



Distribution of As trapping along a ZVI/sand bed reactor

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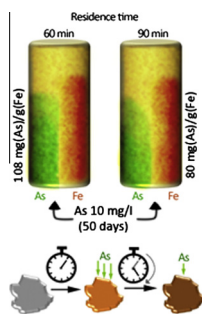
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HIGHLIGHTS

- Arsenic sorption has been studied in a ZVI/sand column for two residence times.
- A heterogeneous arsenic distribution was observed along the ZVI/sand column.
- A very high trapping capacity, $170 \text{ mg(As)} \text{ g}^{-1}(\text{Fe})$, was found at the reactor inlet.
- The reactivity of ZVI byproducts towards arsenic decreased over time.

GRAPHICAL ABSTRACT



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ABSTRACT

Zero-valent iron (ZVI) based reactors are widely used to remove arsenic from water and have been extensively studied, though typically by considering just the inlet and outlet arsenic concentrations. This paper presents the arsenic distribution inside an aerated bed reactor filled with a ZVI/sand support. The removal performance was evaluated through both the classical monitoring of arsenic concentrations and an analysis of the support upon completion of the experiment. Both types of analyses were performed not only at the column inlet and outlet but also at various intermediate sampling points in order to highlight the different behavior encountered along the column. The Thomas model was applied to simulate the breakthrough curves and determine sorption constants. These distinct approaches yielded similar results: while the iron distribution remained homogeneous from one approach to the next, the arsenic removal was five to six times higher at the inlet to the bed reactor than in the final ZVI/sand layer, reaching $170 \text{ mg(As)} \text{ g}^{-1}(\text{Fe})$. The influence of residence time was also studied, revealing that a higher arsenic load in the column led to greater retention within the first column layers.

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1. Introduction

Many technological solutions are developed to obtain As free drinking water [1]. Water treatment processes using iron products (zero-valent iron (ZVI), iron oxides or iron-coated media) have gained popularity over the past few decades, especially for arsenic removal through adsorption and co-precipitation mechanisms [2]. ZVI support is in fact easy to handle, inexpensive and efficient.

Several studies have focused on evaluating the behavior and efficiency of As treatment by means of column filtration using ZVI or iron oxides either on their own [3–5], mixed with sand [4,6–8] or else coated on various supports [9–11]. In these studies, the authors generally determined the maximum sorption capacity, expressed as $\text{mg(As)} \text{ trapped per g(support media)}$ or per g(Fe) . The values reported in the literature can vary over a wide range, mainly due to variations in experimental protocols: As concentration, nature of column filling (ZVI, iron oxides, addition of sand or other supporting media, etc.), column size, and effluent composition. For example, Lien and Wilkin [4] reported an arsenic removal

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capacity of $4.7 \text{ mg(As)} \text{ g}^{-1}(\text{Fe})$ for a 50/50 (v/v) ZVI/sand filling, whereas results obtained by Nguyen et al. [11] led to an 11 mg g^{-1} iron oxide-coated sponge (IOCSp) that contained 12% iron oxide, thus above $90 \text{ mg(As)} \text{ g}^{-1}(\text{Fe})$.

In most cases, such an evaluation is based on column inlet and outlet arsenic concentrations, with no attention being paid to the arsenic distribution inside the column. Only a few studies have reported that arsenic distribution may be very heterogeneous. Results obtained by Abedin et al. [6] showed that in a column filled with ZVI and sand, arsenic retention mainly occurred in the first part of the column. A similar observation was made by Nikolaidis et al. [8] during a long-term field experiment. Melitas et al. [12] indicated that arsenate was more quickly removed close to the column inlet, which could be explained by faster iron corrosion in this area. On the other hand, Lien and Wilkin [4] did not observe any significant variations in reactivity along a column filled exclusively with ZVI. Biterna et al. [7] demonstrated the poor efficiency of arsenite removal by ZVI in column experiments under anoxic conditions. They underscored the advantages of working under oxidative conditions in order to improve iron corrosion and, consequently, arsenic removal.

In the present article, we will investigate arsenic removal along a ZVI/sand-filled column under aerobic conditions so as to compare reactivity of the various column layers. For this purpose, As concentrations will be monitored at different sampling points until reaching saturation. The maximum arsenic trapping capacity will then be determined by estimating the quantity of arsenic retained at each layer, as well as by simulating breakthrough curves using the Thomas model [13]. These layer retention capacity evaluations will be confirmed through analyzing total As and Fe in the ZVI/sand support collected at the end of the experiment.

2. Materials and methods

2.1. Pilot unit

The experimental upflow reactors all have an 8-cm internal diameter and are 40 cm high. Four sampling valves were installed along the reactor height to allow for water collection. These valves were located at 10 (P_1), 18 (P_2), 27 (P_3) and 35 (P_4) cm from the reactor inlet. A diagram of these experimental reactors can be found in the [supplementary data section \(SD1\)](#).

The reactors were filled first with a 5-cm high layer containing sieved sand ($d = 3 \pm 1 \text{ mm}$); the second 20-cm high layer was filled with a homogenous ZVI/sand mixture ($1\% w_{\text{Fe}}/w_{\text{sand}}$, ZVI powder: purchased from Jeulin, purity >99.999%, diameter = $76 \mu\text{m}$, density = 7.86 g cm^{-3} , surface area = $0.01029 \text{ m}^2 \text{ g}^{-1}$), while a third layer (15 cm high) contained just sieved sand. The sand layers were introduced to limit iron losses from the column. 18 g of ZVI were introduced in each pilot unit. The empty bed volume equaled $500 \pm 30 \text{ mL}$. To prevent a decrease in ZVI within the column during filling operations, the sand was slightly wetted.

Throughout the experiment, the reactors were continuously fed with synthetic groundwater from a 400 L tank. Flows were calibrated to control residence times using peristaltic pumps. 8.4 and 5.3 mL min^{-1} flows were applied to generate residence times of 60 and 90 min, respectively.

The synthetic water composition (given in [supplementary data section SD2](#)) corresponded to calcareous water [14] spiked with 10 mg L^{-1} of arsenic (NaAsO_2 , Aldrich, purity >99%). This As concentration may be representative of industrial or mining water or of a highly contaminated groundwater. It was also useful to consider such a concentration in order to determine the maximum As sorption capacity and reach support saturation within a reasonable amount of time.

The reactors and intermediate feeding tanks were maintained at $12 \text{ }^\circ\text{C}$ thanks to their double thermostatic jacket and a cryothermostat (Polystat cc3, Model UC015-3, Huber Company). To avoid any evolution of its composition (especially arsenic speciation), the synthetic water was analyzed daily and renewed every three days.

A constant air flow (60 L h^{-1}) was supplied by air diffusers placed at the bottom of each reactor. The physicochemical parameters (pH, dissolved oxygen, temperature) were monitored both at the reactor outflow and in the feeding tank.

2.2. Total As and Fe

Total As was determined using a Varian SpectrAA 880Z Atomic Absorption Spectrometer equipped with a GTA 100Z Graphite Furnace, with Zeeman background correction (GF-AAS). A 193.7-nm wavelength and a 0.5-nm slit width were selected. Pyrolytic graphite-coated tubes with forked pyrolytic platforms were also used. The total Fe concentration was determined using a Varian SpectrAA 220 Atomic Absorption Spectrometer equipped with a Flame atomizer and a D_2 lamp background correction (F-AAS). The selected wavelength and slit width were 248.3 nm and 0.2 nm, respectively. The limits of quantification were $5 \mu\text{g L}^{-1}$ and $100 \mu\text{g L}^{-1}$ for total As and Fe, respectively.

As speciation was monitored with High-Performance Liquid Chromatography (HPLC), coupled to Atomic Fluorescence Spectrometry (AFS) along with Hydride Generation (HG). The chromatographic conditions consisted of a Hamilton PRP-X100 column with a phosphate buffer solution as the mobile phase ($30 \text{ mmol L}^{-1} \text{ NH}_4\text{H}_2\text{PO}_4$, buffered to pH 6.9 by adding $\text{NH}_3 \cdot \text{H}_2\text{O}$) at a rate of 1 mL min^{-1} . HG was conducted using solutions of NaBH_4 (14 g L^{-1}) and HCl (3 mol L^{-1}), which were injected at a flow rate of 0.8 mL min^{-1} . As was detected by means of Atomic Fluorescence Spectrometry (PSA Analytical EXCALIBUR®). The limits of quantification were set at 0.1 and $0.3 \mu\text{g L}^{-1}$ for As^{III} and As^{V} , respectively.

Prior to determining its As and Fe contents, the solid support collected at the end of the experiment was subjected to microwave-assisted acid digestion. 5 mL of 37% HCl and 5 mL of 65% HNO_3 were added to 1 g of solid sample. The mixture was then exposed to 1400 W for 30 min in a closed PTFE vessel using an Anton Paar Multiwave 3000. After cooling, the liquid phase was filtered ($0.2 \mu\text{m}$) and transferred into a 50-mL volumetric flask completed with ultrapure water. Clean sand samples were also used as analytical blanks.

All analyses were performed in triplicate.

2.3. Solid analysis

The solid was dried at $40 \text{ }^\circ\text{C}$ for 24 h. The product obtained was then crushed in a porcelain mortar and sieved to yield particles of a size smaller than $200 \mu\text{m}$. X-ray Diffraction (XRD) analysis was performed on a Siemens D5000 diffractometer using filtered $\text{Cu K}\alpha_1, 2$ radiation.

2.4. Thomas model

The Thomas model was used to simulate breakthrough curves and determine maximum As sorption capacity [13]. It has already been effectively introduced in the literature for determining the maximum solid-phase concentration of arsenic in column systems filled with iron-coated sand [15] or iron-coated sponges [11].

The Thomas equation corresponds to:

$$\frac{C_{\text{out}}}{C_{\text{in}}} = \frac{1}{1 + e^{(K_{\text{th}}(NX - C_{\text{in}}V_{\text{eff}})/Q)}$$

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