



Nanoscale zero-valent iron-assisted soil washing for the removal of potentially toxic elements

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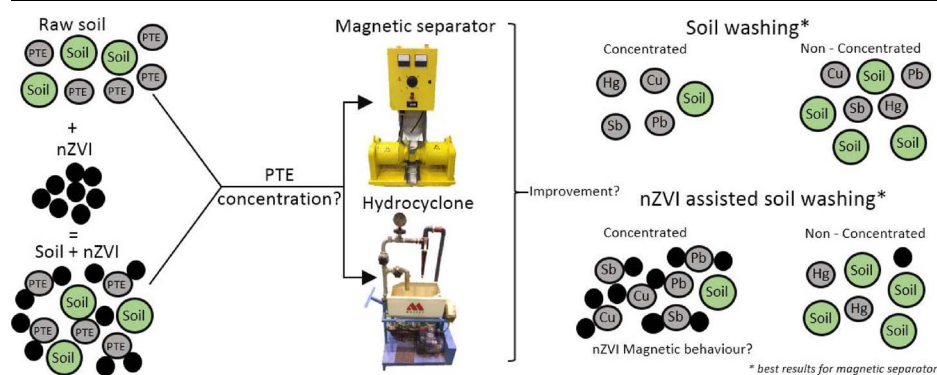
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GRAPHICAL ABSTRACT



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ABSTRACT

The present study focuses on soil washing enhancement via soil pretreatment with nanoscale zero-valent iron (nZVI) for the remediation of potentially toxic elements. To this end, soil polluted with As, Cu, Hg, Pb and Sb was partitioned into various grain sizes (500–2000, 125–500 and < 125 μm). The fractions were pretreated with nZVI and subsequently subjected, according to grain size, to Wet-High Intensity Magnetic Separation (WHIMS) or hydrocycloning. The results were compared with those obtained in the absence of nanoparticles.

An exhaustive characterization of the magnetic signal of the nanoparticles was done. This provided valuable information regarding potentially toxic elements (PTEs) fate, and allowed a metallurgical accounting correction considering the dilution effects caused by nanoparticle addition.

As a result, remarkable recovery yields were obtained for Cu, Pb and Sb, which concentrated with the nZVI in the magnetically separated fraction (WHIMS tests) and underflow (hydrocyclone tests). In contrast, Hg, concentrated in the non-magnetic fraction and overflow respectively, while the behavior of As was unaltered by the nZVI pretreatment. All things considered, the addition of nZVI enhanced the efficiency of soil washing, particularly for larger fractions (125–2000 μm). The proposed methodology lays the foundations for nanoparticle utilization in soil washing operations.

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1. Introduction

Potentially toxic elements (PTEs) are a major cause of contamination in soils of cities and rural areas. The concept of PTE encompasses a wide selection of elements (As, Cu, Hg, Pb, Zn, among others) that in high concentrations might cause severe damage to the environment and also to the human health [1,2]. Their persistence in the environment and the ease with which they bioaccumulate and biomagnify in living organisms make them pollutants of special concern [3]. PTEs may derive from natural sources or anthropogenic sources such as mining, industry or traffic [4,5]. In soils they usually appear linked to industrial and chemical waste, or even atmospheric deposition [6]. They enter tissues via ingestion, breathing and touching and cause severe diseases [7]. For all these reasons, their removal has been widely discussed in environmental research over recent decades [8–10].

Of all the remediation techniques available [11], soil washing is widely used [12,13]. It is based on concentrating the contaminants into a reduced volume fraction of the affected soil (or concentrated fraction), thereby leaving the matrix decontaminated (non-concentrated fraction) [14]. The method embraces two contaminant-removal technologies, namely, physical separation, which is based on mineral processing technologies, and chemical extraction, which is based on hydrometallurgy [15].

Here we focused on physical soil washing, that is to say, those procedures that do not alter the chemical properties of materials [12]. In these cases, separation is achieved by means of differences in the physical properties, namely particle size, density, magnetic susceptibility, or even physicochemical properties, as is the case of froth floatation, between the soil and the contaminant [16]. This technique has several advantages, including ease of deployment and versatility to be combined in sequence with other physical and chemical remediation methods [17]. In this context, some researchers have used remediation techniques that combine soil washing together with phytoremediation [18,19], stabilization [20], electrokinetics [21] or ultrasonics [22].

Moreover, the addition of certain compounds such as surfactants [23] and chelants to soil washing enhances PTE recovery [24]. In this respect, nanoscale zero-valent iron (nZVI) is the most commonly used nanomaterial for remediation purposes in Europe and the United States [25,26]. It is a non-toxic reactive metal (as a result of its large surface area, among other factors) that has found wide applications due to its abundance, low cost and ease of production [27]. This remediation material has been successfully applied for the removal of PTEs not only from soils [28–30] but from groundwater [31–34] and water runoff [35].

The applications of nZVI for PTE decontamination of soil are diverse. In this regard, this nanomaterial can be used to immobilize, sorb and capture these compounds [36]. Within this context, nanoparticles enhance soil washing by adsorption of the PTE-containing particles, thereby causing the formation of larger and heavier aggregates which are easier to separate [37]. Regarding magnetic separation, the high magnetic susceptibility of the newly formed aggregates allows the separation of otherwise non-magnetic particles [11,12].

This study aims to evaluate the effect of nZVI as a pre-treatment to a subsequent soil washing process. Thus, the specific objectives were as follows:

- To introduce a procedure that allows the measurement of the amount of natural Fe and nZVI present in each studied fraction.
- To develop a metallurgical accounting correction that circumvents the dilution effect that the addition of nZVI entails, thus facilitating the comparison of results between experiments with and without pretreatments.
- To ascertain the trace elements for which nZVI is selective, on the basis of their behavior in the separation equipment.

2. Materials and methods

2.1. Site description and soil sampling

Soil samples were collected from the old Hg mine of Olicio, in the surroundings of the Picos de Europa National Park (Asturias, Spain). The geology of the area is framed within the Cantabrian zone, specifically in the Ponga mantle [40]. The lithology comprises mainly paraconglomerates, white quartzites and siltstones from the Ordovician period [41]. The first evidence of cinnabar dates back to the late 19th century, but it was not until 1965 when underground mining began, persisting until the early 1970s, when the Hg crisis occurred. During these years, the extracted mineral was treated in a retort furnace, and ashes and tailings were mindlessly dumped in the confined valley of the Bregues stream [42].

These mining activities covered approximately 8000 m² of the valley with waste, thus enriching the surrounding soils in several PTEs, particularly Hg and As. Within this context, 25 bulk soil samples were collected at a depth of between 0 and 30 cm using a Dutch auger. These samples were then pooled into a single “macro sample” of about 50 kg, which was subsequently sieved through a 2-cm screen to remove rocks, gravel, and other large material.

2.2. Soil characterization and chemical analysis

This macro sample was divided obtaining representative subsamples of 500 g each, which were subjected to wet sieving in order to obtain particle-size fractions of < 125, 125–500 and 500–2000 µm. Thus, normalized sieves were placed in a column, and batches of 100 g of the material were placed in a sieve shaker for 5 min with a water flow of 0.3 l/min (ASTM D-422-63, Standard Test Method for Particle-Size Analysis of Soils). pH was measured with a glass electrode in a suspension of soil and deionized water (1:2.5).

Fractions were then laid out on glass trays, dried at 30 °C to prevent Hg volatilization, and finally weighed. Once all the material was meshed, each fraction was split into two equal and representative masses, which were used to perform the experiments with and without nZVI pretreatment.

To standardize the conditions used for chemical determinations, samples > 125 µm were ground in a RS100 Resch mill at 400 rpm for 40 s. Then, 1-g representative subsamples of the diverse origins (soils, grain-size fractions, etc.) were subjected to a 1:1:1 “Aqua regia” digestion. The total concentrations of Ag, Al, As, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, Hg, K, La, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Sc, Sr, Th, Ti, Tl, V, W and Zn in the digested material were determined by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) at the accredited (ISO 9002) Bureau Veritas Laboratories (Vancouver, Canada).

Powder X-Ray diffraction (PXRD) patterns measured on a PANalytical X’Pert Pro MPD diffractometer with Cu $k_{\alpha 1}$ radiation (1.540598 Å) were used to determine the mineralogical composition of the soil. After determining the position of Bragg peaks observed over the range of $2\theta = 5\text{--}90^\circ$, the minerals were identified using databases of the International Centre for Diffraction Data.

2.3. Nanoscale zero-valent iron pretreatment

A commercial air-stabilized aqueous solution of nZVI (NANOFER STAR-W), supplied by Nano Iron Rajhrad (Czech Republic), was used. This product comprises Fe (0): 14–18%, magnetite (Fe²⁺Fe₂³⁺O₄²⁻): 2–6%, carbon (C): 0–1% and about 80% of water. None of these components are classified as hazardous according to 67/548/EEC and Regulation (EC) N° 1278/2008 (CLP). As quoted by the manufacturer, this product is optimal for the preparation of slurries for in-situ remediation purposes [43].

The addition of nanoparticles followed the same procedure for each of the three grain-sizes. Thus, the nZVI drum provided by the

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