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RESEARCH ARTICLE

The potential for constructed wetlands to treat alkaline bauxite-residue leachate: *Phragmites australis* growth

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Abstract High alkalinity (pH > 12) of bauxite-residue leachates presents challenges for the long-term storage and managements of the residue. Recent evidence has highlighted the potential for constructed wetlands to effectively buffer the alkalinity, but there is limited evidence on the potential for wetland plants to establish and grow in soils inundated with residue leachate. A pot-based trial was conducted to investigate the potential for *Phragmites australis* to establish and grow in substrate treated with residue leachate over a pH range of 8.6–11.1. The trial ran for 3 months, after which plant growth and biomass were determined. Concentrations of soluble and exchangeable trace elements in the soil substrate and also in the aboveground and belowground biomass were determined. Residue leachate pH did not affect plant biomass or microbial biomass. With the exception of Na, there was no effect on exchangeable trace elements in the substrate; however, increases in soluble metals (As, Cd and Na) were observed with increasing leachate concentration. Furthermore, increases in Al, As and V were observed in belowground biomass and for Cd and Cr in aboveground biomass. Concentrations within the vegetation biomass were less than critical phytotoxic levels. Results demonstrate the ability for *P. australis* to grow in bauxite-residue leachate-inundated growth media without adverse effects.

Keywords Hyperalkaline wastes · Macrophytes · Metal removal · Mine water · Passive treatment · Red mud

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Introduction

Bauxite residue is the waste by-product generated by the extraction of alumina from bauxite ore via the Bayer process. The annual global production of bauxite residue is about 120 million tonnes, with an estimated global stockpile of three billion tonnes (Klauber et al. 2011). The high alkalinity of bauxite residue presents challenging conditions for the long-term environmental management of bauxite-residue disposal areas (BRDAs). Where BRDAs are not managed correctly, there is potential for leakage to the surrounding environment representing a high environmental risk (Wang et al. 2015). Requirements for the management and treatment of such drainage waters may persist for many decades following closure (Hua et al. 2015).

Treatment of drainage waters by conventional methods is likely to be expensive, especially if it is to be continued for many decades after closure. Recent research has focused attention on the potential for low-cost, passive technology (constructed wetlands) to treat the alkaline leachates from BRDAs (Hua et al. 2015; Buckley et al. 2016), but the focus of these experiments has been on batch trials at laboratory scale.

Wetlands have been effective in buffering alkaline steel-slag leachates (Banks et al. 2006; Mayes et al. 2008), and there is emerging evidence of their ability to treat bauxite-residue leachate (Hua et al. 2015; Buckley et al. 2016). The ability of vegetation to establish within the stressful environment of bauxite-residue leachate wetlands is unknown. Vegetation of wetlands treating alkaline leachates serves several functions such as flow baffling, provision of a large surface area for inorganic precipitates to be lost from solution and provision of a continued carbon source for the microbial decomposers responsible for elevating CO_2 and thereby, the partial pressure of CO_2 (pCO_2) in wetlands (Mayes et al. 2008). Establishment and continued growth of macrophytes are seen as an essential component for an effective wetland system to treat bauxite-residue leachates. Potential impacts of alkaline loading on wetland ecosystems include the following: reduced solubility of micronutrients, reduced microbial activity, reduced availability of potassium and direct toxicity of the OH^- (Mayes et al. 2009).

Alkaline steel-slag leachate reduces shoot production and shoot growth and causes higher root: shoot weight ratios in both *Phragmites australis* and *Thypha latifolia* (Lawson 2004). Similarly, Mayes et al. (2009) observed reduced *T. latifolia* biomass in calcareous substrates receiving lime spoil drainage (pH up to 12.7). Reduced growth in both of these studies has been attributed to reduced nutrient availability in high pH substrates (Mayes et al. 2009). Other characteristics that can inhibit vegetation growth in the steel-slag wetland environments include low-organic matter content, calcareous crusts, poor physical structure and elevated boron (Mayes et al. 2009). Microbial community structure and activity within wetlands play important roles in pollutant removal (Vymazal 2005; Truu et al. 2009). Microbial activity also consumes alkalinity, but microbe activity within wetlands is often overlooked in short-term laboratory trials (Mayes et al. 2009). The effects of residue leachate loading on soil microbial populations are unknown.

In addition to high alkalinity (ca. pH 13 Buckley et al. 2016), bauxite-residue leachates often present high salinity (up to 160 mS cm^{-1}) (Mayes et al. 2011). Several oxyanionic forming elements are very soluble at alkaline conditions and high concentrations of Al (500–1000 mg L^{-1}), As (3–5 mg L^{-1}) and V (5–10 mg L^{-1}) can occur in the leachate (Burke et al. 2012; Hua et al. 2015). Contamination of soils with "red mud" bauxite residue has resulted in increased levels of trace elements (Rutyers et al. 2011; Lehoux et al. 2013), but there is limited knowledge on the impact of residue leachate to growth media and soils.

For constructed wetlands to be considered as a potential mechanism for buffering bauxite residue-derived leachates, the impact of leachate on plant establishment and growth needs to be assessed. We determined whether residue leachate (pH range 8.6–11.1) affects *P. australis* biomass and nutrient uptake in a 10-week pot-based experiment. Potential phytoavailability of residue-associated trace elements was also assessed by measuring soluble and exchangeable fractions. Substrate microbial biomass was also determined as an indicator of leachate effects on soil microbial properties.

Materials and methods

Bauxite residue leachate was collected from an operating residue disposal area, filtered (0.45 μm), and trace element content was determined using a Perkin-Elmer Elan DRCII inductively coupled plasma-mass spectrometer (ICP-MS; As and Cr) and an Optima 5300 DV inductively coupled plasma optical emission spectrometer (ICP-OES; all other elements) (Table 1). Dilutions were prepared in the laboratory representing the anticipated range of closed BRDA leachate (Buckley et al. 2016) (Table 2).

Juvenile *P. australis* plants were provided by a local nursery and were planted in 1-L pots. Growth medium used was a proprietary all-purpose compost (pH 6, organic content 86 %), as a previous study by Buckley et al. (2016) demonstrated greater potential for buffering pH of NaOH solutions in high-organic matter substrates. All plants were pre-grown for 1 month to acclimatize them to their new environment, which was in a nutrient solution containing the following: KNO_3 (1.5 mmol L^{-1}), $Ca(NO_3)_2$ (1 mmol L^{-1}), $NH_4H_2PO_4$ (0.5 mmol L^{-1}), $MgSO_47H_2O$ (0.25 mmol L^{-1}), KCl (1 mmol L^{-1}), H_3BO_3 (25 mmol L^{-1}), $CuSO_45H_2O$ (0.1 mmol L^{-1}), $(NH_4)_6Mo_7O_{24}4H_2O$ (mmol L^{-1}) and Fe(Na)EDTA (0.1 mmol L^{-1}) (Matthews et al. 2005). Following the acclimatization period, replicate 1-L pots ($n = 5$) were placed in basins and subjected to inundation with residue leachate treatments (Table 2). Following the method of Matthews et al. (2005), leachate treatments were fed to the plants at the same volume and on a continuous basis over a 3-month period ensuring the plant pot was submerged in solution at all times.

Measurements of leaf length were carried out weekly, and root length was determined at the end of experiment. The length of the longest root was measured, by measuring with a ruler from the initial point of growth to the endpoint of the root at the tip. Dry weight measurements of aboveground and belowground biomass were determined at harvest.

Post treatment, the wetland plants were carefully removed from the plastic pots to ensure all roots stayed

Table 1 Selected parameters of bauxite-residue leachate

	This study	Literature values*
pH	13.2	12.6–13.1
EC (μS cm^{-1})	67,400	–
Al (μg L^{-1})	1,077,000	352,000–833,500
As (μg L^{-1})	3125	6325–8140
Cd (μg L^{-1})	3.2	–
Cr (μg L^{-1})	148	65–188
Ni (μg L^{-1})	35	–
V (μg L^{-1})	13,500	8977–15,600

* Burke et al. 2012, 2013

Table 2 Dilution rates and pH of residue leachate treatments

Treatment	Dilution	pH
Control	–	6.9
1	1:400	8.5
2	1:300	8.8
3	1:200	9.05
4	1:100	9.9
5	1:75	10.4
6	1:40	11.1

intact. The majority of the compost substrate mass was removed by hand before the roots were washed. Plant material was placed in an oven at 60 °C for 48 h. Once dry, the plant aboveground and belowground mass was recorded. Plant samples were acid digested in HNO_3, and elemental content determined by ICP (see above).

Compost substrate samples were split and one-half were air-dried prior to physico-chemical analysis. Substrate pH and EC were determined in 1:5 solutions, and elemental content was determined in water soluble and exchangeable (1 mol NH_4OAc) extractions. Microbial biomass C was determined on field fresh (<2-mm sieve) substrate using the fumigation-extraction procedure (Jenkinson and Powlson 1976) using the correction factor K_{EC} of 0.45 (Vance et al. 1987; Joergensen and Wichern, 2008).

Data were analysed statistically using one-way analysis of variance, descriptive measures and Pearson's bivariate correlations on SPSS, version 19.0. Following the implementation of the Kolmogorov–Smirnov one-sample normality tests on SPSS, differences between the residue leachate treatments on substrate and plant properties were individually determined by one-way ANOVA, and differences were calculated at the 5 % level using Tukey's test. Figures were constructed using GraphPad Prism, Version 6.

Results

After the 3-month growth trial, alkaline-residue leachate caused a significant increase in substrate pH that increased with leachate concentration (Fig. 1). At the highest concentration rate of residue leachate (1:40 dilution), pH increase was 0.6 pH units. Substrate EC increased significantly in all treatments with an EC high of 0.72 mS cm^{-1} recorded for the highest leachate application rate (3.5 × that of the control). Soluble levels of Mg and Ca were decreased with increasing application rate of leachate and soluble Na was increased. Increased soluble As and Cd was observed with increasing amounts of leachate additions, but this increase was only significant for Cd with the highest leachate pH loading. With the exception of Na, leachate pH did not significantly affect exchangeable elements (Ca, Mg, Cr and Al) (Table 3). Soluble and exchangeable V was below detection limits.

Although decreased root length was observed with increasing concentrations of leachate pH, the difference were not significant (Table 4). There were also no significant differences between treatments for shoot length. Similarly, there were no significant differences between treatments for belowground and aboveground biomass. No significant differences were observed for microbial biomass.

There was no significant difference between the treatments for *P. australis* nutrient content in the belowground samples (Fig. 2). Na content significantly increased between each treatment with the highest leachate application displaying results higher than all other treatments, but no significant differences were observed for aboveground samples.

For aboveground biomass samples, increased leachate pH did not result in any significant differences for N, K, Na and Mn content (Fig. 3). Significantly lower values for Ca, Mg, S and Mn were observed with increased residue leachate pH.

No significant differences were observed for Ni, Cd or Cr content in belowground biomass (Fig. 4). There was a pattern of increased concentrations with increased rate of leachate for Al, As and V with significant differences for the higher pH treatments. Significant differences were also observed for As and Al content at the highest application rates. For aboveground samples, only Cd and Cr displayed significant differences, and these were at the higher application rates (Fig. 5).

Discussion

Residue leachate was both alkaline and saline with pH marginally higher than values previously reported (Burke et al. 2012, 2013). Elements determined were also in the range of the previously reported values with the exception of Al which was higher (Burke et al. 2013), possibly due to the slightly higher pH. Whilst there have been no published studies on application of residue leachates to soils and substrates, Lehoux et al. (2013) and Mišík et al. (2014) reported similar findings (increase in pH, increase in salinity and increase in aqueous metal(-loid) concentrations) upon addition of residue to soils.

Al and V (except for pH 11.1) concentrations in the current study were below the limit of detection, and soluble levels for other elements determined were below values previously reported (Czop et al. 2011; Lehoux et al. 2013) over the same pH range. This may be due to the buffering capacity of the organic substrate used in our study. The most effective soil for buffering residue additions was attributed to those with high-organic carbon content (Lehoux et al. 2013; Buckley et al. 2016). Reduction in solution pH is a critical factor for removing trace elements from alkaline-leachate water (Hua et al. 2015).

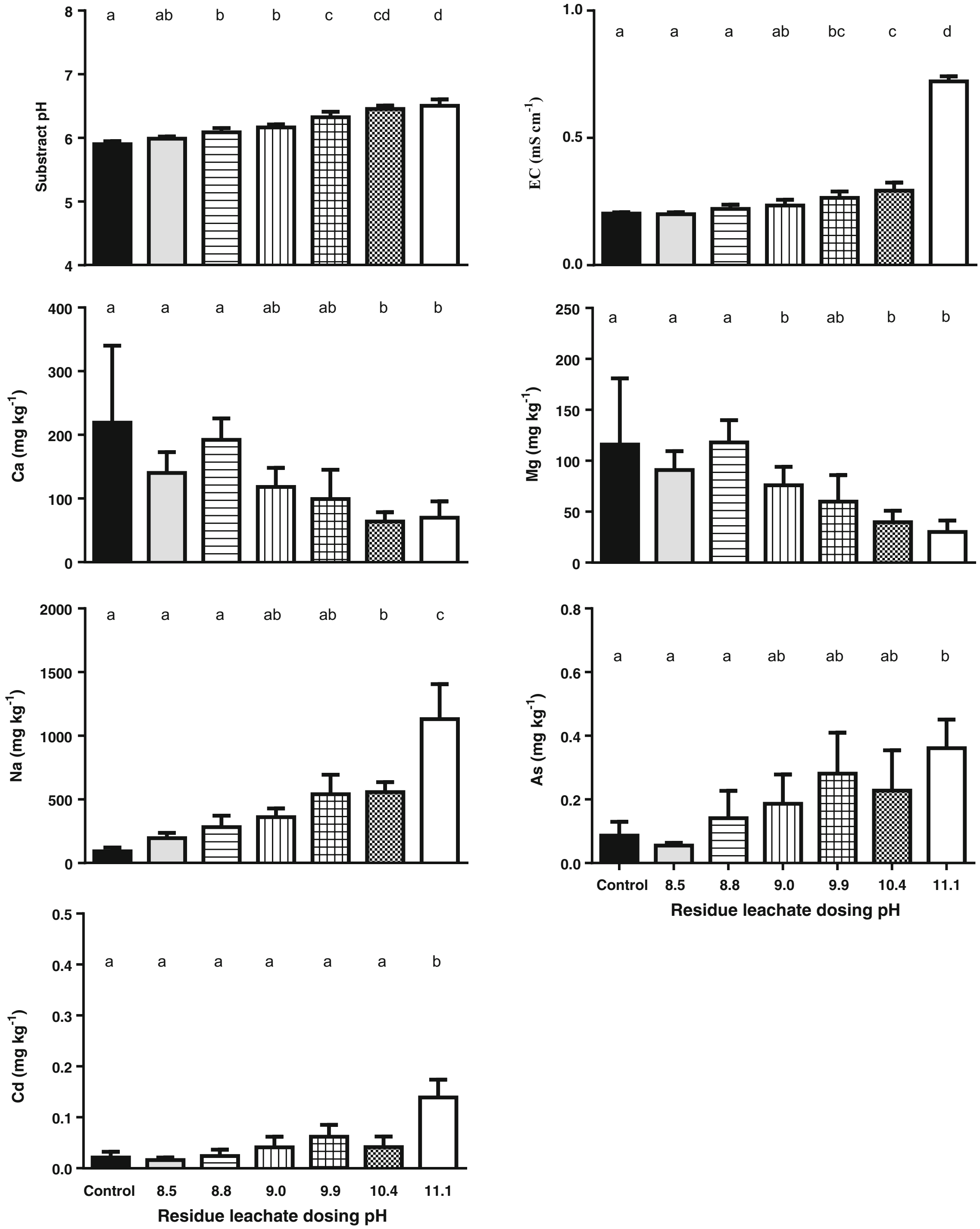

Fig. 1 Substrate pH, EC and soluble elements in residue leachate treatments. *Means followed by the same letter* are not significantly different at $P \leq 0.05$

Adsorption of arsenate and vanadate to mineral surfaces occurs at circumneutral pH (Peacock and Sherman 2004), and aluminate becomes highly insoluble below about pH 10.5 and precipitates as an amorphous oxyhydroxide phase (Burke et al. 2012). Significant decreases in aqueous Al, As and V concentrations with red mud additions were observed below approximately pH 8.5 and resulted from an enhancement in both sorption (As and V) and precipitation (Al) that effectively inhibited metal(loid) release to solution (Lehoux et al. 2013). Soluble V in the pH 11.1 treatment was a magnitude of ten times lower than values reported likely to cause genotoxic effects in plants (Mišík et al. 2014). Although leachate additions in the current study raised substrate pH levels, all values were considerably lower than pH 9. Mišík et al. (2014) suggested that pH control of soil to pH 9 in residue-affected soils would mitigate high aqueous concentration phytotoxic effects.

Table 3 Ammonium acetate extractable elements (mg kg^{-1}) in bauxite-residue leachate-treated substrate following *Phragmites australis* growth trial (means ± SE, *n* = 5)

Treatments	Ca	Mg	Na	Al	As	Ni	Cr
Control	4480 ± 228a	1280 ± 156a	9.5 ± 3a	2.5 ± 0.1a	0.19 ± 0.02a	4.8 ± 0.7a	0.25 ± 0.01a
pH 8.5	3810 ± 229a	1280 ± 25a	93 ± 7.5a	2.4 ± 0.1a	0.35 ± 0.04a	5.0 ± 0.6a	0.26 ± 0.01a
pH 8.8	5300 ± 122a	1830 ± 76a	194 ± 18ab	3.8 ± 0.7a	0.24 ± 0.02a	4.4 ± 0.1a	0.26 ± 0.01a
pH 9.0	4400 ± 201a	1550 ± 119a	243 ± 33b	2.6 ± 0.1a	0.33 ± 0.03a	4.7 ± 0.08a	0.26 ± 0.01a
pH 9.9	4110 ± 206a	1420 ± 71a	461 ± 55bc	2.6 ± 0.1a	0.46 ± 0.02a	4.9 ± 0.2a	0.26 ± 0.01a
pH 10.4	4850 ± 221a	1870 ± 127a	848 ± 105c	3.1 ± 0.3a	0.20 ± 0.04a	5.4 ± 0.2a	0.29 ± 0.02a
pH 11.3	4330 ± 131a	1620 ± 111a	2180 ± 174d	2.5 ± 0.1a	0.27 ± 0.03a	5.1 ± 0.1a	0.27 ± 0.01a

Means followed by the same letter in a column are not significantly different at $P \leq 0.05$

Increased alkalinity, salinity and sodicity has led to inhibition of root growth in a range of plants growing in bauxite-residue aqueous extracts (Courtney and Mullen 2009) and residue additions to soil resulted in decreased plant growth and increased trace element uptake (Ruyters et al. 2011). Decreases in plant growth in alkaline wetland substrates were previously attributed to reduced N and P availability in the high-pH substrates (Lawson 2004; Mayes et al. 2009). Increased levels of soluble and exchangeable Na in the compost substrate highlight concern for long-term soil quality and performance in constructed wetlands. Soils with high Na content (sodicity) can have reduced permeability, and this may impede wetland performance.

Deficiency levels for macronutrients in *P. australis* of 1.45 % for N, 0.06 % for P and 0.7–0.75 % for K were reported by Allen and Pearsall (1963). Although leachate additions resulted in a trend of decreased N content, this was not significantly different, and indeed, N values of >1.5 % were recorded for the second highest leachate application rate. No treatment displayed nutrient deficiencies in the aerial portions for P and K. Although significant differences were recorded for Ca, Mg and S content, the Ca and Mg content for aboveground samples were within the normal range reported by Vymazal and Šveha (2012) for natural wetlands.

Concentrations of Ca for adequate growth in plants are normally around 0.5 % shoot dry matter (Batty and Younger 2004). For leachate applications of 1:100, this value was not reached. The lower concentrations in the higher pH-loading treatment occurred despite exchangeable reserves for all treatments being adequate although soluble Ca was significantly decreased. The presence of elevated concentrations of Na in soluble and exchangeable form may inhibit uptake of Ca into root tissues. Mean ranges for Na in belowground tissues in natural wetlands are 0.14–0.27 and 0.27–0.75 % for constructed wetlands (Vymazal and Šveha 2012). Even with the higher application rates, the Na content of belowground samples is at the lower end of the constructed wetland values. Transfer of Na into aerial portions as a result of higher leachate loadings was not evident.

Out of six non-nutrient elements detected in plant samples, concentrations in belowground biomass were several magnitudes higher than that in aboveground for Al, As and V. A trend of higher content in belowground compared to aboveground was also observed for Cd, Cr and Ni. Restriction of translocation to aboveground biomass is believed to be a strategy of metal tolerance, and plants can avoid the potential effects of high metal concentrations on the photosynthetic tissue (Bragato et al. 2006).

Bonanno (2011) has proposed *P. australis* as a potential bioindicator of Al trace content in wetlands. In the current study, results showed higher concentrations in belowground

Table 4 Plant growth parameters for *Phragmites australis* and microbial biomass in substrate samples (means ± SE, *n* = 5)

Treatments	Root biomass (g/pot)	Root length (cm)	Shoot biomass (g/pot)	Shoot length (cm)	Microbial biomass (μg C/g)
Control	7.6 ± 1.6a	53 ± 10a	1.3 ± 0.3a	56.4 ± 4.9a	780 ± 152a
pH 8.5	5.8 ± 1.1a	43 ± 7.5a	1.2 ± 0.2a	56.5 ± 9.9a	583 ± 278a
pH 8.8	6.5 ± 1.5a	42 ± 5.7a	1.3 ± 0.2a	56.4 ± 4.8a	604 ± 199a
pH 9.0	8.6 ± 2.6a	36 ± 3.7a	2.2 ± 0.5a	74.2 ± 8.6a	460 ± 376a
pH 9.9	8.5 ± 1.6a	38 ± 5.2a	1.9 ± 0.4a	63.5 ± 6.1a	798 ± 178a
pH 10.4	5.7 ± 1.4a	29 ± 2.3a	1.0 ± 0.3a	48.4 ± 8.1a	900 ± 285a
pH 11.3	7.1 ± 1.0a	32 ± 4.2a	1.9 ± 0.2a	68.7 ± 8.3a	516 ± 145a

Means followed by the same letter in a column are not significantly different at $P \leq 0.05$

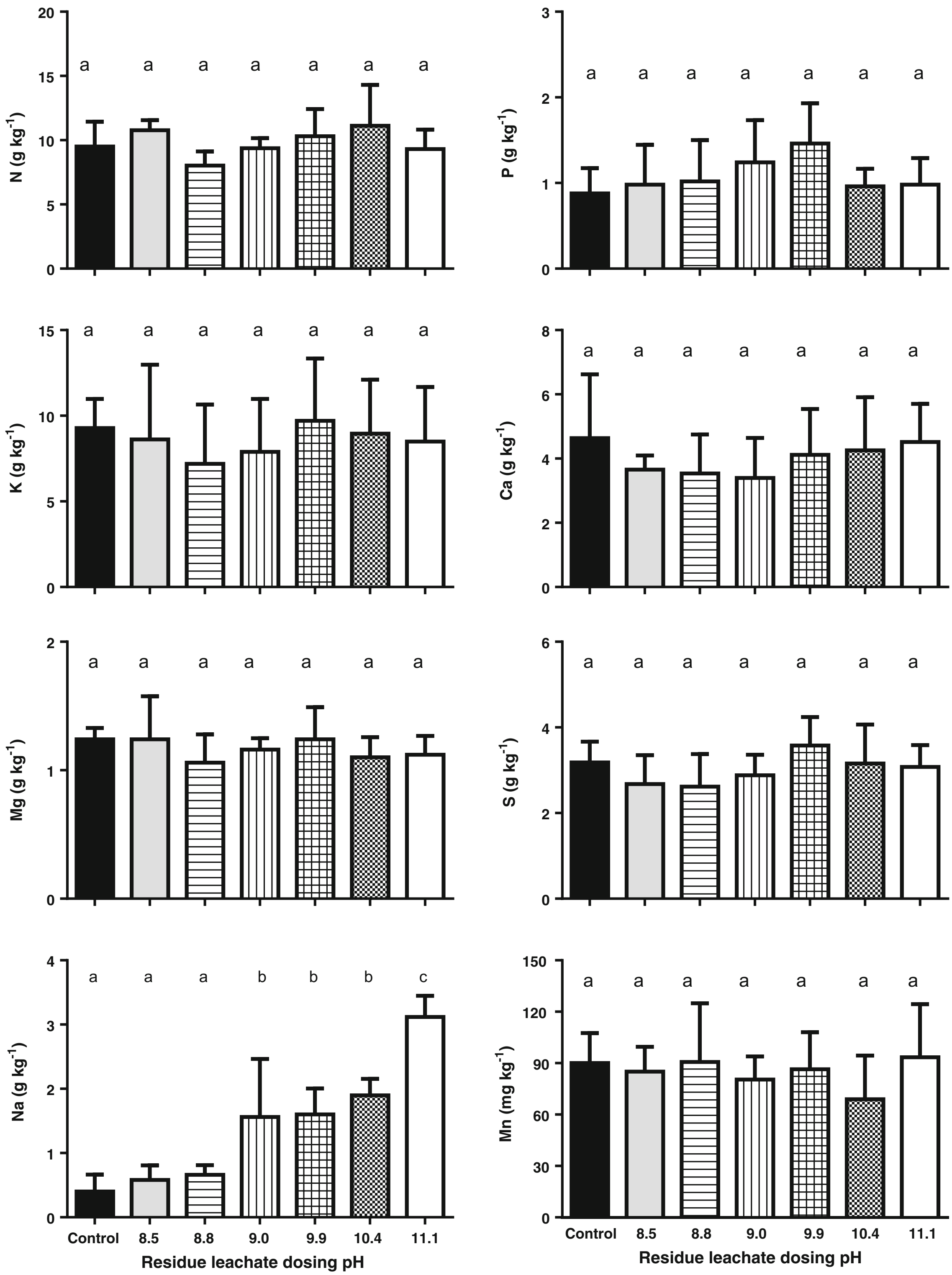

Fig. 2 Nutrient and Na content in *Phragmites australis* belowground biomass. *Means followed by the same letter* are not significantly different at $P \leq 0.05$

samples with low mobility through the aerial portions. This phenomenon is well reported as Al is immobilized in the roots of metallophytes (e.g. Baker et al. 2000; Vymazal et al. 2009; Bonanno 2011). Vegetation content of Al has also been shown to be affected by concentrations in water, and sediment with lower concentrations for both sediment and plant content reported with distance from influent (Lesage et al. 2007). Whilst additions of residue leachate increased levels of Al in

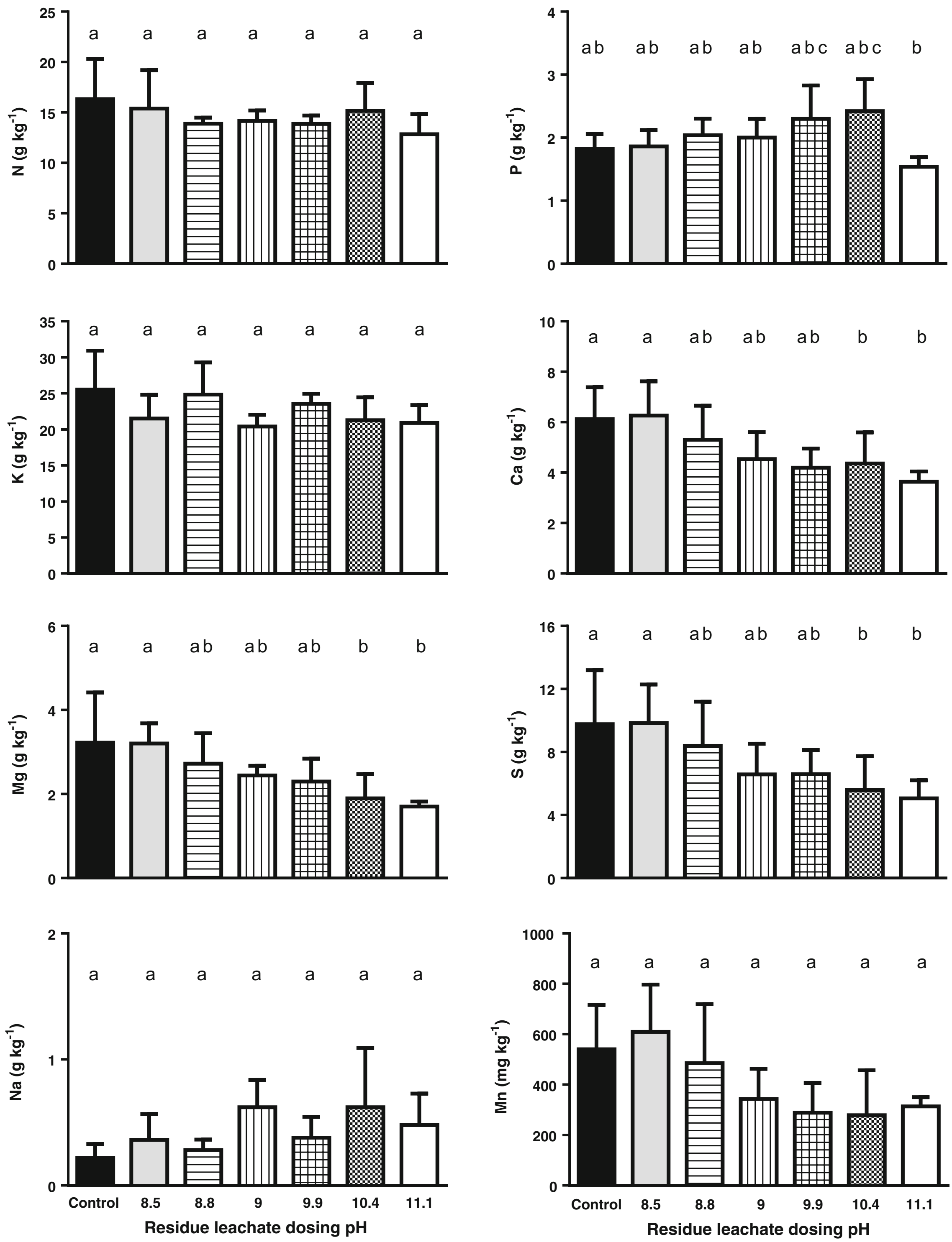

Fig. 3 Nutrient content with Na in *Phragmites australis* aboveground biomass. *Means followed by the same letter* are not significantly different at $P \leq 0.05$

belowground biomass, all concentrations were below those reported for other constructed wetlands treating domestic wastewater (Lesage et al. 2007; Vymazal et al. 2009) and were much lower than the 1000–3000 mg kg^{-1} phytotoxic levels cited by Kabata-Pendias and Pendias (2001).

Cd content in aboveground biomass recorded was significantly increased with leachate applications, but values detected were much lower than values reported for constructed wetlands (Lesage et al. 2007; Vymazal et al. 2007) and river areas (Bonanno and Giudice 2010) and

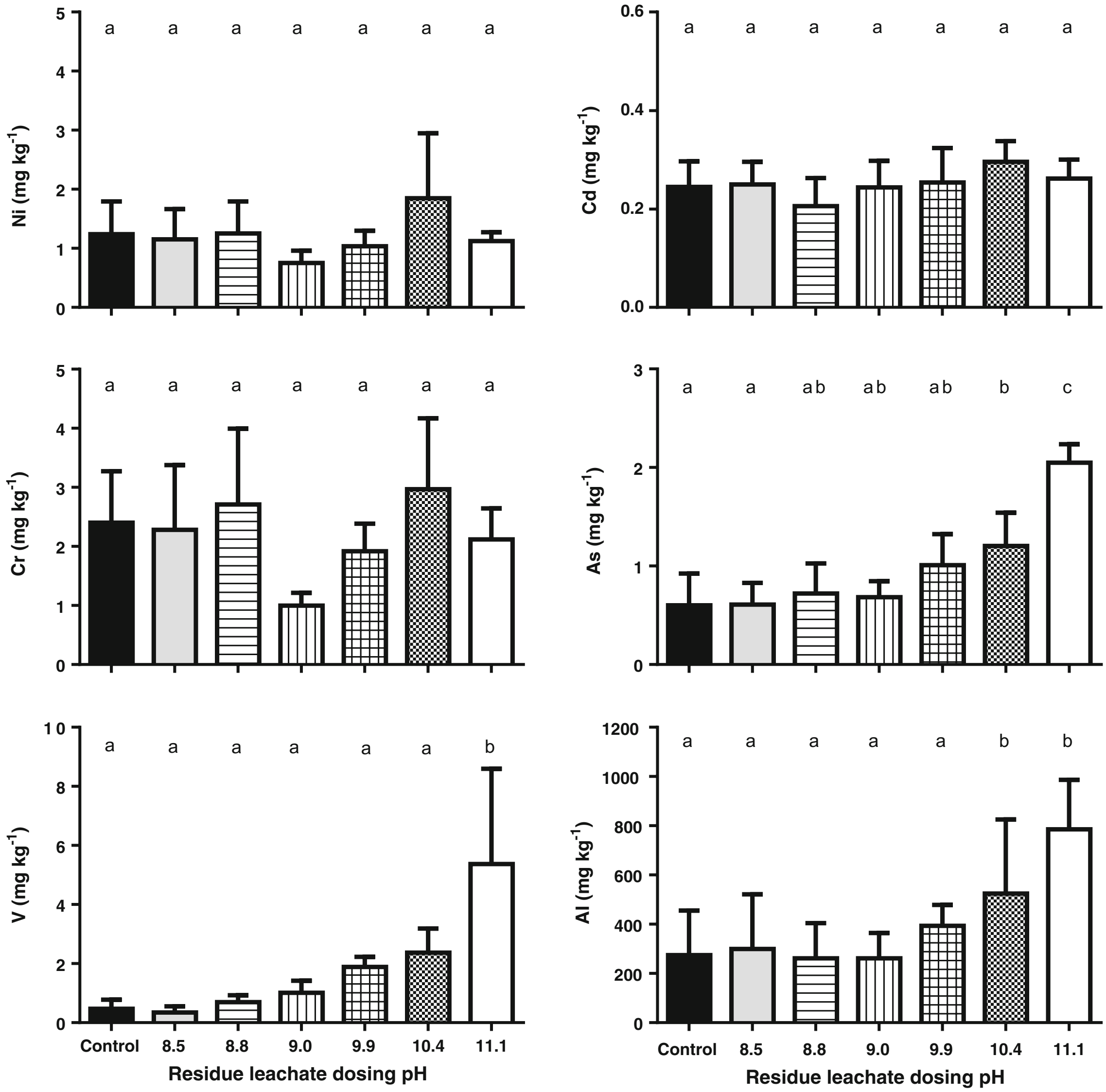

Fig. 4 Trace element content in *Phragmites australis* belowground biomass. *Means followed by the same letter* are not significantly different at $P \leq 0.05$

are well below the phytotoxic range (5–700 mg kg^{-1}) reported by Chaney (1989).

Leachate additions had no effect on belowground Cr levels, and values are considerably lower than the 15–22 mg kg^{-1} reported by Lesage et al. (2007) for *P. australis* in a constructed wetland. Leachate additions resulted in increased levels of Cr in aboveground biomass, fall within the same range reported by Lesage et al. (2007) and are at the lower end of the range reported by Vymazal et al. (2007) for a range of constructed wetlands and natural stands. The toxicity range of Cr levels is 5–30 mg kg^{-1} (Kabata-Pendias and Pendias 2001), and all values are well below the threshold.

Bonanno (2011) reported that roots in *P. australis* may have inherent protective mechanisms to prevent V from penetrating into other organs. Such exclusion mechanisms have been demonstrated in several plant species (e.g. Baker 2000), and Qian et al. (2014) have suggested the limited translocation of V to plant aerial parts provides a selective advantage during colonization and establishment. Soluble forms of V in sediment appear to be readily taken up by roots, and soluble vanadium was below limits of detection in all treatment except treatment 6. Although this treatment yielded the highest V content in belowground samples (ca 5.4 mg kg^{-1}), this is less than the values reported by Bonanno (2011) for the riverside *P. australis* in an urbanized area and is considerably lower than values recorded for brownfield root content in *P. australis* (200 μg g^{-1}). Similarly, the low V content in aboveground biomass (< 0.26 μg g^{-1}) is several magnitudes lower than those recorded by Qian et al. (2014).

Whilst Ni levels above 5 mg kg^{-1} are considered toxic (Allen 1989) by the end of the growing period, leaves can accumulate up to 60 mg kg^{-1} (Bragato et al. 2006). Concentrations in the current study are at the lower end of the range reported for *P. australis* growing in wetlands range

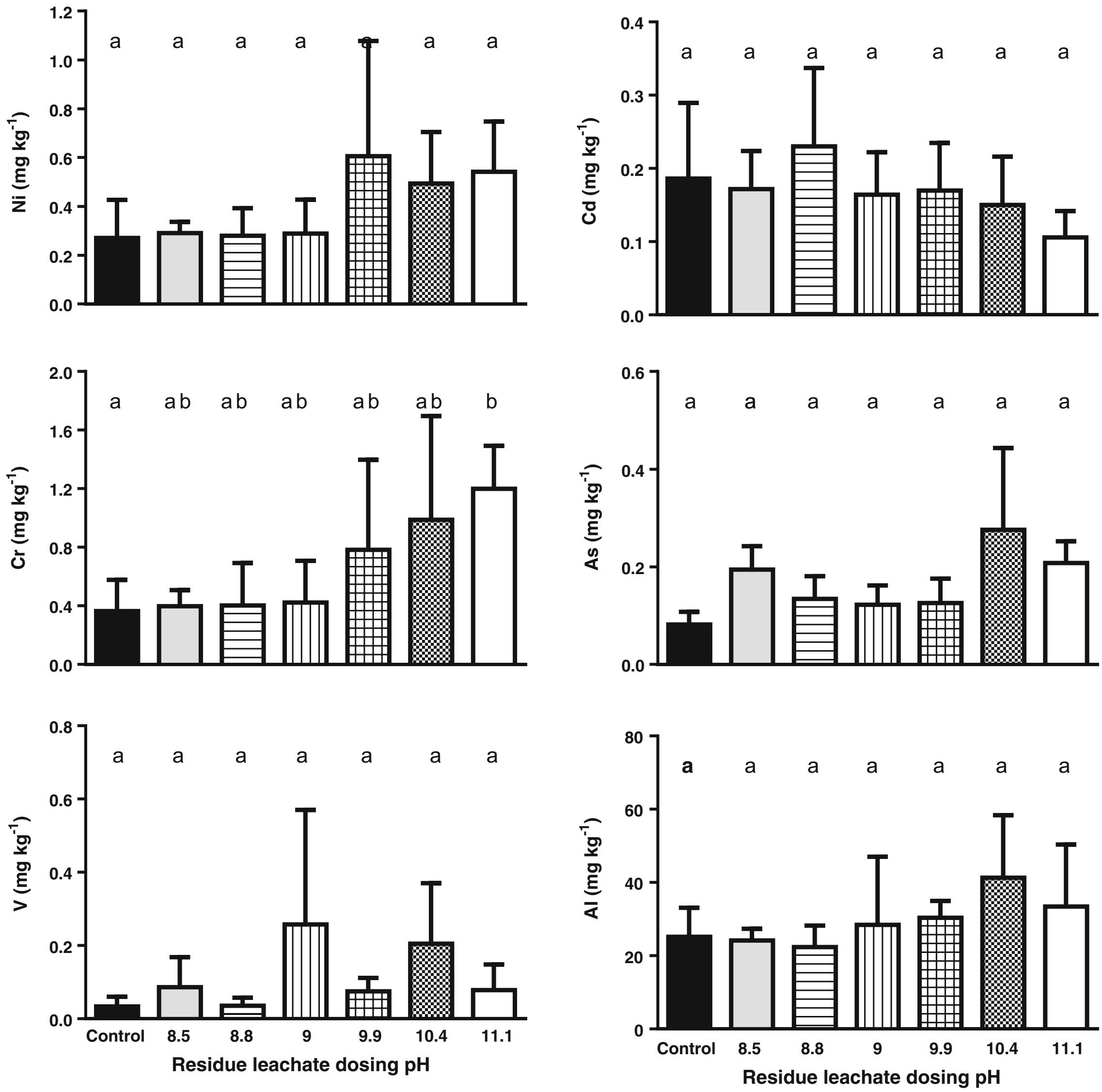

Fig. 5 Trace element content in *Phragmites australis* aboveground biomass. *Means followed by the same letter* are not significantly different at $P \leq 0.05$

from 0.5 to 9 mg kg^{-1} (Bonanno and Giudice 2010) and 0.6 to 11 mg kg^{-1} (Vymazal et al. 2007).

As content in the current study are similar to those reported by Vymazal et al. (2009) for constructed wetlands but are considerably lower than reported by Allende et al. (2014) which examined acidic wastewater. Results are in agreement with Vymazal et al. (2009) and Allende et al. (2014) that roots of *P. australis* have greater As concentration than shoots. Allende et al. (2014) attributed elevated As in wetland plants to the immature state of the wetland system.

Buckley et al. (2016) demonstrated the ability of organic soils to buffer high pH of NaOH solutions. Similarly, buffering of high-pH residue leachate allows for such soils to support plant growth and avoid excessive uptake of trace elements. Trace element levels in *P. australis* detected in the current study are lower than levels reported by Ruyters et al. (2011) for plants growing in red mud affected soils. Ruyters et al. (2011) reported that although these trace elements were detected, they did not exceed toxic limit and hypothesised Na as the prime cause which affected plant growth. Although not investigated in the current study, chlorophyll content (indicator of stress) has been found to have correlations with total N content (Lippert et al. 2001). N content did not significantly vary between treatments or display any negative correlations with trace element uptake.

Factors influencing plant uptake in wetland systems include age of wetland system and period of acclimatization, duration of exposure, the type of vegetation and the type of substrate. Accumulation of Na and metal(loids) in wetland substrates may be problematic in their long-term application for treating hyperalkaline-residue leachates. Analysis of stream sediment samples following the red mud spill at Ajka, Hungary, demonstrated that the bulk of trace elements were in forms that were not readily bioavailable (Mayes et al.

2011). Further work is recommended to investigate trace element accumulation and potential bioavailability in constructed wetlands treating bauxite-residue leachate.

Conclusions

Growth of *P. australis* (below- and aboveground growth and biomass) was not adversely affected in bauxite-residue leachate treatments (pH 8.5–11.1). Whilst some substrate pH, EC and Na content were increased these were not to levels of concern, a pattern of increased trace element content in vegetation was found with increased leachate rates, but no treatments were at levels of concern.

Previously, batch trials have shown the potential for wetland mechanisms to decrease pH and trace element concentration in bauxite-residue leachate and NaOH solutions. Plant growth and biological activity are integral components of constructed wetlands treating mine waters. The ability of *P. australis* to grow in the residue leachate treatments provides strong encouragement for constructed wetland approaches to effectively treat alkaline bauxite-residue leachate.

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