Effects of fulvic acid and ferric hydroxide on removal of Fe$^{2+}$ and Mn$^{2+}$ by oxidation and aerated/submerged ultrafiltration membrane system

Turkan Ormanci Acar, Sevgi Gunes Durak and Nese Tüfekci

ABSTRACT

The purpose of this study is to evaluate the effects of fulvic acid (FA) and ferric hydroxide on the Fe$^{2+}$ and Mn$^{2+}$ removal by oxidation and aerated/submerged membrane systems. Firstly, the oxidation kinetics of Fe$^{2+}$ and Mn$^{2+}$ in the presence of FA and ferric hydroxide are discussed and then the contribution of the oxidation step in membrane experiments is determined. All experiments are conducted with synthetic drinking water and the concentration of contaminants is determined by characterization of two different drinking water sources in the city of Istanbul: Ömerli Dam Lake and Danamandira Village groundwater. The addition of ferric hydroxide helps to mitigate fouling and enhances the rejection of Mn$^{2+}$ by up to 90% by developing a secondary filtration layer on the membrane surface which has been considered a challenge in drinking water treatment plants.

Key words | drinking water, filtration, heavy metals, iron and manganese removal, membranes

INTRODUCTION

Iron and manganese are the most commonplace chemicals found existing together in natural water bodies. Although there is no reported health-based guideline value for iron, it may cause staining of laundry and plumbing fixtures (Barloková & Ilavský 2010) when its concentration exceeds 0.3 mg/L (World Health Organization 2011). For manganese, concentrations below 0.1 mg/L are generally acceptable to consumers (World Health Organization 2011), but above this concentration, manganese causes pipe clogging, laundry staining and unpleasant taste (Bamforth et al. 2006; Mak & Lo 2011). Also the health-based value for manganese concentration is 0.4 mg/L (World Health Organization 2011).

Natural organic matter (NOM) is a ubiquitous component of surface and groundwater sources as well. In particular, fulvic acid (FA) is one of the predominant fraction of NOM because of its higher solubility and stability in water sources (Reckhow et al. 1990; Tang et al. 2014a, 2014b). It is known that NOM affects many chemical and biological processes in drinking water treatment plants and their designs, mostly through the complexion of metals (Li et al. 2012) and adsorption of organics by its various functional groups (such as carboxyl, phenol, hydroxyl and amine).

Formation of iron-NOM and manganese-NOM complexes can prevent oxidation of Fe$^{2+}$ and Mn$^{2+}$. In the case of complexation, oxidation of Fe$^{2+}$ and Mn$^{2+}$ is prevented and it becomes quite difficult to remove Fe$^{2+}$ and Mn$^{2+}$ by conventional filtration (Tang et al. 2014a, 2014b). Furthermore, NOM plays an important role in the formation of potentially harmful disinfection by-products (DBPs). Humic substances in the natural waters are especially reactive to a variety of oxidants and disinfectants that are used in the purification process of drinking water. Chlorine is one of the most commonly used disinfectants in treatment plants that is susceptible to the production of carcinogenic trihalomethanes, haloacetic acid, and other halogenated DBPs in reaction with humic substances (Bellar et al. 1974; Reckhow et al. 1990; Oxenford & Amy 1996).
Membrane separation technology, as an alternative method to conventional separation technology, has become increasingly popular in the elimination of FA (Tang et al. 2014a, 2014b) and metals such as Fe$^{2+}$ and Mn$^{2+}$ from drinking water sources (Choo et al. 2005; Mamtani et al. 2014), but it suffers from drastic flux decline due to fouling. Mamtani et al. (2014) studied membrane fouling in a cross flow capillary UF membrane by four different foulants including iron, manganese, sodium alginate and humic acid. The fouling was rated from highest to lowest in the sequence of sodium alginate, humic acid, iron and manganese. So, instead of using the (pressurized) membrane alone, it has been utilized simultaneously with a combination of conventional processes such as aeration, coagulation and adsorption as a submerged membrane system in order to overcome such problems and to enhance its performance (Côté et al. 1998; Farahbakhsh & Smith 2002; Choi et al. 2008; Gray 2008; Kimura et al. 2008).

Côté et al. (1998) compared two different filtration processes including low-pressurized membrane-based treatment and an immersed membrane in an oxidation tank by studying the results for iron and manganese removal. Although little membrane fouling was observed in the low-pressurized membrane system at temperatures as low as 1°C, greater removal efficiency of manganese and lower energy consumption were observed for the immersed membrane system.

In another study, Rahman et al. (2000) investigated the effects of aeration, pH increment, and H$_2$O$_2$ coupled with UV irradiation on the removal of manganese by a pressurized Microfilter membrane and found that high manganese removal efficiency could be achieved with an increased pH of about 9.7, whereas the removal percentage was reduced when the mechanical aeration was applied.

Reaching an optimum state in removing manganese has remained a challenge for both the membrane based process and non-membrane based process. The problems could be fouling (Chen et al. 2014; Nerger et al. 2015), high energy consumption costs (Singh 2015), high chemical usage (Sharma 2014), and feasibility issues (Bandyopadhyay 2016).

In this study, the adsorption ability of ferric hydroxide magnetic particles was used in an aerated/submerged ultrafiltration membrane system in order to help solve these problems. Ferric hydroxide magnetic particles were used by Funes et al. (2014) as a separating agent for removal of manganese as high as 95% even at pH < 9. So, to the knowledge of the authors it is the first time that magnetic particles have been utilized in a submerged-membrane system to overcome the associated issues. In this context, the contribution of ferric hydroxide and FA to iron and manganese removal was investigated for three-day periods in aerated/submerged membrane systems and the results of Fe$^{2+}$ and Mn$^{2+}$ removal and fouling were analyzed by measuring pressure, dissolved organic carbon (DOC), UV$_{254}$, and iron and manganese concentrations.

**MATERIALS AND METHODS**

**Chemicals**

FeSO$_4$.7H$_2$O and MnCl$_2$.2H$_2$O were used to prepare 1 g/L Fe$^{2+}$ and Mn$^{2+}$ stock solutions respectively. Suwannee River fulvic acid (SRFA) was supplied from the International Humic Substances Society, University of Minnesota, and 50 mg/L ferric hydroxide stock solution was obtained by aerating Fe$^{2+}$ for 3 or 4 hours with an alkalinity of 8×10$^{-3}$ eq/L.

**UF membrane module**

Zee Weed-10 (ZW-10) membrane was purchased from GE Water & Process Technologies. The properties of ZW-10 membrane are given in Table 1. For each trial different membrane modules were prepared from the ZW-10 UF

<table>
<thead>
<tr>
<th>Membrane type: capillary, hydrophilic</th>
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<tr>
<td>Surface area, m$^2$</td>
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<td>Membrane material</td>
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<tr>
<td>Nominal membrane pore size, nm</td>
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<tr>
<td>Capillary outer diameter, mm</td>
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<td>Capillary inner diameter, mm</td>
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<tr>
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<tr>
<td>Transmembrane pressure</td>
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<td>Flux, L/m$^2$sa</td>
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membrane module. The surface area of the membrane was calculated as 0.0235 m² and 12 fibers were used to prepare the membrane module.

**Experimental procedure of oxidation**

The oxidation of Fe²⁺ and Mn²⁺ was studied in a batch scale reactor, and the effects of FA and ferric hydroxide were determined by the oxidation experiment. Iron, manganese and FA were selected because they are common in drinking water sources and are difficult to treat. The experimental setup used in this study is given schematically in Figure 1(a).

The reaction vessel was vigorously mixed using a Yellow Line OST 20 Basic type of mixer. NaHCO₃ was added to the distilled water to obtain a solution with an alkalinity equal to 8 × 10⁻³ eq/L. Air was added into the solution using fine bubble diffusers. The pH of the solution was measured by a JENWAY model 3040 type of ion analyser unit and kept constant at pH 8.5. The dissolved oxygen levels were monitored using a WTW Oxi 538 oxygen meter. Temperature was kept constant at 25 °C by immersing the reaction vessel in a water bath (ORDEL OC770). Samples were taken at the specific time interval and filtered with 0.22 μm membrane filters before the measurement. The concentration of Fe²⁺ was determined by a UV-Visible spectrophotometer according to the method of Tamura et al. (1976). The Mn²⁺ concentration was determined by an inductively coupled plasma–mass spectrometer (ICP-MS).

The experimental combinations for oxidation are given in Table 2.

**Experimental procedure of membrane filtration**

Membrane experiments were conducted with synthetic solutions. Every experiment lasted for three days and before the experiments feed solution had been aerated for a day along with adding NaHCO₃ to obtain a solution with an alkalinity equal to 8 × 10⁻³ eq/L. Synthetic solutions were fed into a 10 × 20 × 45 cm-sized Plexiglas reactor by a peristaltic pump. During the experiment, air was provided at the bottom of the reactor. Backwash was maintained through feedback of filtered water with a backwash pump.

![Figure 1](https://iwaponline.com/ws/article-pdf/17/6/1712/204711/ws017061712.pdf)
The pH of the solution was measured by a JENWAY model 3040 type of ion analyser with a sensitivity of 0.001 pH unit and kept constant at pH = 8.5 during a day of aeration and experiment. Pressures were measured by a Hach Lange SC 1000 data logger and transferred to the computer every five minutes. The samples were taken every day regularly from the feed tank, raw water and inside the reactor and permeate, and filtrated with 0.22 μm filter paper. DOC (dissolved organic matter) and UV254 were measured in the raw and treated water to evaluate the FA removal. DOC was measured with a Schimadzu TOC-VCPH instrument using the high-temperature combustion method (5310B) (Standard Methods for the Examination of Water and Wastewater 2005). UV254 adsorbance was measured at 254 nm wavelength by a Perkin Elmer Lambda 25 UV visible spectrophotometer. Inductively coupled plasma optical emission spectrometry (ICP-OES) was used to determine iron and manganese concentrations. The aerated/submerged membrane setup is given in Figure 1(b).

In order to evaluate the independent impact of FA and ferric hydroxide, different combinations of iron, manganese, FA and ferric hydroxide were used for the experiments and are given in Table 2.

### RESULTS AND DISCUSSION

#### Seasonal characterizations of Ömerli Dam Lake and Danamandıra village drinking water sources

Danamandıra village (DV) and Ömerli Dam (ÖD) drinking water sources were selected, and were seasonally characterized to determine the composition of synthetic water. DV is specified as a good reference for a groundwater source and ÖD for surface drinking water. Specific and relevant parameters which are important for drinking water were analyzed and the results are given in Table 3.

These results were taken as a reference to minimize the number of experiments and select the best representative concentrations of FA, iron and manganese. Experimental combinations and concentrations of contaminants which are given in Table 2 were determined. Iron and manganese

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### Table 2

<table>
<thead>
<tr>
<th>Combination of iron, manganese, FA and iron hydroxide for oxidation and membrane filtration experiments</th>
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<td>Oxidation experiments</td>
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<td>Aerated/submerged membrane experiments</td>
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### Table 3

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<td>7.57</td>
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<td>7.8</td>
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<td>2.9</td>
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<td>2.1</td>
<td>2.3</td>
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<td>Total alkalinity (mg/L CaCO₃)</td>
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<td>181</td>
<td>64</td>
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<td>79</td>
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<td>TOC (mg/L)</td>
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<tr>
<td>Total Fe (mg/L)</td>
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<td>1.0</td>
<td>0.642</td>
<td>1.1</td>
<td>1.6</td>
<td>0.96</td>
<td>0.93</td>
<td>1.05</td>
<td>0.96</td>
<td>1.1</td>
<td>0.84</td>
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<tr>
<td>Total Mn (mg/L)</td>
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<td>0.99</td>
<td>0.9</td>
<td>1.4</td>
<td>1.9</td>
<td>1.8</td>
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<td>1.5</td>
<td>1.1</td>
<td>2.08</td>
<td>0.99</td>
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</tr>
<tr>
<td>UV254 (1/cm)</td>
<td>0.196</td>
<td>0.06</td>
<td>0.164</td>
<td>0.05</td>
<td>0.247</td>
<td>0.044</td>
<td>0.261</td>
<td>0.045</td>
<td>0.251</td>
<td>0.034</td>
<td>0.157</td>
<td>0.055</td>
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</table>
were measured as roughly equal to 1 mg/L. UV254 was found to be between 0.034 and 0.247 which is equal to concentrations of almost 1 and 7 mg/L of FA. When preparing the feed solution, manganese and FA were kept at their original concentrations of 1 mg/L and 1–7 mg/L, respectively. As it is hard to measure Fe2+ at 1 mg/L due to its rapid oxidation rate under a pH of 8.5, it was decided that the concentration in the feed solution should be 5 mg/L for better observation of its oxidation.

Removal of Fe2+ and Mn2+ by oxidation

The oxidation rate of iron is rapid in the range of pH 6.5–6.7 (Tüfekci & Sarıkaya 1996). The pH in this study was chosen to be 8.5 to simulate actual drinking water treatment conditions. The reaction rate constant and reaction time for 5 mg/L Fe2+ at pH 8.5 were calculated as 1.3821 and 3.6 min respectively from the slopes of the lines on semi-logarithmic plots of Fe2+ versus time. As can be seen from Figure 2, in the case of high pH (pH = 8.5), the oxidation rate of Fe2+ was found to be quite high.

Several factors such as Fe2+ concentration, oxygen concentration, pH, temperature, presence of organic matter and other ions in solution affect the oxidation of Fe2+. It has been demonstrated that many organic compounds are capable of significantly affecting the rate of Fe2+ oxidation (Jobin & Ghosh 1972; Theis & Singer 1974). Other research has revealed that the addition of organics changes the kinetics remarkably, reducing the half-life of Fe2+ from hours to minutes (Pham et al. 2004). In contrast to previous studies, addition of FA or ferric hydroxide has negligible effects on the oxidation of Fe2+. According to the results of the reaction rate constant (k) and reaction time (Table 4), adding FA or ferric hydroxide did not affect the reaction time or the rate constant (k) remarkably.

According to Figure 3 which represents the removal rate of Mn2+, it was confirmed that the removal rate of Mn2+ was notably low which is in parallel with the fact that oxidation of Mn2+ is likely to be negligible at pH lower than 9 (Aydin et al. 2001), and it increased with the addition of FA and ferric hydroxide (see also Table 4). As ferric hydroxide has a negative charge at a pH of 8.5 so it can be inferred that the reduction in Mn2+ concentration happened not only by oxidation but also by adsorption. This adsorption originates from its amphoteric functional group (–OH) and it brings electrokinetic properties to the ferric hydroxide surface (Funes et al. 2014).

Removal of Fe2+ and Mn2+ by the aerated/submerged membrane reactor

The three-day removal efficiency of Fe2+ is shown in Figure 4. As mentioned above, oxidation of Fe2+ at pH 8.5 was found to be extremely high. So the contribution of membrane filtration to Fe2+ removal could be ignored.

Oxidative removal of Mn2+ with O2 occurs eventually in homogeneous solutions at pH 8. But at this pH, the half-life for Mn2+ removal may be estimated to be as long as 200 to 300 days (Morgan 2005). In this study Mn2+ removal efficiency was found to be extremely low, about 11–19%.
without the contribution of Fe\(^{2+}\), FA and ferric hydroxide (Figure 5). However, adding 5 mg/L Fe\(^{2+}\) enhanced Mn\(^{2+}\) removal by up to 50% on the third day of operation. This enhancement could be related to the Mn\(^{2+}\) adsorption capacity of ferric hydroxide generated by the aeration of the operation tank. Fe\(^{2+}\) can be found in drinking waters as a dissolved form in the presence of NOM or as a precipitated form which is formed by oxidation of inorganic hydroxyl complexes of Fe\(^{2+}\) (Pham et al. 2004). The effect of FA as organic matter on Mn\(^{2+}\) removal is negligible. Although a general enhancement was observed by adding FA and Fe\(^{2+}\), the organic matter did not have a positive effect on the removal of Mn\(^{2+}\) as its removal declined from 36% to 30% by increasing the FA concentration from 1 mg/L to 7 mg/L. Consequently it is assumed that the increment of Mn\(^{2+}\) removal originated mostly from the presence of the ferric hydroxide which occurs by oxidation of Fe\(^{2+}\). The highest Mn\(^{2+}\) removal efficiency was achieved when the feed solution contains 5 mg/L Fe\(^{2+}\), 1 mg/L Mn\(^{2+}\), 1 mg/L FA and 50 mg/L Fe(OH)\(_3\). The most effective additive was found to be ferric hydroxide (Fe(OH)\(_3\)) and almost 92% Mn\(^{2+}\) removal was observed. We estimate that this phenomenon originated from the electrokinetic surface characteristics of ferric hydroxide particles.

### Removal of fulvic acid by the aerated/submerged membrane reactor

In this study, FA concentration was determined by measuring DOC and UV. Results of DOC removal are given in Figure 6. No considerable changes were found in DOC removal by increasing the concentration of FA. However the presence of iron and manganese, especially iron, increased the removal efficiency of DOC. The highest removal efficiency (60%) was obtained by adding 50 mg/L ferric hydroxide to 5 mg/L Fe\(^{2+}\), 1 mg/L Mn\(^{2+}\) and 1 mg/L FA. As expected, the ferric hydroxide affected the removal efficiency in a positive way.

FA removal by UV (254 nm wavelength) is illustrated in Figure 7. The results of UV\(_{254}\) demonstrated greater removal of FA in comparison with the results from DOC measurements. This could be attributed to strong UV absorbance of aromatic groups of FA at 254 nm (Ha et al. 2004). Also, sorption abilities of iron and ferric hydroxide on removal of FA were testified from the results of UV\(_{254}\) removal.
Almost 67% UV$_{254}$ removal rate was observed for solutions containing ferric hydroxide.

Membrane fouling mechanism and pressure gradient change

Pressure change is an instantaneous indicator of membrane fouling (Figure 8). Two types of fouling mechanism were observed in this study. The first one occurred through clogging of the membrane’s pores. According to the results, Mn$^{2+}$ was the main source of this type of fouling. The membrane pressure change was reached at approximately 200 mbar in the presence of Mn$^{2+}$, as Mn$^{2+}$ does not oxidize precisely at pH 8.5 and it can be readily passed through the membrane pores and occludes the pores. Because of Mn$^{2+}$, a thin and thick cake layer occurred on the surface of the membrane (see Figure 9(b)) and that layer could not be removed by backwashing in operation time. So an irreversible fouling occurs and there is no possible method to clean this membrane without causing damage. However in the presence of iron, the fouling effect of Mn$^{2+}$ diminished to approximately 75–80 mbar. Formation of iron (ferric hydroxide) prevented membrane fouling by sorbing Mn$^{2+}$. FA was also responsible for irreversible fouling. Therefore FA was found to be the second most important pollutant for membrane fouling, at $\approx$ 150 mbar at 7 mg/L FA concentration and $\approx$ 60 mbar at 1 mg/L FA concentration (Figure 8). Scanning electron microscope (SEM) images of 1 mg/L FA (Figure 9(d)) and 7 mg/L FA (Figure 9(e)) show that the increase in FA concentration thickened the membrane cake and increased the fouling. However, in the presence of iron, manganese and FA together, the pressure change of the feed solution, which consists of 1 mg/L and 7 mg/L FA, was decreased to $\approx$ 50 mbar and $\approx$ 70 mbar respectively (Figure 8). This phenomenon can be explained by the oxidation of Fe$^{2+}$ and the ferric hydroxide formation and possible iron and FA complexation.
The second type of fouling occurred on the surface of the membrane and it can be removed easily with backwashing or striking. The rapid oxidation ability of Fe$^{2+}$ at pH 8.5 generated surface fouling. In the presence of Fe$^{2+}$, 40–60 mbar pressure changes were obtained and it was found to be quite low. But the presence of Mn$^{2+}$ along with Fe$^{2+}$ augmented membrane fouling to $\approx$80 mbar. Formation of ferric hydroxide or adding prepared ferric hydroxide prevented fouling also in some instances; it served as a second membrane layer (Figure 9(f) and 9(e)). Addition of ferric hydroxide (Fe(OH)$_3$) to feed solutions which contain Fe$^{2+}$, Mn$^{2+}$ and FA at 1 or 7 mg/L scaled down the pressure change to 40 and 50 mbar respectively.

According to SEM images, a thick and dense cake layer occurred in the presence of ferric hydroxide. From an

Figure 9  |  SEM images of membranes after membrane filtration experiments: (a) 5 mg/L Fe$^{2+}$, (b) 1 mg/L Mn$^{2+}$, (c) 5 mg/L Fe$^{2+}$ – 1 mg/L Mn$^{2+}$, (d) 1 mg/L FA, (e) 7 mg/L FA, (f) 5 mg/L Fe$^{2+}$ – 1 mg/L Mn$^{2+}$, (g) 5 mg/L Fe$^{2+}$ – 1 mg/L Mn$^{2+}$ – 7 mg/L FA, (h) 5 mg/L Fe$^{2+}$ – 1 mg/L Mn$^{2+}$ – 1 mg/L FA – 50 mg/L Fe(OH)$_3$, (i) 5 mg/L Fe$^{2+}$ – 1 mg/L Mn$^{2+}$ – 7 mg/L FA – 50 mg/L Fe(OH)$_3$. 

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evaluation of all the results, it can be inferred that membrane fouling was largely due to the removable cake layer.

CONCLUSION

In this paper, the effects of FA and ferric hydroxide (Fe(OH)₃) on Fe²⁺ and Mn²⁺ removal were investigated. Firstly, oxidative removals of Fe²⁺ and Mn²⁺ were examined and in the light of this conventional method, an aerated/submerged hybrid membrane system was designed and operated. As oxidation of Fe²⁺ was found to be high at a pH of 8.5, the membrane separation process step can be skipped for Fe²⁺ removal. On the other hand, iron gained electrokinetic features by oxidation and ferric hydroxide was produced. Ferric hydroxide can be evaluated as a sorbent material, and makes a contribution to removal of Mn²⁺.

Mn²⁺ oxidation was found to be notably slow at a pH of 8.5. Complexation with FA improved removal efficiency and the sorption ability of ferric hydroxide raised the Mn²⁺ removal rate up to almost 92%. In addition to iron and manganese removal, it was important to remove FA which is followed by measuring DOC and UV₂₅₄. Because of aromatic groups of FA, UV₂₅₄ removal efficiency (67%) was found to be higher than DOC (60%) in the presence of ferric hydroxide.

By analyzing the results, it can be concluded that ferric hydroxide had a catalytic effect on aeration, in addition to adsorptive removal. Ferric hydroxide reduced membrane fouling by abatement in increasing the vacuum pressure gradient. So, it could be said that ferric hydroxide tends to prolong the useful life of membranes and enables important removal efficiencies for iron, manganese and FA (UV₂₅₄ and DOC) at drinking water treatment plants. For future studies, ferric hydroxide can be utilized to prevent membrane fouling and can be tested for other metals in drinking waters.

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