



## Mobility of arsenic, cadmium and zinc in a multi-element contaminated soil profile assessed by *in-situ* soil pore water sampling, column leaching and sequential extraction

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Mobility of arsenic, cadmium and zinc in a polluted soil can be realistically interpreted by *in-situ* soil pore water sampling.

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### ABSTRACT

Three methods for predicting element mobility in soils have been applied to an iron-rich soil, contaminated with arsenic, cadmium and zinc. Soils were collected from 0 to 30 cm, 30 to 70 cm and 70 to 100 cm depths in the field and soil pore water was collected at different depths from an adjacent 100 cm deep trench. Sequential extraction and a column leaching test in the laboratory were compared to element concentrations in pore water sampled directly from the field. Arsenic showed low extractability, low leachability and occurred at low concentrations in pore water samples. Cadmium and zinc were more labile and present in higher concentrations in pore water, increasing with soil depth. Pore water sampling gave the best indication of short term element mobility when field conditions were taken into account, but further extraction and leaching procedures produced a fuller picture of element dynamics, revealing highly labile Cd deep in the soil profile.

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### 1. Introduction

The characteristics of soils in urban, contaminated and brown-field areas are poorly defined, especially in relation to the stability and mobility of inorganic pollutants. Some urban soils, in areas of recent or historical perturbation, are difficult to classify as soils in the traditional sense because they are heavily disturbed and have a variable content of imported materials, such as construction residues or wastes (Lehmann and Stahr, 2007). In such soils, many of which are heavily contaminated with a variety of pollutants, we have limited knowledge of the characteristics that regulate both the residence time and potential movement of metals and metalloids. Field based techniques to measure the mobility of trace elements such as the sampling of interstitial pore water, could have significant potential for ecotoxicology testing (Tiensing et al., 2001) at such sites, although there are only a few reports of the use of this method in field experiments (Farley and Fitter, 1999; Geibe et al., 2006; Buckingham et al., 2008; Clemente et al., 2008). In the case of

soils that are derived from sediments or have a substantial component that originates from waste products, such as in former industrial or urban areas, the examination of element behaviour and movement in soils should be as representative as possible of processes naturally occurring *in-situ*, which could be unique to individual substrates or soils. In these cases, measurements taken in the field could provide valuable supplementary information when used in conjunction with laboratory based methods to examine the behaviour of trace elements.

The aim of the present study was to assess the labile fractions of cadmium, arsenic and zinc in a multi-element contaminated soil profile, in relation to the short and longer term behaviour of these elements, by directly comparing two commonly used laboratory methods with measurements taken on the same soil under field conditions. After a sequential extraction procedure to identify the geochemical associations of the elements, a laboratory leaching test was carried out to investigate longer term mobility. Soil pore water taken *in-situ* from the soil profile was analysed to examine element mobility and to provide a linkage between the results of the laboratory tests and the results of the data obtained under real world conditions in the field.

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## 2. Methods

### 2.1. Study site

The study site was located on an embankment that separates two canals, constructed in the first decades of the 19th century in Kidsgrove, Staffordshire, UK (Lat/Long: 53°05'23.0"N, 02°15'05.7"W). The site is composed of dredged sediment, originating from the iron-rich waters of the lower of the two canals that intersect at this location. The history of the pollution in the soil is probably associated with an adjacent manufacturer, whose effluent containing Cd and Zn may have been discharged or spilled into the lower of the two canals. The origin of the As is not known, although one possible source is the former Kidsgrove gasworks that was located near to the study site at the southern end of the embankment on the outskirts of Kidsgrove. Canal sediment was dredged and dumped onto the embankment without further treatment at an unknown period within the last century and has since become spontaneously vegetated. Today the area supports a variety of shrubs (mainly *Salix caprea* and *Salix viminalis*), and perennial herbs (Lepp and Madejón, 2007). In a previous study by Hartley et al. (2004) to examine immobilization of arsenic by soil amendments, cadmium leaching was found to increase when this soil was treated with iron based additives. However, *S. caprea* growing here has been shown to selectively accumulate Cd in stems and leaves without apparent phytotoxic effects (Lepp and Madejón, 2007) despite high soil Fe content (Hartley et al., 2004). Further to this, Hartley et al. (2008) found no obvious lack of key functional invertebrate groups in this soil, leading to the conclusion that the elevated concentrations of metals have not adversely affected soil biodiversity. In the case of arsenic very limited transfer from soil to plant has been observed (Madejón and Lepp, 2007).

### 2.2. Soil sampling and analysis

Two adjacent trenches were excavated to a depth of 100 cm in an undisturbed area of the site that previous studies had identified as contaminated with As and Cd. One trench was used for bulk soil removal (for subsequent column leaching and laboratory extraction procedures) and the other for sampling of soil pore water *in-situ*. Bulk soil samples were taken from three faces of the soil trenches providing triplicate samples of approx. 500 g each at depths of 0–30 cm, 30–70 cm and 70–100 cm.

In the laboratory, larger debris was removed and the remaining soil was crushed and sieved to <2 mm prior to the following analysis. Particle size distribution was determined using a Beckman Coulter LS 13 laser diffraction particle size analyser. Samples were carefully pre-treated with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub> 30%) to remove organic matter prior to analysis. Soil pH was determined using a Jenway 3051 pH meter and SIS electrode in 1:10 suspension of soil in de-ionised water, shaken for 3 h and allowed to settle for 45 min at room temperature (22 °C). Total organic carbon (TOC) in solid samples was determined using an SSM-5000A/TOC-VE analyser (Shimadzu, Tokyo). Water extracts were prepared using 1:10 suspensions, shaken for 3 h, centrifuged for 10 min at 3000 rpm and screened using a fine nylon mesh to remove any suspended particulate material which could interfere with analysis. These extracts were analysed for total water-soluble carbon (WSC) using a TOC-VE water analyser (Shimadzu, Tokyo). Aliquots of soil (0.2 g) were microwave digested in concentrated 14 M, GPR grade HNO<sub>3</sub> and analysed for pseudo-total As, Cd, Zn, Fe and Al using ICP-MS (XSERIES 2 ICP-MS; Thermo Scientific, MA, USA). Certified reference material (CMI 7004) was used to verify accuracy, with 78%, 83% and 94% recoveries of Zn, As and Cd respectively.

The sequential extraction procedure used here followed that of Shiowatana et al. (2001) with minor modifications. This procedure was developed for arsenic fractionation but was also used in the present study for Cd and Zn, primarily because a standardized water-soluble extraction was required for all elements. Triplicate soil samples from each depth were bulked together and mixed thoroughly before the following analysis:

For step 1 (Water-Soluble; pH 6), 1 g soil plus 30 ml of deionized water was shaken for 16 h. The soil suspension was centrifuged at 3000 rpm for 15 min and the supernatant filtered through Whatman No. 42 filter paper. For step 2 (Sodium Bicarbonate-Extractable/surface adsorbed; pH 9) the soil residue from Step 1 was re-suspended in 30 ml of NaHCO<sub>3</sub> (0.5 M) and shaken for 16 h. The soil was then centrifuged and filtered as in step 1. For step 3 (Sodium Hydroxide-Extractable/Fe and Al associated; pH 13) the soil residue from Step 2 was re-suspended in 30 ml of NaOH (0.1 M) and shaken for 16 h, then centrifuged and filtered as in step 1. For step 4 (Hydrochloric Acid-Extractable) the soil residues from Step 3 was re-suspended in 30 ml of HCl (1 M) and shaken for 16 h, then centrifuged and filtered as in step 1. In the final step, step 5 (Residual), the remaining soil residue from step 4 was oven-dried at 60 °C for 48 h and finely ground in an agate mortar. The HNO<sub>3</sub> digestion procedure described previously for pseudo-total soil concentrations was used to determine element concentration for this step. This differed from Shiowatana et al. (2001) who used hydrofluoric acid digestion for the final step.

### 2.3. Leaching column test and pore water sampling

#### 2.3.1. Leaching column test

Duplicate glass columns (XK50, Pharmacia Biotech), 20 cm long with a 5 cm internal diameter were packed with 400 g of air dried soils from each depth (Fig. 1A)

from one of the soil trenches. Columns were leached upwards from their base with de-ionised water (pH 5.5) at a 0.1 ml min<sup>-1</sup> continuous flow maintained by a peristaltic pump. The standard method (Dutch Environment Agency; NEN 7343: Determination of leaching of inorganic components from granular materials) was modified so that a 9 fraction series over 63 days (see Table 1) was employed rather than the standard 7 fractions. Additionally, leaching water of pH 5.5 was used instead of acidified water (pH 4) to represent as realistically as possible natural precipitation. The overall aim of this test is to simulate leaching over a long term period (3–5 years). Cellulose nitrate membrane filters (1.2 µm) were placed at the base and top of each column to remove colloidal material from leachate. Before leachate collection commenced, soils were fully saturated.

#### 2.3.2. In-situ pore water sampling

In the second trench compact rhizon pore water samplers (Eijkelkamp Agri-search Equipment, The Netherlands) were inserted at 4 depths within the soil profile (10, 25, 50 and 75 cm; Fig. 1B). Samplers were placed in triplicate at each depth and left for 1 month to equilibrate, after which pore water was collected over a period of 2 weeks using removable needles and vacuum tubes. This 2 week period was required to ensure that sufficient volume of pore water was collected for subsequent laboratory analysis. Dissolved organic carbon (DOC) in pore water was determined using a TOC-VE water analyser (Shimadzu, Tokyo) and both leaching column eluate and soil pore water were analysed for arsenic, cadmium and zinc using ICP-MS (as detailed above) with an internal rhodium standard to compensate for instrument response changes during analysis.

After analyses of all samples, the proportion of the pseudo-total As, Cd and Zn extracted by each method was calculated. In the column test, the weight of dry soil in each column (400 g) was used to calculate the total amount of element removed per kg of soil (as presented in Fig. 2). For pore water data a transformation using soil moisture measurements was calculated, following Clemente et al. (2008). In a modification to this method, triplicate soil moisture measurements were taken in



Fig. 1. Leaching column set up in the laboratory (A) and rhizon pore water samplers in the soil profile in the field (B).

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