouling.
- Protein-like substances, which were demonstrated to be the major foulants for subsequent UF, could effectively be removed by SBAC.
- 3) The particle load in the SBAC effluent was reduced by 88.0% and their changed size distribution resulted in a thinner cake, with favourable outcome for fouling.

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