



Water treatment residuals as soil amendments: Examining element extractability, soil porewater concentrations and effects on earthworm behaviour and survival



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ABSTRACT

Drinking water treatment residuals (WTRs), the by-product of water clarification processes, are routinely disposed of via landfill however there is a growing body of research that demonstrates the material has great potential for beneficial use in environmental applications. Application to agricultural land is one option showing great promise (i.e. a low cost disposal route that provides organic matter input to soils and other potential benefits), however questions remain as to the impact such applications may have on earthworm survival and behaviour and also on the potential effects it may have on soil porewater chemistry. This study examined the leachability of elements within two types of WTRs (one Al- and one Fe- based) from England via 0.001 M CaCl₂ solution, at varying pH, and via the Community Bureau of Reference (BCR) sequential extraction scheme. Earthworm avoidance, survival, growth, reproduction and element concentrations were examined in WTR-amended sandy soils (0%, 5%, 10%, 20% w/w), while soil porewaters were also recovered from experimental units and examined for element concentrations. The results revealed leachable element concentrations to be very low in both types of WTRs tested and so element leaching from these WTRs would be unlikely to pose any threat to ecosystems under typical agricultural soil conditions. However, when the pH was lowered to 4.4 there was a substantial release of Al from the Al-WTRs (382 mg/kg). Soil porewater element concentrations were influenced to some degree by WTR addition, warranting further examination in terms of any potential implications for nutrient supply or limitation. Earthworm avoidance of WTR-amended soil was only observed for Al-WTRs and only at the maximum applied rate (20% w/w), while survival of earthworms was not affected by either WTR type at any application rate. Earthworm growth and reproduction (cocoon production) were not affected at a statistically significant level but this needs further examination over a longer period of exposure. Increased assimilation of Al and Fe into earthworm tissues was observed at some WTR application rates (maximum fresh weight concentrations of 42 mg/kg for Al and 167 mg/kg for Fe), but these were not at levels likely to pose environmental concerns.

1. Introduction

Clarification of drinking water supplies is commonly achieved by treatment with aluminium (Al) or iron (Fe) salts, which remove impurities through coagulation and co-precipitation into a sludge like material referred to as drinking water treatment residuals (WTRs). Thus WTRs are primarily composed of Al(OH)₃ or Fe(OH)₃ plus organic matter, clay particles and other components (e.g. nutrients, contaminant metals and other impurities) removed from the raw water (Bugbee and Frink, 1985; Graveland et al., 1993). Vast quantities (i.e. millions of tons) of WTRs are produced globally (Babatunde and Zhao,

2007), with the majority disposed of via landfill. However, landfill disposal is increasingly expensive and may be wasting a potentially useable material; an increasing array of potential beneficial uses of WTRs have been researched and demonstrated over the last two decades, including use in constructed reedbeds or as a soil amendment to manage phosphorus (P) mobility within catchments (Babatunde et al., 2011; Ippolito, 2015; Oliver et al., 2011), land application to increase organic matter and water holding capacity and related soil parameters (e.g. Ahmed et al., 1998; Bugbee and Frink, 1985), and most recently as a way of remediating polluted soils through immobilisation of contaminants by WTRs (Garau et al., 2014, 2017; Wang et al., 2012).

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Beneficial use of WTRs is therefore an attractive option that offers financial advantages and facilitates development of a more circular economy with greater levels of materials recycling. However, while land applications of WTRs can be beneficial there are uncertainties that remain, including the mobility of elements within WTRs (particularly Al) and any ecotoxicological impacts on soil ecosystems linked to that or other changes brought about by WTR addition. For this reason there are still tight controls on where WTRs can be applied (e.g. in the UK it is only permitted on soils with pH > 6.0). Some studies have found no negative impacts on plants or plant yield increases following WTR application to 'clean' agricultural soils (Ahmed et al., 1998; Geertsema et al., 1994), while others have noted plant yield reductions that were attributed to restrictions in bioavailable P (Lombi et al., 2010; Oladeji et al., 2007). While a number of studies have investigated the effects on microbes following soil amendment with WTRs (e.g. Garau et al., 2017), very few, if any, have examined the influence of WTR application on earthworms. This is a major gap in current understanding of the risks and benefits of using these materials in agricultural soils, especially considering that earthworms are widely recognized as essential ecosystem engineers that provide a host of advantages for soil health and development (e.g. creation of pore channels, improved aeration and hydraulic conductivity, nutrient cycling, etc). The aims of the present study were to fill this gap, and to further scientific understanding of the behaviour and ecological effects of WTR components when the materials are applied to soils, by examining two WTR types from central England, UK, and determining i) the leachability of elements via single solution extraction at varying pH, ii) the fractionation of key elements within WTRs, iii) the influence of WTRs on the survival, growth and reproduction of the earthworm *Eisenia fetida*, and iv) the influence of WTR application on soil porewater element concentrations (because the majority of soil biota assimilate nutrients and contaminants via the soil porewater).

2. Methods

2.1. Water treatment residuals and soil

Partially dewatered WTRs from two water treatment plants in Staffordshire, England, one of which primarily uses Al salts (producing Al-WTRs; once dry, pH 7.34 ± 0.06 , OM $28.0 \pm 0.1\%$, Al $11.64 \pm 1.08\%$, Fe $0.91 \pm 0.08\%$, w/w) and the other primarily Fe salts (producing Fe-WTR; pH 7.37 ± 0.01 , OM $25.9 \pm 0.2\%$, Al $0.71 \pm 0.12\%$, Fe $17.69 \pm 0.19\%$) in their respective water treatment processes, were supplied by Severn Trent Water. The original 'as received' water content was high (~ 80% of total mass, determined on subsamples oven dried at 105 °C) so the WTRs were air-dried with the assistance of an oven set at 30 °C. During the ~ 2 week drying period required to reach stable mass, the WTRs were broken down to small pieces by hand on a daily basis to avoid large clods forming that, once dried, would present difficulties for hand crushing using a pestle and mortar. Once dried, the WTRs were crushed to pass a 2 mm sieve. Organic matter content was determined by loss on ignition at 450 °C, pH was determined in 0.001 M CaCl₂ extracts using a Jenway 3510 pH meter and probe, and pseudo total element concentrations were determined via microwave (CEM Mars 6) assisted mineral acid digestion (0.3–0.5 g solid; 9 ml HCl + 3 ml HNO₃, i.e. *aqua-regia*, n = 3) and analysis via ICP-OES (Optima 5300 DV instrument, Perkin Elmer, UK) as per USEPA method 3052 (see Supplementary information Table 1). Due to the high organic matter content, samples were combusted for 4 h at 450 °C prior to digestion. All acids used in the digestions were trace analysis grade (e.g. Aristar and Primar plus) and a certified reference material (CRM033 Loamy Sand; Trace Metals - Loamy Sand 10, Sigma-Aldrich) was digested and analysed alongside samples for quality assurance purposes. Measured values for relevant elements in the CRM closely matched certified values (e.g. 97–117% for Fe, Pb and Zn).

A sandy soil from Sevenoaks, Kent, UK, provided by a commercial

supplier (Bourne Amenity) and known to be free from contaminants, was used in the experiments. A sandy soil was selected because this would maximize the likelihood of identifying elements that leach from the WTRs into the soil and therefore into soil porewater. Organic matter content (1.1%) and pH (6.78 ± 0.1) were determined while particle size distribution (1% clay, 2% silt and 97% sand) was determined by first combusting at 450 °C, soaking in calgon solution and then analysing on a Coulter LS230 optical laser particle size analyser. Water holding capacity (WHC) was determined as 0.37 ± 0.02 ml/g by fully saturating 100 g, allowing to drain and then measuring retained water.

2.2. pH buffering capacity and element leachability

The pH buffering capacity of WTRs and their extractable element contents were determined in 0.001 M CaCl₂ (3 g solid; 30 ml solution; n = 3) extracts (Degryse et al., 2007; Hamels et al., 2014) that had been adjusted to varying acid levels. For Fe-WTR samples, the solutions were adjusted to four acid levels using high purity HCl (0, 0.013, 0.032 and 0.064 M HCl), while for Al-WTRs three acid levels were imposed (0, 0.013 and 0.064 M HCl). Once solutions were added samples were sealed, shaken by hand for 30 s, then shaken for 48 h on an end-over-end shaker, centrifuged and then a portion used for pH measurement and the remainder filtered (0.45 µm cellulose acetate syringe filter) before analysis by ICP-OES and ICP-MS (Agilent 7500ce).

2.3. Element fractionation (BCR sequential extraction)

Many sequential extraction schemes have been devised that attempt to identify fractions within soils and sediments with which elements of interest are associated. All have limitations and all generate operationally defined fractions (see review by Bacon and Davidson (2008), but they are nonetheless useful for identifying easily extractable vs recalcitrant element contents and for comparative purposes. The scheme devised by the Community Bureau of Reference (BCR) (Ure et al., 1993) has been employed extensively to examine metal fractionation in river sediments (Martinez-Santos et al., 2015; Pulford et al., 2009), aquaculture sludges (Nematī et al., 2011), sewage sludge (Scancar et al., 2000), urban soils (Gál et al., 2008; Madrid et al., 2007), agricultural soils (Kosolsaksakul et al., 2014), upland peat soils (Bacon et al., 2006), battlefield soils (Oliver et al., 2008) and in soils were pollution remediation trials (e.g. immobilisation with biochar or by zeolite formation) have been conducted (Belviso et al., 2010; Ippolito et al., 2017), hence it was chosen for this study. In the BCR procedure, 1.0 g oven dry equivalent samples are subjected to the following extraction regime. Step 1 (targeting the 'exchangeable' fraction): 40 ml 0.11 M acetic acid, shaken over-night, centrifuged, supernatant removed and filtered (0.45 µm cellulose acetate syringe filter) before analysis by ICP-OES. Step 2 (targeting the 'reducible fraction', indicative of Fe/Mn oxide-bound): 40 ml 0.1 M hydroxyl ammonium chloride adjusted to pH 2.0 with concentrated (15.8 M) HNO₃ is added to the residue from step 1, shaken over-night, then centrifuged, with the solution removed, filtered and analysed as in step 1. Step 3 (oxidisable fraction, indicative of organic matter bound): residues from step 2 were treated with 10 ml hydrogen peroxide (> 30% w/v, added as supplied), left to stand at room temperature for 1 h, heated in a water bath at 85 °C for 1 h then reduced to near dryness (< 1 ml volume). Each sample then received 40 ml 1.0 M ammonium acetate (adjusted to pH 2.0 with 15.8 M HNO₃) and was shaken over-night followed by extraction, filtration and analysis performed as above. Step 4 (residual fraction): this additional recommended (Rauret et al., 1999), and widely adopted, step to the original BCR procedure enables assessment of element mass balances (i.e. sample recoveries). Here, residues from the above 3-step sequence were digested in *aqua-regia* as described in Section 2.1 and analysed by ICP-OES. Analyses of BCR fractions were conducted using matrix-matched standards (range 0.1–100 mg/L).

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