



Quantification of changes in zero valent iron morphology using X-ray computed tomography

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Abstract

Morphological changes within the porous architecture of laboratory scale zero valent iron (ZVI) permeable reactive barriers (PRBs), after exposure to different groundwater conditions, have been quantified experimentally for different ZVI/sand ratios (10%, 50% and 100%, W/W) with the aim of inferring porosity changes in field barriers. Column studies were conducted to simulate interaction with different water chemistries, a synthetic groundwater, acidic drainage and deionised (DI) water as control. Morphological changes, in terms of pore size and distribution, were measured using X-ray computed tomography (CT). CT image analysis revealed significant morphological changes in columns treated with different water chemistries. For example, 100% ZVI (W/W) columns had a higher frequency of small pores (0.6 mm) was observed in ZVI grains reacted with typical groundwater, resulting in a porosity of 27%, compared to 32% when exposed to DI water. In comparison, ZVI grains treated with the acidic drainage had higher porosity (44%) and larger average pore size (2.8 mm). 10% ZVI PRB barrier material had the highest mean porosity (56%) after exposure to any water chemistry whilst 100% ZVI (W/W) columns always had the lowest (34%) with the 50% ZVI (W/W) in between (40%). These results agree with previously published PRB field data and simultaneously conducted geochemical monitoring and mass balance calculation, indicating that both the geochemical and hydraulic environment of the PRB play an important role in determining barrier lifespan. This study suggests that X-ray CT image analysis is a powerful tool for studying the detailed inter pores between ZVI grains within PRBs.

Key words: porosity change; X-ray computer tomography; mass balance; permeable reactive barrier; zero valent iron; acidic drainage; typical groundwater; pore-clogging

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Introduction

Zero valent iron (ZVI) is a popular material for use in Permeable Reactive Barriers (PRBs) (US EPA, 2002). It has been widely exploited to remove chlorinated solvents, metals and radionuclides from contaminated groundwater through mechanisms such as, reductive precipitation, sorption, ion exchange and biodegradation (Bartzas et al., 2006; Jin suk et al., 2009). To date no failures based on barrier porosity have been reported at commercial ZVI PRB sites, however the literature suggests that many barriers decline in performance over time (e.g. Bartzas and Komnitsas, 2010). Pore clogging of a ZVI barrier by mineral precipitates (Li and Benson, 2010), bio-fouling (Lampron et al., 2001), barrier material deposition (Lee et al., 2009) and gas production (Kamolpornwijit and Liang, 2006) can all, alone or in combination, reduce the

available porosity, increase preferential flow, reduce residence time and reduce treatment success quantification of morphological changes in ZVI grains over time and under different physical and chemical conditions is therefore key to understanding and predicting long-term performance of a ZVI PRB.

A number of studies have previously attempted to detect and quantify pore clogging process in PRBs. Wilkin et al. (2003) estimated pore volume loss at the Denver Federal Centre (DFC, Lakewood, Colorado, USA) via monitoring the geochemical changes of groundwater and characterising the barrier mate Kamolpornwijit X-ray diffraction (XRD) and SEM-XPS (X-ray photoelectron microscopy). Kamolpornwijit et al. (2003) conducted a column study to quantify mineral formation using both pore water mass balance and Thermogravimetric Analysis (TGA). Other methods such as the geochemical model code PHREEQC (Bartzas et al., 2006; Bartzas and Komnitsas, 2010; Sass et al., 2002), the groundwater flow modelling MODFLOW

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(Mayer et al., 2002; Li et al., 2006), the reactive transport model RT3D (Clement, 1997) and tracer tests (Wilkin and Puls, 2003; Lai et al., 2004; Parbs et al., 2004) have also been successfully employed to evaluate porosity change at PRB sites. These approaches however require a full understanding of chemical reactions between groundwater and barrier material. Wantanaphong et al. (2006) used Scanning Electron Microscopy (SEM) to directly quantify pore clogging process by imaging individual grains of reactive media before and after reaction with contaminated groundwater. However, the method is limited to the identification of the associated pore characteristics on the surface of grains rather than these between grains. X-ray computed tomography (CT) is a non-destructive technology that enables *in-situ* direct measurement of pore characteristics within porous structures, i.e. between grains. It has been successfully employed to investigate bulk density, moisture content, porosity of soils and sediments and the mechanical properties of rocks in 2D and 3D (e.g. Dulu, 1999). For example, it can adequately characterise the major components in soils, such as water, pore space, organic material, and soil minerals (Mooney, 2002; Mooney and Morris, 2008; Atkinson et al., 2007). To date no previous studies have used X-ray CT to identify changes in the porous architecture of ZVI grains, possibly due to the beam hardening artefacts associated with scanning dense materials. The high-density of ZVI can reduce X-ray penetration which impacts on image quality (Walter et al., 2004). Some of these issues can be reduced by the use of filters (e.g. Al, Ca) in front of the X-ray source to correct for beam hardening.

In this study, we used X-ray CT to visually examine the pore characteristics between ZVI grains and to quantify porosity in a PRB. Column studies were conducted with different water chemistries to understand how groundwater chemistry might affect PRB longevity. The possible reasons for porosity differences were validated by data from mass balance calculation and Scanning Electron Microscopy (SEM).

1 Materials and methods

1.1 Zero valent iron characterization

Milled ZVI grains (Peerless Metal Powders & Abrasive

Co., USA) with density 7.2 g/cm³, surface area 1.8 m²/g and grain size between mesh 8 and 50, i.e. ≥ 90% of the ZVI grains size were between 2.38 and 0.30 mm, was used in this study. In addition to Fe, the grains had 2.6%–3.0% C, 2% Si, minor calcite (CaCO₃), dolomite and Mn-sulphide inclusions and trace amounts of Ti and Nb according to the manufacturer.

1.2 Synthetic water preparation

Two water chemistries were selected in addition to DI water (**Table 1**); a typical groundwater (pH 8.32) representing an unpolluted freshwaters with all ions in the middle of the normal concentration range and an acidic drainage water (pH 3.35) (Appelo and Postma, 2005).

1.3 Column experiments

Column experiments were used to simulate reactions between ZVI grains and water at the entrance zone of a PRB barrier under medium flow conditions (2 mL/min). This is 56 times greater than the average flow rate at a PRB installation (0.04 mL/min, 0.16 m/day) (ITRC, 2005). At this flow rate, 16 days represents an equivalent reaction period of 2.2 years under typical field conditions. Polypropylene columns of 7 cm in length and 4.3 cm in internal diameter were first packed with 1 cm of acid washed sand (2–4 mm grain size), followed by 5 cm of the ZVI/sand mixture and a final 1 cm layer of sand. The ZVI was wet sieved before packing to remove fine grains that may migrate through the column and block the influent. Three ZVI mixing ratios of the barrier material (100%, 50% and 10%, W/W) were tested for each water type in triplicate. When the water flow was terminated, columns were allowed to drain for two days before oven drying at 50°C for 10 days. All columns were sealed in plastic bags prior to X-ray CT scanning. After scanning, samples from columns exposed to typical groundwater and control (DI water) were taken from Philips XL30 Scanning Electron Microscope – Dispersive X-ray Spectroscopy (SEM-EDX) analysis following the procedure demonstrated in Wantanaphong et al. (2006).

1.4 X-ray computed tomography image acquisition and analysis

Columns were scanned using an X-Tek (now Nikon) Venlo heavy duty scanner fitted with a 300-mm linear array

Table 1 Geochemistry of column influent and effluent collected after reaction with different water chemistries (unit: mg/L)

Groundwater type		pH	Ca	Fe	K	Na	Mg	HCO ₃ ⁻	Cl ⁻	SO ₄ ²⁻
Control (DI)	Influent	5.91	0.08	0.02	0.00	0.50	0.00	0.00	0.47	0.50
	Effluent	5.77	0.20	0.80	0.00	0.00	0.00	0.00	0.67	0.58
Typical groundwater	Influent	8.32	20.6	0.02	5.39	8.90	18.3	230	55.8	9.80
	Effluent	8.03	12.2	0.05	5.56	9.11	19.9	195	56.2	8.12
Acidic drainage	Influent	3.35	54.0	0.02	3.84	2.18	23.3	0.00	2.30	9.51
	Effluent	5.60	55.6	4.77	3.92	2.09	22.9	0.00	2.03	7.40

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