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Effectiveness and longevity of a green/food waste derived compost packed column to reduce Cr(VI) contamination in groundwater

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ABSTRACT

PAS100 accredited compost derived from green and food waste sources was used to remediate groundwater containing Cr(VI) at a historically contaminated site in Falkirk, Scotland, UK. The compost was mixed with gravel at a ratio of 1:1 (v/v) to provide a reactive bedding material in an upflow column. The Cr(VI) concentration in the groundwater (inlet) ranged from 0.5 to 7.8 mg L⁻¹ during the 168 d trial period. After an acclimation period of 54 d, the flow rate was increased in the column from 5.8 to 8 mL min⁻¹. Cr(VI) in the outlet was less than 100 μ g L⁻¹ up to 134 d, after which the concentration steadily increased till 168 d. Compost analysis following completion of the trial confirmed that Cr(VI) was captured within the column. Anaerobic microbial reduction of Cr(VI) to Cr(III) was thought to be a key mechanism responsible for the longevity of the system to remove Cr(VI) from the groundwater. Requiring no additional organic carbon or nitrogen during the trial period, this setup represents a cost-effective treatment approach for low flow-through systems.

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1. Introduction

Hexavalent chromium (Cr(VI)) is regarded as a contaminant of concern as chromate and dichromate anions such as $HCrO_4^-$ and $Cr_2O_7^{2-}$ are highly toxic and potential carcinogens and mutagens [1]. The toxicity of Cr(VI) is in part due to its high solubility, which results in its ability to pass across cell membranes [2]. Being highly soluble, Cr(VI) is found in polluted water from industrial processes including leather tanning, electroplating, paint manufacture, metal manufacture and processing, as well as, agricultural runoff [3]. In a number of global regions including the central belt of Scotland, UK, deposited chromium ore process residue, the waste product from historical 'high lime' chromium smelting, is a major source of groundwater contamination [4].

In contrast to Cr(VI), Cr(III) is far less toxic and relatively insoluble, therefore, approaches to treat Cr(VI) contaminated groundwater are usually dependent on reducing Cr(VI) to Cr(III) [5]. Traditionally, this has been accomplished by chemical reducing agents such as sodium bisulphite, Fe(II) compounds, sulphur dioxide, amongst others [6]. Although effective, these reducing agents and the process plants used to deliver them are considered expensive [7].

A potential alternative to chemical reducing agents is microbial mediated reduction of Cr(VI) [8]. Towards this aim, a number of aerobic and anaerobic micro-organisms have been isolated [9,10] and in some cases their Cr(VI) reductase enzymes purified [8]. Under anaerobic conditions, dissimilatory metal-reducing bacteria utilise Cr(VI) as a terminal electron acceptor [11]. However, common metabolites of anaerobes such as H_2S and Fe(II), from sulphate reducing bacteria and iron reducing bacteria respectively, can also reduce Cr(VI) indirectly [8], suggesting a wide diversity of commonly found micro-organisms capable of Cr(VI) reduction.

Recently, there has been an increasing interest in the use of lowcost organic materials such as composts and seaweed, as well as, exhausted coffee grounds and coconut coir for remediation of Cr(VI) contaminated groundwater [6,7,12–15]. The majority of these have studied the removal of Cr(VI) in batch and column experiments with an emphasis on adsorptive removal mechanisms [7,12,16,17]. However, recently, it has been demonstrated that such organic materials can act as reductants of Cr(VI) [18]. In addition, many of these organic materials, being rich in organic carbon and nutrients, can stimulate conditions for indigenous microbial Cr(VI) reduction without the need for costly cell isolation or enzyme purification. Finally, the use of organic materials in permeable reactive barriers or fixed bed biofilm reactors could lead to low-cost technologies for Cr(VI) contaminated groundwater [19,20].

Despite the potential advantages, few studies have assessed the effectiveness of organic materials as part of microbial mediated Cr(VI) reduction. Boni and Sbaffoni [2] assessed green waste compost for Cr(VI) remediation in an upflow fixed bed column, however, the column was fed with an artificial Cr(VI) solution, with additional carbon and nutrients added periodically. In addi-

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Tuble	-
Green	food compost (GFC) characterisation

Parameter	Units ^a	GFC		
рН		7.32		
Moisture content	%	42.8		
Chromium (total)	mg kg ⁻¹	8.6		
Ammonia as NH4 ⁺	mg kg ⁻¹	5700		
Total ammonia	mg kg ⁻¹	4500		
Phosphate	mg kg ⁻¹	11,000		
Potassium	mg kg ⁻¹	5500		
TOC by ignition in O ₂	%	5.3		

^a mg kg⁻¹ equals mg kg⁻¹_{dry weight}, unless otherwise stated.

tion, as far as we are aware, compost derived from green and food waste sources has not been studied in this manner. Green/food waste compost is known to possess certain benefits over green only compost, including higher nutrient (such as nitrogen) levels. Therefore, the aims of our study were (1) to assess the effectiveness of green/food waste derived compost for the treatment of Cr(VI) contamination groundwater, and (2) determine the longevity of the system without carbon and nitrogen amendment other than from the compost or present in the inlet water. Finally, compost material meeting the British Standards Institute Publically Available Specification (BSI PAS) 100 (hereafter referred to as PAS100) was chosen for the study. PAS100 is a UK national compost benchmark to ensure compost is processed to minimum specifications and prepared from suitable materials. For the purpose of this and future studies PAS100 will help to ensure material is of consistent quality and composition.

2. Materials and methods

2.1. Materials

Commercial compost prepared from a mixture of green and food waste feedstocks (designated green/food compost or 'GFC') and accredited as PAS100 material was obtained from Natural Organic Fertiliser Company Ltd. (Perth, UK) in April 2009. Compost characterisation from the sampled GFC material can be found (Table 1). Siliceous gravel (8 mm size) was washed with 10% (v/v) nitric acid and rinsed with deionised water. The gravel (2.5 kg) was mixed with 2.5 kg of GFC to create the column bed material. All chemicals used were purchased from Fisher Scientific UK Ltd. (Loughborough, UK), except diphenylcarbazide (DPC) pillows which were supplied by Hach Lange Ltd. (Manchester, UK).

2.2. Experimental system setup

An experimental column was setup alongside a full scale chromium groundwater treatment plant in Falkirk, Scotland. The site was a former chemical, iron and pre-cast concrete works and is now a vegetated spoil heap. Chromium in groundwater from the spoil heap impacts a local burn eventually leading into the River Carron. The current chemical treatment system operates to a discharge limit of $0.1 \text{ mg L}^{-1} \text{ Cr(VI)}$.

Groundwater characterisation from samples taken in April 2009 can be found (Table 2). The column was run from April to September 2009, and consisted of a Perspex cylinder (100 cm length, 8.4 cm internal diameter) equipped with two sampling ports, in addition to the inlet and outlet (Fig. 1). The column was filled with a 1:1 mix (w/w) of GFC and gravel.

Cr(VI) contaminated water was pumped to the column from an intermediate bulk container receiving water directly from the historically contaminated site. A multi-channel peristaltic pump was used to achieve an initial flow rate within the column equal to $5.8 \text{ mL} \text{min}^{-1}$ (phase 1). One pore volume corresponded to approxi-

Table 2	
Groundwater characte	erisation.

Analyte	Units	Value
рН		6.6-7.4
Arsenic, soluble	$\mu g L^{-1}$	7.2-12
Calcium, soluble	mgL^{-1}	63-90
Chromium, soluble	mgL^{-1}	1.9-7.5
Iron, soluble	mgL^{-1}	0.11-0.42
Lead, soluble	$\mu g L^{-1}$	0.7-1.1
Magnesium, soluble	$mg L^{-1}$	16-23
Manganese, soluble	$mg L^{-1}$	< 0.010
Nickel, soluble	$\mu g L^{-1}$	0.5-10
Phosphorus, total	mgL^{-1}	<0.20
Potassium, soluble	mgL^{-1}	9-21
Sodium, soluble	mgL^{-1}	22-57
Hexavalent chromium	$mg L^{-1}$	1.6-6.6
Sulphate as SO4 ²⁻	$g L^{-1}$	0.08-0.2
Ammonium as NH4 ⁺	$mg L^{-1}$	< 0.05
Chloride as Cl	$mg L^{-1}$	8-14
Nitrate as N	mgL^{-1}	<2.0
Sulphide as S	$\mu g L^{-1}$	<10
TOC (filtered)	mgL^{-1}	8.7-67

mately 2760 mL, equating to a hydraulic retention time of 8 h. After day 54, the flow rate was increased to 8 mL min⁻¹ (phase 2), thereby reducing the hydraulic retention time to 5.75 h.

Every 7 d, samples were taken from the inlet, outlet, bottom and top sampling ports. Samples were analysed immediately or stored at 4 °C prior to analysis (maximum ten days storage).

2.3. Groundwater analysis

Liquid samples were analysed for the following:

- (i) pH, conductivity, oxidative-reduction potential (ORP), dissolved oxygen (DO) and groundwater temperature were determined immediately onsite using a multi-parameter probe (HI9828; Hanna Instruments Ltd., Bedfordshire, UK). The instrument was calibrated prior to every use.
- (ii) Cr(VI) was determined using the diphenylcarbizide assay [21] and analysed on a spectrophotometer (DR2800; Hach Lange Ltd., Manchester, UK) following the manufacturers' instructions. Total suspended solids (TSS) were also determined using a spectrophotometer (DR2800; Hach Lange Ltd., Manchester, UK) following the manufacturers' instructions.



Fig. 1. Schematic of pilot column setup.



Fig. 2. Cr(VI) concentration at each sampling port of the column during phases 1 (0–54 d) and 2 (54–168 d).

(iii) All other parameters were analysed by an external UKAS accredited laboratory. Specifically, chromium (total) was analysed by inductively coupled plasma mass spectroscopy and total organic carbon (TOC) by UV persulphate oxidation.

2.4. Solid phase characterisation

GFC was analysed prior, as well as, on completion of the trial for the parameters outlined (Table 1). pH was determined as a 1:5 water extraction and moisture content as % of fresh weight. All other parameters were analysed by an external UKAS accredited laboratory. Specifically, metals were analysed by inductively coupled plasma optical emission spectrometer (ICP-OES) and TOC by ignition in oxygen.

3. Results

3.1. Chromium (total) and (VI) removal from water

Cr(VI) concentration in the groundwater ranged from 0.5 to 7.8 mg L^{-1} during the 168 d trial (Fig. 2). With the exception of the period 106–152 days, the concentration was in the range of $4.6-7.8 \text{ mg L}^{-1}$.

For the experimental phase 1 (0–55 d), the groundwater flow rate to the column was maintained at 5.8 mLmin^{-1} . The concentration of Cr(VI) in the outlet was 0.15 mg L^{-1} at 1 d, after which it was less than the detection limit (0.02 mg L^{-1}) of the colorimetric method. The concentration of Cr(VI) in bottom and top ports was also found to be below the detection limit, except on day 43 when 0.2 mg L^{-1} Cr(VI) was detected in the bottom port. At day 55, the flow rate was increased to 8 mLmin^{-1} , corresponding to phase 2 of the experiment. At day 57, Cr(VI) was detected in the bottom port, after which the concentration increased till 98 d before largely replicating the inlet Cr(VI) concentration. The top port and outlet showed similar patterns in experimental phase 2 (due to compaction of the compost/gravel to a level just below the top port). In these sample locations, Cr(VI) was less than 0.1 mg L^{-1} up to 134 d, except on 98 d when the top port recorded 0.24 mg L⁻¹.

Analysis of Cr(total) by ICP-MS meant that the chromium concentration in the groundwater could be determined to a detection limit an order of magnitude lower than for (CrVI) at $2 \mu g L^{-1}$. As a result, the lowest Cr(total) found in the outlet was $9 \mu g L^{-1}$. In general, Cr(total) in the outlet was less than 100 $\mu g L^{-1}$ from 14 to 134 d, except for samples at 111 d (106 $\mu g L^{-1}$) and 125 d (104 $\mu g L^{-1}$),



Fig. 3. Cr(total) concentration for inlet and outlet of the column at times 0-168 d.

respectively (Fig. 3). After 134 d, the Cr(total) concentration steadily increased till the end of the experiment (168 d).

3.2. Dissolved oxygen

Dissolved oxygen (mgL^{-1}) was determined in samples taken immediately from the column from 0 to 152 d (Fig. 4). No values were reported for samples at 168 d due to failure of the dissolved oxygen probe. In general, dissolved oxygen showed a trend of decreasing concentration from inlet to outlet, with the greatest difference between inlet and bottom port. Dissolved oxygen (mgL^{-1}) concentrations were relatively stable at the different sample locations until 120 d when a significant drop was observed, most prominent for the inlet. The drop in dissolved oxygen readings is thought to be associated with ultimate failure of the probe rather than a change within the column system.

3.3. Oxidative reduction potential (ORP)

ORP at the inlet was generally positive throughout, except during the period 21-70 d when slightly negative values were recorded (maximum -77.5 mV). ORP were initially positive (80-150 mV) for all sampling locations, after which the bottom port, top port and outlet became increasingly negative during the first 55 d (Fig. 5). A maximal value of -269 mV was achieved in the outlet at 35 d. ORP became less negative in the bottom port, top port and outlet



Fig. 4. Dissolved oxygen for each sampling port of the column at times 0-152 d.



Fig. 5. Oxidative-reduction potential (ORP) for each sampling port of the column at times 0–168 d.

between 55 and 134 d, although they were still in the region of -80 to -200 mV. After 134 d, a sharp increase to positive ORP values was observed in these sample locations.

3.4. Groundwater temperature

The temperature of groundwater through the column fluctuated between 13.6 and 20.5 $^{\circ}$ C except at highs of 25.5 and 21.3 at 55 and 70 d, respectively.

3.5. Total organic carbon (TOC)

A measure of soluble organic carbon, TOC was initially (1 d) 6.3 mg L^{-1} in the inlet and 13.4 mg L^{-1} in the outlet (Fig. 6). The outlet TOC remained higher than the inlet concentration until 50 d. After this period, the inlet and outlet TOC concentrations were similar.

3.6. Solid phase analysis

Following completion of the trial, the solid column material was analysed for Cr(total) and Cr(VI) in order to provide further information on the fate of Cr(VI). Solid column material was sampled from two locations, corresponding to the bottom and top thirds of the column, respectively. In the bottom sample, Cr(total) was



Fig. 6. Total organic carbon (TOC) for inlet and outlet of the column at times 0–168 d.

 $7700~mg~kg^{-1}$ and Cr(VI) was 0.83 mg $kg^{-1},$ respectively. In the top sample, Cr(total) was 1400 mg kg^{-1} and Cr(VI) was 0.4 mg kg^{-1} .

4. Discussion

As outlined previously, the aims of this pilot trial experiment were (1) to assess the effectiveness of green/food waste derived compost for the treatment of hexavalent chromium contamination groundwater and (2) determine the longevity of the system without carbon and nutrient amendment other than from the compost or naturally present in the inlet water. Towards these aims, we found that an upflow fixed bed column consisting of PAS100 accredited compost was effective at removing Cr(VI) from groundwater. The Cr(VI) concentration in the outlet was below the detection limit of the colorimetric method (0.02 mg L^{-1}) during virtually the entire first phase of the experiment. In fact, the Cr(total) analysis showed that chromium in the outlet was as little as 0.009 mg L⁻¹ (method detection limit 0.001 mg L^{-1}) on two sampling points during the first phase. Chromium concentrations as low as these are comparable with those achieved previously by others using compost derived from green waste only [2].

In terms of longevity, after 7 d, our system achieved concentrations below 0.1 mg L^{-1} for at least 100 d. Likewise, Boni et al. [2] achieved less than 0.1 mg L^{-1} at port three (comparative hydraulic retention time of 5.16 h) for around 100 d using green waste derived compost. However, these authors supplied acetic acid and ammonium hydroxide from 28 d, representing additional carbon and nitrogen supply to their system. As compost from green/food waste contains a considerably higher amount of nitrogen then green only compost (Aspray, unpublished); this may have been an important factor in the longevity of our system. In terms of carbon, green/food waste and green only waste compost contain comparable amounts of organic carbon, therefore, the longevity of the system in the absence of additional carbon was less likely to be due to green and food waste compost properties. Instead, the contaminated inlet groundwater contained a significant amount of TOC (at times higher than the outlet) which may have helped to sustain the system. The source of the contaminated groundwater, an artificial mound ('bing') covered in woodland may have produced groundwater rich in organic carbon from tree root exudates. Although the TOC data presented suggests that organic carbon was not a limiting factor in later stages; changes in TOC composition from labile (compost) to comparatively non-labile (groundwater) carbon sources may be responsible. As this theory is currently only speculative, further research will be needed to characterise TOC composition in groundwater at this site. In terms of groundwater carbon sources, influence of weather and season on the concentration and composition of such carbon sources also requires investigation.

In order to enhance the longevity of the system in future work, we sought to understand the mechanism(s) responsible for Cr(VI) removal. The fact that no significant Cr(total) or Cr(VI) was detected in the outlet water during the majority of the trial suggested that the Cr(VI) was reduced to insoluble Cr(III). This was confirmed by analysis of the solid column material following completion of the trial which showed high concentrations of adsorbed Cr(total), with relatively little Cr(VI). By comparison, the original PAS100 compost material contained 8.6 mg kg⁻¹ Cr(total).

Microbial reduction was thought to play a major role in Cr(VI) removal over direct reduction by compost organic matter. We have found in other column experiments that reduction by compost organic matter alone is comparatively short-lived (Aspray, unpublished). In terms of microbial reduction mechanisms, it is likely that Cr(VI) was reduced indirectly in the column by metabolic end-products including Fe(II) and H₂S [22,23]. Certainly the analysis of H₂S in water samples (data not shown) during phase II sampling,

suggests the presence of sulphate reducing bacteria and the possibility for indirect Cr(VI) reduction by this metabolic end-product. ORP was recorded in the-200 mV range during 43-125 d for the outlet and top ports providing further support of this [24]. However, H₂S was thought not to be the only microbially mediated reduction mechanism. In the bottom port during the period 43–125 d and in the entire column prior to this period, the ORP was in the 0 to $-100 \,\mathrm{mV}$ range suggesting that other mechanisms including dissimilatory metal reduction were also responsible. Certainly, the reduced oxygen concentration in the column would have favoured solubility of metal ions such as Fe(II) and Mn(II) [25]. Characterisation of the microbial community present can help to confirm the mechanisms operating in the column [26]. Work is currently underway to characterise the bacterial and fungal community members' present, initial data confirms the presence of iron and sulphate reducing bacteria.

5. Conclusions

In conclusion, we have demonstrated that green/food waste compost represents a cost effective material for remediation of Cr(VI) contamination in low flow-through systems. The higher nutritional level of green/food waste compost over green only compost, particularly in terms of nitrogen, appears to have advantages in its application to this area. Further work is required to determine how closely compost based systems can compete with chemical reducing agents in higher flow treatment systems.

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