



Drinking water treatment by *in situ* generated ferrate(VI)

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ABSTRACT

The present study aims to investigate the efficiency of on-site produced ferrate(VI) for drinking water treatment as well as for the removal of micro pollutants. The study examined and validated the laboratory electrochemical production of ferrate(VI) and tested water treatment efficiency of the resulting ferrate(VI) in comparison with that of FeClSO_4 and poly-aluminium chloride (PACl). The performance of water treatment was compared by the removal efficiency of turbidity, dissolved organic carbon, microbial count and trace organic micro pollutants. Major observations were that 0.5 mg/L of ferrate(VI) as Fe was sufficient to remove most of the contaminants and achieve complete disinfection irrespective of the original microbial counts. Ferrate(VI) has considerable oxidation potential to degrade commonly occurring trace organic micropollutants (other than melamine) in the Danube River water. In contrast, FeClSO_4 or PACl cannot remove any trace micro pollutants.

Keywords: Coagulation; Disinfection; Drinking water treatment; Ferrate(VI); Removal of micro pollutants

1. Introduction

Iron commonly exists in the oxidation states of 2+ and 3+. However, under strong oxidizing and alkali conditions, iron can possess higher oxidation states (6+) such as ferrate(VI). Reduction-oxidation (redox) potential of ferrate(VI) has been estimated to be 2.2 under acidic conditions, which is the highest among the practical used water treatment chemicals.

Ferrate(VI) has multifunctional functions. When dosed into water, it plays a role of disinfection and

oxidation and it is reduced to ferric species including ferric hydroxide which can be served as coagulant/flocculent [1], and then it is considered to be a green chemical for water treatment.

Only a few researches have been reported to use ferrate(VI) for drinking water treatment [e.g. 2]. In contrast, several application cases of wastewater treatment using ferrate(VI) have been reported [3,4], where the multifunctional functions of ferrate(VI) were demonstrated. More suspended solids, colour (as Vis400-abs) and COD were removed and more bacteria was reduced by ferrate(VI) in comparison with that removed by aluminium sulphate or ferric

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sulphate. Also, for a very low dose (<0.2 mg Fe/L), ferrate(VI) can effectively remove phosphate from raw sewage [5,6]. De Luka et al. [7] reported that ferrate (VI) can be used to replace several chemicals for the control of sewage sludge odour, mainly caused by ammonia and sulphides through the formation of precipitates with iron compounds. The ferrate(VI) oxidation process also significantly improves the biodegradability of alachlor and acetanilide herbicide in wastewater [7].

Moreover, the oxidation capability of ferrate(VI) to degrade various contaminants in water and waste water has been demonstrated [8]. These pollutants include ammonia, cyanide, thiocyanate and sulfide [9], iodide [10], arsenic [11], carbohydrates [12], alcohols, toluene and cycloalkanes, ketones and hydroquinones, phenols, aminobenzene, estrogens [13], Bisphenol A, pharmaceuticals [14], triclosan and benzotriazoles [15].

A number of studies on the ferrate(VI) have focused on waste water treatment and environmental remediation but a few of published studies reported using online generated ferrate(VI) for drinking water treatment. This study thus aims to investigate the efficiency of on-site produced ferrate(VI) for drinking water treatment by examining the ferrate(VI)'s performance of oxidation, disinfection, coagulation as well as that of the removal of micro pollutants.

2. Materials and experimental procedures

2.1. Raw water quality

Raw water from the Danube River was collected and stored in a reservoir for a set of experiments.

Table 1 displays the quality characteristics of the test water utilized by this study.

2.2. Chemicals

For jar-test experiments ferric chloride sulphate (FeClSO_4 , $[\text{Fe}] = 185 \text{ g/L}$) was obtained from a commercial source. Four different kinds of commercially available poly aluminium chloride (PACl) with 100–160 g/L Al were used. Polyacrylamide solution was used as conventional coagulant aid with concentration of 2 g/L obtained from the large-scale plant in Langenau. Other chemicals used were of analytical grade for water quality analysis. For the micro pollutants analysis, analytical standards were purchased from Dr Ehrenstorfer (Augsburg, Germany) or Sigma–Aldrich (Steinheim, Germany). Ultra pure water was produced from deionized water using Pur-elab Ultra (Elga, Celle, Germany). Methanol and acetonitrile were of LC–MS grade and from Carl Roth (Karlsruhe, Germany). Ammonium acetate, ammonium carbonate and acetic acid were of analytical grade and from Sigma–Aldrich (Steinheim, Germany).

2.3. Online ferrate production

Ferrate(VI) production was carried out by a pre-setup electrochemical reactor system [4] consisting of four iron electrodes made of steel iron plates for both anodes and cathodes, a DC power supply (Hi-tech power controller) and a digital multi-meter (Isotech) to monitor the operating current and the temperature. Concentrated sodium hydroxide solution was used as an electrolyte. The dimensions of the reactor are 22 cm

Table 1
Quality characteristics of test raw water

Parameter		Danube River raw water	
		Average	(Min/Max)
pH value	–	8.0	(7.1/8.4)
Conductivity	$\mu\text{s/cm}$	524	(301/667)
Alkalinity	mmol/L	4.30	(3.08/5.07)
Total alkalinity	mmol/L	2.5	(1.4/3.0)
Calcium	mmol/L	2.0	(1.0/2.5)
Ammonia, NH_4^+	mg/L	0.08	(<0.01/0.25)
Total coliforms	1/100 ml	11,600	(800/130,000)
DOC	mg/L	2.8	(1.3/8.7)
UV ₂₅₄ absorbance	1/m	7.9	(2.1/19.3)
Absorbable org. halogenides (AOX)	mg/L	0.016	(<0.005/0.035)
Turbidity	FNU	16	(2/>500)
Suspended solids	mg/L	17	(Approx. 1 ... > 500)

in height, 2 cm in width and 5 cm in length. Electrodes have 99% iron and 0.1–0.12% of carbon content, with thickness of 2 mm. Reactor has maximum capacity of 75 mL for the electrolyte. Fifty millilitres of sodium hydroxide was used for each preparation to avoid the fluids overflow caused by H₂ gas bubble evaporation. Ferrate(VI) samples were collected at regular time intervals with standard pipette for the measurement of its concentrations. Cleaning of anodes and cathodes was required due to the formation of ferric oxide salts especially during higher current density. These layers were scrapped out by tissue papers and the electrodes were stored in dilute hydrochloric acid (<1%) to avoid the rust formation and to keep electrode active for further experiments. The resulting ferrate(VI) was measured by standard absorbance method at 510 nm with a molar absorptivity of 1,100 L/mol cm. To dilute the concentrated stock solution produced during electrolysis, 4M sodium hydroxide was used.

2.4. Jar test procedures

Standard jar test procedures and a scale down jar test method were adapted to evaluate overall coagulation and disinfection performance. To neutralize the pH in most of the jar tests, 5 M sulphuric acid was used. The supernatant was collected in fresh bottles and stored at 4°C for water quality analysis. All jar tests were duplicated and the resulting samples were used to measure the various water quality parameters. Detailed two jar test methods are listed in Table 2.

2.5. Analytical methods for the general water quality measurement

Standard methods were used in the measurement of turbidity, UV-254 absorbance, pH, DOC, residual

iron, aluminium, sodium and bacterial count presented in the duplicated samples as stated above. Results were presented in average with the standard deviation of <±5%. Supernatant was filtered with 0.45 µm glass fibre filter and measured at 254 nm in UV range by a spectrophotometer (Perkin Elmer). Similar filtered samples were used for the measurement of the dissolved organic carbon by HACH IL 500-TOC analyser. Unfiltered samples were used to measure turbidity by Dr Lange meter at 800 nm with units measured as Formazin Nephelometric Unit (FNU). Dionex *coli* count method was used to measure number concentrations of total coliforms and *E. coli* present in 100 ml of water sample. Samples were also filtered by the microfiber (0.45 µm) in order to measure residual concentrations of iron, aluminium, sodium, magnesium and calcium by the inductively coupled plasma mass spectrometry.

2.6. Analytical methods for the micro pollutants

The concentration of following compounds were analysed due to their regular occurrence in water samples, including melamine, acesulfame, diatrizoic acid, iopamidol, iohexol, iomeprol, iopromide, sulfamethoxazol and diclofenac. The determination of the selected organic micro pollutants was performed by direct injection of samples into high performance liquid chromatography tandem mass spectrometry (HPLC-MS/MS), (Agilent, 1100 Series and 1200 Series, respectively, Waldbronn, Germany). The triple quadrupole mass spectrometer (AB Sciex API 4000 Qtrap) and (API 5000) (Darmstadt, Germany), respectively, were applied as mass analysers. Quantification of the analytes was performed by external calibration, whereas matrix effects were checked for each sample by the analysis of an additional spiked sample. The most sensitive mass transition per analyte was used

Table 2
Two jar test methods

	Stage	Description	Stirrer speed in rpm	Time in s
Standard method	1	Fast mixing (coagulant aid addition)	300	60
	2	Slow mixing	35	1,200
	3	Sedimentation	0	1,800
Scaled down method	1	Fast mixing (coagulant aid addition)	220	16
	2	Microflocculation	140	82
	3	Microflocculation ripening	100	305
	4	Coagulant aid addition	150	93
	5	Macroflocculation	30	171
	6	Macroflocculation ripening	10	245
	7	Sedimentation	0	1,800

for quantification and the second mass transition was used to verify the identity of the analyte. For measuring various compounds, three methods were used and the detailed information on these methods is listed in Table 3. The mass spectrometric details for the selective detection of each analyte are given in Table 4.

3. Results and discussion

3.1. Comparative coagulation performance of ferrate(VI) vs. other coagulants

Figs. 1 and 2 show the percentage removals of turbidity and UV-254 absorbance by ferrate(VI) and ferric chloride sulphate using the standard and scaled-down coagulation methods. Results demonstrated that there were no great difference for the performance achieved by two jar test procedures; both standard and scaled-down methods produced close results and then, future results presented are those gained from the standard jar test procedures. Secondly, ferrate(VI) with

pH adjustment was superior to ferric chloride sulphate in removing turbidity and reducing UV-254 absorbance.

Fig. 3 compares DOC removal by three coagulants. For the dose range of 0.2–1.0 mg Fe/L, 10–15% more DOC can be removed by the ferrate(VI) in comparison with that by the other two coagulants.

Promising performance of ferrate(VI) was also shown in its extremely high reduction of total *coliform* (Fig. 4). For a dose between 0.3 and 1.0 mg Fe/L, ferrate(VI) can achieve 99.9–99.99% reduction while other two coagulants can only reduce 5–30% for the same dose range (0.3–1 mg/L as Fe or Al).

This study aims to examine the performance of coagulants at lower doses (e.g. <1 mg/L as Fe). The outstanding performance of ferrate(VI) in drinking water treatment is consistent with that reported in the previous studies, where, ferrate(VI) performed better than other coagulants for reducing the turbidity, UV254-abs, DOC and total *coliform* [2]. Achieving good performance at lower doses should attract interest from water industries as this first will reduce the chemical consumptions and secondly reduce the sludge production and ultimately, save the operating cost.

Table 3
Chromatographic details and ionization details

HPLC	Analytical runs		
	Melamine	Acesulfame	Diatrizoic acid, Iopamidol, Iohexol, Iomeprol, Iopromide, Sulfamethoxazol, Diclofenac
Instrument	1100 Series	1200 Series	1200 Series
Analytical column	Restek Ultra Aqueous C18, 5 μ m, 250 \times 4.6 mm	Zorbax Eclipse XDB-C18, 1.8 μ m, 50 \times 4.6 mm	Zorbax Eclipse XDB-C18, 1.8 μ m, 50 \times 4.6 mm
Eluent A	Water, 5 mmol/L ammonium acetate	Water, 2 mmol/L ammonium carbonate	Water, 1 mL/L acetic acid
Eluent B	Methanol, 5 mmol/L ammonium acetate	Methanol, 2 mmol/L ammonium carbonate	Methanol, 1 mL/L acetic acid
Flow rate	0.8 mL/min	0.5 mL/min	0.6 mL/min
Gradient	0–3 min 5% B, 3–10 min 5–90% B, 10–13 min 90% B, 13–13.1 min 90–5% B, 13.1–19 min 5% B	0–7 min 2–75% B, 7–9 min 75% B, 9–9.1 min 75–2% B, 9.1–15 min 2% B	0–7 min 5–20% B, 7–11 min 20–90% B, 11–15 min 90% B, 15–15.1 min 90–5% B, 15.1–20 min 5% B
Temperature	25 $^{\circ}$ C	25 $^{\circ}$ C	25 $^{\circ}$ C
Injection volume	50 μ L	25 μ L	100 μ L
MS Ion source			
Instrument	API 4000 Qtrap	API 5000	API 5000
Mode	ESI+	ESI-	ESI+
Curtain gas	25 psi	30 psi	25 psi
Temperature	550 $^{\circ}$ C	650 $^{\circ}$ C	600 $^{\circ}$ C
Ion spray voltage	5,000 V	–4,200 V	5,300 V
Gas 1	60 psi	40 psi	60 psi
Gas 2	50 psi	50 psi	60 psi

Table 4
Mass transitions for selected micro pollutants

Analyte	RT [min]	Precursor	Product ion	Dwell time [ms]	Declustering potential [V]	Collision energy [V]	Collision cell exit potential [V]
Melamine	9.6	127	85	150	91	25	5
		127	68	150	91	43	5
Acesulfame	2.8	162	82	100	-35	-20	-11
		162	78	100	-35	-42	-1
Iopamidol	2.9	778	559	150	96	33	14
		778	687	150	96	31	32
Iohexol	5.1	822	804	150	101	29	42
		822	302	150	101	81	18
Iomeprol	5.2	778	405	150	106	63	24
		778	532	150	106	41	30
Diatrizoic acid	4.1	615	361	150	101	27	18
		615	233	150	101	55	12
Iopromide	7.4/7.8	792	573	150	111	31	16
		792	300	150	111	77	14
Sulfamethoxazole	10.3	254	156	25	41	21	15
		254	108	25	41	30	15
Diclofenac	12.8	296	250	25	51	21	14
		296	214	25	51	45	10

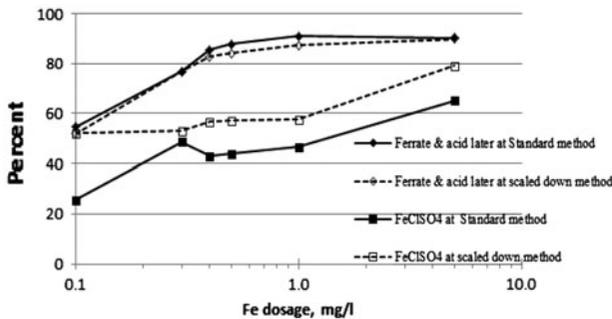


Fig. 1. Turbidity removal vs. two jar test procedures and iron doses.

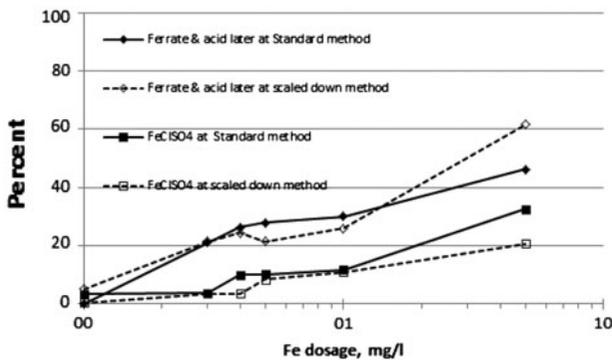


Fig. 2. UV-254 absorbance removal vs. two jar test procedures and iron doses.

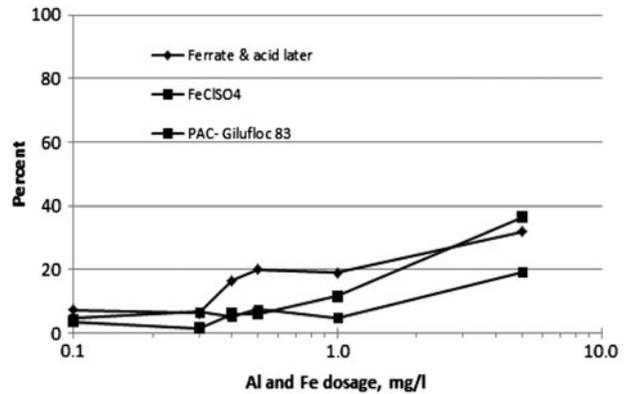
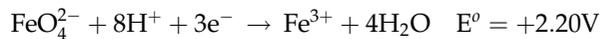


Fig. 3. Comparative DOC removal for three coagulants and doses.

Superior performance of ferrate(VI) can be attributed to its high redox potential and multiple functions. Under acidic conditions (see Eq. (1)), the redox potential of ferrate(VI) is the strongest ($E^0 = +2.20$ V) among all oxidants/disinfectants used for water and wastewater treatment. Even under neutral conditions (Eq. (2)), the redox potential of ferrate ($E^0 = +0.72$ V) is still greater than that of permanganate (MnO_4^-) which is a strong oxidant.



(1)

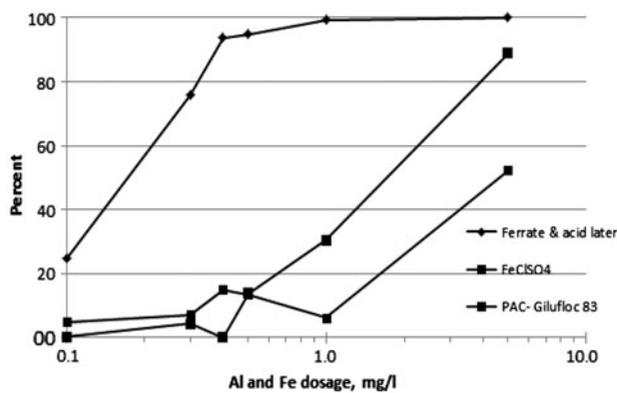
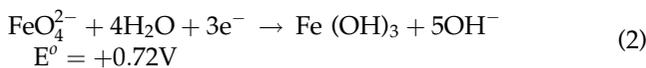


Fig. 4. Comparative total coliform reduction by three coagulants and doses.



Therefore, very low doses of ferrate(VI) (e.g. 0.2 mg/L as Fe) can perform superiorly to other coagulants. High redox potential alone could not explain the superior performance of the ferrate(VI), there must be other reasons behind such phenomena. First, oxidation by ferrate(VI) could change the surface property of suspended solids and organic pollutants making them readily removable through coagulation and precipitation. Secondly, the byproducts of the ferrate(VI) oxidation are ferric ion or ferric hydroxide (see Eqs. (1) and (2)) which are basic coagulant resources. Therefore, ferrate(VI) also performs coagulation after it degrades the organic matter and micro organisms.

3.2. Degradation of organic micro pollutants

As stated previously that ferrate(VI) has strong redox potential and then possesses capability to degrade organic micro pollutants such as pesticides and X-ray contrast medium. This adds an additional advantage of using ferrate(VI) in drinking water treatment when it performs both coagulation and oxidation to degrade organic pollutants.

The concentrations of micro pollutants for the Danube River raw water were measured and the data for the year of 2013 are presented in Table 5. The sampling site, Leipheim, was approximately 13 km downstream of Ulm. The degradation of micro pollutants by the ferrate(VI) can be seen in Fig. 5, where three micropollutants, diclofenac, sulfamethoxazol and iopromide, can be completely removed by ferrate(VI) for most operating conditions. Four other compounds, iomeprol, iohexol, iopamidol and amidotrizowsaure,

Table 5
Concentrations of micro pollutants in the raw water which were used for this study

	Diatrizoic acid ($\mu\text{g/L}$)	Iopamidol	Iohexol	Iomeprol	Iopromide	Melamine	Acesulfame	Sulfa-methoxazole	Diclofenac
Min	<0.025	<0.025	<0.025	<0.025	<0.025	0.05	0.15	<0.025	<0.025
Max	0.12	0.98	0.09	0.38	0.12	1.6	0.79	0.25	0.07
Average	0.05	0.12	0.04	0.19	0.04	0.80	0.47	0.03	<0.025
Std. dev.	0.03	0.16	0.02	0.10	0.02	0.57	0.24	n.a.	n.a.
N	38	38	38	38	38	5	5	38	39

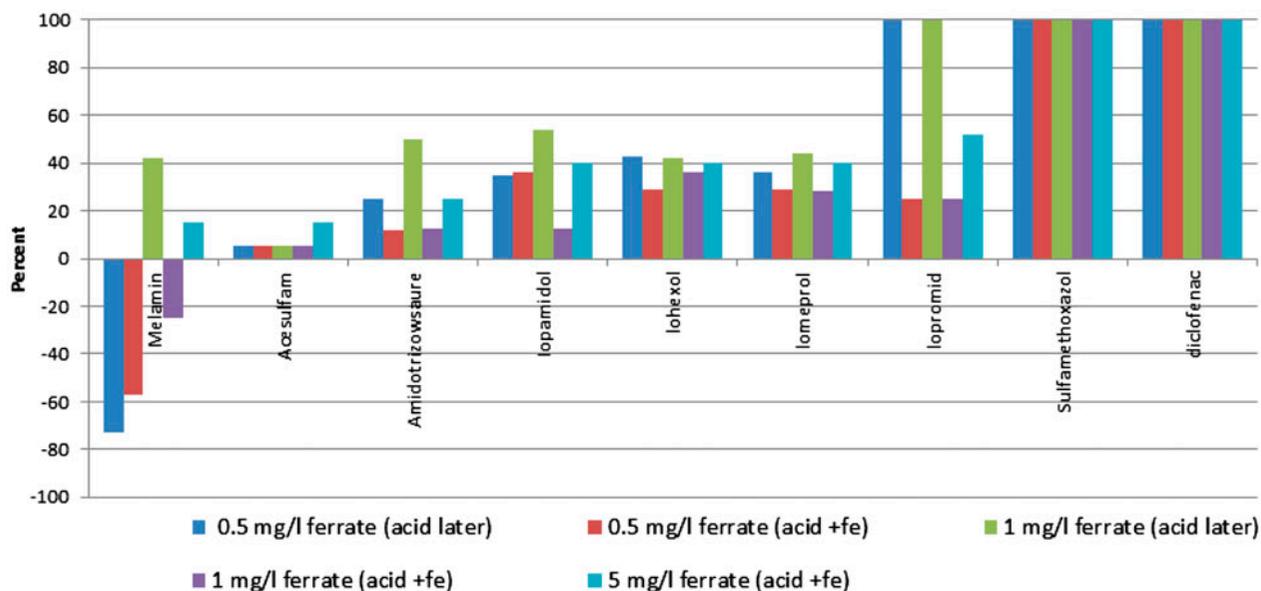


Fig. 5. Degradation or removal of various micro pollutants from drinking water sources.

Table 6

Performance and reaction rate constants of ozonation in the treatment of micro pollutants

Pollutant	Treatment performance	Reaction rate constants of kinetics (M^{-1}/s)	Ref.
Macrolide & sulphonamide antibiotics, estrogens (EE2), diclofenac, naproxen, indomethacin and ICM	O_3 dose >2 mg/L, except ICM, over 90% of pollutants removed	pH 7, 20°C, for diclofenac, EE2, sulfamethoxazole: $k_{(O_3)} = (1-3) \times 10^6$; carbamazepine, naproxen: $(2-3) \times 10^5$; roxithromycin: 7×10^4 ; bezafibrate: 590 ± 50 ; clofibric acid, diazepam, ibuprofen, iopromide: <20, pH 7, 25°C, $vk_{(\bullet OH)} = (3.3-9.8) \times 10^9$	[18]
Caffeine, progesterone, medroxyprogesterone, norethindrone, and levonorgestrel	CT value 2 mg min/L, >80% of caffeine, PPCPs and EDCs removal	pH 8.1, 20°C, $k_{(O_3)} = 2,215 \pm 76$ (norethindrone); $1,427 \pm 62$ (levonorgestrel); $(558 \pm 9)-(650 \pm 22)$ (others)	[19]
Sulphonamides	pH 7.0, $[O_3] = 3.2$ mg/L, more than 90% removed	pH 2, 22°C, $k_{(O_3)} > 2 \times 10^4$	[20]
Triclosan	pH 7, complete ozonation triclosan: ozone = 1:5 in molar ratio	n.a.	[21]

can be removed by 15–50%. Acesulfame can be removed by 5–15%, and melamin can only be removed for some conditions by 15–40%. In general, for a dose of 0.5–1.0 mg/L as Fe with pH adjustment later, ferrate(VI) has comparable oxidation effects as ozone to reduce concentrations of most micro pollutants studied.

The removal efficiency of diclofenac, sulfamethoxazol and iopromide was the highest among all compounds listed, while acesulfame and melamin were relatively difficult to be removed by ferrate(VI).

Especially melamin was not degraded by ferrate(VI) for some conditions. The selective removal of micro pollutants by ferrate(VI) as demonstrated in this study is consistent with that from others [e.g. 16]. Ferrate(VI) can degrade electron-rich organic moieties [16,17] of micro pollutants such as diclofenac and then achieve high percentage removals but difficult to oxidize others such as melamin. This is also the case when using ozonation. Table 6 shows selected reaction rate constants of ozone in the treatment of a number of micro pollutants in wastewater; ozone can selectively oxidize

micro pollutants effectively, with higher rate constants up to 10^2 – 10^9 M^{-1}/s .

Nevertheless, in drinking water treatment, ferrate(VI) should achieve the removal of some emerging micro pollutants as well as enhance the overall treatment performance such as lowering concentrations of suspended solids and DOC and killing *E. coli* and total coliform [2].

3.3. Assessment of the toxicity of ferrate(vi) treated water

When considering the application of ferrate(VI) for water treatment it is important to determine whether the ferrate(VI) treated water contains any toxic or mutagenic substances. The Ames test has been applied to ferrate(VI) treated water and a preliminary study demonstrated negative results [22], suggesting that ferrate(VI) does not produce mutagenic by-products for the study conditions. Moreover, in a recent study [23], the toxicity of the ferrate(VI) treated waste water effluent was assessed and compared with that of raw wastewater effluent by the zebrafish embryos model. Raw wastewater effluents possessed toxicity as the evidence of reducing the zebrafishes hatching rate but ferrate(VI) treated effluents had no adverse effects.

However, a number of other studies have reported the potential formation of harmful by products [24,25]. Adsorbable organic halogens (AOX) in the ferrate(VI) treated wastewater effluents were observed although the AOX concentration rise was lower than that in the chlorinated effluents [26]. We are currently studying the formation potential of harmful oxidation products during ferrate(VI) treatment and should report the study results accordingly.

Initial concentrations of micro pollutants in the raw water samples can be seen in Table 5.

4. Conclusions and future work

The study discovers that

- Ferrate(VI) at a dose of 0.5 mg/L as Fe will be sufficient to remove most of pollutants for the study conditions.
- Complete disinfection can be achieved by ferrate(VI) for the dosage no more than 0.5 mg/L irrespective of the original microbial count.
- Ferrate(VI) has considerable oxidation potential to degrade commonly occurring organic micro pollutant in Danube river water. In contrast, $FeClSO_4$ or PACl can not remove any such pollutants. This will be beneficial to use ferrate(VI) as a coagulant.

- The selective removal of micro pollutants by ferrate(VI) is demonstrated in this study which is in agreement with that from other studies.
- Ferrate(VI) has potential to replace conventional coagulants and disinfection reagents for drinking water treatment. Future work is needed to study possible toxicities of the water treated by ferrate(VI), and to investigate approaches to reduce and make the ferrate(VI) operating cost to be less than that of conventional coagulants.

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