



Synergistic effects of sulfate reducing bacteria and zero valent iron on zinc removal and stability in aquifer sediment



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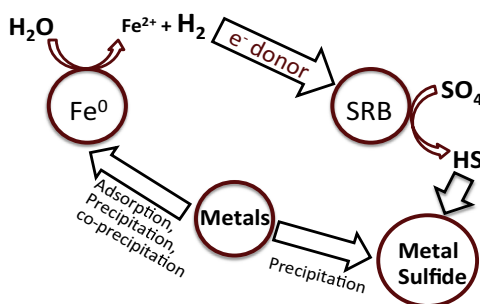
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HIGHLIGHTS

- Zn was efficiently removed in biotic and abiotic conditions.
- Synergistically using Fe⁰ and SRB for metal removal is effective approach in forming more stable precipitates.
- Change in ORP is more likely to cause leaching of Zn than change in pH in subsurface environments.

GRAPHICAL ABSTRACT



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ABSTRACT

We conducted a series of flow-through column experiments using aquifer sediment treated with zero valent iron (Fe⁰) with or without active microbial sulfate reduction. The aim of this study was to investigate the stability of Zn removed by Fe⁰ and impact of sulfate reducing bacteria (SRB). The leaching of Zn was assessed by flushing the aquifer in vertical glass columns with groundwater that was pre-treated with mixture of gases to simulate natural changes (pH and ORP) in subsurface environment (N₂ + CO₂ for pH and N₂ + O₂ for ORP) along with a control (N₂ flushed). Zinc removed synergistically by Fe⁰ and SRBs was found to be more stable and did not leach from the aquifer sediment with pH or redox changes. We also observed that the Zn leaching in the aquifer treated only with Fe⁰ was more influenced by change in redox than by pH changes. These results were also confirmed by sequential metal extractions and scanning electron microscopy observations in the aquifer sediments. Mineral phases were predicted using geochemical modeling tool CHESS confirming that the metal removed in Fe⁰ + SRB conditions formed more stable precipitates and did not show any signs of leaching back to groundwater upon change in pH and redox conditions.

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1. Introduction

Groundwater resources are vital for meeting current and future global water demands, but are threatened by extensive contamination originating from geogenic and anthropogenic sources. Due to

potential influences on the human food chain, presence of heavy metals in groundwater is a global concern. Heavy metal contamination in groundwater is caused mostly by leaching of metals by infiltrating rainwater from contaminated soil to the groundwater, or vertical displacement and eventual leaching through soil profiles from non-ferrous industrial plumes. Metals initially migrate from the unsaturated zone into the saturated zone, sorbing on to the aquifer material until all the available binding sites are saturated. Once the sorption capacity of soil is exhausted, remaining

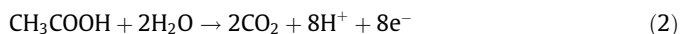
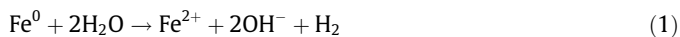
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metal concentrations can be transported through the groundwater [4].

In recent years, extensive research has been carried out to design efficient and cost effective techniques for treating groundwater contaminated with heavy metals e.g. chemical precipitation, electrochemical separation, ion exchange etc [29]. Most of these approaches are based on pump-and-treat (above-ground) methods. These are relatively easy to implement but consume high amounts of energy for pumping the water, which increases the treatment cost and produce considerable amount of waste and water that further needs to be discharged [30]. These constraints make pump-and-treat method highly impractical for large volumes of groundwater. These problems encouraged researchers to further explore possibilities for *in situ* treatment of groundwater e.g. *in situ* chemical precipitation, *in situ* bio-precipitation (ISBP), natural attenuation, permeable reactive barriers (PRBs), and reactive material injections to form reactive zones.

Zero valent iron (Fe^0) has recently attracted attention as an efficient reactive material used in subsurface applications to treat chlorinated compounds as well as others contaminants including heavy metals [16,23,31,12,15]. Fe^0 is a strong reducing agent, which while in contact with water, oxidizes and produces OH^- ions that further elevates the pH of the system and also generates water born H_2 that can play an important role in biogeochemical interactions:



It is generally believed that metal removal by Fe^0 from groundwater is primarily due to surface adsorption and reductive mineral precipitation and co-precipitation [27,5,35]. Evolution of microbial communities in these reducing environments has often been reported [9,14]. Sulfate reducing bacteria (SRB) in particular are already well known for heavy metal precipitation as relatively stable metal sulfides (reaction 2–4) [3]. But SRBs in natural attenuation could take very long time in overall site remediation and success is often limited due to lack of suitable environmental conditions and/or availability of electron donor [15]. However, these limitations can be overcome by using an integrated Fe^0 + SRB treatment approach for subsurface applications. Fe^0 oxidation creates strongly reducing and oxygen depleted environment required by SRBs for optimum growth and also SRBs can utilize the H_2 produced by iron oxidation as an efficient electron donor [11,36,15]. H_2 consumption by SRBs can help in avoiding the fouling and over pressure within the PRBs, which further adds to the longevity of the treatment process [9,34]. Integration of SRB- Fe^0 systems can be synergistically beneficial for heavy metal removal as Fe^0 can remove metals efficiently, and eventually SRBs can form relatively stable metal sulfides. There have already been few reported studies discussing the importance of microbial communities in Fe^0 based treatment systems [25,15]. Although Fe^0 is highly efficient in metal removal, the stability of these removed metals in subsurface environment is still poorly understood. Sudden changes in environmental conditions i.e. pH or oxidation–reduction potential (ORP) could lead to undesired changes in performance of the treatment system over time and even to release of already immobilized metals back to the groundwater.

In the present study, we investigated the effectiveness of Fe^0 + SRB integrated groundwater treatment systems in terms of stability of the removed Zn. The primary aims of this study were (1) to

evaluate the impact of SRBs on the stability of Fe^0 -immobilized metals in subsurface systems and (2) to understand the impact of changing environmental conditions (pH, ORP) on the immobilized Zn. Two parameters, pH and ORP were considered important in these systems, as they can fluctuate due to human activities or seasonal changes and [19,18]. A low pH aquifer sediment and groundwater from a Zn contaminated site in Belgium was used in this study. Earlier in a batch performed on this aquifer sediment, it was shown that (1) Zn removal only occurred in the presence of Fe^0 acting as pH and redox mediator, and (2) SRBs were only activated after addition of glycerol together with Fe^0 [15]. Subsamples of the aquifer from this batch test were further subjected to (1) sequential extraction to identify different metal binding phases in sediment, (2) Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) for solid phase characterization, and (3) Geochemical calculations performed using Chemical Equilibrium of Species and Surfaces (CHESS) to predict the formed phases during the batch incubation. Finally, (4) a series of vertical flow-through columns were setup in parallel to evaluate the stability of the formed Zn precipitates under different pH and ORP conditions.

2. Materials and methods

2.1. Batch test

Contaminated aquifer material and groundwater from a low pH, heavy metal contaminated site in Belgium were incubated with Fe^0 in batch microcosms under anaerobic conditions. SRBs were stimulated in selected microcosms by providing glycerol as an extra C-source. This batch experimental setup has been previously explained in detail elsewhere [15]. Briefly, microcosms were designed using 200 mL of groundwater mixed with 40 g of sediment in glass bottles (250 mL) under nitrogen atmosphere. Two different size of Fe^0 were used i.e. granular zero valent iron (gFe^0 , Gotthard Maier, Germany) and micro zero valent iron (mFe^0 , Högenäs, Sweden) with an average particle size of 0.25–2 mm and 20–40 μm and available surface area of ≈ 0.8 and 4 m^2/g , respectively. Five different experimental conditions were setup i.e. (i) incubation with gFe^0 (ii) incubation with gFe^0 + glycerol (iii) incubation with mFe^0 , (iv) incubation with mFe^0 + glycerol and (v) a control (aquifer material without Fe^0 and glycerol). After 120 days of incubation, the aquifer material was retrieved from batch microcosms and was further subjected to sequential metal extraction, SEM–EDX analysis and column leaching tests. These results are discussed in detail in this study.

2.2. Geochemical and microscopic analysis

2.2.1. Sequential extraction

Aquifer material retrieved from batch microcosms was subjected to sequential metal extraction using a procedure modified from Tessier et al. [24]. For this study, only three fractions were extracted i.e. Carbonate fraction, Fe–Mn oxide fraction (reducible) and organic fraction using 1 M CH_3COONa ; 0.04 M $\text{NH}_2\text{OH.HCl}$ in 25% acetic acid; 3.2 M $\text{CH}_3\text{COONH}_4$, respectively. Further details on this analysis methods are described elsewhere [22].

2.2.2. Microscopic analysis

Aliquots of aquifer material were dried under nitrogen environment and examined under scanning electron microscope (SEM) combined with energy dispersive X-ray (EDX) imaging, using a JEOL-JSM 6340-F scanning electron microscope equipped with a X-FLASH silicon drift detector. Microscope was operated at a 20 kV electron accelerating potential and a beam current of

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