



## Treatment of Iron from Groundwater by Ozonation: Influence of Hardness as a Scavenger

Apiradee Sukmilin<sup>1</sup>, Ratsamee Sangsirimongkolying<sup>2,\*</sup>

<sup>1</sup> Environmental Science and Technology Program, Faculty of Science and Technology,  
Phranakhon Rajabhat University, Bangkok 10220, Thailand

<sup>2</sup> Chemistry Program, Faculty of Science and Technology,  
Phranakhon Rajabhat University, Bangkok 10220, Thailand

\* Corresponding author: [gratsamee@gmail.com](mailto:gratsamee@gmail.com)

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### Abstract

This study assesses the efficiency of using ozone technology to treat iron from groundwater while hardness acts as a radical scavenger. Batch experiments were conducted to determine the effects of initial pH 6, 7 and 8, and hardness of 300, 400 and 500 mg L<sup>-1</sup> and kinetic analysis. The optimum conditions were found to be initial pH of 8 and hardness of 300 mg L<sup>-1</sup>. The removal efficiency of iron by ozonation was 99.97%, which was higher than that of air without ozone applied (83.67%). However, the presence of hardness and tert-butanol (TBA) had a negative effect on the oxidation of iron from groundwater. By increasing the carbonate hardness concentration, the oxidation rate of iron by ozone was reduced. The results of kinetic analyses indicated that the reactions of ozonation to remove iron from groundwater followed a pseudo-first order kinetic model with a rate constant of 0.0881 min<sup>-1</sup>, while air without ozone applied (aeration) was 0.0051 min<sup>-1</sup>. This finding suggests the potential use of ozone to remove iron from groundwater. Iron concentration from groundwater samples treated by ozonation have met the World Health Organization (WHO) guidelines (0.3 mg L<sup>-1</sup>).

**Keywords:** Groundwater; Hardness; Iron; Ozonation

### Introduction

Iron is one of the most abundant elements of the earth's crust. Groundwater easily gets contaminated with iron (II). The presence of iron in groundwater above a certain level makes the water unusable, mainly with regards to aesthetics such as giving the water a reddish color, metallic taste, odor and turbidity [1]. Moreover, iron

oxides, which are formed in reservoirs upon aerial oxidation of dissolved iron, promote growth of microorganisms in water. Thus, the World Health Organization (WHO) has set a guideline value of 0.3 ppm of iron in drinking water [2]. Chaibadan Piphat College, Phranakhon Rajabhat University is located in Lopburi, a province located in the central part of Thailand

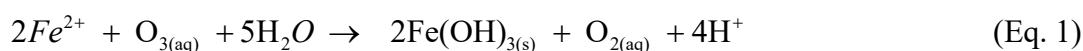
at 15°12'0"N latitude and 101°7'59.99"E longitude. This college has problem with water supply and drinking water due to drought. Frequently, the tap water does not flow. The surface water is also limited, leading to an insufficient water supply for use in the college. Thus, groundwater plays an important role in the water supply at Chaibadan Piphat College. However, the groundwater used in this college has a reddish color and distinct odor.

Conventional treatment methods such as ion exchange, water softening, adsorption and electrocoagulation are expensive [3]. Oxidation, which involves the transfer of electrons from iron (II) to iron (III), is an effective method. The most common chemical oxidants in water treatment are chlorine, chlorine dioxide and potassium permanganate [4]. Chlorination is also used for oxidation of divalent iron [5]. However, excess chlorine that remains in treated water may create problems such as trihalomethanes formation. Trihalomethanes are carcinogenic, mutant and toxic compounds with major negative effects to human health [6]. Potassium permanganate is normally more expensive than chlorine and must be carefully controlled. Permanganate can also form precipitates that cause mudball formations on filters. These are difficult to remove and compromise filter performance [7]. Another method of providing oxidation is to use the oxygen in the air as the oxidizing agent. No chemical dosing is required, which allows for unattended operation. This method is not effective for water in which the iron forms complexes with humic materials or other large organic molecules because oxygen is not strong enough to break the strong complexes formed between iron and large organic molecules [2].

Ozone can react with various organic and inorganic compounds in solutions in two ways, including direct molecular ozone with a redox potential of 2.07 V and radical-type reaction involving hydroxyl radicals induced by ozone decomposition with a redox potential of 2.33 V [8]. Ozone may be used for iron oxidation and it leaves no environmentally harmful residues. Iron (II) can be easily oxidized by ozone, which is shown in Eq. 1 [9].

Water hardness depends on the amount of calcium and magnesium cations in water. Hardness is mostly expressed as milligrams of calcium carbonate (CaCO<sub>3</sub>) equivalent per liter and also can be mentioned in terms of carbonate (temporary) and noncarbonated (permanent) hardness [10]. Hydroxyl radicals may react preferentially with carbonate and bicarbonate to produce radicals with weaker oxidation potentials. Therefore, if present, both carbonate and bicarbonate will behave as scavengers to eliminate indirect oxidation reactions in the ozonation processes [11]. The rate of ozone decomposition decreases in such conditions, resulting in a smaller ozone oxidation iron in the groundwater. However, the study of the influence of carbonate/bicarbonate in groundwater on oxidation of iron via ozonation is scarce.

For this reason, the objectives of this study are to study the influence of carbonate hardness as scavengers to remove iron by ozonation. Factors affecting iron removal by ozonation, including pH and hardness, were investigated. Reaction kinetics of ozonation were analyzed and compared with aeration. Radical scavenger studies were carried out by using TBA.



## Materials and methods

### 1) Materials

All reagents used in this experiment, including sodium hydroxide (Ajax), and nitric acid (Merck), were analytical grades. Sodium thiosulfate (Kemaus) was used to quench residual ozone, while potassium iodide (Gammaco) was used to determine ozone concentration and to trap unreacted ozone. tert-butanol (TBA) (Sigma), was used as a free radical scavenger to confirm the performance of hydroxyl radical ( $\text{OH}^\circ$ ) in oxidation of iron.

### 1) Study area of groundwater collection, preservation and characterization

Groundwater (20 L) was suddenly collected by grabbing from a groundwater pipe well at a depth of approximately 80 m at Chaibadan Piphat College, in Lopburi Province, which is located in central Thailand. The sampling point and topography of the study area are shown in Figure 1. The initial pH of the groundwater at Chaibadan Piphat College was 7.5. Groundwater samples were preserved by adding 6 M nitric acid until pH was 2. Iron concentration in groundwater was analyzed by an inductively coupled plasma optical emission spectrophotometer (ICP-OES, Perkin model optima 2100 DV).



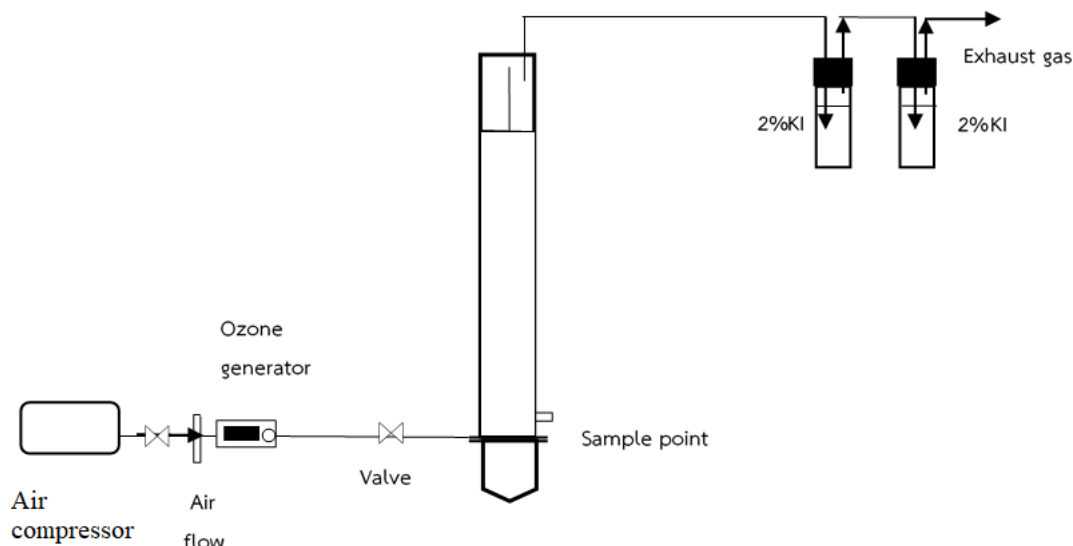
**Figure 1** The sampling point and topography of the study area.

### 3) Ozonation experiment

Ozonation experiments were carried out in batch mode using a cylindrical reactor. Ozone was generated by a laboratory ozone generator (ZON027), and oxygen was supplied from an air compressor (CS-51) as a reactant. The ozone generation rate measured by the iodometric method was  $0.57 \text{ mg min}^{-1}$ . In the ozonation procedure, 2.7 L of groundwater at an initial iron concentration of  $109 \text{ mg L}^{-1}$  was added to the cylindrical reactor. The pH of the solution was adjusted to 6, 7 and 8 using 1 M nitric acid and 1 M sodium hydroxide. Hardness concentration (The groundwater with initial hardness concentration of  $304 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$ ) was adjusted to 400 and  $500 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$  by adding calcium carbonate. The ozone flow was introduced into the reactor via air diffusers. Aliquot samples of 50 mL were collected at the specified time. A few drops of sodium thiosulfate solution were added to quench the residual ozone, while iron concentration was determined by using ICP-OES. All experiments were performed in triplicate. The schematic diagram of ozonation system is shown in Figure 2. Control experiments were conducted simultaneously with ozonation experiments and under the same conditions. The control was fed compressed air instead of ozone gas, which was excluded from the protocol. Data presented in figures and tables represent averages calculated from three initial values.

### 4) Effect of radical scavenger

TBA was used as a radical scavenger at varied concentrations of 1, 2 and 3 mM. A number of parameters were controlled, such as  $\text{O}_3$  generation =  $0.57 \text{ mg min}^{-1}$ , iron concentration =  $109 \text{ mg L}^{-1}$ , and pH 8. Data presented in figures and tables represent averages calculated from three initial values. Total hardness, total dissolve solid (TDS) and alkalinity were measured by standard methods for water and wastewater treatment [12].



**Figure 2** Schematic diagram of the ozonation system.

### 5) Analytical methods

The ozone generation rate was measured by iodometric method using 2% KI solution in absorption of ozone gas, and then subsequently titrated against sodium thiosulfate titrant. The total iron was determined by ICP-OES. The detection limit of iron by ICP-OES was  $0.1 \mu\text{g L}^{-1}$ . The instrument was calibrated using standard solutions. The curve obtained with regression coefficient,  $R^2 = 0.9999$ . Deionized water was used as blank. pH was measured by using pH meter (Eutech Model CyberScan pH1100).

## Results and discussion

### 1) Characterization of groundwater

In the study area, the groundwater iron concentration was found to be higher than the acceptable standard of  $0.3 \text{ mg L}^{-1}$  for drinking water. The chemical properties of the groundwater are presented in Table 1.

Based on previous research, the groundwater in Lopburi Province had a pH of 7.4–7.7 and hardness of  $210\text{--}330 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$  [13]. The most common sources of iron in groundwater occur naturally from the weathering of iron bearing minerals and rocks. Hardness concen-

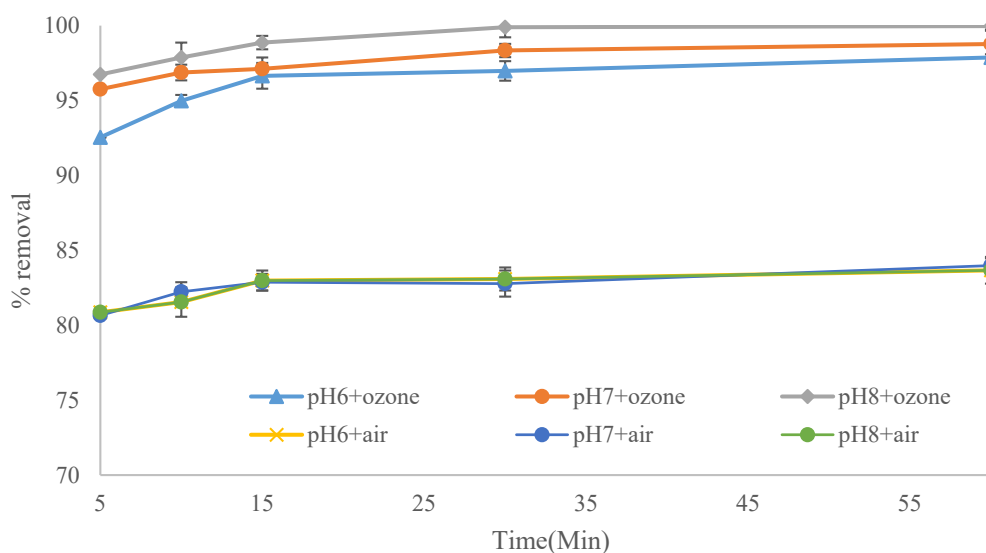
trations in natural water are up to  $100 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$ , depending on the associated sources [10]. The water quality hardness standard in Thailand divides hardness into following categories: soft water  $0\text{--}75 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$ , medium water  $75\text{--}150 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$ , hard water  $150\text{--}300 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$ , and very hard water  $300 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$ , and greater [10]. Thus, this groundwater at Chaibadan Piphat College is classified as very hard water. Alkalinity measures the ability of a solution to neutralize acids to the equivalence point of carbonate or bicarbonate. Alkalinity  $284 \text{ mg L}^{-1}$  referred to carbonate and bicarbonate dissolved in the groundwater. High iron concentration and total hardness not only effect health, but also contribute to inefficient and costly operation, which may cause scale deposition in the distribution system.

### 2) Effect of initial pH on iron removal

The pH of groundwater in Thailand is normally in the range of 6–8. The solution pH is an important factor affecting ozone decomposition because hydroxide ion ( $\text{OH}^-$ ) has been found to be an initiator of the chain reaction in ozone decomposition process [12].

**Table 1** Chemical properties of groundwater

Chemical properties	WHO maximum contaminant level for drinking water [22]	
Iron (mg L <sup>-1</sup> )	109	0.3
pH	7.5	6.5–8.5
Total hardness (mg L <sup>-1</sup> as CaCO <sub>3</sub> )	304	300
TDS (mg L <sup>-1</sup> )	557	500
Alkalinity (mg L <sup>-1</sup> )	284	200



**Figure 3** Effect of pH on oxidation of iron by ozonation and air (Experimental conditions: O<sub>3</sub> generation = 0.57 mg min<sup>-1</sup>, [iron] = 109 mg L<sup>-1</sup>).

As shown in Figure 3, when the solution pH increases, the oxidation of iron increases as well. At 60 min and pH of 6, 7, and 8, removal efficiencies of iron by ozonation were 97.88%, 98.77%, and 99.77%, respectively. At pH 6, 7 and 8, air oxidized iron, without ozone being applied, can remove 85.67% of iron. In the aeration process, there were no difference iron oxidation because air was not a strong oxidant and, the groundwater might contain natural organic matter. This organic matter might inhibit of iron oxidation by aeration [14] and iron with natural organic matter in the groundwater might not be transformed to ferric hydroxide by changing pH [15].

In the ozonation process, high pH can contribute to the decomposition of ozone, and, consequently, the generation of hydroxyl radicals

(OH<sup>•</sup>), which are very powerful radicals [16] that can oxidize iron efficiency because of high redox potential.

### 3) Effect of hardness on iron removal

The total hardness was one of the important parameters for assessing groundwater quantity, suitability for drinking/domestic uses, and treatment technologies. The high level of total hardness in the groundwater cause problems such as scale deposits in hot water pipes and difficulty in producing lather with soap. To determine the total hardness effect of ozonation, the total hardness concentration was studied at 300, 400 and 500 mg L<sup>-1</sup> as CaCO<sub>3</sub>, which is shown in Figure 4.

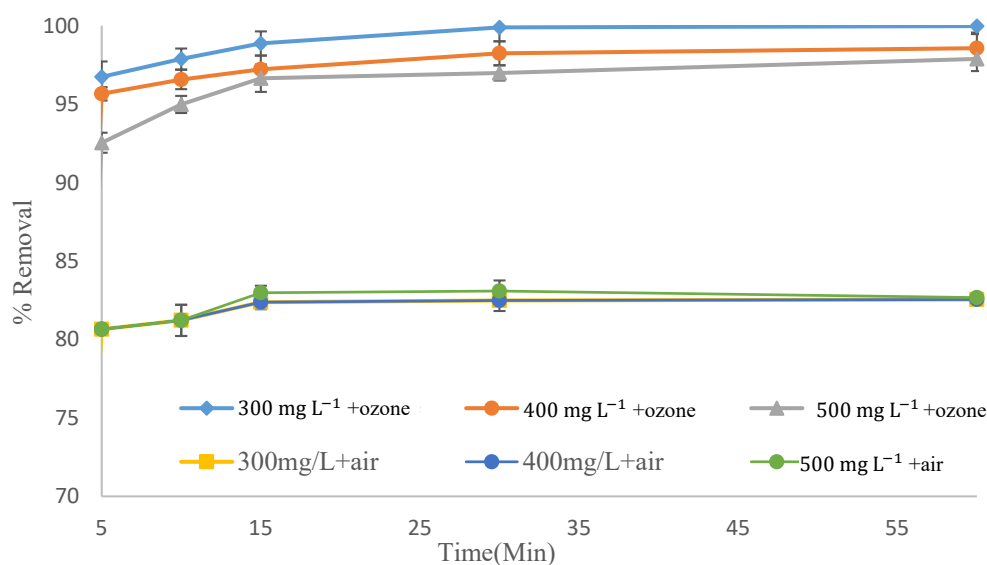
As shown in Figure 4, at 60 min and hardness of 300, 400, and 500 mg L<sup>-1</sup>, removal efficiencies of iron were 99.97, 98.57, and 97.87 %, respectively.

respectively. Hardness can be classified as carbonate and non-carbonate hardness. Carbonate hardness refers to calcium and magnesium bicarbonate. Non-carbonate hardness is caused primarily by calcium and magnesium nitrates, chlorides, and sulfates [17]. From Table 1, alkalinity was  $284 \text{ mg L}^{-1}$ , referred to carbonate and bicarbonate dissolved in the groundwater. While, non-carbonate was  $20 \text{ mg L}^{-1}$  as  $\text{CaCO}_3$ . For this reason, we focused on carbonate and bicarbonate effect in the ozonation. Regarding the previous study, carbonate and bicarbonate can act as ozone radical scavengers [18] that can act with hydroxyl radicals. When the hardness increased means high carbonate and bicarbonate in the groundwater. Thus, ozone decomposition decreased. So, the oxidation of iron decreased as well. According to Eq. 2 and Eq. 3, bicarbonate and carbonate are strong scavengers for hydroxyl radicals [18].

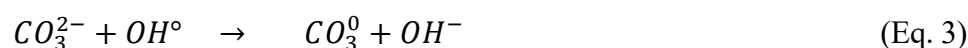
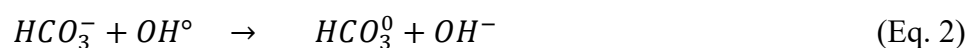
#### 4) Effect of radical scavengers on iron removal

In general, the key active species in ozonation at alkaline condition were  $\text{OH}^\circ$  generated during ozone decomposition. TBA is a commonly used hydroxyl radical scavenger [19] which was employed here to study the mechanism of iron oxidation during ozonation since it has a reaction rate constant of  $6 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$  with hydroxyl radicals and only  $3 \times 10^{-3} \text{ M}^{-1} \text{ s}^{-1}$  with ozone [20]. In this study, therefore, TBA was used as a radical scavenger at varied concentrations of 1–3 mM to prove the formation of  $\text{OH}^\circ$  from ozone decomposition and its performance on iron removal during ozonation with hardness.

Therefore, the ozonation process with high hardness slowed down the removal of iron from groundwater.



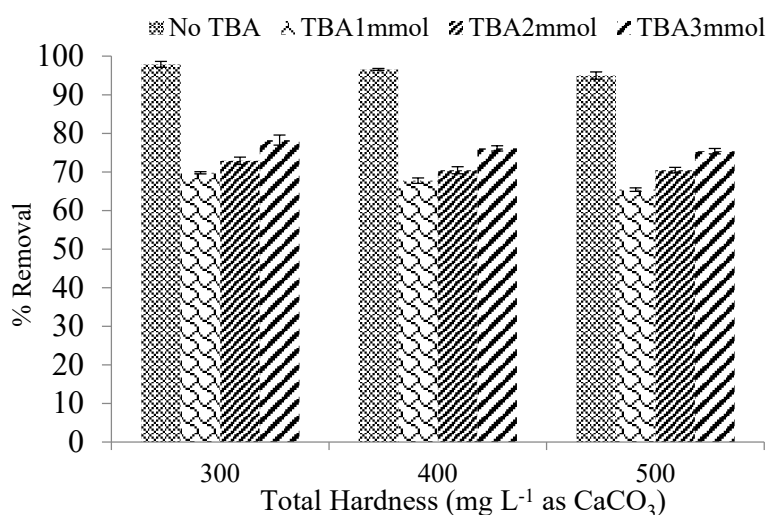
**Figure 4** Effect of hardness on oxidation of iron by ozonation and air (Experimental conditions:  $\text{O}_3$  generation =  $0.57 \text{ mg min}^{-1}$ ,  $[\text{iron}] = 109 \text{ mg L}^{-1}$ ,  $\text{pH}=8$ ).



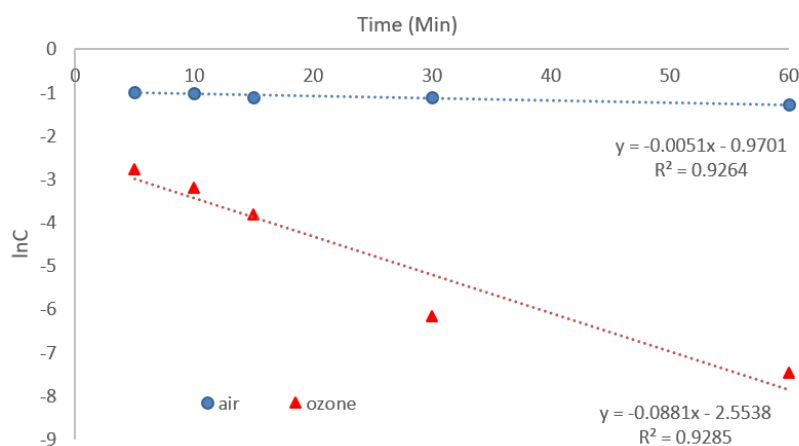
As shown in Figure 5, when the concentration of TBA increased, the percent iron removal simultaneously decreased. The findings confirm that hydroxyl radicals play an important role in oxidation of iron from groundwater. The inhibition imparted by TBA here indicates the iron removal efficiency is predominantly driven by hydroxyl radicals. Additionally, bicarbonate or carbonate can also react with hydroxyl radicals to decrease iron removal efficiency. The results indicate that the presence of TBA obviously inhibited the oxidation rate of iron in ozonation with carbonate hardness. This finding confirms that carbonate hardness acts as a hydroxyl radical scavenger in ozonation.

### 5) Reaction kinetics of ozonation

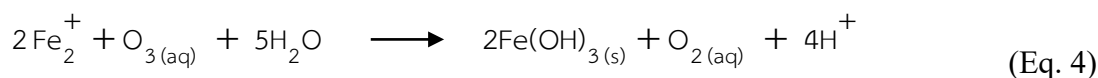
The reaction kinetic of ozonation and air without ozone applied was analyzed. Plots of  $\ln([C]/[C]_0)$  versus reaction time of the ozonation and aeration are shown in Figure 6. A good linear fit was observed for each of these ozonation reactions. The experimental data fit well with a pseudo-first order kinetic model [21], where  $[C]$  was iron concentration in molar. The value  $K_{obs}$  is an apparent reaction rate coefficient, which indicated that iron oxidation in both ozonation and air without ozone applied was described by the pseudo-first order kinetic model [12]. The oxidation rate of iron in both aeration and ozonation is expressed in Eq. 4 and Eq. 5.



**Figure 5** Effect of TBA in ozonation at various concentration of hardness (Experimental conditions:  $O_3$  generation =  $0.57 \text{ mg min}^{-1}$ ,  $[\text{iron}] = 109 \text{ mg L}^{-1}$ , pH 8).



**Figure 6** Kinetic study of iron oxidation by compared with air without ozone applied (Experimental conditions:  $O_3$  generation =  $0.57 \text{ mg min}^{-1}$ ,  $[\text{iron}] = 109 \text{ mg L}^{-1}$ , solution pH = 8).



$$\frac{d[\text{Fe}^{2+}]}{dt} = -K[\text{Fe}^{2+}] \quad K = k_H[\text{O}_2]_{\text{aq}} \quad (\text{Eq. 5})$$

According to the kinetic analysis results shown in Figure 6, the experimental data fits well with the pseudo-first order kinetic model with apparent rate constants ( $K_{\text{obs}}$ ) of  $0.0051 \text{ min}^{-1}$  and  $0.0881 \text{ min}^{-1}$  for the use of aeration and ozonation, respectively. The results confirm that the use of ozone is efficient for iron oxidation.

### 6) Portability of the treated water

To compare iron concentration with untreated groundwater, filtration, air without ozone applied, ozonation and maximum contaminant level for drinking water of WHO [22] are presented in Table 2.

The results from Table 2, shown that ozonation can oxidize iron in groundwater from Chaibadan Piphat College, Lopburi in order to meet the WHO guidelines for drinking water. The average iron concentration of the ozonation was  $0.28 \text{ mg L}^{-1}$ , meeting the maximum contaminant level for drinking water (less than  $0.30 \text{ mg L}^{-1}$  of iron) [22], while the average iron concentration of the aeration was still  $0.42 \text{ mg L}^{-1}$ , higher than the maximum contaminant level for drinking water. However, it should be

noted that the laboratory-scale test should thus be an integral part of the design process, and the technical and economic feasibility studies of ozonation in the full-scale application are required for further studies. To elucidate the application of ozonation for further treatment of groundwater in Chaibadan Piphat College, Phranakhon Rajabhat University, further studies on the pathogen inactivation efficiency are strongly recommended to validate the technical feasibility and its performance. For ozone applied in Chaibadan Piphat College are that it is no chemicals need to be added, greater disinfection against bacteria and virus, also effective oxidation with natural organic matter. However, to increase confidence for consumers, other parameters of drinking water should be examined. For example, manganese, fluoride and total organic carbon should be investigated. Those parameters are always higher than the standard with causing health effect and operation cost. For further study, ozonation should be apply in pilot scale. In addition, the efficiency of disinfection and economic aspect such as operation cost and investment cost should be investigated.

**Table 2** Comparing iron concentration of untreated groundwater, aeration, ozonation, and maximum contaminant level for drinking water

Parameter	Untreated groundwater	Aeration	Ozonation	Maximum contaminant level for drinking water [22]
Iron ( $\text{mg L}^{-1}$ )	109	0.42	0.28	0.30

### Conclusions

Ozonation was successfully used to oxidize iron from groundwater. At the optimum conditions, i.e. pH of 8, hardness of  $300 \text{ mg L}^{-1}$ ,

removal efficiency of iron was 99.97%, which is higher than air without ozone applied (85.67%). By increasing the hardness concentration, the oxidation rate of iron by ozone was



reduced. These results confirm that the oxidation of iron by ozone in groundwater follows a radical mechanism (TBA) and carbonate hardness also acts as a radical scavenger. Kinetic studies showed that the ozonation and aeration followed pseudo-first order kinetic models with the apparent rate constants ( $k_{obs}$ ) of 0.0881 and 0.0051  $\text{min}^{-1}$ , respectively. It can be concluded that ozonation is able to oxidize iron in groundwater effectively with a radical mechanism. Importantly, the iron concentration of the water after ozonation treatment met the WHO drinking water standard. The results of this research exhibit iron removal by ozonation which might be applied in the case of Chaibadan Piphat College, Phranakhon Rajabhat University, in Lopburi Province.

### Acknowledgements

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