



Insights into mixed contaminants interactions and its implication for heavy metals and metalloids mobility, bioavailability and risk assessment



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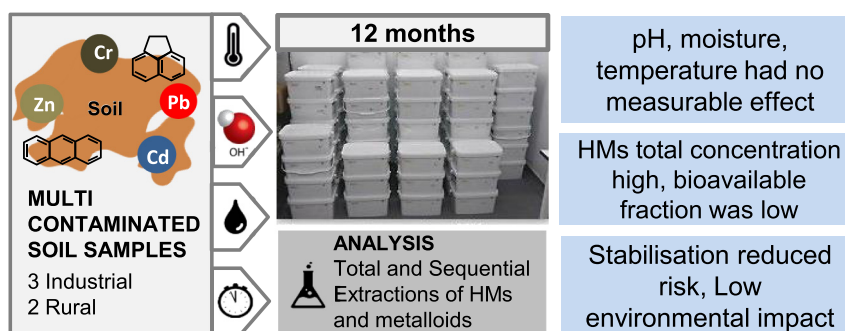
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HIGHLIGHTS

- Assessing HMs fractionation is key for understanding their environmental impact.
- HMs total concentration exceeded the SGVs but bioavailable fraction was low.
- pH, moisture, and temperature did not affect HMs bioavailable fraction over time.
- Cement stabiliser was able to reduce HMs solubility and bioavailability, reducing risk.

GRAPHICAL ABSTRACT



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ABSTRACT

Mobility of heavy metals at contaminated sites is mainly influenced by the soil physicochemical properties and environmental conditions, therefore assessing heavy metals (HMs) and metalloids fractionation can provide insights into their potential risk and the mechanisms that regulate bioavailability. A 12-months mesocosms experiment was setup to investigate the effect of physicochemical factors (pH, moisture, and temperature) and weathering (time) on HMs and metalloids fractionation in three different multi-contaminated soil matrices (low, medium, and high contamination) collected from a soil treatment facility located in the United Kingdom, and two rural contaminated soil samples. The study demonstrates that even though Pb and Zn were found associated with the exchangeable fraction in the soil with the highest contamination (total average Pb 3400 mg/kg, and total average Zn 2100 mg/kg in Soil C), neither the condition applied nor the weathering caused an increase in their mobility. Although it was expected that lower pH (4.5) would favour the dissociation of HMs and metalloids, no significant differences were observed, potentially due to the initial alkaline pH of the genuine-contaminated soil samples. The results show that even though total concentration of Pb, Cu, and Zn exceed the soil standards and guideline values, HMs were predominantly associated with the non-exchangeable fraction, while only 5% were dissolved in the pore water fraction (potentially bioavailable). In addition, the mobility and bioavailability of HMs remained constant over the 12 months monitoring, suggesting that these soils pose negligible risk to the environment.

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1. Introduction

Anthropogenic activities such as mining, waste disposal, combustion of leaded fuels, the use of fertilizers and pesticides, and petrochemical spills all contribute to the presence, accumulation, and persistence of heavy metals (HMs) in soil (Tóth et al., 2016; Suresh et al., 2012; Wuana and Okieimen, 2011). While organic contaminants might be degraded as they persist in the environment, inorganic contaminants, such as heavy metals and metalloids, are non-degradable and display long-term persistence in soils (Lu et al., 2017), which can potentially cause risk for plants, animals, and humans (Bolan et al., 2014).

European environmental regulatory frameworks, to manage HMs pollution, define risk based on the total extractable concentration of metals in soil. This approach does not consider how likely an HM is to be bioavailable, which can lead to an over/under estimation of risk (Cipullo et al., 2018). In relation to contaminated land risk assessment and remediation, bioavailability can be interpreted as the fraction of contaminant that is freely available in the environment (not sorbed or sequestered), and mobile (extractable by mild extraction), thus the most likely to lead to receptor exposure (Adedigba and Semple, 2015; Dean and Scott, 2004).

Sorption and desorption are the main processes controlling bioavailability of HMs (Caporale and Violante, 2016); in particular soil components responsible for the sorption includes; amorphous materials, silicates, clay minerals, carbonates, and organic matter (Leleyter et al., 2012). How a HM interacts with the different soil compartments will influence its bioavailability, and it is bioavailability that can inform the likelihood that a HM might leach into the broader environment (Ashraf et al., 2012). For example, HMs that are dissolved in pore water can be easily mobilised, and are considered readily available for uptake by plants (Chang et al., 2014) or available for interaction with biological systems (Hodson et al., 2011), while those dissolved in labile fractions are potentially bioavailable, if physico-chemical conditions were to change (e.g. pH decrease) (Di Bonito et al., 2018). Many physico-chemical factors such as soil pH, composition, organic carbon content, and redox potential, can impact partitioning between soil-solid phase and pore water, which will consequently have an impact on HMs bioavailability (Islam et al., 2015; Venegas et al., 2016). In contrast, HMs associated with non-exchangeable or non-mobile soil fractions are characterised by a stronger binding (weaker reversibility), therefore unlikely to leach into the surrounding environment.

Despite the recent shift toward a risk-based approaches for assessing contaminated sites, risk characterisation remains a conservative approach (Harmsen and Naidu, 2013; Naidu et al., 2015; Ortega-Calvo et al., 2015), because it relies on total contaminant concentration, rather than assessing the fraction of the total (bioavailable) that can potentially interact with biological and environmental targets. For bioavailability to be implemented and support regulatory decisions, the bioavailable estimation should rely on standardized methods, however, to date there exists no systematic method of assessment (Alvarez et al., 2011; R.Y. Kim et al., 2015). A number of techniques have been developed over the past two decades, and are still used to estimate HMs bioavailability in soil; including diffusive gradient in thin films (Agbenin and Welp, 2012; Menegário et al., 2017; Parker et al., 2016; Ren et al., 2015), ion exchange (Ge et al., 2005; Qian and Schoenau, 2002), single-step extractions (R.Y. Kim et al., 2015; Pinto et al., 2015; Sakan et al., 2016), and sequential extractions (Cox et al., 2013; Fernández-Ondóño et al., 2017; Palumbo-Roe et al., 2013; Reis et al., 2014; Sungur et al., 2014). Sequential extractions, in particular, are simple low cost methods, that can be applied to different soil types (Rosado et al., 2016), and can help understanding HMs and metalloids leachability, solubility, and mobility (Kaakinen et al., 2015), providing the most information about the fate, transport, and behaviour of HMs in soil. However, most studies focus on (1) assessing effects and toxicity of one contaminant in isolation (Cui et al., 2005), (2) using sequential chemical extraction on synthetic models or spiked samples (R.-Y. Kim et al., 2015; Ma et al., 2015) rather than genuine contaminated soil

samples (Ma et al., 2015). Limitations associated with these approaches include; metal transfer among phases (Bermond, 1992) when performing sequential extractions resulting in an overestimation of metals concentration and risk, and the inability of an artificial contamination to reproduce the actual geochemistry encountered in real site conditions (Ribeiro and Mexia, 1997). While it is challenging to establish a one-size fits all approach for assessing HMs behaviour in contaminated soil, the choice of procedure should be based on a more realistic prediction of elemental mobility and characterisation of their association with the soil matrix.

In this study a modified version of a non-specific sequential extraction method coupled to chemometric analysis known as the Chemometric Identification of Substrates and Element Distributions (CISED), was used and applied to five different genuine contaminated soils. Our objectives were (1) to apply a sequential extraction technique in genuine-contaminated soil samples and identify the common soil phases, (2) to evaluate the spatial distribution of HMs/metalloids and potential changes over time in order to ascertain the bioavailability of HMs/metalloids and potential risk, (3) to determine the influence of different physico-chemical factors on HMs/metalloids solid phase distribution and bioavailability, and (4) to evaluate the effect of co-occurrence of hydrocarbons on HMs/metalloids partitioning in soil samples. The novelty of this study lies in the fact that it highlights the importance of taking into account the effects of a range of environmental stressor conditions (pH, moisture, and temperature) and weathering (time), on HMs/metalloids potentially labile fractions; including both dissolved elements (pore water), and exchangeable fraction in genuine co-contaminated soil samples. A special emphasis on the effectiveness of this protocol with multi-contaminated samples of different nature (with and without stabilisation treatment), origins (industrial and rural), and with a wide range of HMs contents has also been verified. This information can be used as additional lines of evidence to support risk-based decisions about endpoint remediation and to evaluate potential reuse of remediated soil.

2. Materials and methods

2.1. Sample collection and preparation

Since soil contaminants are often present in the environment in a complex mixture, including both inorganic and organic compounds as by-products of industrial activities (Wawra et al., 2018), this study investigates five multi-contaminated soil samples. Three genuine contaminated soils, denoted as Soil A, Soil B (treated), and Soil C, were collected from a soil treatment facility located in the United Kingdom. Information regarding original location of the soil samples collected, and specific details regarding the treatment applied (soil B), were not disclosed to maintain anonymity and confidentiality. Two additional samples were collected from a rural site contaminated by HMs/metalloids and diesel range organic (DRO) compounds (nC_{10} – nC_{24}) (Soil D), and HMs/metalloids mineral oil range organic (MRO) compounds (nC_{22} – nC_{34}) (Soil E). The mutual presence of organic and inorganic contaminants in these soil samples could potentially enhance (or inhibit) HMs transport by

Table 1
Soils samples and soil characteristics used in the mesocosms experimental setup.

Soil	Treatment	Soil type	Contamination type	Soil matrix
Soil A	Pre-treatment ^a	Industrial	TPHs > 1000 mg/kg (high), HMs > 800 mg/kg	Sandy loam
Soil B	Post-treatment ^b			
Soil C	No treatment	Industrial	TPHs < 1000 mg/kg (medium), HMs > 6200 mg/kg	Sandy loam
Soil D	No treatment	Rural	TPHs < 500 mg/kg (low), HMs > 800 mg/kg	Clay loam
Soil E	No treatment			

TPHs: total petroleum hydrocarbons, HMs: heavy metals.

^a No stabiliser.

^b Application of cement stabiliser.

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