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RESEARCH ARTICLE

Effectiveness of a constructed wetland for treating alkaline bauxite residue leachate: a 1-year field study

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Abstract Increasing volumes of bauxite residues and their associated leachates represent a significant environmental challenge to the alumina industry. Constructed wetlands have been proposed as a potential approach for leachate treatment, but there is limited data on field-scale applications. The research presented here provides preliminary evaluation of a purpose-built constructed wetland to buffer leachate from a bauxite residue disposal site in Ireland. Data collected over a 1-year period demonstrated that the pH of bauxite residue leachates could be effectively reduced from ca. pH 10.3 to 8.1 but was influenced by influent variability and temporal changes. The wetland was also effective in decreasing elemental loading, and sequential extractions suggested that the bulk of the sediment-bound metal inventory was in hard-to-leach phases. Elemental analysis of *Phragmites australis* showed that although vegetation displayed seasonal variation, no trace elements were at concentrations of concern.

Keywords Constructed wetland · Alkaline bauxite residue leachate · *Phragmites australis*

Introduction

The alumina industry produces some 150 MT of bauxite residue each year with an estimated global stockpile of 3 billion tonnes (Evans 2016). Bauxite residue and associated leachate is inherently alkaline (pH 9–13), with many refineries now pursuing applications to reduce residue pH (e.g. seawater neutralisation, gypsum amendment, carbonation) (Menzies et al. 2004; Courtney and Kirwan 2012; Courtney et al. 2013; Evans 2016). The current best practice in the industry is disposal in engineered bauxite residue disposal areas (BRDAs), increasingly using dry stacking methods and management steps such as mud farming to aid dewatering of the mud in order to compact and consolidate the residue, and treatment of any leachates that are produced (Evans 2016; Gomes et al. 2016; Zhu et al. 2016).

In some instances, where industry best practice is not achieved, there is potential for leakage of residue leachate to the surrounding environment (Wang et al. 2015; Kong et al. 2017) and requirements for the management and treatment of such drainage waters may persist for many decades following closure (Hua et al. 2015; Olszewska et al. 2016). Neutralisation of residue leachate is an important step in reducing the overall burden of alkalinity and soluble trace elements on the aqueous environment (Burke et al. 2013; Wang et al. 2015; Kong et al. 2017). Discharge of high pH residue leachate to Kinghorn Loch, Scotland, resulted in As contamination of lake sediment and long-term cycling of As in macrophytes (Olszewska et al. 2016).

For discharge to receiving environment, waters must be ≤pH 9 and treatment of alkaline drainage waters by conventional methods, such as chemical neutralisation, is likely to be expensive, especially if it is to be continued for many decades after closure. Passive treatment systems such as constructed wetlands have been used extensively over the past 30 years to treat mine and circum-neutral discharges (Batty and Younger

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2004) with recent evidence of the potential for alkaline leachates (Banks et al. 2006; Mayes et al. 2006). More recently, their use was suggested by Burke et al. (2013) as a possible polishing treatment for residue leachate prior to discharge.

With $Ca(OH)_2$-rich alkaline steel slag leachates, Ca^{2+} (the dominant cation) is precipitated to calcite crusts during wetland treatment due to high partial pressure of CO_2 (Mayes et al. 2009). For bauxite residue, leachate Na^+ dominates and removal efficiency through constructed wetlands is unknown but thought to be problematic as most Na compounds are highly soluble (Hua et al. 2015). While no field data exists on the potential for constructed wetlands to treat residue leachate, the mechanisms conferred by constructed wetlands have been assessed for their potential to treat bauxite residue/NaOH alkalinity and trace element removal (Hua et al. 2015; Buckley et al. 2016). Decreases in alkalinity were associated with soil characteristics, carbonate formation and diluting water quality, with removal of target ions effective at circum-neutral pH (Hua et al. 2015; Buckley et al. 2016).

For constructed wetland technology to be considered as a potential mechanism for treating bauxite residue-derived leachates, the field performance of such a system needs to be validated. The purpose of this study was to evaluate influent and effluent water qualities of a constructed wetland in order to assess treatment performance on bauxite residue leachate representative of a closed BRDA. Residual risk associated with metal(loid) uptake by biota was also assessed. Efficiency of the system to buffer alkalinity, trace element loading and possible mechanisms for alkalinity removal are presented.

Methods

Study site

A pilot-scale constructed wetland was constructed on the bauxite residue disposal area at Aughinish, Southwest Ireland (52° 37′ 06″ N, 9° 04′ 19″ W). This comprised of a horizontal surface flow constructed wetland cell of 44 m^2 (4 × 11 m) and a programmable logic controller (PLC) leachate mixing system. Local soil was excavated, mixed and placed in the lined cell system to a depth of 200 mm and representative samples characterised (Supplementary Information; Table 1). Wetland vegetation (*Phragmites australis*, *Typha latifolia* and *Sparganium erectum*) were supplied by a local wetland nursery (FH Wetland Systems Ltd.) and planted (February) and given an acclimatisation period of approximately 6 months during which the cell received fresh water only.

The PLC mixing system was constructed for the preparation of a diluted leachate solution that could then be fed to the constructed wetland at pre-set pH and flow rate. The leachate was diluted to represent a residue leachate and surface runoff mix that could manifest from a closed BRDA (Residue solutions 2007; Buckley et al. 2016). The mixing system was composed of three 1000-L tanks: a leachate tank, a mixing tank and a dosing tank. Leachate from the leachate tank was pumped into an adjacent mixing tank along with water. The mixing tank continuously added more water and leachate until the target pH value of the solution was met. Once the target pH has been reached, the diluted leachate was then pumped into the dosing tank where it is ready to be discharged to the constructed wetland. The flow rate of the residue leachate through the wetland was controlled by setting a flow rate valve on the mixing unit and by the valves on the outflow pipes from the wetland (Fig. 1). Tracer testing conducted during summer 2014 indicated residence time of 4–6 days.

Following the acclimatisation period, bauxite residue leachate discharge to the wetland commenced in August 2013 and the wetland subsequently received residue leachate for 1 year (August 2013–August 2014).

For sampling purpose, the wetland was divided into a front section, middle section and bottom section, with each monitoring section approx. 3 m in length. Sample pH and electrical conductivity (EC) were determined daily by field probe (monthly averages reported). Influent and wetland-treated effluent samples were taken on a monthly basis for elemental analysis and filtered (with 0.45-μm cellulose nitrate filters) prior to analysis. Al, As, Ca, Cd, Cr, Mg, Na and V content was determined by inductively coupled plasma (ICP-MS).

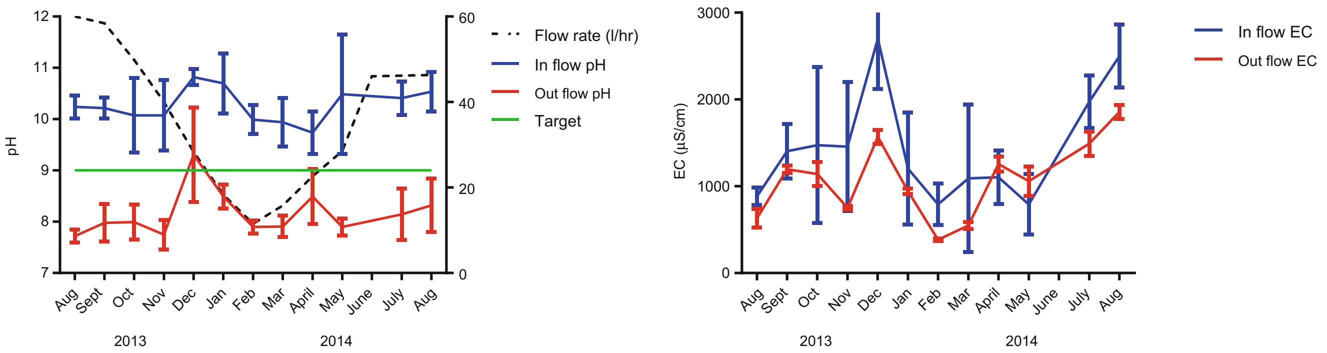

Fig. 1 Monthly average pH, flow rate and EC values of wetland inflow and outflow—August 2013 to August 2014

Vegetation and soil samples were taken on a seasonal basis. Aerial portions of the vegetation were harvested, rinsed thoroughly with DI water, oven dried, acid digested in HNO_3 and elemental content determined by ICP-MS. Soil samples (0–10 cm) were taken prior to leachate dosing commenced (baseline) and seasonally thereafter. Samples were air-dried and sieved (<2 mm) prior to analysis. Soil pH and EC were determined on 1:5 soil/solution. Elemental content was determined by sequential extraction using a modified Tessier procedure as shown in Table 1 (Tessier et al. 1979). The reported values for the different fractions are operationally defined, and while there is the possibility of some of the metal(loid)s investigated changing chemical states, of repartitioning during the extraction stages and being otherwise transformed physically as well as chemically, a similar fractionation procedure was used by Mayes et al. (2011) investigating river sediments contaminated with bauxite residue.

Results

Leachate feed (inflow) monthly mean pH varied between 9.7 and 11.1 (mean 10.3) with treated leachate (outflow) ranging from 7.7 to 9.3 with a mean of 8.1 (Fig. 1). Electrical conductivity showed high variability with an inflow range of 790–2698 µS cm^{-1} (mean 1447) with outflow 383–1856 µS cm^{-1} (mean 1069). The dominant soluble cation in the leachate inflow was Na^+ with much lower values for Ca^{2+} and Mg^{2+} (Fig. 2), which is typical of bauxite residue leachates. The maximum flow rate through the system was 60 L h^{-1} which corresponded with the summer/late summer period. Substantially lower flow rates (~10 L h^{-1}) were required during the winter period to achieve an outflow pH of <9, and flow rates steadily increased for spring/summer months.

Neat bauxite residue leachate exhibited elevated levels of elements Al, As, Cr and V (Supplementary Information Table 2). Diluted leachate feed (representing the expected closure scenario) produced lower values for the selected trace elements. As with pH and EC, there was considerable variation in concentrations for these parameters in wetland input (Fig. 2). This was attributed to the variation in bauxite residue leachate source and feed pH from the PLC system. Despite the variability in leachate inflow, the constructed wetland (CW) outflow produced consistently lower values for the target parameters. Reductions in trace element (mean values) were Al 97%, As 84% and V 86%. Reductions in Cr content went from 12.6 µg L^{-1} to below the detection limit. Changes in the major cation contents were much less: Ca (53%), Mg (44%) and Na (31%).

Wetland soil pH, EC and elemental content varied significantly both temporally and spatially during the 1-year monitoring period. Increases in soil pH and EC were most evident in the front and middle sections of the wetland with increases from the baseline pH 6.7 to pH 7.8 for winter sampling and reaching pH 8.6 by the autumn sampling period (Fig. 3). Changes in soil EC were increased throughout the wetland and were apparent in all sections. By the summer sampling period, EC in the front end of the wetland had increased 6-fold.

Results of sequential extractions are shown in Fig. 4. Significant increases in all sodium soil fractions were observed over time with particular loading to front and middle sections. Water-soluble (F1) Na accounted for 9% in baseline soil with front and middle parts of the wetland increased to ~20% soluble by winter sampling and 40–52% by spring and summer sampling. In the same time period, the ammonium acetate-carbonate (F3) Na increased from non-detectable levels in baseline soil to 1.8–4% in front and middle sections with the lower end increased to ~0.5%. Significant increases were observed for Ca content in the front and middle sections of the wetland. Increases were particularly evident for ammonium acetate-carbonate (F3) and oxide-bound (F4) Ca. While the total amount of Mg did not vary over time, the relative proportion of Mg that was bound to oxides increased for the front and middle sections of the wetland.

Changes in water-soluble Al fractions in the soil were evident by the end of the sampling period with increases for front

Table 1 Modified sequential extraction procedure of wetland soil samples

Fraction	Reagent	Soil/solution ratio	Condition
Soluble (F1)	Deionised water	1:20	Shake 1 h, room temperature
Exchangeable (F2)	1 M $NH_4C_2H_3O_2$	1:8	Shake 1 h, room temperature
Carbonate (F3)	1 M $NH_4C_2H_3O_2$ adjusted to pH 5 with CH_3COOH	1:8	Shake 5 h, room temperature
Oxide (F4)	0.04 M $HONH_2{\cdot}HCl$ in 25% (*v/v*) CH_3COOH	1:20	Occasional agitation 6 h, 96 ± 3 °C
Organic (F5)	0.02 M HNO_3 + 30% H_2O_2 (pH 2 with HNO_3)	1:8	Occasional agitation 2 h, 85 ± 3 °C
	30% H_2O_2 (pH 2 with HNO_3)	1:3	Occasional agitation 3 h, 85 ± 3 °C
	3.2 M ($NH_4C_2H_3O_2$) in 20% (*v/v*) HNO_3	1:5	Shake 30 min, room temperature
Residual (F6)	Acid digest		

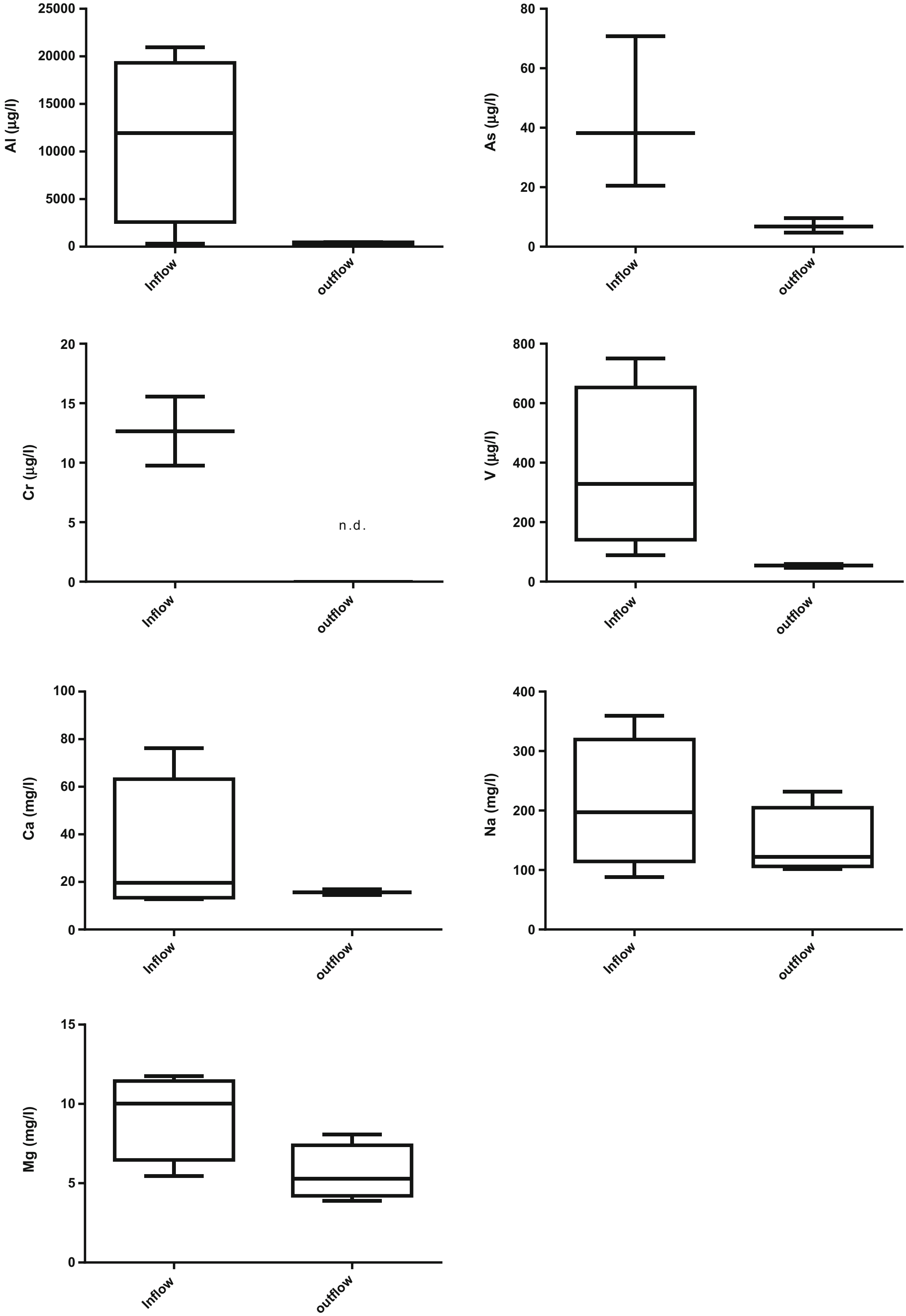

Fig. 2 Distribution of the inflow and outflow (*box plot*) concentrations for trace elements (filtered) in wetland

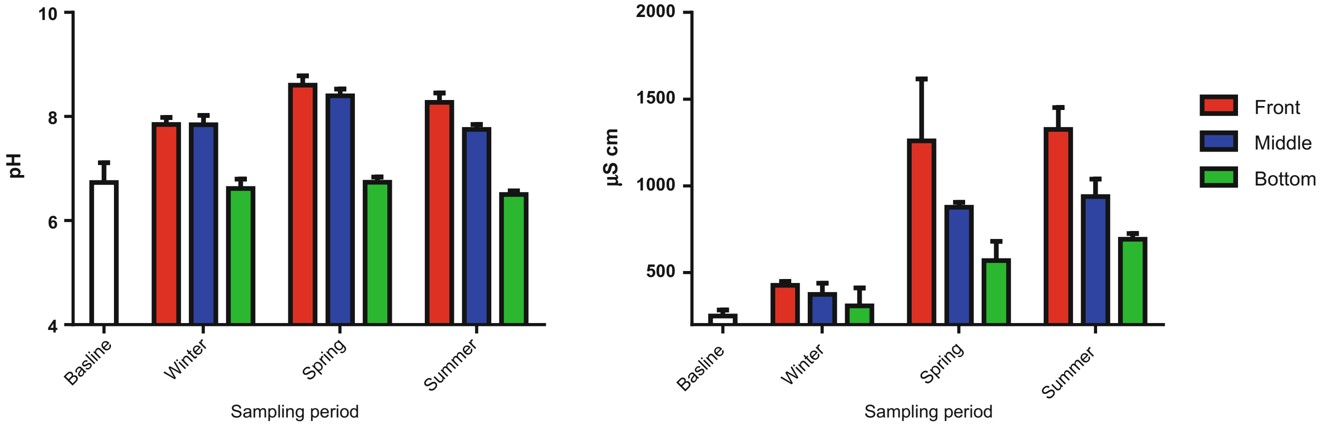

Fig. 3 Soil pH and EC in wetland soils for baseline and spatial/temporal changes

and middle section. The percent of Al that was mobilised by nitric acid extraction (F5) also increased over time for all sections of wetland. The relative proportion increased from 2% for baseline soil to 5–9% for wetland soils. Water-soluble As increased 4-fold from baseline values to winter sampling period and 10-fold by the end of sampling period. Oxide-bound As increased from 3 to 8% by the end of sampling. A similar pattern was observed for V with increases in the soluble content. Oxide and organic-bound fractions also increased. Ammonium acetate-carbonate-bound V only increased for the front section of wetland (from nd to 0.4%). The Cr content in fractions F2–F4 did not alter in the soil samples for the sampling period. There were increases in both soluble and organic fraction.

Baseline values for trace elements and nutrient content in aerial portions of *P. australis* were taken during the summer months prior to leachate dosing commenced. Winter values were not taken for As, Cr and V, and Al values had increased 3-fold (Fig. 5). Summer sampling values had decreased to baseline values and were the lowest recorded for the monitoring periods. Values increased again for all parameters by autumn with a pattern of higher values for As and Cr in the top end of the wetland. Conversely, V values were slightly higher at the bottom section of the wetland.

The sodium content was below the limit of detection for baseline sampling and had increased to detectable levels by the end of the following summer. By the autumn sampling period, the front section of the wetland exhibited the highest Na content in *P. australis*.

Nutrient content was also determined for vegetation (Supplementary information) with seasonal variation observed for all nutrients.

Discussion

Evidence of CWs treating alkaline leachates has received significantly less attention than for acidic waters (Mayes et al. 2009). Batch trials had previously demonstrated the potential for CW mechanisms to reduce the pH of NaOH solutions (Buckley et al. 2016) and trace element removal (Hua et al. 2015). The primary objective of the current trial was to test the ability of CW to treat bauxite residue leachate representative of a closed BRDA under field conditions. Results show that CW can effectively reduce alkaline bauxite residue leachate to a pH suitable for discharge (pH ≤ 9). Variation in pH at the CW outlet is likely due to a combination of variability in leachate feed pH (Fig. 1) and the seasonal effect of the wetland. In a study treating alkaline steel slag leachate, Mayes et al. (2006) attributed improved wetland performance over a summer period to improved microbial performance and growth of vegetation.

Removal of Ca and Na from leachate loading to the CW was evident through their increases in soluble, exchangeable and carbonate fractions within the wetland soils. The proposed mechanism for CW in the removal of alkalinity in steel slag leachates is the formation of calcium carbonate (Eq. 1) (Mayes et al. 2008).

$$Ca^{2+} + CO_2 \leftrightarrow CaCO_3 \tag{1}$$

The accumulated amounts of carbonate-bound Ca and Na in wetland soils illustrate the findings of Mayes et al. (2006) in the role of carbonate in removing alkalinity. Buckley et al. (2016) reported formation of calcite in Ca-loaded NaOH solutions following carbonation, and it was observed that calcite acted as a site for nucleation for sodium carbonate. Similarly, sites for nucleation within wetland systems include the soils, plant and organic debris. Precipitation of sodium carbonates (e.g. trona $NaHCO_3 \cdot Na_2CO_3 \cdot 2H_2O$) has been predicted for several alkaline natural waters (pH 10.13–10.19), and trona has been observed precipitating from high alkalinity natural waters in the pH range from 9.8 to 10.2 (Marion 2001).

Trace element loading to the wetland was higher than those reported by Mayes et al. (2008) for steel slag-derived alkaline leachates but is lower than values reported for the bauxite

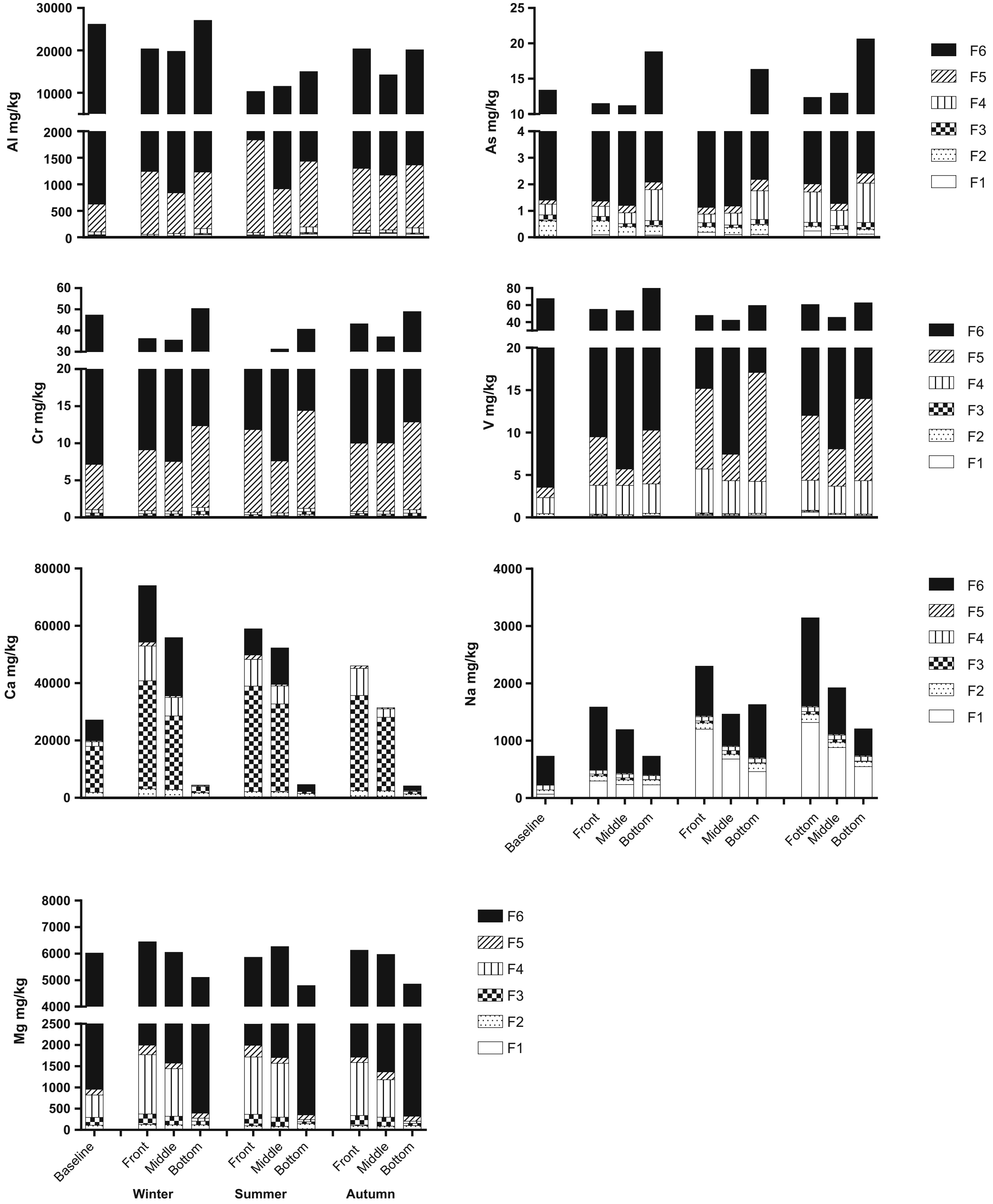

Fig. 4 Sequential extraction of selected major and trace elements in wetland soil samples with time

residue (red mud) event in the Torna creek at Ajka, Hungary (Burke et al. 2012). Hua et al. (2015) proposed that for bauxite residue-derived leachates, a pH of 6.0–7.0 is required for effective removal of trace elements. Removal of trace elements

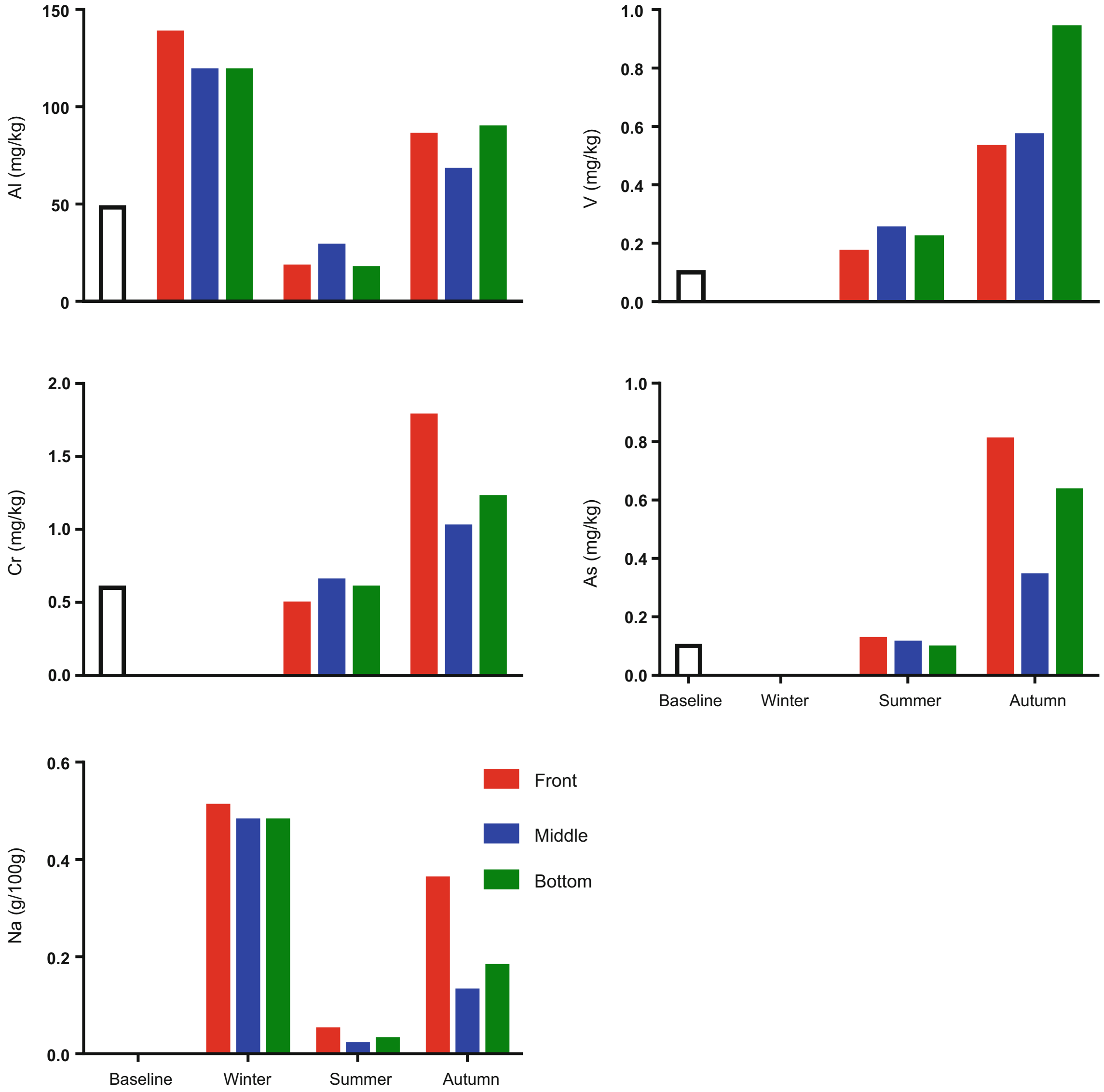

Fig. 5 Trace element and Na content in *Phragmites australis* aboveground biomass

in neutralised bauxite residue leachate has met with varied success. Hua et al. (2015) suggested that pH of below 8 is required to cause Al precipitation as it is present predominantly as Al(OH)$_4$ in a pH range of 8.0–8.6. Conversely, Burke et al. (2012) reported 99.7% removal efficiency of Al in neutralised residue leachate (pH 8.3).

High Al removal in horizontal flow CWs was reported (Vymazal and Krása 2003; Lesage et al. 2007; Kröpfelová et al. 2009) and is attributed to the formation of insoluble compounds through hydrolysis and/or oxidation which leads to the formation of a variety of oxides, oxyhydroxides and hydroxides (Kröpfelová et al. 2009). Monitoring of three constructed wetlands by Vymazal and Krása (2003) reported a decrease in Al concentration from 45 to 91.1% and Kröpfelova et al. (2009) from 80 to 91% removal. Similarly, high removal rates in the current study were achieved for much higher Al concentrations. While there were increases in the carbonate and oxide-bound Al, the fraction with the greatest increases was that that was organically bound.

Neutralisation of residue leachate yields high rates for As removal but were much less for V (70%) and Cr (26%) (Burke et al. 2012), although there is some evidence that the presence of additional sorption sites helps to reduce aqueous V concentration. The use of CW for removal of trace element loadings has received considerable interest (e.g. Vymazal and Krása 2003; Lesage et al. 2007; Kröpfelová et al. 2009). For the elements of concern in the current study, the wetlands have achieved varying levels of success.

Cr levels encountered in the current study were much lower for effluents going to constructed wetlands (Hadad et al. 2006; Khan et al. 2009) and those reported by Burke et al. (2012). However, removal rates in CW are typically efficient with findings of 82–89% removal reported (Hadad et al. 2006; Khan et al. 2009).

There is limited information on the removal of V in CWs with removal of 0–48% reported for three different wetlands (Kröpfelová et al. 2009). Loading values reported were much lower than for the current study. Removal efficiency of As and V from residue leachate is dependent on pH and filter media characteristics (Hua et al. 2015), and at circum-neutral pH, immobilisation by sorption or coprecipitation with oxides is likely to be an important factor. Immobilisation and attenuation of V by sorption to readily extractable oxides and carbonate phases were evident through the wetland (Fig. 4) and consistent with findings reported by Mayes et al. (2011) for stream sediment attenuation. In constructed wetland environments, arsenic is retained mostly in sediments (Ye et al. 2003). Findings for the current study were in agreement with Singhakant et al. (2009) who reported that predominant fractions within the sediment/soils are residual with oxidisable (bound to organic matter and sulphides) the second most important. Increases in soluble As were also noted towards the latter stages of monitoring.

Addition of bauxite residue and residue leachate to soils and freshwater sediments can result in increased levels in trace elements with potential transfer to vegetation (Ruyters et al. 2011; Olszewska et al. 2016; Higgins et al. 2016). Levels of trace elements in *P. australis* encountered in the current study displayed seasonal variation which is attributed to the dynamic growth cycle of the plant as well as variations in the element levels and availability (Bonanno and Giudice 2010).

While the amount of trace elements in the aboveground parts of plants growing in constructed wetlands represents only a very minor portion of the inflowing load (Vymazal et al. 2009), Bonanno (2011) has proposed *P. australis* as a potential bioindicator of Al trace content in wetlands. Seasonal flux has been observed for Al (Batty and Younger 2004), and levels encountered ranged from 20 mg kg^{-1} (summer) to 130 mg kg^{-1} (winter) and are considerably within range for constructed wetlands treating domestic wastewater (Vymazal et al. 2009) and much lower than the 1000–3000 mg kg^{-1} phytotoxic levels cited by Kabata-Pendias and Pendias (2001). The Cr content is within the same range reported by Lesage et al. (2007) and is at the lower end of the range reported by Vymazal et al. (2009) for a range of constructed wetlands and natural stands. The toxicity range of Cr levels is 5–30 mg kg^{-1} (Bonanno 2011), and all values are well below the threshold. Vegetation As concentrations were much lower than for a range of macrophyte plants growing in a lake which received residue leachate discharge (Olszewska et al. 2016). Values reported for summer sampling were similar to previous summer (baseline values) and those reported by Vymazal et al. (2009).

Although there were some increases in the more mobile phases of trace elements in the wetland soil, the main contents were in the more recalcitrant and residual fractions. In addition, there was no evidence of excessive element content in *P. australis* through the wetland. This suggests that bioavailability of these elements remained low over the 1-year operation and monitoring period and agrees with findings of Mayes et al. (2011) who reported that abundance of trace element residue-affected sediments appeared to be associated principally with recalcitrant residual phases that are relatively immobile. Increases in Na content in *P. australis* were evident for summer sampling (same season as baseline), and by the end of the study, the front end of wetland exhibited higher Na content. As soils also exhibited increases in soluble and exchangeable Na, this result is not surprising.

Ruyters et al. (2011) suggested that a possible effect of trace element from bauxite residue loading is nutrient deficiency. Seasonal flux has been reported for nutrient elements in wetlands (Batty and Younger 2004; Bragato et al. 2006), and values for the current study did not indicate deficiency.

Results presented here demonstrate that a reduction in residue leachate pH and element loading is possible through passive treatment systems. Data also indicates that there are no significant increases in bioavailable levels of trace elements and adverse effects to plant growth.

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