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Simultaneous removal of algae and its odor metabolites in raw water by potassium ferrate

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ABSTRACT

The removal of algae and odorous metabolite dimethyl trisulfide (DMTS) in water using potassium ferrate (K_2FeO_4) was investigated. The treatment effect was compared with polyferric chloride (PFC) and potassium permanganate ($KMnO_4$) pre-oxidation followed by PFC. Factors such as pH, oxidation contact time, and turbidity were also analyzed. The results show that K_2FeO_4 is more effective in algae elimination than PFC, with a higher removal ratio of 92.4% when dosage is equivalently measured by Fe. Removal of DMTS by K_2FeO_4 is better than pre-oxidized $KMnO_4$, with decreased oxidation contact time. The removal ratio of DMTS by K_2FeO_4 was 92.5% with contact time set to 1 min; in comparison, $KMnO_4$ had a removal ratio of only 74.6% after 10 min of contact.

Keywords: Algae removal; Odor contaminant; Dimethyl trisulfide; Potassium ferrate; Water purification

1. Introduction

Critically contaminated water sources around cities, and the aqueous micro-pollutants contained in them have brought difficulties and insecurities in water supply system, which pose a serious problem in China. Prevalent and frequent breakout of algae and its metabolites is conspicuous, and since those algal metabolites deteriorate water quality, they have resulted in increased concern of government officials, researchers, and citizens [1–5]. Numerous metabolites, such as microcystins, taste and odor compounds (T&O), extracellular organic matters (EOM), and so on, have been detected in raw water drawn from reservoirs or rivers containing high algae concentration seasonally. Among these metabolites are T&O compounds, which are small and volatile molecules that bypass conventional treatment processes, infiltrate into supply networks, and induce disgusting odors due to their low odor threshold values. These compounds bring about problems, such as public suspicion and panic, due to the unpleasant taste and odor of the water.

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Dimethyl trisulfide (DMTS) [6] is a specific type of odorous contaminant that induces a pungent, disgusting odor into the water even at low concentrations (at ng/L^{-1}). It is mainly generated by *microcystis*, *oscillatoria* and decayed organisms. Thio-ethers including DMTS were the primary cause of the water supply crisis in Wuxi, China in 2007; and finally one of the most serious water supply accidents caused by an uncertain complex related to algae bloom, was solved by potassium permanganate ($KMnO_4$) together with powder carbon [1,7,8].

Fe(VI), in the form of Potassium ferrate (K_2FeO_4), is a powerful oxidant in a wide pH range and can be applied as an oxidant in water and wastewater treatment. The standard oxidation potential of Fe(VI) varies from +2.20 V to +0.72 V in acidic and basic solutions [9,10]. During aqueous oxidation reaction, Fe (VI) is reduced finally to Fe(III) or to an insoluble end-product, ferric hydroxide ($Fe(OH)_3$) [11] which can aid in the removal of micro particles. Hence, K_2FeO_4 can act as not only an oxidant but also a coagulant. Fe(VI) is proved to be an environmentally friendly oxidant and safe to be used in drinking water production [12,13], and which acts as an optimum alternative to current water treatment chemicals such as $KMnO_4$ or chlorine because it can avoid the production of effluent color and byproducts of disinfection [14–19].

In the current paper, *oscillatoria* and DMTS are selected to simulate waters polluted by high algae and T&O metabolites. K_2FeO_4 is applied to remove algae and DMTS simultaneously. Its dual function of oxidation and coagulation is then evaluated by comparison with that of common pretreatment process of $KMnO_4$ pre-oxidation followed by polyferric chloride (PFC). Other factors influencing the reaction are also analyzed.

2. Materials and methods

2.1. Experimental reagents and devices

The reagents used in this study included the following: DMTS (⩾95%, TCI, Japan), n-hexane (⩾99%, HPLC grade, CNW, Germany), sodium chloride and sodium thiosulfate (⩾99.5%, analytical grade, Nanjing Chemical Regent Co., Ltd), *oscillatoria* (pure algae, Institute of Hydroecology, MWR & CAS), PFC ($B = 1.0$, self-made), $KMnO_4$ (⩾99.5%, analytical grade, Nanjing Chemical Regent Co., Ltd), K_2FeO_4 (⩾50%, self-made), MF memberane (0.45 μm, whatmam, American) and pure water (Milli-Q, 18 MΩ).

The equipments used in this experiment included the following: gas chromatograph (GC-2014, SHIMADZU, Japan), optical microscope (XSP-2C, optical instrument factory, Shanghai), portable turbidimeter (2100P, HACH, USA), and portable pH meter (SensION, HACH, USA).

2.2. Procedure

Kinetic experiments of DMTS oxidation by K_2FeO_4 were carried out in a 15-L self-made homogeneous reactor. Artificial water solutions were prepared through the addition of standard DMTS chemical to pure water and make concentrations of up to 550–650 μg/L (the measured value was taken as valid). To avoid rapid decomposition of K_2FeO_4, and also to simulate the real algal-laden water, the solutions were adjusted to pH = 10.2 using a sodium bicarbonate-sodium carbonate buffer. All reactions were carried out by stirring at 200 r/min at 25℃. Samples were taken at serial specified intervals and residual oxidant was neutralized by sodium thiosulfate immediately.

Simultaneous removal experiments of algae and DMTS by K_2FeO_4 and combined process of $KMnO_4$ and PFC were conducted in 1-L beakers with 800 mL samples at 25℃. The water samples were prepared through addition of *oscillatoria* and DMTS into simple treated river water. *Oscillatoria* was purchased from the Institute of Hydroecology (MWR and CAS) and was cultured with a BG11 medium. Raw water, drawn from a little river inside the campus, was filtrated by MF memberane and then diluted until algae concentration ranged from 10^5 cells/L to 10^6 cells/L. DMTS was added to make concentrations of up to 550–650 μg/L (the measured value was taken as valid). Afterward, the prepared solutions were adjusted to the designated pH value by using a sodium bicarbonate-sodium carbonate buffer and to the designated turbidity by using kaolin. The quality parameters of artificial water are shown in Table 1.

In simultaneous removal experiments, contact oxidation was carried out by stirring at 100 r/min for 1 min for K_2FeO_4 and 10–20 min for $KMnO_4$. Subsequently, a certain amount of PFC was added into solutions oxidized by $KMnO_4$. For both methods, rapid mixing was conducted at 250 r/min for 1 min and slow stirring at 50 r/min for 10 min. About

Table 1
Quality parameters of artificial water

Index	Oscillatoria (cells· L^{-1})	DMTS (μg· L^{-1})	UV_{254} (cm^{-1})	Turbidity (NTU)	pH
	6.4×10^5	555–600	0.035	62–65	10.8

100 mL of upper liquid was sampled after 30 min of precipitation for target compound and algae analysis.

2.3. Analytical methods

One milliliter hexane was adopted to analyze DMTS by liquid-liquid extraction. Five gram of NaCl, which was dried in advance, was added to 100-mL water samples. After rapid mixing at 1,200 r/min for 10 min (by magnetic stirring) and 30 min (by static setting), upper hexane was analyzed by gas chromatograph (splitless) equipped with a flame ionization detector (injector: 250℃). An RT-5 column (30 m) with an ID of 0.25 mm and a film thickness of 0.25 μm was used. The oven temperature was increased firstly from 50 to 150℃ at a rate of 15℃/min and then to 240℃ at a rate of 30℃/min. Carrier gas was set at a flow rate of 1.5 mL/min and injection sample at 1 μL. The DMTS was held for 3.419 min. The algae were counted using a phytoplankton count plate under an optical microscope after being stabilized by Lugol's solution. The average of the final value was obtained using reflex sympathetic dystrophy controlled below 15%.

3. Results and discussion

3.1. DMTS oxidation kinetics by K_2FeO_4

Initial concentration of DMTS was held at a constant of 610 μg/L and oxidized by different K_2FeO_4 dosages (i.e. 2.0, 2.5, 3.0, and 4.0 mg/L, measured by Fe).

Generally, the removal of DMTS increased when oxidation time extended and oxidant dosage enhanced. When dosage was 4 mg/L, 52.4% DMTS was decomposed in 60 s and 65.0% in 600 s. It can be concluded that the contact oxidation time was very short and the removal of DMTS completed mainly before 60 s (in Fig. 1). In order to describe the removal more effectively, the oxidation processes were divided into two stages with the first one (i.e. the rapid oxidation stage) occurring from 0 to 30 s and the second stage (i.e. the slow oxidation stage) occurring from 60 to 600 s. Sharma et al. [20] discussed organosulfur compounds oxidation by ferrate(VI) through three possible steps, in which two related to Fe(VI): (1) $1-e^-$ transfers to form Fe(V) and Fe(V) oxidizes the compound; and (2) one $2-e^-$ transfers to yield Fe(IV) and Fe(IV) oxidizes the compound. According to the report, it was assumed in this paper that the two stages of DMTS oxidation were dominated by different Fe(VI), Fe(IV) and Fe(V) to summarize the kinetics model.

With respect to the reaction ratio of DMTS to K_2FeO_4 and the dosages, K_2FeO_4 was added excessively, and based on the situation, DMTS degradation curves presented characteristics of pseudo-second order and pseudo-first order kinetics in the first and second stages, respectively. The oxidation kinetics equations of DMTS degradation under different initial K_2FeO_4 dosages are shown in Figs. 2, 3 and Table 2.

Generally, the DMTS degradation curves fit the oxidation kinetics equation well with R^2 values of more than 0.9. From 0 to 30 s, the reaction rate increased from 0.00001 to 0.00005 s^{-1} with the increased K_2FeO_4 dosage from 2.0 to 4.0 mg/L, while the half-lives decreased from 164 to 31 s (Fig. 2 and Table 1). This indicated that the reaction rate was related to K_2FeO_4 concentration and increased with oxidant dosage enhancement. From 60 to 600 s, the half-lifes were longer than that in the first stage (Fig. 3

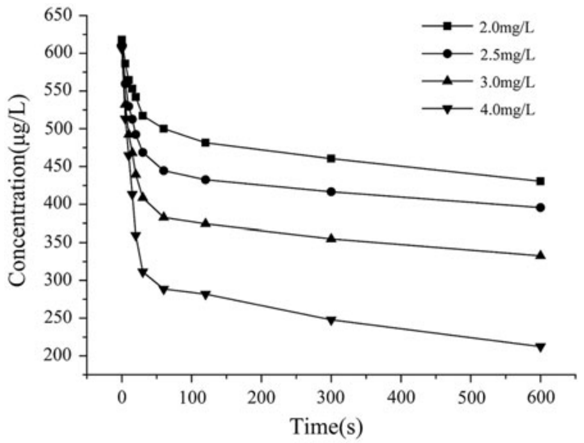

Fig. 1. Oxidation reaction of DMTS under different K_2FeO_4 dosages.

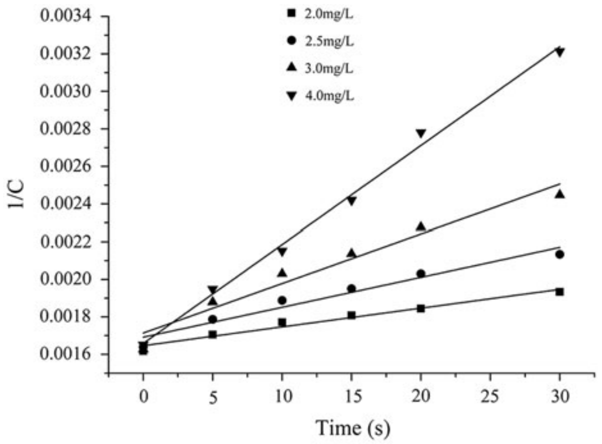

Fig. 2. Oxidation fit curves of DMTS under different K_2FeO_4 dosages (0–30 s).

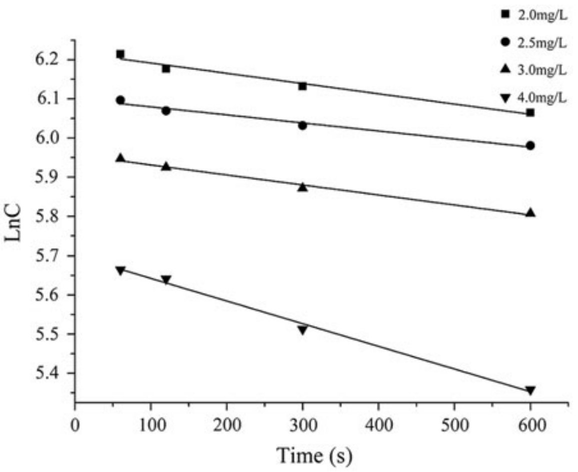

Fig. 3. Oxidation fit curves of DMTS under different K_2FeO_4 dosages (60–600 s).

and Table 2). In this phase, along with increased oxidant dosage, the reaction rate and half-life fluctuated and were irregular with possible reason being K_2FeO_4's consumption, transformation and decomposition. Since most of the DMTS was removed in the first phase, therefore, a short contact oxidation time of one minute was chosen for K_2FeO_4.

3.2. Simultaneous removal of algae and DMTS by K_2FeO_4

Different dosages K_2FeO_4 (2.8, 5.6, 8.5, 11.3, 14.0 and 17.0 mg/L) were used to remove algae and odor DMTS simultaneously from simulated algal-laden water. Results illustrated that dosage served as a key influencing factor. The removal ratio of DMTS was only 44.5% when the dosage was 2.8 mg/L, while the dosage increased to 11.3 mg/L, leading to a rapid increase of up to 92.5% in the removal ratio. Subsequent increases in the dosage, however, led to a slow increase of up to 93.3%. High DMTS removal ratios showed that Fe(VI) is a powerful oxidizing agent to effectively decompose DMTS in a short contact time of 1 min. However, compared with the oxidation effect of K_2FeO_4 on single DMTS in Section 3.1, the oxidation capacity was greatly weakened due to the existence of algae and natural organics. As negative particles and electron acceptors, algae and organics can consume oxidants. Since the rapidly increasing tendency of removal turn almost constant after a dosage of 11.3 mg/L, it can be considered that for the simulated level of contaminant of algae and odor, a threshold limit value of above 11.3 mg/L may be adequate (Fig. 4).

In the same process, elimination of algae by K_2FeO_4 was superior compared with the elimination of the odor compound. For algae removal, the ratio was 87.5% when the dosage was at 2.8 mg/L. As the K_2FeO_4 dosage increased, the ratio also slowly increased to the maximum point of 92.8%. Algae were eliminated by $Fe(OH)_3$ colloids formed from Fe(III) as a reduction product of Fe(VI). $Fe(OH)_3$ acted as a coagulant, first precipitating on the algal surface and making obvious changes to algal surface properties, thereby modifying the setting characteristics and eventually removing the particles [21,22]. In addition, the turbidity caused by the inorganic particles was removed simultaneously in order to reduce the residual turbidity of effluent water. An increase in ferrate dosage decreased residual turbidity to 1.5 NTU when dosage was at 16.8 mg/L.

3.3. Elimination of algae and odor by $KMnO_4$–PFC

3.3.1. PFC coagulation

PFC is a kind of polymerized inorganic polymer flocculant that has a polyhydroxy oxide bridge and a high positive charge [23]. It is used as a coagulant in eliminating particles (e.g. algae) through charge

Table 2
Kinetics parameters of DMTS under different K_2FeO_4 dosages

	Dosage of K_2FeO_4 ($mg\cdot L^{-1}$)	Fitting equation	K_{obs} (s^{-1})	R^2	$t_{1/2}$(s)
0–30s	2.0	$1/C = -1.0 \times 10^{-5}t + 0.0016$	1.0×10^{-5}	0.9699	164
	2.5	$1/C = -1.6 \times 10^{-5}t + 0.0017$	1.6×10^{-5}	0.9577	66
	3.0	$1/C = -2.6 \times 10^{-5}t + 0.0017$	2.6×10^{-5}	0.9608	63
	4.0	$1/C = -5.3 \times 10^{-5}t + 0.0017$	5.3×10^{-5}	0.995	31
60–600s	2.0	$\mathrm{Ln}C = -2.6 \times 10^{-4}t + 6.2180$	-2.6×10^{-4}	0.9751	2,665
	2.5	$\mathrm{Ln}C = -2.0 \times 10^{-4}t + 6.1003$	-2.0×10^{-4}	0.9772	3,465
	3.0	$\mathrm{Ln}C = -2.6 \times 10^{-4}t + 5.9567$	-2.6×10^{-4}	0.9899	2,665
	4.0	$\mathrm{Ln}C = -5.8 \times 10^{-4}t + 5.7006$	-5.8×10^{-4}	0.9931	1,195

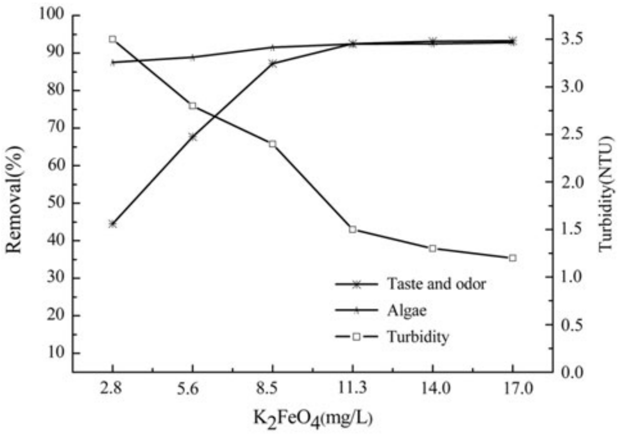

Fig. 4. Removal ratios of algae and odorous DMTS with different dosages of K_2FeO_4.

neutralization. The removal variations of algae and DMTS under different dosages of PFC are shown in Fig. 5. To compare with oxidation products of K_2FeO_4, which are both iron-derived chemicals, amounts of PFC under equivalent dosages of ferrate for PFC were analyzed.

The removal ratio of algae was 85.3% when the PFC dosage was at 7.0 mg/L (Fig. 5). As the dosage increased to 14.0 mg/L, the removal ratio slightly increased up to 90.6%. Nevertheless, after a flat period, the removal ratio decreased as the dosage increased, indicating that the stability of the algae colloids was destroyed by the Fe(III) produced in the hydrolysis process of PFC. As negative charges covering the algal surface were neutralized, more algae were removed. However, as the dosage continually increased and excess positive charges were adsorbed on the algal surface, algae colloids stabilized again, thereby leading to worser effluent water [23,24]. Similar variations were observed in the removal tendency of turbidity. From the two curves, the inflection point was identified at 14.0 mg/L. At that point, optimal removal of algae was 90.6%, whereas residual turbidity was only 0.76 NTU.

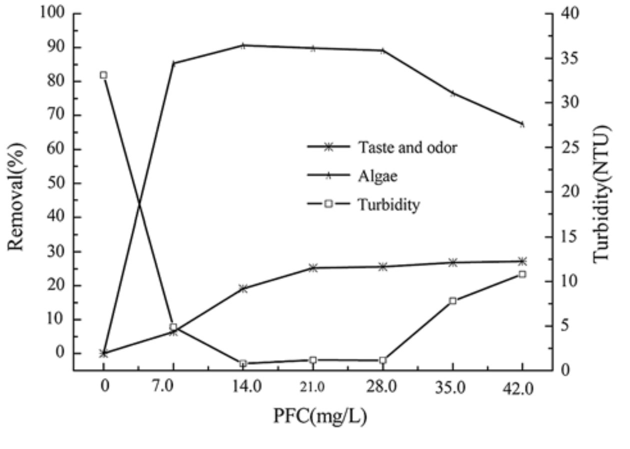

Fig. 5. Removal ratios of algae and odorous DMTS with different PFC dosages.

DMTS cannot be removed by single coagulation process. Given that it is a sulfide compound with small molecules, it can hardly be removed by coagulation [25]. But there was a significant decline of DMTS residual concentration when PFC was added at 35.0 and 42.0 mg/L, which was lost possibly due to DMTS volatilization during stirring (Fig. 5).

3.3.2. *Combined process of $KMnO_4$–PFC*

$KMnO_4$–PFC is commonly applied in treatment of algal-laden water. Thus it was selected for comparison with K_2FeO_4; the dosage of $KMnO_4$ ranged from 1.0 to 6.0 mg/L, followed by 14.0 mg/L PFC of coagulation.

Removal of DMTS and algae by $KMnO_4$– PFC through oxidation and coagulation, respectively. For algae removal, it was obvious that addition of $KMnO_4$ had enhanced removal ratio compared with single PFC process. It had a removal ratio of above 90% and a maximum of 94.8%, which was better than the rate of 90.6% obtained by using PFC alone. Pre-oxidized $KMnO_4$ causes little change on algal cell surface charges; however, $KMnO_4$ puts stress on algae and induces the release of EOM, and these EOM complexes, with MnO_2 colloids coated on algal cells, result in algae cell aggregation enhancement [26].

As oxidation dosage increased, DMTS removal ratio increased to 90.9%. $KMnO_4$ is an oxidant with an oxidation potential of +1.7 V and +0.59 V in acid and basic solutions, respectively; thus it can oxidize sulfide-type compounds [27,28]. In the experiments, the DMTS removal curve increased continuously with the inflection point, indicating that adequate amount of oxidant could not be generalized (Fig. 6).

$KMnO_4$–PFC showed fine turbidity removal effect. But as the oxidant dosage increased, the residual turbidity also increased when excess $KMnO_4$ was reduced to MnO_2 colloids. In this experiment, considering the color and residual turbidity of the effluent, more amounts of $KMnO_4$ were no longer added.

3.4. *Comparisons of K_2FeO_4 and $KMnO_4$–PFC*

Comparisons of K_2FeO_4 and $KMnO_4$–PFC were determined and different influences such as pH, contact time, and turbidity were analyzed. Agents' dosages were invariable with K_2FeO_4 and PFC

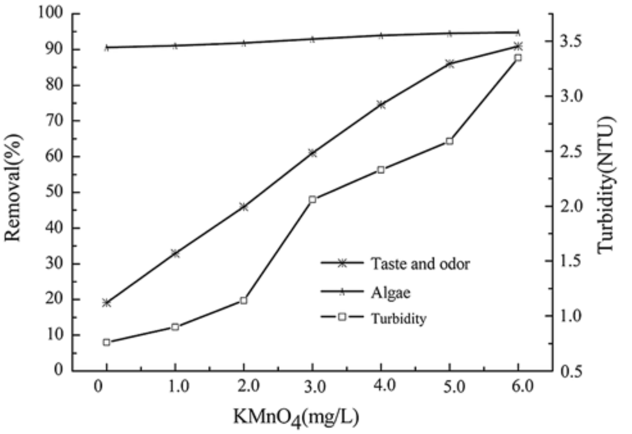

Fig. 6. Removal ratios of algae and odorous DMTS with different $KMnO_4$ dosages in $KMnO_4$–PFC process.

(equivalently measured by Fe) at 14.0 mg/L and $KMnO_4$ of 4.0 mg/L.

3.4.1. *Removal of algae and DMTS by K_2FeO_4 and $KMnO_4$–PFC*

The algae removal ratios of PFC, $KMnO_4$–PFC, and K_2FeO_4 were 90.6, 94.8, and 92.4%, respectively (Fig. 7). Algae were removed mostly by coagulation; however, such oxidants as K_2FeO_4 and $KMnO_4$ also facilitated coagulation by breaking algal cell structures or changing their surface characteristics. IOM or EOM was also released into the water when the algae and oxidants interacted; these organic matters sometimes worked as coagulation aids, enhancing algae cell aggregation [22]. From highest to lowest, the sequence of the three methods based on their algae removal ratios was $KMnO_4$–PFC > K_2FeO_4 > PFC. For residual turbidity, the sequence of the three methods was $KMnO_4$–PFC > K_2FeO_4 > PFC.

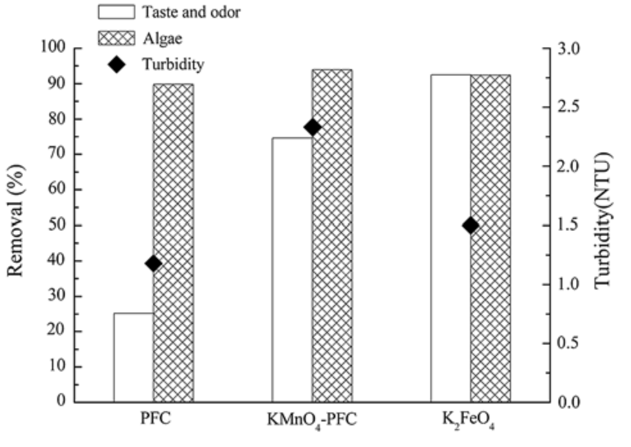

Fig. 7. Comparison of the algae and odorous DMTS removal by PFC, $KMnO_4$–PFC, and K_2FeO_4.

PFC almost had no effect in decomposing DMTS, whereas K_2FeO_4 and $KMnO_4$–PFC easily and effectively decomposed DMTS. The removal ratio of 92.5% when using K_2FeO_4 was much better than 74.6% obtained using $KMnO_4$–PFC. In natural aqueous system, massive growth of algae usually causes high pH of above 10 which is propitious to the stability of K_2FeO_4. In addition, the oxidation-reduction potential of Fe(VI) was higher than that of Mn(VII) in alkaline solutions; thus, DMTS removal by K_2FeO_4 was better than $KMnO_4$.

3.4.2. *Effect of pH on removal of algae and odorous DMTS*

The pH had little influence on the removal of algae in both methods, but had obvious influence on DMTS removal (Fig. 8). DMTS removal by $KMnO_4$ was stable under the selected pH with ratios above 70% and a maximum of 75.2%. K_2FeO_4 was severely affected compared with $KMnO_4$. The DMTS removal ratio was 35.4% when pH was at 5.2; it then reached up to 78.5% when the pH increased to 9.1, showing better effect than $KMnO_4$. This phenomenon corresponded to the characteristics of K_2FeO_4 under different pH concentrations. Acidic environments accelerated rapidly decomposition K_2FeO_4 and reduced its oxidation capacity, whereas alkaline environments decreased K_2FeO_4 oxidation potential. However, the oxidation potential of K_2FeO_4 in an alkaline environment was

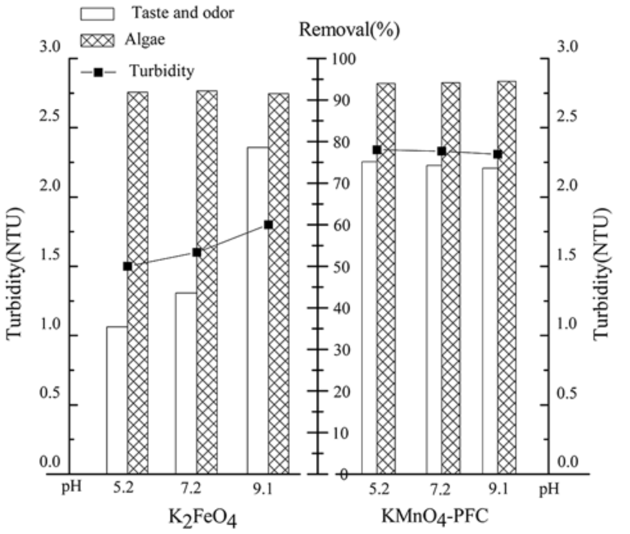

Fig. 8. Influence of pH value on the removal ratios of algae and odorous DMTS by $KMnO_4$–PFC and K_2FeO_4.

still higher than $KMnO_4$, and along with less decompositions, led to DMTS better removal [14].

Generally, during an explosive growth of algae, the pH of water turns alkaline and usually can reach a pH value of up to 10. Thus, in the current paper, all experiments were designed and carried out under alkaline circumstances. The oxidation potential of both K_2FeO_4 and $KMnO_4$ decreased as pH increased, but the oxidation power of K_2FeO_4 remained higher than $KMnO_4$. Furthermore, in the alkaline solutions, K_2FeO_4 was comparably stable than in an acidic solution. Considering both oxidation and coagulation purposes in achieving a fine control effect of both algae and DMTS, K_2FeO_4 was effective and adapted well to highly alkaline algae raw water [26].

Water turbidity treated by K_2FeO_4 was also better than $KMnO_4$–PFC under the same original turbidity. This phenomenon was possible because coagulation by the reduction product of K_2FeO_4 was better than PFC. Furthermore, the reduction product of $KMnO_4$, which was MnO_2 colloids, raised residual turbidity.

3.4.3. *Effect of pre-oxidation time on removal of algae and odorous DMTS*

The removal ratio of DMTS by K_2FeO_4 after 1 min of contact oxidation was 92.5%. However, the removal ratio of $KMnO_4$ after 20 min was only 79.5% (Fig. 9). Previous studies have indicated that DMTS is mainly degraded by K_2FeO_4 in the first 30 s; in the next 60–600 s, DMTS is degraded slowly. Less contact time was needed for K_2FeO_4 than $KMnO_4$ to achieve the same level of DMTS removal. For algae removal, there was little discrimination between K_2FeO_4 and $KMnO_4$ as both decreased more than 90% of the original algae. However, K_2FeO_4 was better than $KMnO_4$ in terms of efficiency.

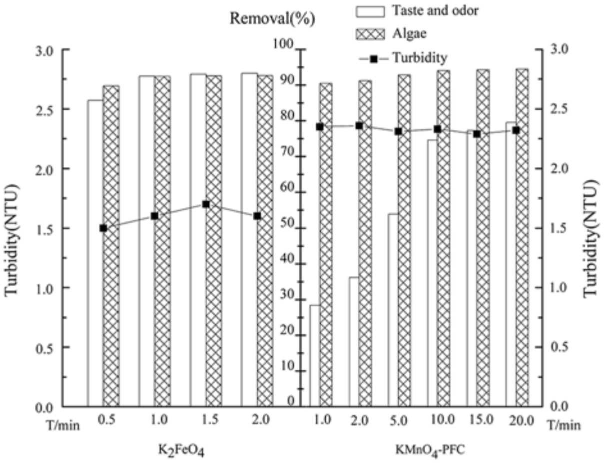

Fig. 9. Influence of pre-oxidation time on the removal ratios of algae and odorous DMTS by $KMnO_4$–PFC and K_2FeO_4.

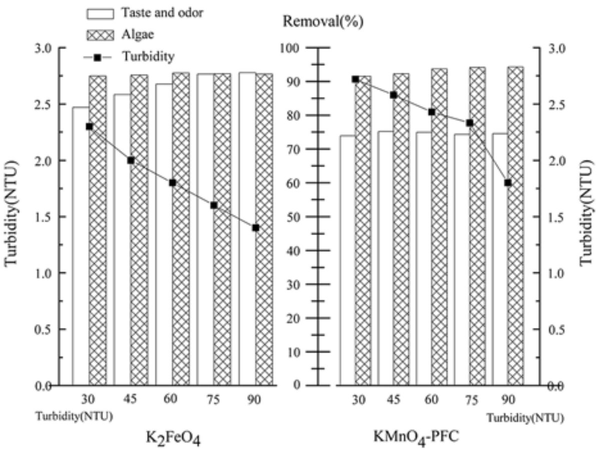

Fig. 10. Influence of the turbidity of raw water on the removal ratios of algae and odorous DMTS by $KMnO_4$–PFC and K_2FeO_4.

3.4.4. *Effect of turbidity on the removal of algae and odorous DMTS*

Turbidity almost had no influence on the removal ratio of algae and DMTS using K_2FeO_4 and $KMnO_4$–PFC (Fig. 10) because the surface of kaolin covered with negative charges, was similar to that of algae cells. In similar levels of turbidity, the removal of algae and odorous DMTS by K_2FeO_4 were better than $KMnO_4$; the residual turbidity was also lower in K_2FeO_4 compared with $KMnO_4$–PFC. Moreover, K_2FeO_4 had advantages in terms of oxidation rate, residual turbidity, and negative impact compared with $KMnO_4$–PFC.

4. Conclusions

The powerful oxidation capability of K_2FeO_4 and its coagulant reduction product results in the stable and ideal removal of algae and odorous DMTS as well as low residual turbidity. At an equivalent dosage, the removal ratio of algae is higher by K_2FeO_4 than by single coagulation with PFC. K_2FeO_4 has advantages in terms of oxidation rate, residual turbidity, and negative impact compared with $KMnO_4$. Therefore, K_2FeO_4 can be an emergency control measure for the outbreak of algae in raw water.

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