The role of ferrate(VI) in the remediation of emerging micro pollutants

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Abstract

The adverse effects of micro pollutant residues in water on the environment and human health could take place under a very low concentration range; from several μg L⁻¹ to ng L⁻¹. Since there is no efficient unit process to remove these pollutants, efficient technologies are sought to treat them. Ferrate (VI) exhibits high oxidation/reduction potentials and has many advantages because of its dual functions of oxidation and coagulation. Removal of micro pollutants by ferrate(VI) was pH dependant and this was in coordinate to the chemical/physical properties of the pollutants and ferrate(VI) speciation. Promising performance of ferrate(VI) in the treatment of real waste water was observed. It is important to determine whether the ferrate(VI) treated water contains any toxic or mutagenic substances as this should relieve public health concerns when a new chemical is employed for water treatment. The toxicity studies on the ferrate(VI) treated effluent were carried out initially via Ames tests and recently via zebrafish embryos tests conducted at author’s group. These results suggest that ferrate(VI) reagents do not produce mutagenic by-products for study conditions. However, a recent other study showed the formation of adsorbable organic haloids (AOX) as by-products in the ferrate(VI) treated waste water effluents. Obviously, more researches are needed to investigate the formation potential of harmful by-products during ferrate(VI) treatment. Other future work are suggested in order to implement ferrate(VI) into full-scale water treatment and other environmental remediation requirements.

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1. Emerging micro-pollutants in water and wastewater

Pharmaceutical and personal care products (PPCPs) and endocrine disrupting chemicals (EDCs) are classified as emerging micro-pollutants, which have been a significant issue of environmental and public health concern because they may be significant adverse environmental and human health effects although the occurrence of these pollutants in the environment is usually in a very low concentration range; from μg L⁻¹ to ng L⁻¹.

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Pharmaceuticals such as antibiotics, anti-inflammatory drugs, β-blockers and X-ray contrast media are widely used; these pharmaceuticals and their metabolites cannot be fully utilized by human beings or animals and are inevitably emitted into the waters by excretion [1-4] and/or through the discharge of industry effluents and hospitals waste waters [5, 6]. Results of toxicology studies have revealed that some pharmaceuticals are suspected to have direct toxicity to certain aquatic organisms [7-9] and they could accumulate slowly, and finally lead to irreversible change on wildlife and human beings [10]. The adverse environmental and human health effects could take place under a very low concentration range; from several μg L⁻¹ to ng L⁻¹.

Endocrine disrupting chemicals (EDCs) are defined as the natural and/or synthetic compounds which would affect endocrine systems of fishes and other aquatic animals. Since the middle of last decade, a variety of adverse effects of EDCs on the endocrine systems of animals have been observed [11, 12]. These effects may be cumulative, possibly will only appear in subsequent generations, and then the resulting effects may be irreversible, threatening the human's sustainable development. Most EDCs are synthetic organic chemicals being introduced to the environment by anthropogenic inputs, (e.g., bisphenol A) but they can also be naturally generated estrogenic hormones, e.g., estrone (E1) and 17β-estradiol (E2), and therefore are ubiquitous in aquatic environments receiving wastewater effluents.

Personal care products (PCPs) represent a large group of compounds which include non-prescription and prescription pharmaceuticals for human and veterinary use, and the active and inert ingredients for personal care purposes. Examples of PCPs include analgesics, lipid regulators, synthetic hormones, steroids, fragrances, sun screens, shampoos and cosmetics. Most PCPs, in their original or biologically altered form, are discharged into wastewater and make their way to wastewater treatment plants. Possible fates of PCPs and their metabolites within a wastewater treatment plant are: 1) mineralization to CO2 and water; 2) retention to the solids portion (sludge/biosolids) if the compound entering the plant or the product of biologically mediated transformation is lipophilic; 3) release to the receiving water either as the original compound or as a degradation product.

The presence of emerging micro pollutants and their potential toxicity are challenge to the global water industries as there is no unit process specifically designed to remove these pollutants; activated sludge and secondary sedimentation in most wastewater treatment works (WWTWs) seems to be inefficient to eliminate them [13-17]. Thus, a number of recent studies have been carried out to explore suitable technologies to treat pharmaceutical residuals from water and wastewater. Ozonation was found to be effective to remove pharmaceuticals in municipal WWTWs [18]. Nanofiltration (NF) and reverse osmosis (RO) membrane filtration have been applied at bench, pilot and full scale [19]. Activated carbon adsorption [20] has also been proved as an efficient process to remove pharmaceuticals; addition of 5 mg L⁻¹ of powder activated carbon with a 4-h contact time removed 50% to >98% of the volatile PPCPs and 10% to >95% of the polar PPCPs [21].

2. Ferrate(VI) and its application in water and wastewater treatment and in degradation of emerging micro pollutants

A promising technique which can address the concerns on emerging micro pollutants is ferrate (VI) which exhibits many advantages because of its dual functions for the oxidation and coagulation [22-26] and it has green chemical properties [27]. Ferrate(VI) has been successfully applied into water remediation processes [28-32] and to the oxidation of carbohydrates [33] and nitrogen-containing pollutants [34]. The removal of pharmaceuticals and other micro pollutants by ferrate(VI) have been extensively studied [35-40].

2.1. General water and waste water treatment by ferrate(VI)

Ferrate(VI) can act as coagulant, disinfectant and oxidant. An early study conducted by the author [24]
demonstrated in comparison with ferric sulfate (FS), ferrate(VI) can achieve high organic matter removal as DOC (see Fig. 1).

![Graph showing DOC removal](image1)

Fig.1. Removal of DOC for drinking water treatment with various ferrate dosage compared with ferric sulfate [24].

In a study [31] using ferrate(VI) for raw sewage treatment, much smaller dose of ferrate(VI) was required, in comparison with ferric sulfate (FS, Fe$^{3+}$), to efficiently remove suspended solids (SS), chemical oxygen demand (COD), biochemical oxygen demand (BOD) and phosphorous (P). Dashed lines in Fig. 2 are low and upper limits of the required percentage removal for controlling quality parameters in post-sedimentation. The removal efficiency of SS, COD, BOD and P exceeded the high requirement by ferrate(VI) at lower doses.

![Graphs showing SS, COD, BOD, and P removal](image2)

Fig.2. Comparison Fe$^{6+}$ with Fe$^{3+}$ for (a) SS, (b) COD, (c) BOD and (d) P% removal [31].
Ferrate(VI) is efficient in inactivating *Escherichia coli*, total *coli forms* and *f2* coliphage viruses [41]. Fig. 3 shows significant fast rates of *E. coli* inactivation by ferrate(VI) with smaller *Ct* values required to achieve 4 Log inactivation at a low dose, 1.5 mg L\(^{-1}\) and in neutral pH range, 6.8 – 7.2 [42].

Ferrate(VI) has been used as an oxidant in conjunction with coagulation for algal removal [43]. The combined use of ferrate(VI) ands alum, algal removal increased significantly in comparison with that using alum alone (Fig. 4).

![Fig. 3. Inactivation level of *E. coli* (log(N0/N)) and exposure amount of *E. coli* to Fe(VI) at various pH values [42].](image1)

![Fig.4. Effect of ferrate(VI) oxidation on the removal of algae by alum [43].](image2)

Although potentials of ferrate(VI) to act as coagulant, disinfectant and oxidant for water and waste water treatment have been widely investigated, researches will still be required to assess treatment efficiency and cost effectiveness considering simultaneously removing turbidity, dissolved organic carbon, particles and lowering residual iron, microbial activity in drinking water treatment and the removal of SS, COD, BOD, phosphorous and micro pollutants in waste water treatment.

The ferrate(VI) efficiency to remove 68 selected endocrine disrupting chemicals (EDCs) and pharmaceuticals and personal care products (PPCPs) spiked in a waste water matrix were studied in two wastewater treatment plants (WWTPs) [37]. Thirty-one target EDCs and PPCPs were detected in the effluents of the two WWTPs with concentrations ranging from 0.2 to 1156 ng L\(^{-1}\). Ferrate (VI) treatment resulted in selective oxidation of electron rich organic moieties of these target compounds, such as phenol, olefin, amine and aniline moieties. But Ferrate (VI) failed to react with triclocarban, 3 androgens, 7 acidic pharmaceuticals, 2 neutral pharmaceuticals and erythromycin-H\(_2\)O.

In a recent study by the author’s team, selected pharmaceuticals were spiked into the effluent samples with concentration of 10 \(\mu\)g L\(^{-1}\) for each compound. Results showed that removal efficiencies of ciprofloxacin and naproxen were up to 70% and 50%, respectively, for ferrate(VI) doses up to 5 mg L\(^{-1}\). Except ciprofloxacin and naproxen, raising ferrate(VI) dose did not improve the removal of other pharmaceutical significantly (Fig. 5). The relative high reactivity of ciprofloxacin and naproxen with Fe(VI) may be attributed to electron donation by the methoxy group to the naphthalene moiety [37, 44]. The acidic pharmaceuticals compounds (such as ibuprofen) showed less reactivity with ferrate(VI) because a carboxylic group is an electron-withdrawing functional group, which can depress the reaction of aromatic ring with Fe(VI). Therefore, the reactivity of Fe(VI) with carboxylic acids is usually slow.

The rate constant for the reaction of ferrate(VI) with selected EDCs and PPCPs can be seen in Table 1. The data indicate relative reaction activities between ferrate(VI) and various compounds. Ferrate(VI) can degrade most listed EDCs at rapid speed except for Buten-3-ol which has low reactivity with ferrate(VI). For the PPCPs, atenolol, carbamazepine, ibuprofen have low reactivity especially ibuprofen has the slowest reaction rate with ferrate(VI). On the other hand, the relative high reactivity of most EDCs and
PPCPs with ferrate(VI) may be attributed to electron donation by the specific group to the naphthalene moiety [37, 44].

Table 1. Second order rate constants $k$, M$^{-1}$s$^{-1}$ for the reaction of ferrate(VI) with selected endocrine disrupting chemicals (EDCs) and pharmaceuticals and personal care products (PPCPs)

| Group                | Contaminant               | pH | Rate constant $k$, M$^{-1}$s$^{-1}$ | Half life $t_{1/2}$ | Reference |
|----------------------|---------------------------|----|-------------------------------------|---------------------|-----------|
| Endocrine disruptors | $17\alpha$-Ethinylestradiol | 7  | $7.3 \times 10^2$                   |                     |           |
| Endocrine disruptors | $17\alpha$-Ethinylestradiol | 8  | $4.2 \times 10^2$                   |                     |           |
| Endocrine disruptors | Bisphenol A              | 7  | $6.4 \times 10^2$                   |                     | [45]      |
| Endocrine disruptors | $\beta$-estradiol        | 7  | $7.7 \times 10^2$                   |                     |           |
| Endocrine disruptors | Phenol                    | 7  | $7.7 \times 10^1$                   |                     |           |
| Endocrine disruptors | Bisphenol-A              | 7  | $7.7 \times 10^1$                   |                     |           |
| Endocrine disruptors | $17\beta$-estradiol       | 7  | $7.6 \times 10^2$                   |                     |           |
| Endocrine disruptors | $17\beta$-estradiol       | 8  | $4.6 \times 10^2$                   |                     |           |
| Endocrine disruptors | 4-methyphenol             | 7  | $6.9 \times 10^2$                   |                     |           |
| Endocrine disruptors | 4-methyphenol             | 8  | $3.3 \times 10^2$                   |                     |           |
| Endocrine disruptors | Buten-3-ol                | 7  | 12                                  |                     | [44]      |
| Endocrine disruptors | Buten-3-ol                | 8  | 3                                   |                     |           |
| PPCPs                | Atenolol                  | 8  | 7                                   | 0.7 s               | [46]      |
| PPCPs                | Bisulfite                 | 8  | $1.90 \times 10^4$                 | 0.2 s               | [47]      |
| PPCPs                | Bisulfite                 | 7  | $8.24 \times 10^4$                 |                     |           |
| PPCPs                | Carbamazepine             | 8  | 16                                  |                     | [47]      |
| PPCPs                | Ciprofloxacin             | 7  | $4.7 \times 10^2$                   | 29.4 s              |           |
| PPCPs                | Enrofloxacin              | 7  | $4.6 \times 10^1$                   | 300 s               | [45]      |
| PPCPs                | Ethionine                 | 8  | $8.3 \times 10^2$                   | 17 s                | [47]      |
| PPCPs                | Glycolic acid             | 8  | $4.0 \times 10^3$                   | 1.39 h              | [48]      |
| PPCPs                | Glycolic acid             | 12.4 | $7.2 \times 10^3$         |                     | [27]      |
| PPCPs                | Glycylglycine             | 7  | $8.2 \times 10^2$                   | 17 s                | [49]      |
| PPCPs                | Ibuprofen                 | 8  | 0.4                                 |                     | [46]      |
| PPCPs                | Iodide                    | 7  | $6.67 \times 10^3$                 | 2.1 s               |           |
| PPCPs                | Iodide                    | 8  | $1.54 \times 10^3$                 | 9.0 s               | [47]      |
| PPCPs                | Sulfamethizole            | 7  | $4.1 \times 10^3$                   | 33.9 s              | [50]      |
| PPCPs                | Sulfamethoxazole          | 8  | 95                                  |                     | [44]      |
| PPCPs                | Sulfamethoxazole          | 7  | $1.3 \times 10^3$                   | 10.4 s              | [50]      |
| PPCPs                | Sulfisoxazole             | 7  | $1.5 \times 10^3$                   | 9.2 s               |           |
3. Assessment of the toxicity of ferrate(VI) treated water and waste water

It is important to determine whether the ferrate(VI) treated water contains any toxic or mutagenic substances as this should relieve public health concerns when a new chemical is employed for water treatment. The Ames test has been applied to ferrate(VI) treated water and a preliminary study demonstrated negative results [51], suggesting that ferrate(VI) reagents do not produce mutagenic by-products for the study conditions. Moreover, in a recent study by the author’s group in Glasgow, UK, the toxicity of the ferrate(VI) treated waste water effluent was assessed and compared with that of raw waste water effluent by the zebrafish embryos model. The zebrafish, a small tropical fish native to the rivers of India and South Asia [52], has become one of the most popular model organisms in developmental genetics and (eco) toxicology [53-55]. The zebrafish embryos represent an attractive model for environmental risk assessment of chemicals since they offer the possibility to perform small-scale, high-throughput analyses. The results of both mortality of zebrafish embryo tests and microscopic images demonstrated that raw waste water effluents possessed toxicity to zebrafish but ferrate (VI) treated effluents had no adverse effects.

As shown in Table 2, a number of other studies have reported the potential formation of harmful by-products. Most recently, A study showed the formation of adsorbable organic haloids (AOX) as by-products in the ferrate(VI) treated waste water effluents although the AOX concentration rise was lower than that in the chlorination process [56].

Obviously, more researches need carried out to investigate the formation potential of harmful by-products during ferrate(VI) treatment. For example, it is to be studied under which operating conditions and for which original pollutants, that harmful by-products would be formed.
Table 2. Harmful by products formation in the ferrate(VI) treated waste water

| Compound      | By-product                                      | Ref.     |
|---------------|-------------------------------------------------|----------|
| Carbohydrates | Aldehydes                                       | [57]     |
| Aniline       | Azobenzene and Nitrobenzene                     | [58]     |
| Phenol        | Pbenzoquinone and Biphenols                     | [58]     |
| Methanol      | Formaldehyde                                    | [59-61]  |
| Carbamazepine | Aldehyde, Ketone, and Carboxyl groups           | [62]     |
| Sulfamethoxazole | Methyl group compounds                       | [50]     |
| Trimethoprim  | 3,4,5,-trimethoxybenzaldehyde and 2,4-dinitropirimidine | [63]     |

4. Concluding remarks - feasibility of ferrate(VI) treatment in full scale water and waste water treatment

The high oxidation properties of ferrate(VI) were discovered a long time ago but systematic studies of oxidising a number of organic compounds or pollutants using ferrate(VI) can be dated back to the early 1970s. Oxidation of micro pollutants existing in water and waste water by ferrate(VI) has been studied extensively since the beginning of this century. The advances in analytical chemistry theory and instrument allow tiny and low level concentrations of micro-pollutants could be detected which helps the new legislated regulations could be setup and quality of water could be monitored. Water industries have to meet the requirement of stringent water and waste water quality regulations and therefore, alternative technologies are sought by water industries.

A number of laboratory based studies have investigated schemes and mechanisms, possible by-products formation, and kinetics or rate constants of the degradation of micro pollutants; these have advanced knowledge of the use of ferrate(VI) for the environmental remediation. In comparison with these fundamental studies, relative smaller number researchers have focused on the practical application of ferrate(VI) for water and waste water treatment or the environmental remediation such as odour removal and sewage sludge treatment. Nevertheless, a few of cases have been reported that ferrate(VI) have been used in full scale applications so far.

Studies on the overall efficiency of ferrate(VI) as a coagulant, an oxidant or a disinfectant have been carried out for water and waste water treatment. However, there are some fundamental issues which have not yet been studied thoroughly and are critical to implement ferrate(VI) into full-scale water treatment and other environmental remediations. This author suggests following future work to be carried out:

1. To classify and assess the toxicity of the potential degraded by-products when ferrate(VI) is used to oxidise various micro-pollutants;
2. To study the effects of dosing points, dosing methods, dosing farcicalities and mixing schemes on the ferrate(VI) performance in water and wastewater treatment;
3. To investigate the impact of water quality characteristics on the ferrate(VI) efficiency as a disinfectant and as an oxidant;
4. To assess the effect of ferrate(VI) dose and pH on the reduction of various micro-pollutants and on the inactivation of bacteria and virus in sewage sludge treatment, and finally;
5. To carry out a full-scale trial to validate the treatment performance obtained in the laboratory studies and to evaluate economic suitability of using ferrate(VI) comprehensively.

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