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Composition of ashes from heating plants and solubility of ash-forming elements determined by BCR sequential extraction

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ABSTRACT

Wood ash from small-scale (<1 MW) wood-fired heating plants represents a valuable secondary resource containing essential plant nutrients such as Ca, K, and P, but may also harbor potentially hazardous elements. Its current utilization and disposal are typically based solely on total elemental composition, disregarding element mobility and environmental behavior. This study introduces element mobility as an additional criterion for assessing ash quality and suitability for recycling. Sequential extraction using a modified BCR (Community Bureau of Reference) protocol with four solvents (water, acetic acid, hydroxylamine hydrochloride, and ammonium acetate) was applied to evaluate the leachability of 26 elements. Results indicate high extractabilities for several potentially hazardous elements—Sr (54 %), B (48 %), V (44 %), Cd (38 %), and Tl (27 %)—suggesting potential environmental risks despite low total concentrations. In contrast, key plant nutrients, especially P (15 %) were less mobile but could be mobilized under oxidizing conditions. While dissolution of K (74 %) and Ca (12 %) in water and weak organic acid indicates partial plant-availability already in the untreated ash, 26 % resp. 88 % remained in the residual fraction, indicating only long-term plant availability through weathering. The findings demonstrate that both total concentrations and leaching behavior must be considered in environmental assessments. For fertilizer production, pretreatment to remove Tl and enhance P solubility is recommended. From a waste management perspective, controlled ash processing can promote nutrient recovery and support circular economy objectives. These insights contribute to optimizing wood ash management strategies and minimizing environmental risks associated with its reuse.

1. Introduction

Heat generation from wood-fired heating plants is a widespread and important pillar of sustainable energy supply. The ash from these plants contains valuable elements, including important plant nutrients such as Ca, K and P, and should be utilized in the most ecological and economical way possible. However, ash may also contain potential pollutants such as heavy metals, even when only natural wood is used as a fuel [1–3]. In addition to ecological aspects, ash management is also a cost factor for plant operators. A sensible and cost-efficient ash utilization in the sense of the circular economy is increasingly becoming the focus of researchers and practitioners [4].

1.1. Utilization options for ash from wood-fired boilers <1 MW and regulatory framework

The ash quality defined by its elemental composition determines ash utilization or disposal paths. The composition of wood ash is very heterogenous, depending on multiple factors such as the type of fuel used, the combustion temperature and the flue gas cleaning technology [5–9].

Typically, different fuel qualities are used in plants of different size classes. Due to higher fuel demands and a more robust technology, larger plants often use lower quality fuel such as landscape maintenance material or waste wood. These materials are characterized by higher ash contents (e.g. if it contains a high share of bark) or higher contents of non-wood material (e.g. coatings, adhesives, metal parts etc.) and therefore also higher loads if minor and trace elements compared to

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Abbreviations:

BCR	Community Bureau of Reference
CI	Confidence interval
EF_i	Extractable fraction of an element i
ISSA	Incinerated sewage sludge ash
MSWI	Municipal solid waste incineration

natural wood. Smaller plants <1 MW typically burn natural, untreated wood in the form of wood chips or pellets in which generally a low content of potential pollutants can be expected [4,10].

Next to small proportions of unburnt fuel, wood ash consists mostly of the inorganic, non-combustible part of the fuel remaining after complete combustion, i. e. the mineral fraction of the original biomass [11]. During incineration, environmentally relevant elements such as heavy metals accumulate in the ash. Finer ash fractions tend to be more heavily contaminated than coarse fractions. Though lower in concentration, environmentally relevant elements can also be present in the coarse ash fraction [11].

Usually, one, two or three ash fractions are removed from wood fired heating plants. A coarse ash fraction is removed from the combustion chamber, i.e. as grate ash. Finer ash fractions are removed from the flue gas, i.e. as fly ash removed via a cyclone or filter [12]. It is recommended that the ash fractions be removed and disposed separately as far as possible [4]. While this is done in larger plants, smaller plants such as the ones considered in this study often produce only one ash fraction consisting of bottom ash and boiler ash.

Legally, ash is defined in Germany as waste according to § 3 (1) of the German Circular Economy Act which transposes the requirements of the European Waste Framework Directive into German Law [13,14]. Depending on the ash properties, it is classified according to the Waste Catalogue Ordinance. Therein, waste codes are defined [15]. Generally, ash is covered by the waste codes of group 10 (wastes from thermal processes), more precisely 1001 (wastes from power stations and other combustion plants). Ashes from the incineration of untreated wood usually fall under the waste codes 10 01 01 (grate and boiler ash, slag and boiler dust) or 10 01 03 (filter dust from peat firing and firing with untreated wood).

In the waste hierarchy, the circular economy law lays down principles for waste prevention and waste management that follow the following order of priority: Waste prevention > preparation for reuse > recycling > other options, in particular energy recovery and backfilling and lastly, disposal. Ash can be disposed in surface or underground landfills of different standards (landfill classes 0 - IV according to the landfill ordinance [16]). This method still accounts for 70 % of the wood ash produced globally [17]. Other options include mountain backfilling or the co-incineration if the ash in municipal solid waste incinerators (MSWI), where any remaining combustible components are converted and the remainder is treated together with the MSWI ash. The main paths for wood ash reuse and recycling can be categorized as (I) soil amendment and fertilization in forest or agricultural systems, and (II) production of construction or industrial materials [5,18]. Biomass ashes provide plant nutrients and might be a valuable substitute for traditional liming agents such as $CaCO_3$ [19]. While biomass ash application in forestry can offer liming effects and recirculate nutrients to forest soil, it may also result in undesired releases of contaminants such as Cd. Therefore, further research is needed for ash utilization both in forestry and as fertilizing material in agriculture [6,20]. As described in Mayer et al. [6], ash quality criteria and the total quantity that can be used is regulated by the biowaste and fertilizer ordinances [21,22]. However, analyses showed a high variety of the bulk composition depending on the ash origin, ash stream and ash management practices, pointing out the need for further investigation and regulation of ash utilization in soils [23]. Further, wood ashes can be utilized as a partial cement replacement in concrete production as long as material properties, particularly stability are not compromised [24–26]. By using the waste material ash instead of conventional raw materials, the envorinmental impact of concrete production, especially regarding CO_2-emissions can be minimized [27–29].

1.2. Ash characterization through element solubility

As described by Eichermüller et al. [30], wet chemical sequential extraction can be used for the selective separation of elements. The method provides information on the solubility of the elements under different environmental conditions and gives an indication of the chemical speciation of the elements contained in a sample.

Based on Tessier et al. [31], the elements can be divided into five classes. The first element fraction contains water-soluble elements (exchangeable), where changes in ionic composition of water affect sorption and desorption mechanisms. The second element fraction contains elements that are mainly bound as carbonates and are soluble in (mild) acids. The solubility of this element fraction is especially affected by changes in pH. The third element fraction is mainly bound to Fe and Mn (oxyhydr)oxide minerals and soluble under reducing conditions. This means that compounds are unstable under anoxic conditions. The fourth element fraction is mainly bound to organic matter and soluble under oxidizing conditions. The residual fraction consists mainly of primary and secondary minerals. Elements are not expected to be released from the crystal structure over a reasonable time span under conditions as encountered in nature.

As shown in Table 1, the mobility of elements has been increasingly studied in recent years. The materials investigated vary widely and include wastes from mining and metallurgical processes, different types of ash from the combustion of sewage sludge, MSWI, and biomass, as well as contaminated soils. Research in this field has often focused on the mobility of metals within these matrices in order to assess their environmental relevance. The leaching procedures applied are based on different principles and standards and are frequently adapted to the specific medium under investigation.

Tests on element extraction of wood ash were carried out in a number of leaching experiments. Previous studies investigated element leaching from wood ash under different test conditions such as sequential extractions, batch leaching tests and percolation tests [23, 32–38] Other studies investigated influencing factors on element solubility, such as pH [39] and ash pre-treatment through ageing or so-called hardening or weathering [40,41]. Ageing has been shown to both reduce and enhance the leaching of specific elements from the hardened ash. For example, Ca, Ba, Pb, Zn were found to be less mobile after ash ageing, while the mobility of As, P, Sb, Se. Mg increased after hardening [40,41]. The pH of the surrounding environment, e. g., during storage, utilization or disposal, has been found to have a major influence on element mobility. Ash is typically alkaline, and under high-pH conditions, many constituents remain relatively immobile. Acidification increased aqueous concentrations of B, Ca, Cd, Co, Cu, Mg, Mn, Ni, Sr, and Zn, whereas Cr, Hg and Mo showed decreased concentrations [39]. However, the results are difficult to compare due to varying extraction conditions.

Maresca et al. [20] point out that a consistent assessment of the leaching properties of ashes is essential as a foundation for a future regulatory framework aimed at reducing the landfilling of wood ashes. Ash reuse and utilization is associated with potential environmental risks related to mobilization of undesired elements in ecosystems. Still, there is little information available in the current literature on biomass ash leaching properties [50].

As shown in Table 1, leaching studies to date have predominantly focused on the mobility of heavy metals, with strong emphasis on environmental risks and, to a lesser extent, on the potential for element recovery. While these aspects are crucial for assessing the environmental

Table 1
Overview of recent studies on element mobility in various waste types and biomass sources, including applied extraction procedures.

Sources	Authors	Focus	Leaching procedure
Incinerated sewage sludge ash (ISSA)	Li et al., 2017 [42]	Metal mobility in residual ISSA after chemical P extraction	Synthetic precipitation leaching procedure (SPLP, US EPA Method 1312), European Council Waste Acceptance Criteria (ECWAC)
Metallurgical waste	Mizerna 2016 [43]	Metal mobility in zinc slag and copper slag	EN 12457:2002 (CEN)
Processed municipal solid waste incineration residues	Abramov et al., 2018 [44]	Heavy metal mobility and valuable contents	Modified four step BCR sequential extraction method
Biomass bottom ash (from wheat straw, corn straw, and forestry waste)	You et al., 2024 [45]	Chemical speciation, leaching behaviour and environmental risk assessment of ash forming elements	Five step sequential extraction following Tessier
Waste wood fly ash and Municipal solid waste fly ash	Wolffers et al., 2021 [46]	Heavy metal recovery	FLUWA process
Municipal solid waste incineration ash	Oehmig 2021 [47]	Trace element mobility before and after plasma vitrification	Toxicity characteristic leaching procedure TCLP (EPA Method 1311), SPLP (EPA Method 1312)
Industrial and hazardous incineration ashes	Kasina et al., 2024 [48]	Element fractionation and leaching potential and environmental implications	Three step BCR sequential extraction
Contaminated soil	Vollprecht et al., 2024 [49]	Heavy metal mobility before and after thermal soil treatment	pH-dependent leaching tests according to EN 14997

implications of ash utilization, comparatively little attention has been directed toward the presence, transformation, and bioavailability of plant-relevant nutrients in ash. This knowledge gap is particularly relevant, as nutrient recovery could provide an important secondary resource for sustainable agriculture and contribute to circular economy strategies. Therefore, a comprehensive understanding of both nutrient and metal contents, together with their mobility and bioavailability in wood ash, is essential to fully evaluate the risks and opportunities associated with its utilization. Therefore, the leaching approach is considered appropriate, in addition to the assessment of total element concentrations, for the evaluation of biomass ash utilization options to provide a more realistic assessment of element availability.

1.3. Aim of the study

This study aimed to determine the elemental composition of wood ash from a plant <1 MW and to determine the solubility of the ash-forming elements. A sequential extraction protocol using four solvents (water, acetic acid, hydroxylamine hydrochloride, and ammonium acetate with hydrogen peroxide) was applied to assess the solubility of ash-forming elements. Element solubility was examined from two perspectives: (1) the potential for elements to leach under environmentally relevant conditions, and (2) the possibility of recovering valuable or harmful elements through defined extraction steps. The results are to be used to discuss utilization options of the ash with respect to regulatory requirements.

2. Material and methods

2.1. Ash origin and sample collection

The investigated ash originates from a combustion plant (PYROT rotary furnace 100–540 kW, KOB, Viessmann Group GmbH & Co. KG, Allendorf (Eder), Germany) in southern Germany, which is fired exclusively with natural forest wood chips. Three ash samples were taken individually from the plant's ash collection container on three days in November 2022. All samples were analyzed in the laboratory of the Rottenburg University of Applied Forest Sciences (Germany).

2.2. Sample preparation and analytics

Three ash samples were collected after leaving the plant in water-free state and were not ground for the extraction experiments. On average, 74 wt.-% of the three single samples were finer than 0.5 mm, 12 wt.-% between 0.5 and 1 mm and 14 wt.-% > 1 mm.

Sequential extraction according to the BCR (Community Bureau of Reference) method was used to simulate the dynamic processes in soils [51,52]. Following Tong et al. [53], the standard BCR protocol was adapted for highly alkaline ash, with the acetic acid extraction step repeated to offset the influence of alkaline components. Compared to alternative leaching methods, the adapted BCR protocol was selected because it represents a multi-stage process simulating four environmental conditions (water-soluble, acid-soluble, reducible, and oxidizable phases). In contrast, DIN EN 12457–4:2003–01 [54] is a single-stage leaching test using only water, and TCLP [55] employs acetic acid and sodium hydroxide to simulate landfill conditions. Thus, the adapted BCR provides a more comprehensive assessment of element mobility under varying environmental scenarios.

The extraction procedure was carried out in quadruplicate for each ash sample as described in Ref. [30]. In addition to the extractable element fractions, the total concentration of elements in the ash was determined by microwave-assisted aqua regia digestion in quadruplicate (Multi-wave GO 3000, Anton Paar, Graz, Austria). Aqua regia digestates and leachates from sequential extraction were measured with inductively-coupled optical emission spectroscopy (ICP-OES) following [56–59] (Spectro Blue, ASX-260 Auto-Sampler, SPECTRO Analytical Instruments, Kleve, Germany).

Multi-element ICP standard solutions (ROTI Star and ROTI Star IV, Carl Roth GmbH + Co. KG, Karlsruhe, Germany) were used for both calibration and as a standard reference material. Calibration curves for the 26 elements analyzed in this study were prepared using the following concentration levels: 0 (blank), 0.001, 0.01, 0.1, 1, 10, 25, 50, 100, 500, and 1000 ppm. Accuracy of the calibration was checked using SRM check standards at 0.05, 0.5, 4, and 15 ppm, with recoveries ranging from 95 to 105 %. Repeatability was assessed by analyzing triplicate measurements from each sample, yielding relative standard deviations (RSD%) below 5 % for all elements. The limit of detection (LOD) for all elements was determined to be 0.001 ppm.

Five sequential extraction steps were carried out (Fig. 1). The exposure time for each solvent was 16 h (GFL 3005 orbital shaker, GFL Gesellschaft für Labortechnik, Burgwedel, Germany at 350 rpm, Appendix B). After each extraction step, the solid and liquid phases were separated by centrifugation (Rotofix 32 A, Hettich GmbH & Co. KG, Tuttlingen, Germany, 20 min at 4000 rpm). The leachates were decanted (Appendix C) and pH was determined (WTW ProfiLine pH3110 Set 2 pocket pH meter incl. Sentix 41 pH electrode, WTW, Weilheim, Germany). The remaining solids were further treated with the following extraction agent according to the extraction protocol. The decanted leachates were stabilized with 0.2 ml HNO_3 before analysis by ICP-OES.

First, 1 g of ash was treated with 40 ml of deionized water (H_2O).

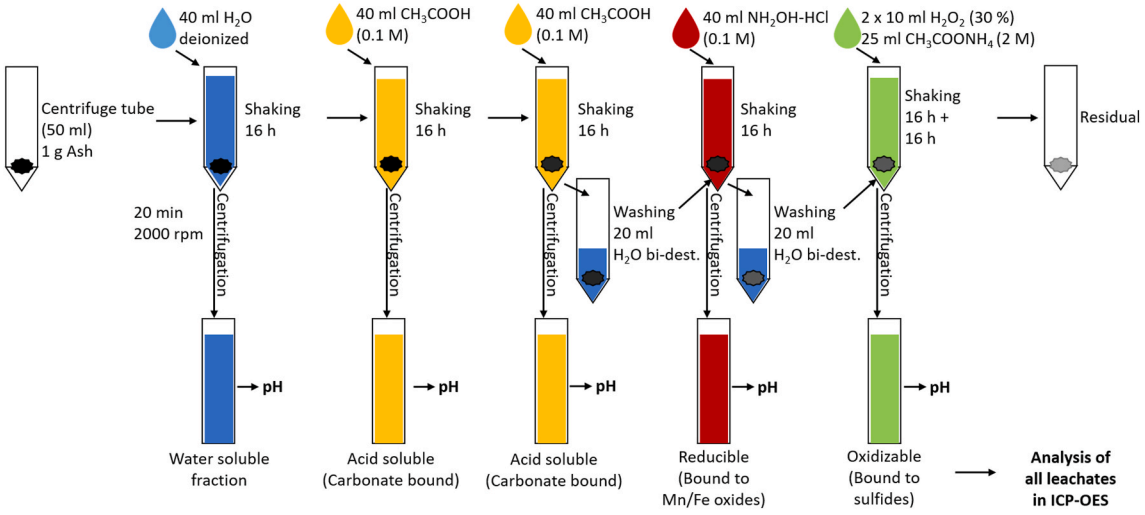

Fig. 1. Sequential extraction protocol [51–53].

Then, the remaining solid after centrifugation and separation of the water-based leachate was treated with 40 ml of acetic acid (CH_3COOH, 0.1 M, pH 2.8). This step was carried out twice due to the high alkalinity of ash. The remaining solid was then treated with 40 ml of NH_2OH-HCl (0.1 M, pH 3.4) as reduction agent. The vessels were closed during the extraction in water, acid and reducing agent. The last extraction step under oxidizing conditions was carried out in open vessels. The oxidizing agents were added in steps to avoid violent reactions. 20 ml H_2O_2 (30 %) were added in 2 steps of 10 ml each, with a waiting time of approx. 10 min in between. After an exposure time of 16 h, 25 ml CH_3COONH_4 (2 M, pH 6.9) was added to the mixture of ash and H_2O_2 for another 16 h exposure time. Before the reduction and oxidation step, the solid was washed with H_2O bi-dest. for 20 min (GFL 3005 orbital shaker, GFL Gesellschaft für Labortechnik, Burgwedel, Germany at 350 rpm). The solids were centrifuged again and the wash water was discarded.

2.3. Data evaluation

The total element content was determined by analysis of the aqua regia digestate. The measured concentration of an element i was defined as 100 % (Equation (1)). This value serves as a reference value for the solubility of elements in the extraction steps. Theoretically, the total element content as concentration in mg kg^{-1} in the ash is calculated as the sum of the fractions that can be extracted in the individual steps and the residual content that cannot be washed out. The concentration of an element i in the residual fraction $c_{i,\ res}$ (i. e., in the remaining solid after all extraction steps) is calculated from the initial concentration $c_{i,\ total}$ minus the sum of the concentrations of the element i in leachates across all extraction steps (Equation (2)). The extractable fraction of an element i (EF_i) describes the sum of the element quantities washed out in the extraction steps $c_{i\ \sum extr}$ in relation to the total element concentration $c_{i,\ total}$ (Equation (3)).

$$\text{Total element concentration } c_{i,\ total} = c_{i,\ aqua\ regia} = 100\ \% \quad (1)$$

with $c_{i,\ aqua\ regia}$ defined as element concentration measured after digestion in aqua regia

$$\text{Element concentration in the residual fraction } c_{i,\ res} = c_{i,\ total} - c_{i\ \sum extr} \quad (2)$$

with $c_{i\ \sum extr}$ defined as sum of the element quantities washed out in the extraction steps

$$\text{Extractable fraction of an element i } EF_i = c_{i\ \sum extr} / c_{i,\ total} \quad (3)$$

For error analysis, the statistical mean, standard deviation, and 95 % confidence interval (Equation (4)) were calculated for the measured values.

$$\text{Confidence Interval CI} = \bar{x} \pm z^* \frac{\sigma}{\sqrt{n}} \quad (4)$$

with $\bar{x}$ defined as the sample mean, σ defined as the standard deviation, z defined as the z-score corresponding to the chosen confidence level (95 %), and n defined as the sample size.

3. Results and discussion

3.1. Elemental composition of the ash

Elements in solid fuels and ashes can be divided into three groups, depending on their concentration, major elements (>1 wt% or > 10,000 mg kg^{-1}), minor elements (0.01–1 wt% or 100-10,000 mg kg^{-1}) and trace elements (<0.01 wt% or < 100 mg kg^{-1}) [60]. Fig. 2 shows the elemental concentrations in the ash samples investigated in this work. In total, 26 elements were analyzed via ICP-OES. In the order of magnitude of major elements, Al, Ba, Ca, Fe, K, Mg, Mn, Na, P were detected. In the order of magnitude of minor elements, B, Cr, Cu, Li, Ni, Pb, Sr, Ti, Zn were found and in the order of magnitude of trace elements, As, Bi, Cd, Co, Mo, Se, Tl, V are to mention, whereby Cd was below the detection limit.

The measured ash composition is comparable to values reported in the literature [2,7]. However, the content of potentially critical elements such as As, Cd, Ni, Pb, Tl and Zn, for which limit values e.g. from the German Fertilizer Ordinance also exist, can vary considerably. This applies both to the variation within the multiple determination of one sample as can be seen from Fig. 2 (heterogeneity of ash from one plant) and to ash from different plants as reported by Bachmaier et al. [7]. Possible sources of these fluctuating levels of potential pollutants are the fuel itself and any contamination from fuel processing or the plant.

In previous studies on ash characterisation and ash management options [6,7], as well as in the regulatory framework [21], the elemental composition of the ash is the decisive criterion for possibilities of ash utilization, e.g. as fertilizing material. In addition to the total element content, the solubility of the elements from the ash and thus their environmental mobility or bioavailability are the key properties for a sensible and safe utilization of the ash in the sense of a circular economy.

3.2. Soluble element fractions under different conditions

Fig. 3 shows the initial element concentrations in the analyzed ash and the distribution of the dissolved elements across sequential

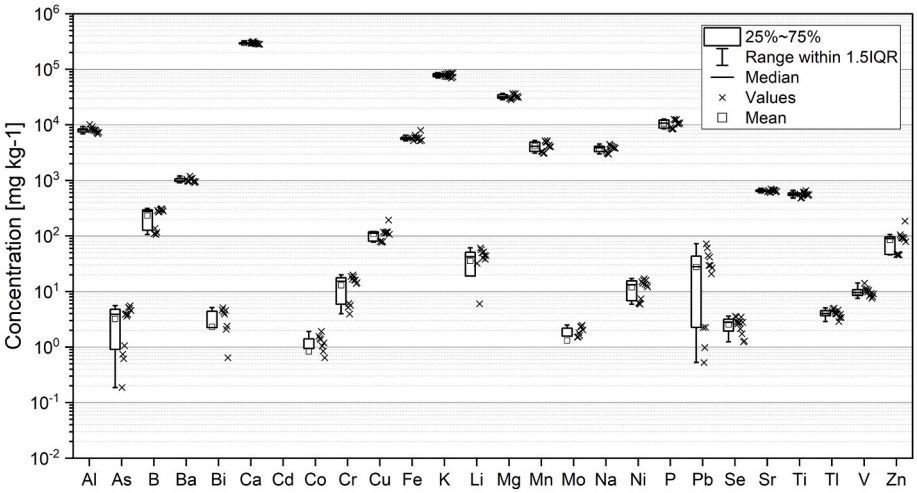

Fig. 2. Element concentrations in wood ash from a grate-fired furnace <1 MW using natural wood as a fuel (n = 12). Boxplots with 25 % and 75 % quantiles (box). Outliers were determined at 1.5 interquartile range (whisker). Individual measurements are marked with 'x'.

extraction steps: (I) water, (I) acetic acid, (III) hydroxylamine hydrochloride, and (IV) ammonium acetate with hydrogen peroxide. The solubility of elements varies considerably, both overall (across all extraction steps) and in relation to the various extraction conditions. The element solubility across all extraction steps can be measured as the extractable fraction of an element i (EF_i, Equation (3))

Highly soluble elements across all extraction steps are S (92 % $_{EF}$), K (74 % $_{EF}$), and Sr (54 % $_{EF}$). As moderately soluble elements across all extraction steps, B (48 % $_{EF}$), V (44 % $_{EF}$), Mo (43 % $_{EF}$), Na (41 % $_{EF}$), Mn (41 % $_{EF}$), Cd (38 % $_{EF}$), Mg (37 % $_{EF}$), Se (37 % $_{EF}$), Ba (33 % $_{EF}$), and Cr (30 % $_{EF}$) and Tl (27 % $_{EF}$) can be categorized. Poorly soluble elements are As (19 % $_{EF}$), Ni (17 % $_{EF}$), P (15 % $_{EF}$), Pb (14 % $_{EF}$), Cu (14 % $_{EF}$), Li (13 % $_{EF}$), Ca (12 % $_{EF}$), Co (11 % $_{EF}$), Zn (7 % $_{EF}$), as well as Fe, Ti, Al, and Bi (≈0 % $_{EF}$).

The extraction conditions during the sequential extraction procedure range from strongly alkaline to slightly acidic, depending on the extraction step. Due to the high alkaline contents in ash, very basic conditions are found in the water based leachate (pH 12.6 ± 0.1). The use of acetic acid lowers the pH value of the leachates, down to pH 8.8 ± 0.7 after the first acid step, and pH 6.8 ± 0.9 after the second step. Leachates from the reduction (pH 6.3 ± 0.2) and oxidizing stages (pH 7.3 ± 0.1) show slightly acidic to neutral conditions.

It can be assumed that the elements that can be mobilized in the sequential extraction by water, acid, reducing and oxidizing agents, are also leachable in the environment under natural conditions to a certain extent. The residual fraction is considered to be leachable only in the long term and therefore has a low environmental mobility or bioavailability [30]. According to their leachability in the respective solvents, the elements can be categorized as primarily water soluble (K, Mo), acid soluble (Ca, Mg, B, Sr, Li), reducible (Pb, Se, As, Co, Tl, V) or oxidizable (Zn, V).

The measured values from the series of experiments on solubility using sequential extraction were statistically evaluated. The table in Appendix A (supplementary material) shows the concentrations in the initial material, in the leachates from the various extraction steps and in the residual material after washing for each element examined. The dispersion of the mean values of the measurements (n = 12) was quantified using the standard deviation. In addition, a 95 % CI was calculated for each mean value. This interval indicates the range of values in which the actual mean value of the population lies with 95 % probability.

3.3. Utilization options of ash and leachates in the context of legal limits

As discussed in Mayer et al. [6] and as can be seen from Fig. 3, ash from wood combustion contains plant nutrients. However, the ash contains also potentially hazardous elements such as heavy metals. A safe utilization as fertilizing material is therefore difficult to guarantee. Leaching results as presented in this work help to gain a better understanding of the potential for elements to escape under environmentally relevant conditions. When ash is landfilled, valuable raw materials are lost from the utilization circle. It has been estimated [6] that ashes from wood-fired heat and power plants in southwestern Germany could substitute the need for fertilizer raw materials in the state of Baden-Wuerttemberg (3.1 % of P, 7.5 % of K and 22.8 % of Ca fertilizer currently used). In order to utilize this potential, adequate processing methods to separate nutrients from potentially harmful elements are required. The tests carried out in this study on the leachability of the ash-forming elements are a first step towards the development of such a process.

The concentrations of selected elements in the untreated ash (Initial), the loaded leachates (water soluble, acid soluble, reducible, oxidizable) and the leached solid material (residual) are shown in Figs. 4 and 5. In order to evaluate the element mobility from the untreated ash as well as the effect of a possible ash treatment through leaching, the values are set in relation to legal limit values.

Firstly, the content of plant nutrients in the ash and in the extraction products (leachates) is assessed. Fig. 4 shows the measured concentrations of Ca, K and P calculated as CaO, K_2O and P_2O_5 as a percentage of the minimum required concentration in a commercial solid mineral compound fertilizer defined in the German Fertilizer Ordinance [21]. The legally defined minimum contents are indicated as 100 %.

It should be noted that the comparison to legally defined minimum nutrient contents in commercially available fertilizers only serves as a point of reference for the quality of the ash and the extraction products. The concentrations were measured in the untreated leachtes. The levels of nutrient content could e. g. easily be increased by evaporation.

The initial content of CaO in the untreated ash is high, exceeding the legally required content in fertilizers by more than tenfold. Despite this high initial content, the overall leachability of CaO is low, with the majority remaining in the residual fraction. This indicates low environmental mobility. Calcium is therefore only potentially bioavailable in the long term, primarily through washing out under acidic conditions. K_2O is also present in the ash at levels more than ten times higher than the legal requirement. In contrast to CaO, potassium exhibits high mobility, with only a small proportion retained in the residual fraction.

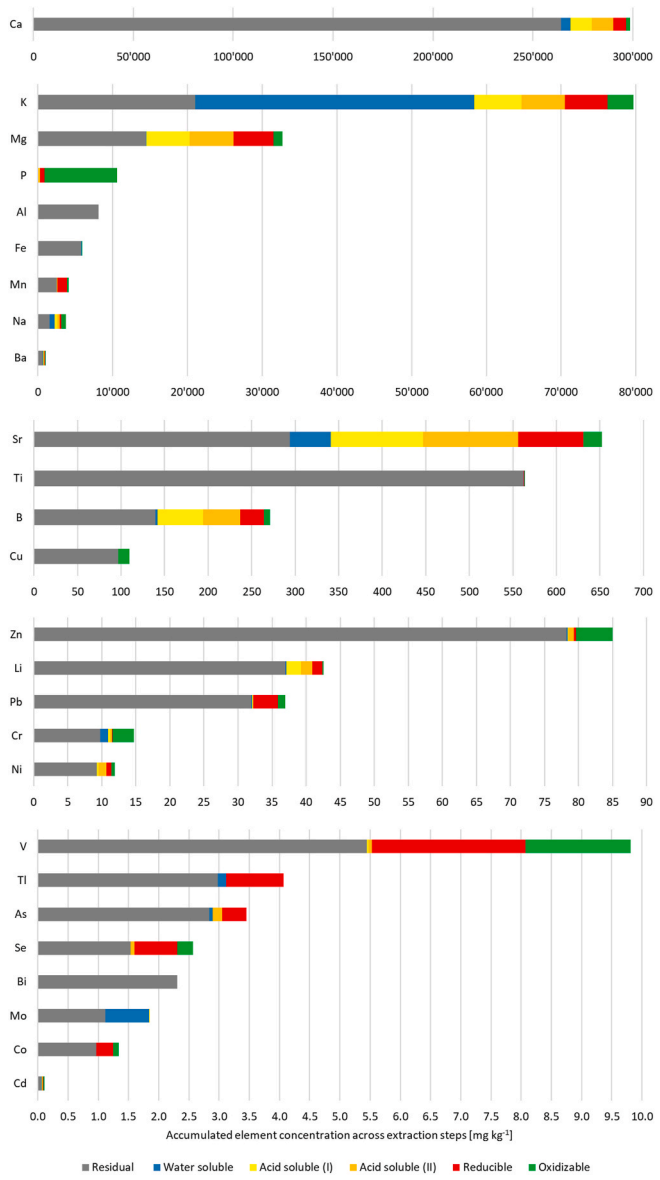

Fig. 3. Element concentration in the analyzed ash and distribution of the dissolved elements across sequential extraction steps. Dissolved concentrations [mg kg^{-1}] in aqueous (Water), acidic (acetic acid in two steps), reducing and oxidizing conditions. Calculated concentration remaining in the solid phase after extraction (residual fraction).

Due to its predominantly water-soluble form, K_2O is readily bioavailable. Additional fractions can also be mobilized under acidic, reducing, and oxidizing conditions.The initial concentration of P_2O_5 in untreated ash is comparable to the legal threshold for fertilizers. However, the total leachability of phosphorus is low, with most of it remaining in the residual fraction. Significant mobilization of phosphorus was only observed under oxidizing conditions, suggesting limited short-term availability in natural soil environments.

Secondly, the content of metals in the ash and in the extraction products is assessed. Fig. 5 shows the measured concentrations of As, Cd, Ni, Pb, Tl and Zn as a percentage of the maximum allowed concentration according to the German Fertilizer Ordinance [21]. The legally defined maximum contents are indicated as 100 %.

The initial content of As, Cd, Ni, Pb and Zn is low. For As, Ni and Pb, the concentration in the raw ash is approx. 10 % of the permitted maximum content. For Zn, the concentration is well below 10 % of the permitted maximum, with the exception of one outlier. The concentration of Cd in the initial material is below the detection limit.

Due to the low concentrations in the starting material, the concentrations of extracted elements in the individual leachates are also relatively low. The concentrations of As, Cd, Ni and Zn in all leachates (water, acid, reducing and oxidizing agents) are in the range of 1 % or well below 1 % of the limit value.

Pb could be mobilized from the ash especially under reducing and oxidizing conditions, leading to concentrations in the range of 1–5 % of the limit value in the reducible and oxidizable leachate fractions. In the aqueous and acidic solutions, Pb was hardly detectable.

Tl is the only element from this list of potentially hazardous ash constituents whose concentration in the source material (initial) exceeds the values permitted by the Fertilizer Ordinance. The Tl content of the analyzed ash is approx. 300 % of the permitted value. This relatively high concentration in ash from untreated wood can be due to various factors, e. g. the environmental conditions during the growth of the biomass (pollution of soil, water or air), contamination during fuel preparation or in the combustion plant itself (metal abrasion). As shown in chapter 3.2, Tl is a very mobile element with a high leachability (100 % of initial element content can be washed out). The highest proportion of the soluble fraction can be leached under reducing conditions. As a result, a relatively high Tl concentration which corresponds to appoximately 100 % of the limit value is found after leaching in the reducing agent. The Tl concentration in all other leachates (pregnant water, acid and oxidizing leaching agents) remains below the legal limit.

Thallium pollution resulting from anthropogenic activities is an increasing environmental concern. Elevated Tl concentrations are commonly observed in soils affected by mining, but they can also arise from the use of fertilisers and from by-products of energy production. Tl is considered highly toxic because it can substitute for potassium in biological processes, thereby disrupting essential metabolic functions. In addition, Tl exhibits particularly high mobility and bioavailability, which further increase its risk towards (agricultural) ecosystems and groundwater [61–63]. Current methods for Tl removal from (waste) water include adsorption, redox-driven precipitation, solvent extraction, and ion exchange [64]. Due to the significant environmental and health implications of Tl contamination, there is an increasing focus on researching Tl remediation [65–67].

In summary, with the exception of the wash water after the reduction step, all extraction liquids are harmless with regard to the content of As, Cd, Ni, Pb, Tl and Zn. Plant nutrients can be released from the ash matrix by washing. Most of the Ca remains in the residual fraction after extraction (88 %). But due to the high concentration in the starting material, considerable amounts of Ca are still found in the leachates, mainly mobilized by acid (amounting to 100 % of the legal requirement). Potassium is more mobile and readily available to plants, with only 26 % remaining in the residual fraction. The highest share of K is water soluble, resulting in >100 % of the legally required concentration for K fertilizer in the aqueous leachate. Phosphorus is very difficult to mobilize in the first stages of extraction. Acids and reducing agents contain <10 % of the amount expected in fertilisers by law. P can be specifically released from the ash by oxidation, resulting in >10.000 % of the legally required concentration for P fertilizers in the leachate after oxidation. As a result, no P is left in the residual fraction.

Results show that oxidation effectively mobilizes P from wood ash, which can be attributed to the chemical forms in which P is stabilized within the ash. In wood ash, P is often present in stable mineral forms such as Ca-, K- or Mg-phosphates. These minerals can exist as discrete phosphate minerals (e.g., $Ca_3(PO_4)_2$) or be occluded within Al- and Fe-oxides [68,69]. A possible formation route during combustion and flue gas cleaning is that organically bound P from the fuel is assumed to create reactive P(V)oxide, subsequently forming Calcium phosphate with the Ca abundant in wood and wood ash [70]. Such phosphate compounds are generally resistant to dissolution under mild acidic or reducing conditions but can be released under oxidizing conditions [71]. Under these conditions, associated organic matter and mineral matrices

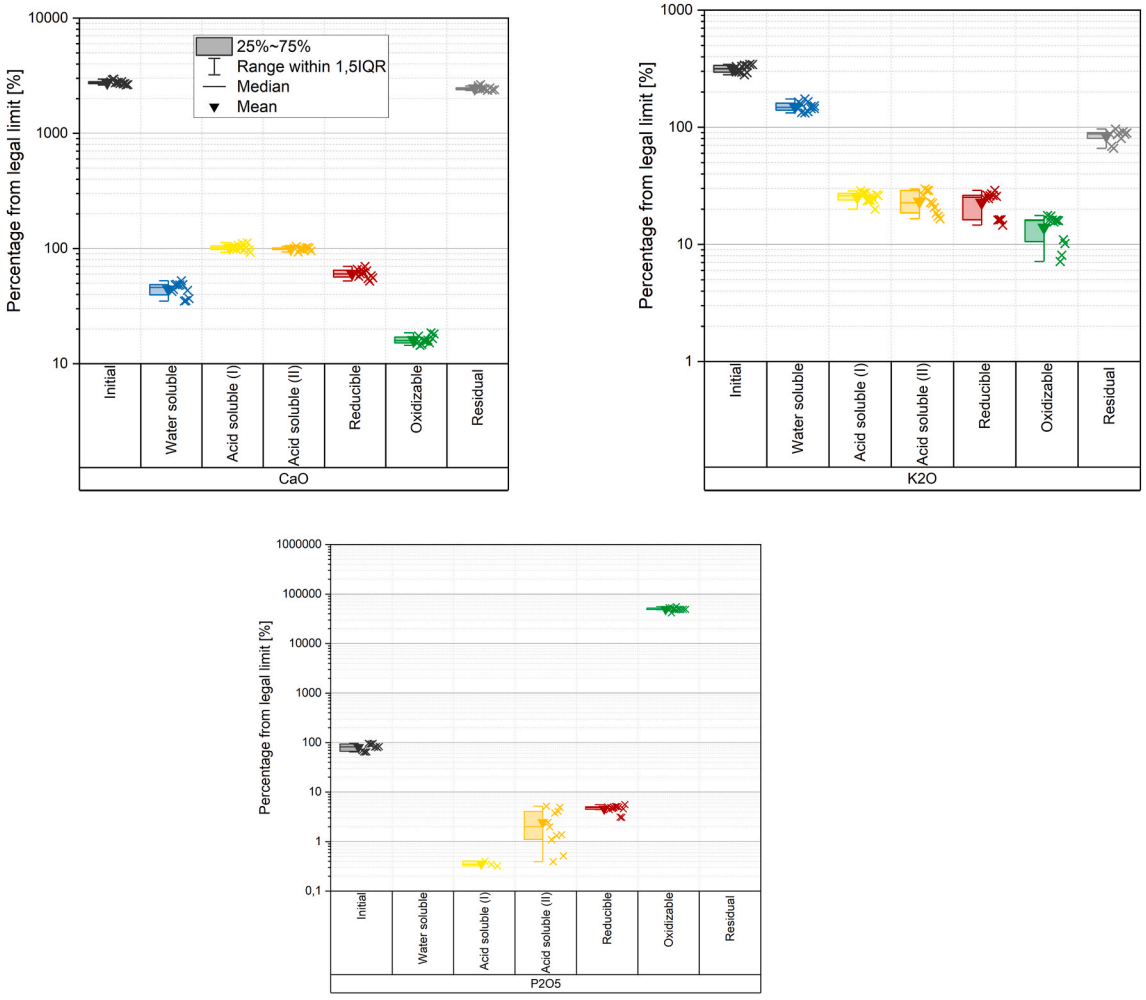

Fig. 4. Concentrations of Ca, K and P shown as CaO, K_2O and P_2O_5 calculated as percentage of the minimum required concentration according to the German Fertilizer Ordinance [21] for commercial solid mineral compound fertilizers and lime fertilizer. The legally defined minimum contents are indicated as 100 %. Boxplots with 25 % and 75 % quantiles (box) and outliers determined at 1.5 interquartile range (whisker). Individual measurements are marked with 'x'.

are degraded, liberating P previously bound within these fractions. Phosphate compounds are not readily bioavailable, e.g. to plants. Similar as it has been found for sewage sludge ash [72], direct land application would be limited in fertilization value and further processing is necessary.

Lab results cannot be transferred directly into field conditions. For example, element mobility is influenced by soil conditions such as pH and redox potential, which are linked to factors such as water availability and soil organic matter content [73–75]. Soil acidification, for example, can significantly increase the mobility of potentially hazardous metals such as Cd, Zn and Pb contained in soil [75]. The transferability to environmental scenarios needs to be evaluated in a separate study.

With respect to environmental risk management, the concentrations of metals such as As, Cd, Ni, Pb, and Zn in the initial wood ash can generally be regarded as uncritical, as they amount to only about 10 % of the legally defined maximum limits. The only exception is Tl, which has already been discussed in detail due to its higher environmental relevance. However, these study only reports on one specific case and does not represent a cross-section of wood ash from different plants. The actual metal content of ash depends strongly on the specific fuel used, as well as on combustion conditions and flue gas cleaning technologies. Continuous quality control is therefore essential to ensure that the ash does not exhibit elevated concentrations of potentially hazardous metals. In addition to the absolute metal content, the mobility of potential contaminants represents a crucial factor for environmental risk assessment. The results of the sequential extraction indicate relatively high mobilities under natural conditions for several elements, including Sr (54 % EF), B (48 % EF), V (44 % EF), Cd (38 % EF), and Tl (27 % EF). Despite the low total concentrations in the raw material, this mobility poses a potential risk for the release and dispersion of these elements into the environment. Consequently, environmental management strategies should not only consider total concentrations but also take into account the leaching potential and long-term behavior of these elements in soils and aqueous systems.

Regarding fertiliser formulation, the safe use of ash in fertilizer production requires the prior removal of potential contaminants, particularly toxic metals such as Tl. Furthermore, the plant availability of nutrients is a key criterion in the design of ash-based fertilizers. The results demonstrate that macronutrients such as Ca and K can be partially dissolved in water and weak organic acids, indicating that they are already relatively plant-available in the untreated ash. Still, a proportion of Ca and K remains in the residual fraction after sequential extraction and is considered to be plant available only in the long term. P, by contrast, can only be released from the ash matrix under oxidizing conditions, as discussed above. Therefore, pretreatment of the ash is necessary to increase the plant-available fraction of P.

From the perspective of waste management policy, the processing and valorization of ash offer a promising opportunity to reduce overall waste volumes and promote the recovery of secondary raw materials. Ash fractions that are currently unsuitable for material use due to

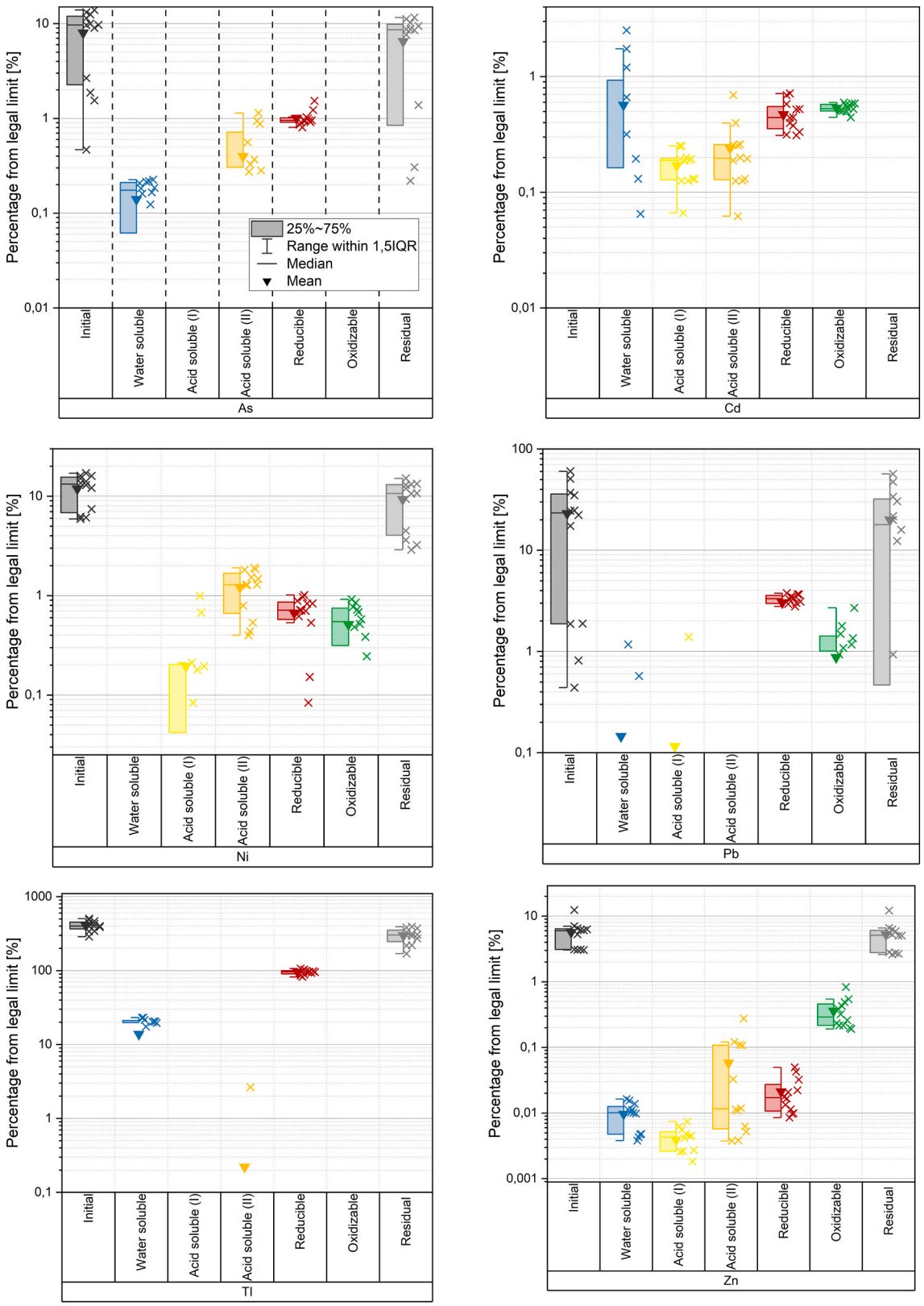

Fig. 5. Concentrations of As, Cd, Ni, Pb, Tl and Zn calculated as percentage of the maximum allowed concentration according to the German Fertilizer Ordinance [21]. The legally defined maximum contents are indicated as 100 %. Boxplots with 25 % and 75 % quantiles (box) and outliers determined at 1.5 interquartile range (whisker). Individual measurements are marked with 'x'.

elevated metal contents could become viable resources after appropriate treatment. This approach, however, requires a more differentiated assessment of the material ash itself. To effectively transform waste into a secondary raw material, waste management strategies should not only consider the total elemental composition but also the mobility of critical elements and the potential for their recovery through leaching or extraction processes. Such an integrated approach would support both environmental protection and circular economy objectives by facilitating the sustainable and safe utilization of ash residues.

Future studies should broaden the scope by analyzing wood ash from multiple plants and diverse fuel types to improve the generalizability of findings on composition and element mobility. Investigations are also needed to assess the transferability of laboratory results to field conditions, considering soil properties, hydrology, and long-term environmental dynamics. Additionally, research should explore targeted recovery of valuable elements from ash leachates, including nutrients and critical metals, to support sustainable resource utilization. Expanding these research horizons will enhance understanding of ash behavior, inform environmental risk management, and promote the development of integrated strategies for safe and circular use of wood ash.

4. Conclusion

In this study, the elemental composition of wood ash from a plant <1 MW and the solubility of the ash-forming elements in water, acetic acid, reducing and oxidizing agents were determined by experimental investigations. The results are used to discuss utilization options of the ash with respect to regulatory requirements.

Concerning the elemental composition, both plant nutrients (Ca, K, P) and and potential pollutants (As, Cd, Ni, Pb, Tl, Zn) are present in the ash in the expected concentrations. The individual ash-forming elements show a wide range of leachability, which is defined as the ratio of all leachable elements to the concentration in the starting material.

Comparing the analytical results to legal requirements allows a classification of the ash quality with regard to possible use as a fertilizer. Some of the plant nutrients are hardly leachable. A high proportion of Ca and K remains in the residual fraction after the extraction and is considered to be plant available only in the long term. Due to the high contents in the starting material, the extraction liquids nevertheless contain Ca and K in concentration ranges comparable to commercially traded fertilizer. The critical element P is initially difficult to mobilize under aqueous, acidic and reducing conditions. However, P can be dissolved from the ash matrix through oxidation. The environmentally critical elements show a wide range of leachability, from 7 % (Zn) to 100 % (Tl). The only element present in the ash in critical concentration is Tl.

Sequential extraction results highlight that several elements (e.g., Sr, B, V, Cd, Tl) exhibit relatively high mobility, indicating potential environmental risks despite low total concentrations. Therefore, environmental assessments and management strategies should address not only total metal contents but also leaching behavior and long-term stability in soils and aqueous systems.

However, it must be noted that laboratory results on ash behavior cannot be directly transferred to field conditions, as element mobility is strongly influenced by soil parameters such as pH, redox potential, water availability, and organic matter content.

For fertilizer applications, pretreatment to remove contaminants, especially Tl, and to enhance the plant-available fractions of nutrients, particularly P, is required. Macronutrients such as Ca and K are already partly soluble and thus readily available to plants.

From a waste management perspective, ash processing and valorization can reduce waste and recover secondary resources. Integrated strategies considering composition, mobility, and recovery potential are essential to ensure the environmentally safe and sustainable utilization of ash residues.

CRediT authorship contribution statement

Johanna Eichermüller: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Visualization, Writing – original draft. **Vanessa Heilmann:** Investigation, Writing – original draft. **Andreas Kappler:** Conceptualization, Supervision, Writing – review & editing. **Harald Thorwarth:** Conceptualization, Funding acquisition, Supervision, Writing – review & editing.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.biombioe.2025.108527.

Data availability

Data will be made available on request.

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