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Color composition in a water reservoir and DBPs formation following coagulation and chlorination during its conventional water treatment in northeast of China

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ABSTRACT

In this research, the source of reservoir type water supply in northeast of China was determined through Minitan^{TM} ultrafiltration system and three-dimensional excitation emission matrix, providing the relationship between color, molecular weight cut off (MWCO) of natural organic matter (NOM), and disinfection byproducts (DBPs). Removal efficiencies of color, turbidity, and DBPs by conventional water treatment processes were also investigated. The results showed that MWCO of NOMs were mainly between <1 kDa and 5–10 kDa. There were two typical EEM peaks in reservoir water, in which peak A (Ex/Em: 260/450 nm) representing fulvic acid (FA)-like substances (1–5 kDa) and peak C (Ex/Em: 330/450 nm) standing for humic acids (HA)-like substances (MWCO > 5 kDa). HA contributed more to color than FA at high dissolved organic carbon. Macromolecular HA contributed more to haloacetic acids (HAAs) and small molecular FA made more contribution to trihalomethanes (THMs) and HAAs. During conventional water treatment processes, about 30% organic matters (OMs) (MWCO > 5 kDa) could be removed by coagulation–sedimentation process and total removal efficiency was more than 60% (HA mainly) after filtration process. However, only 22% OMs (MWCO < 3 kDa) could be removed limitedly, indicating the left OMs was small molecular.

Keywords: NOM; DBPs; Color; 3D-EEM; MWCO

1. Introduction

Natural organic matter (NOM) is a heterogeneous mixture of complex organic compounds in surface water, consisting of different chemical compositions and molecular sizes (from largely aliphatic to highly colored aromatics, both hydrophobic and hydrophilic compounds), and normally most of NOM are negatively charged [1,2]. Hydrophilic NOM includes more

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aliphatic carbon and nitrogenous compounds (e.g. carbohydrates, proteins, and so on). While hydrophobic NOM contains a higher proportion of aromatic carbon, processing phenolic structures, and conjugated double bonds. The hydrophobic/hydrophilic rate of NOM can influence the effect of the coagulation process [3]. Furthermore, in aquatic NOM, the main component was hydrophobic acids, occupying more than 50% dissolved organic carbon (DOC) in water [2], which could be described as humic substances (HS).

HS can generally be divided into three fractions: humins, humic acids (HAs), and fulvic acids (FAs) [4]. Humins contain the insoluble humic components of aqueous solutions at all pH values. HAs represent soluble components in alkaline and weakly acidic solutions, but they precipitate when pH value is lower than 2.0. FAs are soluble in aqueous solutions at all pH values. HS are toxic by themselves as they can be responsible of blackfoot disease [5], and may cause color and taste problems in water supply. HS could also reduce the effectiveness of water treatment processes which employ membranes or microporous adsorbents [6,7]. Furthermore, during the disinfection process of water treatment, HS present in water supplies are considered to be potentially harmful pollutants for human health, due to halogenation reactions with chlorine. Chlorine is still commonly used as disinfectant because of its low cost, but it contributes into carcinogenic disinfection byproduct (DBPs) formation, for example trihalomethanes (THMs) and haloacetic acids (HAAs). Consequently, it is necessary to know the characteristics of HS and remove them before disinfection with chlorine in conventional water treatment as much as possible. The United States Environmental Protection Agency (USEPA) has identified enhanced coagulation (EnC) as a best available technology (BAT) for reducing DBP precursors (e.g. HS) in conventional water treatment plants. Their removal by EnC depends on a variety of factors, including but not limited to, pH, alkalinity, coagulant type and dosage, and the type and concentration of NOM [8].

HS molecular weight cut off (MWCO) could be determined by high-pressure size exclusion chromatography (HPSEC) [9], gel permeation chromatography (GPC) [10], and ultrafiltration [11]. HS Removal from aqueous solutions has been investigated by a number of researchers [12–16]. Furthermore, the application of ultrafiltration (UF) and other membrane-based techniques have shown great potential for treatment of HS and other organic matters [17]. Nowadays, many previous studies have illustrated that 3D-EEM fluorescence spectroscopy is a powerful tool for investigating the chemical and physical characteristics of dissolved organic matter (DOM) and for interpreting the formation of DBPs and membrane fouling [17]. 3D-EEM fluorescence spectroscopy shows many advantages, such as high sensitivity, good selectivity, easy operation, short measurement time, non-destruction of sample, little or no sample pre-treatment, and inexpensiveness. Meanwhile, many researchers try to remove or reduce the content of DBPs and its precursors by all kinds of methods [18–22].

However, most researches only focus on MWCO of NOM in all kinds of water supplies and removal efficiency of NOM, DBPs, and DBPs precursors by different water treatments. There is little research on the relationship of MWCO of NOM and DBPs. In this research, the source of reservoir type water at cold area in northeast of China is determined through Minitan^{TM} ultrafiltration system and 3D-EMM fluorescence spectroscopy technique, providing the relationship between NOM MWCO of reservoir type water and DBPs formation. Removal efficiencies of color, turbidity, and DBPs by conventional water treatment processes are also investigated at the same time.

2. Materials and methods

2.1. Condition of experiments

The research is carried out in a reservoir type water treatment plant in northeast of China (Wu Chang, Heilongjiang province). Water treatment quality of the plant is $9 \times 10^4\,m^3/d$ and it contains two same water treatment systems. The plant is a representative one of the scale, the characteristics of influent, the treatment processes, and the usage of effluent. The average characteristics of raw water are shown in Table 1. The research is operated from November 2012 to March 2013.

The water treatment processes are the conventional drinking water treatments, including coagulation, sedimentation, filtration, and disinfection. Mechanical mixing tanks have four series and each series has two tanks

Table 1
Average characteristics of raw water

Temperature (°C)	pH	DOC (mg/L)	Turbidity (NTU)	Color	NH_4-N (mg/L)	Alkalinity (mg/L)	Hardness (mg/L)
11.1	6.81	5.05	2.28	32.7	0.26	22.7	33.3

(mixing time: 1 min). Horizontal axis mechanical stirring flocculation tanks have three series (average coagulation time: 8.67 min, $G = 60.47\ S^{-1}$, $GT = 1.068 \times 10^5$). Poly aluminum Chloride (PAC) is used as the coagulant and the dosage is up to 80–120 mg/L (containing 7% aluminum).

Opposite flow tube settlers have four series, and each series has two settlers. The upflow speed of clear water in sedimentation tanks is 1.2 mm/s.

In water purification district, there are four sets, and each set has six cells of shutter filters with double layer filter materials. These filters are arranged in two parallel rows and the filtration flow speed is 7.0 m/h operated in the mode of constant water level filtration.

Liquid chlorine is chosen to disinfect and the dosage is about 1–2 mg/l. The disinfectant was added automatically in the mode of post-chlorination. The dosing point located in the middle of main clean water pipe after filtration and clear water tank. Hydraulic retention time (HRT) of clear water tank after post-chlorination is more than 30 min. By this way, residual chlorine kept at 0.5–0.8 mg/L in finished water and kept higher than 0.05 mg/L in the end of pipe network.

2.2. Analysis methods

2.2.1. MWCO of NOM

MWCO of NOM is carried out using cell filters and dead end batch UF apparatus (Minitan^{TM} UF system). UF experiments are performed with a stirred cell (Amicon, Model 8400), which is composed of a 400 mL methyl methacrylate glass holder. In this study, the sequential filtration is carried out using UF membranes (modified cellulose acetate membrane), with pore sizes corresponding to the nominal molecular weight of 30, 10, 5, 3, and 1 kDa.

2.2.2. 3D-EEM fluorescence spectroscopy

Three-dimensional fluorescence spectrum is measured using a Cary Eclipse fluorescence spectrophotometer (Varian, USA). The excitation–emission matrix (EEM) is generated for each sample by scanning over an excitation wavelength (Ex) between 200 and 450 nm at an increment of 10 nm, and an emission wavelength (Em) between 200 and 800 nm at an increment of 10 nm. The excitation and emission slit widths are set at 10 nm, and a scanning speed of 1,200 nm/min is applied for all of the measurements.

Certain organic matters generate characteristic fluorescence by excitation wave or emission wave, and then 3D-EEM spectra could indicate the particular molecular conformation of organic compounds in water. Fluorescence active compounds in environmental water include enzyme, coenzyme, pigmented and metabolic products, which generate characteristic fluorescence under ultraviolet light. DOC in cold area water includes protein-like, fulvic acid-like, humic acid-like, hydrophilic acids, carboxylic acid, amine acid, and carbohydrates. The 3D-EEM spectra contain a great quantity information of functional group structure and construction of dissolved organic matters (DOMs). The 3D-EEM spectra characteristics of common DOMs in water are shown in Table 2.

2.2.3. Other analysis methods

DOC (prefiltration through 0.45 micro meter) is detected by TOC analyzer (Phoenix 8000, Tekmar-Dohrmann, American).

DBPs (Trichloromethane (TCA), Trihalomethanes (THMs), Dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), HAAs, choral hydrate (CH)) are measured according to U.S. EPA 551 standard methods [23,24]. An automated miniaturized liquid–liquid extraction gas chromatography–mass spectrometry (MLLE-GC–MS) method is developed to detect DBPs [25].

An Agilent 6890 Plus gas chromatograph (GC) coupled to a 5973 mass-selective detector (MSD) (Agilent Technologies, Sanata Clara, CA, USA) is used for all analysis. The GC capillary column is HP-5MS (0.25 mm × 0.25 μm × 30 m). High purity of helium is used as the carrier gas.

Table 2
Characteristics of 3D-EMM fluorescence spectroscopy of DOMs in water

Peak	Ex/nm	Em/nm	Substance
δ	205	370	Aromatic proteins [35,36]
	280	330	[37]
B_1	225–237	305–310	Tyrosine-like [36,38,39]
T_1	225–237	340–350	Tryptophan-like [36]
T_2	275	340	[36]
	230–255	400–460	Ultraviolet FA [40]
	280–310	380–455	Visible FA [40]
D	230	465	FA-like [35]
C	237–260	400–500	HA-like [36]
	300–370	400–500	HA-like [36]
E	245	440	HA-like [35]
B_2	275	310	Tyrosine-like [36]
A	285	340	Protein-like [35]
α	320	415	FA-like [37]
B	355	440	Visible HA [35]

3. Results and discussion

3.1. Characteristics of raw water

Recently, researches on humus characteristics increased more and more, humus characteristics of water supplies in different regions were not all the same [26,27].

Reservoir raw water was classified by Minitan™ UF system, and the concentration of DOC fluctuated between 3.92 and 6.05 mg/L (average 5.2 mg/L), which showed there was enough nutrition in the water supplies [28].

Relationship between DOC and color was researched in Fig. 1. The regression coefficient (R^2) was 0.9786, which showed DOC has a very good correlation to color. So it could be determined that color matters mainly derived from DOC in the influent.

3.1.1. 3D-EEM fluorescence spectroscopy

The source of reservoir water was analyzed through three-dimensional fluorescence spectrometry in Fig. 2. The 3D-EEM fluorescence results of reservoir water samples indicated that NOM component of natural organic matters appeared in 3D-EEM fluorescence. In a certain range of concentration, the fluorescence intensity of humus samples strengthened with increase of concentration [29,30]

There were two main peaks (Ex/Em 240–260/400–450 nm, and Ex/Em 300–330/400–440 nm) in the fluorescence spectrum. Peak A was rich in FA-like substances (Ex/Em: 260/450 nm), and peak C represented HA-like substances (Ex/Em: 330/450 nm) in the fluorescence spectrum. Fig. 2(a) showed that fluorescence intensity of peak C was stronger than that of peak A, which proved that the main component of raw water organic matters was micro molecular FA.

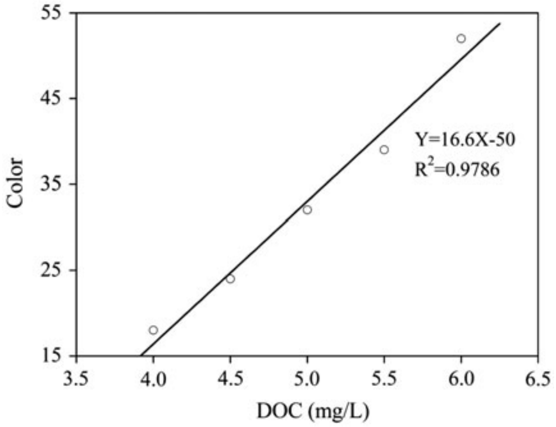


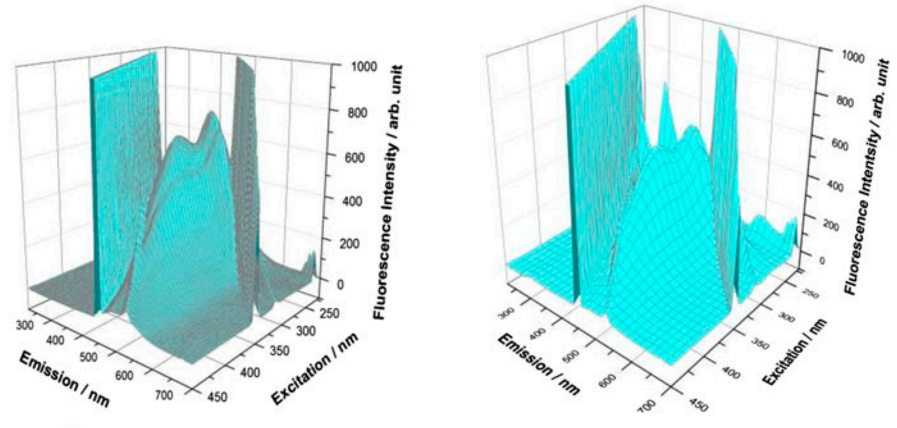
Fig. 1. Relationship between DOC and color.

The 3D-EEM fluorescence results obtained from untreated water samples indicated that fluorescence intensity of peak C decreased from 679.2 to 503.2, and fluorescence intensity of peak A decreased from 726.8 to 536.5 through UF membrane (30 kDa) in Fig. 2(b). Fluorescence intensity of peak C decreased from 501.2 to 457.1, and fluorescence intensity of peak A decreased from 536.5 to 498.5 through UF membrane (10 kDa) in Fig. 2(c). Fluorescence intensity of peak C decreased from 457.1 to 250.1, and fluorescence intensity of peak A decreased from 498.5 to 239.6 through UF membrane (5 kDa) in Fig. 2(d). The decreased phenomenon of peak C intensity was nearly disappeared, and fluorescence intensity of peak A decreased from 239.6 to 235.5 through UF membrane (3 kDa). Meanwhile, a new peak appeared at Ex/Em 225–237/340–350 nm in Fig. 2(e). The 3D-EEM characteristic of the new peak was similar to T2. The decrease phenomenon of peak C continued through UF membrane (1 kDa), and two new weak peaks appeared at Ex/Em 275/340 nm and 225–237/340–350 nm in Fig. 2(f). The 3D-EEM characteristic of the previous peak was similar to T1 and that of the later peak was similar to T2. These peaks mainly meant tryptophan-like and FA-like substances.

It could be shown that the source of the reservoir included HA, FA, and tryptophan-like substances. Based on the intensity of peak, it could be seen that the content of FA was high. The content of HA was less than FA and the content of tryptophan-like was low. In view of MWCO of organic matters (OMs) in the reservoir source, it could be seen that the intensity of HA peak was the most strong, when MWCO of OMs was larger than 5 kDa. However, it disappeared when MWCO of OMs was smaller than 3 kDa. The intensity of FA peak existed universally when MWCO of OMs was larger than 1 kDa. And the intensity of FA peak changed obviously when MWCO of OMs was 5–1 kDa. The result showed that FA existed mostly in this MWCO interval. While micro molecular matters (<1 kDa) mainly included micro molecular tryptophan-like and fulvic acid-like substances.

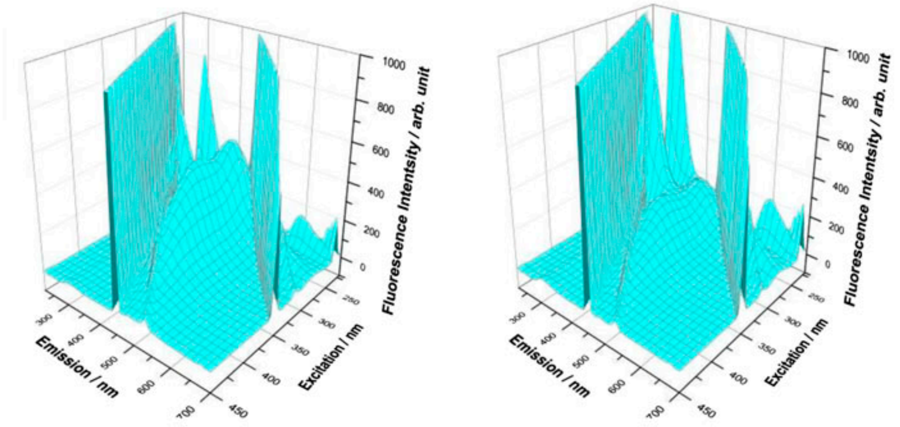
3.1.2. MWCO

MWCO of the OMs in the influent was classified, when color was 24, 32, and 42, respectively in Fig. 3. Fig. 3 shows MWCO mainly concentrated in MWCO <1 kDa and 5–30 kDa. Micro molecular FA-like substances were the majority, while macromolecular humic acid was the minority. MWCO of NOM could be determined by UF system. Pelekani et al. [31] compared UF system and high-performance size exclusion chromatography (HPSEC) technique in



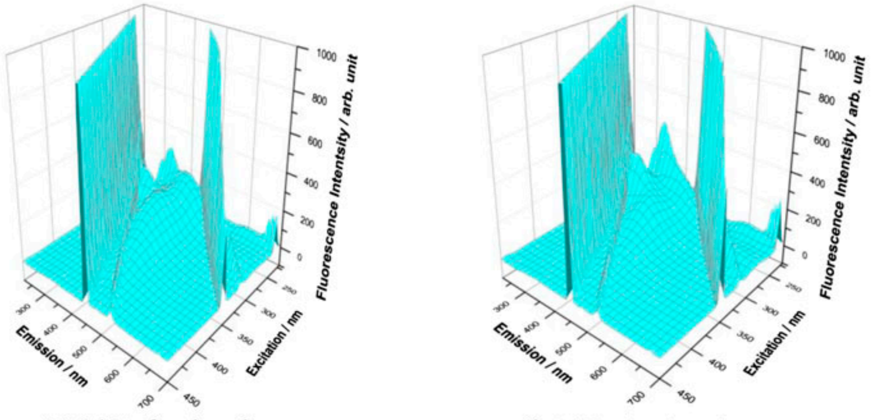
(a) Raw water

(b) 30 kDa fractionation



(c) 10 kDa fractionation

(d) 5 kDa fractionation



(e) 3 kDa fractionation

(f) 1 kDa fractionation

Fig. 2. 3D-EEM fluorescence spectroscopy of different MWCO in raw water.

determining MWCO, and they also found UF system was more suitable for the determination of small molecular weight substances. It also could be found that OMs accounted for 43.5% with low color when MWCO was <1 kDa, while they accounted for 32.5% with high color at the same MWCO interval. The color was higher, the content of macromolecular humic acid was more, while, the contribution of micro molecular fulvic to color was little. Moreover, in view of MWCO fractionation, it also could be found that HS got a

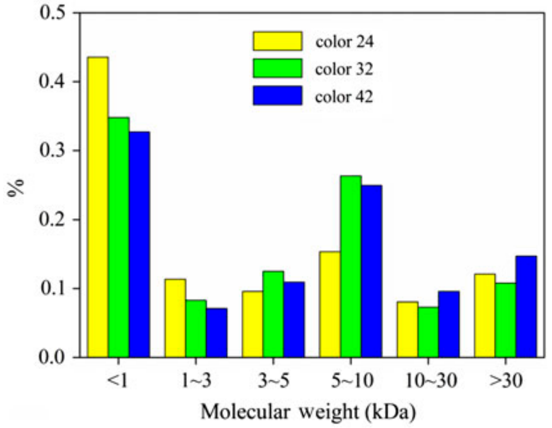


Fig. 3. MWCO of influent at different colors.

smaller molecular trend, especially in the frozen period. At the same DOC level, when the content of organic matters (<1 kDa) continued to grow, the color had a downward trend.

3.2. Relationship between color, MWCO and DBPs

Based on above mentioned experiments, the main source of color substances in water had already been determined. However, whether the color substances were the DBPs precursors after chlorination disinfection, it was needed further experiments to be proved. After the source water was disinfected by chlorination as shown in Fig. 4, various DBPs changed obviously with increased color and the increase percentage of HAAs was higher than that of THMs. In the condition of high color, chlorinated DBPs increased significantly, especially CH increasing the fastest, while the reducibility of water bodies significantly enhanced. These results showed that increased macromolecular HA was an important factor causing increased DBPs at high color.

Although the experiments showed that high color with increased high HA led to the rise of DBPs, the effect of the color substances with different MWCO fragments (measured by DOC) on DBPs formation potential was unclear. Therefore relevant experiments were carried out as shown in Fig. 5. The results showed that small molecular matters (<1 kDa) contributed much more for the formation of TCM and HAAs. TCM formation potential was lower at large molecular fragment and HAAs got much more formation potential when MDCO > 10 kDa and MDCO < 1 kDa. Meanwhile, macromolecular HA supplied much more contribution for TCM and micro molecular FA contributed much more for THMs and HAAs.

3.3. Removal efficiency of water treatment

3.3.1. 3D-EEM fluorescence spectroscopy

Based on the characteristics of 3D-EEM fluorescence spectroscopy during water treatment as shown in Fig. 6, the results indicated that the intensity of peak A and peak C decreased obviously after sedimentation, as the intensity of peak C decreased 35%, and the intensity of peak A decreased 32%. These two peaks continued to decrease with limited reduced degree after filtration, as the intensity of peak C decreased 39%, and the intensity of peak A decreased 35%. Bieroza et al. [32] also found fluorescence peak C intensity was found to be a sensitive and reliable measure of OM content. The research results of [33] Gone et al. indicated a high significant correlation between measured DOC removal and fluorescence-inferred DOC removal calculated for peak A, peak C, peak T

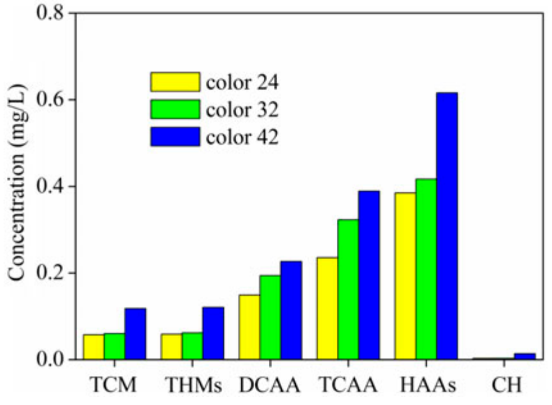


Fig. 4. DBPs at different colors.

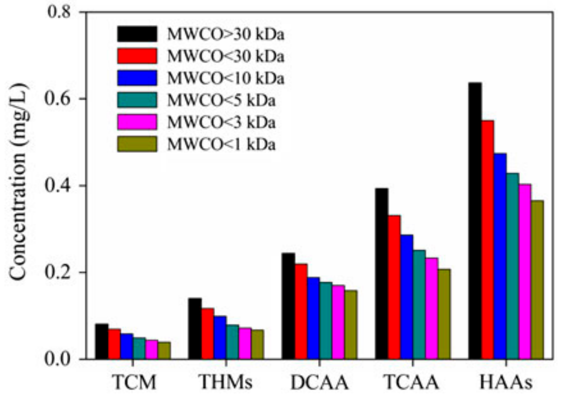
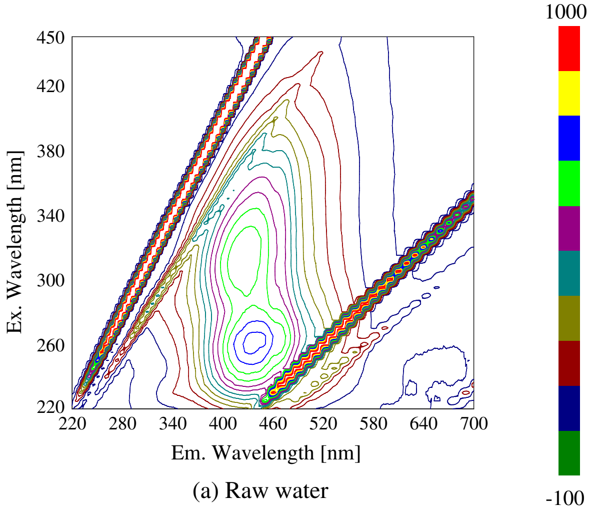
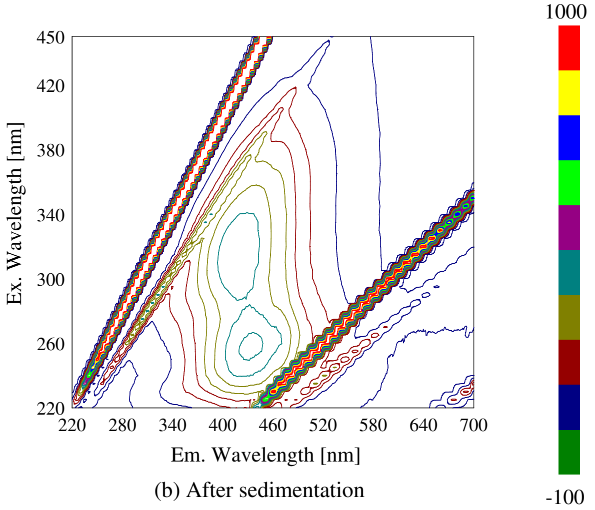


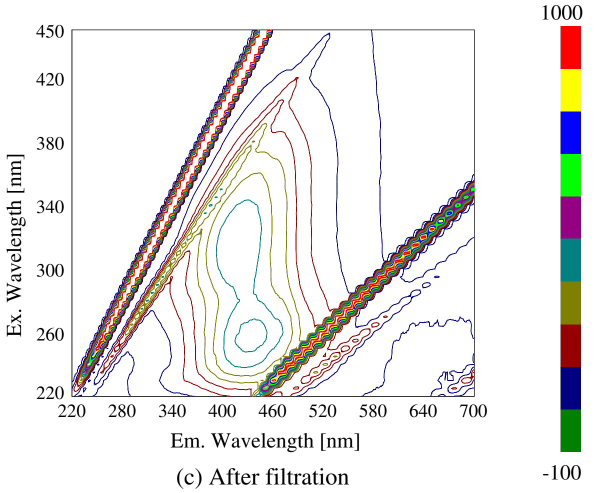
Fig. 5. DBPs at different MWCO.



(a) Raw water



(b) After sedimentation



(c) After filtration

Fig. 6. 3D-EEM of water during water treatment.

indicating a strong linear relationship between DOC removal and fluorescence intensities.

3.3.2. MWCO

Effluent MWCO fractionation variation during water treatment was shown in Fig. 7. The results showed that the process of coagulation–sedimentation played a dominant role in the removal of NOM and the filtration process had limited ability to further remove NOM. The whole water treatment system removed less micromolecular NOM (<5 kDa) and removal ability of other NOM (>5 kDa) was strong. It could be determined that conventional water treatment had a certain removal effect of HA, which was the main color substances, and FA removal capacity was poor. Therefore, the remaining color and DOC mainly derived from FA and the left FA resulted in DBPs formation produced by chlorination. Myllykangas et al. also found the sum of the molecular size fractions of the lake water was reduced by 47% during the bank filtration process, and the molecular size fractions of the artificially recharged groundwater was reduced by 55% during the process in the water treatment plant [34].

3.3.3. Color and turbidity

The above results showed that the conventional water purification process exhibited limited removal ability of color substances, especially NOM. Removal efficiency of color and turbidity during different purification process was compared in Fig. 8 (raw water, water after sedimentation, and water after filtration during four typical water periods).

The results showed that removal efficiency of color during each purification process was relatively stable. Coagulation–sedimentation process showed the highest removal efficiency of color, which was the core color control section, and removal efficiency was about

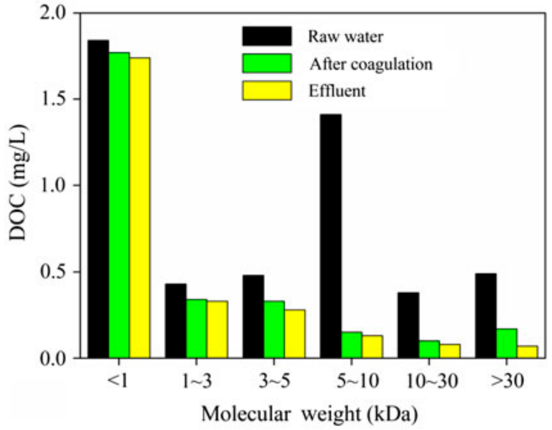
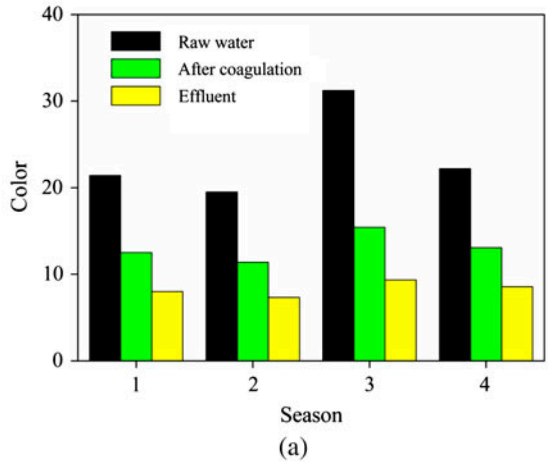


Fig. 7. MWCO variation during water treatment.



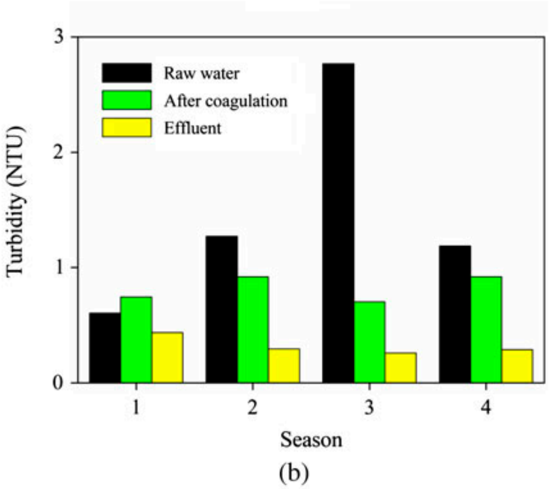


Fig. 8. Removal efficiency (a: color; b: turbidity) at different periods.
Notes: 1: ice-bound period; 2: variable period of Spring; 3: stable period of Summer; 4: variable period of Winter.

40–50%, average 44.8%. Final effluent color could generally controlled at about 10°, and total removal efficiency of color was about 65.4%

There was big difference about removal efficiency of turbidity during different period, especially in the effluent of sedimentation tank. When the turbidity of raw water was low, especially during ice-bound period, the turbidity of effluent after sedimentation increased again contrarily, for incomplete hydrolysis reaction of coagulant at low temperature. However, although the turbidity of raw water was high during the stable period of summer, the turbidity of effluent after sedimentation and final effluent was still very low. The results were proved indirectly that color substances and turbidity substances in the reservoir water were not entirely consistent. Color substances were mainly composed of NOM, with relatively stable removal efficiency, and a large amount of coagulant should be added to achieve the desired effect, when there were turbidity substances in the raw water. However, NOM had little effect on complicated turbidity composition and it was difficult to remove under low temperature and low turbidity conditions.

4. Conclusion

Reason for color composition in a water reservoir and DBPs formation following coagulation and chlorination during its conventional water treatment in northeast of China was elaborated and effect of color on water treatment process was also investigated. The results show that:

(1) MWD of NOM in the reservoir water were mainly between <1 kDa and 5–10 kDa. There were two typical EEM peaks in reservoir water, which was peak A (Ex/Em: 260/450 nm) representing FA-like substances (1–5 k Da) and peak C (Ex/Em: 330/450 nm) standing for HA-like substances (MWCO > 5 kDa).

(2) In the reservoir water, the higher concentration of DOC was, the more content of macromolecular HA was, contributing to the color the most, while small molecular FA contributed less to the color. When the color increased, various DBPs changed obviously and the increased proportion of HAAs was larger than THMs. DBPs formation potential of TCM was lower at larger MWCO, while DBPs formation potential of HAAs was larger when MWCO was more than 10 kDa and less than 1 kDa. It could be determined that macromolecular HS contributed more to HAAs and small molecular fulvic acid made more contribution to THMs and HAAs.

(3) In traditional water treatment process, coagulation–sedimentation process could remove OMs whose MWCO were more than 5 kDa by about 30% and after filtration process, removal efficiency was more than 60% (HA mainly). However, coagulation–sedimentation process could only remove OMs whose MWCO was less than 3 kDa (FA) by about 22% limitedly, indicating the left DOC in the water was small molecular organics.

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