



Research article

Investigations of water-extractability of As in excavated urban soils using sequential leaching tests: Effect of testing parameters

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ABSTRACT

Excavated soils with low-level As contamination obtained from construction projects during city development have been of great concern in Japan. Water-extractable As represents the most easily mobilized and ecotoxicologically relevant fraction in the soil environment. In the present study, the water-extractability of As in excavated alkaline urban soils was assessed using sequential leaching tests (SLTs) with a focus on the effects of test parameters. In addition, the potentially water-leachable As over an extremely long period was assessed using the pollution potential leaching index (PPLI), from which one can estimate the number of extractions required to reduce the As in the cumulative leachates to below the Japanese environmental standard ($10 \mu\text{g L}^{-1}$). Total As concentrations varied from 6.75 to 79.4 mg kg^{-1} , and As was continuously detectable among replicate SLT experiments. The water-extractable As obtained in the first step of the SLT accounted for 0.41%–7.60% of total As (average: 2.36%), while the cumulative released As in the SLTs corresponded to 1.30%–21.6% of the total (average: 10.6%). The variability of the water-soluble fractions was sensitive to the test conditions. The shaking time at each SLT step had the largest effect on the As water-extractability; followed by sample storage, shaking speed and shaking interruption. A longer shaking time in the standard leaching test of excavated soils is suggested for regulatory purposes in Japan. The use of the PPLI concept for quick estimation of the potential As leachability from excavated soils was supported by the good reproducibility of PPLI results obtained from SLTs under different test parameters.

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1. Introduction

Soil excavation is a fundamental step of infrastructure development. Many infrastructure construction projects occur during city development. As a result, excavated surplus soils (or rocks) are generated in large quantities. For example, it is estimated that more than 140 million m^3 of excavated soils were generated in Japan in 2012 (MLIT, 2014). Reuse of excavated soils either on or off site has been promoted (Katsumi, 2015; Magnusson et al., 2015). However, the potential contamination by heavy metal(loid)s (HMs) must be

considered prior to reuse of such materials (Cui et al., 2017; Tabein et al., 2012, 2013). Although soil contamination is commonly assessed in terms of total element concentrations, the total amount of HMs in soils can be a poor indicator of their environmental impact (Davidson et al., 1998; Hartley et al., 2004). Consequently, various leaching tests have been suggested because they better reflect the potential risk of HMs release into the water phase and their subsequent migration and/or uptake by biota. This leads to unique trends in which, despite high levels of HMs in soils, the leachability or mobility is often low because of the relatively strong bonding (Li et al., 2014; Madrid et al., 2004), as well as to conditions in which substantial HMs leaching from soils occurs, irrespective of their extremely low total contents. Previous studies have reported HMs contamination, particularly arsenic (As), in excavated surplus

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soils or rocks, even though the total contents were at low levels (e.g., Li et al., 2016, 2017; Tabelin et al., 2013, 2014a,b, 2017).

Water-soluble As represents the most easily mobilized and ecotoxicologically relevant fractions of As in the soil environment. Water-extractable As has most often been used to estimate As water-solubility and is typically assessed using an accelerated leaching test when soils are too dry and no pore water can be collected (Meers et al., 2006). Several leaching tests using water as the only extracting liquid have been developed, including a batch test (e.g., American Society of Testing and Materials (ASTM) (Eisenberg et al., 1986), the United Kingdom leaching test (Hartley et al., 2004), the test described in Japanese Notification No. 46 (Environmental Agency of Japan, 1991)) and a percolation or column test (e.g., ISO/TS 21268-3, 2007; PrEN 14405, 2015). Column tests yield more representative information regarding actual leaching under field conditions (e.g., Jackson et al., 1984; Grathwohl and Susset, 2009) and therefore allow a detailed risk assessment of contaminated soils; however, they are costly and time-consuming. Conversely, batch tests can be used as compliance tests for routine testing because of their low costs, simple design and low duration. However, they do not reflect real world conditions because of friction between particles during mechanical agitation, centrifugation, and filtration of samples prior to analysis (Li et al., 2016; Naka et al., 2016). In addition, the sequential leaching test (SLT) (Grathwohl and Susset, 2009) has been developed for specific purposes. In the SLT, soils can be successively extracted by deionized water. The first step in water extraction can be used as a compliance test for environmental regulation. For instance, single-step water extraction (Japanese Notification No. 46) has been used for evaluation of As contamination in contaminated soils in Japan (Environmental Agency of Japan, 1991); however, its suitability for assessment of As contamination in excavated urban soils (or rocks) is still under discussion (Igarashi et al., 2008; Katsumi, 2015; Tabelin et al., 2014a). On the other hand, the total amount of As released in SLT reflects the As availability based on an extremely long-term perspective (Astrup et al., 2006).

To date, many studies have evaluated the water-extractability of As in excavated soils (or rocks) from construction projects using SLTs (e.g., Li et al., 2016, 2017; Tabelin et al., 2012, 2014b, 2017). However, no studies have systematically compared the effects of SLT test parameters on the water-extractability of As. Therefore, the present study was conducted to investigate the water-extractability of As in excavated urban soils using sequential leaching tests with different test parameters. The data under each test were compared with the prescribed regulatory limit of As in Japan and interpreted to comprehensively understand the long-term As leaching. This study will facilitate evaluation of the effectiveness of the currently used standardized leaching test for As contamination assessment and provide supporting information regarding the potential As leachability in excavated urban soils.

2. Materials and methods

2.1. Experimental samples

Excavated soil samples were obtained from construction projects in Tokyo, Japan. Four excavated subsurface soils (A, B, C and D) were randomly collected from four different construction projects and then transported to the laboratory. Upon arrival, part of each sample was air-dried immediately, while the rest was stored in airtight containers at room temperature for 18 months, then air-dried. For analysis, all samples were crushed, sieved through a 2 mm mesh, and then homogenized for the subsequent leaching tests. To investigate the effects of soil particle size on As leaching, each homogenized soil sample after immediate air-drying (<2 mm)

was further ground to pass through a 0.15 mm mesh. The physical and chemical characteristics of the four soils (<2 mm) have been reported in our previous studies (Li et al., 2016, 2017, Table S1 in the Supplementary Material (SM)). The pH of soils A, B, C and D was 8.5, 9.6, 10.2 and 10.0, respectively, suggesting the presence of basic salts. The total carbon contents of the four soils ranged from 9.8 to 15.1 g kg⁻¹. In addition, soils A, B, C and D were light clay, sandy clay loam, clay loam and loam and the total As contents were 9.22, 79.4, 8.81 and 6.75 mg kg⁻¹, respectively. These soils were not considered As-contaminated because the total contents were below the Japanese environmental quality standard (Li et al., 2017).

2.2. Sequential leaching tests

A series of SLTs were conducted to investigate the effects of test parameters (sample storage, L/S at each step, experimental device size, soil particle size, shaking method, speed, time and interruption between two steps, centrifugation speed and filter size) on As release from the studied excavated urban soils. As shown in Table 1, experiment T0 was the control test and the detailed test parameters of each step were slightly modified according to Japanese Notification No. 46. Briefly, 3 g of sample was mixed with 30 mL deionized water (Milli-Q[®] Gradient A10[®] water filtered with a Millipak[®] Express 20, 0.22 μm filter) in a 50 mL polypropylene centrifuge tube. The tubes were then shaken using a lateral-reciprocating shaker (Double Shaker NR-30, Taitec, Saitama, Japan) at 200 rpm for 6 h at room temperature, after which the pH values of the suspensions were measured immediately using a pH electrode (Navi F-52; Horiba, Kyoto, Japan). The suspensions were then centrifuged at about 6000 rpm (4700 g) for 15 min, which was followed by filtration through 0.45 μm membrane filters. The residual soils remaining in the tubes were then stored at 4 °C overnight, after which they were re-suspended in fresh deionized water for the ensuing leaching step. This procedure was repeated 10 times.

The effects of other parameters on As release were considered in experiments T1–T11 (Table 1). Test T1 investigated the effects of sample storage and drying on As leaching. The immediately air-dried samples described in Section 2.1 were used in T0, while the samples air-dried after 18-months of storage were used in T1. In T3, 250 mL polypropylene centrifuge tubes were used, in which 10 g aliquots of soil were mixed with 100 mL deionized water. Other experimental parameters in T1 and T3 were the same as those in T0. Similarly, all leaching procedures in T1–T11 were repeated 10 times, except for T2, in which the water extraction was repeated 20 times. All SLTs were carried out in triplicate. Arsenic, Fe and Al concentrations in the leachates were measured by inductively coupled plasma mass spectrometry (ICP-MS 7500c, Agilent Technologies, Tokyo, Japan) after appropriate dilution. Following the experiments, the residual soils in selected tests (T0, T4, T8 and T11) were air-dried and then digested for As measurement. The accountability of As (%) in SLTs, as determined by $100 \times [(\text{cumulative mass of released As} + \text{residual mass of As}) / \text{total mass of As}]$, was found to range from 91% to 105%.

2.3. Pollution potential leaching index (PPLI)

The pollution potential leaching index (PPLI) was applied to estimate the potentially water-leachable As and assess the contamination risk to the water environment (Li et al., 2018). If all leachates from SLT were mixed together, the average As in the cumulative leachate was calculated by the ratios of cumulative released As (mg kg⁻¹) to cumulative liquid to solid ratio (L/S) value (L kg⁻¹) (Fig. S1). Initially, the As in the first step exceeded the environmental standard of Japan (Soil Leachate Standard in Soil Contamination Countermeasures Law, 10 μg L⁻¹), but the As in the

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