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Recovery of potassium and phosphorus from biomass-derived ash

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ABSTRACT

Sustainable fertilizer production is crucial for society, and ensuring food security remains a global challenge. Virgin mining still dominates the production of potassium and phosphorus fertilizers. Exploiting secondary sources, in particular those associated with waste to energy plants and biomass refining, is an attractive alternative and complement for fertilizer production. This study is an in-depth material characterization of seventeen types of ash from the incineration of nutrient-containing biostock. This provides a novel and extensive comparison of ash outputs from a variety of industrial processes. Ash samples after the incineration of poultry litter, fish litter, and from the Kraft process were studied. Total elemental analysis, XRD, TGA, SEM/EDS, leaching and leachate titration investigations were done to identify the most promising materials for recycling potassium and phosphorus. Poultry litter fly ash was identified as a promising secondary source of nutrients due to its phosphorus (3.0 wt%) and high potassium content (24.4 wt%). Efficient and selective separation of potassium from phosphorus was possible using water leaching at ambient temperature. This generated an alkaline solution with sodium, potassium, chlorides, sulphates ions, and very low heavy metal content; and a solid residue in which phosphorus was up-concentrated to 11.3 wt%. We briefly outline processes described in the literature to separate salts from the aqueous leachate and produce potassium fertilizers; and methods to process the phosphorus enriched solid. These offer more sustainable routes to produce fertilizers by exploiting secondary sources and allow for nutrient circularity in the industries that generate these materials.

1. Introduction

Potassium, phosphorus, and nitrogen are the principal elements for sustaining agricultural productivity and ecosystem equilibrium. This is due to their role in enhancing soil health, optimizing plant nutrition, and ensuring global food security. Current production of fertilizers is through the production of ammonia from air and processing of large amounts of phosphorus- and potassium-containing ores, neither of which are sustainable long-term approaches (Manning, 2010; Öborn et al., 2005; Ushakova et al., 2023).

No substitutes exist for potassium and phosphorus as essential plant nutrients and in nutrition for animals and humans (U.S. Geological Survey, 2023). Increased population and food scarcity have led to an increasing demand of fertilizers. In the EU Critical raw materials report from 2023, phosphate rock was deemed critical due to its high economic importance and supply risk (European Commission, 2023). Potash (mined and manufactured salts containing potassium in water-soluble form) was not classified as critical despite its high economic importance, due to a lower supply risk.

Potassium and phosphorus mines and deposits are not evenly distributed globally (al Rawashdeh and Maxwell, 2014). Most of the world potash reserves are concentrated in three countries: Canada, Russia, and Belarus, whereas the largest consumers are China, Latin America, North America, and India. Deficits of potassium production in densely populated areas have been reported, e.g., in India, Africa (Ciceri et al., 2015; Manning, 2010) and Europe (Öborn et al., 2005). The EU is highly dependent on imports of phosphorus fertilizers due to the lack of significant phosphate rock deposits within Europe (Grohol and Veeh, 2023). A more localized production of fertilizers can be enabled by processing nutrient-containing secondary sources, including wastes that are currently under-utilized or disposed. This promotes sustainability by decreasing the need for virgin phosphorus mining and intercontinental

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transportation of fertilizers. Reclaiming nutrients like potassium and phosphorus from secondary sources can partly overcome some concerns associated with virgin mining, e.g., salinization of surface water and groundwater, contamination with hazardous metals, landscape degradation and land disturbance, and increased local releases of radionuclides and particulate matter (Ushakova et al., 2023).

There are multiple streams containing nutrients, foremost those linked to bioprocesses e.g. food, animal, pulp and paper, forestry (Zhai et al., 2021). Their potential is not always exploited, and several such streams are discarded due to organic or heavy metals contamination. Incineration of biomass, sewage sludge, animal manure, or animal by-products, holds significant promise for energy and nutrient recovery. This is due to their high caloric value, reduction in moisture content and volume after incineration and up-concentration of nutrients in the resulting ash (Billen et al., 2015). While direct application of such ash (e.g., spreading on arable land) is straightforward, this comes with drawbacks. Such ash generally has a high pH and contains other soluble constituents apart from nutrients (Kan et al., 2024). The presence of heavy metals above regulatory limits often prevents their direct use as fertilizer (Luyckx et al., 2020). Chemical processing including decontamination allows for a better use of the nutrient value and enables production of pure fertilizers with well-defined chemistry. Furthermore, this limits the addition of insoluble ash constituents without nutritional value to soils, and of chlorides that increase salinity.

In contrast with the recovery of precious or technology metals from secondary sources (e.g., processing of electronic wastes to recover gold and platinum-group metals (PGMs), copper, cobalt, nickel, lithium, rare earths, tantalum etc.), the recovery of nutrients has not received as much attention. While there are studies on the recovery of phosphorus from sludges and sludge ash (Chen et al., 2024; Fang et al., 2018; Mochiyama, 2019, Adam et al., 2009; Guedes et al., 2014; Atienza–Martínez et al., 2014; Patents US20240117462A1, US8425872B,US10329153B2 and EP2137101B1), wastewaters (Ichihashi and Hirooka, 2012; Li et al., 2024; Moulessehoul et al., 2024; Yetilmezsoy and Sapci-Zengin, 2009; Zhang et al., 2022), food waste (Han et al., 2024; Huang et al., 2022; Li and Li, 2017), waste acids (Jang et al., 2023; Kim, 2006; Park et al., 2006; Shibata et al., 2003), or manure wastewater and ash (Cempa et al., 2022; Qaramaleki et al., 2023), the recovery of potassium nutrients is less documented (Atsbha et al., 2025; Cohen, 2018; Pan et al., 2024). From an EU perspective, part of the driving force behind the efforts to recover nutrients is the rise of mandatory legislation that targets recovery of phosphorus from sewage sludge (EU Directive 2024/3019, 2024). This will be in force in Germany in 2029 (AbfKlärV, The German Sewage Sludge Ordinance,2017), with Austria (AVV, 2024 AVV, 2024. In: A.A,2024) and Switzerland (ADWO, 2014) having similar legislation with other deadlines. However, the regulations do not yet mandate processing of other nutrient-containing wastes or recovery of nitrogen and potassium. For example, poultry manure, a combination of poultry droppings, feathers and bedding material, has high caloric value and generates a nutrient-rich stream after incineration (Cempa et al., 2022; Faridullah et al., 2009). It is estimated that worldwide poultry meat production will increase by 15 % from 2023 to 2032 (OECD/FAO, 2023). With the increase in production, the amount of poultry litter will also increase, which presents a favourable opportunity to use this material as an alternative source for fertilizer production. Direct usage, without incineration, can lead to emissions of greenhouse gases such as methane and ammonia and increased possibility of water contamination and eutrophication (Dróżdż et al., 2020). Incineration eliminates biological hazards (pathogens) and odour issues and reduces the volumes of waste and greenhouse gas emission potential (Billen et al., 2015). Incineration of such wastes in a fluidized bed reactor is well-described (Luyckx et al., 2020; Billen et al., 2015). This generates several ash fractions (bottom, boiler and fly ash) in which the nutrients distribute. The bottom ash is collected under the fluidized bed and contains a significant amount of sand from the fluidized bed. The boiler ash contains lighter particles carried by the flue gas from the fluidized bed, as well as solids after condensation and solidification of evaporated salts. This is typically obtained separately from the bottom ash. Fine fly ash particles carried by the flue gas are recovered in the flue gas cleaning system, the bulk being collected by electrostatic precipitators. There are studies that highlight the advantages of using fly ash for stabilizing heavy metals in ash after municipal solid waste incineration (Fahimi et al., 2020) or as supplementary cementitious material (Castillo et al., 2022).

Since potassium is an integral constituent of plants, processing and incineration of plant bio-stock, or side streams from pulp and paper industries also generate ash with potassium (Zhai et al., 2021). Ash with 4–20 wt% chlorine and potassium is obtained in the Kraft process (method for producing paper pulp by cooking wood chips) and can be potentially used as alternative source to produce nutrients. Extraction of potassium from pulp and paper ash is not driven by the recovery of nutrients but to avoid buildup of potassium chloride. Chlorine and potassium harm operation (fouling and corrosion of heat transfer tubes in recovery boilers and lowers the melting temperature of fly ash), and therefore their bleed from recirculated streams is desired (Tran and Earl, 2004). Cleaning of potassium chloride from sodium sulphate ash in the Kraft process is documented (Begley and Anthony Pecoraro Thomas, 2016; Ebner and Ebner, 1989) and commercial units based on selective leaching and crystallization are available (Tran and Arakawa, 2001; Knutsson et al., 2002; Ferreira et al., 2003).

The present study focuses on material characterization and leaching techniques to achieve selective nutrient recovery from ash linked to bioprocesses. Seventeen samples, including various ash fractions after the incineration of poultry litter, streams from the Kraft process, and fish litter and ensilage, are compared. Total element analysis, combined with leaching and titration studies, and characterization methods such as X-ray Diffraction (XRD), Thermalgravimetric analysis (TGA) and Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy (SEM/EDS) were used to characterize and assess the potential for selective recovery of potassium and phosphorus. This provides a clear overview of how these different materials compare against each other, and which of these are the most promising secondary sources for nutrients. Applicable methods for the separation and production of potassium and phosphorus fertilizers described in the literature are briefly discussed for the most promising waste stream investigated (poultry litter ash). We show that industrially relevant concentrations of potassium (e.g., tens of g/L) can be leached from all types of poultry litter ash (fly, bottom and boiler ash). There are very few papers available on the characterization and resource recovery from poultry litter ash. Characterization studies (Fahimi et al., 2020; Luyckx et al., 2020) focus on the production of ash (the incineration process) and the physical and chemical properties of various poultry litter ash fractions; and do not address resource recovery or provide comparisons with ash from other industries. Studies on nutrient recovery from poultry litter ash target phosphorus but do not focus on its recovery as a chemical compound. Instead, emphasis is placed on using the ash as fertilizer after conditioning it with acid to make the insoluble phosphates more plant available (Kan et al., 2024). The research presented here contributes to the development of more circular and sustainable fertilizer systems by addressing some of this missing information.

2. Materials and methods

2.1. Ash samples and chemicals

Seventeen samples were screened (Table 1; also see Figure S1 in supplementary material). Samples marked PL are ash after the incineration of primarily poultry litter, samples marked FL originate from fish litter and fish ensilage, and samples named KP originate from the Kraft process. Samples PL-F1, PL-Bt1 and PL-Bo1 come from the same facility, from a traveling grate boiler that was fed with poultry litter mixed with < 25 wt% plant residues. Samples PL-F2, PL-Bt2 and PL-Bo2 are ash after the incineration of poultry litter in a fluidized bed incinerator and

Table 1
Ash samples screened.

Place of origin	Ash	Type	Incinerator type	Main source	Physical characteristics
A	PL-F1	Fly ash	Traveling grate boiler	Poultry litter **	Homogenous white-grey, low-density, dry
B	PL-F2	Fly ash	Bubbling fluidized bed	Poultry litter **	Homogenous white-grey, low-density, dry
C	PL-F3	Fly ash*	Fluidized bed	Poultry litter	Homogenous white-grey, low-density, dry
C	PL-F4	Fly ash	Fluidized bed	Poultry litter	Homogenous white-grey, low-density, dry
A	PL-Bt1	Bottom ash	Traveling grate boiler	Poultry litter **	Nonhomogeneous, wet, larger clumps, brown
B	PL-Bt2	Bottom ash	Bubbling fluidized bed	Poultry litter **	Nonhomogeneous, large stones, black, white and grey, wet
C	PL-Bt3	Bottom ash	Fluidized bed	Poultry litter	Nonhomogeneous, mix of small and larger stones, light to dark grey, dry
A	PL-Bo1	Boiler ash	Traveling grate boiler	Poultry litter **	Nonhomogeneous, black and grey mix, hydrostatic, dry
B	PL-Bo2	Boiler ash	Bubbling fluidized bed	Poultry litter **	Nonhomogeneous, black and grey mix, hydrostatic, dry
C	PL-Bo3	Boiler ash	Fluidized bed	Poultry litter	Homogenous, brown, low-density, dry
Various	KP−1	Fly ash	Boiler	Kraft process	Homogenous, brown, clumps, dry
	KP−2	Fly ash	Boiler	Kraft process	Homogenous, white, small particles, low density, dry
	KP−3	Mixture	Traveling grate boiler	Kraft process	Homogenous black, low-density, wet
	KP−4	Fly ash	Traveling grate boiler	Kraft process	Homogenous white, low-density, dry
	KP−5	Fly ash	Unknown	Kraft process	Homogenous, dark grey, small particles, low density, dry
	FL−1	Mixture	Conventional oven	Fish litter and ensilage	Homogenous, dark grey, small particles, low density, dry
	FL−2	Mixture	Conventional oven	Fish litter and ensilage	Homogenous, dark grey, small particles, low density, dry

* Mixed with reaction products and unreacted lime after lime dosing for acid removal from the flue gas ** Less than 25 wt% addition of plant residue

are also mixed with plant residue. Samples PL-F3, PL-F4, PL-Bt3 and PL-Bo3 are ash after the incineration of poultry litter (no addition of plant matter) in a fluidized bed incinerator at a different facility. Samples KP-1 to KP-5 originate from various types of incinerators and are connected to the Kraft process and incineration of plant constituents used in the Kraft process. Of these, KP-3 and KP-4 are mixtures of different fractions with high carbon content (incomplete combustion). Sample FL-1 and FL-2 are ash after incineration of fish sludge (fish litter and fish ensilage) in conventional ovens at approximately 900 ºC.

Samples PL-Bt1, PL-Bt2, KP-3, KP-4 were received wet and were dried at 105 ºC in a conventional oven prior to investigations. Deionized water was used in all the leaching tests. 1 M HCl solution (Merck Millipore Titrisol) was used in the titration experiments.

2.2. Characterization techniques and analytical procedures

For total elemental analysis, the ash samples underwent microwave-assisted digestion in a mixture of nitric acid, hydrochloric acid, and hydrofluoric acid according to standard SS-EN13656:2003. The total element content in the resulting solution was measured with Inductively Coupled Plasma Sector Field Mass Spectrometry (ICP-SFMS; Thermo Scientific Element XR). Spectrophotometric determination of total nitrogen was performed according to the Kjeldahl method (CSN ISO 11261). Determination of the total content of bound nitrogen (TN_b) after oxidation to nitrogen oxides was measured based on EN ISO 20236. The halogen (F, Cl, and Br) content in the ash was measured according to standard SS-EN 15408:2011. SEM observations were performed utilizing a Hitachi FlexSEM 1000-II with an acceleration voltage set at 5–20 kV, a spot size of 20–50 nA, and a working distance of 5–10 mm and a secondary electron detector. For EDS, an acceleration voltage of 20 kV, a spot size of 50 nA, and a working distance of 10 mm were employed using the Aztec software. TGA was performed with a Perkin-Elmer Pyris-1 instrument in air atmosphere. X-ray Diffraction analysis was done using a Bruker D8 Advance system using a scan time of 30 min. per sample and interpretation of the data was done using the EVA software and the ICDD database. Quantification of elements in ash leachates was done using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES; Agilent 5800 VDV). A Thermo Scientific Orion 9617BNWP Ionplus Chloride Electrode was used to measure the aqueous chloride concentrations. pH measurements and titration of the leachates were done using an automatic titrator (Mettler Toledo Titrando 888). 20 ml of each leachate was titrated with 1 M HCl solution (Merck Millipore Titrisol) to pH < 3.

2.3. Leaching

Batch leaching tests were designed based on the CEN EN 12457–2 standard. For every ash, 10 g sample was mixed with 100 ml deionized water in a glass beaker using magnetic stirring. A temperature sensor attached to the device was used to control and monitor the temperature of the slurry during leaching. Leaching was performed at 20 °C for 30 min. The slurry was filtered through Munktell Grade 3 filter paper using a conventional vacuum filtration setup. The undissolved residue was washed with 100 ml deionized water (20 °C). The residue was dried in an oven at 105 °C and weighed.

To study the up-concentration of potassium in solution using sequential leaching, 30 g of PL-F1, PL-Bt1 and PL-Bo1 ash were first leached with 90 ml of deionized water for 60 min. at 20 °C and liquid:solid (L:S) ratio of 3:1 based on weight (w/w). After filtration, each leachate was used to leach fresh ash using similar operating conditions. A third leaching was performed for samples PL-Bt1 and PL-Bo1 using the leachates produced in the second leaching step.

To study the dissolution speed of water-soluble elements, sample PL-F1 was leached with deionized water for 120 min. at 30 °C and L:S 3:1 w/w (150 ml water and 50 g ash). During this time, several aliquots (1–2 ml) were collected, filtered using 0.45 μm PVDF syringe filters and analysed.

The dissolution behaviour of ash PL-F1 at different L:S ratios were investigated by leaching this ash with the corresponding amount of water for 60 min. at 20 °C and L:S 3:1 w/w. The L:S ratios investigated were in the range 1:1 – 10:1 w/w.

3. Theory/calculation

The ash mass loss was calculated using Eq. (1):

$$Ash\ mass\ loss(wt.\%) = \frac{m_{residue}\quad (g) * \quad DS_{residue}(\%)}{m_{ash}\quad (g)} \tag{1}$$

Where the m_{ash} is the weight of the ash used for the experiment, $m_{residue}$ is the weight of the ash residue after leaching, and $DS_{residue}(\%)$ is the dry solid content of this residue (measured by weighing the residue before and after drying at 105 °C).

4. Results and discussion

4.1. Elemental composition

The elemental composition of the ash samples is given in Table 2 (major elements), Table 3 (minor elements) and Table S3 in the supplementary material (total nitrogen, halogens and carbons)

Poultry litter ash consists of complex mixtures of various mineral relics, neo formed crystals, amorphous inorganic phases and char (Fahimi et al., 2020). The ash samples are rich in potassium (6.5–24.5 wt%), phosphorus (3.0–8.9 wt%), and calcium (8.0–23.6 wt %). These values are similar to what those reported in other studies. For example, ash with 0.2–20 wt% potassium and phosphorus levels of 0.25–7.6 wt% are reported by (Fahimi et al., 2020), and ash with 9.4–15.5 wt% potassium and 2.3–10.9 wt% phosphorus is described in (Luyckx et al., 2020). The fly ash samples have the highest potassium content (13–24.5 wt%), followed by the boiler (9.3–12 wt%) and bottom ash samples (6.5–11.2 wt%). This is due to the volatilization of potassium chloride at the temperatures used in the incineration process (Tran and Earl, 2004), which leads to an up-concentration of potassium salts in the light (fly) ash fraction. As a result, collection of fly ash towards the end of the flue gas treatment unit will generate more concentrated potassium fractions. In contrast, phosphorus compounds were up-concentrated in the boiler ash compared to the fly or bottom ash.

Ash PL-F1 and PL-F2 have higher sodium content compared to other fly ash samples, which is due to the co-incineration of poultry litter with plant materials (more sodium-rich). Other macro-elements commonly found in the poultry litter samples were aluminium (0.25–2.3 wt%), chloride (0.9–10 wt%), iron (0.4–1.7 wt%), sodium (1.1–11.2 wt%), magnesium (2.2–5.1 wt%), sulphur (0.6–7.8 wt%), and silica (1.7–14.6 wt%). The major heavy metals were copper (230–640 mg/kg), manganese (1900–3500 mg/kg) and zinc (900–3900 mg/kg). Mercury was below detection limit in all poultry litter ash, while arsenic and cadmium were present in trace amounts, generally < 10 mg/kg. The presence of heavy metals emphasizes the downside of directly applying the material to soil.

The Kraft process produces ash with diverse compositions. Ash KP-2 contains low amounts of nutrients (1.3 wt% phosphorus and 0.2 wt% potassium) and rich in calcium carbonate. Ash KP-4 has high amounts of sodium, sulphur, and potassium. This is a sodium sulphate and carbonate ash which also contains potassium (5.3 wt%) and chlorides (1.1 wt%). Other ash samples from the Kraft and papermaking industries have noticeable concentrations of potassium (4.8–7.5 wt%), phosphorus (0.9–1.5 wt%) and especially calcium (18.0–20.2 wt%).

Ash FL-1 and FL-2, derived from fish and fish ensilage, have high calcium content. The nutrient concentrations varied between the two ash samples, with FL-2 containing potassium and phosphorus levels approximately 10 and 4 times higher, respectively, compared to FL-1. These differences are likely attributed to variations in the sampling points of the sources and differences in the combustion processes.

All ash samples contain low concentrations of nitrogen (< 1000 mg/kg, Table S3 in supplementary material), which is typical for such incineration processes due to the high volatility and gaseous nature of nitrogen compounds. However, poultry litter boiler ash (PL-Bt1, PL-Bt2 and PL-Bt3) has the highest nitrogen concentration, likely due to an incomplete combustion of the fraction analysed here. This material also had a characteristic odour supporting this. The contained carbon is primarily in inorganic form. A few of the ash are rich in carbonates, and this is further addressed in section 4.3.

4.2. Leaching experiments

The analysis results from the water leaching experiments at L:S ratio 10:1 w/w are presented in Table 4. All leachates were alkaline (pH>11.5). The high pH is mainly caused by elements such as calcium, sodium and potassium occurring as chlorides and calcium oxide, the latter generating alkaline calcium hydroxide in contact with water. Calcium oxide is present in ash from the decomposition of calcium carbonate in the sand bed (which decomposes at higher temperatures) and from the injection of lime in the flue as cleaning process.

Due to their higher soluble potassium content, poultry litter fly ash materials generated leachates with significantly higher potassium concentrations compared to their bottom and boiler poultry litter ash counterparts. Aqueous potassium concentrations as high as 23,000 mg/L were obtained at L:S ratio 10:1 w/w (sample PL-F1). As further discussed in 4.5, more concentrated solutions can be prepared using a L:S ratio that is more relevant for industrial operations, e.g., L:S 3:1, which allows for good stirring and solid-liquid separation using conventional filters. The high leaching efficiencies seen over 30 min. also indicates that the kinetics are favourable for industrial applications, especially for continuous processes where long residence times are undesirable (also see Figure S7 in supplementary material). The leaching selectivity of potassium over the other nutrient element, phosphorus, was excellent. Low aqueous concentrations of phosphorus (< 12 mg/L) were measured in all solutions. The absence of water-soluble ammonium phosphates (Table 4; very low concentration of nitrogen and phosphorus in the leachates), and the high alkalinity of the ash-water slurry keeps the phosphates in the solid fraction as compounds with low water solubility.

Table 2
Concentrations of major elements in the ash (typical concentrations of > 1 g/kg ash).

Ash/Element (g/kg dry)	K	P	Al	Ca	Cl	Fe	Na	Mn	Mg	S	Si	Zn
PL-F1	244	30.2	2.53	80.4	100	4.11	67.4	1.94	22.4	77.3	17.1	3.88
PL-F2	172	48.3	3.06	115	65.6	5.65	112	2.37	28.6	39.9	36.0	2.66
PL-F3	129	77.5	20.0	193	18.7	8.7	24.4	3.10	48.5	16.6	72.8	2.35
PL-F4	217	65.2	15.8	114	67.4	4.56	27.1	2.50	36.0	41.6	42.8	3.12
PL-Bt1	64.7	32.2	13.6	236	5.5	16.6	11.6	1.99	30.1	6.13	87.7	0.95
PL-Bt2	112	57.4	5.03	206	25.6	6.03	25.2	2.92	31.5	21.8	124	2.34
PL-Bt3	114	44.4	9.30	143	14.3	6.05	16.2	1.98	28.6	27.9	146	1.50
PL-Bo1	119	67.0	7.78	192	5.40	5.65	22.4	3.80	46.8	16.0	92.9	1.78
PL-Bo2	93.7	55.8	4.66	181	5.80	7.36	17.7	2.45	26.4	26.6	127	1.71
PL-Bo3	120	89.0	22.6	171	9.0	8.70	25.8	3.45	51.8	13.1	65.1	2.78
KP−1	48.4	15.8	54.0	187	3.1	5.20	9.93	12.4	29.4	22.9	93.7	2.55
KP−2	2.27	12.6	0.56	385	1	54.0	12.7	0.75	6.75	2.90	1.49	0.26
KP−3	75.4	10.6	4.31	202	< 0.1	0.51	0.70	14.4	16.8	9.91	4.40	3.48
KP−4	53.3	< 0.05	0.02	< 0.1	10.9	5.32	277	0.10	0.10	157	< 0.4	0.07
KP−5	60.8	9.2	37.2	185	11.8	0.02	6.83	2.71	23.1	14.6	132	5.24
FL−1	5.84	15.7	5.21	314	< 0.1	37.2	14.2	1.56	20.0	5.15	13.7	0.81
FL−2	57.2	63.9	16.4	197	< 0.1	16.4	22.2	1.5	30.7	6.87	128	0.9

Table 3
Concentrations of minor elements in the ash samples (typical concentrations of < 1 g/kg ash). For all samples, beryllium was measured as below 2 mg/kg, cobalt and tungsten below 10 mg/kg, niobium and yttrium below 20 mg/kg and vanadium below 60 mg/kg. Their concentrations can be found in the supplementary material Table S1.

Ash/Element (mg/kg dry)	As	Ba	Cd	Cr	Cu	Hg	Mo	Ni	Pb	Sb	Sc	Sn	Sr	Ti
PL-F1	5.47	71.1	2.51	30.4	411	< 0.05	31.3	25.5	6.27	0.54	1.08	1.03	141	184
PL-F2	8.26	91.1	3.32	30.4	412	< 0.05	22.3	35.1	9.53	1.08	1.38	0.56	249	178
PL-F3	< 3	177	1.87	37.5	570	< 0.05	26	29.9	9.31	0.56	1.37	0.74	296	446
PL-F4	3.11	112	3.26	22.2	632	< 0.05	34.2	30.7	16.1	0.67	1.41	0.67	198	379
PL-Bt1	< 3	119	0.16	64.4	232	< 0.05	13	36	2.59	0.49	2.59	113	283	633
PL-Bt2	4.47	120	0.40	33.6	429	< 0.05	24.2	44.1	3.76	0.83	1.09	0.56	349	275
PL-Bt3	3.28	138	< 0.05	19.3	363	< 0.05	19.1	21.7	1.97	0.36	1.25	0.88	223	296
PL-Bo1	< 3	153	0.32	67.7	457	< 0.05	30.1	46.4	1.8	0.45	2.05	2.07	279	526
PL-Bo2	4.81	93.5	< 0.05	25.6	244	< 0.05	19.7	42.1	1.08	0.68	< 1	0.76	356	249
PL-Bo3	< 3	181	1.88	21.3	624	< 0.05	21.3	40.7	10.3	0.52	1.65	0.89	301	606
KP−1	3.88	2950	20.7	101	92.1	0.35	7.21	55.0	42.9	1.63	2.11	6.86	786	716
KP−2	< 3	833	1.23	18.9	1.02	< 0.05	< 0.5	8.31	< 1	< 0.05	< 1	0.22	460	72.7
KP−3	< 3	2770	8.24	25.6	84.4	0.06	0.72	18.1	15	0.64	< 1	0.32	769	46.5
KP−4	< 3	3.36	3.82	< 1	< 1	< 0.05	1.31	< 2	3.55	0.15	< 1	< 0.2	< 2	< 2
KP−5	13.2	5290	18.9	143	474	0.05	6.13	90.8	700	21.3	6.13	40.4	901	7400
FL−1	10.1	254	0.35	70.4	404	< 0.05	17.9	45.7	169	3.86	1.92	12	815	1210
FL−2	11.1	225	0.67	81.2	297	0.06	13.3	41	92.8	81.6	4.19	7.22	924	1070

Table 4
Concentration of the leachates and pH (25 ± 2 °C, L:S 10:1 w/w), and ash mass loss (Eq. 1). For all samples magnesium, strontium, and lead was measured to be below 10 mg/kg, arsenic, cadmium, chromium was measured below 1 mg/kg; iron and molybdenum below 3 mg/kg; and silicon was below 60 mg/kg. Their concentrations can be found in the supplementary material Table S2. Blanked cells (-): no measurement performed.

Element	Ash mass loss (wt%)	pH	K (mg/L)	P (mg/L)	Ca (mg/L)	Na (mg/L)	S (mg/L)	Cu (mg/L)	Zn (mg/L)	Cl (mg/L)	N (mg/L)
PL-F1	70	11.7	22,500	1.76	79.2	5400	8000	0.22	0.66	9800	3.25
PL-F2	54	12.8	14,550	11.4	56.1	9700	4050	< 0.05	0.30	6710	6.3
PL-F3	16	12.8	5,400	0.09	1190	360	1500	< 0.05	1.68	1900	2.36
PL-F4	37	13.0	14,950	0.04	690	950	4000	< 0.05	6.47	5720	1.67
PL-Bt1	13	12.6	5070	1.5	12.0	710	1050	1.94	0.07	610	49.1
PL-Bt2	19	12.4	7930	0.06	135	980	2700	< 0.05	< 0.05	2510	3.04
PL-Bt3	20	12.8	7980	< 0.05	490	520	2820	< 0.05	< 0.05	1530	1.81
PL-Bo1	12	12.9	3940	0.39	1230	320	1450	< 0.05	0.24	496	2.98
PL-Bo2	13	13.0	4680	< 0.05	1270	560	1940	< 0.05	0.14	529	1.56
PL-Bo3	12	13.0	3820	< 0.05	860	280	1080	< 0.05	0.14	860	1.88
KP−1	24	12.8	2970	< 0.05	44.0	530	830	< 0.05	0.1	171	2.5
KP−2	2	12.1	81.0	< 0.05	32.0	410	230	< 0.05	0.09	14.0	4.06
KP−3	75	12.5	530	< 0.05	190	4.03	3	< 0.05	0.20	20.7	-
KP−4	100	11.6	4800	2.6	24.0	29,000	1730	< 0.05	0.61	1000	8.84
KP−5	11	12.9	3030	< 0.05	1500	110	980	1.1	10.4	1160	17.8
FL−2	7	12.5	1100	2.3	660	63	1000	< 0.05	0.46	-	-

The high ash mass loss of the fly ash, e.g., 70 wt% mass loss for PL-F1, leads to a significant up-concentration of phosphorus in the undissolved ash residue. This is further discussed in 4.5.2. The leaching selectivity of potassium over other elements apart from sodium, chloride and sulphur was also high, with many ‘impurities’ being reverted to, and up-concentrated in the undissolved residue. Very low concentrations of heavy metals were leached alongside potassium. The aqueous concentrations of arsenic and cadmium were below the detection limit of the instrument (< 0.1 and < 0.5 mg/L, respectively). Only sub-mg/L amounts of copper and zinc were detected in poultry litter ash leachates.

Ash KP-4 (Kraft process) dissolved almost completely in water and has a lower potassium and much higher sodium content compared to poultry litter fly ash. In contrast, ash KP-2 showed very low ash mass loss which can be explained by the low water solubility of its calcium constituents. Ash FL-2 also had low ash mass loss in water.

4.3. Alkalinity of the ash leachates

To determine the predominant alkaline species, the leachates were titrated with 1 M HCl solution (Fig. 1). A typical titration curve for carbonates presents two inflection points: the first at pH 8.3, where the carbonate compound reacts with acid to form a hydrogen carbonate (Eq. 2) and the second at pH 3.7, where the hydrogen carbonate reacts, producing CO_2 (Eq. 3). Titration of alkaline metal hydroxides (NaOH and KOH) (Eq. 4) results in a curve with one inflexion point at neutral pH.

Samples PL-F2 and KP-4 consumed most acid and the curves showed two inflection points at pH 8.3 and pH 3.7, indicating the presence of a higher concentration of soluble carbonates compared all other samples. The presence of carbonates was confirmed by the generation of gas (carbon dioxide bubbling out of the solution) during titration. In this case, production of potassium fertilizers from such ash needs to consider the presence of carbonate species alongside sulphate and chloride. Addition of hydrochloric or sulphuric acid will remove carbonates present in solution. The other ash leachates had lower alkalinity and contained less carbonates, as seen in Fig. 1.

$$CO_3^{2-} + H^+ \rightarrow HCO_3^- \tag{2}$$

$$HCO_3^- + H^+ \rightarrow CO_2 + H_2O \tag{3}$$

$$MOH + H^+ \rightarrow M^+ + H_2O \tag{4}$$

The ash mass loss vs. the potassium content in the leachate for every ash is mapped in Fig. 2. The size of each dot is proportional to the

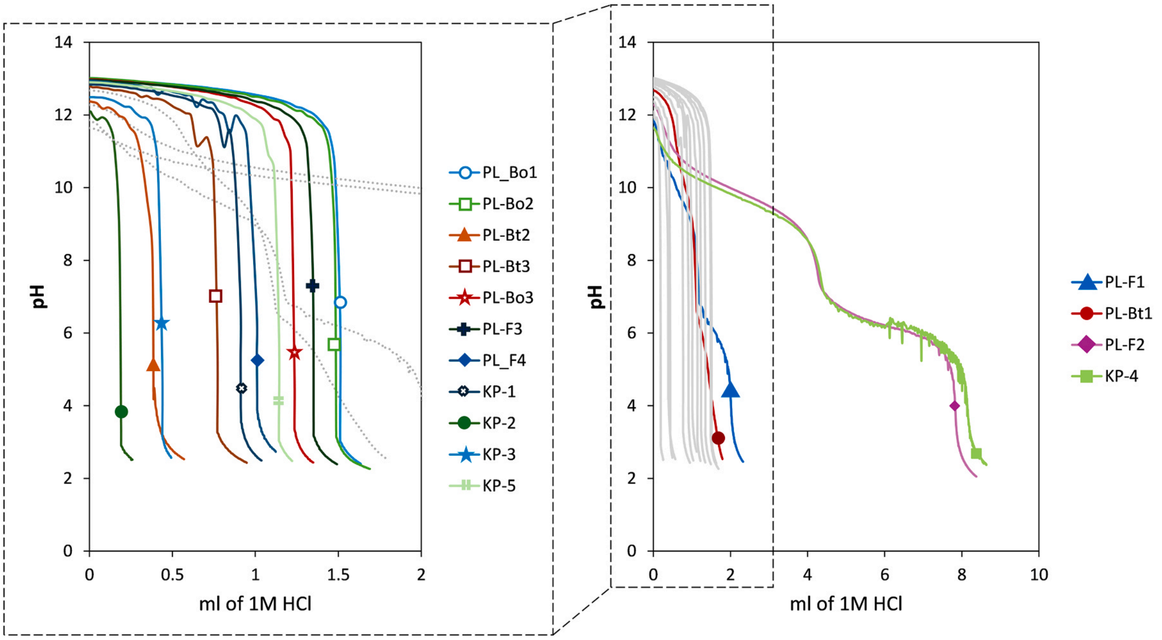


Fig. 1. Titration curves for leachates produced by leaching ash with water at L:S ratio of 10:1 w/w. 20 ml leachate was titrated with 1 M HCl.

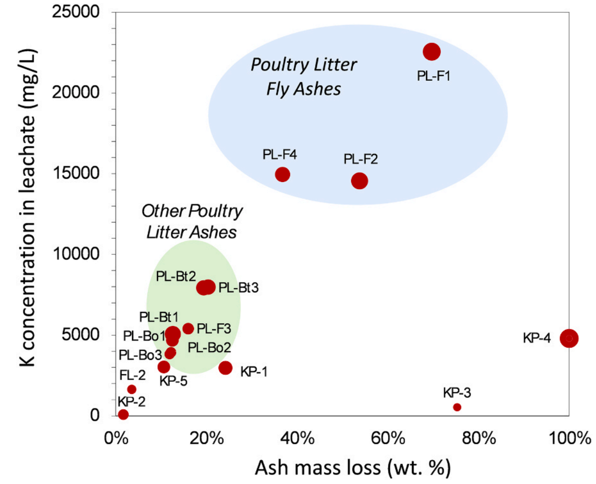


Fig. 2. Potassium concentration in ash leachates (obtained from leaching at L:S ratio 10:1 w/w) vs. the ash mass loss. Ash samples at the top right corner of the graph are most attractive for nutrient recovery (highest water-soluble potassium content and least amount of solid residue generated). The dot size is proportional to potassium leaching efficiency.

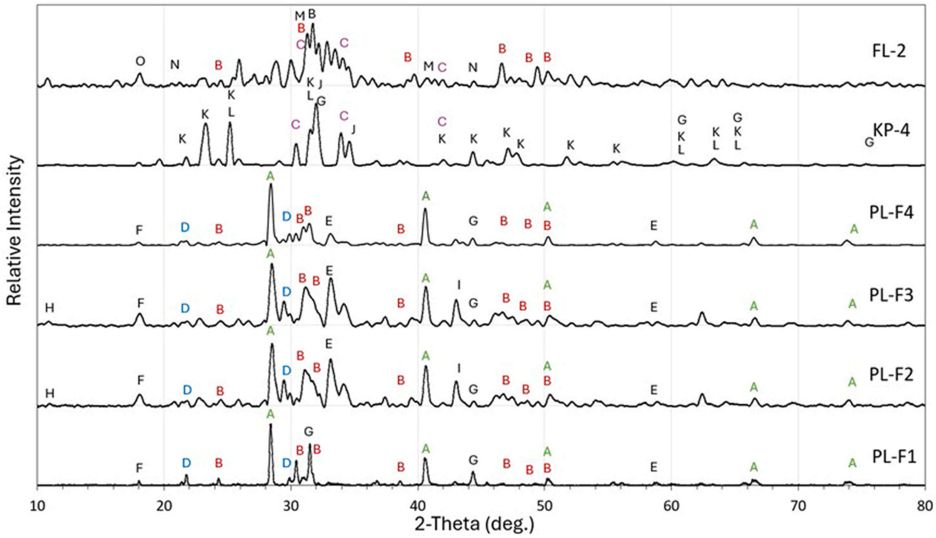
leaching efficiency of potassium (% potassium dissolved, calculated from data in Table 2 and Table 4, leaching efficiency = potassium concentration in leachate * L:S ratio / potassium concentration in ash). Ash with larger markers positioned closer to the top right corner of the graph indicate better choice for nutrient recovery. The poultry litter fly ash outputs appear to be the most promising materials among those studied, followed by ash KP-4. These ash samples were investigated more in detail with SEM, EDS, XRD and TGA analysis, presented in 4.4. Bottom and boiler poultry litter ash also exhibit similar characteristics and are grouped together in the graph.

4.4. XRD, TGA and SEM/EDS analysis

The major components identified in poultry litter fly ash are sylvite and hydroxyapatite (Fig. 3). Additional minerals such as collinsite and aphthitalite were also seen. Ash KP-4 comprised a mixture of thenardite and aphthitalite. Carbonates such as burkerite and hankesite were also detected. These compounds are highly water-soluble, which aligns well with the previous experimental results. In contrast, in ash FL-2 the potassium was found mainly in the form of silicates (waedeite and leucite). This mineral composition explains the low concentrations of potassium leached from this ash with water.

Fig. 4 shows the TGA for more interesting ash samples. A small decrease in weight (< 1 wt%) was observed at temperatures below 200 °C, corresponding to the loss of humidity in the ash. Above 500 °C, the weight decrease becomes noticeable, and this is due to the decomposition of the different carbonates in the samples (calcium, sodium, potassium etc.). According to (Georgieva et al., 2013), calcium carbonate typically decomposes in the range 550–750 °C. Decomposition of sodium and potassium carbonate occurs at higher temperatures, with sodium carbonate decomposing at around 800 °C and potassium carbonate at 1000 °C, as reported by (Siriwardane et al., 2011; Lehman et al., 1998). Halide decomposition also occurs at elevated temperature, with potasium and sodium chloride starting decomposing at above 800 °C (Broström et al., 2011). Hydroxyapatite, the other predominant component in poultry litter ash, decomposes gradually during heating, leading to a steady and incomplete mass reduction in the temperature range studied (Lazić et al., 2001). In contrast with samples PL-1, PL-2 and PL-3, the major weight loss for ash KP-4 occurs at higher temperature, above 750 °C. This is due to its significantly different chemistry (absence of calcium carbonate, presence of primarily sodium species, and a low K:Na ratio), which results in TGA with a lower number of weight loss steps.

Figure S2 to Figure S5 in supplementary material show the SEM/EDS analysis for four of the ash samples. The poultry litter fly ash appears to comprise of particles with varying morphology, e.g., spherical, spheroidal, rod-like and flake-like. The EDS analysis of these morphologies revealed that potassium is present as porous agglomerates in which sulphur, chloride and oxygen are also present (potassium sulphate and chloride, also confirmed by XRD analysis). These agglomerates adhere to and can cover particles of other morphology, such as spherical particles containing calcium, phosphorus, magnesium, and oxygen (Figure S2 in supplementary material, A and B); or rod-like silicon dioxide particles (Figure S2 in supplementary material, C). Adherence on calcium rich particles is mostly noticeable in sample PL-F3, a fly ash



Symbol	Mineral	Chemical Formula	Symbol	Mineral	Chemical Formula	Symbol	Mineral	Chemical Formula
A	Sylvite	KCl	*F*	Portlandite	$Ca(OH)_2$	*K*	Burkeite	$C_{0.61}Na_3O_3S_{0.89}$
B	Hydroxiapatite	$Ca_5(PO_4)_3OH$	*G*	Halite	NaCl	*L*	Hanksite	$C_2ClKNa_2O_4S_9$
C	Aphthitalite	(K,Na)3Na(SO4)2	*H*	Gmelinite-K Kaolinite	$Al_2Si_2O_5(OH)_4$	*M*	Calite	$CaCO_3$
D	Aranite	K_2SO_4	*I*	Grattarolite	Fe_3O_7P	*N*	Wadeite	$K_2O_9Si_4$
E	Collinsite	$Ca_2(Mg,Fe^{2+})(PO_4)_2 2H_2O$	*J*	Thenardite	Na_2O_4S	*O*	Leucite	$AlKO_6Si_2$

Fig. 3. XRD pattern of ash samples FL-2, KP-4, PL-F4, PL-F3, PL-F2 and PL-F1, indicating major crystalline phases present.

from the flue gas treatment where lime is dosed to remove sulphur oxides and hydrochloric acid, which results in lime particles collected with the ash. The particle size varies, with particles and agglomerates above 100 microns being observed alongside flakes and particles of only a few microns in size. Due to the varying morphology and the clumping and adherence of potassium species on larger particles, up concentrating a potassium fraction and removing part of the calcium and magnesium particles by, e.g., sieving, will lead to losses of potassium. Ash KP-4 (rich in sodium sulphates and carbonates, free from calcium and with a lower content of chlorides and potassium) has a significantly different morphology. This ash contains mostly flake-like particles of a few microns, which adhere to form agglomerates of tenths of microns or larger. A more diverse morphology was observed for ash FL-2, where needle and rod-like particles, spheroids and flakes were seen alongside spheres rich in calcium, potassium, silicon, oxygen, iron, and aluminum.

4.5. Leaching of potassium from poultry litter ash and up-concentration of aqueous potassium

Ash from incineration of poultry litter stand out due to their high content of water-soluble potassium (>20 wt% for fly ash). The water solubility of potassium sulphate is advantageous for direct application of these materials on soils. However, the leachability of sodium and chloride ions alongside potassium sulphate and the high alkalinity of the leachates limits this. While the heavy metals showed low solubility in water, their long-term presence and accumulation in soils is undesirable, and is an environmental concern. The low solubility of phosphorus means this nutrient will have low plant availability. A chemical process that removes the contamination (heavy metals) and enables the selective recovery of potassium and phosphorus as pure compounds is desired. Such processes greatly improve the sustainability of industries that generate such streams, having a significant positive contribution towards fulfilling various UN Sustainable Development Goals, e.g., 6. Clean Water and Sanitation, 9. Industry Innovation and Infrastructure, 12. Responsible Consumption and Production, 13. Climate action, and 15. Life on Land. Chemical processing enables the production of high-purity fertilizers with defined and preferred chemistry. For example, potassium sulphate is commercially more desired and preferred over potassium chloride by crops sensitive to chloride ions, or crops that need high sulphur content (Zehler et al., 1981).

Table 4 shows that potassium and phosphorus can be selectively separated by leaching the ash with water. We will address the fate of phosphorus in the undissolved residue later, and for now focus on the water-soluble elements. Leaching of ash samples obtained after co-incineration of poultry litter with plant residues (PL-F1, PL-Bt1, PL-Bo1) was investigated in more detail. Leaching was done at L:S ratio 3:1 w/w, which is more representative for industrial applications. This ensures higher content of solids in the slurry, which enables filtration using conventional solid-liquid separation units (filter press, belt filter), and production of more concentrated leachates. Fig. 5 shows the concentration of potassium, sodium, sulphur (as sulphate) and chloride in the bottom (PL-Bt1), boiler (PL-Bo1) and fly ash (PL-F1) leachates. Due to their lower soluble salt content, the bottom and boiler ash leachates were less concentrated. A second leaching step in which fresh ash was leached with leachate from the first ash leaching increased the aqueous concentrations of some elements. For fly ash, this second leaching only marginally increased the potassium concentration (from 67.4 g/L to 71.9 g/L). This is because the original water leachate was already close to saturation (solubility of potassium sulphate in pure water at 20 °C is 111 g/L (Söhnel, Novotny.,1985) which is equivalent to 49.8 g/L K in solution). In this case, enrichment of potassium in solution is not needed. A third leaching step for the bottom and boiler ash using previously obtained leachate showed that further enrichment of potassium in solution is possible. The aqueous phosphorus content remained low, e.g., < 6 mg/L after a third leaching of the boiler and bottom ash.

The leaching behaviour of potassium, sodium and sulphur (as sulphate) is presented in Figure S7 in supplementary material. Potassium was leached within the first 20 min., the bulk already before the 10 min. mark. Sodium and sulphates dissolved faster with only marginal increases in their aqueous concentrations after 10 min. This is an

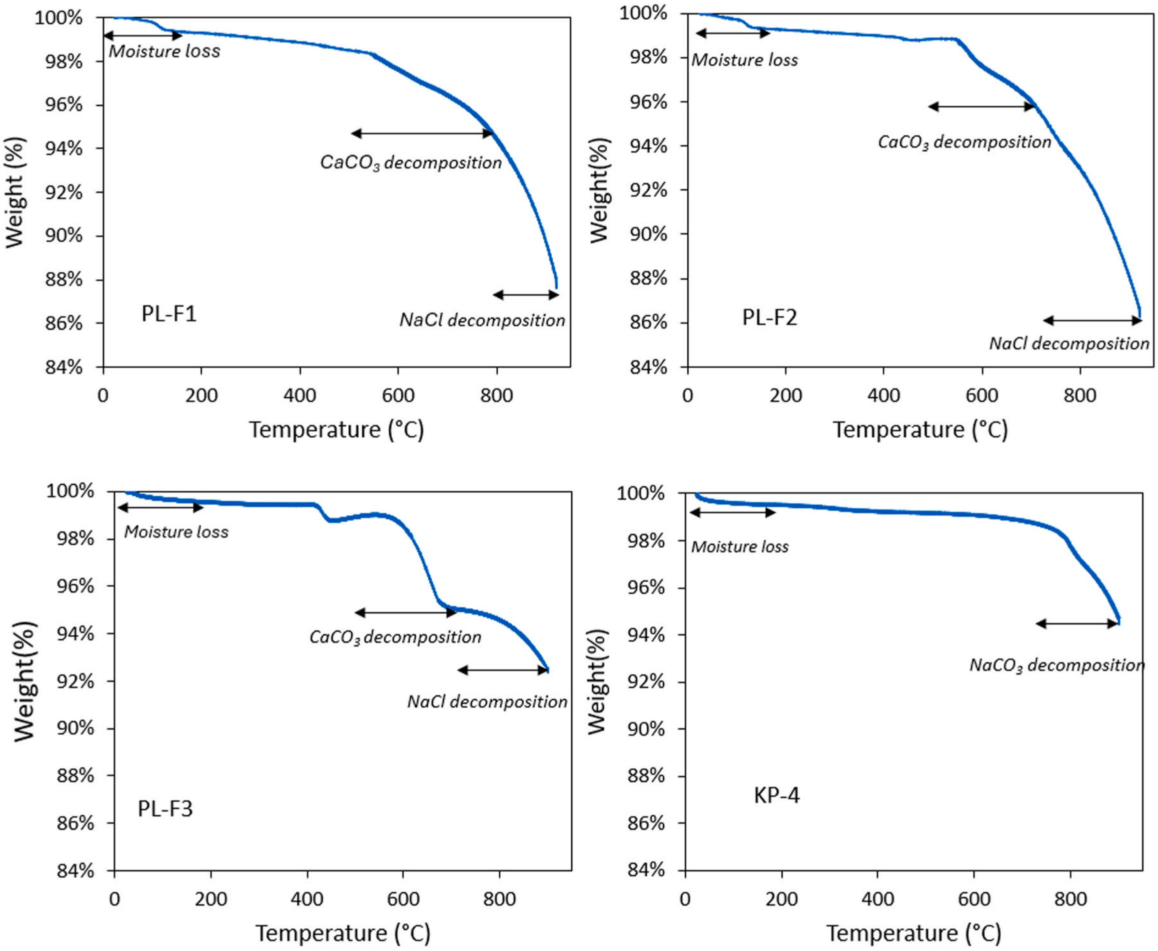


Fig. 4. TGA for ash PL-F1, PL-F2, PL-F3 and KP-4.

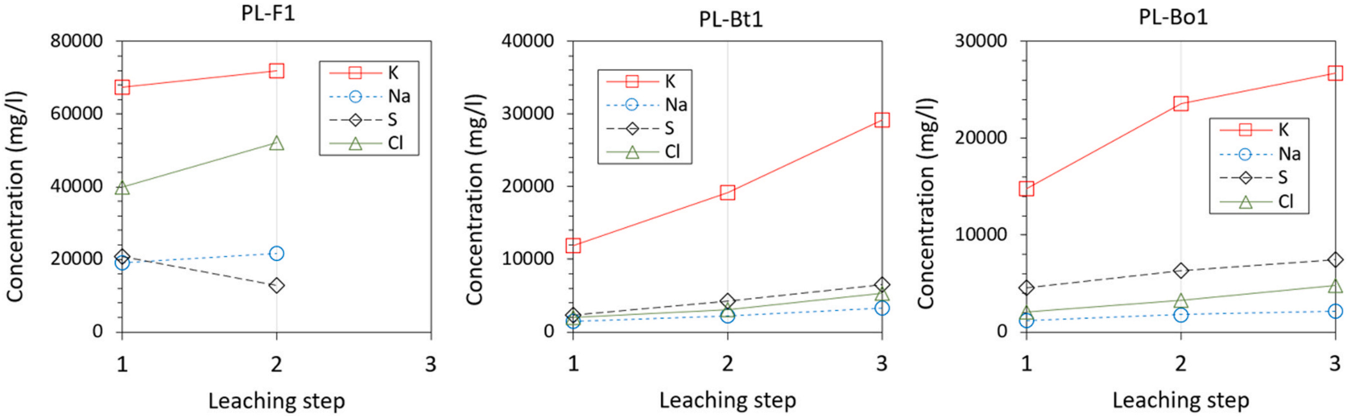


Fig. 5. Concentration of potassium, sodium, sulphur (as sulphate) and chloride in bottom (PL-Bt1), boiler (PL-Bo1) and fly ash (PL-F1) leachates after several leaching steps. The initial leaching was done with water at L:S ratio 3:1 w/w (60 min., 20 °C). The subsequent leaching steps were done with the previously obtained leachates.

advantage for industrial operations, where continuous processing and short retention times are desired. Because these species are leached at the same time, leaching selectivity cannot be exploited to separate potassium.

The PL-F1 ash mass loss and the aqueous potassium concentrations at different L:S ratios are presented in Fig. 6. Leaching at low L:S ratios ($<$ 3:1) is not recommended as this will not effectively dissolve the salts. About 21 wt% of the ash dissolved at L:S 1:1 w/w and 42 wt% at L:S 2:1 w/w. In both cases, the aqueous potassium concentrations were about 66–67 g/L. Leaching at L:S 3:1 w/w produced a leachate with similar potassium content. The mass loss was close to the values seen at higher L:S ratios indicating high leaching yield. The lower amounts of ash dissolved at low L:S ratios were corelated to the saturation of potassium sulphate in the leachate. Among the soluble salts, potassium sulphate has the lowest solubility at ambient temperatures, 120 g/L in water at 25 °C (Table S4 in supplementary material).

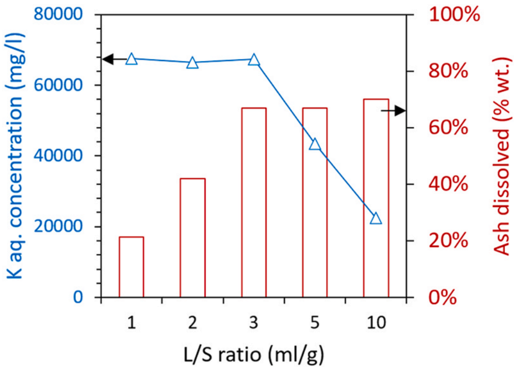


Fig. 6. Ash mass loss (wt%) and aqueous potassium concentrations for sample PL-F1 at L:S ratios between 1:1–10:1. Leaching was done for 60 min. at 20 °C.

4.5.1. *Potential methods to recover potassium and sodium salts based on existing literature data*

Relevant literature data that can be exploited to recover salts from poultry litter ash and possible recovery scenarios are presented in Fig. 7.

We have shown in Fig. 5 that, for ash with lower potassium content (bottom and boiler fractions), up-concentration of potassium in the leachate can be done by re-using the leachate to subsequently leach fresh ash. The high pH of the leachate ensures that heavy metal contamination is minimal since their solubility at such pH values, e.g., pH 11–12 is very low. If further decontamination is needed, treatment of the leachate with sodium hydrogen sulphide or sodium sulphide will effectively precipitate heavy metal ions as sulphides (Bhattacharyya et al., 1979; Marchioretto et al., 2005). There is potential to selectively separate salts from the resulting solution by exploiting their different solubilities (Table S4 in supplementary material). Water soluble carbonates, if any, can be converted to sulphates or chlorides by adding sulphuric or hydrochloric acid, respectively. Potassium sulphate has the lowest solubility among the expected sulphate and chloride compounds. A common method to recover potassium sulphate from solution is via the selective crystallization of glaserite, a double sulphate salt of potassium and sodium (3:1 stoichiometric ratio) (Grzmil and Kic, 2005a). There are seeveral publications dealing with crystalization of glaserite from solution (Grzmil and Kic, 2005b; Mazzarotta, 1989; Zisner et al., 1996). Precipitation of glaserite from leachates comprising of potassium and sodium chlorides and sulphates, e.g. by evaporative crystallization or antisolvent crystallization, will remove the soluble sulphates from solution and produce a chloride brine. Sodium chloride and potassium chloride can be separated from each other by taking advantage of their different solubilities at different temperatures (i.e., sodium chloride is less soluble at higher temperature and vice versa). This is further detailed in Patent US 10773971B2.

Glaserite can be converted to sodium sulphate and potassium sulphate as described in patents US4215100A, US7604792B2, US1936070, KR102378526B1, RU2107028C1 or RU220579C2. For example, patent US7604792B2 describes methods to split glaserite by dissolution in hot water to produce a saturated solution that is afterward cooled; and addition of glaserite to a saturated solution of glaserite in water and mixing at a temperature where glaserite will decompose. Further separation of potassium sulphate from sodium sulphate using froth floatation is also described. Patent US1936070 describes splitting of glaserite by leaching with saturated potassium chloride solution to replace the sodium in glaserite with potassium.

4.5.2. *Potential methods to process the phosphorus-enriched fraction based on literature data*

Leaching of the potassium and other water-soluble elements dissolves the bulk of the fly ash, significantly lowering its mass (about 70 % of the mass for PL-F1, Fig. 6). This up-concentrates phosphorus in the undissolved residue, henceforth denoted as ‘ash residue’. Table S5 in supplementary material shows the analysis of such a residue produced by leaching ash PL-F1 with water. The resulting material contained 11.3 wt% phosphorus as low water-soluble phosphate. Sodium (1.98 wt %) and potassium (4.28 wt%) were quantified, likely present as compounds with low water solubility, e.g., silicates. Calcium (23.1 wt%), magnesium (6.87 wt%), zinc (1.4 wt%), aluminium (0.72 wt%), silicon (4.6 wt%) and iron (1.1 wt%) were the other major elements. The up concentration of heavy metals together with phosphorus makes decontamination important for a sustainable use (or recovery) of the fertilizer. The SEM and EDS analysis of the ash residue is presented in Figure S6 in the supplementary material.

A survey of the literature shows that acidic leaching can be used to make the phosphates water soluble and plant available. Kan et al., 2024 describes a process in which poultry litter ash (without prior removal of potassium) are treated with sulphuric acid and phosphoric acid to improve the nutrient solubility. As shown above, a prior removal of water-soluble salts will generate a fertilizer with a higher phosphate content. However, this does not negate the downsides associated with

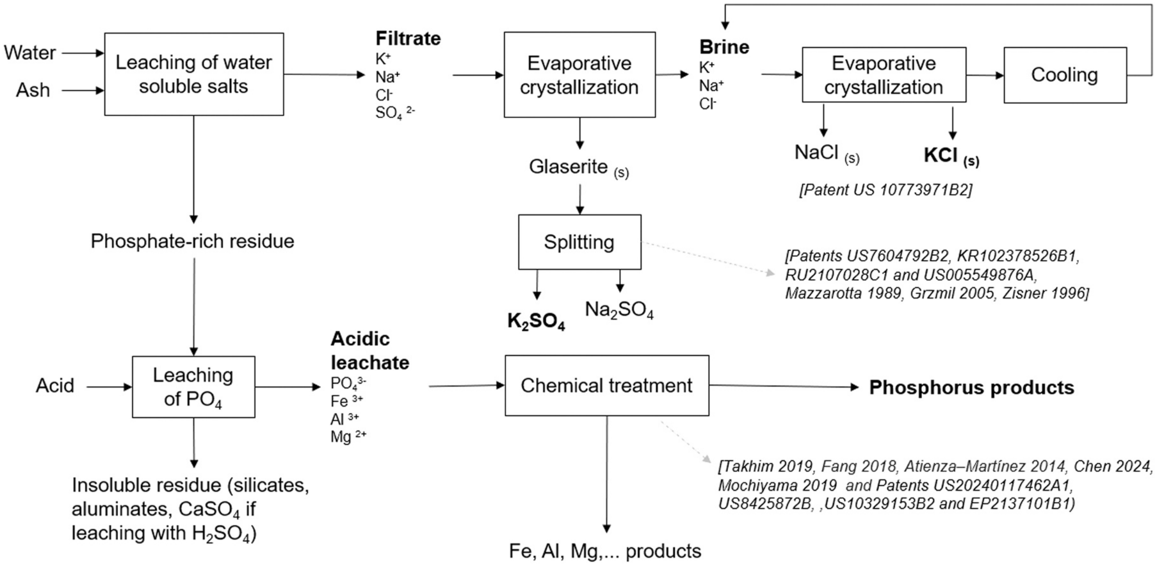


Fig. 7. Possible pathways to recover potassium and phosphorus from poultry litter ash based on existing literature studies.

the presence of the remaining non-nutrient elements, particularly heavy metals.

Acid treatment, depending on conditions (concentration, temperature, pH, reaction time), will also improve the solubility of other compounds apart from silicates and other mineral oxides that have high acid chemical resistance, e.g., aluminium oxide. Chemical processing of impure phosphor-rich acidic leachates to selectively separate phosphate ions from aluminium, magnesium, iron and other ions are described in the literature, and are applicable to such materials. It was reported (Levlin, 1999) that the solubility of phosphate compounds in sewage sludge increases with decreasing pH, with formation of phosphoric acid occurring $<$ pH 2.15. An effective recovery of phosphorus with mineral acids will require a low leachate pH.

Leaching of the phosphorus-rich ash residue with sulphuric acid generates a stream without calcium, which precipitates as gypsum. Selective acidic dissolution of phosphates can be done by controlling the leaching pH. Leaching with hydrochloric acid at low pH produces a leachate with phosphoric acid and metal chlorides, notably calcium, magnesium, zinc, aluminium and iron chlorides. This solution is chemically similar to hydrochloric acid leachates of sewage sludge ash, already acknowledged to be an attractive material for phosphorus recovery (Ohtake and Tsuneda, 2019). Solvent extraction with tributyl phosphate can be employed to selectively recover phosphoric acid and ferric chloride as described in Patent US2024/0117462A1. Precipitation with lime at various pH can also be used to selectively recover calcium phosphate, iron chloride, magnesium hydroxide and aluminium compounds as detailed in patent US 2010/0068113 A1. Other approaches are possible, for example Patent US8425872B2 suggests leaching phosphorus with phosphoric acid and removing the dissolved impurities using ion exchange (Takhim et al., 2019); or production of calcium hydrogen phosphate after leaching the material with raw phosphoric acid, lime and sulphide, and subsequent removal of calcium from solution as gypsum using sulphuric acid (Patent US10329153B2).

After treatment of ash residue with acid to recover/remove acid soluble constituents, the resulting solid fraction comprises of undissolved silicates, aluminates etc., and can be used in construction applications. Residues after leaching of fly ash have been used in such applications, for example in concrete (Hemalatha and Ramaswamy, 2017; Thomas, 2007).

5. Conclusions and future perspectives

There is high potential to recover nutrient elements like potassium and phosphorus from ash after the incineration of biostock. This promotes circularity and offers alternative sources for these elements, which are mainly produced through virgin mining. Among the ash samples evaluated in the present paper, fly ash after the incineration of poultry litter is most promising due to its high potassium content ($>$20 wt%), which is present mainly as water soluble salts like chloride and sulphate. It was possible to selectively leach potassium, sodium, sulphate, and chlorine species from phosphorus, calcium, iron, aluminium, silicates, and magnesium, which were left undissolved in the resulting solid. For poultry litter ash fractions with lower potassium content (i.e., bottom and boiler ash), enrichment of potassium in the leachate was possible through sequential leaching. Leaching selectivity over heavy metals was very good, enabled by the high pH of the leachate. If still needed to guarantee the recovered potassium fertilizers meet the necessary standard requirements (e.g., set in the EU by the Fertilising Product Regulation 2019/1009), a sulphide-based treatment step for heavy metals can be implemented after leaching. Removal of heavy metal sulphides is effectively carried out in alkaline media without generation of hazardous hydrogen sulphide by dosing, e.g., sodium sulphide or sodium hydrogen sulphide. Future work needs to investigate the selective separation of sodium and potassium chlorides and sulphates in the resulting ash leachate. This was not studied before for such ash, but a survey of the literature shows that separation is possible by exploiting the differences in their aqueous solubilities, in particular the lower solubility of potassium sulphate or glaserite, a double sulphate salt of potassium and sodium. Investigations can target the feasibility to remove the aqueous sulphates as glaserite via evaporative crystallization; and subsequent chemical conversion of the glaserite to potassium sulphate and sodium sulphate.

For poultry litter fly ass, the high ash mass loss during leaching of potassium (70 wt%) resulted in a solid fraction with 11.3 wt% phosphorus, present mainly as calcium phosphate. This is also an attractive secondary source for nutrient recovery. Future investigations can target recovery of phosphorus employing hydrometallurgical techniques used to process and decontaminate apatite or other ash, for example sewage sludge ash. These rely on a combination of acid leaching, precipitation and/or solvent extraction and ion exchange.

Chemical processing of nutrient-containing ash offers significant benefits compared to landfilling or spreading on land (with or without conditioning), foremost decontamination (removal of heavy metals), and recovery of concentrated compounds with defined chemistry and application area (e.g., fertilizers tailored for selected crops or soil types). Legal restrictions can limit the direct use of such ash as fertilizer. Therefore, processing them into clean commercial products is key to comply with regulations, minimize landfilling and promote circularity by providing a sustainable secondary source for nutrients.

Supplementary material

Provided materials include: The description of appearance of the ash samples (Figure S1), Concentrations of additional minor elements in the ashes (Table S1), Concentrations of additional minor elements in the leachates obtained by leaching the ashes with water at L:S 10:1 w/w. (Table S2). Other analysis of on the ash, including TN (Total Nitrogen), Halogens (fluorine, chlorine and bromine), Total Carbon (C), Inorganic Carbon (IC) and Total Organic Carbon (TOC) (Table S3) SEM images and EDS analysis from the ash PL-F1 (Figure S2) SEM images and EDS analysis from the ash PL-F3 (Figure S3) SEM images and EDS analysis from the ash KP-4 (Figure S4) SEM images and EDS analysis from the ash FL-2 (Figure S5) Solubilities of sodium and potassium sulphates and chlorides at 25 °C (Table S4) Elemental analysis of the residue obtained after leaching ash PL-F1 with water at 20 °C and L:S 3:1 w/w for one hour (Table S5). SEM/EDS of the residue obtained after leaching ash PL-F1 with water (Figure S6) Leaching behaviour of potassium, sodium and sulphur and chloride from sample PL-F1 with water at 20 °C and L:S 3:1 w/w for 120 min (Figure S7).

CRediT authorship contribution statement

Ehsan Moslehi: Writing – original draft, Visualization, Validation, Investigation, Formal analysis, Conceptualization. **Gabriella Mastantuoni:** Visualization, Validation, Investigation, Formal analysis. **Elin Larsson:** Writing – review & editing, Validation. **Seisenbaeva Gulaim:** Writing – review & editing, Supervision. **Cristian Tunsu:** Writing – original draft, Project administration, Methodology, Conceptualization. **Yariv Cohen:** Writing – review & editing, Supervision. **Vadim Kessler:** Supervision.

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Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Ehsan Moslehi reports financial support was provided by The Ragnar Sellberg Foundation. Cristian Tunsu reports financial support was provided by BioInnovation. Ehsan Moslehi has patent #SE-21141344 pending to EasyMining Sweden AB. Cristian Tunsu has patent #SE-21141344 pending to EasyMining Sweden AB. Yariv Cohen has patent #SE-21141344 pending to EasyMining Sweden AB. Elin Larsson has patent #SE-21141344 pending to EasyMining Sweden AB. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.clwas.2025.100428.

Data availability

Data will be made available on request.

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