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Research article

Leaching of arsenic, copper and chromium from thermally treated soil

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ABSTRACT

Thermal treatment, if properly performed, is an effective way of destroying organic compounds in contaminated soil, while impact on co-present inorganic contaminants varies depending on the element. Leaching of trace elements in thermally treated soil can be altered by co-combusting different types of materials. This study aimed at assessing changes in mobility of As, Cr and Cu in thermally treated soil as affected by addition of industrial by-products prior to soil combustion. Contaminated soil was mixed with either waste of gypsum boards, a steel processing residue (Fe₃O₄), fly ash from wood and coal combustion or a steel abrasive (96.5% Fe⁰). The mixes and unamended soil were thermally treated at 800 °C and divided into a fine fraction <0.125 mm and a coarse fraction >0.125 mm to simulate particle separation occurring in thermal treatment plants. The impact of the treatment on element behaviour was assessed by a batch leaching test, X-ray absorption spectroscopy and dispersive X-ray spectrometry. The results suggest that thermal treatment is highly unfavourable for As contaminated soils as it increased both the As leaching in the fine particle size fraction and the mass of the fines (up to 92%). Soil amendment with Fe-containing compounds prior to the thermal treatment reduced As leaching to the levels acceptable for hazardous waste landfills, but only in the coarse fraction, which does not justify the usefulness of such treatment. Among the amendments used, gypsum most effectively reduced leaching of Cr and Cu in thermally treated soil and could be recommended for soils that do not contain As. Fly ash was the least effective amendment as it increased leaching of both Cr and As in majority of samples.

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1. Introduction

Soils at industrial sites usually contain both organic and inorganic contaminants. Former wood impregnation sites is an example of this combination of contaminants, since the use of creosote and pentachlorophenol as an organic impregnation chemical was replaced with inorganic chemicals such as chromated copper arsenate (CCA). Such sites pose difficulties selecting effective in situ remediation options and soil is usually excavated for further treatment ex situ and/or landfilling. Highly contaminated soils not always comply with the regulations and cannot be accepted at landfills even for hazardous waste if the leaching of contaminants exceeds the leaching limit values. Moreover, according to the EU landfill directive (Council Directive 99/31/EC), waste must be treated by physical, thermal, chemical or biological means before

being landfilled. Combined techniques and stepwise remediation are often required to destroy, remove or immobilise contaminants in such soils, often followed by secondary waste treatment (e.g. process waters, concentrates, separated wood residues) (Coudert et al., 2014; Reynier et al., 2013).

Thermal treatment, if properly performed, is an effective way of destroying organic compounds (Marani et al., 2003; Desai et al., 2007), while impact on co-present inorganic contaminants varies depending on the element. Previous studies show that soil pH increases after thermal treatment and so does the mobility of oxyanions of arsenic (As) and chromium (Cr), while leaching of copper (Cu) decreases (Nordmark et al., 2011).

Leaching of trace elements can be altered by co-combusting different types of materials and modifying combustion conditions (Skoglund et al., 2016). It is therefore expected that increased amount of certain fuel constituents (e.g. Ca, Si, S, Fe) can improve the stability of trace elements in thermally treated soils and reduce contaminant leaching.

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The aim of this study was to assess changes in mobility of As, Cr and Cu in thermally treated soil as affected by addition of industrial by-products prior to soil combustion. Three of the selected amendments: waste of gypsum boards, steel processing residues and fly ash from wood and coal combustion are waste products containing components, such as sulphates, oxides and silicates, which can be beneficial for trace element stabilisation. In addition, restrictions or specific requirements for disposal are applied for some waste, e.g. gypsum boards due to generation of hydrogen sulfide (Montero et al., 2010). Therefore, using these waste materials for contaminated soil treatment is beneficial from resource management perspective. The fourth amendment, metallic iron, which is among the most efficient materials for As immobilisation (Kumpiene et al., 2008; Komárek et al., 2013), was selected as a reference amendment.

2. Materials and methods

2.1. Soil and amendments

Soil samples were collected at a former industrial wood impregnation site contaminated with creosote and chromated copper arsenate (CCA) chemicals. Samples were air dried homogenised and split into required portions first using coning and quartering and further using a riffler splitter.

Bulk soil was tested for total solids and loss on ignition (SS 028113, 1981), pH, electrical conductivity and redox potential in soil-water suspensions (1:2 v/v). Water holding capacity (WHC) was estimated from the mass of water remaining in drained soil after its saturation.

The industrial by-products used as soil amendments were: i) waste of gypsum boards (CaSO_4) containing 20.2% Ca, 15.5% S, 2 mg kg^{-1} Cu and the concentrations of As and Cr were found to be below the instrument detection limits; ii) a steel processing residue (Fe_3O_4) with 70% Fe_3O_4 , 28% Fe^0 , 1.2% Cr, 0.5% Mn; iii) fly ash from wood and coal combustion (FA) with 35.7% of CaO; other elements were (in mg kg^{-1}) Fe 6; Al 4; Cu 71; Cr 19; pH = 12.4 and iv) a commercially available product, steel abrasive with 96.5% of zero-valent iron (Fe^0). Each amendment was mixed with soil at 5 wt% and left for 3 weeks at 50% of WHC to condition the soil before thermal treatment.

The total element concentrations in soil prior and after thermal treatment were determined after microwave digestion in 5 ml HNO_3 + 0.5 ml H_2O_2 (Pavličková et al., 2003; Shumo et al., 2014) by inductively coupled plasma sector field mass spectrometry (ICP-SFMS) by an accredited laboratory ALS Scandinavia. A certified reference material soil CRM027-050 was used for quality control along with extraction and instrument blanks. In addition, quality control standard solutions were run periodically between the samples.

2.2. Thermal treatment

Soil mixtures and soil with no additives (control) were treated in 1 kg batches in silicon carbide crucibles at 800 °C in a pre-heated carbolite oven CSF 1200 in stagnant air for 20 min. Samples were cooled at room temperature and divided into a fine fraction <0.125 mm and a coarse fraction >0.125 mm to simulate particle separation occurring in thermal treatment plants.

2.3. Leaching test

Batch leaching test at a liquid to solid (L/S) ratio 10 (EN 12457/2) was performed in triplicates with all the soil samples, i.e. control, amended soil without thermal treatment and thermally treated soil

with and without amendments to evaluate the changes in element mobility. Briefly, samples were leached using double-distilled water for 24 h using an end-over-end shaker, filtered through 0.45 μm nitrocellulose membrane filter and immediately analysed for pH, EC and redox potential. Dissolved element concentrations were measured with ICP-OES. Limits of detection were 3 $\mu\text{g l}^{-1}$ for As, 0.5 $\mu\text{g l}^{-1}$ for Cr and 0.4 $\mu\text{g l}^{-1}$ for Cu, and limits of quantification were 0.02 mg l^{-1} for As, 1 $\mu\text{g l}^{-1}$ for Cr, 2 $\mu\text{g l}^{-1}$ for Cu. The leached concentrations (in mg kg^{-1} dry weight) were compared to the leaching limit values for waste acceptance at EU landfills (EU Council, 2003/33/EC Annex II).

2.4. X-ray absorption spectroscopy

Both particle size fractions of control soil and soil amended with Fe^0 prior and after thermal treatment were analysed using Extended X-ray absorption fine structure spectroscopy (EXAFS) at the wiggler beamline I811 at MAX-lab, Lund University, Sweden. The beamline was equipped with a Si[111] double crystal monochromator, operated at 1.5 GeV and a maximum current of 250 mA. Iron K-edge EXAFS spectra were collected in fluorescence mode using a Lytle detector. Iron metal foil was used as an energy calibration reference and was run simultaneously with the samples. Higher-order harmonics were reduced by detuning the maximum intensity of the second monochromator crystal by 50%. A Mn filter was used to reduce Fe fluorescence and scattering contributions. Spectra for standard Fe oxides were collected separately at the same beamline. The air-dried samples were mounted on aluminium frame holders using Kapton tape and fixed at 48° angle to the incident beam to avoid XRD peaks in the scans. EXAFS data were collected in the X-ray energy range between 6960.75 and 7910.75 eV. In total 548 points per scan with an exposure time of 0.5 s per point were collected in the entire X-ray energy interval. Five scans were collected per sample. All measurements were carried out at ambient-room temperature.

The collected XANES data were processed using Athena software (Ravel, 2008) including data normalization based on the Cromer-Lieberman calculations, background removal using the AUTOBK algorithm, alignment and merge of scans. Linear combination fitting was performed for $\chi(k)$ spectra with five standard compounds: zerovalent iron, ferrihydrite, goethite, lepidocrocite and hematite. The variable weights were constrained to be between 0 and 1, but not forced to sum to 1 in order to accommodate compounds that were absent from the set of used standards.

2.5. Dispersive X-Ray spectrometry

The distribution of As and Fe was analysed in Fe^0 -amended soil prior and after the thermal treatment using an Energy Dispersive X-Ray Spectrometer (EDS) X-Flash 4030 detector (Bruker Quantax). Samples were analysed under a controlled pressure water steam atmosphere. A 133 eV (at Mn K) energy resolution at 100,000 cps was achieved with a 30 mm^2 area solid state drift detector with a Peltier cooling device.

2.6. Statistics and geochemical modelling

The data was processed by analysis of variance (ANOVA) and regression analysis using the software *Statgraphics Plus 5.1*. A two-sample *t*-test ($p < 0.05$) was applied to differentiate between the sample means. Multivariate data analysis (MVDA) was performed using *SIMCA-P+ 11.5* (Umetrics). Principle component analysis was used to identify clusters of observations within the data set comprising total and leached concentrations of elements in the untreated and thermally treated soil.

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