Influence of nitrite on the removal of Mn(II) using pilot-scale biofilters
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ABSTRACT
Two pilot-scale biofilters were used to systematically investigate the influence of nitrite on biological Mn(II) removal. Gibbs free energy change (ΔG) of the redox reaction between MnO₂ and NO₂⁻ was 122.28 kJ mol⁻¹ in 298 K, suggesting that MnO₂ could not react with NO₂⁻. When nitrite in the influent was increased from 0.05 to 0.5 mg L⁻¹, manganese oxides did not react with nitrite in anaerobic conditions; nitrite was quickly oxidized and biological Mn(II) removal was slightly affected in 2 h in aerobic conditions. When nitrite was accumulated in the biofilter by increasing ammonia concentration, nitrite existed for more than 3 d and biological Mn(II) removal was affected in 3 d. When Mn(II) and ammonia in the influent were about 2 and 1.5 mg L⁻¹, respectively, both of them were completely removed and the oxidation-reduction potential was increased with the depth of the filter from 16 to 122 mV. Biological Mn(II) removal followed the first-order reaction, and the k-value was 0.687 min⁻¹.

INTRODUCTION
Groundwater is often mildly acidic and devoid of dissolved oxygen (DO) (Azher et al. 2008), so when groundwater flows through soils, minerals and rocks, soluble Fe(II) and Mn(II) are present (Jusoh et al. 2005), either in dissolved mineral form, or associated with various organics, minerals or chelating agents. In addition, the predominant form of Mn(II) at low or neutral pH values is Mn²⁺, which occurs primarily as a free cation in natural waters (Nealson et al. 1988). Continuously increasing ammonia concentration in groundwater has been observed in the past years, owing to the discharge of waste from both industry and bank-side residents without adequate pre-treatment and sub-optimal conditions of the catchments (Okoniewska et al. 2011; Akkera et al. 2012).

When Fe(II) and Mn(II) are present in drinking water at concentrations exceeding the permitted limits of 0.2 and 0.05 mg L⁻¹, respectively (Tekerlekopoulou & Vayenas 2007), Fe(II) and Mn(II) are objectionable for the following reasons: (a) Fe(II) and Mn(II) give a metallic taste in water systems (Azher et al. 2008); (b) iron and manganese deposits build up in pipelines reducing the pipe diameter in the distribution systems and eventually clog the pipe; (c) in water distribution systems, Fe(II) and Mn(II) are substrates for bacteria growth (Azher et al. 2005), hence when the bacteria die and slough off, bad odors and unpleasant tastes may be produced (Kontari 1988; Gouzinis et al. 1998). In addition, when Mn(II) exceeds the permitted limit, Mn(II) has been found to affect the central nervous system (Sharma et al. 2003). The presence of ammonia in drinking water treatment could affect the chlorination process and Mn(II) biofiltration system (Hasan et al. 2013). Ammonia will react with chlorine to form disinfection by-products (Richardson & Postigo 2012), which could damage the human nervous system (Nieuwenhuijsen et al. 2009), cause a deterioration in the taste and odor of water (Richardson et al. 2007), and reduce the disinfection efficiency (WHO 1996). Furthermore, ammonia can interfere with the Mn(II) biofiltration process by consuming excessive...
oxygen during nitrification, resulting in moldy and earthy tasting water (WHO 1996). So groundwater which contains high concentrations of Fe(II), Mn(II) and ammonia needs to be treated before it is used for industry and humans. Chemical methods could be used to oxidize Fe(II), Mn(II) and/or ammonia, but chemical oxidation may produce potential hazardous by-products and/or introduce other pollutants into the produced water. Moreover, it is difficult to simultaneously remove ammonia and manganese with only one chemical (Han et al. 2013). The advantages of the biological oxidation process over the conventional physicochemical methods include high filtration rates, and low operation and maintenance costs. As single stage filtration is used, it is not necessary to provide additional chemicals, and the volume of the generated sludge is appreciably smaller and easier to handle (Pacini et al. 2005; Han et al. 2013).

It is reported that achieving simultaneous removal of ammonia and Mn(II) would be very difficult (Hasan et al. 2012; Han et al. 2013), since biological Mn(II) removal can only take place after complete nitrification because of the necessary evolution of the oxidation-reduction potential (ORP) (Frischherz et al. 1985; Vandenabeele et al. 1995; Hasan et al. 2012). Thus the start-up period of biofilters for Mn(II) and ammonia removal needs 3–4 months (Frischherz et al. 1985), while the biofilters for Mn(II) removal is only 1–2 months (Frischherz et al. 1985; Vayenas et al. 1997). It is not clear how biological Mn(II) removal and biological ammonia removal are linked. A better understanding of the interactions between the two phenomena is important from an economic point of view (Vandenabeele et al. 1993). Nitrification (biological ammonia oxidation) is carried out by two different consecutive microbial processes, nitritification and nitratification (Vayenas et al. 1997). In nitritification process, ammonia is oxidized to nitrite by the bacterial genera Nitrosomonas; and in nitratification process, Nitrobacter converts nitrite to nitrate. In addition, Nitrosonomas and Nitrobacter are aerobic and autotrophic bacteria. The nitritification rate is faster than the nitratification rate, resulting in nitrite accumulating in the biofilters during the start-up period. Vandenabeele et al. (1995) investigated the influence of nitrite on Mn(II) removal using PYM medium (Ehrlich & Zapkin 1989); however, few researchers have investigated the influence of nitrite on Mn(II) removal using a biofilter.

In this study, the $\Delta G$ of the redox reaction between MnO$_2$ and NO$_2$ was calculated. Two pilot-scale biofilters were established to investigate the redox reaction between MnO$_2$ and HNO$_2$ in anaerobic conditions, and the influence of added and generated nitrite on biological Mn(II) removal in a biofilter. The main objectives of this study were to verify whether the reaction between MnO$_2$ and NO$_2$ could occur, and find the reason why the start-up period of biofilters for Mn(II) and ammonia removal was much longer than the biofilters for Mn(II) removal.

**MATERIALS AND METHODS**

Two pilot-scale biofilters were developed in a groundwater treatment plant (GWTP), which is located in Harbin city, P.R. China. The height and diameter of filters 1 and 2 were $300 \times 25$ and $300 \times 15$ cm, respectively, and the effective working volume was 74 and 26 L, respectively (Figure 1). At the top of each filter, the incoming waters were firstly mixed in the mixing chamber and then they flowed into the filter. Meanwhile, at the bottom of each filter, an underdrain system was used to collect the treated water and any biological solids which detached from the media. Along each filter depth there were 20 sampling ports at 10 cm intervals for Fe(II), Mn(II), ammonia, nitrite and ORP concentration measurements in the bulk liquid. Tank 1 (volume was 2,000 L) was used to collect raw groundwater (DO was about 0.2 mg L$^{-1}$) or aerated raw groundwater (DO was about 8.5 mg L$^{-1}$), which were obtained from the GWTP. Tank 2 (volume was 200 L) was used to collect the effluent water of filter 1. In order to increase the concentration of Mn(II), nitrite and ammonia in the influent stock solutions of 20 g L$^{-1}$ Mn(II), 2 g L$^{-1}$ NO$_2$-N and 20 g L$^{-1}$ NH$_4$-N were prepared in tanks 3, 4 and 5 (volume was all 50 L) by diluting MnSO$_4$·H$_2$O, NaNO$_2$ and NH$_4$Cl, respectively.

Filters 1 and 2 were packed with manganese sand at a height of 150 cm and a diameter of 0.8–1 mm. It should be noted that the biofilters were operating about 18 months before this experiment. Real groundwater, which was extracted from the wells with a depth of 40–50 m, in Harbin city, P.R. China, was used throughout this experiment. The compounds in the real groundwater are
shown in Table 1. The concentration of total iron, Mn(II) and ammonia in the raw groundwater was about 8–13, 1.1 and 1.2 mg L\(^{-1}\), respectively. The temperature was approximately 8°C. Downward gravity flow was adopted in the biofilters, and the amount of the flow was controlled at the entry point. Due to pore clogging from bacteria growth and iron and manganese precipitation on the support materials surfaces, regular backwashing was performed approximately every 2 days using high-water up flow velocities to wash out dead bacteria and maintain the activity of the system at a high level. The sampling time was 8:30 am every day, and the backwashing time was 10:00 am.

The redox reaction between nitrite and Mn(IV)

In order to verify whether the reaction between MnO\(_2\) and NO\(_2^-\) could occur under the conditions of this experiment, the \(\Delta G\) of the redox reaction between MnO\(_2\) and NO\(_2^-\) was calculated with a thermodynamic temperature of 298 and 281 K and an atmospheric pressure of 100 kPa.

The raw groundwater in tank 1 was pumped into the mixing chamber of filter 1, and then flowed into the filter; simultaneously DO in the raw groundwater in filter 1 was increased to about 3 mg L\(^{-1}\). The concentration of total iron, Mn(II) and ammonia in effluent of filter 1 was about 0.1, 1.5 and 0.7 mg L\(^{-1}\), respectively, and DO was about 0.2 mg L\(^{-1}\). The effluent water of filter 1 in tank 2 and the stock solutions of Mn(II) and nitrite in tanks 3 and 4, respectively, were pumped to the mixing chamber of filter 2 which was sealed, mixed and flowed into filter 2. The flow rates of the stock solutions of Mn(II) and nitrite were regulated to ensure that Mn(II) in the influent was approximately 2 mg L\(^{-1}\) and nitrite was approximately 0.05, 0.1, 0.2 and 0.5 mg L\(^{-1}\), respectively.
The influence of added and generated nitrite on Mn(II) removal in a biofilter

In order to investigate the influence of added nitrite on Mn(II) removal using a biofilter, the experiment was operated as in the previous section except that the DO in the influent of filter 2 was above 10 mg L\(^{-1}\). In order to investigate the influence of generated nitrite on Mn(II) removal using a biofilter, the concentration of ammonia in influent of filter 1 was increased from approximately 1.1 to 1.5 mg L\(^{-1}\) by adding the stock solution of ammonia in tank 5 (DO in the influent of filter 1 was approximately 11 mg L\(^{-1}\)).

The concentration profiles of ORP in simultaneous Mn(II) and ammonia removal

The aerated raw groundwater in tank 1 was pumped into filter 1, and the concentration of total iron, Mn(II) and ammonia in effluent water of filter 1 was lower than 0.1, 0.05 and 0.1 mg L\(^{-1}\), respectively. The effluent water of filter 1 in tank 2 was aerated and DO was increased to approximately 11 mg L\(^{-1}\), then the effluent water in tank 2 and the stock solutions of Mn(II) and ammonia in tanks 3 and 5, respectively, were pumped to filter 2. The concentration of Mn(II) and ammonia in the influent of filter 2 was approximately 2 and 1.5 mg L\(^{-1}\), respectively.

Kinetics of biological Mn(II) oxidation

The effluent water in tank 2 (as in the previous section) and the stock solution of Mn(II) in tank 3 were pumped into filter 2. In addition, the concentration of Mn(II) in the influent was approximately 4 mg L\(^{-1}\). The determination of the empty filter contacted time (EFCT) of the groundwater in the biofilter was based on the following equation:

\[
EFCT = \frac{\text{filter height (m)}}{\text{linear velocity (m h}^{-1})}
\]

Analysis methods

The pH, ORP and DO measurements were conducted using a pH meter (Ultra BASIC UB-10), an ORP meter (pH 315i-WTW) and a DO meter (Oxi 315i-WTW), respectively. The ammonia, total iron, Mn(II) and nitrite concentration measurements were according to the Standard Methods for the Examination of Water and Wastewater (Rittmann & Snoeyinck 1984).

RESULTS AND DISCUSSION

The redox reaction between nitrite and Mn(IV)

\[
\text{MnO}_2 + 2\text{NO}_2^- = \text{Mn}^{2+} + 2\text{NO}_3^-
\]

An equation of Gibbs free energy change:

\[
\Delta G = \Delta H - T\Delta S
\]

where \(\Delta G\) is Gibbs free energy change (kJ mol\(^{-1}\)), \(\Delta H\) is free enthalpy change (kJ mol\(^{-1}\)), \(T\) is thermodynamic temperature (K), and \(\Delta S\) is entropy change (kJ mol\(^{-1}\) K\(^{-1}\))

When the thermodynamic temperature was 298 (25 °C) and 281 K (8 °C), the \(\Delta G\) was 122.28 and 120.92 kJ mol\(^{-1}\) in 100 kPa, respectively, which suggested that MnO\(_2\) cannot react with NO\(_3\) under these conditions.

When nitrite (approximately 0.05, 0.1, 0.2 and 0.5 mg L\(^{-1}\)) and Mn(II) (approximately 2 mg L\(^{-1}\)) were added to filter 2 and DO in the influent was approximately 0.2 mg L\(^{-1}\), only a small part of nitrite was oxidized by DO in depths of 0–0.1 m of the filter, while Mn(II) was obviously decreased in depths of 0–0.2 m (Figure 2). The activity of manganese oxidizing bacteria (MnOB) was higher than nitrite oxidizing bacteria (NOB) in very low DO conditions. In depths of 0.2–1.5 m, DO was lower than 0.1 mg L\(^{-1}\), therefore nitrite and Mn(II) could not be oxidized by DO. Nitrite and Mn(II) were not varied in depths of 0.2–1.5 m, suggesting that nitrite did not react with manganese oxides in the biofilter in anaerobic conditions, which corresponds with the result of the \(\Delta G\).

The influence of added and generated nitrite on Mn(II) removal in a biofilter

When the concentration of nitrite and Mn(II) in the influent of filter 2 was approximately 0.05 and 2 mg L\(^{-1}\), respectively, nitrite and Mn(II) were completely oxidized in depths of 0–0.1 m of the filter after 1 d (Figure 3). The filter was...
used to remove Mn(II) and ammonia before this experiment, and an abundance of MnOB and NOB existed in the filter, which quickly oxidized Mn(II) and ammonia. Then the concentration of nitrite was increased to about 0.1, 0.2 and 0.5 mg L\(^{-1}\), respectively, and nitrite and Mn(II) were also completely removed in depths of 0–0.1 m after 1 d.

Before nitrite was added to filter 2, Mn(II) was completely removed in depths of 0–0.1 m of the filter. When nitrite was added to filter 2 with a concentration of approximately 0.05 mg L\(^{-1}\) (Figure 4(a)), Mn(II) was increased to 0.23 mg L\(^{-1}\) after 1 h in depths of 0.1 m (Figure 4(b)), and then decreased to lower than 0.05 mg L\(^{-1}\) after 2 h. When the concentration of added nitrite was increased to approximately 0.5 mg L\(^{-1}\) (Figure 4(c)), Mn(II) was increased to 0.18 mg L\(^{-1}\) after 1 h in depths of 0.1 m (Figure 4(d)), and then decreased to lower than 0.05 mg L\(^{-1}\) after 2 h. When nitrite was added to filter 2, biological Mn(II) removal was affected slightly, this is attributed to the presence of NOB, which quickly oxidized the added nitrite, and the MnOB adapted to the nitrite presented conditions.

When ammonia in the influent of filter 1 was increased from approximately 1.1 to 1.5 mg L\(^{-1}\), nitrite accumulated in the filter. Nitrite rapidly increased in depths of 0–0.4 m of the filter, and increased in depths of 0.4–0.8 m, then decreased to approximately 0.1 mg L\(^{-1}\) in depths of 1.5 m after 1 d (Figure 5(d)). The concentration of nitrite in the effluent was decreased to 0.045 and 0.02 mg L\(^{-1}\) after 2 and 3 d, respectively. Ammonia in depths of 0–0.8 m was obviously increased after 1 d (Figure 5(b)) and then quickly decreased. Mn(II) in depths of 0.8 m was 0.046, 0.159, 0.091 and 0.046 mg L\(^{-1}\) after 0, 1, 2 and 3 d, respectively (Figure 5(a)), while total iron was almost unchanged along the filter depth (Figure 5(c)). When ammonia in the influent was suddenly increased and nitrite was accumulated, biological Mn(II) removal was obviously affected, while Fe(II) removal was almost not affected. The reasons are as follows: Fe(II) was chemically and biologically removed in depths of 0–0.2 m where the nitrite was relatively low; while most of the Mn(II) was removed in depths of 0.2–0.8 m where the nitrite was high.

When nitrite was generated in the filter, Mn(II) removal was affected in 3 days; however, when nitrite was added to the filter, even the concentration of nitrite was much higher, Mn(II) removal was affected in only 2 h. The reason was because the added nitrite was quickly

![Figure 2](https://iwaponline.com/jwrd/article-pdf/7/3/264/376045/jwrd0070264.pdf)  
**Figure 2** Nitrite (a) and Mn(II) (b) concentration profiles along depth of filter 2 for nitrite feed concentration of approximately 0.05, 0.1, 0.2 and 0.5 mg L\(^{-1}\), respectively, and Mn(II) feed concentration of approximately 2 mg L\(^{-1}\) in anaerobic conditions.

![Figure 3](https://iwaponline.com/jwrd/article-pdf/7/3/264/376045/jwrd0070264.pdf)  
**Figure 3** Mn(II) (a) and nitrite (b) concentration profiles along depth of filter 2 for Mn(II) feed concentration of approximately 2 mg L\(^{-1}\), and nitrite feed concentration of approximately 0.05, 0.1, 0.2 and 0.5 mg L\(^{-1}\), respectively, in aerobic conditions.
oxidized to nitrate by NOB, but the generated nitrite needed much longer to be completely oxidized. So in order to shorten the start-up period of biofilter for Mn(II) and ammonia removal, the suitable inoculated bacteria were the biomass obtained from biological Mn(II) and ammonia removal filter, because the presence of NOB

Figure 4 | The variation of nitrite concentration profiles along depth of filter 2 for nitrite feed concentrations of approximately 0.05 (a) and 0.5 mg L\(^{-1}\) (c), respectively, in 1, 2 and 3 h after nitrite is added to the filter, respectively, and Mn(II) concentration profiles in 0, 1, 2 and 3 h (b) nitrite was approximately 0.05 mg L\(^{-1}\) and (d) nitrite was approximately 0.5 mg L\(^{-1}\), respectively.

Figure 5 | The variation of Mn(II) (a), ammonia (b) and total iron (c) concentration profiles along depths of filter 1 in 0, 1, 2 and 3 d after ammonia increased from approximately 1.1 to 1.5 mg L\(^{-1}\), respectively, and nitrite (d) concentration profiles in 1, 2 and 3 d, respectively.
could quickly oxidize nitrite to nitrate, and MnOB adapted to the nitrite presented conditions.

The concentration profiles of ORP in simultaneous Mn(II) and ammonia removal

When the concentration of Mn(II) and ammonia in the influent was approximately 2 and 1.5 mg L\(^{-1}\), respectively, ORP was 16 mV in influent, quickly increased to 75 mV in depths of 0.2 m of the filter, increased to 94 mV in depths of 0.4 m, and slowly increased to 122 mV in the effluent; Mn(II) and ammonia were decreased to 0.069 and 0.086 mg L\(^{-1}\) in depths of 0.1 m, respectively. Tekerlekopoulou & Vayenas (2008) investigated the ORP profiles along the depth of the biofilters for Fe(II), Mn(II) and ammonia removal, and found that ORP increased along the filter depth from 150 to 600 mV, depending on the feeding concentrations. In their investigation, ORP was much higher than in filter 2, because DO in their filter was 7–8 mg L\(^{-1}\); however, DO in the effluent of filter 2 was lower than 1 mg L\(^{-1}\).

Kinetics of biological manganese oxidation

The removal kinetics of contaminants during water treatment is considered an important issue, because it can provide information about the required time that the specific contaminant needs to be removed efficiently, which is necessary in sizing treatment units (Katsoyiannis & Zouboulis 2004). The concentration of Mn(II) in groundwater in China was normally lower than 3.5 mg L\(^{-1}\). In this experiment, Mn(II) in influent was 4.17 mg L\(^{-1}\) and decreased to 0.069 and 0.000 mg L\(^{-1}\) in depths of 0.4 and 0.5 m of the filter (Figure 6(a)), respectively. From the obtained results, the kinetics of Mn(II) oxidation could be calculated by assuming that all the soluble Mn(II) was oxidized, and then removed by filtration. By keeping DO constant and at a constant pH value, the Mn(II) depletion rate would be first order, i.e.

\[
\frac{d[Mn(II)]}{dt} = K[Mn(II)]
\]

A plot of ln\([Mn(II)]_t/[Mn(II)]_0\) versus time (EFCT) would be linear if the kinetics of Mn(II) oxidation were indeed first order, and the slope of such a plot would be \(-k\)-value (Figure 6(b)). The results indicated that the ln\([Mn(II)]_t/[Mn(II)]_0\) versus time (EFCT) was linear. The value of \(k\) was 0.687 min\(^{-1}\) and the half-life time for the depletion of Mn(II) was 1.010 min in the pilot-scale biofilter. The experiment was carried out at the actual pH value of the groundwater, i.e. 7.0.

CONCLUSIONS

\(\Delta G\) of the redox reaction between MnO\(_2\) and NO\(_2^-\) in 298 (25 C) and 281 K (8 C) was calculated and the results suggested that MnO\(_2\) cannot react with NO\(_2^-\). In the biofilter, nitrite could not react with manganese oxides in anaerobic conditions. Biological Mn(II) removal was affected by nitrite, and the longer the nitrite was present in the biofilter, the longer the Mn(II) removal was affected. In the start-up period, the presence of nitrite in the biofilter was the main reason for the start-up period of biofilters for Mn(II) and
ammonia removal being much longer than biofilters for Mn(II) removal. When Mn(II) and ammonia in influent were 2 and 1.5 mg L\(^{-1}\), respectively, ORP increased along the filter depth from 16 to 122 mV. Biological Mn(II) removal followed the first-order reaction, the \(k\)-value was 0.687 min\(^{-1}\) and the half-life time for the depletion of Mn(II) was 1.010 min.

ACKNOWLEDGEMENTS

This work was kindly supported by the Scientific Research Foundation of CUIT (KYTZ201511) and the Program of Education Department of Sichuan Province (16ZB0221).

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First received 12 December 2015; accepted in revised form 8 April 2016. Available online 15 May 2016