

The application of ICP-MS and ICP-OES in determination of micronutrients in wood ashes used as soil conditioners

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Received 15 September 2005; received in revised form 8 December 2005; accepted 18 May 2006

Available online 27 June 2006

Abstract

In the present paper, the elemental composition of wood ashes obtained by the combustion of wood in a fireplace was determined with the use of ICP-MS and ICP-OES techniques. Wood ashes may find a potential application as deacidifying agents and soil conditioners, since they contain calcium (in the form of CaCO_3 and CaO), potassium (in the form of K_2SO_4 and K_2CO_3) and significant levels of micronutrients. However, if applied to soil, it is important to assess the bioavailability of particular elements to plants. This process can be simulated by proper extraction procedures.

Various species of wood were combusted in a firestove in a single-family house. The ashes underwent multielemental analyses with ICP-MS Varian Ultra Mass 700 (Australia) and ICP-OES Vista-MPX from Varian (Australia) in order to determine the content of macro- and micronutrients as well as toxic elements. Ashes were also extracted with solutions of 0.1 M NaNO_3 and water in order to simulate the process of elemental transfer from ash (used as soil conditioner) to soil solution and consequently to plants. Also, the environmental impact of ash supplementation to soil was assessed in these experiments. Soil was supplemented with 0–20% of ash. After elution, the eluent underwent multielemental analysis by ICP-MS and ICP-OES techniques to determine the content of macronutrients (P, K, Mg), micronutrients (Fe, Mn, Co, Mo, Zn, Cu and Ti) and toxic elements (Hg, Pb, As and Cd).

It was shown that fireplace ashes can be applied for deacidification of homestead gardens. Ash may be described as a valuable soil conditioner with N:P:K formula 0:1:3. It is concluded therefore that in order to achieve full fertilization, additional supplementation with nitrogen fertilizer would be necessary.

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Keywords: Wood ashes; Macronutrients; Micronutrients; Toxic elements; Soil conditioners; Soil deacidification; ICP-MS; ICP-OES

1. Introduction

The combustion of wood has the potential to become more common way of heating houses. The use of biofuels conforms to recent political tendencies. In Kyoto protocol, 39 industrialized countries (USA, European Union, Russia, East European countries, Japan, Canada, Switzerland) have assigned CO_2 -emission limit. The combustion of wood enables to balance CO_2 emission with consumption. Therefore, in the future, forests will be exploited for wood more extensively [1] and this would lead to a depletion of nutrients in forest soils. One potential way to prevent this would be to recycle the nutrients taken up by burnt wood back to the forest [2]. This would

also be consistent with the policy of sustainable development [3].

Ash from the combustion of wood was sent in the past to landfills, however this method of disposal became expensive. Therefore, the possibility of applying wood ash in agriculture and forestry is being intensively investigated. There are several problems that need to be resolved in order to utilize this by-product in practice. First of all, bioavailability of the nutrients to plants should be assessed. Also, the environmental impact of wood ash supplementation to soil should be investigated, in particular the content and availability of toxic elements and the effect on microbial communities should be studied [4].

Wood ash contains all the nutrients that were taken up by trees from soil, except nitrogen and sulfur that volatilized during the combustion process. Therefore, when used as soil amendment, nitrogen fertilizers should also be applied. When introduced to soil, wood ash acts also as a liming agent by increasing soil pH.

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Table 1
The operating parameters of determination of elements by ICP-OES

Method parameters	Al	Ca, Mg, Na, K	P value
RF power (kW)	0.95	0.80	1.10
Plasma flow (l min ⁻¹)	15.0	12.0	16.5
Nebulizer Flow (l min ⁻¹)	0.69	0.76	0.79
Viewing height (mm)	12	15	15
Pump rate (rpm)	15	15	15
Rinse time (s)	10	10	10
Auxiliary flow (l min ⁻¹)	1.5	0.75	1.5
Replicates	3	3	3
Replicate read time (s)	10	5	5
Instrument stabilization (s)	15	15	15
Sample uptake delay (s)	30	25	23

Element	Emission line (nm)
Al	396.152
Ca	396.847
K	766.491
Na	589.592
P	213.618

Table 2
The operating parameters of determination of elements by ICP-MS

Method parameters	Value
Power (kW)	1.40
Plasma flow (l/min)	16.0
Nebulizer flow (l/min)	0.95
Sampling depth (mm)	5.5
Extraction lens (V)	-450
First lens (V)	-260
Second lens (V)	-11.2
Third lens (V)	0.4
Fourth lens (V)	-60
Photon stop (V)	-10.0
Entrance plate (V)	0.4
Exit plate (V)	0
Pump rate (rpm)	20
Rinse time (s)	10
Auxiliary flow (l/min)	1.25
Replicates	3
Replicate read time (s)	10
Instrument stabilization (s)	10
Sample delay uptake (s)	50

For this reason, it would be advantageous to apply wood ash to soil in the area of acid rain deposition [4].

The composition of wood ash varies and depends on both: tree species and combustion conditions [1]. Wood ash is considered as a rich source of potassium, calcium, magnesium and phosphorus, as well as micronutrients. The average reported composition is: Ca 180 g kg⁻¹, K 27.9 g kg⁻¹, Mg 9.7 g kg⁻¹, P 4.2 g kg⁻¹, N 0.6 g kg⁻¹. It was found that the availability of

potassium was the same as in potassium fertilizers, but phosphorus was lower when compared with phosphorus fertilizers. In the literature [3,11], there is inconsistency in the reports on the availability of magnesium. It is thought that bioavailability of micronutrients (Fe, Mn, Zn and Cu) is low due to increased pH. Wood ash is also a good source of boron [4]. It was found that the level of organic contaminants (PAHs, PCBs, chlorobenzenes and chlorophenols) was low [5,6].

Table 3
The comparison of recommended and information (*) values obtained for Polish Certified Reference Material for multielement trace analysis Fine Flye Ash (CTA-FFA-1)

Element	Certified concentration		Obtained result		Recovery (%)
	Mean	Uncertainty	Mean	Uncertainty	
Al (%)	14.87	0.39	12.64	5.06	85
As (mg kg ⁻¹)	53.6	2.7	46.1	16.1	86
Be (μg kg ⁻¹)	27*	–	28	14	105
Ca (%)	2.29*	–	2.24	0.90	98
Cd (mg kg ⁻¹)	2.8*	–	2.6	0.9	93
Ce (mg kg ⁻¹)	120	7	116	41	97
Cr (mg kg ⁻¹)	156	8	161	56	103
Cu (mg kg ⁻¹)	158	9	153	54	97
Fe (%)	4.89	0.14	4.94	1.98	101
Ga (mg kg ⁻¹)	49*	–	48	17	97
K (%)	2.20*	–	2.18	0.87	99
La (mg kg ⁻¹)	60.7	4.0	58.9	20.6	97
Mg (%)	1.55*	–	1.52	0.61	98
Mn (mg kg ⁻¹)	1066	41	1034	362	97
Na (%)	2.19	0.08	2.28	0.91	104
Ni (mg kg ⁻¹)	99.0	5.8	105.9	37.1	107
P (%)	0.0725	0.0074	0.07	0.02	96
Pb (mg kg ⁻¹)	369	46	362	127	98
Rb (mg kg ⁻¹)	185	5	176	62	95
Sb (mg kg ⁻¹)	17.6	2.5	17.1	6.0	97
Th (mg kg ⁻¹)	29.40	0.70	27.64	9.67	94
Ti (mg kg ⁻¹)	0.58*	–	0.56	0.19	96
U (mg kg ⁻¹)	15.10	0.80	14.04	4.92	93
V (mg kg ⁻¹)	260	10	255	89	98
Zn (mg kg ⁻¹)	569	58	563	197	99

Literature reports that the supplementation of wood ash to soil should be moderate [9]. If applied according to limestone needs, wood ash would be considered a valuable soil amendment, not causing additional soil pollution [4]. It is also advantageous to use wood ash as amendment to acidic forest soil [7,8] usually applied in the dosage 1–7 Mg ha⁻¹ [9].

Hallenbarter et al. [3] studied the combined effect of wood ash and liquid fertilization on nutritional status and growth of Norway spruce in a forest. The authors observed increased growth, but no major shifts in the levels and ratios of nutrients were detected. Arvidsson et al. [9] examined effects of the application of wood ash on ground vegetation of Norway spruce in the dosage 3 Mg ha⁻¹. No visible negative response to the ash application was recorded. Voundi Nkana et al. [10] studied chemical effects of wood ash on growth of rye grass in tropical acid soils. Plants grown on ash-amended soil showed higher biomass production than plants grown on lime and control treatments. Steenari et al. [11] studied the release of mineral nutrients and other species from soils amended with wood ash. Low leach-

ing rates of important plant nutrients were recorded (Mg, Fe and other metals) and rapid release of alkali metals (K, Na) was observed.

Mahmood et al. [12] investigated the effect of wood ash on microbial activity, plant growth and nutrients uptake by ectomycorrhizal spruce seedlings. The authors found that ash treatment had a highly significant positive effect on plant growth as well as on shoot and root concentrations of K, Ca and P irrespective on mycorrhizal status. Zimmermann and Frey [1] examined the effect of wood ash on soil and microbial respiration in soils fertilized with wood ash in the dosage 8 Mg ha⁻¹. Higher biomass and bioactivity of soil microorganisms were observed after supplementation with ash due to increase of pH as well as the level of nutrients, therefore increased mineralization of organic matter was detected. Perkiomaki and Fritze [13] studied the effects of wood ash on boreal forest humus microbial community. The authors found that the supplementation with wood ash resulted in increase of microbial activity. Similar results were obtained by Yrjälä et al. [14], who studied

Table 4
The elemental composition of ashes from various tree species and soils

Element	Literature data		Oak	Oak II	Birch	Apple tree	Ash	Coniferous tree	Soil
	Etiegni et al. [17]	Huang et al. [18]							
Ag (mg kg ⁻¹)			4.19	3.91	6.53	3.65	4.56	4.84	13.4
Al (%)	2.36	1.30	0.85	0.20	0.23	0.15	1.76	0.72	0.64
As (mg kg ⁻¹)			22.0	21.3	19.1	19.8	23.6	23.2	63.9
B (mg kg ⁻¹)	8	127	345	222	511	257	521	274	24.3
Be (μg kg ⁻¹)			535	76	383	255	473	601.	469
Bi (μg kg ⁻¹)			1.37	0.05	60.3	≤0.0005	≤0.0005	177	4.21
Co (mg kg ⁻¹)			7.29	7.450	13.34	4.79	7.230	15.23	4.897
Ca (%)	31.74	10.94	31.1	36.1	29.4	35.1	24.3	19.8	2.32
Cd (mg kg ⁻¹)	21	3	7.8	4.2	23.3	1.7	3.2	16.0	0.27
Ce (mg kg ⁻¹)			1.8	1.9	2.4	0.91	2.9	4.9	6.5
Cr (mg kg ⁻¹)	86	14	75.9	6.07	16.3	5.79	5.69	82.8	25.3
Cu (mg kg ⁻¹)	145	78	111	153	143	96.8	176	123	24.2
Fe (%)	1.95	0.33	0.58	0.56	0.53	0.56	0.48	2.36	0.970
Ga (mg kg ⁻¹)			7.75	73.6	84.8	66.3	87.9	84.6	77.4
Ge (mg kg ⁻¹)			1.60	18.5	24.1	24.5	20.7	43.4	30.2
Hg (μg kg ⁻¹)			6.30	≤0.0001	0.01	≤0.0001	≤0.0001	≤0.0001	95.29
K (%)	4.13	2.86	4.85	8.22	6.25	5.16	12.26	3.43	0.454
La (mg kg ⁻¹)			1.11	5.16	2.31	0.666	1.99	10.4	3.20
Mo (mg kg ⁻¹)			2.81	0.334	0.960	1.28	0.419	1.95	1.74
Mg (%)	2.25	1.62	0.96	3.33	2.51	5.16	2.77	1.35	0.251
Mn (%)	0.669	0.347	0.999	1.23	1.69	0.191	0.236	0.775	0.047
Na (%)	0.34	0.16	0.21	0.15	0.16	0.19	0.24	0.86	0.122
Nb (μg kg ⁻¹)			521	464	419	362	316	8107	1397
Ni (mg kg ⁻¹)	47	12	87.7	63.4	37.8	47.0	102	53.1	10.8
P (%)	1.40	0.69	0.85	1.52	1.97	1.15	2.19	2.11	0.169
Pb (mg kg ⁻¹)	130	66	32.1	87.1	103	25.2	42.6	690	15.2
Pd (μg kg ⁻¹)			653	494	448	580	719	538	567
Rb (mg kg ⁻¹)			47	103	141	37.2	158	177	22.3
Rh (μg kg ⁻¹)			20.4	11.6	≤0.0006	29.2	12.0	11.5	≤0.0006
Sb (mg kg ⁻¹)			2.63	1.13	0.516	1.02	0.49	14.1	4.63
Sn (mg kg ⁻¹)			1.76	0.316	0.351	0.346	0.230	2.32	1.37
Th (μg kg ⁻¹)			≤0.0006	≤0.0006	≤0.0006	≤0.0006	≤0.0006	≤0.0006	0.0015
Ti (mg kg ⁻¹)			259	302	294	237	256	2941	675
Tl (μg kg ⁻¹)			75.6	17.2	0.05	0.00	0.06	119	15.2
U (μg kg ⁻¹)			28.8	13.5	15.5	6.45	18.0	40.0	47.5
V (mg kg ⁻¹)			0.00	2.71	≤0.03	≤0.03	≤0.03	20.3	5.12
Zn (mg kg ⁻¹)	700	794	228	401	4903	756	577	5020	117
Zr (mg kg ⁻¹)			1.08	1.66	1.21	0.770	0.769	7.54	6.53

Table 5

The comparison of the composition of wood ash and soil (a), extraction efficiency (b) and bioavailability (c) of elements from ash, soil and soil supplemented with ash

(a)				
Macronutrients and micronutrients the level of which was higher in wood ash when comparing with soil Macronutrients: K (15 times higher content in ash than in soil), Ca (13 times), Mg (11 times), P (10 times), Na (2.5 times) Micronutrients: Cd, Pb, Bi, Ni, Tl.	Elements the level of which was similar in ash and in soil Cr, Nb, La, Ti, Al, Pd, Ga Fe, Be, V, Sb, Ge, Sn, Mo	Toxic elements the level of which was lower in ash when comparing with soil Hg (91 times), Zr (3 times), As (3 times), Ag (3 times), Ce (2.5 times), U (2 times)		
(b)				
The extraction efficiency of the following elements increased after addition of wood ash to soil K, Sb, Mo, Rb, Ge, B, V, Ga, Na, Cr, U	The extraction efficiency of the following elements reached maximum at intermediate level of ash supplementation (5–10%) Al, Cd, Zr, Ni, Nb, Co, Zn, Mn, Pb, La, Ce, Be, Rh	The extraction of the following elements decreased with increase of ash supplementation Ca, Mg, P, Tl	The extraction efficiency of the following elements did not change after addition of wood ash to soil As, Cu, Fe, Ti, Bi, Sn	
(c)				
Availability of the following elements increased with the increase of the level of ash supplementation Co, Zr, U, Sb, V, Cu, Ce, La, Ge, Ti, Nb, Fe, Sn, Ga, As, Pb, Cd, Pd, Ni, Mn, Zn	Availability of the following elements decreased with the increase of the level of ash supplementation Rb, Ti, Cr, Al, Ca, P, Mg, Bi	Availability of the following elements did not changed with the increase of the level of ash supplementation Mo, K, B	The following elements were available from soil (extraction efficiency was higher than 5%) Mo, K, Sb, V, Ti, Rb, Cr	The following elements were available from ash (the level of supplementation 5%) Mo, K, Rb

changes in *Archaea* from coniferous forest humus after ash treatment.

The aim of this study was to investigate the applicability of wood ash as a soil conditioner and deacidifying agent. The composition of wood ash and bioavailability to plants of macro-, micro- and toxic elements from soil supplemented with ash (as the function of the level of ash supplementation to soil that was in the range 0–20%) to plants were estimated. Also, the environmental impact was simulated under laboratory conditions. In the present study, the level of 39 elements was tested in the studied ash material as well as in leached extracts. The concentration of many elements was very low and therefore could be detected only with the use of the very sensitive methods of ICP-MS and also ICP-OES.

2. Materials and methods

The investigation into the micronutrient content of ash, soil and soil supplemented with ash and their potential availability to plants was assessed directly with the use of multielemental analysis and also after extraction with 0.1 M NaNO₃ (bioavailability test) and water (environmental impact test). Previously [15], several extraction solutions were investigated (1 mol/l MgCl₂, 0.1 mol/l NaNO₃, 0.1 mol/l K₂P₂O₇, 2% (w/v) CH₃COOH, 2% (w/v) HCOOH, 2% (w/v) ammonium citrate, 2% (w/v) citric acid, 0.05 mol/l Na₂EDTA, 0.1 mol/l EDTA, 0.05 mol/l CaCl₂, mixture of HCl and HNO₃) in order to select the extraction system that would enable to simulate the bioavailability of elements to plants. The extracting agent that showed the highest value of correlation coefficients between the composition of

soil extract and plant was 0.1 mol/l NaNO₃ (average correlation coefficient: 0.649). For this reason, in the present study this solution was used to estimate bioavailability of elements from soil, soil supplemented with wood ash and wood ash to plants.

2.1. Wood ash and soil

Wood ash was obtained through the combustion of oak wood in a single family house in a fireplace (Supra, France) having the following characteristics: power 15 kW, the rate of wood combustion was typical (0.06–0.07 m³ wood day⁻¹). After the combustion, the ash was collected, grinded and mixed. Soil used in the experiment was standard commercially available garden

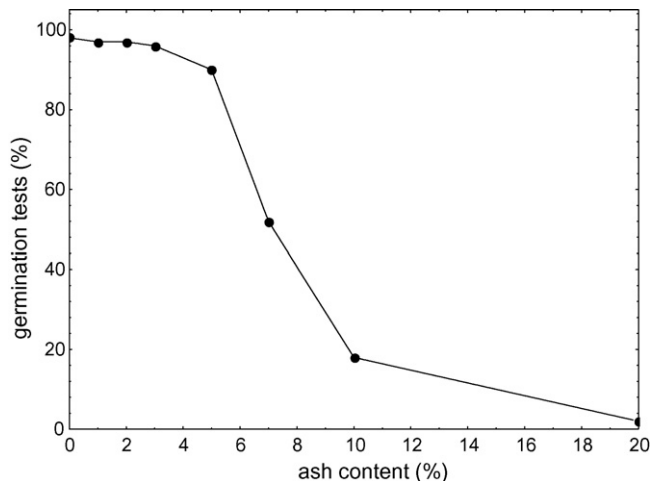


Fig. 1. Germination tests on soil supplemented with ash.

soil that was not enriched with neither organic nor mineral fertilizers. The ash was introduced to the soil in quantities ranging from 0 to 20%. The mixture underwent microwave digestion and afterwards the content of macronutrients, micronutrients and toxic elements was determined. The effect of ash supplementation on plants was also studied by germination tests that were performed on Petri dishes with the use of radish seeds. Germination efficiency was calculated as the fraction of seeds that germinated. Also, the composition of wood ashes originating from other tree species (birch, apple tree, ash tree and coniferous tree) was determined.

2.2. Elution

The ash and the mixture of ash and soil underwent the process of extraction with 0.1 M NaNO_3 solution and water. The composition of the eluents obtained after extraction (4 h) of 10 g of ashes in 100 ml of extractant was determined with the use of both: ICP-OES and ICP-MS. The eluents were analyzed in order to collect data on elemental solubilities in solutions considered to be commonly used extraction solutions and to determine the level of bioavailability to plants. High correlations between

the concentration of the majority of elements in the solution of 0.1 M NaNO_3 after extraction and in plants have been previously reported [15].

2.3. Analytical methods

The samples of ash and soil underwent microwave digestion by samples decomposition in Teflon bombs with the use of Milestone MLS-1200 MEGA microwave digestion system. For digestion of 0.1 g ash and 0.5 g soil, a mixture of ultra pure concentrated acids (2.5 ml of nitric acid and 7.5 ml of hydrochloric acid from Merck) was used. Afterwards, the samples were diluted with demineralized water to the volume 75 ml. The extracts were analyzed directly, without further digestion.

The digested samples of ash, soil mixed with ash, as well as solutions of the extractants after elution underwent multielemental analyses with the use of plasma spectrometry: macronutrients and Al were analyzed with the use of ICP-OES Varian Vista MPX (Australia) (Ca, K, Mg, Na, P and Al). The concentration of micronutrients and toxic elements (except Hg) was determined with ICP-MS Varian Ultra Mass 700 (Australia). Mercury was analyzed with Mercury analyzer AMA 254 (the

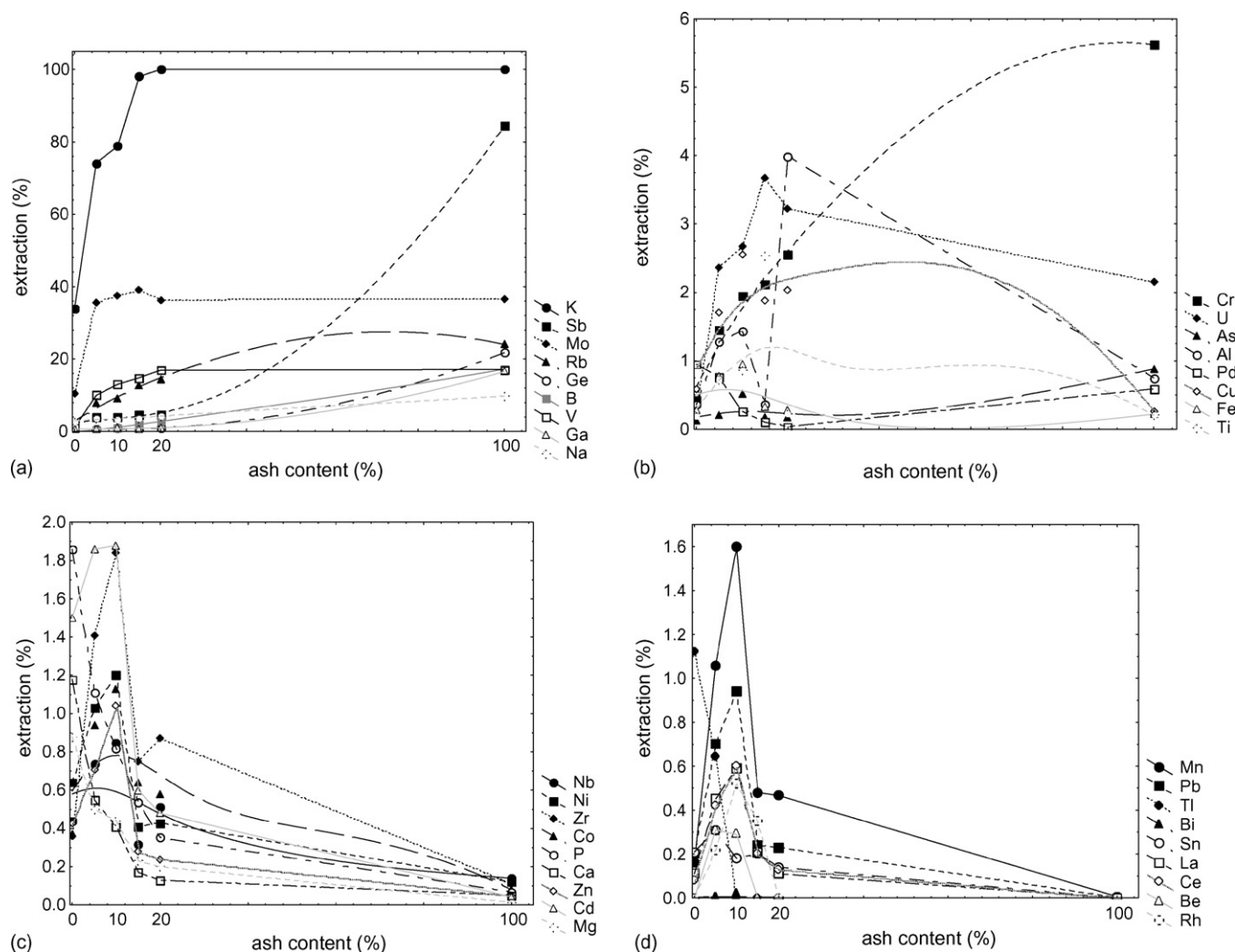


Fig. 2. Elution of elements from soil supplemented with ash with water.

operating parameters: sample mass: 0.2 g, drying time: ash and soil, 10 s, extract, 50 s, decomposition, 200 s, waiting: 60 s), atomic absorption spectrometer (Czech Republic). The operating parameters for ICP-OES are shown in Table 1 and ICP-MS in Table 2. The instruments were calibrated with the use of multielement standard solutions prepared from single elements standard solutions (Ultra Scientific ICP standards 1000 and 10,000 $\mu\text{g ml}^{-1}$). The analytical process was validated by the use of an ash matrix standard. Polish Certified Reference Material for multielement trace analysis Fine Flye Ash (CTA-FFA-1) from Institute of Nuclear Chemistry and Technology (Poland) was analyzed and the comparison of obtained results and certified concentrations is shown in Table 3. Uncertainty and recovery were calculated according to international standards [16].

3. Results and discussion

Many authors report [1,4] that the composition of wood ash varies in dependent on not only tree species, but also on local conditions (i.e. soil content) on which a tree was growing. In the present study, the analysis of wood ash from various tree species (oak, birch, apple tree, ash tree and coniferous tree) showed large

differences (Table 4). Also, differences in the composition of wood ash originating from the same tree species were observed. The average composition of wood ash was compared with the composition of the soil to which it was introduced. The comparison of ash and soil content is presented in Table 5a. It was found that wood ash is a source of macro- and micronutrients. The level of toxic elements was found to be similar. This data support the theory that wood ash if supplemented to soil is a rich source of macro- and micronutrients. It contains also some toxic elements, however since wood ash affects soil pH it would probably decrease bioavailability of elements (supplied by ash and also present in soil) to plants.

When introducing wood ash to soil, it is important to estimate the appropriate dosage. High dosages of ash might be dangerous to plants and the microbial community, due to pH changes. In the present study, the garden soil was supplemented with ash in the quantity 0–20%. We examined the effect of ash level supplementation on germination efficiency of radish seeds (Fig. 1). We also studied the effect of ash content in soil on elemental transfer to the aqueous environment in order to assess the potential transfer of toxic elements to ground and surface waters, as well as to determine the loss of fertilizer components: macro-

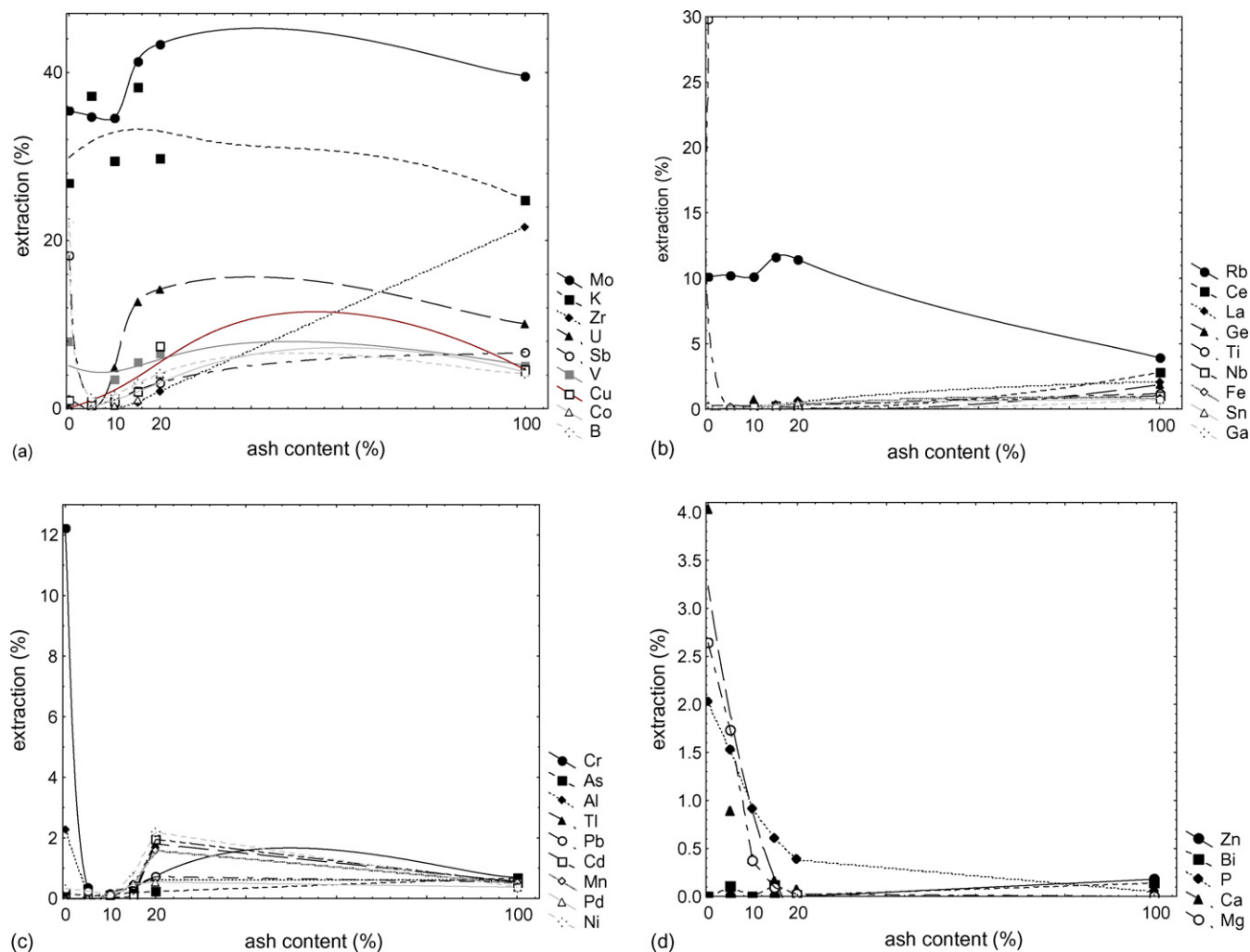


Fig. 3. Elution of elements from soil supplemented with ash with 0.1 M NaNO_3 .

and microelements (Fig. 2). Also the bioavailability of elements to plants was investigated (Fig. 3).

The level of ash supplementation had a substantial effect on seeds germination. It was found that ash supplementation at the level 1–5% did not have strong adverse effect on germination of radish. Introduction of 20% ash resulted in almost total growth inhibition (Fig. 1).

The environmental impact of ash supplementation was studied by extraction with water (Fig. 2). Extraction of ash, soil and soil supplemented with ash was performed. We found that the level of ash supplementation had an effect on elements transfer to water (Fig. 2). The results of experiments on the extraction efficiency are presented in Table 5b. Bioavailability of elements to plants was examined through the extraction with 0.1 M NaNO₃. Different results were obtained when comparing with the extraction with water. Availability of elements is discussed in Table 5c.

4. Conclusions

In order to investigate the theoretical bioavailability of elements from soil and soil supplemented with ash, extraction procedures were also utilized. The environmental impact of elemental transfer to ground and surface water was also investigated. The level of 39 elements was determined in wood ash, soil and extracts using the analytical techniques of both ICP-OES and ICP-MS.

The experimental results showed that the supplementation of wood ash to soil resulted in increase of the levels of macroelements, microelements and toxic elements, the solubility in water and availability to plants was low. Wood ash was found to be a rich source of macronutrients: the concentration of K, Ca, Mg and P was 10–15 times higher than in soil. Also, wood ash supplied micronutrients. Over 15 times higher levels of Mn, Zn and B were detected. The level of some toxic elements was higher (Cd, Pb), but the concentration of other (Hg, As, U) was lower when compared with soil. Although ash was the source of various elements it did not mean that they were readily available to plants and environment. We found that only potassium showed high solubilities in water, unlike the remaining macronutrients (Ca, Mg and P), the solubility of which significantly decreased. The solubility of micronutrients supplied by the ash: Mn and Zn increased at intermediate levels of ash supplementation and decreased at higher levels. The effect on boron solubility was reverse. The solubility of toxic elements supplied by ash (Cd and Pb) was very low: in the case of Cd

less than 2% and in the case of Pb less than 1%. Bioavailability tests showed that the level of ash supplementation did not affect the availability of K. Bioavailability of the remaining macronutrients (Ca, Mg and P) was greatly reduced. The availability of the majority of micronutrients and toxic elements decreased. This showed that ash acted as a fertilizer that released nutrients slowly. This also showed reduced environmental impact, since nutrients were not eluted to ground and surface waters. By taking into consideration germination efficiency of plants, the dosage of ash should not exceed 5%. Therefore, we concluded that the safe level of ash supplementation, when taking into consideration germination efficiency, bioavailability to plants and environmental safety would be on the level lower than 5%.

Summarizing, our data showed that it is possible to use ash obtained by heating a single family house (annual ash production 150 kg ash year⁻¹).

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