

CHANGES IN LEACHING AND CHEMICAL FRACTIONATION OF ARSENIC, CHROMIUM AND COPPER IN SOIL AFTER THERMAL TREATMENT

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SUMMARY: Thermal treatment is used for remediation of soils contaminated with both organic and inorganic contaminants. The target is destruction of organic contaminants while the residue containing the inorganic contaminants has to be further treated. The effects of thermal treatment on As, Cr and Cu in contaminated soil are evaluated in laboratory and field tests, using leaching test, As speciation and sequential extraction test. In laboratory tests on four particle size fractions, the leaching of As and Cr increased by factors of 18-40 and 2-23, respectively, while the mobility of Cu decreased 12-14 fold after thermal treatment. The concentration of As(V) in the finest soil fraction increased 19 fold, while As(III) remained constant. Arsenic, Cr and Cu associated to the reducible soil fraction decreased, possibly due to enhanced crystallisation of Fe oxides and reduction of available adsorption sites. In the field test, the concentration of As, Cr and Cu was 1-4 times higher in the filter fraction compared to the treated soil.

1. INTRODUCTION

Soil contamination with various chemical substances is common at former wood preservation sites due to leakage and spillage of impregnation solutions during the impregnation process. The types of preservative used have changed over time, leading to the occurrence of mixed organic (e.g. creosote and pentachlorophenol (PCP)) and inorganic impregnation chemicals (e.g. chromated copper arsenate (CCA)) in the soil. Due to the complex contamination situation, the standard approach to remediate such co-contaminated sites is to excavate and landfill the soil. In Europe, this is permitted as long as the soil fulfils the EU criteria for waste acceptance at landfills (EC, 2003) However, landfilling does not solve the contamination problem in a long term perspective. If the soil does not fulfil the criteria, the remediation options are to treat the soil before landfilling.

Single remediation techniques useful for co-contaminated soils are limited. Usually a treatment sequence in several steps is required to destroy the organic contaminants and immobilize the inorganic contaminants.

Thermal treatment is an established and recommended technique for treatment of the organic contaminants in co-contaminated soils from wood preservation sites (US-EPA, 1995). During thermal treatment, organic contaminants will be separated, concentrated and destroyed, while most of the trace elements from the inorganic contaminants will remain in the treated soil. The fate of inorganic contaminants (trace elements) during thermal treatment depends on thermal process conditions, soil characteristic and thermodynamic properties of the elements (e.g. Eddings et al., 1994; Rizeq, 2000).

The mobility of trace elements in a soil is governed by sorption/desorption processes with organic matter (OM), clay minerals and metal oxides. Amongst the metal oxides, Fe oxides are the most important ones controlling these processes. The processes are dependent on factors such as pH, redox potential (E_h), and microorganism activity. Thermal treatment of a contaminated soil greatly affects these factors, mainly because of morphological (Merino et al., 2003) and chemical changes occurring in the clay minerals (Brindley and Lemaitre, 1987) and Fe oxides (Cornell and Schwertmann, 2003). The transformations affect the potential mobility of trace elements as As, Cr and Cu (Thurnau and Fournier, 1992; Tack and Verloo, 1993; Thurnau, 1996; Wei and Hsieh, 2006) and hence, the environmental impact of trace element emissions. The changes in the leaching potential of trace elements have to be considered concerning the next separation, concentration and immobilization step in a treatment sequence.

Changes in the redox potential may affect the speciation of As and Cr. As(III) and Cr(VI) are considered to be more toxic, more mobile and, hence, of greater environmental concern than As(V) and Cr(III). The association of trace elements with different chemical soil fractions (e.g. exchangeable, acid-soluble, reducible, oxidizable and residual fractions) and hence, differences in the binding strength and potential mobility of the trace elements will also be affected by thermal treatment (Tack and Verloo, 1993). Because the mineral composition between particle size fractions shifts in a soil, the changes in the associations with certain chemical soil fractions also differs between different particle size fractions (Lombi et al., 2000; Cai et al., 2002).

This study evaluates the effect of thermal treatment on the mobility, speciation and chemical fractionation of As, Cr and Cu in different particle size fractions in CCA contaminated soil using one-step batch leaching test, As speciation and sequential chemical extraction test. The study consists of both laboratory experiments and a field test.

2. MATERIAL AND METHODS

2.1 Soil and thermal treatment

Soil from a former wood preservation site contaminated with CCA was used in the laboratory experiments. The soil was dried to constant weight at 25 °C in a ventilated heating cabinet, homogenized, divided into sub-samples using a riffle splitter and sieved to < 10 mm.

Determination of particle size distribution of the soil (< 10 mm) was done according to Swedish standard methods SS 27123 and 027124 (SIS, 1992b, 1992a).

One sub sample of the soil was separated into four particle size fractions by dry sieving (< 0.125, 0.125-0.250, 0.250-0.500 and 0.500-1 mm). About 500 g of each fraction was thermally treated at 800 °C for 20 minutes in stagnant air in a muffle furnace. After treatment the soil was cooled to room temperature and stored in a desiccator before analysis.

2.2 Analytical evaluation methods

Total element concentrations (in reality, pseudo total element concentrations) were analysed on soil extracts prepared by digestion of 1 g of soil in 10 mL of aqua regia using a microwave

digester.

The effect of thermal treatment on the leaching of trace elements was assessed using the one-step batch leaching test EN 12457-2 (CEN, 2002). This test is the prescribed method in the Landfill Directive to regulate waste acceptance at landfills (EC, 2003). The leaching test was performed at a liquid to solid ratio of 10 L/kg dry weight (L/S 10), scaled to fit reduced sample sizes.

A sequential chemical extraction procedure described by Tessier et al. (1979) was carried out to operationally define four elemental fractions in the soil (fraction I-IV). A fifth residual fraction (fraction V) was determined through digestion in aqua regia of the residue from the fourth extraction step, using a microwave digester. The five operationally defined fractions were: (I) exchangeable, (II) acid-soluble (associated to carbonates), (III) reducible (associated to Fe and Mn oxides), (IV) oxidizable (associated to OM and sulphides) and (V) residual.

Elemental analyses of digestates, leachates and extracts were filtered through 0.45 µm filters, acidified with nitric acid (HNO₃) when necessary, and analysed by inductively coupled plasma optical emission spectroscopy (ICP-OES). All analyses were done on both untreated and thermally treated soil fractions.

Speciation of As(III) and As(V) in soil pore water was determined on the finest (< 0.125 mm) treated and untreated soil fraction. The soil (100 g) was packed into wide opened plastic bottles (ø6 cm) containing 5 cm soil moisture samplers and moistened with deionised water to reach 23% soil humidity level (slightly below the water-holding capacity (WHC) of the soil). Soil pore water was extracted five days later by the soil moisture samplers connected to 30 mL glass vacuum bottles. Analysis of As(III) and As(V) was carried out using ion chromatography followed by inductively coupled plasma mass spectrometry (IC-ICP-MS). Samples were stored at 4 °C before the analyses.

2.3 Chemical analyses

Total solids (TS) and loss on ignition (LOI) were determined according to Swedish standard SS 28113 (SIS, 1981) at 110 °C and 550 °C, respectively. The water-holding capacity (WHC) of the soil (< 10 mm) and the soil fraction (< 0.125 mm) was determined gravimetrically as the water remaining in the saturated soil after drainage (Shinner et al., 1996). Electrical conductivity (EC), pH and redox potential (E_h) of the soil were determined in soil–deionised water suspensions (1:2 w/w) after 30 min of mixing. Total organic carbon (TOC) of the soil fractions was analyzed using a TOC-SSM-500A analyzer according to European standard EN 13 137 for determination of TOC (CEN, 2001). Dissolved organic carbon (DOC) in leachates was analyzed using a TOC-VCPH/CPN instrument according to European standard EN 1484 for water analysis of TOC and DOC (CEN, 1997).

2.4 Field test

A full-scale thermal treatment experiment was performed on three batches of soil (30 tons each) contaminated with CCA and polycyclic aromatic hydrocarbons (PAH). A rotating kiln with continuous feed and with the soil in direct contact with the heat source was used for the experiment. The temperature interval in the kiln ranged from 800 °C in the inlet to 200 °C in the outlet. The feeding of each batch into the kiln took about 30 minutes. During the treatment process fine particles were separated from the bulk soil and transported with the air stream to cyclones and bag filters. The two soil streams, here after called "treated soil" and "filter fraction", were sampled apart from each other after the treatment.

The experiment was evaluated by analysis of the two soil fractions by total As, Cr and Cu concentrations, one-step batch leaching tests at L/S 10 L/kg dw, with subsequent analysis of As,

Cr and Cu in the leachate and finally, As speciation on the soil pore water. The main characteristic of the soil fractions were analysed by particle size distribution, total solids (TS), loss on ignition (LOI), pH, electrical conductivity (EC) and redox potential (E_h). The same analytical methods as in the laboratory tests were used. The untreated soil used in the experiment will be analysed further.

3. RESULTS AND DISCUSSION

3.1 Laboratory experiment

3.1.1 Soil

For both untreated and treated soil the highest contents of As, Cr and Cu were found in the finest fraction (< 0.125 mm), with the exception of As in the treated soil where no statistically significant difference between the < 0.125 mm fraction and the 0.500-1.0 mm fraction ($p < 0.05$) could be observed (Table 1). The fraction < 0.125 mm consisted of 41% sand (> 0.06 mm), 54% silt (0.002-0.06 mm) and 5% clay.

The concentrations in the untreated soil were about two times higher in the finest fraction compared to the other fractions. These results were expected since trace elements tend to be enriched in the clay fraction of a soil (e.g. Qian et al., 1996; Lombi et al., 2000). In the untreated soil fractions, pH varied between 6.0 and 6.9. After treatment the pH increased and varied between 7.6 and 8.8 (Table 1).

3.1.2 Trace element leaching

The effect of thermal treatment on the leaching of trace elements was assessed by a one-step batch leaching test, EN 12457-2 (CEN, 2002). The use of deionised water and oxic test conditions lead to the mobilisation of trace elements that are mainly soluble under oxidising conditions. Low redox conditions, which may be important for the mobility of As, were not considered. The leaching of As and Cr from the soil increased 18-40 and 2-23 times, respectively, while the leaching of Cu decreased by 12-14 times after thermal treatment (Figure 1).

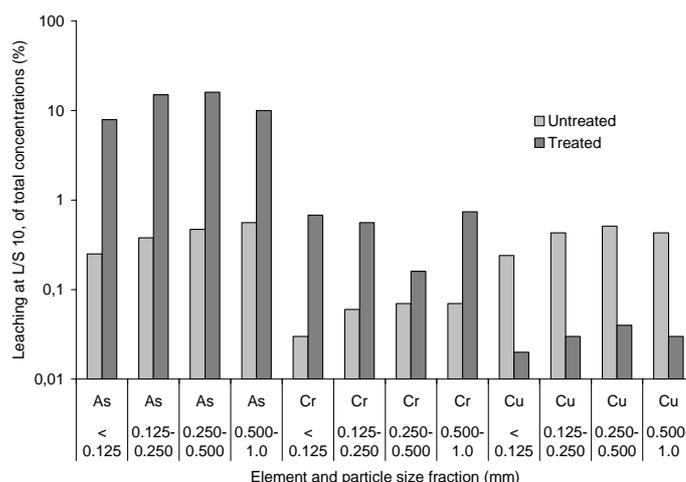


Figure 1. Relative leaching of As, Cr and Cu from untreated and treated soil fractions at L/S 10, as percentage of the total concentration in each soil fraction (n=3).

Table 1. Main characteristics of untreated and thermally treated soil fractions (\pm SD, n=3).

Soil properties	Unit	Untreated soil				Soil treated at 800 °C			
		< 0.125	0.125-0.250	0.250-0.500	0.500-1.0	< 0.125	0.125-0.250	0.250-0.500	0.500-1.0
		mm				mm			
pH	-	6.8	6.2	6.0	6.0	8.8	7.6	7.7	8.5
EC	μ S cm ⁻¹	1056	508	253	164	847	422	443	317
E _h	mV	447	420	422	434	427	418	433	438
TS	g kg ⁻¹	992	996	996	997	1000	1000	1000	1000
LOI	wt. %	3.0	1.7	1.6	1.2	ND	ND	ND	ND
TOC	wt. %	0.51	0.32	0.83	0.70	0.39	0.05	0.06	0.06
WHC	wt. %	39	ND	ND	ND	37	ND	ND	ND
Total element concentration									
As	mg kg ⁻¹ dw	352 \pm 7	191 \pm 4	179 \pm 23	148 \pm 33	359 \pm 2	187 \pm 3	187 \pm 23	297 \pm 82
Cr	"	155 \pm 2	93 \pm 2	93 \pm 15	82 \pm 22	158 \pm 1	89 \pm 4	83 \pm 8	108 \pm 4
Cu	"	94 \pm 1	49 \pm 1	43 \pm 5	40 \pm 6	76 \pm 1	35 \pm 1	33 \pm 2	45 \pm 3

ND = not determined

Before the treatment the partition between As(III) and As(V) was about 1:1.5 in the soil pore water. After treatment the partition between the species was 1:25. The concentration of As(III) remained constant during the treatment. The elevated level of As(V) in the soil pore water after thermal treatment could be due to an enhanced crystallization of less crystalline or amorphous Fe oxides during the treatment. Arsenic has a high affinity to Fe oxides in native soil, but high temperatures increase the crystallinity of the oxides and hence, available binding sites for As are reduced (Sørensen et al., 2000; Cornell and Schwertmann, 2003) and cause the release of bound As. The thermal treatment increased the leaching of As from a level where the soil could have been landfilled at a landfill for non-hazardous waste to a level where it exceeds the limit value (25 mg kg⁻¹ dw) for waste acceptable at landfills for hazardous waste (EC, 2003).

The changes in leaching coincide with studies of e.g. Thurnau and Fournier (1992) and Wei and Hsieh (2006) who observed an enhanced leaching of As and Cr respectively due to thermal treatment, and Wei et al. (2001) who reported a decrease in Cu leaching from a contaminated soil. According to Thurnau (1996), the leaching of As but not Cr is negatively correlated to the treatment temperature in the temperature interval 316-648 °C.

3.1.3 Fractional distribution

A sequential chemical extraction test was applied to assess five operationally defined chemical fractions of the trace elements in the soil. Operationally defined means that the obtained data are dependent on the experimental approach adopted. This test provides useful information about the binding form of the trace elements and the changes in solubility that occurred after the thermal treatment. The changes in fractional distribution of the trace elements due to thermal treatment at 800 °C are summarized in Table 2. The dominating changes for As are transitions from the reducible fraction (III) to the more easily soluble exchangeable (I) and to the acid-soluble fraction (II). The dominating translocations for Cr and Cu are from the reducible fraction (III) to the more recalcitrant residual fraction (V). Extractable Cr from the exchangeable fraction (I) increased slightly. The weakened role of Fe oxides for trace element sorption in thermally treated soil is evidenced by the significantly decreased trace element fraction associated with Fe and Mn oxides compared to the untreated soil.

Table 2. Changes in trace element extraction from elemental fractions determined by sequential extraction after thermal treatment at 800 °C. The changes are represented by the average of the changes in the particle size fractions < 0.125, 0.125-0.250, 0.250-0.500 and 0.500-1.0 mm.

Trace element	Changes in fractional distribution				
	I	II	III	IV	V
As	++	++	---	+/-	+/-
Cr	+/-	+/-	---	--	+++
Cu	-	-	--	-*	+++

--- = 25-50 percentage points (pp) decrease, -- = 10-25 pp decrease, - = 2-10 pp decrease, +/- = 2 pp increase/decrease in different fractions, + = 2-10 pp increase, ++ = 10-25 pp increase, +++ = 25-50 pp increase, *Standard deviation exceeds the mean value (n=4) more than 4 times.

3.2 Field test

The partition between the treated soil and the filter fractions was about 70 to 30% by weight, respectively. The particle size distribution of the filter fraction showed that 95% of the fraction was smaller than 0.125 mm. The concentration of trace elements in the filter fraction was 1-4 times higher than in the treated soil (Table 3), which confirms the findings from the laboratory experiment. The leaching of trace elements was higher from the filter fraction compared to the treated soil as shown in Figure 2a. The relationship between As(III) and As(V) was 1:11 in the filter fraction and 1:25 in the treated soil. In the untreated soil the relationship was 1:3.

Arsenic had the highest relative leaching (3-4%) compared to the total concentrations, while Cr and Cu from the treated soil had a relative leaching < 0.5% (Figure 2b). Compared to the leaching limit value (2 mg kg⁻¹ dw) of As at L/S 10 stipulated by the Council of European Union for acceptance of waste to landfills (EC, 2003), the treated soil slightly exceeded the limit value for waste acceptable at landfills for non-hazardous waste (Figure 2a).

The relative leaching of As from the large scale thermally treated soil and filter fraction was about 3 times lower than the relative leaching of As from all particle size fractions of the thermally treated soil in the laboratory experiment. It is possible that reduced zones were developed in the furnace during the laboratory experiment because the experiments were conducted in stagnant air and As become more mobile at low oxygen levels during thermal treatment. In a large scale thermal treatment experiment by Thurnau and Fournier (1992) an oxygen level of 11% decreased the As leaching about 4.6 times compared to an oxygen level of 7%.

Table 3. Main characteristics of full scale thermally treated soil (\pm SD, n=6 in treated soil, n=3 in filter fraction).

Soil properties	Unit	Batch 1		Batch 2		Batch 3	
		Treated soil	Filter fraction	Treated soil	Filter fraction	Treated soil	Filter fraction
pH	-	8.3	8.4	7.9	8.2	7.8	8.3
EC	μ S cm ⁻¹	110	269	109	259	105	386
E _h	mV	427	474	480	474	463	475
TS	g kg ⁻¹	1000	997	1000	1000	999	1000
LOI	wt. %	3.3	4.3	2.8	4.9	2.2	4.6
Total element concentration							
As	mg kg ⁻¹ dw	65 \pm 1	165 \pm 9	61 \pm 6	249 \pm 2	68 \pm 17	274 \pm 4
Cr	"	83 \pm 34	116 \pm 5	52 \pm 5	140 \pm 2	50 \pm 11	148 \pm 6
Cu	"	42 \pm 7	60 \pm 2	35 \pm 2	81 \pm 0	40 \pm 13	87 \pm 3

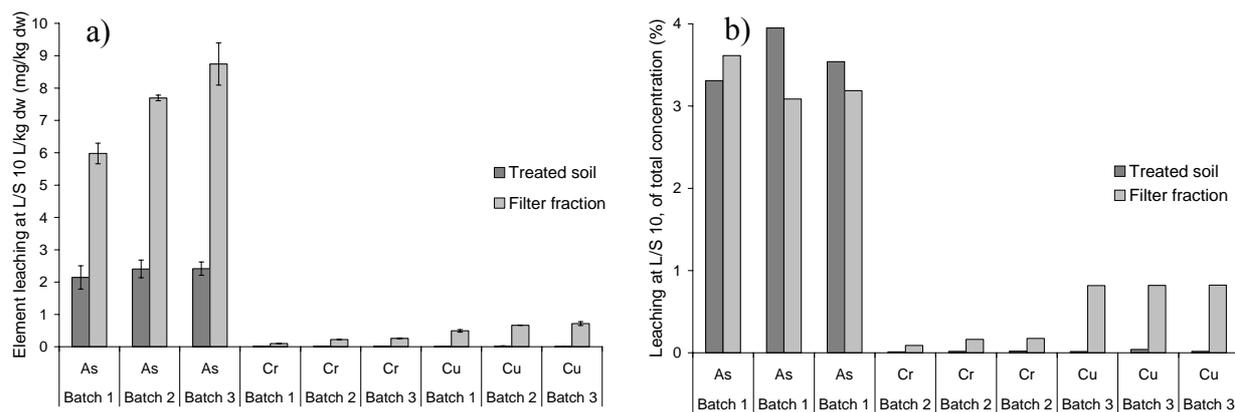


Figure 2. Trace element leaching from large scale thermally treated soil and filter fractions (a) and relative leaching of trace elements compared to total concentrations from thermally treated soil and filter fraction (b) at L/S 10 (n=6 for treated soil, n=3 for filter fractions).

Full-scale and pilot-scale thermal treatment tests have been used by others to evaluate the fate of As in rotary kiln facilities. The amount of As found in the treated residual soil varied between 80 and 90% of the total amount of As recovered in nine tests performed by Waterland et al. (1991).

An increase of the kiln exit temperature from 816 °C to 927 °C increased the amount of As in the flue gases and to the scrubber liquor during a thermal treatment test performed by Thurnau and Fournier (1992). Between 89 and 97% of the total recovered As was found in the treated residual soil after treatment. No relationship between chlorine content and As vaporisation was observed. A similar test with thermal treatment between 316 and 648 °C showed that 96% (average of 12 tests) of the feed As concentration was recovered in the residual soil (Thurnau, 1996).

3.3 Further research

The possibility to reduce the leaching of As by co-combustion of the contaminated soil with stabilising amendments containing Fe, Al and Ca will be investigated in laboratory tests.

4. CONCLUSIONS

The increased mobility of As after thermal treatment and the high concentration of trace elements in the finest soil particle size fraction have to be considered when thermal treatment is used on trace element contaminated soil.

Thermal treatment at 800 °C of a CCA contaminated soil affected the mobility and chemical fractionation of As, Cr and Cu. The leaching of As, with As(V) being the dominant specie, and Cr increased after treatment, while the leaching of Cu decreased.

The result from the sequential chemical extraction test on the soil before and after thermal treatment indicated that As, Cr and Cu associated to the reducible fraction decreased after treatment, most likely because of a reduction of available binding sites in Fe oxides due to the increased crystallisation of Fe oxides occurring at high temperatures. An increase of As associated to the more easily soluble exchangeable and acid-soluble fractions increases the potential mobility of As. Chromium associated to the exchangeable fraction increased slightly after thermal treatment even if it was the residual fraction that increased the most. This means that the mobility of Cr will increase in a short time perspective, but in a long term, Cr might

become more stable. Thermal treatment caused changes in the Cu fractionation which increased its stability.

A high oxygen level during the treatment process may reduce the leaching of As. Reduced zones in the thermal treatment facility must be avoided as far as possible.

In rotary kiln facilities, cyclone and filter fractions can be separated from the residual (coarse) soil. Due to the trace element enrichment in the filter fraction the treated soil and the filter fraction should be treated separately after the thermal treatment.

ACKNOWLEDGEMENTS

The study was financed by the European Union (Interreg III A Nord), the Finnish Funding Agency for Technology and Innovation (Tekes), Digipolis OY, the County Administrative Board of Norrbotten, Norrbottens Research council and EU Structural Funds and Objective 2, North Sweden Soil Remediation Center. The support and co-operation of Savaterra Oy and RagnSells AB is gratefully acknowledged.

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