

Hydrogen sulfide and organic compounds removal in municipal wastewater using ferrate (VI) and ultraviolet radiation

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Abstract

Background: In this study, ferrate (VI) and ultraviolet (UV) radiation were employed to remove hydrogen sulfide from municipal wastewater resulting in a reduction in chemical oxygen demand (COD). Although ferrate (VI) and UV have been used individually for the removal of a few pollutants from urban and industrial wastewater, there exists no study to investigate the effectiveness of simultaneous utilization of both methods for the removal of hydrogen sulfide and reducing COD.

Methods: This study aims to compare the application of UV, ferrate (VI) and UV/ferrate (VI) for the removal of hydrogen sulfide and COD from municipal wastewater in batch mode. Moreover, the effect of many parameters such as ferrate (VI) concentration, temperature, hydraulic retention time (HRT) and pH on ferrate (VI) oxidation power, were investigated.

Results: The results of this study demonstrated that for pH less than 2, higher pollutant removal efficiency was obtained. COD removal efficiency could increase up to 68% by adding 1.68 mg/L of ferrate (VI), almost 100% of hydrogen sulfide was removed by the same concentration of ferrate (VI). Both hydrogen sulfide and COD removal efficiencies increased as temperature increased to 50°C; nevertheless, further increase in temperature had negative effect on the removal efficiency. The use of UV/ferrate (VI) increased the removal efficiency of both hydrogen sulfide and COD when compared with the use of UV and ferrate (VI) individually. UV method was not effective in the removal of hydrogen sulfide.

Conclusion: The research findings shed new light on wastewater treatment systems employing UV/ferrate (VI) to decrease both the hydrogen sulfide and COD of municipal wastewater. This new findings will assist in the inaccurate design and effective operation of such systems which can be employed to maintain or improve environmental quality.

Keywords: Ferrate (VI), Hydrogen sulfide removal, Organic compounds removal, Ultraviolet

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Introduction

Anaerobic reactors are among the most important wastewater treatment systems which are capable of removing recalcitrant organic compounds. Anaerobic reactors have many advantages like high pollutant removal efficiency, low energy consumption and less sludge production when compared to aerobic reactors. As a result, anaerobic reactors are common to be used for a diversity of industrial wastewater. Nevertheless, anaerobic systems suffer from odor production (1,2). Controlling odor production in wastewater collection and treatment systems is one of the most important research areas in the field of environmental engineering (3). Hydrogen sulfide is a malodorous compound emitted from wastewater collection and treatment systems (4). This gas is extremely

toxic for humans at levels above 500 ppm (1). In addition to odor problem, hydrogen sulfide gas causes corrosion of facilities (1). Wastewater collection systems made of concrete are seriously damaged by microbial corrosion (5,6). Corrosion in wastewater collection systems occurs because different forms of sulfur (such as mineral and organic sulfur) are changed to hydrogen sulfide (1). The activities of sulfur-reducing bacteria, which exist in anaerobic conditions caused as a result of oxygen deficiency, produce hydrogen sulfide (7). Hydrogen sulfide evaporates from wastewater and accumulates in the upper space of wastewater collection pipes (8). Sufficient oxygen and moisture in the upper space of wastewater collection pipes generate a perfect condition for growing sulfur oxidizing bacteria, which absorb hydrogen sulfide and



convert it to sulfuric acid (9). This is the reason behind the extensive corrosion problems in concrete pipes (1,7,8). Coagulation, oxidation and disinfection are three important processes in water and wastewater treatment technology (10). Coagulant compounds agglomerate non-settling colloidal particles in the form of large particles. These large particles can be eliminated by settling or filtration. Disinfection processes have been designed to remove microorganisms harmful to human health (11). Sometimes, disinfectants are used not only for the removal of microorganisms but also for odorous compounds (4). Several different oxidizers and disinfectants like chlorine, sodium hypochlorite, chlorine dioxide and ozone are used in water and wastewater treatment (12). Chlorine oxidizes wastewater organic compounds and produces harmful by-products called trihalomethanes. Although chlorine dioxide and ozone do not suffer from secondary pollutant production, they are expensive (13). As environmental regulations for releasing wastewater into the environment are being more strict annually, new efficient coagulants, oxidizers and disinfectants are needed to meet these stringent restrictions. Ferrate (VI) is an oxidizing compound capable of fulfilling both tasks of disinfection and degradation, while it does not produce any harmful secondary pollutant (6,14). Ferrate (VI) is the most powerful oxidant ever known (4). In acidic conditions, it is the most powerful oxidant ever known in water and wastewater treatment systems (12). Ferrate (VI) is converted into ferric(III) which is a coagulant reagent (15). So, ferrate (VI) can be utilized simultaneously, in a single unit for oxidation, disinfection and coagulation. There are different methods for the production of ferrate (VI) such as wet method, dry method and electrolysis method (16). Since ferrate (VI) solution is an unstable product, wet method is not a suitable method for the production of ferrate (VI) (17). Dry method has risk of explosion, so, it is not suitable for use in industrial usage. Electrochemical method is a simple method for producing ferrate (VI) (7). Talaiekhosravi et al reported that in electrolysis method, the highest amount of ferrate (VI) production was observed when 14M sodium hydroxide solution was used (4).

This study aims to evaluate the effectiveness of ferrate (VI) and UV methods in the removal of hydrogen sulfide and chemical oxygen demand (COD) from municipal wastewater. Ferrate (VI) was produced employing

electrolysis method. In this study, the effects of different parameters like pH, temperature, COD and hydrogen sulfide concentrations and hydraulic retention time (HRT) on the removal efficiency were investigated.

Materials and Methods

Production of Ferrate (VI)

Two rectangular iron with dimension of 60×24 mm and thickness of 0.63 mm were employed as anode and cathode electrodes, respectively. The DC voltage employed was within the range of 1–24 V. An electrolysis system was assembled to produce ferrate (VI) as demonstrated in Figure 1. In this study, 56 g of sodium hydroxide was dissolved in 100 mL of distilled water to prepare a 14M solution. Thereafter, the electrolysis container as illustrated Figure 1 was filled with 100 mL of 14M sodium hydroxide. Subsequently, electrodes were charged using DC current with voltage of 9 V and amperage of 1 A for 30 minutes. Given that ferrate (VI) is converted to ferric (III) as time goes by, the prepared ferrate (VI) solution must be used as soon as possible.

Determination of removal efficiency

As the first step of this study, 10 L of wastewater were obtained from the municipal wastewater collection system of Fooladshahr, Iran. Collected wastewater was carried in a cool box to maintain the temperature at 4°C and was transferred to the chemistry laboratory of Jami Institute of Technology, Isfahan, Iran as soon as possible. Thereafter, the collected wastewater's hydrogen sulfide and COD concentration were measured. Subsequently, 8 Erlenmeyer flasks, each containing 40 mL of collected wastewater, were prepared. Thereafter, certain amounts of the ferrate (VI) solution were added to each Erlenmeyer flask to prepare ferrate (VI) concentrations of 0.56, 0.7, 0.84, 0.98, 1.26, 1.4, 1.54 and 1.68 mg/L. Given that ferrate (VI) oxidizing ability is significantly increased for pH of less than 2, the pH of all solutions in the flasks was adjusted to 2 by the addition of a suitable amount of hydrochloric acid. After stirring for 30 minutes, the hydrogen sulfide and COD concentrations of all solutions in the Erlenmeyer flasks were determined. Finally, hydrogen sulfide and COD removal efficiencies were calculated using Eq. 1, as follows:

$$RE = \left(\frac{C_f - C_i}{C_f} \right) \times 100 \quad (1)$$

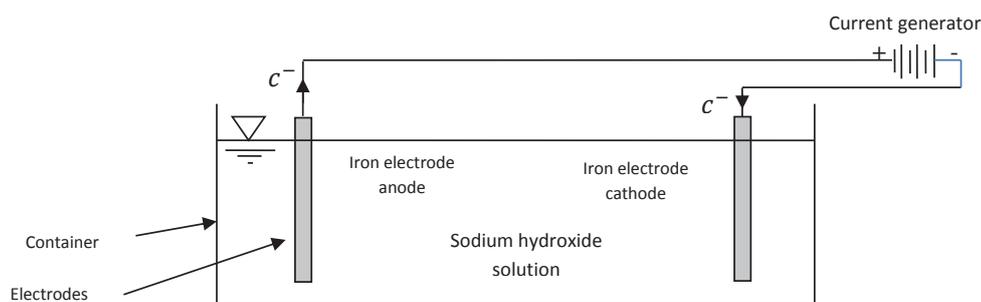


Figure 1. Schematic of the electrochemical cell for the synthesis of ferrate(VI).

Evaluation of hydraulic retention time effect

To the best of our knowledge, based on an extensive literature survey, there exists a shortage on the information about the efficiency of hydrogen sulfide and COD removal by ferrate (VI). So, in this section of the study, the effect of HRT on the removal efficiency of hydrogen sulfide and COD was investigated. For this purpose, 6 Erlenmeyer flasks, each containing 40 mL of wastewater, were prepared. A certain amount of ferrate (VI) was added to each Erlenmeyer flask to obtain a ferrate (VI) concentration of 0.5 mg/L. Subsequently, hydrogen sulfide and COD concentrations in the Erlenmeyer flasks were measured at HRTs of 10-60 minutes. Eq. 1 was utilized to calculate hydrogen sulfide and COD removal efficiency.

Evaluation of pH effect

Providing low-pH condition is vital in retaining ferrate (VI) oxidizing power as high as possible (4). In this section, the effect of pH on ferrate (VI) ability to remove hydrogen sulfide and COD was investigated. For this purpose, 6 Erlenmeyer flasks, containing 40 mL of the wastewater each, were prepared. Thereafter, a certain amount of ferrate (VI) was added to each Erlenmeyer flask to increase ferrate (VI) concentration to 0.5 mg/L. Subsequently, by adding suitable amounts of hydrochloric acid to each Erlenmeyer flask, the pH values of 1.4, 5.5, 7.2, 8.6, 10.9 and 13.2 were prepared. All Erlenmeyer flasks were stirred for 15 minutes at 23°C and then their hydrogen sulfide and COD concentrations were measured. Eq. 1 was used for calculating hydrogen sulfide and COD removal efficiency.

Evaluation of temperature effect

Temperature is an effective factor influencing the rate of chemical reaction. In this section, the effect of temperature on hydrogen sulfide and COD removal by ferrate (VI) was investigated. Temperature can be effective on the degradation rate of ferrate (VI) to ferric (III) and the oxidation rates of hydrogen sulfide and other organic compounds. So, this section was categorized into 2 sections, 1) evaluating the effect of temperature on the rate of degradation of ferrate (VI) and 2) evaluating the effect of temperature on the removal of hydrogen sulfide and COD.

In first set of experiments, 6 Erlenmeyer flasks, containing 40 mL of the wastewater each, were prepared. Thereafter, ferrate (VI) was added to each Erlenmeyer flask in the amount needed to increase its concentration to 0.5 mg/L. Erlenmeyer flasks were preserved at temperatures of 23.5, 45, 60, 70 and 80°C for 15 minutes. Finally, the concentrations of ferrate (VI) were determined in all Erlenmeyer flasks.

For the second set of experiments, 6 Erlenmeyer flasks, containing 40 mL of the wastewater each, were prepared. Thereafter, ferrate (VI) was added to each Erlenmeyer flask in the amount needed to increase its concentration to 0.5 mg/L. Erlenmeyer flasks were preserved at temperatures

of 23.5, 45, 60, 70 and 80°C for 15 minutes. Given that the increase in temperature may cause evaporation of volatile organic compounds in wastewater, each Erlenmeyer flask was considered as a control sample. Finally, the concentrations of hydrogen sulfide and COD were determined in all Erlenmeyer flasks. Eq. 1 was utilized to calculate hydrogen sulfide and COD removal efficiency.

Evaluation of UV/ferrate (VI) method effectiveness

In this section, 3 sets of experiments were performed. In first set of experiments, 6 Erlenmeyer flasks, containing 100 mL of the wastewater each, were prepared. Thereafter, a certain amount of ferrate (VI) was added to each Erlenmeyer flask to increase ferrate (VI) concentration to 0.5 mg/L. Subsequently, by adding a suitable amount of hydrochloric acid, the pH value of each Erlenmeyer was increased to 2. Thereafter, concentrations of hydrogen sulfide and COD in the Erlenmeyer flasks were measured at HRTs of 10–60 minutes.

In the second set of experiments, 6 containers holding 100 mL of the wastewater each were prepared. Thereafter, the containers were located under UV beam at 8 W power. The distance between the containers and the UV lamp was 15 mm. Subsequently, the concentrations of hydrogen sulfide and COD were measured at HRTs between 1 and 45 minutes.

In the third set of experiments, 6 containers, each containing 100 mL of the wastewater, were prepared. Thereafter, a certain amount of ferrate (VI) was added to each Erlenmeyer flask to increase ferrate (VI) concentration to 0.5 mg/L. Then, by adding a suitable amount of hydrochloric acid, the pH value of each Erlenmeyer flask was increased to 2. Subsequently, the containers were beamed by a UV lamp. Finally, the concentrations of hydrogen sulfide and COD at HRTs between 1 and 45 minutes were measured.

Analytical methods

The concentration of wastewater organic compounds was measured by the COD method, according to standard methods for examining water and wastewater (6). Iodometric titration was employed to measure hydrogen sulfide in wastewater (18). In iodometric titration method, sulfide was reacted with iodine in acidic condition. Thereafter, the amount of iodine remaining was measured by titration with sodium thiosulfate in the presence of starch as an indicator. The end of the titration was indicated by change in solution color from blue to light-straw color. A blank is treated exactly the same way as the samples. Thereafter, Eq. 2 was used for calculating hydrogen sulfide.

$$H_2S \left(\frac{mg}{l} \right) = \frac{(A \times B) - (C \times D) \times 16000}{mL \text{ sample}} \quad (2)$$

where, *A* is used iodine in mL, *B* is normality of iodine solution, *C* is used sodium thiosulfate in mL, and *D* is normality of sodium thiosulfate. The amount of ferrate (VI) was measured by the methods introduced by

Talaiekhosani et al (15).

pH value was determined using a AZ pH meter. All chemicals were obtained from Merck (Germany). Microscopic photos of the electrodes were taken by a Chinese metallurgical microscope Model VJ-2006B.

Results

Ferrate (VI) can be produced using three different methods, namely dry method, wet method and electrolysis method (4). Dry method for ferrate (VI) production is not a popular method owing to the risk of explosion. Although wet method is not dangerous, ferrate (VI) produced employing this method is highly unstable. The electrolysis method is simple, allowing *in situ* ferrate (VI) production. In this study, the effect of electrolysis process on electrode morphology was investigated using metallurgical microscope. Figure 2 illustrates the

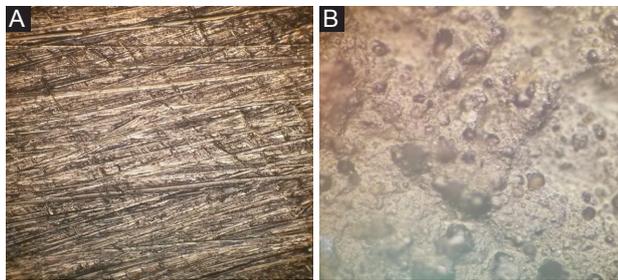


Figure 2. Microscopic figures of an iron electrode (A) before and (B) after electrolysis.

microscopic appearance of the electrodes before and after use showing high roughness on the electrode after the electrolysis process is completed. This roughness on the electrodes is created when iron is changed into ferrate (VI) and released into water.

Production of ferrate (VI) can generate a purple color in water. The colors of water before and after electrolysis are illustrated in Figure 3.

In this study, the effect of the presence of various metallic salts, like FeCl_2 , NaOH , NaCl , BaCl_2 and MgCl_2 , on the production of ferrate (VI) by electrolysis was investigated. The results are illustrated in Figure 4A.

Figure 4B illustrates the effect of temperature on the production of ferrate (VI). Figure 4C shows the effect of voltage on the production of ferrate (VI) and Figure 4D demonstrates the variation of hydrogen sulfide and COD removal efficiencies with concentrations of ferrate (VI).

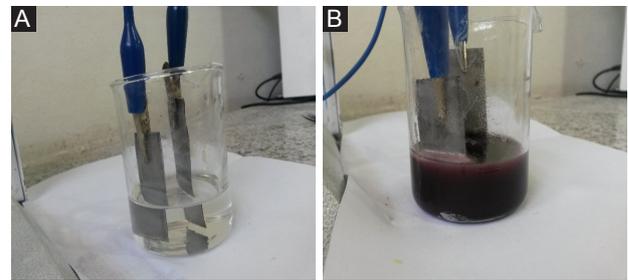


Figure 3. Change in the color of the electrolyte solution (A) before and (B) after electrolysis process.

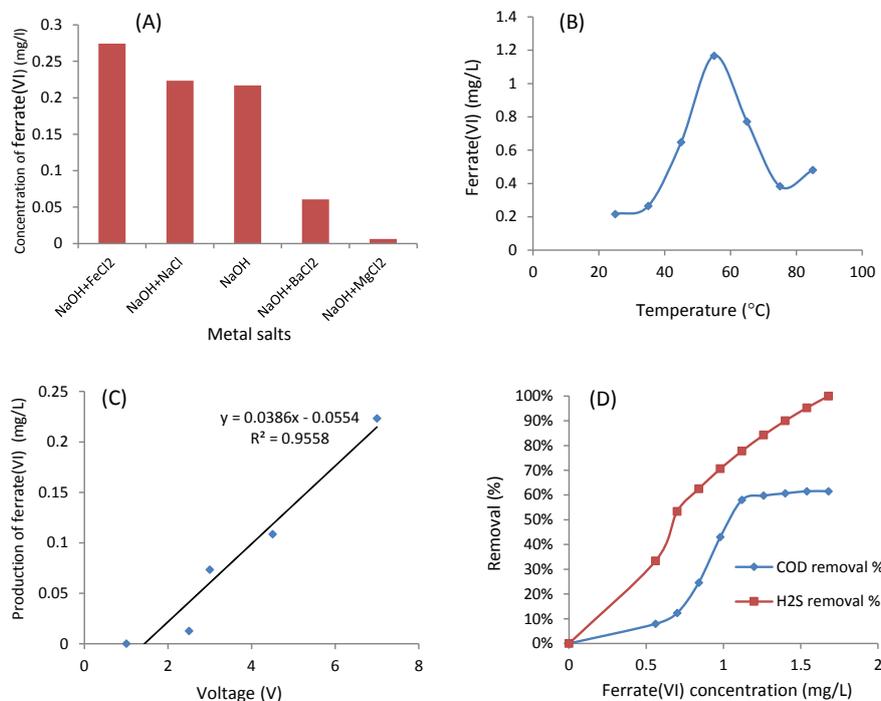


Figure 4. (A) Effect of the presence of metallic ions on ferrate(VI) production during 30 minutes of electrolysis; (B) Effect of temperature on ferrate(VI) production; (C) Effect of voltage on ferrate(VI) production and (D) Efficiency of hydrogen sulfide and COD removal with different concentrations of ferrate(VI).

Furthermore, the effects of pH, HRT and temperature on COD and hydrogen sulfide removal are shown in Figure 5A, 5B, 5C and 5D, respectively. Ferrate (VI) is degraded over time (Figure 6A and 6B) that the rate of its degradation under different temperatures can be seen in Figure 6A. Given that UV is a powerful oxidizing method, its effect on COD and hydrogen sulfide removal was investigated. The results of this investigation are illustrated in Figure 7. These results indicate that using combination of UV/ ferrate (VI) can improve the removal of both COD and Hydrogen sulfide.

Discussion

Ferrate (VI) production

In this study, the effect of the presence of a set of salt such as FeCl_2 , NaCl , BaCl_2 and MgCl_2 as well as NaOH on the rate of ferrate (VI) production was investigated. This effect is important because in real-world wastewater treatment applications, there exist constituents other than Na and Fe ions in ferrate (VI) production mixture, especially in case of using industrial-grade NaOH . The results are illustrated in Figure 4A. The amount of ferrate (VI) production can be increased (20%) by the addition of FeCl_2 to the electrolyte basic solution. Such an increase in ferrate (VI) production supports the idea that Fe^{6+} production is a two-step process. In first step, Fe^{3+} is produced and then converted to Fe^{6+} . Both steps occur on the surface of cathode electrode. Thus, the addition of Fe^{3+} to the solution can enhance the production rate. This mentioned

notion needs more detailed investigation to be confirmed. Another reason that can be attributed to such an increase in ferrate (VI) production rate is the rise in ionic strength due to the addition of an ionic compound. Increasing ionic strength can enhance electrical conductivity leading to an increase in electrical current and hence ferrate (VI) production rate (6). Nevertheless, this idea can be rejected by examining the results of ferrate (VI) production rate when ionic compounds other than FeCl_3 are used. For example, the addition of MgCl_2 and BaCl_2 deteriorated the production rate although the ionic strength of the solution was raised.

Temperature is an affecting parameter on the chemical reactions. In this study, a 14M solution of NaOH was electrolyzed for 15 minutes under various temperatures to investigate the effect of temperature on ferrate (VI) production. Figure 5B demonstrates that the increase in temperature in the range of 25 to 55°C led to an increase in ferrate (VI) production. Nevertheless, further increase in temperature led to a dramatic reduction of ferrate (VI) production rate. So, the optimum temperature for the production of ferrate (VI) employing electrolysis method is approximately 55°C.

Voltage is another effective parameter on the production of ferrate (VI) when utilizing the electrolysis method. The increase in voltage can increase the production rate of ferrate (VI) as demonstrated in Figure 4C. Increasing voltage results in increasing electrical current that passed through the circuit and hence ferrate (VI) production rate.

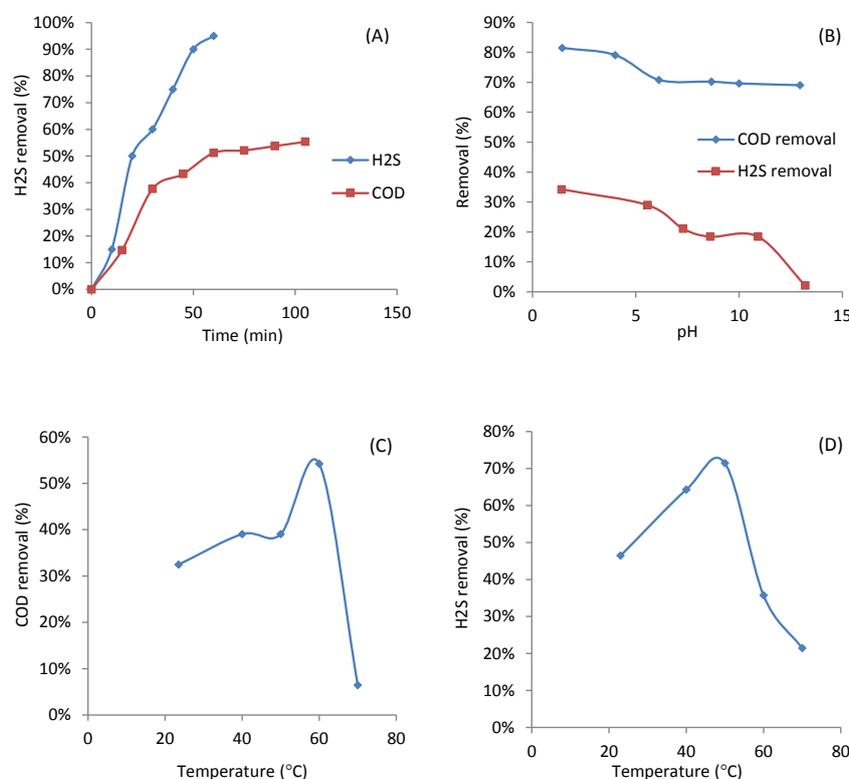


Figure 5. (A) Efficiency of hydrogen sulfide and COD removal over time; (B) Efficiency of hydrogen sulfide and COD removal at different pH values; (C) The variation of COD removal efficiency by using ferrate(VI) under different temperatures and (D) Variation of hydrogen sulfide removal efficiency by using ferrate(VI) under different temperatures.

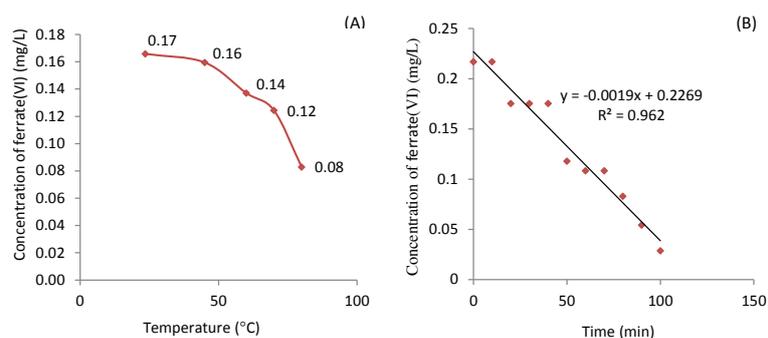


Figure 6. (A) Evaluation of ferrate(VI) degradation rate during 15 minutes under different temperatures and (B) evaluation of ferrate(VI) degradation under temperature of 23.5°C over time.

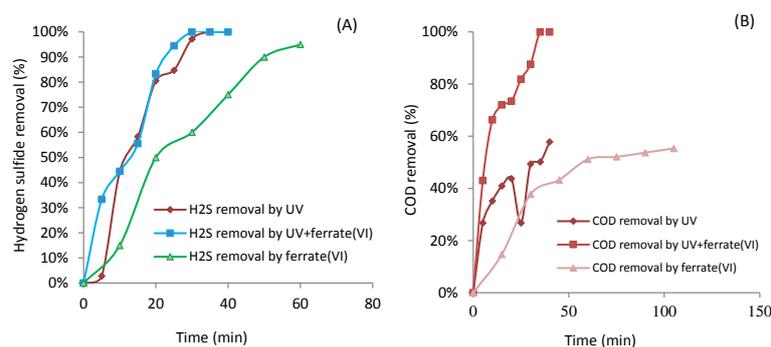


Figure 7. (A) Comparison of using UV, ferrate(VI) and their combination in the removal of hydrogen sulfide from municipal wastewater and (B) comparison of using UV, ferrate(VI) and their combination on COD removal from municipal wastewater.

Given that ferrate (VI) is a strong oxidizer, evaluating the effect of ferrate (VI) concentration on the removal of hydrogen sulfide and COD is highly important (4). As shown in Figure 4D, the removal efficiency of COD increases to a maximum value of about 60% when ferrate concentration reaches 1 mg/L. Nevertheless, further increase in oxidizer concentration has no significant effect on COD removal efficiency. This trend was not observed for hydrogen sulfide removal efficiency. The removal efficiency could reach about 100% when ferrate concentration increases to more than 1.5 mg/L. All results illustrated in Figure 4D were obtained for the residence time of 30 minutes.

Hydrogen sulfide removal and COD reduction

In the following sections, the results of the experiments pertaining to hydrogen sulfide and COD removals are discussed in terms of influencing parameters.

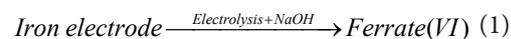
Hydraulic retention time

The contact time between an oxidizer and pollutants plays a critical role on the efficiency of pollutants' removal from wastewater (4). The results of this study demonstrate that hydrogen sulfide efficiency strongly depends on HRT (Figure 5A), and a large portion of this compound can be degraded after 50 minutes. Given that most damage caused by hydrogen sulfide occurs in wastewater collection systems, addition of adequate amount of ferrate (VI) to the starting point of the collection system can be

considered as a promising solution for the problems of corrosion and malodor of the hydrogen sulfide.

pH

As demonstrated in Figure 4B, pH has an adverse effect on the removal efficiency. It implies that in acidic environments, the reaction rate of removal process is higher. This fact can be explained by examining the degradation rate of ferrate (VI). According to reaction 1, ferrate (VI) is produced during electrolysis of iron electrode in the presence of NaOH and distilled water.



Production of ferrate (VI) can generate a purple color in water. Ferrate (VI) in water can appear in two forms, including FeO_4^{2-} and HFeO_4^- . According to Eq. 3, the rate of ferrate consumption can be found by adding the consumption rates of these two species:

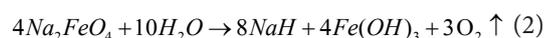
$$K[\text{Ferrate}] = K_1[\text{HFeO}_4^-] + K_2[\text{HFeO}_4^{2-}] \quad (3)$$

The rate constant for HFeO_4^- consumption ($K_1=1.24 \times 10^7$ M/S) is dominant when compared to that of FeO_4^{2-} ($K_2=8.41 \times 10^2$ M/S). Consequently, the reaction rate of HFeO_4^- and hydrogen sulfide is greater than that of FeO_4^{2-} (17). Therefore, HFeO_4^- is responsible for the removal of hydrogen sulfide rather than FeO_4^{2-} . In acidic environment, where pH is low, the dominant species is

HFeO_4^- ; while in basic conditions ferrate is mainly in the form of FeO_4^{2-} . Because the rate constant of ferrate acidic species degradation is much higher than the basic species, ferrate (VI) is much more effective in acidic condition.

Temperature

As shown in Figure 5C and 5D, the removal efficiencies increase as temperature increases to 50°C. Nevertheless, the removal efficiencies are reduced for higher temperature. This fact can be justified by examining the results shown in Figure 4B which is the variation of ferrate (VI) concentration versus temperature in ferrate (VI) production section. Ferrate (VI) is gradually converted to ferric (III) due to self-degradation according to reaction 2 (4):



The ferrate can be degraded due to both reactions with organic compounds and self-degradation. The rates of both reactions are accelerated by temperature. Nevertheless, as seen in Figures 4B and 6A, the self-degradation reaction speeds up significantly when the temperature exceeds 50°C. Thus, a significant portion of ferrate (VI) is consumed by self-degradation, and it decreases the contribution of ferrate to organic compounds degradation.

Ultraviolet radiation

UV radiation is a powerful means of degrading the molecules of organic compounds (15). In this study, the effect of UV radiation on the removal of hydrogen sulfide and COD from wastewater was investigated in the presence and absence of ferrate (VI) via a set of experiments. The results of these experiments are demonstrated in Figure 7A and 7B. As shown in Figure 7B, almost 100% of organic compounds was removed after 40 minutes when both UV and ferrate (VI) methods were used simultaneously. Nevertheless, when UV and ferrate (VI) methods were employed individually, 60% and 40% of hydrogen sulfide was removed, respectively, after the same time. Similar trend can be observed for the removal of hydrogen sulfide according to Figure 7A. These results show that UV and ferrate (VI) create synergy in the degradation process of organic compounds. Salari reported similar results for the removal of formaldehyde from synthetic wastewater by UV/ferrate (VI) (19). She reported that UV has a synergic effect on the removal of formaldehyde by ferrate (VI).

Conclusion

In this study, the removal of hydrogen sulfide and COD from municipal wastewater employing ferrate (VI) and UV was investigated. It was found that temperature, pH, HRT, ferrate (VI) concentration and UV radiation are effective factors in removing hydrogen sulfide and COD from municipal wastewater. The optimum condition for obtaining the highest hydrogen sulfide and COD removal is a pH value of less than 2, HRT of 60 minutes and a maximum temperature of 50°C. It was also concluded

that employing UV radiation in addition to ferrate (VI) enhanced the removal efficiency. Using UV radiation and a combination of UV/ferrate (VI) to remove hydrogen sulfide shows that the combination of both methods is able to remove almost 100% of hydrogen sulfide in only 30 min, while it takes 60 minutes to obtain the same results using ferrate (VI) individually. Hydrogen sulfide is a serious issue in wastewater collection and treatment systems causing not only the production of offensive odor but also severe corrosive condition. The results of this study demonstrate that the combination of ferrate (VI) and UV radiation can be considered as a promising method to resolve the issue of hydrogen sulfide in wastewater collection systems.

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Ethical issues

Authors are aware of, and comply with, best practice in publication ethics specifically with regard to authorship (avoidance of guest authorship), dual submission, manipulation of figures, competing interests and compliance with policies on research ethics. Authors adhere to publication requirements that submitted work is original and has not been published elsewhere in any language.

Competing interests

The authors declare that there is no conflict of interest that would prejudice the impartiality of this scientific work.

Authors' contributions

All authors of this study contributed equally for data collection, data analyses and manuscript writing.

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