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Manganese release linked to carbonate dissolution during the start-up phase of a subsurface iron removal well in Khabarovsk, Russia

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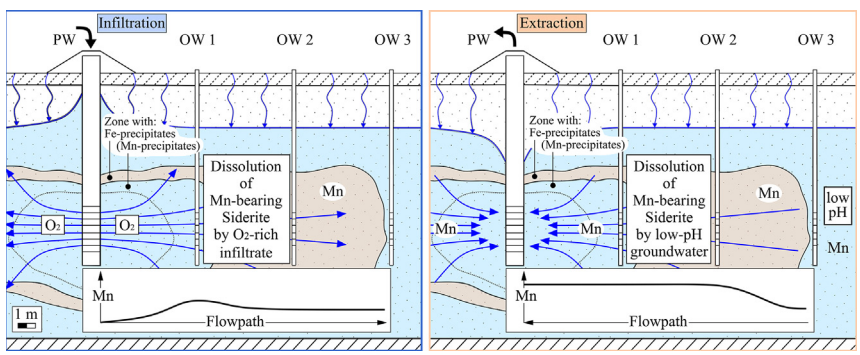
HIGHLIGHTS

- An unexpected Mn release was observed during the start-up phase of a SIR well.
- Water quality data of the first 194 infiltration & extraction phases were evaluated.
- Column-experiments & closed-bottle batch tests with aquifer sediment were conducted.
- Aquifer materials' grain size and groundwater pH primary controlled the Mn release.
- Dissolution of Mn-bearing carbonate minerals was identified as primary Mn source.

GRAPHICAL ABSTRACT



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ABSTRACT

Subsurface iron removal (SIR) is a proven pre-treatment technology for removing dissolved iron and manganese from groundwater. The start-up phase of a SIR well and the proper development of the reaction zone around the well are crucial for its subsequent performance. This study evaluates the start-up phase of a SIR production well in Khabarovsk, Russia during the first 194 SIR cycles. A strong release of manganese was observed, which led to concentrations twice as high as the background value of the pristine groundwater. Regular monitoring of the production well and its three adjacent observation wells showed that iron removal began immediately after start-up and that the reaction zone was completely formed within 25 SIR cycles. Closed-bottle batch tests and a flow-through leaching test revealed that the grain size of the aquifer material and groundwater pH are the primary controls on manganese release. During infiltration phases the dissolution of manganese-bearing carbonate minerals was caused by direct oxidation by O_2, whereas the low groundwater pH of 6 seemed to be responsible for the manganese release during extraction.

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1. Introduction

Subsurface iron removal (SIR) is a widely used and proven pre-treatment technology for removing dissolved iron and manganese from groundwater (Hallberg and Martinell, 1976; Rott et al., 2002). The principle is that aerated water is injected into an anoxic aquifer to form a so-called oxidation or reaction zone. Within this zone, dissolved and adsorbed iron, Fe(II), and manganese, Mn(II), are oxidized to Fe(III) and Mn(IV) and deposited as metal(hydr)oxides. After a short storage period of a few hours or days, the water can be recovered. Due to the large exchange capacity of the previously

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formed metal(hydr)oxide layers, dissolved Fe or Mn are going to adsorb at the metal(hydr)oxide surfaces and the water can be extracted without containing dissolved iron and manganese (Rott et al., 2002; van Halem et al., 2012).

One major advantage of the SIR process is the avoidance of any waste products above the surface. Contrary to conventional aboveground water treatment plants, where Fe and Mn are removed by e.g. aeration, flocculation and filtration, the oxidation products of iron and manganese remain in the aquifer. Hence, no cost-intensive disposal or treatment of waste products is required and SIR is often considered to have lower investment and operating costs (Rott et al., 2002). In addition, due to the aging-related conversion of the hydrous hydroxide precipitates to less voluminous oxides and oxide hydrates and comparably small amount of precipitates, clogging of aquifer pores will not lower the SIR efficiency in the long term (Mettler et al., 2001; Rott et al., 2002). Nonetheless, to ensure a satisfying treatment effectiveness of this technique, the initial start-up phase and the formation and position of the reaction zone are of major importance. Several studies showed that iron removal started within a few infiltration phases, whereas several months or years passed until manganese removal was completed (e.g. Meyerhoff, 1996; Rott and Friedle, 2000b; Roessner et al., 2017).

Problems that interrupt the SIR process were observed when the reaction zone was insufficiently developed and/or the extracted volume was increased too early, such that the existing treatment capacity was exceeded. The consequence was a breakthrough of Mn that surpassed the background concentration of the groundwater (Meyerhoff, 1996; Roessner et al., 2017). Furthermore, the periodic precipitation and dissolution of (Mn-)bearing carbonate minerals was found to impact the manganese concentration during SIR (Mettler et al., 2001). Antoniou et al. (2012, 2014) observed that the infiltration of oxygen-rich water caused the release of Mn from (Mn-)bearing carbonates by direct oxidation from O_2 and indirectly by pyrite oxidation, which forces buffering reactions of the (Mn-bearing) carbonate minerals. Also, since Fe^{2+} can chemically reduce existing Mn(hydr)oxides, incomplete iron removal can cause the release of Mn and lead to a delayed start of Mn removal (Meyerhoff, 1996).

In the city of Khabarovsk, SIR is used as pre-treatment at a recently built waterworks. After Fe removal and partial Mn removal were successfully initiated at >25 wells, an unexpected release of Mn was observed during the infiltration and (especially) the extraction phases at several production wells (PW). This study evaluates the initial start-up of a SIR production well during the first 194 SIR cycles, which took 650 days altogether. At this particular well a strong release of Mn was observed, resulting in Mn concentrations twice as high as the background concentration in the pristine groundwater. Based on a detailed evaluation of data from the regular monitoring of the PW and three adjacent observation wells (OW), the spatial and temporal formation of the reaction zone is described and the primary Mn source is identified. The resulting characterization is reinforced by results of additional column experiments, closed-bottle batch tests and a flow-through leaching test. Finally, results of a sequential extraction of aquifer sediments and from regular monitoring were used to estimate the expected future time span for Mn release.

2. Materials & methods

2.1. Field site Khabarovsk

The city of Khabarovsk is located in the Far East of Russia within the Central Amur River Basin at the confluence of the Ussuri and Amur Rivers (Fig. 1). Groundwater from the 400 km^2 large Tungusska reservoir is used for drinking water production. The base of the Central Amur River Basin begins at 120 m depth and consists of silt layers, silicate rock, sand, argillite and aleurolite (Neumann, 2007). A 50 to 70 m thick layer of low diagenized, carbonaceous rocks overlies the base. The upper part is formed by Pliocene-Quaternary unconsolidated rocks and is 50 to 60 m thick. Within the unconsolidated rocks are water-bearing layers of medium to coarse sand. These have a thickness of 40 to 50 m and are covered by a 5 to 7 m thick clay layer. Solid material analysis identified siderite ($FeCO_3$) as dominant carbonate mineral. This is probably "contaminated" with Mn and Ca and has the approximate composition $(Ca_{0.15}Mn_{0.08}Fe_{0.77})CO_3$ (Bilek, 2016). Rhodochrosite ($MnCO_3$) was not found as pure mineral. In addition, pyrite (FeS_2) was not found in noticeable amounts within the aquifer (Neumann, 2007). The pristine groundwater contains 26 ± 3 mg/l Fe, 1 to 3 mg/l Mn and has a low pH of 5.9 (Table 1). It also has a low to medium high buffer capacity with 150 to 165 mg/l bicarbonate and a high CO_2 concentration (155 to 215 mg/l).

To control elevated Fe concentrations in the pristine groundwater, SIR (Subterra technique) is applied as a pre-treatment step (Kulakov et al., 2011; Kulakov, 2013; Herlitzius et al., 2012; Herlitzius, 2015; Braun et al., 2016). All production wells (PW) alternately serve as infiltration and extraction wells. The efficiency coefficient assesses the effectiveness of the process and reflects the ratio of extracted water volume (V_{extr}) to (re-)infiltrated water volume (V_{inf}). Depending on the aquifer and raw water conditions the efficiency coefficient can reach values between 2 and 12 (Rott et al., 2002). This study focuses on one particular PW, where a strong release of Mn was observed already at an efficiency coefficient of 3. The PW was equipped with three observation wells (OW), which were located 6 m (OW 1), 12 m (OW 2) and 18 m (OW 3) apart from the well. The PW had a casing diameter of 0.35 m, a 0.1-m-thick filter gravel pack and a filter screen height of 5 m. The infiltration time was 48 h at an infiltration rate of 80 m^3/h (Fig. 2). Pretreated groundwater (aerated, chlorinated) from previous extraction phases served as infiltrate. To increase the treatment capacity, the infiltrate was enriched by technical oxygen up to a dissolved oxygen concentration of ≈25 mg/l. Extraction time was initially 48 h and increased after up to 72 h at a rate of 160 m^3/h. After 165 completed SIR cycles (450 days, observation period 1) with continuous operation the PW was shut down due to operational issues (Fig. 2). Following a 100 days lasting rest period the PW was restarted and further monitored for another 27 SIR cycles (observation period 2). Additional column experiments were conducted during observation period 2.

2.2. Regular monitoring

During infiltration phases, relevant water samples for this study were taken after total infiltrated volumes of 200, 2000 and 3840 m^3. Sampling during extraction phases was conducted after total extracted volumes of initially 100, 1000 and 4000 m^3 (48 h extraction) as well as after 8840 and 11,520 m^3 at 72 h extraction. Temperature (T), dissolved oxygen (O_2), pH and electrical conductivity (EC) were determined using a WTW Multi 3430 and appropriate electrodes (WTW, Weilheim, Germany) in a flow-through cell during water sampling.

2.3. Calculation of saturation indices and alkalinity

Because the infiltrate at the PW has roughly pH 8, precipitation of Mn as its divalent carbonate mineral rhodochrosite ($MnCO_3$) could occur (Mettler et al., 2001). To check saturation of Mn^{2+} with respect to $MnCO_3$ (Eq. (1), Table 2), saturation indices (SI) for rhodochrosite were determined with PHREEQC (Parkhurst and Appelo, 2013). Because solid material analysis identified $FeCO_3$ as dominant carbonate mineral, SI were determined for siderite, too (Eq. (2.1), Table 2). Since the equilibrium constant of the mineral actually present in Khabarovsk is unknown, Eq. (2.2) was not used for saturation calculations. SI were calculated for all observation points after infiltrated volumes of 200 m^3 (start) and 3840 m^3 (end) as well as after extracted volumes

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