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Characterization of DOM and disinfection by-products precursors in biological activated carbon filter backwash water from drinking water treatments

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ABSTRACT

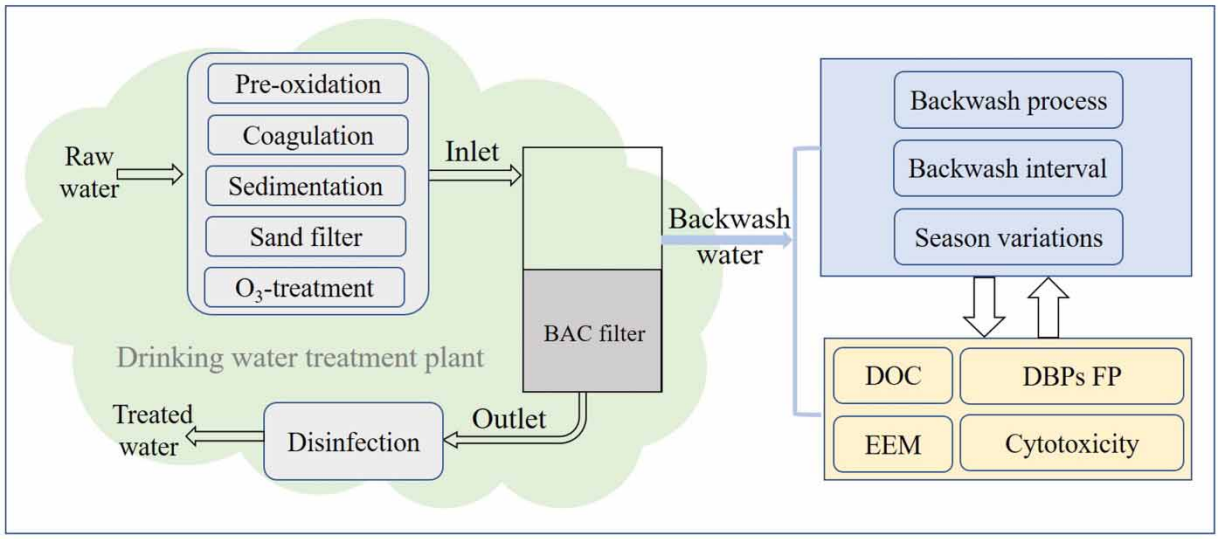
Biological activated carbon filter backwash water (BAC FBW) removes pollutants from BAC filters in drinking water treatment plants. Characterizing the dissolved organic matter (DOM) in BAC FBW is of great significance for the recycling and treatment of backwash water. DOM pollution was monitored over a year using three-dimensional excitation–emission matrix fluorescence spectra. The dissolved organic carbon (DOC) concentration and fluorescence results showed that the DOC concentration reached the lowest value of 1.95 mg/L in the summer. The relative abundances of fluorescence components had a direct relationship with temperature variations. The soluble microbial products were enhanced greatly during cold seasons, while fulvic-acid-like and humic-acid-like compounds remained constant. Disinfection by-products (DBPs) formation potential and cytotoxicity were evaluated. The total haloacetonitriles (HANs), trihalomethanes (THMs), halonitromethanes (HNMs), and haloacetaldehyde (HALs) formation potentials ranged from 35.5 to 47.3 μg/L, 95.1 to 126 μg/L, 4.95 to 8.06 μg/L, and 43.4 to 53.0 μg/L in BAC FBW, respectively. Among all DBPs included in the calculation, the order of contribution to cytotoxicity was HANs > HALs > THMs > HNMs, respectively. Especially, HANs were much higher than the other DBPs, which should be controlled to avoid the biological risk of effluent quality.

Key words: backwash water, biological activated carbon, cytotoxicity, DBPs, fluorescence spectra

HIGHLIGHTS

- DOC concentration of backwash water reached its lowest value in the summer.
- Seasonal variations impacted the composition of DOM due to microbial activity.
- THMs were the main DBPs in the backwash water.
- The order of contribution to cytotoxicity was HANs > HALS > THMs > HNMs in backwash water.

GRAPHICAL ABSTRACT



1. INTRODUCTION

Biological activated carbon (BAC) filtration is used as the main advanced drinking water treatment process in China. Nowadays, drinking water resources are deteriorating with contamination by anthropogenic chemicals. Dissolved organic matter (DOM) is ubiquitous in natural aquatic environments and engineered systems, which is a complex mixture of different compounds such as carbohydrates/polysaccharides, amino acids/peptides/proteins, lipids, humic substances, and anthropogenic organic pollutants (Shi *et al.* 2021; Zhang *et al.* 2022). BAC filtration can efficiently improve the water quality by removing DOM through adsorption and microorganism biodegradation (Ho *et al.* 2007; Thuptimdang *et al.* 2021). It is reported that the turbidity, COD_{Mn}, NH_3-N, UV_{254}, taste, and odor can be further decreased by the BAC filter (Xiang *et al.* 2013).

In our previous research, DOM is an important DBP precursor (Chu *et al.* 2011). Most DBPs have potential carcinogenic, teratogenic, and mutagenic toxicity, so it is important to study those (Richardson *et al.* 2007). BAC filtration also can remove the emerging contaminants, which are recalcitrant to the conventional water treatment, such as pharmaceutical and personal care products (PPCPs), endocrine-disrupting compounds (EDCs), pesticides, and herbicides (Benner *et al.* 2013; Chys *et al.* 2017; Fu *et al.* 2019). The disinfection by-products (DBPs) and their precursors can be controlled by BAC filtration and the performance of the O_3-BAC filter for DBP control was higher than that of the conventional process (Liu *et al.* 2017; Yu *et al.* 2021). BAC filtration is extensively applied to ensure drinking water safety.

The BAC filtration treatment requires backwashing at regular intervals to control the growth of the biofilm. A prolonged usage of BAC filter would reduce water quality due to the activated carbon saturation and the biomass overgrowth. Uncontrolled BAC treatment exposes downstream water consumers to the risk of pathogenic and/or infectious diseases associated with microbial breakthroughs into the distribution system (Simpson 2008; Shen *et al.* 2018). The regular backwash process ensures the normal operation of the BAC filter. As a result, large volumes of water are produced during the backwashing in drinking water treatment plants (DWTPs) (Loret *et al.* 2013). A common practice in DWTPs is recycling the BAC filter backwash water (BAC FBW) to the raw water with or without pretreatment (Tan *et al.* 2017). The process of backwashing removes considerable biodegradable organic matter and some decaying micro-organisms from the BAC filter. Most BAC filter backwash water contains abundant contaminants, including suspended solids, natural organic matter (NOM), bacteria, and inorganic metals (e.g., Fe, Mn, and Al) and the quality of backwash water will vary due to the differences in raw water quality and treatment train design (McCormick *et al.* 2010; Korotta-Gamage & Sathasivan 2017).

While the characteristics of NOM serving as precursors for trihalomethanes (THMs) have been well studied in raw water and sand filter backwash water (Walsh *et al.* 2008; McCormick *et al.* 2010; Lin *et al.* 2017), little is known about the unregulated DBPs in BAC FBW that are recycled to the head of DWTPs. Moreover, there are few studies on the influence of seasonal variation and backwash intervals on NOM in BAC FBW. Therefore, it is necessary to comprehensively monitor

the change in backwash water quality of BAC filters and study the change of organic matter in different seasons and backwash intervals. The backwash interval of BAC filters in water treatment plants in Shanghai is 3–5 days in summer and 5–7 days in winter. There is no regulation for the backwash intervals in spring and autumn, so it is necessary to study the backwash water at different backwash intervals in spring or autumn. Finally, it can provide support for the long-term treatment and recycling of backwash water in DWTPs.

In this study, the BAC FBW in a DWTP was monitored over one year. Fluorescence spectroscopy was used to characterize and analyze the chemical composition of DOM in BAC FBW in different seasons and backwash intervals. The 17 DBPs formation potential (DBPs FP) including THMs, haloacetonitriles (HANs), haloacetamides (HAMs), haloacetaldehydes (HALs), and their cytotoxicity of BAC FBW were also investigated. The research offers insight into the DOM characteristics of the BAC filter backwash water in DWTPs.

2. MATERIALS AND METHODS

2.1. Sample collection in DWTPs

The layout and wastewater recycling process of the drinking water treatment plant are shown in Figure 1. The raw water of the DWTP is taken from the Jinze reservoir in Shanghai, China. There is a pre-oxidation unit followed by coagulation, sedimentation, sand filter, BAC filter, and disinfection in this DWTP. O_3 is used as pre-oxidation agent for pre-oxidation, with the dosage of 0.5 mg/L. The disinfectant used in the water plant is NaClO, the dosage is about 2.2 mg/L and the residual chlorine in the treated water is controlled at around 1.05 mg/L. Table 1 shows the quality characteristics of raw water and treated water.

The backwash parameters of the BAC filter are 4 min of air scouring followed by 8 min water rinse. The backwash water consumption of each BAC filter is about 200 m^3. The water production capacity of the water plant is 60,000 m^3. There are six BAC filters and the backwash water consumption accounts for 0.4%–0.9% of the water production. The backwash water is firstly discharged into the reuse pool and then recycled to the head of the DWTP.

The sampling control interval of backwash water of BAC filters is to take samples every 1 min during backwashing. The samples were collected in 1 L mild-cleaned plastic bags and transported to the laboratory freezer at 4 °C and processed within 24 h. The samples were filtered through a pre-washed 0.45 μm cellulose acetate membrane filter before analysis.

2.2. Chemicals and materials

THMs, HANs, halonitromethanes (HNMs), and HALs mixture standards (Supelco 47904) were supplied from Sigma-Aldrich (St Louis, MO, USA). Methyl tert-butyl ether (MTBE) and anhydrous sodium sulfate were purchased from Aladdin Industrial Inc. (Shanghai, China). All other chemicals with analytical grade were obtained from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). Ultrapure water was produced using the Millipore Milli-Q gradient water purification system (Milli-Q, Merck Millipore, USA).

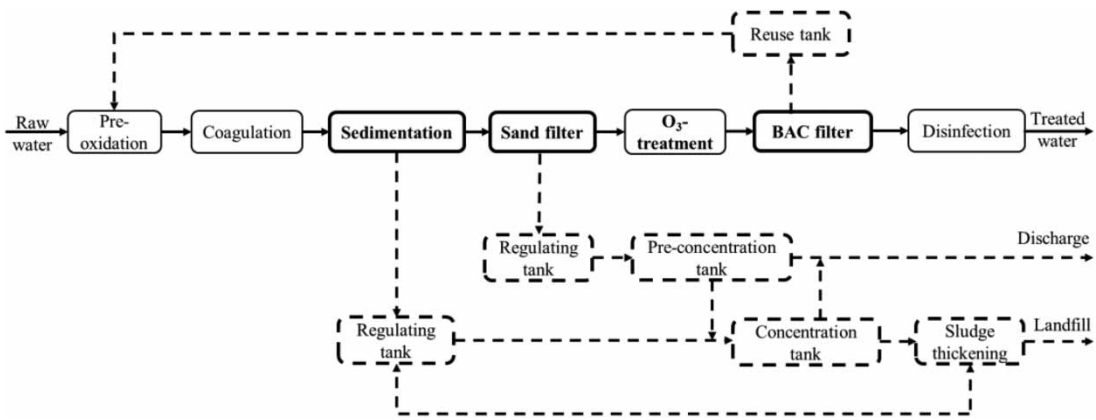


Figure 1 | The layout and wastewater recycling process of the DWTP.

Table 1 | Raw water and treated water characteristics

	pH	Turbidity (NTU)	COD_{Mn} (mg/L-O_2)	DOC (mg/L-C)	NH_3-N (mg/L-N)	NO_2^- (mg/L-N)	Fe (mg/L)	DO (mg/L-O_2)
Raw water	7.82–7.95	12.09–34.38	3.50–3.98	3.51–3.88	0.03–0.33	0.006–0.04	0.49–1.20	3.37–9.36
Treated water	7.05–7.45	0.06–0.08	1.4–1.9	2.12–2.45	0.32–0.43	<0.001	<0.05	/

2.3. Dissolved organic carbon (DOC) and fluorescence spectral analysis

Samples filtered through 0.45 μm cellulose acetate membrane filter were stored at 4 °C until further analysis for dissolved organic carbon (DOC) and fluorescence excitation–emission matrix (EEM) spectra. The DOC concentration was measured with a TOC/TN analyzer (TOC-VCPH, Shimazu, Japan). Three independent replications were carried out and the average value was presented.

EEM spectra were measured using a fluorescence spectrophotometer (F-2710, Hitachi, Japan). The excitation wavelength (λ_{ex}) was varied from 220 to 450 nm, and the emission (λ_{em}) was varied from 220 to 550 nm, each in 5 nm increments. The scan speed was set to 12,000 nm/min.

Using an analytical approach termed fluorescence regional integration (FRI), the EEM spectra were divided into five regions representing specific organic matter components (Chen *et al.* 2003): region I (representing aromatic protein-like compounds such as tyrosine); region II (representing aromatic protein-like compounds such as tryptophan); region III (representing fulvic acid-like compounds); region IV (representing soluble microbial products including tryptophan-like and biologically-related tyrosine-like compounds); and region V (representing humic acid-like compounds). The integral volume (Φ_i) of a specific fluorescent region was calculated and normalized to the volume of the full EEM, resulting in an integrated standard volume ($\Phi_{i,n}$) of the specific fluorescent region. Relevant calculations are given by Equations (1)–(4):

$$\Phi_i = \int_{ex}\int_{em} I(\lambda_{ex}\lambda_{em})d\lambda_{ex}d\lambda_{em} \tag{1}$$

$$\Phi_{i,n} = \mathrm{MF}_i\Phi_i \tag{2}$$

$$\Phi_{T,n} = \sum \Phi_{i,n} \tag{3}$$

$$P_{i,n} = \frac{\Phi_{i,n}}{\Phi_{T,n}} \times 100\% \tag{4}$$

where $I(\lambda_{ex}\lambda_{em})$ is the fluorescence intensity (au) at each excitation–emission wavelength pair, $\Phi_{T,n}$ is the total fluorescence area integrated to produce a volume (au·nm^2), MF_i is a multiplication factor equal to the ratio of total integrated area to the ith integrated fluorescence area, and $P_{i,n}$ is the proportion of the ith integrated volume to the total integrated standard volume of the fluorescence area (%).

2.4. DBPs FP analytical methods

Seventeen halogenated DBPs FP were analyzed, including four THMs: chloroform (TCM), bromodichloromethane (BDCM), dibromochloromethane (DBCM), and bromoform (TBM); five HANs: trichloroacetonitrile (TCAN), dichloroacetonitrile (DCAN), bromochloroacetonitrile (BCAN), dibromoacetonitrile (DBAN), and tribromoacetonitrile (TBAN); three HNMs: dichloronitromethane (DCNM), trichloronitromethane (TCNM), and tribromonitromethane (TBNM), and five HALs: dichloroacetaldehyde (DCAL), trichloroacetaldehyde (TCAL), bromodichloroacetaldehyde (BDCAL), dibromochloroacetaldehyde (DBCAL), and tribromoacetaldehyde (TBAL).

The DBP FP test method and the dosage of chlorine addition were performed according to the methods detailed in Krasner *et al.* (2006). Fill a 250 mL triangular flask with a water sample, keep the pH at 7.0 ± 0.2 and add 750 μL sodium hypochlorite solution with 5.2% effective chlorine. Make the content of available chlorine 20 mg/L, place it in a 25 °C water bath to react for three days in a dark place, and control the residual chlorine at 3–5 mg/L at the end of the reaction. Residual chlorine was quenched using excess sodium thiosulfate. Duplicate samples, along with laboratory and field blanks, were extracted for DBP analysis. After that, the water sample was extracted by liquid–liquid extraction and the formation potential of DBP was

determined by gas chromatography with an electron capture detector (GC-ECD). DBP was separated via RTX-5MS column (30 m × 0.25 mm × 0.25 μm). The specific analysis conditions of DBPs are shown in Table 2.

2.5. Calculation of water sample toxicity

The cytotoxicity of DBP was characterized by the ratio of the formation potential of DBP to the LC_{50} value of corresponding Chinese hamster ovary (CHO) cells (Zeng *et al.* 2016). After summing, the cumulative cytotoxicity of various DBPs was obtained to explore the effects of different water samples on cytotoxicity. The LC_{50} value of CHO cells used for calculation was obtained from previous studies (Yang *et al.* 2013; Wagner & Plewa 2017).

3. RESULTS AND DISCUSSION

3.1. DOM variations in different seasons

3.1.1. DOC concentration

DOC was measured to reveal the total concentration of DOM in the BAC FBW. Figure 2 shows the average DOC concentrations in four seasons. DOC concentrations varied from 1.95 to 3.13 mg/L seasonally. The concentration of DOC in raw water is 3.5–3.9 mg/L, in the BAC inlet water it is 3.0–3.5 mg/L, and in the BAC outlet water it drops below 2.5 mg/L. The content of DOC in backwash water is lower than that in raw water, mainly because the BAC filter mainly degrades rather than intercepts DOM, so the enriched DOC content is lower. The concentration of DOM reached its lowest value

Table 2 | GC column and oven temperature programs for DBPs analysis

DBPs	Injector temperature (°C)	Detector temperature (°C)	Oven program
THMs	200	300	30 °C held for 10.5 min 14 °C/min increase to 72 °C held for 1 min 40 °C/min increase to 200 °C held for 2 min
HALs	180	200	34 °C held for 8 min 20 °C/min increase to 74 °C held for 1 min 50 °C/min increase to 200 °C held for 2 min
HANs	180	200	30 °C held for 10 min 7 °C/min increase to 72 °C held for 1 min 40 °C/min increase to 200 °C held for 2 min
HAMs	250	300	40 °C held for 3 min 20 °C/min increase to 110 °C held for 1 min 10 °C/min increase to 220 °C held for 2 min

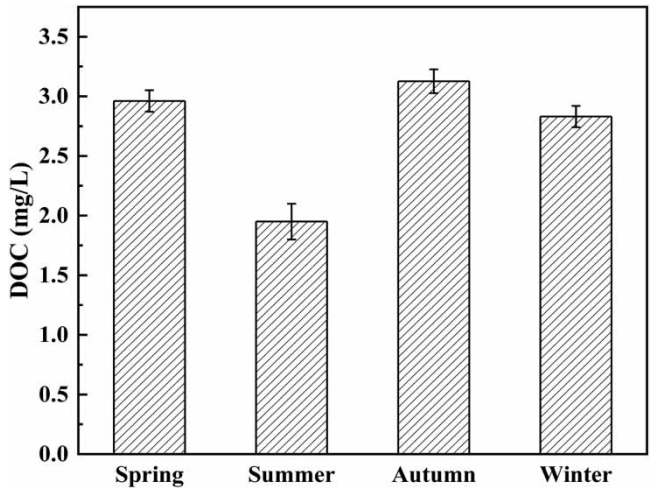


Figure 2 | Seasonal variation of DOC concentrations in BAC FBW.

of 1.95 mg/L in the summer because the microbial activity was intense in the BAC filter. When the operation of BAC FBW is evaluated, the temperature variation should be taken into consideration.

3.1.2. FRI EEM spectra

FRI EEM was used to elucidate the composition of DOM in the BAC FBW. The organic composition and the chemical structure of NOM in BAC filter backwash water were reported to be complex (Feng *et al.* 2020). Characterizing the DOM composition can give an insight into the BAC filter backwash water quality. Backwash water was sampled in different seasons and the influence of seasonal variations on backwash water DOM dynamics was investigated. Figure 3 reveals the FRI EEM spectra of BAC filter backwash water in four seasons. $P_{i,n}$ is the proportion of the ith integrated volume to the total integrated standard volume of the fluorescence area (%).

The results showed that the relative abundances of fluorescence components had a direct relationship with seasonal variations. EEM spectra exhibited high intensities in regions I and II, with corresponding area volumes of 20%–30% and 38%–48% of the sum of all the regional volumes, respectively. These two regions represented aromatic protein-like compounds, which was possibly due to the stimulated microbial activity in the BAC filter (Ly *et al.* 2017). With the change of seasons, the abundance of each region changed significantly. The proportion of regions I and II was the highest in summer, up to 75%, and the lowest was only 52% in winter. Region IV represented the soluble microbial products, including tryptophan-like and biologically related tyrosine-like compounds, which were the lowest in summer and the highest in winter. The dynamic change of fluorescence area was mainly due to the different microbial activities in the BAC filter under the change of temperature consistent with the DOC analysis.

3.2. DOM variations in backwash water

The DOC concentration and DOM composition in the BAC filter backwash water changed with the backwash interval. The backwash interval impacted the pollutant accumulation in the BAC filter bed and the growth of biomass as well. The study on the concentration of DOC in backwash water in different seasons shows that the concentration of DOC in spring and autumn is higher, so the effect of the backwash interval and backwash process in spring was also investigated to analyze the variation of BAC FBW.

3.2.1. Backwash intervals

Figure 4 shows the DOC concentration is highest when the backwash interval is five days. Under different backwash intervals, the proportion of each component of the fluorescence integral had little effect on the change of organic matter composition in BAC FBW.

3.2.2. Backwash process

The first 4 min was the air scour followed by 8 min water rinse during one backwash process. The variation of DOC concentration is presented in Figure 5. At the end of air scouring, the DOC concentration reached its maximum and the discharge of backwash water made the air-scour DOM decrease. With the progress of water rinsing, the DOC concentration in BAC FBW

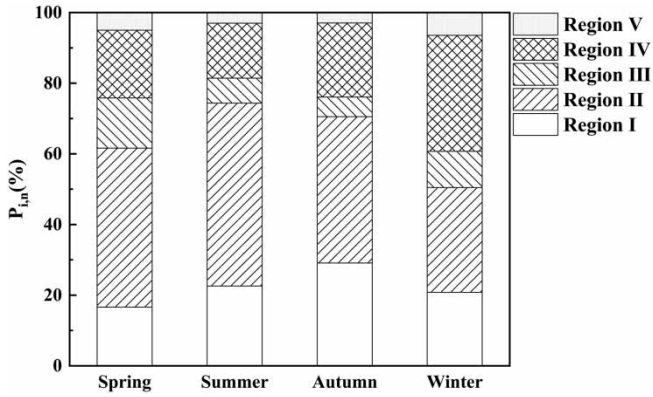


Figure 3 | Seasonal variation of FRI EEM spectra for BAC FBW.

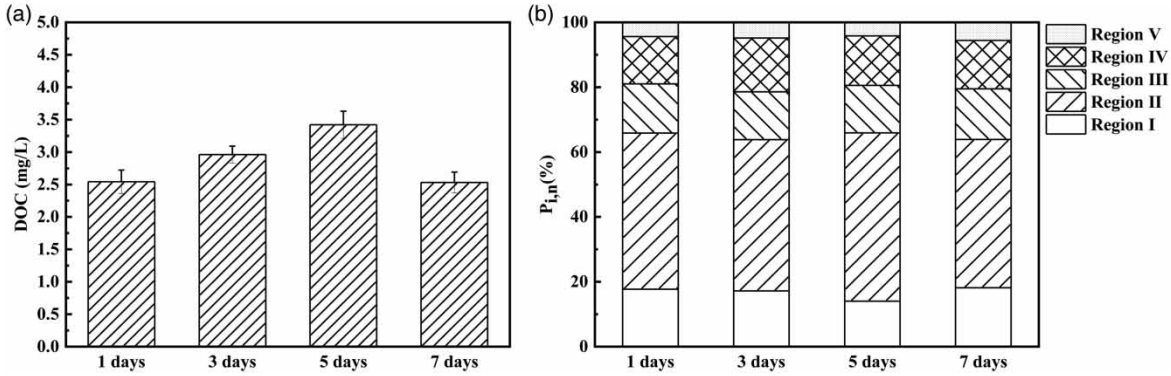


Figure 4 | Variation of DOM with different backwash intervals: (a) DOC concentrations and (b) FRI EEM spectra.

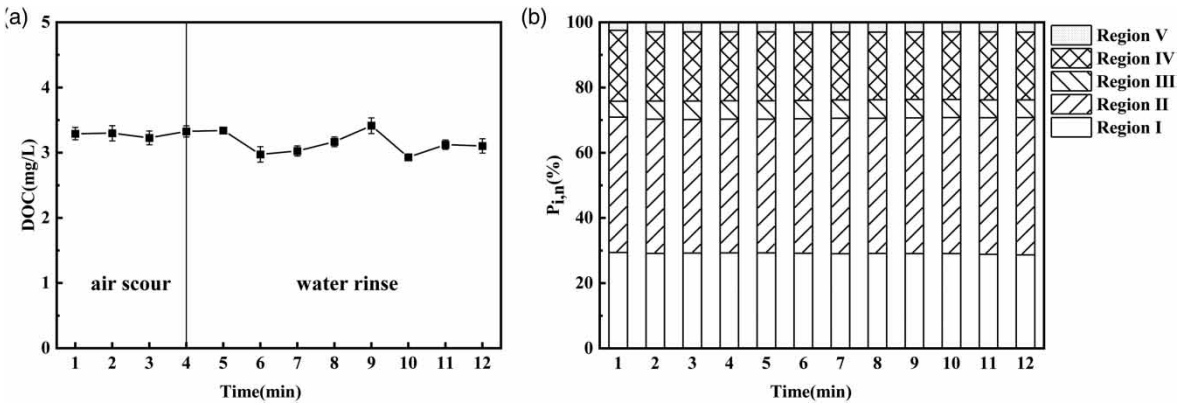


Figure 5 | Variation of DOC concentrations during the backwash process: (a) DOC concentrations and (b) FRI EEM spectra.

increased gradually and then decreased, indicating that the DOM in the filter was washed again and discharged out of the BAC filter during water rinsing. In general, the general change trend of DOM by FRI EEM spectra in the backwash process was not obvious.

3.3. DBP precursors

Four types of DBPs were investigated: THMs, HANs, HNMs, and HALs. Figure 6 shows DBP formation potentials in BAC FBW in different backwash intervals. The formation potential of DBP increased and then decreased with the increase of the backwash interval and reached the highest value of 230 μg/L on the fifth day. With the extension of backwash interval, the growth and reproduction of biofilm increase the generation potential of DBPs (Delatolla *et al.* 2015). THM formation potentials were higher than those of HANs, HNMs, and HALs. Total THM, HAN, HNM, and HAL formation potentials ranged from 95.1 to 126 μg/L, 35.5 to 47.3 μg/L, 4.95 to 8.06 μg/L, and 43.4 to 53.0 μg/L in BAC FBW, respectively. THMs were the main disinfection by-product in the chlorinated backwash water of the BAC filter, while the content of HNM precursors was the lowest (Figure 7).

TCM and BDCM were presented at the highest concentrations, at 65.3 and 29.2 μg/L, respectively, which was consistent with the research results (Hu *et al.* 2021). There were bromines in the backwash water, which was the reason to generate BDCM in the backwash water. Among the five HANs, TBAN and DCAN were the most prevalent, at 27.2 and 8.74 μg/L, respectively. DCNM formed to the greatest extent among the three HNMs, at 5.87 μg/L in chlorinated BAC filter backwash water. Among the five HALs, TCAL was present at the highest concentration at 28.03 μg/L in chlorinated BAC FBW. The backwash interval impacted more greatly on the formation potential of THMs, with an increase from 95.1 to 126 μg/L when the backwash interval was prolonged to five days. The variation was due to the enhancement of NOM with the prolonged backwash interval. The impact of backwash interval variation on the formation potential of the other DBPs was

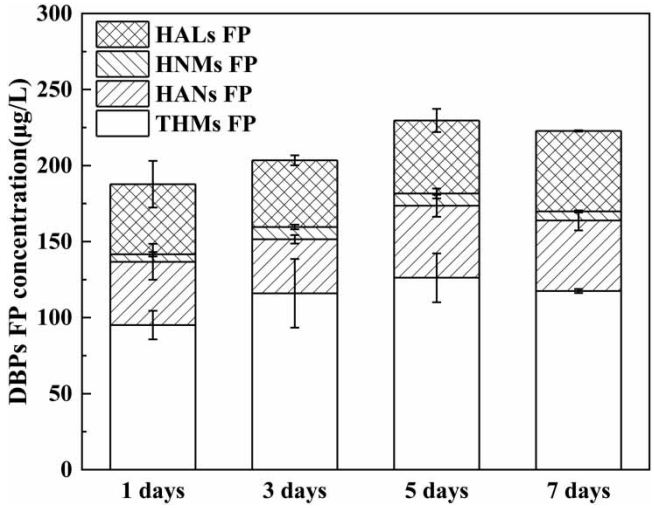


Figure 6 | Precursors of DBP in BAC FBW in different backwash intervals.

not obvious. Some results confirmed the presence of bromide in the raw water of the Jinze reservoir from Tai Lake (Hong *et al.* 2015; Li *et al.* 2018). Moreover, brominated DBPs are produced in BAC FBW after chlorine disinfection, so the presence of bromide in BAC FBW should not be underestimated. Therefore, more attention should be paid to the safety risks of brominated DBPs after backwash water reuse.

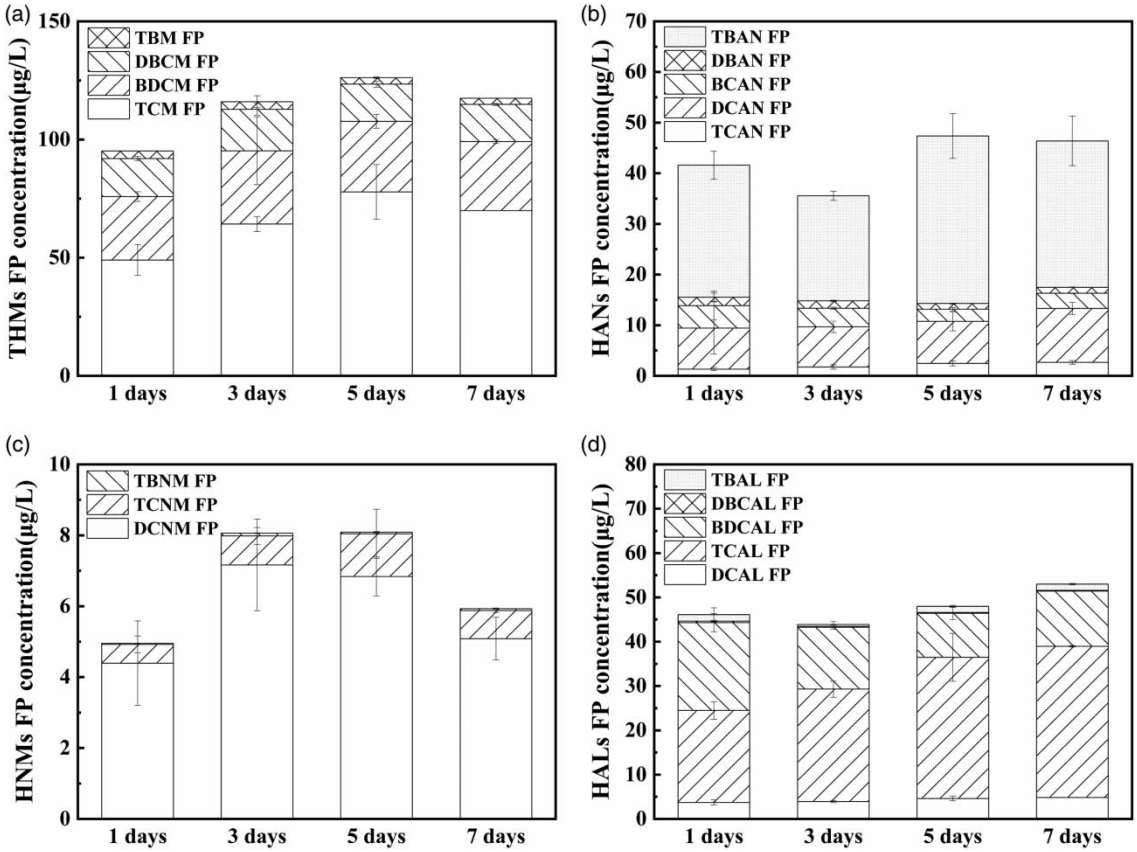


Figure 7 | Formation potential of (a) THMs; (b) HANs; (c) HNMs; and (d) HALs in BAC FBW with different backwash intervals.

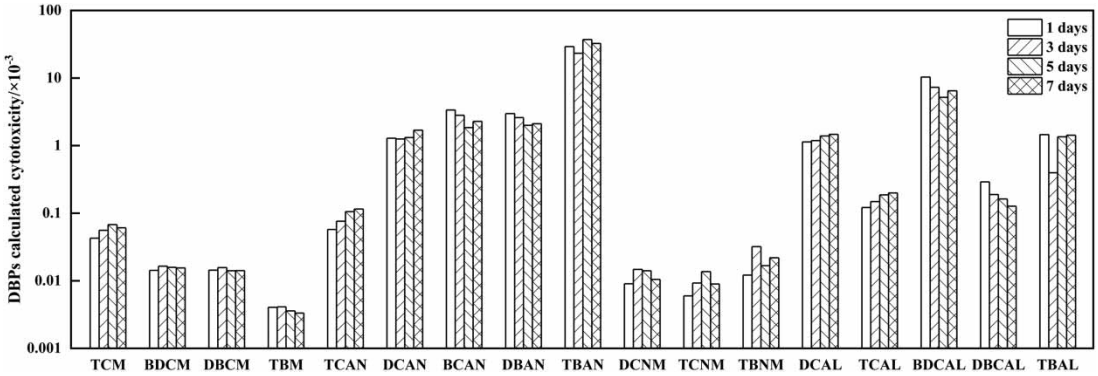


Figure 8 | Cytotoxicity in BAC FBW in different backwash intervals.

3.4. Calculated cytotoxicity of measured DBPs

Cytotoxicity was an important indicator to characterize the biological risk of water, which represented the chronic toxicity of inhibiting cell growth. Among all DBPs calculated in Figure 8, the order of contribution to cytotoxicity is HANs > HALS > THMs > HNMs, respectively. Although THMs had the highest formation potential, HANs and HALs had the highest toxicity to water samples. Especially the cytotoxicity of TBAN was much higher than that of the other DBPs. According to the literature, HANs were detected in drinking water after chlorination and disinfection, which induced organisms to mutate and caused deoxyribonucleic acid (DNA) strand breaks in human lymphocytes and skin tumors (Plewa *et al.* 2004). Their content in recycled BAC FBW should be measured to avoid the biological risk of effluent quality.

4. CONCLUSION

The fluorescence spectroscopy, DBP formation potential, and cytotoxicity of BAC FBW were investigated during different seasons and backwash intervals. The main conclusions of this study are as follows:

(1) The BAC filter backwash water in a DWTP was monitored over one year. The DOC concentration reached its lowest value of 1.95 mg/L in the summer. Aromatic protein-like compounds were found to be the main component of DOM by FRI analysis in all seasons. Seasonal variations in temperature impacted the composition of DOM due to the microbial activity in the BAC filter.

(2) BAC filter backwash intervals changed the DOM quantity in the backwash water. DOC concentration increased with the prolongation of backwash intervals, but decreased over five days. The DBP formation potentials also increased with the prolonged backwash interval. On the premise that the BAC FBW is reused, it is not advisable to use a long backwash interval, so as to ensure the quality of the backwash water of the BAC filter and not cause deterioration of the effluent of the water plant.

(3) THMs were the main DBP in chlorinated BAC FBW. However, the order of contribution to cytotoxicity was HANs > HALS > THMs > HNMs, respectively. In addition to the regulated THMs, the water treatment plants should also pay attention to HANs when considering the reuse of BAC FBW to avoid the biological risk of effluent quality.

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DATA AVAILABILITY STATEMENT

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

CONFLICT OF INTEREST

The authors declare there is no conflict.

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