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CL:AIRE research bulletins describe specific, practical aspects of research which have direct application to the characterisation, monitoring or remediation of contaminated soil or groundwater. This research bulletin summarises the use of a novel technique using electrokinetics to remediate chromium contaminated soil.

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Electrokinetic Ferric Iron Remediation and Stabilisation (FIRS) of Hexavalent Chromium Contaminated Soils: An *Ex Situ* Field Scale Demonstration

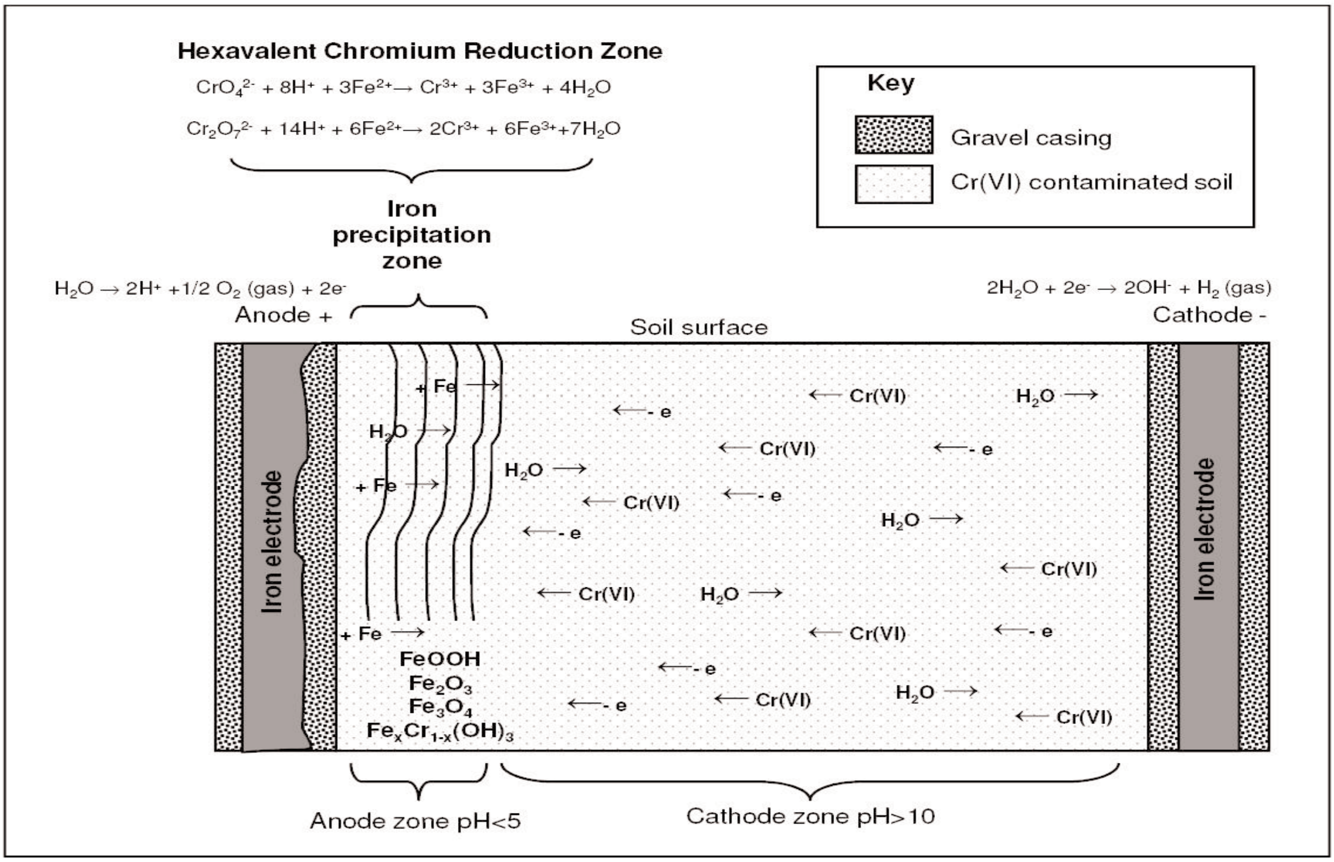

Fig. 1: Schematic diagram depicting the key processes associated with remediation and stabilisation of the Cr(VI) contaminated soil. Localisation of iron mineralisation close to the anode results from the alkaline nature of pore solutions within the contaminated soil.

1. INTRODUCTION

Hexavalent chromium [Cr(VI)] is one of the most toxic and common heavy metal contaminants (e.g., Reddy et al., 1997). A number of techniques have been employed to remediate Cr(VI) contaminated soils. These are comprehensively summarised in CL:AIRE technical bulletin TB14 (Bewley 2007). In essence, reduction of Cr(VI) to trivalent chromium [Cr(III)], which is of low toxicity and mobility is a critical step in the remediation of Cr(VI) contaminated sites (e.g., Reddy et al., 2003; Bewley 2007; Bini et al., 2008). The chemical reduction of Cr(VI) to Cr(III) can be stimulated by adding a variety of sulphur or iron based compounds, as well as various organic compounds, and carbon (e.g., Eary and Rai 1988; Su and Ludwig 2005; Bewley 2007). Many of these techniques employ a two-step process in which Cr(VI) is transformed to Cr(III) by a reducing agent, at an acidic pH, and then precipitated as an insoluble hydroxide at an alkaline pH (Mukhopadhyay et al., 2007). Further, many of the techniques necessitate physical mixing of the reducing agent with the contaminated soil, hence, are prone to be labour intensive. Electrokinetic treatment of contaminated soil, in which a low voltage direct current is passed through the soil may offer an alternative technique to treat Cr(VI) contaminated soils.

Electrokinetics has a proven effectiveness for *in situ* treatment of Cr(VI) contaminated groundwater (e.g., Mukhopadhyay et al., 2007), and potential effectiveness for soil (Bewley 2007). Conversely, many commercial electrokinetic systems are technically complex and energy intensive operating under very specific field or laboratory-based conditions (e.g., Acar and Alshawabkeh 1993; Virkutyte et al., 2002), which has tended to limit the widespread adoption of electrokinetics by industry. Further, up-scaling of electrokinetic systems from bench-top prototypes to industrial scale application on contaminated land in the real environment presents significant challenges.

2. FIRS SYSTEM

Research by staff at the University of Brighton has led to the development of a novel, low energy, natural-analogue-based, electrokinetic system called FIRS (Ferric Iron Remediation and Stabilisation). The FIRS system applies a low magnitude direct electric potential between two or more sacrificial iron-rich electrodes placed at opposing sides of a mass of contaminated soil or sediment. The electric potential generates a strong Eh-pH gradient between the two electrodes, promotes anodic dissolution, Fe^0 and $Fe^{2+}(aq)$ migration, and forces the precipitation of ferric iron oxyhydroxides, hematite, goethite, magnetite and zero valent iron, at near neutral pH values at the interface of the anodic and cathodic domains. The system uses approximately a tenth of the energy requirements of most conventional electrokinetic systems, and has been successfully applied at bench-top scales in a range of contaminated soils and sediments (Hopkinson and Cundy 2003; Cundy and Hopkinson 2005; Faulkner et al., 2005). The system is currently being developed for field-scale application to heavy-metal contaminated soils and wastes in the UK in partnership with Churngold Remediation Ltd (CRL).

3. EXPERIMENTAL RATIONALE

The reduction of Cr(VI) to Cr(III) by the delivery of iron (Fe^0, Fe^{2+}) is relatively well documented (Aoki and Munemori 1982; Rai et al., 1987; Reddy et al., 1997; Batchelor et al., 1998; Eary and Rai 1991; Haran et al., 1995; Powell et al., 1995; Pamukcu et al., 1997; Reddy et al., 2003):

$$CrO_4^{2-} + 8H^+ + 3Fe^{2+} \longrightarrow Cr^{3+} + 3Fe^{3+} + 4H_2O$$

$$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \longrightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

Accordingly, under an applied Direct Current (DC) electric field, stabilisation and remediation of Cr(VI) contaminated soils may be achieved where oxidative dissolution of iron-rich anodic electrodes provides $Fe^{2+}(aq)$ to react with Cr(VI) (Figure 1). The reduction of Cr(VI) by iron is known to result in the generation of relatively benign and stable $Fe_xCr_{1-x}(OH)_3$ solid phases (Batchelor et al., 1998). Bench-top trials of FIRS have been conducted on three varieties of Cr(VI) impacted soils (Hansen et al., 2007; Hopkinson et al., 2008), including chromium ore processing residue (COPR) impacted soil. The experiments were undertaken on 50 kg samples, conducted for 42 days, at potential differences ranging from 75 V(1.5 V/cm) to 150 V (3 V/cm), with opposite polarity electrode spacing of 500 mm. While the experiments showed variable success, primarily as a function of the different buffering capacities of the soils, in all three experiments Cr(VI) underwent a substantive decrease in concentration, ranging from 83.8 to 96.7% in cathodic sections of the experimental cells, and 54.0 to 84.4% in anodic sections of the cells. All cells exhibited a build up of $Fe_xCr_{1-x}(OH)_3$ mineral phases close to the anodes (Hopkinson et al., 2008). In this bulletin, progress is reported on the pilot scale trials of FIRS applied to five tonnes of Cr(VI) impacted soil, at a potential difference of 55 V (0.36 V/cm). All chemical analyses reported here were conducted at a UKAS and MCERTS accredited laboratory.

4. STARTING MATERIAL

A five tonne composite soil sample was transported to the CRL laboratory in Avonmouth from a contaminated site in the north of Britain. The soil was dark brown coloured sandy clay loam (0.1 mm to 2 mm grain size), with frequent small pebbles. Site investigation by CRL indicated that the sampled soil was fairly representative of the site as a whole, with chromium contamination restricted to the first 1.1 m depth below surface (Table 1). Scanning electron microscopy (SEM) observations suggest that the soil has a low calcium carbonate concentration. No specific chromium mineral phase(s) were identified.

Analysis of sampled soil on which the pilot trial was conducted showed pH 10.6. Total chromium concentration Cr(total) was measured at 4700 mg/kg, while Cr(VI) was measured at 710 mg/kg. The measurements are in keeping with those taken during construction of the trial pit from which the sample was taken (Table 1). A conservative estimate of the contaminant load within the five tonne sample is taken as the mean of the readings taken at 1.1 m depth below surface: Cr(total) 3933 mg/kg, Cr(VI) 734 mg/kg (Table 1). At the onset of the experiment pore fluids contained: Cr(total) 290 mg/l, Cr(VI) 280 mg/l.

5. EXPERIMENTAL DESIGN

An industrial skip was converted to a bespoke Treatment Container (TC). This involved insulating the skip with Thixotropic Polyurethane Elastomer, engineering a plumbing system to cater for electro-osmotic flow of pore waters within the soil mass, installation of a pH neutralisation system within holding tanks, with a feedback system to provide controlled supply of neutralised solutions to the anodic domain (Figure 2). The circulation system served three main purposes, namely: it promoted closed system recycling of aqueous solutions, thereby allowing continued electrokinetic treatment of the soil; it facilitated collection of experimentally induced fine grained precipitates, thereby lowering the overall contaminant level within the TC, and permitted water purification. Details on the design of the plumbing system, associated irrigation pipes, etc is commercially sensitive. The electrode array was comprised of two lines of five anodes and five cathodes (each electrode is 1.5 m in length, fabricated from cast iron) connected to a

Table 1: Total chromium, [Cr(total)], and hexavalent chromium [(Cr(VI)] data for the sampled soil and pit from which it was excavated. Note that the soil used in the experiment was acquired from depths ≤1.1 metres below surface level (mbsl).

Sample	Cr(total) mg/kg	Cr(VI) mg/kg
Trial pit 0.3 mbsl	2900	580
Trial pit 1.1 mbsl	4200	920
Experiment sample	4700	710
Mean	3933	734
Trial pit 1.3 mbsl	130	<6

power-pack. The electrodes were implanted in gravel casings (Figure 1) at each end of the contaminated soil mass, to facilitate penetration of water throughout the ~1.5 m depth of the soil mass. The FIRS trial was started on the 29th May 2008 and ran for 42 days.

Samples from the trial were collected from the TC after 14, 28 and 42 days. Interim samples consisted of three composite samples from the anode, three composite samples from the cathode and one composite sample from the middle of the treated soil. The selected sampling intervals were informed by bench-top results (Hopkinson et al., 2009), which showed that the highly alkaline nature of pore solutions in this soil type resulted in the localisation of iron mineralisation close to the anodic array (Figure 1). Pore fluid samples were collected on a weekly basis. Samples were taken from approximately 10 to 25 cm below the soil surface. At the end of the 6 weeks, composite samples were collected 10, 35, 60, 85, 110 and 135 cm from the anode. All composite samples were also analysed for leachability. These samples were taken with a hand auger and were taken at a depth of approximately 75 cm below the surface.

Fig. 2: The Treatment Container (TC) fabricated for the experiment. The connected white tower structure behind the TC holds electro-osmotically mobilized aqueous solutions from which 5 L samples were taken for purification prior to de-alkalisation.

6. EXPERIMENTAL RESULTS

6.1 Soil Remediation and Stabilisation

Within 14 days of activating the TC, hexavalent chromium underwent a relatively uniform ~40% reduction in concentration across the TC, prior to the emergence of a strong Eh-pH gradient, indicating the high mobility of a significant fraction of the contaminant within this particular soil, and its consequent liberation into the aqueous phase (Figure 3). With emergence of a strong Eh-pH gradient, after 28 days Cr(VI) underwent a 92% decrease in concentration in the cathode zone. The high pH and reducing conditions resulting from the electrolysis reaction at the cathode favours Cr(VI) solubility and electro-migration towards the anode (Reddy et al., 2003). The progressive decrease in concentration of Cr(VI) in the anode zone between 28 and 42 days represents a reduction in contaminant levels rising from 64 to 85%, and is consistent with the anodic compartment operating as an increasingly efficient zone of Cr(VI) reduction with increasing experimental duration (Figure 3). The comparatively high levels of Cr(VI) within the centre of the TC (220 mg/kg) at cessation of the experiment, relative to the cathodic and anodic domains (57 mg/kg and 110 mg/kg respectively) presumably indicates that the experiment was terminated during ongoing anodic bound electro-migration of chromate and dichromate ions. Changes in Cr(total) concentration with experimental duration (Figure 4) reflect widespread liberation of Cr(VI) to the aqueous phase, and mobilisation of the contaminants to the anodic compartment, and the holding tank.

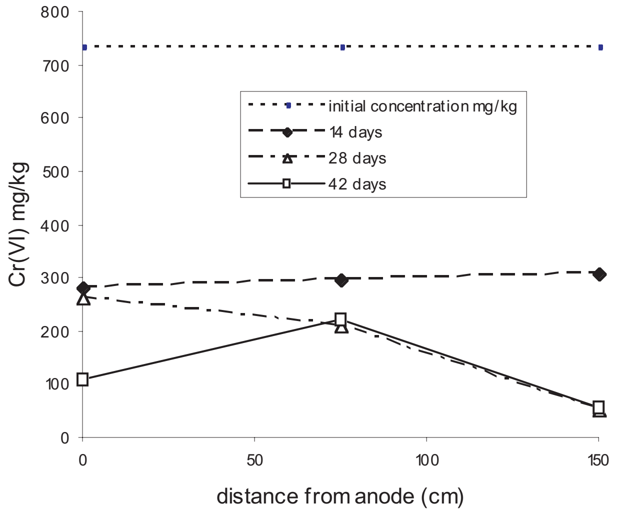

Fig. 3: Composite analyses of Cr(VI) mg/kg content of soil after 14, 28 and 42 days, plotted relative to distance from the anodes.

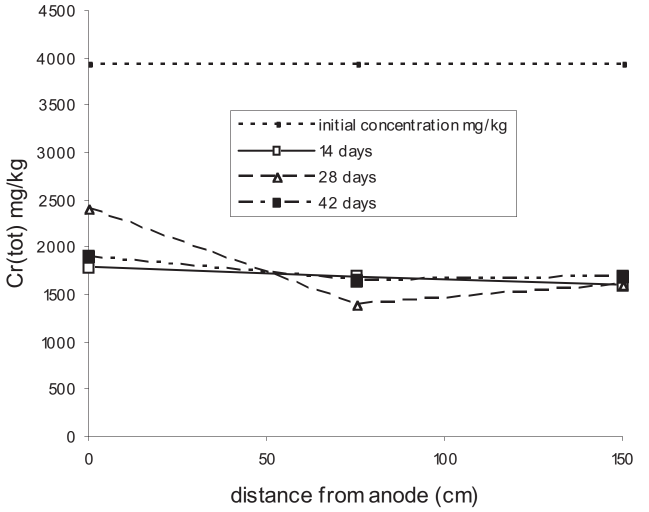

Fig. 4: Composite analyses of Cr(total) mg/kg content of soil after 14, 28 and 42 days, plotted relative to distance from the anodes.

6.2 Soil Pore Water Chemistry

Electro-osmotically purged soil pore waters were sampled periodically during the experiment from a holding tank, prior to de-alkalinisation and gravity fed redistribution to the anodic compartment. A pronounced decrease in pore water Cr(total) and Cr(VI) concentration is evident with increasing experimental duration (Figure 5). The decrease in Cr(VI) from 280 mg/l at the onset of the experiment to 69 mg/l at its termination represents a 75% reduction in the level of Cr(VI) contamination. Figure 6 shows chromium leachate profile at the onset, and termination, of the pilot trial. The data shows that the leachability of chromium in the TC has been reduced by an average of ~94%, providing a strong indication that the FIRS pilot trial was successful and that the highly mobile Cr(VI) has been substantially reduced to Cr(III).

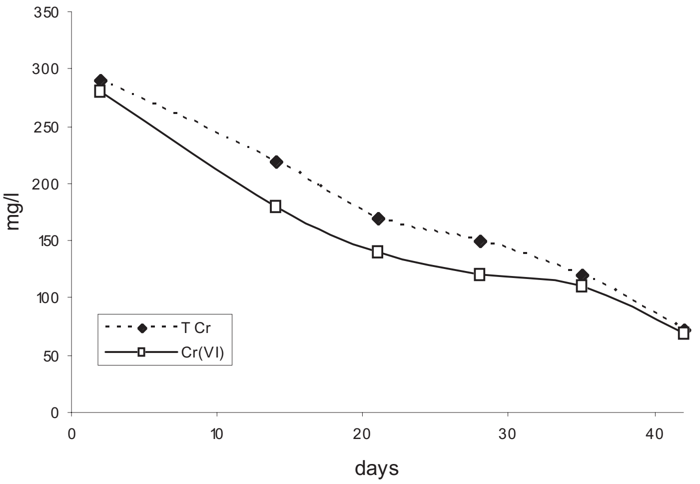

Fig. 5: Evolution of pore water Cr(total) and Cr(VI) mg/L concentrations with increasing experimental duration.

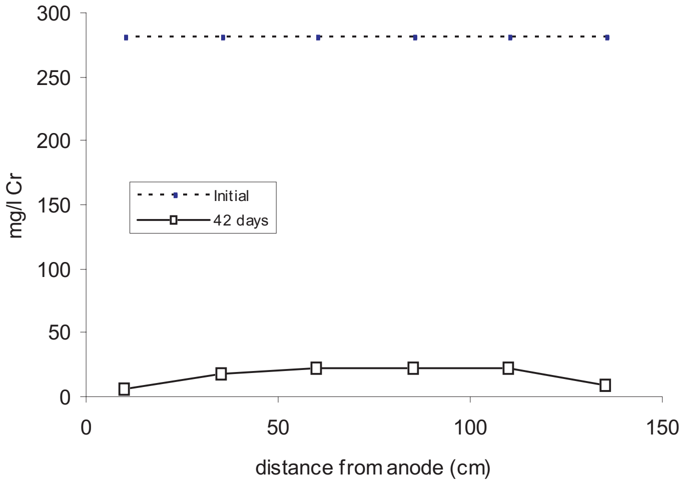

Fig. 6: Chromium leachate data (mg/L) analysed at commencement and cessation of the TC pilot test.

6.3 Electrokinetic Water Purification

To examine the potential for further enhancing, the capabilities of the TC 5 L batches of aqueous solutions were periodically removed and subjected to 50 V potential difference applied between two cast iron plates. Post treatment the 5 L batches were returned to the holding tank after filtering of iron-rich precipitates. When subject to electrokinetic treatment the yellow pigmented waters rapidly clear, and fine grained iron rich material collects in the base of the container (Figure 7). With water clearing comes a drastic reduction in the Cr(VI) concentration. The process evidently goes to completion in less than 24 hours, and, in the process yields water which surpasses the European Union's 0.05 mg/L maximum contaminant level ceiling on Cr(VI) in drinking water (Table 2). SEM analysis of the iron-rich oxide precipitates from the holding tank indicates that the precipitates contain accessory quantities of chromium. Hence, development of an electrokinetic holding cell within the TC plumbing circuit facilitated *in situ* extraction of stabilised chromium waste from impacted soils, thereby resulting in an overall reduction in Cr(total) in the soil as well as yielding clean water.

Fig. 7: Sub-samples of a 5 L sample of Cr(VI) contaminated water, pre (left) and post (right) electrokinetic water purification. The red pigmented sediment at the bottom of the vial contains iron-rich precipitates. The change in colour from left to right is marked by a reduction in Cr(VI) concentration in the aqueous solution to ≤ 0.03 mg/L, i.e. analytical detection limits, i.e. safely within the European Union's 0.05 mg/L maximum contaminant level of Cr(VI) in drinking water.

7. DISCUSSION

The feasibility trial achieved significant reduction of hexavalent chromium in the target soils. FIRS performed at least as well as the bench scale studies documented in Hopkinson et al., (2008). Results show that significant amounts of hexavalent chromium were mobilised into the aqueous phase, leading to a significant overall reduction in contaminant loading in the soil. Once liberated from the soil matrices and dissolved in water, anodic dissolution resulted in the removal of over 99% of hexavalent chromium in ~24 hours. Hence, in the context of Cr(VI) impacted soils, FIRS represents a potentially viable mechanism to simultaneously stabilise and remediate Cr(VI) contaminated soils, in the absence of a need to physically ensure total homogenisation of additives, as is the case with competing technologies such as Calcium Polysulphide (CaS_5), which also require precise calculation of additive requirement. Moreover, given that the applied voltage in the TC could have been increased significantly without leading to excessive soil heating, it follows that electrokinetic treatment times may potentially be significantly reduced, or adjusted to cater for soil specific buffering capacities.

Experimental results have shown that mixed Cr(III) – iron hydroxide complexes result from the FIRS process. Consideration of iron and chromium speciation in Eh-pH space indicates that the $Fe_xCr_{1-x}(OH)_3$ solid phases are likely to be stable under neutral and alkaline, and moderately to strongly oxidising conditions. Further, any scenario

Table 2: Cr(VI) concentrations in water, pre-experiment Cr(VI) concentration is analysis TC start. 12hr, 24hr, etc refers to the length in hours of treatment by iron electrodes. EK stands for electrokinetics. The rapid decrease in Cr(VI) concentration with treatment is clearly evident.

Sample Identity	TC start	12 hr EK	24 hr EK	25 hr EK	48 hr EK	48 hr EK	48 hr EK	Limits of Detection / Units
Iron dissolved (ICPMS)	-	180	-	-	-	-	-	<50 µg/L
Chromium Total (HNO_3 Digest)	210000	1600	76	-	23	2	2	<1 µg/L
Hexavalant Chromium	170	0.47	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03 µg/L

involving reductive dissolution of the mineral phases under acidic conditions means that $Fe^{2+}(aq)$ is available to react with Cr(VI), or else, chromium is already present as Cr(III). Hence, stabilised chromium left *in situ* will most likely show low mobility in the natural environment. Furthermore, electrokinetically remediated iron mineralisation is, in essence, a self limiting process, in which an essentially impermeable ($1x10^{-7}$ m/s or less) iron-rich lithified soil mass develops with time (e.g., Cundy and Hopkinson 2005). The generation of low permeability zones, in which chemically stable Cr(III) is locked in iron-rich mineral phase(s), means that stabilised zones are also likely to be resilient to subsequent reactive flow (i.e. following disposal of stabilised soil wastes), the low permeability also serving to limit potential loss of any Cr(VI) residue adsorbed onto the iron mineral phase(s).

When $Fe_xCr_{1-x}(OH)_3$ is produced solely from the stoichiometric reaction between Fe(II), chromate and water, the value of x is 0.75, and so the ratio of Fe(III) to Cr(III) in the precipitated hydroxide is 3:1 (Batchelor et al., 1998):

$$4H_2O + CrO_4^{2-} + 3Fe^{2+} + 4OH^- \longrightarrow 4Fe_{0.75}Cr_{0.25}(OH)_3\ (s)$$

However, in natural environments soil specific characteristics means that fractions of $Fe^{2+}(aq)$, liberated by anode dissolution, will be rendered unavailable to react with Cr(VI) due to interactions with other solid phases (e.g., Batchelor et al., 1998). This means that site specific models will be required to predict the mass of iron required to stabilise and treat chromium under *in situ* conditions (e.g., Batchelor et al., 1998).

8. INDUSTRIAL PERSPECTIVE

The scale-up to a working full-scale system has been considered in view of the findings from the pilot study. One of the most significant findings of the pilot trial was the treatment of highly impacted pore water extracted from the test soil. This process is very cost effective and means the full-scale system can be completely self-contained, generating a nominal amount of waste. The results of the study have been used to evaluate FIRS application at site scale e.g. treating soil at volumes greater than 500 m^3. The 'scale-up' of the system will be relatively linear for some components whereas economies of scale need to be applied to others. At 'full-scale' the greatest capital expenditure will be the power packs needed to supply the DC current, the provision and installation of electrodes (which are sacrificial) and the associated control system e.g. leachate capture and re-circulation.

Once constructed, a FIRS treatment cell would incur very low running costs, which means the process could continue, if there are no issues with timing, until treatment goals have been met. The largest operational costs would be associated with engineers/scientists operating the system and the disposal of collected leachate/ precipitated iron-chrome sludge. The *in situ* application of FIRS would be the most cost-effective means of using the technology as excavation and *ex situ* processing costs can be avoided. Although FIRS offers inherent hydraulic control, additional infrastructure may be required in very sensitive site settings to ensure components mobilised within the treatment zone are contained.

Although each site needs to be evaluated on its own merits the apparent robustness of FIRS specifically for the treatment of Cr(VI) means its application could suit a wide range of soil types. Based on this assumption, Churngold Remediation Limited, using experience to factor in the scale-up, estimate 'turnkey' cost for applying FIRS to Cr(VI) impacted soil ranges between £35 and £55/tonne. This cost assumes all soil requiring treatment is excavated and placed in *ex situ* treatment cells, that treated soil is retained on site and a minimum treatment volume of 500 m^3. It should be noted that the FIRS treatment can also potentially be applied *in situ* if conditions allow, reducing the treatment cost.

9. CONCLUSIONS

Overall, experimental results suggest that electrokinetically-mediated iron supply and mineralisation, combined with purposeful neglect to control soil alkalisation provides a potentially viable mechanism to stabilise (and potentially simultaneously remediate) Cr(VI) contaminated soils over short durations of time, at industrial scales. Hence, FIRS appears to represent a comparatively simple, low energy, low cost, and low maintenance approach to abate environmental risk from Cr(VI). Treatment time and applied voltage requirements will vary as a function of buffering capacity of the specific soil, and level of contaminant loading. Available evidence suggests that this approach may be extended successfully to treat Cr(VI) impacted soils *in situ*.

As FIRS is one of few solutions available for the treatment of Cr(VI) contaminated groundwater and soil *in situ* it has unique market potential. Adding to this is the additional benefit of the system's relatively passive treatment of contamination once set-up, which can continue at relative low cost and impact to site operations until targets have been met.

Competing Cr(VI) treatment options typically require the mixture of additives, potentially over an number of rounds of application before treatment goals can be met. Therefore active sites or sites where there are space limitations could benefit the most from the use of FIRS. Specific areas of industry where FIRS could be applicable include Metal Manufacturing/Refining Sites, Residential/Industrial areas historically developed on top of mining residuals and Active/Redundant Mining Sites. Further, given that reactions involving iron play a major role in the environmental cycling of a wide range of important organic, inorganic, and radioactive contaminants (e.g., Cundy et al., 2008), it is suggested that electrokinetic driven iron mineralisation may find application in a range of areas.

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Contact Details:

Laurence Hopkinson (corresponding author), Andrew Cundy
School of Environment and Technology, University of Brighton
Moulsecoomb
Brighton, BN2 4GJ
Telephone: 0127 364 2239
Fax: 0127 364 2285
Email: l.hopkinson@brighton.ac.uk, a.cundy@brighton.ac.uk

Ross Pollock
Churngold Remediation Limited
St. Andrews House, St. Andrews Road
Avonmouth, Bristol
BS11 9DQ
Telephone: 0117 916 0510
Fax: 0117 916 0511
Email: ross.pollock@churngold.com