

The Power of Ferrate(VI)

ABSTRACT: The potential of ferrate, specifically Ferrate(VI), a supercharged form of iron with a +6 oxidation state, as an advanced oxidant for water and wastewater treatment is widely documented. While the use of ferrates has been limited due to synthesis difficulties and inherent instability, AMS has developed a novel system for on-site production of Ferrate(VI) to address this limitation. Examining the history of oxidation in water treatment and the role of advanced oxidation processes (AOPs) in oxidizing complex organic constituents, it is evident that Ferrate(VI) can be a strong oxidant with a higher oxidation potential than ozone and chlorine. Despite challenges in treating stubborn organics and the need for efficient and economical AOPs, ongoing study and consideration for Ferrate as an AOP option will reshape the paradigm of advanced oxidation for water and wastewater treatment.

INTRODUCTION

Ferrate (FeO_4^{2-}), first synthesized in Germany in 1715, is a supercharged form of iron with a +6 oxidation state, also known as Ferrate(VI). It is one of the strongest oxidants known, capable of solving a range of treatment challenges unlike other common oxidants. Ferrate(VI) is environment-friendly, and when applied to organic-containing waters, it does not create toxic disinfection by-products (DBPs). Ferrate(VI) rapidly decomposes to ferric iron, precipitating quickly from the solution. Because the residual is non-toxic ferric iron, it can be safely land-applied for disposal or recycled.

Ferrate(VI) has been demonstrated to be a powerful oxidant that can purify water safely from a wide range of pollutants, including viruses, microbes, arsenic, sulfur-containing compounds, cyanides and other nitrogen-containing contaminants, a broad range of organic compounds, and algae. However, commercial utilization of ferrates has been extremely limited primarily because of the difficulty in synthesizing them and their inherent instability. AMS has developed a novel system where ferrate is produced on-site for treatment purposes and contaminants of concern are monitored in real-time.

HISTORY OF OXIDATION IN WATER

Chemical oxidation in water treatment usually involves the use of oxidizing agents such as ozone, hydrogen peroxide or chlorine compounds. Advanced oxidation processes (AOP) are where free hydroxyl radicals are used as a strong oxidant to destroy specific organic constituents that cannot be oxidized by conventional oxidants.

Oxidation-reduction reactions take place between an oxidizing agent and a reducing agent. In a redox reaction, electrons are exchanged, the oxidizing agent is reduced, and the reducing agent is oxidized. Chemical oxidation reactions often require the presence of catalysts for the reaction to proceed or to increase the rate of the reaction. Metal cations, enzymes and pH adjustment can be used as catalysts. The traditional applications of chemical oxidation in water include reducing the concentration of residual organics, controlling odors, removing ammonia, and reducing pathogen content.

Dating back to the early 1900s, the primary purpose of biological treatment has been to remove organic compounds to prevent excessive dissolved oxygen (DO) depletion, remove suspended solids, and inactivate pathogens. Chemical oxidation has been used in municipal water treatment for over a century, primarily for disinfection. In 1908, Jersey City, New Jersey, became the first city to use chlorine as a primary disinfectant for drinking water.¹ Chlorine's oxidative properties were intended to fight waterborne disease; however, they also created potentially toxic DBPs. Ozone was first used in a commercial scale in the Netherlands in 1893.²

AOPs are used to oxidize complex organic constituents that are difficult to degrade. It is not always necessary to completely oxidize a given compound. In many cases, partial oxidation is sufficient to reduce toxicity. AOPs were first proposed for drinking water treatment in 1980,³ and have since been studied as oxidizing treatments for various wastewaters.

AOPs are a set of chemical treatment procedures designed to remove organic, and sometimes inorganic, materials in water and wastewater by oxidation through reactions with hydroxyl radicals (OH). In real-world applications of wastewater treatment, however, this term usually refers more specifically to a subset of such chemical processes that employ ozone (O_3), hydrogen peroxide (H_2O_2) and UV light, or a combination of the few processes. The hydroxyl radical is one of the most active oxidants known. It reacts with dissolved constituents, initiating a series of oxidation reactions until they are completely mineralized. Hydroxyl radicals are capable of oxidizing almost all reduced materials without restriction because the mode of action is nonselective and can occur at normal temperatures.

Since AOPs were first defined in 1987,⁴ the field has witnessed a rapid development both in theory and in application. TiO_2/UV systems, $\text{H}_2\text{O}_2/\text{UV}$ systems, and Fenton, photo-Fenton and Electro-Fenton systems have received extensive scrutiny. There are still many research needs on these existing AOPs. Recent trends are the development of new, modified AOPs that are efficient and economical.

ADVANCED OXIDATION POTENTIAL USING FERRATE

Another AOP option currently being studied and considered is ferrate. Ferrate is one of the strongest oxidants available. Its oxidation potential (Figure 1) is higher than ozone's and almost twice that of chlorine.⁵

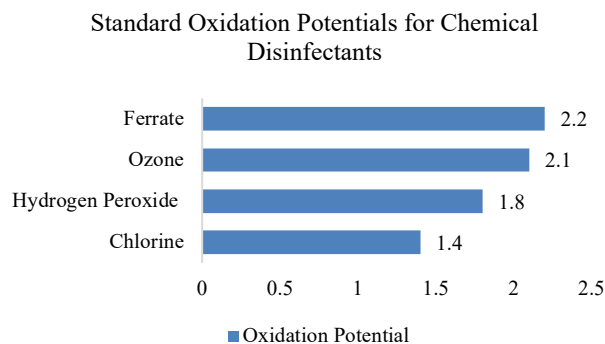


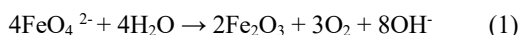
Figure 1. The Standard Oxidation Potential for Chemical Disinfectants Adapted from Metcalf & Eddy (2003), Wastewater Engineering Treatment and Reuse, Fourth Edition, McGraw Hill.

Treatment of recalcitrant organics and inorganics present in wastewater is a major challenge. Conventional biological treatments alone cannot remove these toxic compounds from wastewater. To overcome these problems, AOPs have been used to completely mineralize or transform the organics into simpler compounds,⁶ which can then be treated through biological processes. However, conventional AOPs result in the generation of byproducts, which are known to have higher toxicity. Among various AOPs, ferrate has been gaining popularity because of advantages such as high oxidation potential and non-toxic end-product formation. The end-product (ferric oxide) generated also acts as a coagulant, which thereby enhances the removal efficiency of the contaminant.

Scientific discoveries and developments in pharmaceuticals, pesticides, personal care products (PCPs), and basic sanitation have improved the lifestyle and living standards of humans across the globe. However, these developments come with an environmental cost. Environmental and human health risks associated with traces of pharmaceutical, pesticides and microplastics have been studied by various researchers. Due to the transformation and interaction of these compounds with the environment, they can enhance the resistance of microbes, thus making them resistant to antibiotics. Furthermore, some of the pesticides have been proven to have a carcinogenic effect. With these environmental challenges, there has been a sharp increase in the research for treating and removing these emerging contaminants.

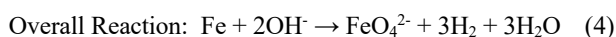
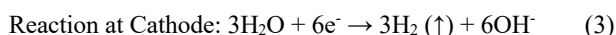
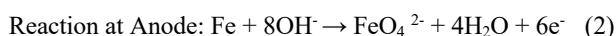
Innovative technologies using alternative sources such as peroxides, ferrates, and ozone can significantly improve the wastewater treatment due to their higher selectivity and efficiency. Among them, Ferrate(VI) is a promising choice for environmental remediation.⁷ It does not produce any harmful byproducts during treatment and provides efficient degradation of organic, inorganics and microorganisms over a wide range of

pH. Typically iron (Fe) exists in two oxidation states either as ferrous (Fe (II)) or ferric (Fe (III)). However, under strong oxidizing conditions, a higher oxidation state (i.e., +IV, +V and +VI) of iron can be achieved, which is referred to as a ferrate. Among the various higher oxidation state, the +VI state of iron is comparatively stable and easier to produce. In the aqueous phase, there are many factors (such as pH, temperature and ferrate concentration) on which the stability of ferrate depends.⁸ Ferrate ions and water molecules reacts to form ferric oxide (Fe₂O₃), oxygen gas, and hydroxyl ions (Equation (1)). Due to the release of hydroxyl ions, the resulting pH of the solution is highly alkaline. The ferric oxide, generated as an end product, acts as a coagulant:



The wet oxidation synthesis of ferrate involves oxidation of ferric to ferrate under high pH conditions.¹⁰ The solution containing ferrate obtained by this procedure is highly unstable, which thereby demands subsequent procedures of precipitation, washing and drying to obtain a stable solid product. The method of producing ferrate by wet oxidation is known since the 1950s.

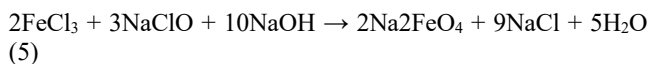
The method involves a reaction between ferric chloride and sodium hypochlorite in the presence of an alkali such as sodium hydroxide. Further recovery of potassium ferrate is obtained by precipitation with potassium hydroxide. Even though a percentage recovery of potassium ferrate as high as 96% could be obtained, the maximum yield percentage obtained by continuous efforts was 75%.⁸ The dry oxidation method for ferrate synthesis is a very old method. However, the method involves high risks as it can lead to an explosion at elevated temperature.¹¹ During the electrochemical synthesis of ferrate, anodic dissolution of iron takes place in a strongly alkaline solution. The current applied during synthesis oxidizes the iron to ferrate in the alkaline solution (KOH or NaOH). The reactions at the anode and cathode are as follows (Equations (2-4)):



Factors such as anode composition, current density and strength of electrolyte govern the production of Ferrate(VI). The major advantage of electrochemical synthesis is its simplicity and no costly chemical requirement.

ON-SITE ELECTROLYTIC FERRATE GENERATION ON DEMAND

Two sodium ferrate generation approaches are suitable for at site reagent production: wet oxidation method and electrolytic method. In wet oxidation approach ferric salt (typically acidic ferric chloride) is oxidized into Ferrate(VI) by strong oxidizers in concentrated caustic solution. Typically, sodium hypochlorite is used as an oxidizer in this process. The reaction equation is shown in Equation (5):



The wet method's disadvantages and limitations result from the reaction stoichiometry and the nature of the ingredients used. As it follows from the reaction it is material intensive and involves multiple reagents including highly acidic ferric chloride solution, concentrated bleach solution as well as relatively high amounts of concentrated caustic solution. Ferric chloride is an acidic and highly corrosive chemical. Bleach solution is unstable and tends to decompose releasing highly dangerous chlorine gas. Both reagents pose environmental and health risks during their transportation, storage and handling. Ferrate produced using this approach is not pure and contains a significant amount of sodium chloride, bleach and chlorine residuals along with potential ferric chloride co-contaminants such as trace metals. Also, chlorine residuals in the ferrate product may cause excessive levels of disinfection by-products in the treated water. For generation of one mole of sodium ferrate five moles of caustic are required. In fact, caustic consumption is even higher because some caustic is consumed by the excessive acidity of ferric chloride reagent. Finally, the wet ferrate generation process is a batch method which requires bulky equipment and has a relatively low ferrate yield resulting in high reagent costs.

AMS's electrolytic approach to on-demand sodium ferrate reagent generation is straightforward and relatively simple. The approach is based on membrane electrolysis of an iron anode in sodium hydroxide electrolyte according to electrode reactions (Equations (2-4)).

In this method the iron anode precursor corrodes under controlled conditions producing high valence sodium Ferrate(VI) selectively into the electrolyte (Equation (2)). As the result of cathode reaction water molecules split into hydrogen gas and hydroxyl ions (Equation (3)). As it is seen from reactions (Equations (2-4)) this electrolytic process requires only three consumables: sacrificial iron anode, ~ 20-40% caustic solution and electricity. It should be noted that the relatively low caustic demand in the electrolytic method is the result of the significant amount of caustic produced during electrolytic process on the cathode (Equation (3)) which makes this approach more economical. The electrolytic approach is a one-step process in which electrolyte is continuously fed into the electrolytic unit while outflowing ferrate reagent is dosed into the treated flow. The high effectiveness of the innovative AMS's ferrate generation approach is due to the optimal both design of flow-through electrolytic device and its operating conditions. The proprietary anode de-passivation mechanism implemented in the electrolytic process allows for long-term anode stability, reliable ferrate generation in a fully automated continuous manner. AMS's ferrate generation modules are equipped with an inline ferrate concentration monitor which ensures a stable ferrate generation process. The compact, modular, and flexible design of the electrolytic unit allows for building ferrate generating capacity to treat virtually unlimited volumes of contaminated water.

Overall, AMS's innovative electrolytic sodium ferrate generation process (part of the SafeGuard™ H2O product line) is relatively simple, safe, and reliable. The entire ferrate generation process parameters are remotely monitored and controlled to ensure stability and integrity.

The SafeGuard H2O technology is an intelligent water treatment system that integrates real-time sensing with an innovative approach to generating water treatment chemicals on-site

and on-demand. SafeGuard H2O generates a ferrate, ferrous, or stannous-based reagent through a patented electrolytic process. The technology is proven to be an economical and sustainable approach for removing a wide range of contaminants. SafeGuard H2O can be used for corrosion control, drinking water purification, resource recovery, and wastewater treatment.

The advanced SafeGuard H2O technology supports reduction greenhouse emission targets by eliminating bulk deliveries and handling. On-site reagent generation is an affordable, non-hazardous and environmentally sustainable solution for broad water treatment needs.

APPLICATIONS FOR ON-SITE GENERATED FERRATE (VI)

The increasing occurrence of emerging pollutants such as pharmaceuticals, dyes, heavy metals, and endocrine disrupting compounds, among others, in wastewater¹² has generated a growing concern. Due to the recalcitrant nature of these compounds, they remain in the environmental matrices for longer duration and are not degraded naturally or by biological reactions. Ferrate(VI) as an oxidizing agent is a promising choice for the remediation of water and wastewater containing recalcitrant pollutants. Highly reactive Ferrate(VI) is capable of oxidizing a broad range of toxic compounds more rapidly than permanganate and chromate.¹³ The oxidizing potential of Ferrate(VI) in water and wastewater treatment mainly depends upon the treated water pH, initial ferrate concentration, presence of co-existing ions and water temperature. As an oxidizing agent, after its reaction with contaminant, Ferrate(VI) is converted into insoluble and non-toxic Fe(III) species and can adsorb organic or inorganic compounds, resulting in its removal. In addition to the higher oxidizing ability of Ferrate(VI), other intermediate oxidation states of iron Fe(V) and Fe(IV) are claimed to help oxidation of organic and inorganic compounds.

PHARMACEUTICALS REMOVAL

The presence of pharmaceutically active compounds in the aquatic environment can cause serious health issues to living organisms. The strong oxidation potential of Ferrate(VI) and its ability to oxidize/remove nitrogen- and sulfur-containing compounds have gained attention for the removal of pharmaceuticals from wastewater. A pilot scale test was performed on drinking water with low dose of Ferrate(VI) (0.1 mg/L). This test achieved a particle removal rate of 93% on raw water and 97% on ozonized water. No pH neutralization was required after dosing. This study also showed 10% removal of metformin, benzotriazole and acesulfam.¹⁴ The efficacy of Ferrate(VI) in the simultaneous removal of emerging micro-pollutants, triclosan, and amoxicillin was conducted in a batch reactor using a dose of 0.2µg/L and 65.5% of triclosan and 76.3% of amoxicillin were removed.¹⁵ Another study analyzed the removal of naproxen and ciprofloxacin from wastewater matrices with concentrations of 10µg/L. Results showed removal rates of between 50 and 70%.¹⁶ The capability of ferrate to remove various pharmaceuticals in a jar test was the subject of another study. It was shown to remove more than 80% of ciprofloxacin at a dose of 1 mg/L and 30% of ibuprofen at a dose of 2 mg/L. Promising performance of ferrate in the treatment of real wastewater effluent at both a pH of 6 and 8 and a dose range of 1–5 mg/L was also observed. Removal efficiency of ciprofloxacin was the highest among the target compounds at 63%, followed by

naproxen at 43%. N-acetyl sulphamethoxazole was the hardest to be removed by ferrate, with a rate of 8%.¹⁷ Another study looked at the removal of sulfamethoxazole over a wide range of concentrations at a pH of 6 with a dose of 2.5 µg/L Ferrate(VI). Removal rates ranged from 79% to 16% depending upon the initial concentration of sulfamethoxazole.¹⁸ In another study, the removal of trimethoprim and sulfamethoxazole was analyzed using Ferrate(VI). This study showed removal rates for trimethoprim of up to 91% and sulfamethoxazole up to 36%. The rates depended on the natural organic matter (NOM) present in the matrix.¹⁹

DYES REMOVAL

The application of Ferrate(VI) for removal of textile dyes is one of the promising technologies because it can act as an oxidant as well as a coagulant at the same time; it enhances the biodegradability property of dye. A study was performed investigating the azo dye orange II removal by ferrate. It was observed that within 30 minutes, maximum discoloration of 95.6% was achieved.²⁰ In another study, a highly stable composite Ferrate(VI) solution was produced for the degradation of azo dye reactive brilliant red X-3B. At a dosage of 2.5 mg/L discoloration of 99% was achieved in 20 minutes.²¹

Another study looked at the removal of methylene blue (MB), remazol black-blue (RBB), and methyl orange (MO) dyes. Degradation rates of close to 100% were achieved with an optimal contact time of 60 minutes using a dose of 2.6 µg/L in a solution with a dye concentration of 10 mg/L.²²

ENDOCRINE DISRUPTORS REMOVAL

The removal of phenolic compounds from environmental matrices has attracted considerable attention in recent years due to their recalcitrant and hazardous properties. One study examined removing four different types of phenolic compounds, such as 2-benzylphenol, phenol, chlorophene and 4-chlorophenol by Ferrate(VI) at pH 8. Under optimal reaction conditions 20.0 µM BPE could be almost completely removed by Ferrate(VI) in 180 seconds.²³ Another study observed the complete degradation of Bisphenol-A within 30 minutes of oxidation under weak acidic pH.²⁴ A third study showed that complete destruction of BPA could occur in 5 minutes when the Ferrate(VI) to BPA molar ratio was 5:1.²⁵ Ferrate(VI) was utilized to treat micro-pollutants — 4-tert-octylphenol (TOP) and 17 α -ethynylestradiol (EE2) — from aqueous solutions. Batch studies were conducted at various pH levels and concentrations of TOP or EE2 using 0.2 mg/L Ferrate(VI). Significant mineralization of 80 – 100% was achieved by Ferrate(VI) treatment.²⁶

PFAS REMOVAL

Perfluoroalkyl and polyfluoroalkyl substances occur in consumer and industrial products. They have been found globally in drinking water, the aquatic environment and in wastewaters. This raises health concerns due to their ability to bioaccumulate and extreme persistency. One study looked at the ability of ferrate to oxidize these compounds. Maximum removals were 34% for perfluorooctanesulfonate and 23% for perfluorooctanoic acid.²⁷ In another study, zero-valent iron (ZVI) and Ferrate(VI) were used as co-milling reagents to degrade Perfluorohexane sulfonate. When ZVI and Ferrate(VI) were used alone, both the degradation and defluorination efficiencies were low.

However, after milling at the optimum ratio the synergistic effect of ZVI and Ferrate(VI) resulted in almost complete degradation (100%) and defluorination (95%).²⁸

PRODUCED WATER TREATMENT

Fracking operations generate produced water that is highly emulsified, viscous and has a high chemical oxygen demand (COD), which makes it difficult to treat and recycle. Ferrate has been adopted to treat this waste stream because it can manage all of these issues. One study demonstrated that ferrate oxidation resulted in demulsification efficiency increase from 56% to 92%, total viscosity dropped from 1.45 centipoise (cp) to 1.10 cp, and total removal of COD increased to over 74%.²⁹ In another study, the ability of Ferrate(VI) to coagulate organic and inorganic compounds to reduce turbidity was assessed under a variety of conditions. Significant reductions in turbidity (95.07% - 97.66% removal) were observed at a variety of hypersaline concentrations and temperature ranges.²³⁰

Another study aimed to determine the effect of ferrate to remove organic matter from shale gas flow back water. A chemical oxygen demand removal efficiency of 57% was achieved under the optimal conditions. The fluorescence intensity of soluble microbial by-product-like matter and acid-like components were reduced by 64% and 43%.³¹ Another study showed a 55% removal of COD from oil well produced water using Ferrate under optimal conditions.³²

PHOSPHOROUS REMOVAL

Dissolved organic phosphorous (DOP) accounts for 26% to 81% of total phosphorous in municipal wastewater. It is difficult to remove with conventional wastewater treatment processes. This inorganic nutrient is then bioavailable threatening the aquatic environment through eutrophication. One study showed that ferrate treatment could effectively destroy and remove 75% of the DOP in secondary effluent from a conventional activated sludge plant.³³ In another study ferrate was shown to reduce phosphate levels in municipal wastewater by 77% with a dose of 7.5 mg/L.³⁴ This study also showed 85% removal of selected micropollutant reinforcing the conclusion that ferrate could be used as an effective enhanced treatment process. Another study showed that ferrate could remove total phosphorous by up to 87% in chemically enhanced primary treatment plants.³⁵ Another study showed that ferrate could remove up to 97.3% of inorganic phosphorous during wastewater treatment through simultaneous oxidation and coagulation.³⁶

CONCLUSION

Ferrate has been shown to successfully oxidize, coagulate and remove a broad range of contaminants of concern including pharmaceuticals, fracking produced water pollutants, PFAS, dyes, and hormone mimicking compounds.

Significant limitation of Ferrate(VI) as a treatment reagent have been overcome by AMS's novel on-site SafeGuard H₂O Ferrate(VI) generation system. The highly reactive and concentrated Ferrate(VI) treatment reagent produced by the SafeGuard H₂O system delivers a unique and cost-effective treatment solution for the most challenging water and wastewater applications.

AUTHORS

Paul Gagliardo* and Vladimir Dozortsev**

*Independent Consultant, 1045 San Dieguito Drive, Encinitas, CA 92024

**AMS, 1225 E. Arques Ave., Sunnyvale, CA 94085

Paul Gagliardo is an independent consultant assisting and advising innovative water sector startup companies. He has held leadership positions in the water and wastewater business for over 30 years at the city of San Diego, American Water and multi-national consulting companies. Paul has been a judge for the Imagine H2O Accelerator since its inception in 2009. He is a registered engineer in California and has a master's degree in

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public health. He is also the host of The Water Entrepreneur podcast.

Vladimir Dozortsev, PhD. is the Senior Product Manager for AMS. Dr. Vladimir Dozortsev holds PhDs in Chemistry and Chemical Engineering. He is the author and co-author on numerous patents related to voltammetry-based analysis of trace metals and the in-situ generation and use of ferrous, ferrate and stannous for water and wastewater remediation. He has published widely in these fields and his work has greatly influenced the development of cost-effective and environmentally sustainable solutions to analyze and treat contaminants.

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