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SOILS, SEC # • RESEARCH ARTICLE

Incorporation of biomass fly ash and biological sludge in the soil: effects along the soil profile and in the leachate water

João Peres Ribeiro[1] • Luís Tarelho[1] • Ana Paula Gomes[1]

[1]Department of Environment and Planning/Centre for Environmental and Marine Studies, University of Aveiro, Portugal

✉ João Peres Ribeiro

joaoperes@ua.pt

Abstract

Purpose: This work aimed to study the effect of the application of biomass fly ash in the soil profile and percolate water, which is a novel feature. The results produced by this work pose a useful contribute for by-products' valorization for the pulp and paper industry, namely fly ash and sludge, diverting them from landfills and achieving significant savings.

Materials and methods: Soil profiles (0.60 m) were collected in the field and into laboratory-scale vessels. Four soil profiles were used in this work. One of the profiles was used as control. To each of the other three, 7.5 $Mg.ha^{-1}$ of biomass fly ash, fly ash combined with sludge (50:50 %wt.) or a conventional liming agent (CaO) were added. A simulation of the daily natural watering of the soils has been made throughout one month, with collection of the daily percolating from the bottom of the vessels. After this period, soil profiles were divided into three equal-sized depth layers (0.20 m each). Soil pH, electrical conductivity and available Ca, Mg, K, P, Na, Mn, Fe, Zn and Cu contents were determined in the three layers for each of the four soil profiles used. A parallel experiment was conducted in which additional pots of soil were prepared with the same amendment. Ryegrass (*Lolium perenne*) was sown in order to evaluate the effect on biomass growth and possible phytotoxicity.

Results and discussion: Amendment with biomass fly ash elevated soil pH slightly, to values within the most adequate range for plant growth. Results showed small raises in the availability of the essential plant macronutrients Ca, K and Mg, especially in the top layer of the soils, where the amendment materials were applied. The mobilization of cations to the groundwater was always minimal, which is promising since it means little contamination to the groundwater. Ash and ash+sludge amendments produced similar plant growth results when compared to the control. However, biomass grown in Cao-amended pots showed the higher root size.

Conclusions: Incorporation in the soil proved to be a viable way to manage fly ash and sludge from pulp and paper industry, which could mean considerable savings. The effect on soil fertilization was similar to the conventional liming agent. No obvious hazardous effect on the soil or groundwater was found.

Keywords Biological sludge • Biomass ash • Leachate water • Nutrients • Soil fertilization

1 Introduction

Ash can be defined as the inorganic incombustible part of the biomass that results from the process of complete combustion and that contains the majority of the original biomass' mineral fraction (Khan et al. 2009; Melotti et al. 2013; Vassilev et al. 2010, 2013). Worldwide, approximately 476 million Mg of biomass ash may be generated *per* year (Vassilev et al. 2013). In Portugal, in 2015, the paper industry generated by itself over 50 000 Mg of biomass burning residues, such as fly ash, slag or dust (CELPA Statistics). Ash application in soils is a current practice in some countries. Sweden and Finland are two examples of good practices regarding this subject, having specific legislation for this purpose. Biomass ash is usually highly alkaline, with pH in the range of 8-13 (Augusto et al. 2008; Basu et al. 2009; Demeyer et al. 2001; Park et al. 2012; Tarelho et al. 2012, 2015). This, alongside their chemical composition, provides a considerable pH correction potential for acidic soils (Ohno 1992; Vance 1996). This potential depends on factors such as combustion temperature or storage period of ash: the lower these factors are, the greater the neutralizing power will be (Augusto et al. 2008; Park et al. 2005).

As regards the elemental composition of biomass ash, it is dominated by (in decreasing order of abundance) O > Ca > K > Si > Mg > Al > Fe > P > Na > S > Mn > Ti, as well as some Cl, C, H, N, amongst other vestigial elements (Girón et al. 2013; Herbert and Krishnan 2016; Lanzerstorfer 2015; Li et al. 2012; Nunes et al. 2016; Rajamma et al. 2015; Tarelho et al. 2015; Vassilev et al. 2013). This way, biomass ash is a direct source of macronutrients, especially P, Ca, Mg and K (Augusto et al. 2008; Demeyer et al. 2000; Matsi and Keramidas 1999; Nkana et al. 2002; Park et al. 2012; Saarsalmi et al. 2012). Some sorts of biomass ash may contain some potentially hazardous elements as well, such as As, Cd, Zn, Cr, Cu, Pb or Hg, which tend to concentrate specially in fly ash (Khan et al. 2009). Even that biomass fly ash can increase those elements' concentration in the soil, their solubility and availability to plants tend to be reduced through pH raise, especially for Fe, Mn, Zn and Cu (*e.g.* Saarsalmi et al. 2012).

According to UNIDO and IFDC's *Fertilizer Manual* (1996), the most relevant factors affecting nutrient availability to plants are: (i) soil pH - in their normal state, soils have pH from about 3.8 to 9, whereas most of the nutrients are more available at pH from 6 to 7.5; (ii) Soil cation exchange ability, which provides the soil the capacity to maintain nutrient ion concentrations at levels conducive to plant growth; and (iii) soil organic matter. The chemical form in which nutrients are brought to soil is equally important. In conventional fertilizers, the majority of the nutrients are provided in the soluble form, which means more likely to be lost by leaching. In biomass ash, only a part of the nutrients occurs in a soluble form, while the other part is progressively liberated through gradual solubilisation, which may favour their utilization by the plants (Gonçalves and Moro 1995).

This work aimed to study the effect of the application in the soil of biomass fly ash and biological sludge, both from pulp and paper industry. The outcome of the amendment was studied through the soil profile (three equal-sized depth layers), as well as in the percolate water, which is a novel feature. This way, the results produced by this work pose a useful contribute for by-products valorization from the pulp and paper industry, namely fly ash and sludge, diverting them from landfills and achieving significant savings.

2 Material and methods

2.1 Collection of soil samples from the field

The collection of the undisturbed soil profiles from the field has been carried out using a cylindrical acrylic plastic sampler open in both ends of dimensions 0.20 m diameter and 0.60 m height. The sampler was vertically introduced in the soil and the sample profile taken out with minimal disturbance. The soil samples were then transferred to laboratory-scale vessels. In order to simulate the natural conditions on the field, the surface of the soil profiles was left open to air and light, whereas the rest of the profile was isolated from solar light.

A cambisol, collected in the central coastal region of Portugal, district of Aveiro (40°45'30.65''N, 8°29'20.11''W) has been studied. The characterization of this soil has been carried out, as well as the characterization of the amendment materials to use. Results from these procedures can be found in Tables 1, 2 and 3.

2.2 Amendment of the studied soil profiles

The amendment materials used in this work were:

i) Fly ash (*A*) from a fluidized bed combustion facility of the pulp and paper industry operating with residual biomass from felling of *eucalyptus* (bark and branches). The ash sample was composed by material collected once a month between January and November 2015 in the cleaning equipment (economiser and electrostatic precipitator);

ii) Thickened biological sludge (*S*) from biological treatment of wastewater from pulp paper industry (from now on designated only as “sludge”);

iii) Calcium oxide (CaO), produced at industrial level and containing a guaranteed minimum of 92% of CaO.

Two types of fly ash application in the soil have been tested: amendment with ash only and amendment with ash mixed with sludge, in a 50-50 mix (%wt., dry basis). Amendment with a conventional liming agent (calcium oxide) was included in the test. One unamended profile was added as control. The application of these materials has been made at the surface of the soil profiles and at 7.5 $Mg \cdot ha^{-1}$ load, since this is the minimum load required to raise this soil pH to the recommended values: 6 to 7.5, to optimize nutrient availability (UNIDO and IFDC 1996). Application of the ash-sludge mix aimed at studying the effect that ash can have on a soil enriched in organic matter, and at the same time find a suitable destination for the biological sludge. The purpose of studying the behaviour of a soil amended with calcium oxide was to quantify the effect on soil fertility that comes strictly from pH raise. A daily addition of water has been performed in order to simulate rainfall. The quantity of water applied aimed at replicating the average precipitation regime in Aveiro, which was estimated to be the equivalent to 0.065 L of water applied per day to each soil profile. This proceeding has been performed during a period of about one month, with collection, at the end of each day, of the percolate water from each vessel. The percolate samples were immediately analysed for pH and electrical conductivity (EC), and then stored at pH≈2 (HNO_3 addition) in weekly combined samples for nutrient analysis.

2.3 Characterization of the amended soil profiles and percolate water

After amendment, all soil profiles were divided into three equal-sized depth layers (0.20 m each), labelled from now on as “Top” (T), “Middle” (M) and “Bottom” (B). “Top” layer corresponds this way to the first 0.20 m of soil profile, while “Middle” layer comprises soil from 0.20 to 0.40 m depth and “Bottom” layer comprises soil from 0.40 to 0.60 m depth. Each of these layers was air-dried to constant mass and sieved to 2 mm. The different soil layers were analysed for pH and electrical conductivity, according to ISO 10390:2005 and ISO 11265:1994, respectively, and then extracted by the Mehlich III (M3) technique. M3 extraction allows the quantification of the exchangeable (and thus plant available) concentration of nutrients and comprises 0.2 M CH_3COOH, 0.015 M NH_4F, 0.013 M HNO_3, 0.001 M ethylene diamine tetraacetic acid (EDTA), and 0.25 M NH_4NO_3. In this procedure, phosphorous is extracted by reaction with acetic acid and fluoride compounds, while exchangeable K, Na, Mg and Ca are extracted by the action of ammonium nitrate and nitric acid. Other elements are extracted by NH_4 and EDTA. Similar procedure was adopted to characterize the ash and sludge applied in the soil. The contents of available sodium (Na), potassium (K), calcium (Ca), magnesium (Mg), iron (Fe), manganese (Mn), aluminum (Al) and zinc (Zn) in the M3 extracted samples were analyzed by atomic absorption/emission spectroscopy technique, with a PerkinElmer AAnalyst 200 – Atomic Absorption Spectrometer. Phosphorous content was determined by colorimetric determination (ascorbic acid method), using a Camspec ® M501 Single Beam Scanning UV/Visible Spectrophotometer. For this purpose, the methodology proposed in Greenberg et al. (1992) was followed.

2.4 Evaluation of ash amendment's effect on plant growth

A second experiment was conducted with the studied cambisol. 2 kg pots were filled with soil samples collected from the field and amended with the same materials (fly ash, A; fly ash+sludge mixture, A+S; CaO), at the same load. Ryegrass (*Lolium perenne*), a feed crop commonly grown in Portugal, was sown in each pot in order to evaluate the stimulation/inhibition effect of the different tested materials in plant growth. Adequate sun light and water conditions were supplied to the pots. Each condition has been replicated three times. Thirty days after seeding, the aboveground biomass (cut at about 1-2 cm above the surface of the soils) was harvested from the different soil pots, dried in an oven (at 105 ºC) for 24h and weighed. The germination index (GI) was calculated for each parcel according to Equation 1, where *m* designates mass.

$$GI = (m_{\text{biomass grown}}/m_{\text{seeds sown}}) / (m_{\text{biomass grown in CT}}/m_{\text{seeds sown in CT}}) \quad (1)$$

The GI has been proved to be a very sensitive index, indicating inexistence of phytotoxicity of the amendment material when greater than 0.8 (Araujo and Monteiro 2005; Tiquia et al. 1996). After harvesting the aboveground biomass, the soil was taken from the pots and root size was measured.

3 Results and discussion

3.1 Characterization of the pre-amendment soil, fly ash and sludge

The studied cambisol had pH suitable for plant growth, especially on its surface (above 6). Both pH and electrical conductivity (EC) showed a tendency to decrease with depth. The soil is rich in organic matter, compared to literature medium values of about 4-5% (UNIDO and IFDC 1996). Biomass fly ash shows higher pH and EC values than biological sludge. Moreover, ash's pH falls perfectly within the literature-appointed range of 8-13.

The mass fractions (mg·kg^{-1}) of the analysed elements present in the untreated soil, in the ash and in the biological sludge are presented in Table 3. The studied soil was essentially rich in calcium, showing substantial quantities of sodium and potassium as well. Apart from Fe, all available elements' concentrations tend to decrease with depth, which is in accordance with the tendency on EC.

Ash and sludge are both rich in plant nutrients, especially Ca and K in the case of ash and P and Ca in the sludge. The sludge is quite rich in Na as well, which could cause considerable rise in soil salinity. These results showed that the ash utilised in this work had a very similar elemental composition to what usually described in the literature (Vassilev et al. 2013). Regarding heavy metals, the studied biological sludge showed a concerning Zn fraction.

3.2 Characterization of the amended soil profiles

The effect on soil pH and electrical conductivity (EC) of the amendments tested is shown in figure 1, for the four soil profiles (three 0.20 m depth layers for each profile). In figure 1 and from now on, "CT" designates the control soil profile, to which no amendment material was applied, "A" designates the soil treated with biomass fly ash, "A+S" designates the soil treated with the 50-50 mix (%wt., dry basis) of ash+sludge and "CaO" designates the soil profile treated with that liming agent.

Ash amendment increased soil pH only at the soil surface layer, where the amendment was performed. The effect was less pronounced when ash+sludge was applied. Amendment with the ash+sludge mix produced the higher EC results for all layers. In fact, the top layer of the soil amended with ash+sludge mixture showed EC four times higher than the control, which may represent an excess on soil salinization, with problems for crop cultures and soil fertility. The high amount of Na in the sludge's composition can help explain these values.

The mass fractions of available macro and microelements in the amended soils (expressed as mg of element·kg^{-1} soil) are represented in Figure 2. All amendments rose availability of Ca, when compared to the control. This effect was recorded especially in the top layer. Ash amendment proved to be the only one capable of marginally increasing potassium's available mass fraction, especially at the bottom layer. This can be explained by the original composition of the ash, much richer in K than the sludge. Given that the original soil was richer in K on its surface, the higher K mass fraction in the bottom layer for every tested soil may be due to the vertical mobilization of K by the added water. Ash amendment produced small raise in magnesium's mass fraction in the top layers of the different profiles. No visible difference was recorded when sludge was combined with ash in the amendment. Moreover, both tests involving ash showed equivalent results to CaO amendment. In what phosphorous is concerned, the variations among profiles are all marginal, and the mass fractions are always below 100 mg.kg^{-1}, so no clear effect can be seen from the amendments tested. In general, it may be concluded that a higher load could be suitable to this soil, in order to further increase macronutrient concentration. Moreover, results showed no evidence of heavy metal enrichment, since the maximum increase verified for those elements was recorded for Fe (below 150 mg.kg^{-1} regardless of amendment). Mn, Zn and Cu's availability stayed vestigial after every amendment, even when the sludge, richer in Zn, was applied. This was possibly due to pH raise causing the inhibition of those elements' availability, as stated in the literature (e.g. Saarsalmi et al. 2012). Moreover, registered values were always below the recommended safety values found in the literature (UNIDO and IFDC 1996).

3.3 Characterization of the percolate water from the soil profiles

The variations in pH and electrical conductivity of the percolate water are represented in Figure 3. Quick pH raise was observed in all percolate samples (10 to 15 days, depending on amendment material). After this period, pH values slowly tend to a stable value. All amendments raised the percolate's pH to a final value of about 6-6.5, against 5 on the control profile, representing some degree of mobilization of elements through the amended soil and into the groundwater. Electrical conductivity showed tendency to rise in all tested scenarios, even the control profile. Such effect may be due to the permanency of the soil at its field capacity for about one month. During this period some degree of element solubilisation may occur, namely Na solubilisation. Ash amendment caused the highest raise in electrical conductivity. The final (stable) value of EC for the percolate water from ash-amended soil was close to 2250 $\mu S \cdot cm^{-1}$, which may pose as a threat for crop development and especially for groundwater.

The concentrations of available elements found in the collected percolate samples from the different soil profiles ($mg \cdot L^{-1}$ percolate) are shown in figure 4.

Ash amendment clearly increased K concentration on percolates when compared to the control soil. Ash and CaO applications showed potential to increase Ca concentration in the percolate. Ash effect on Mg and Na concentration was also noteworthy. A+S and CaO amendments did not produce any clear effect on Mg concentration in the percolate. P concentration was very low in all percolates, meaning low mobilization of this element. The sludge was the amendment material with greater content of available P. However, A+S amendment caused the lower pH raise, which may help to explain these results.

Ash and CaO amendments showed potential to increase Fe concentration in the groundwater, with levels reaching more than twice the ones registered in the CT. The amendment with ash+sludge mixture did not show this behaviour, possibly due to the smaller effect on pH, inducing different solubilisation of Fe. Minor elements Mn and Zn stayed vestigial for all percolate water samples, showing no concerning mobilisation to the groundwater. Cu concentrations were below the detection limit for the selected method. Considering the abundance of each element in the soil and percolate water, it can be stated that the degree of mobilisation was very small, for all studied elements. The element with the greater degree of mobilisation was K, the most soluble one.

3.4 Phytotoxicity assessment

Table 4 compiles the results of the phytotoxicity assessment. Ash and ash+sludge amendments produced germination index (GI) near 1, which mean similar plant growth when compared with the one registered in the control pot. Pots amended with CaO showed an unexpected decrease in plant growth, with GI near 0.8, which is pointed as the limit for phytotoxicity. Given the purity of the CaO used in this experiment, this result is most likely due to slower biomass growth, which means that if we elongated the test in time, probably the effect in pH would allow the soil to produce higher yield of ryegrass. Moreover, biomass grown in Cao-amended pots showed the higher root size after thirty days: 11.5 cm against 10 cm registered in the control pot.

4 Conclusions

Ash amendment raised the soil pH slightly, within the recommended values for optimum plant growth.

All tested amendments raised the availability of essential macronutrients slightly, and mainly on the soil top layers, where the materials were applied. The results obtained with ash amendment were similar to those obtained with CaO amendment. This showed that ash may substitute this type of conventional liming agent. Regarding possible contamination to the groundwater following ash amendment, results showed that the mobilisation of elements, namely heavy metals, to the groundwater by percolate water was minimal.

The results obtained prove that incorporation in the soil is a viable way to manage the two industrial by-products tested: fly ash and sludge. This could benefit industrial sectors such as pulp and paper industry. Further studies are required to evaluate higher loads of application, different ash:sludge mixture ratios, and also different forms of application (e.g. granular application, instead of powder).

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Table 1. Characterization of the studied soil before amendment

Moisture content [%]	Dry bulk density [kg ds/kg H_2O][a]	Total Porosity [L pores/L soil]	Effective Porosity [L mw/L soil][b]	Specific Retention [LH_2O/L soil]	Field Capacity [kg H_2O/kg ds]
27.9	0.60	0.62	0.24	0.38	0.64

[a]ds – dry soil; [b]mw – mobile water, representing the volume of pores in which water can circulate

Table 2. Characterization of the studied soil in terms of pH, EC and organic matter content, before amendment. Top layer comprises the soil profile from 0 to 0.20 m depth; Middle layer comprises the soil profile from 0.20 to 0.40 m depth; Bottom layer comprises the soil profile from 0.40 to 0.60 m depth

	pH	EC [µS.cm^{-1}]	Organic Matter Content [%]
Top layer	6.07	125.6	13.6
Middle layer	5.57	105.4	14.5
Bottom layer	5.53	93.80	16.0
Ash	12.1	7983	NA[c]
Sludge	7.67	1745	68.2

[c]Not applicable

Table 3. Mass fractions (mg·kg^{-1}) of the main elements in the soil, ash and sludge, before amendment. Top layer comprises the soil profile from 0 to 0.20 m depth; Middle layer comprises the soil profile from 0.20 to 0.40 m depth; Bottom layer comprises the soil profile from 0.40 to 0.60 m

	Na	K	Ca	Mg	P	Fe	Mn	Zn	Cu
–Top layer	390	350	800	130	50	80	20	8.90	10.0
–Middle layer	370	310	780	90	40	130	20	8.40	10.0
–Bottom layer	370	290	750	60	20	110	20	9.20	10.0
Ash	2110	3740	8920	300	1.50	BDL[d]	10	5.30	6.0
Sludge	6910	590	1020	200	2730	1020	70	390	10.0

[d]Bellow detection limit

Table 4 Germination index (GI) and medium root size [cm] for the different tested conditions. CT – control soil; A – soil amended with fly ash; A+S – soil amended with fly ash+sludge mix (50:50 %wt.); CaO – soil amended with CaO.

Soil Pot	Medium plant weight [g]	GI	Medium plant height [cm]	Medium root size [cm]
CT	0.302 ± 0.08	1.000±0.000	5.06 ± 0.52	9.86±1.85
A	0.301 ± 0.05	1.031±0.166	7.50 ± 0.74	10.30±1.06
A+S	0.300 ± 0.06	0.983±0.106	5.27 ± 0.49	11.17±1.79
CaO	0.258 ± 0.04	0.843±0.132	3.11 ± 0.20	11.67±1.47

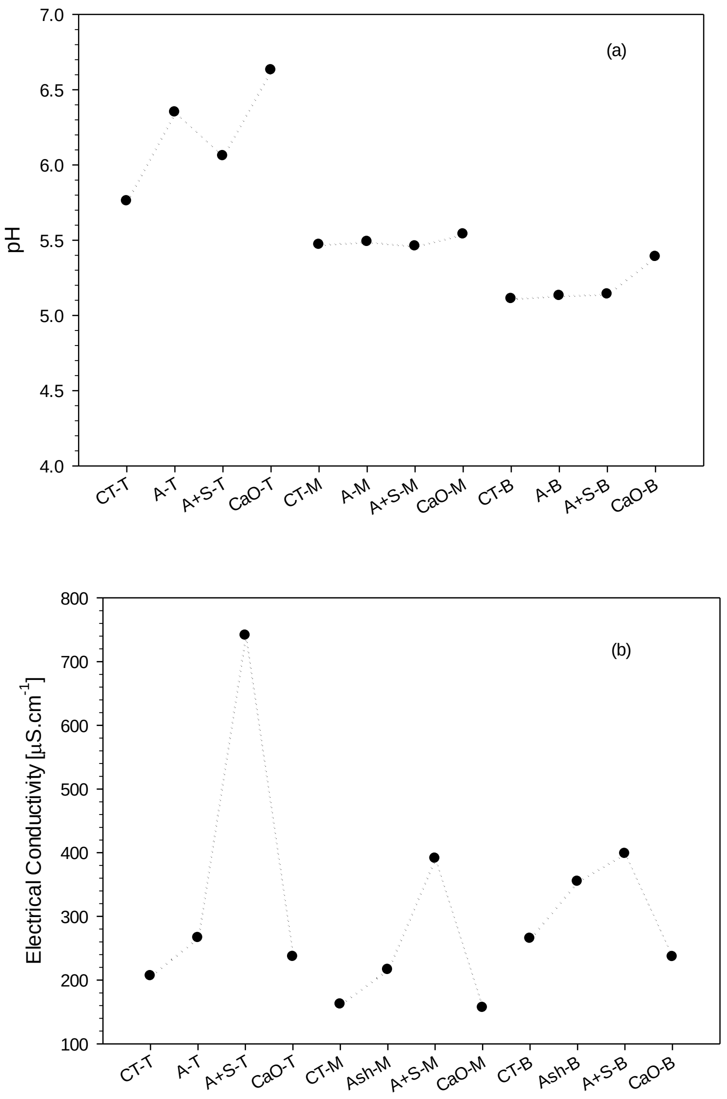

Fig. 1 pH (a) and electrical conductivity (b) of the soil profiles. CT – control soil; A – soil amended with fly ash; A+S – soil amended with fly ash+sludge mix (50:50 %wt.); CaO – soil amended with CaO. Additionally, "T" refers to the top layer of each soil profile, "M" refers to the middle depth layer of each soil profile and B refers to the bottom depth layer of each soil profile

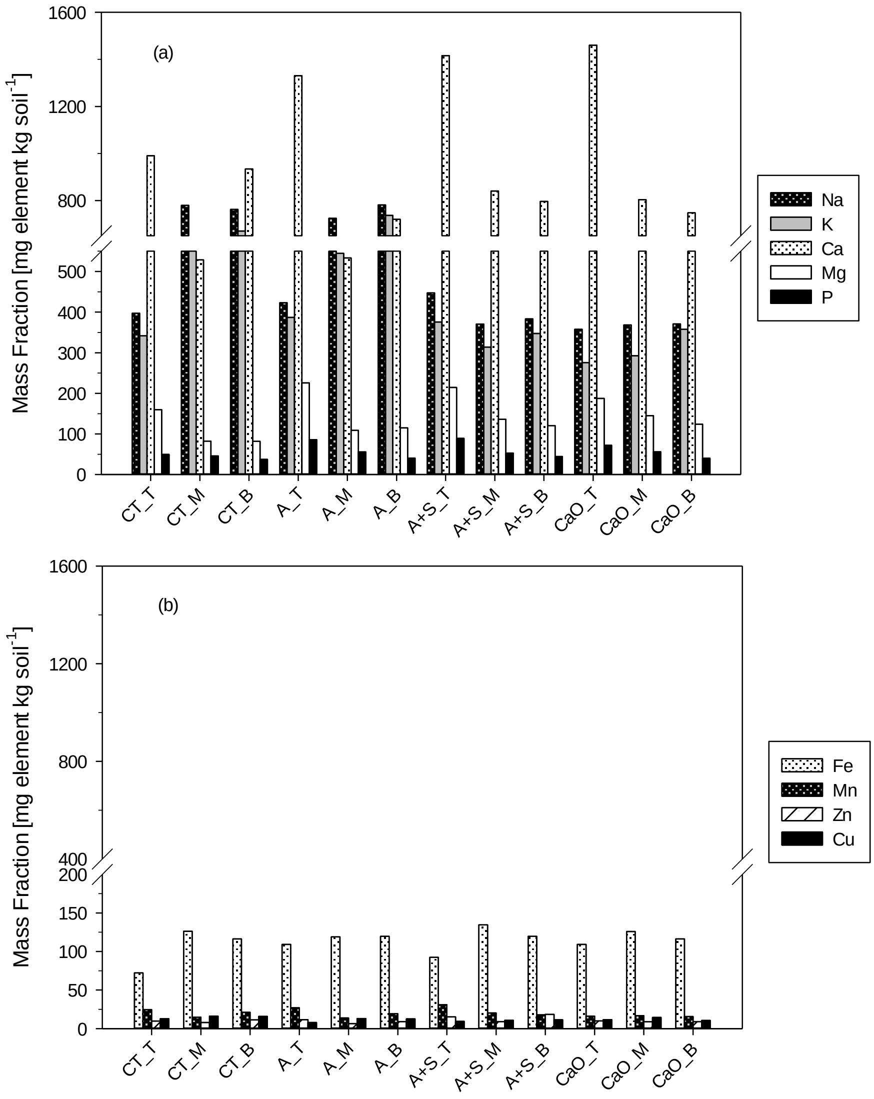

Fig. 2 Mass fraction ($mg\cdot g^{-1}$) (a) of available Na, K, Ca, Mg and P in the soil profiles; Mass fraction ($mg\cdot g^{-1}$) (b) of available Fe, Mn, Zn and Cu in the soil profiles. CT – control soil; A – soil amended with fly ash; A+S – soil amended with fly ash+sludge mix (50:50 %wt.); CaO – soil amended with CaO. Additionally, "T" refers to the top layer of each soil profile, "M" refers to the middle depth layer of each soil profile and B refers to the bottom depth layer of each soil profile

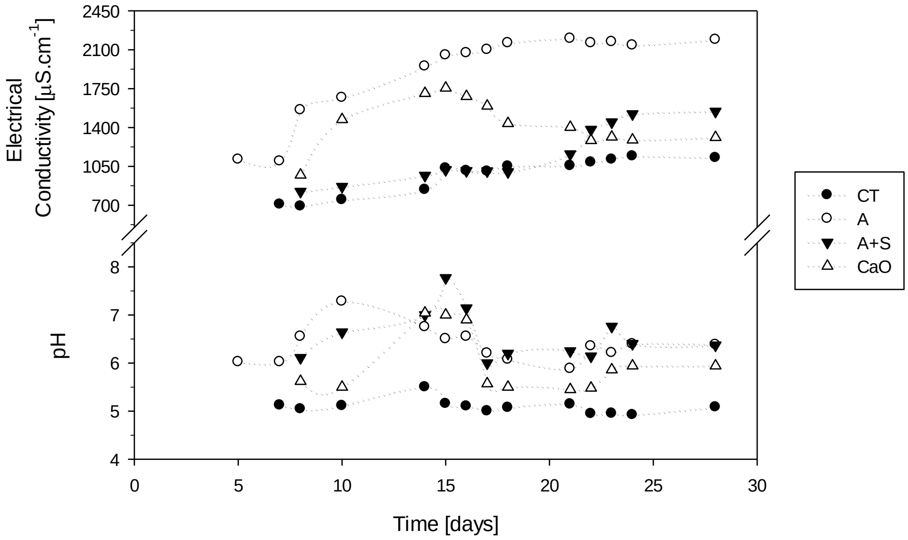

Fig. 3 pH and EC (µS·cm^{-1}) of the soil leachates throughout the leaching process (7.5 Mg·ha^{-1}). CT – control soil; A – soil amended with fly ash; A+S – soil amended with fly ash+sludge mix (50:50 %wt.); CaO – soil amended with CaO.

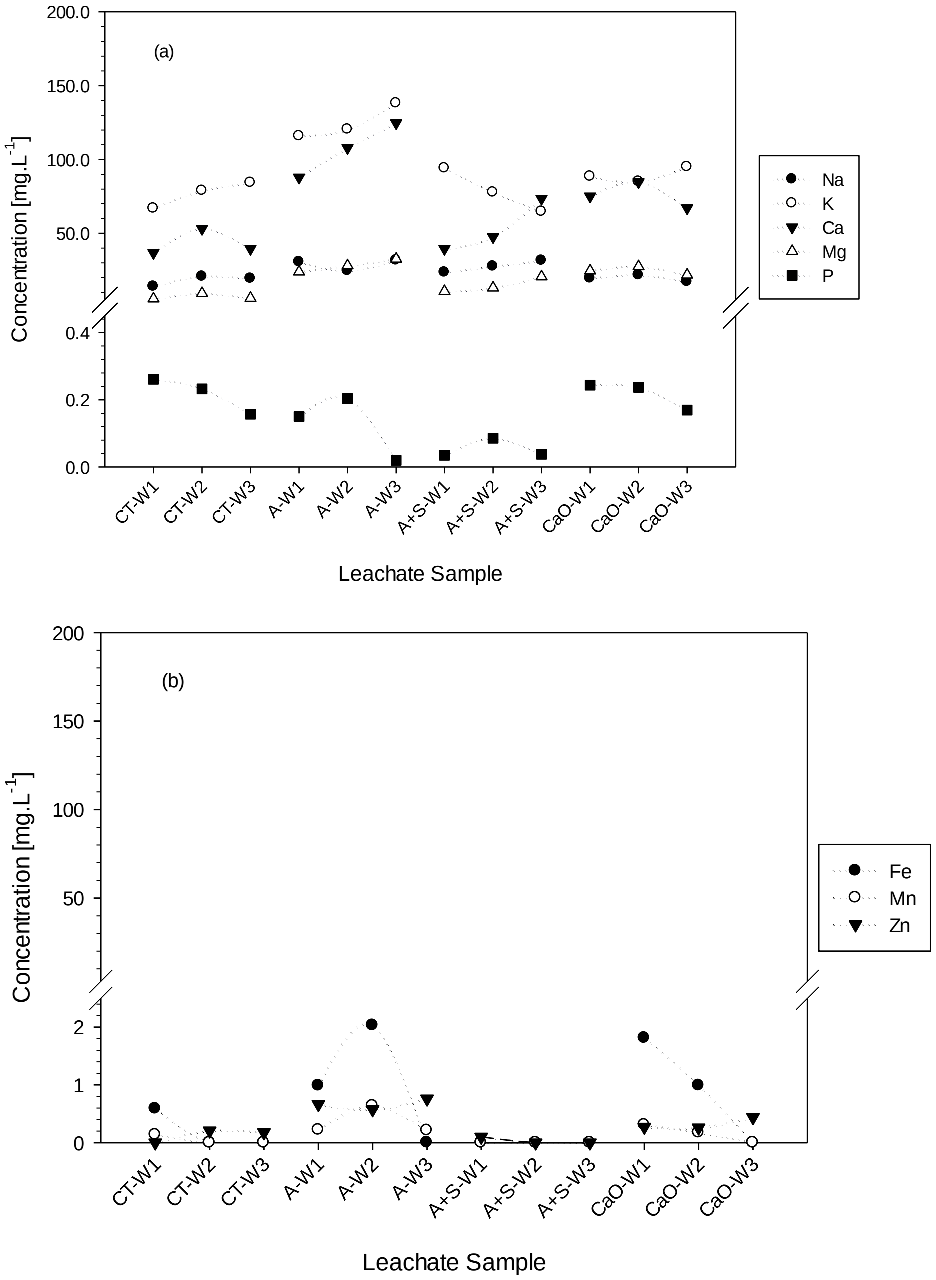

Fig. 4 Concentration (mg·L^{-1}) of Na, K, Ca, Mg, P (a) and Fe, Mn, Zn (b) in the soil leachates for the three consecutive weeks (W1 – week 1, W2 – week 2 and W3 – week 3) of the leaching process. CT – control soil; A – soil amended with fly ash; A+S – soil amended with fly ash+sludge mix (50:50 %wt.); CaO – soil amended with CaO.

386