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Water Treatment of Manganese Oxides and Organic Matter through Pre-Oxidation and Coagulation/Sedimentation †

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Abstract: We investigated the reducing rate of manganese oxides (MnOs) and organic matter in water by using pre-oxidation and coagulation/sedimentation methods with different chemicals. The reduction rate using NaOCl for organic matter was about 11%, while that of manganese was 12%. The reduction rate using chlorine dioxide (ClO_2) was only 7% for organic matter. However, the rate of manganese was 29% when using ClO_2. Potassium permanganate ($KMnO_4$) removed organic matter and MnOs more effectively, with a rate of 18 and 71%. Moreover, aluminum sulfate ($Al_2(SO_4)_3$), ferric chloride ($FeCl_3$), and polysilicate iron (PSI) worked more effectively, with a reduction rate of 99% and 55% for turbidity and organic matter.

Keywords: pre-chlorination; manganese; organic matter; turbidity

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1. Introduction

Drinking water in Taiwan is obtained from natural reservoirs. In summer, strong sunlight and abundant nutrients in water trigger frequent eutrophication, which complicates water purification processes. During the dry season, the storage of water in the reservoir decreases, and dissolved oxygen (DO) decreases, which forms an anaerobic environment at the bottom. The microbial reduction of iron and manganese oxides increases the concentration of Fe^{2+} and Mn^{2+} in water and turbidity, which makes the water body black and worsens the water quality. This also causes discoloration and a metallic taste in drinking water, and residual manganese ions promote the growth and reproduction of microorganisms. An increase in the number of microorganisms allows manganese oxides to be formed and creates clogging in the pipelines [1,2]. The forms of manganese oxides in water are diverse, including particulate manganese (>0.45 μm), colloidal manganese (<30,000 Da~0.20 μm), and dissolved manganese. Colloidal and particulate manganese can be removed using coagulation and filtration. Dissolved manganese requires oxidants (chlorine, ClO_2, and $KMnO_4$) to be removed from water by forming manganese dioxide, which coagulates and can be filtered out.

Natural organic matter is primarily derived from plants, animals, and their degradation products in aquatic, terrestrial, and marine environments [3]. Owing to the diversity of sources, the composition and concentration of natural organic matter in the environment vary depending on backgrounds, locations, climate, and human activities [4]. Natural

organic matter is a complex multiphase mixture of organic substances with different molecular sizes and physical and chemical properties, because it contains various functional groups that include aromatic, aliphatic, phenolic, and quinone structures [5].

In the water-chlorination purification process, the phenols and carboxyls of organic matter in water react with chlorine to generate disinfection byproducts (DBPs) such as total trihalomethane, haloacetic acid, haloketone, and haloacetonitrile. The generation of DBPs is determined by the type of organic matter, temperature, inorganic salts, and pH value [6]. In general, pre-oxidation with coagulants for water purification is carried out to remove organic matter. For this, coagulants such as iron and aluminum salts are hydrolyzed into positively charged metal complex ions, which are then adsorbed onto the negatively charged colloidal surface. Consequently, large polymers containing organic matter are coagulated and deposited on the bottom. As the composition of natural organic matter in different water bodies varies, the removal efficiency of organic matter becomes different.

Therefore, the optimal conditions and strategies for removing manganese oxides and organic matter need to be determined. Thus, we performed related tests under different types and doses of oxidants and coagulants to improve the efficiency of water purification in this study. The result provides a basic reference for further investigation and the development of more effective water treatment procedures.

2. Materials and Methods

The experimental process of this study is shown in Figure 1. The water samples were obtained from reservoirs in south Taiwan. The concentrations of iron, manganese, and total organic carbon (TOC) and the collective property of water were analyzed. The characteristics of organic matter were also analyzed. In the pre-oxidation test, NaOCl, ClO_2, and $KMnO_4$ were used as oxidants to investigate the effect of oxidants on the TOC concentration in water. Changes in the organic matter composition in pre-oxidation were analyzed using the excitation–emission matrix (EEM). The rate of coagulation and sedimentation was measured using coagulants such as $Al_2(SO_4)_3$, $FeCl_3$, and PSI. The pH and turbidity of water samples were measured to identify suitable dosing modes of the coagulants. A simulated distribution system (SDS) was used for chlorination, as shown in Figure 2. The SDS simulated the generation of DBPs in the water pipe after the chlorination of treated water.

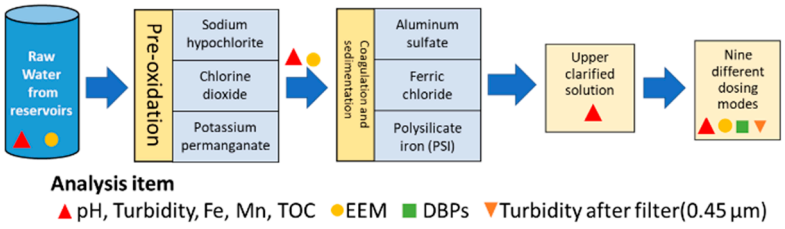


Figure 1. Experimental procedure of this study.

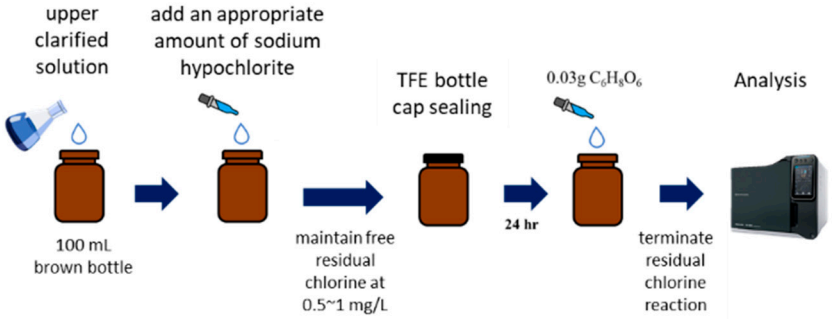


Figure 2. Illustration of SDS chlorination.

Water samples were collected in a 100 mL brown bottle, and an appropriate amount of NaOCl was added to it to control the residual chlorine between 0.5 and 1 mg/L. After the bottle was sealed, the samples were left for 24 h for enough of a reaction to be produced (in real situations, drinking water needs a 24 h wait after NaOCl has been added in a water purification station before reaching domestic homes). After 24 h, ascorbic acid was added to terminate the chlorine reaction.

3. Results

3.1. Effect of Oxidants on Organic Matter

3.1.1. Types and Doses

As the dose of the oxidant increased, the removal rate of TOC increased (Figure 3). However, an excessive amount of the oxidant decreased the removal rate of TOC. References [7,8] pointed out that certain oxidants and excessive oxidants ruptured algal cells, thus resulting in the release of intracellular organic matter. This increased the organic matter content in water, which was reproduced in the results of the experiment in this study. The water samples in this study were not filtered and directly oxidized. The dominant algae species in the water samples were green and blue-green algae.

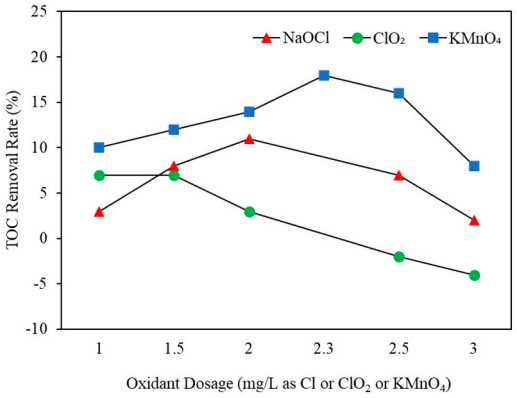


Figure 3. Effect of different oxidants and doses on TOC removal rate.

3.1.2. Effect of Oxidants on Coagulation and Sedimentation

The composition of particulate and dissolved organic matter in water was determined based on the characteristic interval of the EEM. In this study, the excitation and emission wavelength ranges were set to 200–400 and 250–500 nm, respectively. The spectra were classified into four areas, such as by-product-like soluble microbial (Area I, SMP), humic-acid-like soluble microbial (Area II, HA), fulvic-acid-like soluble microbial (Area III, FA), and aromatic proteins soluble microbial (Area IV, AP).

Figure 4 presents the optimal removal dose of the TOC for different oxidants. In this study, 2 mg/L of NaOCl, 1.5 mg/L of ClO_2, and 2.3 mg/L of $KMnO_4$ were added to the water sample for pre-oxidation, and the mixture was stirred for 30 s at 200 rpm. Changes in the organic matter composition before and after stirring were analyzed with EEM. Before oxidation, the spectral intensity of the raw water was the strongest in Area III, followed by Areas II, IV, and I. The organic matter composition in the water was primarily FA-like in composition. With the other three oxidants, the spectral intensities of Areas I, II, and III showed slight changes compared with those before oxidation. Meanwhile, the spectral intensity of Area IV changed significantly after NaOCl was added. Fluorescence data showed a significant weakening trend in Area IV. The decrease in the fluorescence intensity of aromatic proteins was attributed to the generation of DBPs [9]. The addition of ClO_2 and $KMnO_4$ also changed the spectral intensity slightly.

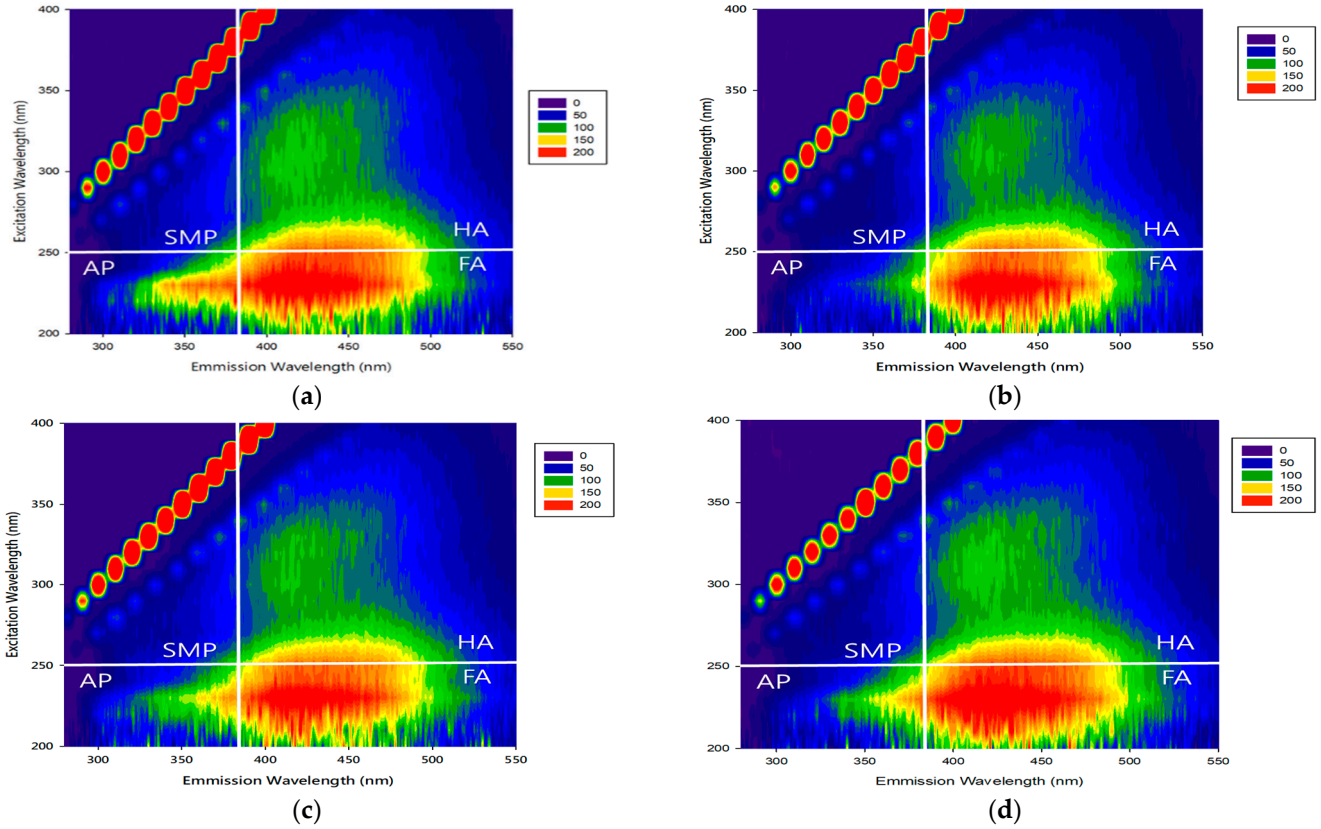


Figure 4. EEM spectra of organic matter oxidized by oxidants: (**a**) no oxidation in water sample; (**b**) 2 mg/L of NaOCl; (**c**) 1.5 mg/L of ClO_2; (**d**) 2.3 mg/L of $KMnO_4$.

3.2. Effect of Oxidants with Coagulants on Coagulation and Sedimentation

In this study, changes in the organic matter content in the water sample were caused by different doses of oxidants. The best treatment method, using oxidants for water purification, completely removed algal cells without rupture, which avoided an increase in dissolved organic matter in water and the generation of DBPs [10]. Therefore, the appropriate doses of oxidants and coagulants needed to be selected for coagulation and sedimentation tests. The selected doses of NaOCl, ClO_2, and $KMnO_4$ were 2, 1.5, and 2.3 mg/L in this study.

Figures 5–8 show the changes in pH and the content of organic matter with three oxidants and the different doses of $Al_2(SO_4)_3$ and $FeCl_3$. When the dose of $Al_2(SO_4)_3$ increased, the removal rate of TOC also increased, while the pH value decreased to 6.7. According to the on-site operation protocol of the water purification plant, the pH of the treated water is maintained above 7 to not exceed the regulatory standards for pH level due to the gradual acidification of drinking water during its transportation in the distribution system. When the dose of $FeCl_3$ increased, the removal rate of TOC kept increasing. The dose of PSI was set independently. From the result, it was found that the appropriate doses of $Al_2(SO_4)_3$, $FeCl_3$, and PSI were 8 mg/L (as 4.2 mg/L Al), 8 mg/L (as 2.8 mg/L Fe), and 3 mg/L (as 3 mg/L as Fe), respectively. In the test for coagulation and sedimentation, the doses of the three oxidants were used with the doses of the three coagulants to produce nine different dosing modes (Table 1). The result was analyzed to investigate the effects of different dosing modes on pH, the concentration of manganese and iron ions, the content of organic matter, and the generation of DBPs. The water samples in this study were not filtered and pH adjusted.

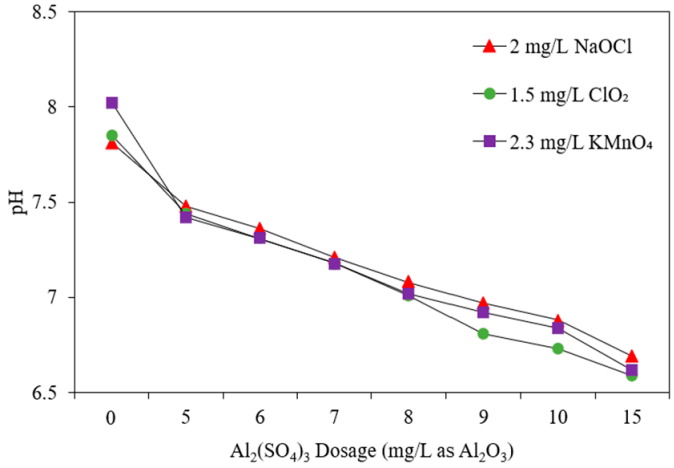


Figure 5. pH changes caused by three oxidants combined with different doses of $Al_2(SO_4)_3$.

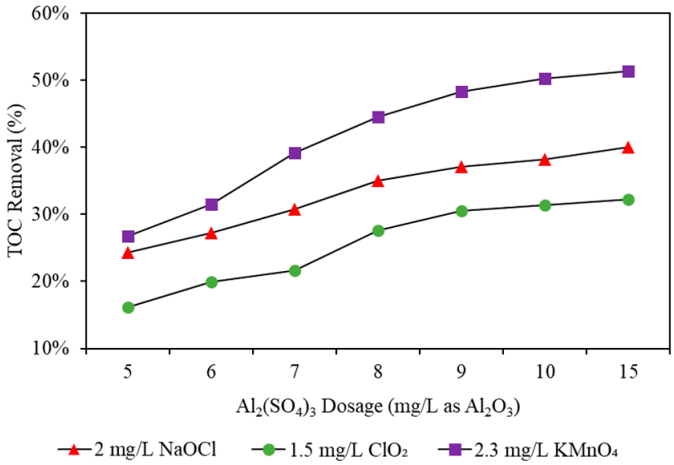


Figure 6. Changes in TOC removal rates caused by three oxidants combined with different doses of $Al_2(SO_4)_3$.

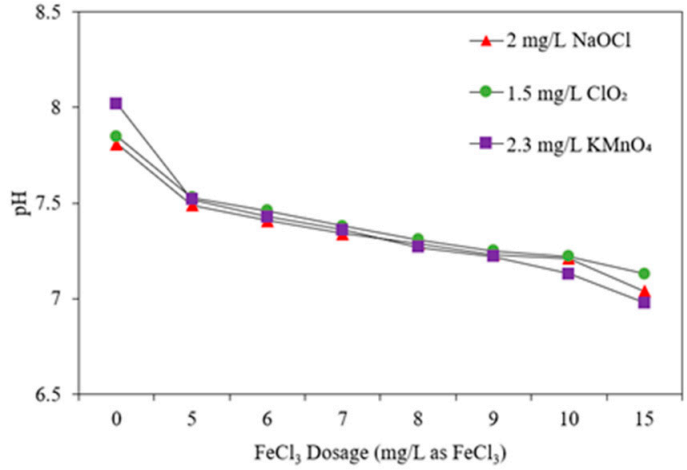


Figure 7. pH changes caused by three oxidants combined with different doses of $FeCl_3$.

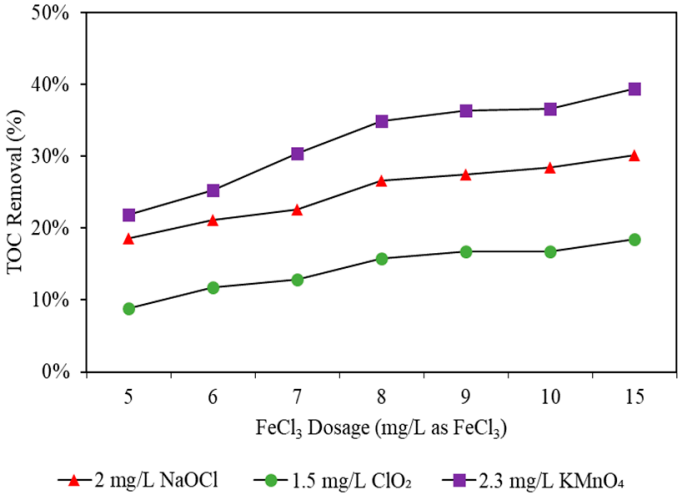


Figure 8. Changes in TOC removal rates caused by three oxidants combined with different doses of $FeCl_3$.

Table 1. Nine different dosing operation modes.

Mode	Dosing Operation
A	2 mg/L NaOCl + 8 mg/L $Al_2(SO_4)_3$
B	2 mg/L NaOCl + 8 mg/L $FeCl_3$
C	2 mg/L NaOCl + 3 mg/L PSI
D	1.5 mg/L ClO_2 + 8 mg/L $Al_2(SO_4)_3$
E	1.5 mg/L ClO_2 + 8 mg/L $FeCl_3$
F	1.5 mg/L ClO_2 + 3 mg/L PSI
G	2.3 mg/L $KMnO_4$ + 8 mg/L $Al_2(SO_4)_3$
H	2.3 mg/L $KMnO_4$ + 8 mg/L $FeCl_3$
I	2.3 mg/L $KMnO_4$ + 3 mg/L PSI

3.2.1. Manganese Removal

Table 2 shows the removal effects of manganese ions with different dosing modes. With no coagulant, manganese ions were removed only via pre-oxidation with NaOCl, and the removal rate was approximately 12%. When $Al_2(SO_4)_3$, $FeCl_3$, and PSI were added, the removal rate of manganese ions increased to 37–50% with coagulation and sedimentation but the concentration of manganese ions still exceeded 0.05 mg/L, which is a drinking water standard. The higher concentration over 0.05 mg/L was maintained because NaOCl could not effectively oxidize manganese ions in pre-oxidation, and a trace amount of manganese ions were not oxidized. Consequently, manganese removal in subsequent coagulation and sedimentation became less prominent. When ClO_2 was used for pre-oxidation, the removal rate of the manganese ions was approximately 30%. If three types of coagulants were added, then the removal rate of manganese ions in coagulation and sedimentation was 63–72%. Correspondingly, the concentration of residual manganese ions in water was lower than 0.05 mg/L. The high oxidizing ability of ClO_2 allowed a considerable removal of manganese ions, which resulted in a better manganese removal effect. When $KMnO_4$ was used for pre-oxidation, the removal rate of manganese ions reached 71%. With $KMnO_4$ and the three coagulants, the removal rate of manganese ions increased to 84–92%. Additionally, the residual manganese concentration was below the drinking water standard. $KMnO_4$ effectively and rapidly oxidized dissolved manganese ions in water to form manganese particles. Coagulation and sedimentation derived an effective removal effect. The experimental results showed that removing manganese ions from water required extensive oxidation in the pre-oxidation process and coagulation and sedimentation. The best oxidant for manganese removal was $KMnO_4$. Appropriate oxidants with coagulants for coagulation and sedimentation guarantee decreased residual manganese in water, which satisfies the quality standards of drinking water.

Table 2. Changes in manganese removal under nine dosing combinations.

Mode	Mn Removal Rate (%)	Residual Mn (mg/L)
Raw water	N/A	0.134
NaOCl	12%	0.118
A	37%	0.085
B	54%	0.062
C	51%	0.065
ClO_2	29%	0.095
D	65%	0.047
E	72%	0.038
F	69%	0.041
$KMnO_4$	71%	0.039
G	84%	0.022
H	92%	0.011
I	89%	0.015

3.2.2. Turbidity Decrease

Table 3 shows the decrease in turbidity with nine dosing modes. Without coagulants, the turbidity of the water sample was decreased only by pre-oxidation. When NaOCl was used as the oxidant, the decrease rate of turbidity was only 3%, and no significant improvement was indicated. With $Al_2(SO_4)_3$, $FeCl_3$, and PSI, the turbidity decreased by 98%. Using coagulants and filtration, turbidity was reduced to 0.46 nephelometric turbidity units (NTU). When ClO_2 was used for pre-oxidation, turbidity decreased to 20%. Pre-oxidation oxidized metal ions in water and produced metal particles that coagulated and sedimented. If three coagulants were added, turbidity decreased by 98–99% through coagulation and sedimentation. Oxidation by ClO_2 did not change the distribution of particle sizes but resulted in the micro-gelation of colloidal particles, thus facilitating particle removal [11]. The maximum decrease rate of turbidity via pre-oxidation using $KMnO_4$ was approximately 36%. Using $KMnO_4$ with the three coagulants, turbidity decreased by 99%, mainly owing to the adsorption of manganese dioxide. Reference [7] discovered that $KMnO_4$ facilitated the removal of organic and inorganic particles. Their experimental results showed that ClO_2 and $KMnO_4$ contributed to the removal of colloidal particles in water. $Al_2(SO_4)_3$ decreased turbidity most significantly. Meanwhile, using ClO_2 and $KMnO_4$ with $Al_2(SO_4)_3$ showed a better effect on turbidity decrease. Using filtration, turbidity could be maintained below the drinking water standard. Therefore, turbidity could be decreased through coagulation, sedimentation, and filtration.

Table 3. Changes in turbidity removal under nine dosing combinations.

Mode	Turbidity Removal Rate (%)	Residual Turbidity (NTU)
Raw water	N/A	213
Only NaOCl	3%	206
A	99%	2.86
B	98%	3.96
C	98%	4.22
Only ClO_2	20%	170
D	99%	1.43
E	98%	3.71
F	98%	3.34
Only $KMnO_4$	36%	136
G	100%	0.11
H	99%	2.92
I	99%	2.69

3.2.3. TOC Removal

Table 4 shows the removal rates of TOC in the water sample using oxidants and coagulants. The results showed that TOC was removed by $KMnO_4$ more than by NaOCl

and ClO_2. Among the coagulants, $Al_2(SO_4)_3$ was the most effective in removing TOC, with a removal rate of up to 55%. No significant difference was found in the removal of TOC between $FeCl_3$ and PSI. As organic matter was composed of various large- and small-molecule organic components, the removal of different types of organic matter by different coagulants could not be determined based on the removal results of the TOC.

Table 4. Changes in TOC removal under nine dosing combinations.

Mode		TOC Removal Rate (%)	Residual TOC (mg/L)
Raw water		N/A	2.82
NaOCl	A	46%	1.51
	B	30%	1.98
	C	28%	2.03
ClO_2	D	38%	1.75
	E	23%	2.17
	F	24%	2.13
$KMnO_4$	G	55%	1.26
	H	40%	1.69
	I	37%	1.78

3.2.4. Effect of Organic Matter Composition on Its Removal

Table 5 shows the effect of oxidants and coagulants on the organic matter with different compositions. The spectral intensity of the raw water was the highest in Area III, and Areas II, IV, and I, in that order. Fluorescence intensities decreased according to the degree of coagulation and sedimentation. The fluorescence intensities in Areas II (HA) and III (FA) decreased most significantly. Thus, it was found that coagulation and sedimentation effectively removed organic matter from water, and in particular large-molecule organic matter. Higher removal rates were achieved with modes A, D, and G, in which $Al_2(SO_4)_3$ was used as a coagulant. Mode G showed the best removal efficiency of organic matter in Areas II (HA) and III (FA), where the fluorescence reduction rates were 60 and 56%, respectively. The fluorescence reduction rates using $FeCl_3$ and PSI were similar to $Al_2(SO_4)_3$. However, the fluorescence intensity in Area IV with mode E removed more organic matter. The high oxidizing ability of ClO_2 allowed the disintegration of large-molecule organic matter containing multiple benzene rings and phenolic groups in Areas II and III into aromatic organic matter with smaller molecular weights. In Ref. [12], the molecular weight changes in organic matter were observed after adding ClO_2. It was discovered that ClO_2 oxidized and disintegrated large-molecule organic matter into smaller molecules. The combination of $KMnO_4$ and $Al_2(SO_4)_3$ enabled the most significant decrease in the fluorescence intensity of water after sedimentation. The reduced form of $KMnO_4$ was adsorbed on the surface of algal cells and organic matter, thus increasing the weight. Using coagulants reduced the coagulation and sedimentation of organic matter. When the pH was greater than 7, the hydrolysis products of $Al_2(SO_4)_3$ became colloidal precipitates of $Al(OH)_{3(s)}$. At this point, the gel plume formed became large [13], and organic matter in the water was removed via sedimentation.

Table 5. Analysis results of raw water EEM under different dosing operation modes.

Organic Matter Type	Raw Water	Mode A		Mode B		Mode C	
	Spectral Intensity (E_0)	Spectral Intensity (E_0)	Removal Rate * (%)	Spectral Intensity (E_0)	Removal Rate (%)	Spectral Intensity (E_0)	Removal Rate (%)
SMP	51.172	34.842	32%	36.286	29%	34.971	32%
HA	75.366	34.634	54%	49.108	35%	48.607	36%
FA	128.23	63.388	51%	88.975	31%	88.355	31%
AP	64.506	49.504	23%	49.140	24%	46.599	28%
Organic Matter Type		**Mode D**		**Mode E**		**Mode F**	
		Spectral intensity (E_0)	Removal rate * (%)	Spectral intensity (E_0)	Removal Rate (%)	Spectral intensity (E_0)	Removal Rate (%)
SMP		33.550	34%	42.301	17%	39.552	23%
HA		35.454	53%	51.184	32%	51.952	31%
FA		62.282	51%	95.687	25%	90.628	29%
AP		43.605	32%	66.207	−3%	46.727	28%
Organic Matter Type		**Mode G**		**Mode H**		**Mode I**	
		Spectral intensity (E_0)	Removal rate * (%)	Spectral intensity (E_0)	Removal Rate (%)	Spectral intensity (E_0)	Removal Rate (%)
SMP		29.337	43%	32.914	36%	31.947	38%
HA		29.959	60%	49.305	35%	47.358	37%
FA		56.034	56%	90.962	29%	87.158	32%
AP		39.681	38%	44.134	32%	39.896	38%

* Removal rate (%) = $(E_0 - E_1)/E_0$.

3.2.5. DBPs

With coagulation and sedimentation in different dosage modes, the number of DBPs in water with the SDS chlorination experiment (24 h) was measured as shown in Table 6. When $KMnO_4$ and ClO_2 were used as the pre-oxidant, the concentration of DBPs was more effectively decreased than with NaOCl, and the decrease in haloacetic acid was significant. If $Al_2(SO_4)_3$ was used as the coagulant, the concentration of DBPs (total trihalomethane and haloacetic acid) was lowered compared to those with $FeCl_3$ and PSI. $Al_2(SO_4)_3$ had the best removal efficiency for organic matter in Areas II and III of the EEM. Since large-molecule organic matter such as HA and FA contained more benzene rings and phenolic groups, their disinfection by-product generation potential was higher than that of small-molecule organic matter [14,15].

Table 6. Generation of DBPs under different dosing operation modes.

Mode	Disinfection By-Product	
	TTHMs	HAAs
A	0.077	0.066
B	0.119	0.091
C	0.129	0.088
D	0.079	0.052
E	0.115	0.074
F	0.119	0.079
G	0.071	0.045
H	0.107	0.078
I	0.117	0.086

4. Conclusions

The experimental results on removing manganese ions from the water samples showed that NaOCl, ClO_2, and $KMnO_4$ were effective oxidants. $KMnO_4$ showed the highest manganese removal rate, followed by ClO_2. The best removal rate of TOC was observed

when using $KMnO_4$. Among the three coagulants, $Al_2(SO_4)_3$ was better at removing TOC. Using ClO_2 and $KMnO_4$ instead of NaOCl as pre-oxidants reduced the generation of trihalomethane (THM) and haloacetic-acid (HAA). The amount of organic matter in Areas II (HA) and III (FA) in the water sample was considerably decreased through coagulation and sedimentation. A 0.45 μm nylon filter was used to simulate the filtration process in this study; filters fabricated using quartz sand, anthracite, or other materials may be used to construct a better water treatment process.

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