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# Mobility and toxicity of heavy metal(loid)s arising from contaminated wood ash application to a pasture grassland soil<sup> $\star$ </sup>



POLLUTION

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#### ABSTRACT

Heavy metal(loid) rich ash ( $\leq$ 10,000 mg kg<sup>-1</sup> total As, Cr, Cu and Zn) originating from the combustion of contaminated wood was subjected to several experimental procedures involving its incorporation into an upland pasture soil. Ash was added to soil that had been prior amended with local cattle manure, replicating practices employed at the farm scale. Metal(loid) concentrations were measured in soil pore water and ryegrass grown on soil/manure plus ash mixtures (0.1–3.0% vol. ash) in a pot experiment; toxicity evaluation was performed on the same pore water samples by means of a bacterial luminescence biosensor assay. Thereafter a sequential extraction procedure was carried out on selected soil, manure and ash mixtures to elucidate the geochemical association of ash derived metal(loid)s with soil constituents. Predictive modelling was applied to selected data from the pot experiment to determine the risk of transfer of As to meat and milk products in cattle grazing pasture amended with ash.

The inclusion of manure to soils receiving ash reduced phyto-toxicity and increased ryegrass biomass yields, compared to soil with ash, but without manure. Elevated As and Cu concentrations in pore water and ryegrass tissue resulting from ash additions were reduced furthest by the inclusion of manure due to an increase in their geochemical association with organic matter. Zinc was the only measured metal(loid) to remain uniformly soluble and bioavailable regardless of the addition of ash and manure. Risk modelling on pot experimental data highlighted that an ash addition of >1% (vol.) to this pasture soil could result in As concentrations in milk and meat products exceeding acceptable limits.

The results of this study therefore suggest that even singular low doses of ash applied to soil increase the risk of leaching of metal(loid)s and intensify the risk of As transfer in the food chain.

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#### 1. Introduction

The majority of studies of wood ash application to land have been carried out on agricultural or forest soils, normally with the feedstock used to generate the ash coming from virgin timber (reviewed in Pitman, 2006). Agricultural soils display welldocumented benefits to their properties after wood ash addition, including an increase in soil pH and nutrient status (Bougnom et al., 2009; Klemedtsson et al., 2010; Pérez-Cruzado et al., 2010; Arshad

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et al., 2012; Bougnom et al., 2012; Saarsalmi et al., 2012; Podmirseg et al., 2013) and resultant improvements in crop biomass and yields (Pérez-Cruzado et al., 2010; Bougnom et al., 2012; Materechera, 2012; Moilanen et al., 2012; Saarsalmi et al., 2012). Combustion of wood that has been previously treated with weatherproof protectants, paints and preservatives, or impregnated/coated with heavy metal(loid)s and organic compounds can result in an ash that is highly concentrated in both trace nutrients and heavy metal(loid) s, arising from the additive treatments (Balasoiu et al., 2001). For example, Solo-Gabriele et al. (2002) measured concentrations ranging from 730 to 99,300 mg kg<sup>-1</sup> total As,  $\leq$ 165,000 mg kg<sup>-1</sup> total Cr and 98,450 mg kg<sup>-1</sup> total Cu in samples of ash produced from wood previously treated with a commonly used anti-fungal/



bacterial treatment 'chromated copper arsenate (CCA)'. Studies that have applied wood ash generated from reclaimed [contaminated] feedstocks have reported that heavy metals from the ash are bioavailable and potentially phytotoxic. Lucchini et al. (2013) studied the addition of a Cu rich ash (~200 g kg<sup>-1</sup>) to two agricultural soils (pH 5-6) finding that increased soil Cu concentrations and uptake to sunflower (Helianthus annuus L.) resulted, compared to the control soil without ash. This study also noted adverse effects to plant biomass and yield resulting from this ash addition. In these experiments the authors noted that the pH increases to soils immediately after ash addition were not maintained throughout the experiment due to the leaching of alkaline metals. Jones and Quilliam (2014) experimented with applications of ash produced from various ratios of contaminated and non-contaminated wood combusted together, to a podzol soil (pH 5). In common with the findings of Lucchini et al. (2013), the addition of Cu contaminated ash to soil resulted in an increased bioavailability of Cu in soil and concomitant uptake to wheat (Triticum aestivum L.). Whilst ash addition to agricultural soils can potentially be beneficial, the application of wood ash from waste wood sources is likely to increase metal(loid) loadings in soils but there is little experimental evidence available related especially to As and Cr fate in soils amended with contaminated wood ashes.

In the following study we examined the fate of the metal(loid)s As, Cr, Cu and Zn which were present in excessive concentration in wood ash generated from the combustion of mixed source waste wood. The aim was to 1) determine the bioavailability and toxicity of metal(loid)s from the ash when added to an agricultural soil, 2) ascertain whether co-application of ash with manure could change the geochemical fractionation of ash-derived metal(loid)s and reduce their bioavailability and phyto-toxic effects, and 3) use modelling to indicate the longer-term risk of As from the ash entering the food chain.

#### 2. Methods

#### 2.1. Soil, amendments and their characterisation

Soil, manure and ash were obtained from a 1,000Ha upland farming estate in north-east Scotland (UK) stocked with 450 Scottish Blackface and 500 crossbred ewes, 50 suckler cows and 100 farmed red deer hinds. Soils on the farm are freely drained humus iron podzols developed on old red sandstone and part of the Strichen Association (Glentworth and Muir, 1963). The soil collected for this experiment was from part of the farm designated for experimental work having received no prior amendment of manure or ash and being lightly grazed seasonally (sandy-loam texture, pH (H<sub>2</sub>O) 5.8; organic carbon (OC) contents of ~7% and nitrogen (N) contents of ~0.6%). After sward removal a horizontal soil pit was excavated and the top 30 cm of soil bagged and returned to the laboratory (A and B horizons). The soil was then air dried at 25 °C for 1 week before it was homogenised by hand and sieved to <10 mm. Ash was sampled from a 70 kW wood fired biomass burner that provides heat for the farm buildings. The feedstock for the biomass boiler had consisted of mixed virgin timber and waste wood such as fencing, pallets and doors (chromated copper

Table 1Metal(loid) concentrations (pseudo-total) of soil, manure and ash. All values are themean of replicates  $(n = 5) \pm s.e.m.$ 

mg $kg^{-1}$	As	Cr	Cu	Zn
Soil	$\begin{array}{l} 4.5 \pm 0.2 \\ 5.4 \pm 0.4 \\ 9259 \pm 649 \end{array}$	23.9 ± 2.1	8.8 ± 0.6	$23.2 \pm 1.4$
Manure		20 ± 2	23 ± 2	$169 \pm 12$
Ash		9914 ± 715	8793 ± 632	$4667 \pm 374$

arsenate-CCA-treated wood). Aside from heavy metal(loid)s, detailed in Table 1, the sampled ash was additionally composed of Ca (20%), K, (7%), Mg (3%), C (2.5%), P (1%), Fe and Al (<1%). The N content was <0.1%. Ash was obtained directly from the boiler's combustion chamber, bagged and returned to the laboratory. The manure, consisting of cattle excreta, was obtained from the farm's manure pit where it had partly decomposed. It was dried at 25 °C for ease of handling and then homogenised by hand. Relevant chemical characterisation of the soil, manure and ash was carried out as follows and is presented in Table 1.

Sub samples of soil, manure and ash were block digested using nitric acid (Meharg et al., 2013) to determine heavy metal(loid) concentrations. In brief, sub samples were oven dried, ball-milled and then 0.1 g was weighed into a glass digestion tube; 2.5 ml of concentrated nitric acid was applied and the sample left overnight. A total of 2.5 ml of hydrogen peroxide was added to the samples prior to being heated on a heating block which was gradually increased to 140 °C and then maintained at 140 °C for 4 h. After digestion the samples were made up to a total volume of 100 ml. In addition to the sample, 3 replicates of reference material (NCS ZC73007) and 3 analytical blanks were also digested and included for quality control. Samples were analysed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS; Agilent Technologies 7500).

A further subsample of ash was subjected to a column leaching procedure to determine the solubility of metal(loid)s in the ash during a pre-determined time. Glass columns (dimensions 8 cm length and 1.5 cm internal diameter) were filled with 10 g of ash (Fig. 1A.). To avoid movement of ash material out of the columns during leaching, glass-wool was packed into the column at both ends after 0.2 µm filter papers had been prior inserted. The filter papers ensured that no particulate matter remained in the sample to be analysed. The columns were leached from their base via a peristaltic pump using de-ionised water as the eluent at a flow rate of 1 ml min<sup>-1</sup>. A second pump was attached to the top of the column, at the same flow rate, in order to maintain neutral pressure in the columns. The purpose of leaching from the base of the column was to eliminate gravitation effects and the preferential passage of eluent through least resistant pathways in the ash inside the column, to ensure as far as possible that the whole mass of ash was leached uniformly. The columns were constantly leached for 2000 min<sup>-1</sup> during which time an auto-sampler distributed 2 ml of leachate into tubes at pre-determined intervals of 10, 100 and 1000 mins<sup>-1</sup> until 2000 mins<sup>-1</sup>. At the end of the experiment samples were analysed using the ICP-MS, as described above. Leachate pH was also determined and reported in Table 2.

The obtained raw data were converted from mg  $l^{-1}$  into cumulative mass of each metal(loid) (mg kg<sup>-1</sup>), based on the volume of eluent passed through the column in each time step and the mass of ash in the column. Therefore a total mass of metal(loid)s extracted from the ash at pre-determined times could be calculated as a percentage of the total extracted throughout the duration of the experiment; as presented in Table 2.

#### 2.2. Experimental pot setup

A total of 4 different treatments involving mixtures of soil, manure and ash were tested in this experiment in addition to 3 controls, with 5 replicates in all cases (see Fig. 1B). To create those treatments, soil and manure (M; 10% v:v) was first mechanically mixed using an end-over-end method to create a homogenous soil and manure mixture. Manure is mixed with ash at the farm scale to prevent ash dispersal by wind-blowing, so this process was replicated here in a controlled fashion. Ash (A) was applied to the soil/ manure mixture at the following 4 vol and mixed in the same way; Download English Version:

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