



Settleability and characteristics of ferrate(VI)-induced particles in advanced wastewater treatment



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ABSTRACT

Ferrate(VI) as an emerging water treatment agent has recently recaptured interests for advanced wastewater treatment. A large number of studies were published to report ferrate(VI)-driven oxidation for various water contaminants. In contrast, very few efforts were made to characterize ferrate(VI) resultant particles in water and wastewater. In this study, jar tests were performed to examine the settleability and characteristics of ferrate(VI)-induced iron oxide particles, particularly the non-settable fraction of these particles, after ferrate(VI) reduction in a biologically treated municipal wastewater. The particle settleability was evaluated through the measurement of turbidity and particulate iron concentration in the supernatant with the settling time. Results showed that a majority of ferrate(VI)-induced iron oxide aggregates remained suspended and caused an increased turbidity. For example, at a Fe(VI) dose of 5.0 mg/L and pH 7.50, 82% of the added iron remained in the supernatant and the turbidity was 8.97 NTU against the untreated sample turbidity (2.33 NTU) after 72-h settling. The poor settling property of these particles suggested that coagulation and flocculation did not perform well in the ferrate(VI) treatment. Particle size analysis and transmission electron microscopy (TEM) revealed that nano-scale particles were produced after ferrate(VI) decomposition, and gradually aggregated to form micro-scale larger particles in the secondary effluent. Zeta potentials of the non-settable ferrate(VI) resultant aggregates varied between -7.36 and -8.01 mV at pH 7.50 during the 72-h settling. The negative surface charges made the aggregates to be relatively stable in the wastewater matrix.

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1. Introduction

Ferrate(VI), i.e. the oxyanion FeO_4^{2-} containing iron in +6 oxidation state, is recognized as an environmentally friendly water treatment agent due to its high oxidizing potential, selectivity, the formation of non-toxic final products (i.e., Fe(III)), and little production of undesirable disinfection byproducts (DBPs) (Sharma, 2002; Jiang, 2007; Lee et al., 2004). Over the past decades, it has been intensively studied as an oxidant or disinfectant for potable water treatment or tertiary wastewater treatment. During advanced wastewater treatment, ferrate(VI) is capable of removing micro-pollutants and precipitating phosphorus from biologically treated municipal wastewater (Lee et al., 2009). It is well acknowledged that multiple treatment mechanisms, including

oxidation/disinfection, coagulation, adsorption and precipitation, may simultaneously occur during ferrate(VI) application (Lee et al., 2004). A majority of previous studies focus on its oxidative/disinfection capability for removal of various contaminants such as color and odor compounds (Sharma, 2002), natural organic matters (Song et al., 2016), regulated toxic organic and inorganic species (Waite and Gilbert, 1978; Costarramone et al., 2004; Fan et al., 2002; Lee et al., 2003; Lim and Kim, 2010; Pucek et al., 2013; Sharma, 2010; Lee and von Gunten, 2010), emerging pollutants (e.g. microcystins) (Anquandah et al., 2013, 2011), as well as pathogenic bacteria and viruses (Jessen et al., 2008; Jiang et al., 2007, 2006; Hu et al., 2012; Schink and Waite, 1980). In contrast, very few efforts were made to appreciate ferrate(VI)-driven coagulation/flocculation behaviors or investigate ferrate(VI)-induced iron oxide particles.

Once electrons are gained in water, ferrate(VI) is reduced to the intermediate high valence iron species (i.e., Fe(V) and Fe(IV)) and eventually to the stable Fe(III) (Jiang, 2007; Sharma et al., 2008; Jiang and Lloyd, 2002; Carr et al., 1985; Carr, 2008; Lee et al., 2014). Accompanied with the ferrate(VI) reduction, Fe(III) is

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produced at an equimolar amount. The *in-situ* formation of Fe(III) is expected to cause coagulation and ensuing flocculation for removal of certain water pollutants, such as suspended particles and algae. Several studies attempted to characterize iron oxide particles produced from ferrate(VI) reduction, and compared them with Fe(III)-induced iron oxide particles (Prucek et al., 2013; Graham et al., 2010; Tien and Graham, 2011; Goodwill et al., 2015; Ma and Liu, 2002; Prucek et al., 2015). Graham et al. (2010) applied a photometric dispersion analyzer (PDA) instrument to study the flocs in a ferrate(VI)-humic acid solution system. PDA is an instrument for observing rapidly changing particle suspensions with an optical technique that analyses the light transmitted through a flowing suspension (Graham et al., 2010). They found that ferrate achieved the comparable, or better, floc formation to ferric chloride but less dissolved organic carbon (DOC) removal. Tien and Graham (2011) introduced kaolin powders to the ferrate(VI)-humic acid water system to further understand the effect of a disperse phase on the floc formation. They reported that the magnitude of floc formation with ferrate was inferior to that with ferric chloride, and the rate of floc growth with ferrate was slower than that with ferric chloride. Ma and Liu (2002) applied ferrate(VI) at a low dose (0.5–1.0 mg/L Fe) to treat three natural waters before aluminum sulfate coagulation, and found that ferrate(VI) pre-oxidation enhanced the aluminum salt coagulation, particularly in the organic-rich water. Goodwill et al. (2015) compared ferrate(VI) and ferric resultant particles produced in deionized carbonate or phosphate buffered laboratory grade water and reservoir water at pH 6.2. Although the ferrate(VI) and ferric resultant particles had similar surface charges at different pH levels, their size distributions were different and the ferrate(VI) resultant particles appeared smoother and more granular. X-ray photoelectron spectroscopy (XPS) analysis suggested that the ferrate(VI) resultant particles comprised Fe₂O₃ that was not detected in ferric chloride resultant particles. In natural water, ferrate(VI) addition produced more nanoparticles with negative surface charges than ferric addition, resulting in a stable colloidal suspension. Therefore, they questioned whether ferrate(VI) may ultimately serve the anticipated dual-role (i.e., oxidation and coagulation) in drinking water treatment, and suggested that application of ferrate(VI) pre-oxidation in water treatment possibly required an additional coagulation step. Furthermore, Prucek et al. (2013, 2015) found that ferrate(VI) decay yielded iron oxide particles with a core (γ -Fe₂O₃)-shell (γ -FeOOH) structure. Unfortunately, these aforementioned studies did not determine the settling properties of these particles or characterize the non-settable fraction of these particles, which can significantly impact the following treatment.

The objective of this study was to examine the settleability and characteristics of ferrate(VI)-induced iron oxide particles produced from ferrate(VI) reduction in a biologically treated municipal wastewater. The term “coagulation” is defined as the addition of a chemical coagulant to water with the objective of destabilizing particles so they aggregate or form a precipitate that will sweep particles from solution or adsorb dissolved constituents (Crittenden et al., 2012). And the term “flocculation” is the aggregation of destabilized particles and precipitation products formed by the addition of coagulants into larger particles (i.e., flocs), which are easier to remove from water than the original particles (Crittenden et al., 2012). In the most cases, these flocs are removed by the inexpensive and commonly used gravity sedimentation (Tchobanoglous et al., 2014). Therefore, the information regarding the settleability of ferrate(VI)-induced particles is essential to understand the performance of coagulation and ensuing flocculation during ferrate(VI) application. In this study, jar tests with ferrate(VI) were carried out under advanced wastewater treatment relevant conditions. After ferrate(VI) decay, ferrate(VI)-induced

particles in the supernatant were quantified over time through the measurement of turbidity and suspended particulate iron concentrations to determine the settleability of these particles. These ferrate(VI)-induced iron oxide particles were compared with the particles produced from Fe(III) salt addition in the control tests. Results would reveal how coagulation and ensuing flocculation behaved in the ferrate(VI) treatment case. Special attention was paid to characterization of non-settable ferrate(VI)-induced particles.

2. Material and method

Reagents and water source. All the reagents used were at least analytical grade, except as noted. Potassium ferrate (K₂FeO₄) (>96%) was purchased from Sigma–Aldrich. Biologically treated municipal wastewater was a secondary effluent collected from a secondary clarifier prior to disinfection at a local municipal wastewater treatment plant (New Jersey, USA) that applies an activated sludge treatment. Once collected, the sample was delivered to Montclair State University’s water treatment laboratory and stored at 4 °C in a refrigerator until use. Basic water quality parameters of the sample are shown in Table 1. A concentrated ferrate(VI) stock solution (200 mg/L as Fe) was prepared by dissolving a certain amount of K₂FeO₄ in distilled water. Ferrate(VI) concentration in the stock solution was confirmed with the ABTS method (Lee et al., 2005). A concentrated (1,000 mg/L as Fe) ferric stock solution was prepared by dissolving an appropriate amount of ferric chloride salt in distilled water. These stock solutions were prepared immediately before the treatment tests.

Tests for ferrate(VI) treatment and particle formation. Ferrate(VI) treatment tests were performed in 2 L square beakers with 2,000 mL secondary effluent on a four paddle programmable jar tester (Phipps & Bird - 7790–950). The treatment was initiated through the addition of an aliquot of K₂FeO₄ from the stock solution to the secondary effluent. Within the first minute, the solution was rapidly mixed at a velocity gradient of 241 s⁻¹ to completely disperse the added ferrate(VI). In the following 29 min, the solution was gently stirred at a velocity gradient of 21 s⁻¹ for the growth of flocs. The initial solution pH was adjusted to 7.50 with 1 M NaOH or 0.5 M H₂SO₄ solution. During the treatment, pH was manually maintained within 7.50 ± 0.10. At designated sampling times, residual ferrate(VI) concentrations were measured. Control tests for one-step ferrate(IV) addition were performed at identical conditions except that ferric chloride (FeCl₃), rather than K₂FeO₄, was added. In the control tests to examine whether a slow Fe(III) release affected the resultant particle formation, the FeCl₃ stock solution was stepwise added to approximate the accumulated Fe(III) release pattern in the ferrate(VI) treatment tests.

Particle characterization. Gravity sedimentation began after the gentle mixing was stopped. At each designated sampling time (0 min, 10 min, 30 min, 1 h, 2 h, 3 h, 6 h, 24 h, 48 h, and 72 h), 30 mL sample was collected through a sampling port on the beaker sidewall. Turbidity, particle size, zeta potential, and total iron in the

Table 1
Basic water quality of the untreated municipal biologically treated wastewater.

Parameters	Value
pH	7.49
Turbidity (NTU)	2.33
TOC (mg/L)	6.98
COD (mg/L)	31.5
UV ₂₅₄ (cm ⁻¹)	0.125
Total phosphorus (mg/L as P)	2.23

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