Thermochemical evaluation of elemental phosphorus recovery from sewage sludge

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Abstract

The FlashPhos process aims to recover white phosphorus (P_4) from sewage sludge by drying, flash combustion/gasification, and carbothermic refining steps. This study evaluates the material behaviour of sewage sludge ash melting and reduction in a refining furnace for the novel FlashPhos process. Thermodynamic modelling in FactSage 8.2 is combined with experiments from a tube furnace to determine the possible phosphorus recovery and minor element behaviour. For the two studied sludges, a yield of 40% and 75% white phosphorus is achieved after 1 hour at 1600°C, corresponding with a 60% and 25% loss to the metal phase. This is slightly above the calculated equilibrium yield of 28%–73%, which depends mainly on the Fe/P ratio of the sludge. Temperature is found to have a significant influence on the final phosphorus yield when leading to incomplete melting of the ash. A clean slag is produced in the process free of heavy metals making it suitable to be used as a cement-like binder material. The main heavy metals that co-evaporate with the phosphorus are Zn, Pb, As, Sb, and Sn. Additional processing steps will be required to remove these to produce a pure P_4 product. The metal alloy consists of mostly Fe and P with smaller amounts of other components such as Mo, Cr, Cu, Mn, Sn, Sb, and V. The final slag consists of CaO, SiO_2, MgO, and Al_2O_3 with virtually complete removal of P and heavy metals.

Keywords

sewage sludge, phosphorus recovery, sustainability, refining process

Introduction

Phosphorus is classified as a critical raw material by the European Commission due to its importance in the agricultural, pharmaceutical, and electronic sector. The current and expected increased demand for phosphorus far exceeds the phosphorus reserves available (Salkunić, et al., 2022). Europe is currently fully dependent on imports for elemental phosphorus (European Commission, 2022). Currently, the main source of phosphorus is primary phosphate rock. Alternative secondary phosphorus sources are therefore critical to ensure the sustainable supply of this resource. Sewage sludge is one potential resource of phosphorus, which is currently only exploited to a limited extent. A country loses approximately 50% of its phosphorus through wastewater (Salkunić et al., 2022). It is estimated that 323 kt/a phosphorus can be recovered from sewage sludge in the EU (Nättorp, et al., 2015). This is enough phosphorus to meet the entire EU demand for elemental phosphorus, which is estimated to be around 100–130 ktP/y (European Sustainable Phosphorus Platform, 2020).

Typically, municipal wastewater is treated to remove particles and precipitate out phosphates and other harmful components from the water before returning it to the environment as clean water (Prasad, Smol, 2023). The solids produced in the treatment process are referred to as sewage sludge. Apart from organic matter, sewage sludge also contains heavy metals and other unwanted components such as pathogens and bacteria from the wastewater (Boniardi, 2018). There are various ways in which the sewage sludge is disposed of or re-used, such as landfill, landscaping, or incineration. The sludge is a sink for most of the impurities and contaminants, such as heavy metals and micropollutants (e.g., pharmaceuticals) in the wastewater (Košnář et al., 2023), which are harmful to the environment. Historically, the primary method of sewage sludge disposal has been landfilling (Inglezakis et al., 2014), which leads to the loss of valuable phosphorus resources and poses environmental risks, such as heavy metal contamination of soil and water. In the EU, landfilling of sewage sludge is no longer permitted under EU Directive 86/278/EEC (Council Directive, 1986). Beyond landfilling, other common sludge management practices include incineration and land application, each presenting its own challenges. Incineration destroys organic matter and limits the options for phosphorus recovery, while land application risks introducing heavy metals and excess nutrients into water systems, exacerbating eutrophication. Given that sewage sludge can contain significant phosphorus concentrations, its management is critical for both environmental protection and resource recovery. It is therefore expected that landfilling or soil applications of this material will soon be more restricted across the world (Inglezakis et al., 2014). This means that most of the material will be incinerated (Cohen et al., 2019). Although costly, this provides the opportunity to recover energy from the combustion of the sludge and produce an inorganic ash. The produced ash will still require further processing for nutrient recovery and safe disposal, as it contains a significant amount of phosphorus along with other contaminants, such as heavy metals.

Sewage sludge is therefore a crucial waste stream that could aid significantly in the phosphorus supply and global sustainability goals. In line with the importance of phosphorus and the high losses in wastewater sludge, some EU countries are implementing limits on wastewater treatment plants to ensure the recovery of phosphorus. Germany is enforcing mandatory phosphorus recovery from 2029 for sewage sludges containing more than 20 g P/ kg dry sludge. In Switzerland, phosphorus recovery from sewage sludge is mandatory from 2026 (Sichler et al., 2022). Furthermore, limits are placed on using sewage sludge directly as fertiliser without further treatment due to the high heavy metal content and pathogens present. Table 1 provides the limits being implemented in Germany and Switzerland (Hermann, Schaaf, 2019). The main focus of these regulations is to ensure phosphorus recovery and removal of heavy metals from the material. If used directly in agriculture without purification, there is a high risk of contamination with heavy metals.

The phosphorus can be recovered at various points in the whole wastewater treatment cycle, each with its advantages and disadvantages. From the wastewater itself, roughly 50% phosphorus removal is possible through a precipitation process. The pH is typically increased to between 8–10 and the phosphorus can be precipitated from the water. Depending on the conditions and reagents used, different phosphorus precipitates are formed. In most precipitation treatment routes, the main product is struvite, which can be used as a fertiliser to some extent. Some heavy metals or other contaminants present in the water are precipitated along with the struvite (Salkunić et al., 2022).

During the production of sludge in the wastewater treatment process, using biological or chemical processes to precipitate phosphorus, the removal can be up to 90%–95% (Salkunić et al., 2022). Typically, the chemicals added are Fe and to a lesser extent Al chloride salts, which increase the metals present in the sewage sludge and the chloride content in the wastewater. To bring the phosphorus in the resulting sewage sludge (or ash after its incineration) to use, a separation process is necessary. Currently, a number of processes are being developed to recover phosphorus from sewage sludge and ash. The two main strategies are wet-chemical and thermochemical (Canziani et al., 2023).

The wet-chemical methods involve leaching the phosphorus from the sludge/ash using an acid or alkaline solution (Egle et al., 2015). Phosphorus can then be precipitated out or concentrated in different forms. In some cases, the phosphorus is kept in solution to produce phosphoric acid. Purification steps are often included to separate contaminants from the product. The wet-chemical process has a higher cost compared to the thermochemical process as well as other challenges to ensure efficient separation of heavy metals and other contaminants (Canziani et al., 2023).

Thermochemical processes involve heating the sludge or ash to higher temperatures (1000 °C–1600°C). In some cases, Cl or S is added to aid in separating the heavy metals from the phosphorus. In these processes, the phosphorus is kept in the slag to produce a fertiliser product (Canziani et al., 2023). Typically, some of the heavy metals (As, Pb, and Zn) will be found in the gas phase due to their high volatility. Others, such as Ni, Cr, Cu, etc., remain in the slag product. As a consequence, these processes cannot completely remove the heavy metals from the final P-product.

A limit in many of the processes (precipitation of e.g., struvite, thermochemical and wet-chemical) is the removal of heavy metals. As the heavy metals content in the wastewater is highly variable, the final phosphorus product may not always meet the criteria to be used directly as fertiliser, or for other purposes such as phosphoric acid (Egle et al., 2015). This makes the processes less robust towards different feed compositions. Nevertheless, these routes allow to recover P in a fertiliser or as phosphoric acid and may cover parts of the largest P applications. However, no route is available for high purity phosphorus products, which typically rely on thermal white phosphorus.

FlashPhos is a novel thermochemical process being developed to recover phosphorus from dried sewage sludge. Unlike the mentioned processes, this process is designed to recover high-quality white phosphorus from sewage sludge. While the FlashPhos process also recovers some phosphorus in a ferrophosphorus alloy, its primary output is high-value P_4. A pilot plant is currently being developed by an international consortium in the FlashPhos project (Flashphos, 2024). Figure 1 provides the flowsheet of the process.

Sewage sludge is first dried and ground to a fine powder. The material is then fed into a flash furnace to gasify and partially combust the material. The combustion energy is used to melt the material in the furnace. Municipal sludge contains between 23%–60% organic material, which can generate an amount of energy of between 8–21 MJ/kg through combustion (Prasad, Smol, 2023), which could significantly contribute to the energy required for the process. The molten material is then reduced with carbon in a refiner furnace to produce phosphorus gas, slag, and a ferrophosphorus alloy. The iron present in the sewage sludge is partly from the wastewater itself as well as from the chemicals (typically, $FeCl_3$) added to precipitate out the phosphate in the water treatment process. White phosphorus is recovered from the gas phase by condensation. The process achieves a better separation of

Table 1

Heavy metal limits for (recycled) fertiliser in Germany and Switzerland (Hermann, Schaaf, 2019)

	Unit	As	Cd	Cu	Hg	Ni	Pb	Zn
Germany	mg/kg	40	4	70	1	80	150	1000
Switzerland	mg/kg		1	100	1	30	120	400

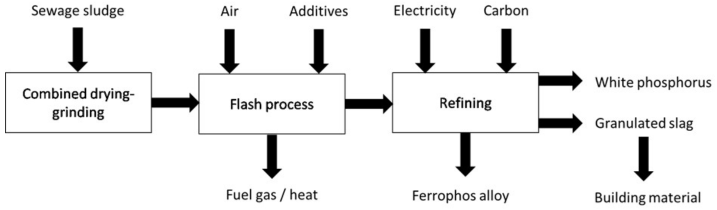

Figure 1—Flowsheet of the FlashPhos process

heavy metals compared to other processes, as many metals remain in the alloy or slag material. Volatile metals, such as arsenic and zinc, that report to the gas phase along with P_4 can be efficiently removed before P_4 condensation, due to their higher condensation points, and different reactivities.

Arnout and others (2023) previously discussed the material behaviour in the flash furnace. Based on this work, a detailed CFD model could be developed to simulate the process discussed by Ortner et al. (2023). Another important aspect is the design and operation of the refiner process. For this reduction process, it is important to understand the chemical behaviour of phosphorus and the relevant impurity elements in sewage sludge, in order to determine the expected phosphorus yield as well as the quality of phosphorus, slag, and alloy. The current work aims to improve this knowledge using a combination of thermodynamic modelling and lab-scale experiments. This work builds on the previous work, which focused on phosphorus behaviour with limited experimental work (Arnout, Nagels, 2016).

Approach and methodology

This study aims to provide insight into the expected efficiency of the refining process, specifically reaction equilibria, component distribution, and product yields. The material reduction behaviour is first studied using thermodynamic calculations. This enables an understanding of the carbon requirement of the process to achieve maximum phosphorus recovery. In addition, the theoretical phosphorus yield and component distribution, including heavy metals, are determined and can be compared with experimental results. For experimental verification of the potential phosphorus yield, 40 g scale experiments are conducted in a tube furnace.

Raw materials

The raw materials used in the study are two types of dry sewage sludge (sourced from Germany and Belgium), which are comprised of organic material, volatiles, and 50% ash. The exact composition is a result of the wastewater characteristics and the nature and efficiency of the treatment operations, as well as the geology of the area. Ash is produced from the sludge by heating the sludge at 1000°C for 24 h in an air atmosphere during which all the organic material is combusted. Table 2 provides the ash composition quantified as oxides analysed with XRF. It is expected that these oxides will be present in combined compounds such as phosphates, however, in the thermochemical calculations the mineralogy of the feed material has no effect on the equilibrium phases.

Thermodynamic calculations

Thermodynamic calculations are carried out with FactSage 8.2 (Bale et al., 2016) using the FactPS, FToxid, FSsteel databases for the gas, slag, and metal phases. The FactPS database is included as it contains all the pure compounds. The FToxid data base is selected to represent the oxide phases and the FSsteel to represent the iron phosphate phase in the process.

Table 2

Sewage sludge ash composition in weight percent

Component	Sludge A wt%	Sludge B wt%
Fe_2O_3	24.4	12
Al_2O_3	23.4	9.7
SiO_2	21.3	37.7
CaO	11.4	8.4
P_2O_5	9.9	20.4
SO_3	3.5	1.3
K_2O	2.4	2.4
MgO	2.2	3
Minor components	1.6	5.1

Table 3

Tube furnace experiments

Sample	Ash	Target basicity (CaO/SiO_2)	Temperature (°C)
1	A	1.2	1 600
2	A	1	1 600
3	A	1	1 500
4	A	0.8	1 600
5	A	0.8	1 500
6	A	0.54	1 600
7	B	1	1 600
8	B	0.6	1600

In the thermodynamic assessment, the reduction progress is evaluated as a function of reductant addition. It also allows the study of the phosphorus yield and the component distribution over the different phases formed. A further parameter considered is the temperature of the process. This provides a better understanding of the expected onset temperature of the reduction process. The calculated results are compared with experimental findings to understand kinetic limitations.

Tube furnace experiments

A set of experiments is completed in a vertical tube furnace (GERO HTRV, model 100-250/18) using graphite crucibles of 45 mm inner diameter. This setup allows for a 40 g sample size. It enables the analysis of the final products and to determine the product yield. The influence of basicity and temperature as process parameters are experimentally evaluated. The formed products are analysed with XRF (Malvern – PANanalytical with OMNIAN software), XRD (Bruker D2 phaser), and ICP-MS (iCAP RQ IFC from Thermo Scientific).

Samples are heated at 5°C/min and kept at high temperature for 60 min, then cooled at 5°C/min. All steps are conducted in an inert Ar atmosphere. Table 3 provides a summary of the samples prepared for evaluation. To increase the basicity, samples 2–7 are prepared by mixing the ash with CaO.

Results and discussion

Thermodynamic calculations

Figure 2 shows the calculated phosphorus distribution as a function of carbon addition for ash A and B at 1600°C. This will allow determination of the required amount of carbon to ensure maximum recovery of phosphorus. As expected, with a higher carbon addition, more phosphorus can be reduced and subsequently evaporated. With 12% carbon addition, 28% and 69% phosphorus yields are theoretically possible for ash A and B, respectively. Increasing the carbon addition further does not improve the phosphorous recovery. The remainder of the P forms a metal alloy with Fe, and only a very small amount of phosphorous is expected to remain in the slag. For ash B the losses to metal are expected to be smaller, this is investigated in more detail in the following section. These results are in line with previously reported calculation results (Arnout, Nagels, 2016).

The maximum phosphorus yield for ash A is achieved at 12% carbon addition, which is defined as the carbon saturation point. Adding additional carbon does not improve the recovery. Figure 3 gives the full elemental distribution at 1600°C with 12% carbon addition for ash A. The following can be concluded: Pb, As, and Zn report to the gas phase (depicted by the white bars). Elements such as Co, Cr, Cu, Fe, Ni, Sn, and V report to the metal phase (grey bars). Al, Ba, Ca, Mg, and Sr remain in the oxidic slag phase (black bars). K, Na, S, and P distribute over metal and gas phase, and Mn, Ti, and Si distribute over slag and metal.

Thus, the main contaminants expected in the final phosphorus-containing gas stream are As, Zn, and Pb. These are all components with high volatility and will evaporate at these temperatures into the gas phase once reduced. To obtain a clean end product, these elements are to be removed from the gas phase before the final condensation of phosphorus. The final slag contains mainly SiO_2, CaO, and Al_2O_3 and is expected to have a low remainder of heavy metals (only barium tends to remain in slag). Potential for the use of this material as a cement-type binder has been shown in experiments (Arnout et al., 2023).

Based on the results it could be seen that the formation of a ferrophosphorus metal alloy limits the potential yield of the process. The formation of the metal alloy consumes reductant, and it captures a substantial amount of P, which can no longer be evaporated. This phenomenon is observed for both Ash A and Ash B. A much higher P recovery is achieved with Ash B due to a higher P_2O_5:Fe_2O_3 ratio compared to Ash A. The influence of the P_2O_5:Fe_2O_3 ratio was further evaluated in Figure 4. It can be seen that the P_2O_5:Fe_2O_3 weight ratio of a sludge or ash is an important parameter for the P-recovery potential (Arnout, Nagels, 2016). As shown in Figure 4, a higher iron content in the ash increases the phosphorus remaining in the metal and subsequently limits the amount of recovered phosphorus. From a P recovery standpoint, it could therefore be beneficial to use biological processes or aluminium salts for P removal from the wastewater (ESPP, 2025).

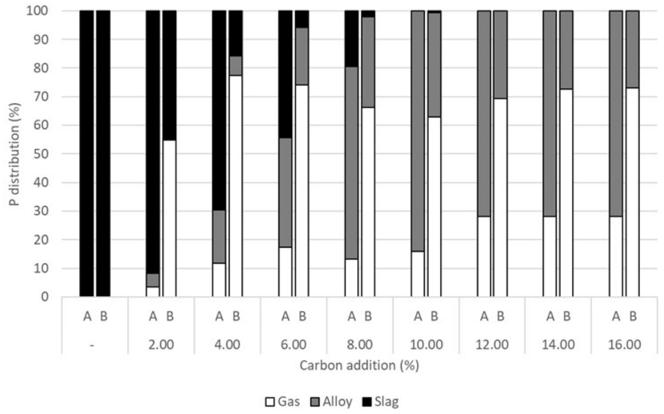

Figure 2—Distribution of phosphorus as a function of carbon addition at 1600 °C of ash A and ash B

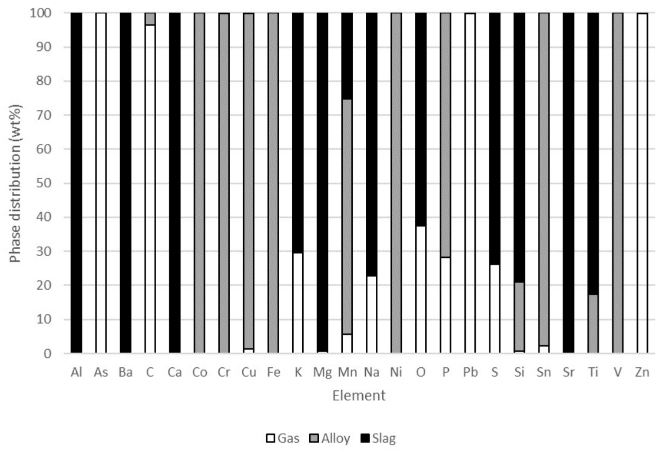

Figure 3—Distribution of elements at 1600°C and 12% carbon addition of ash A

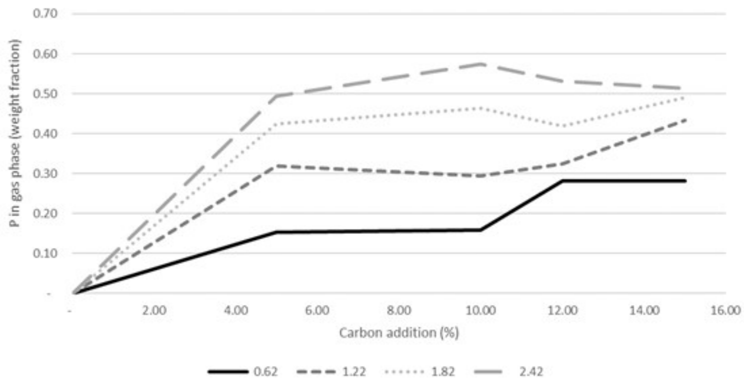

Figure 4—Influence of P2O5/Fe2O3 ratio on phosphorus in gas phase on ash A at 1600°C

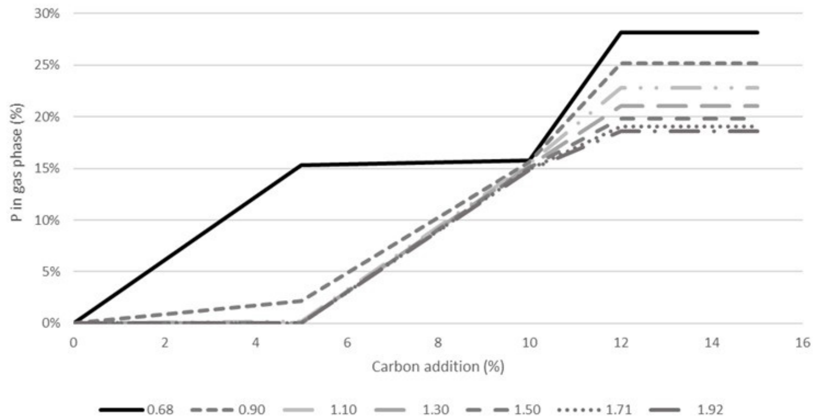

Figure 5—Influence of basicity on phosphorus in gas phase on ash A at 1600°C

Liquid slags are often characterised by their basicity (CaO/SiO_2 ratio) as this has a large influence on the properties of this slag (e.g., melting point, viscosity, activity coefficient) (Verein Deutscher Eisenhüttenleute, 1995). As these properties may influence the reaction extents and kinetics, an experimental validation is needed. In light of this, the influence of basicity on the phosphorus recovery is evaluated in Figure 5 with thermochemical calculations. This is verified experimentally and reported in the next section. The basicity was adjusted by addition of CaO to the feed material in increasing amounts. Based on the calculated theoretical value, a lower basicity achieves the highest phosphorus recovery. However, this is in contrast to experimental work where the recovery is largely independent of the basicity.

Tube furnace experiments

The tube furnace experiments allow a better understanding of the material behaviour and final product quality. Experiments were run with both types of ashes: Ash A, which had a low P_2O_5:Fe_2O_3 ratio of 0.4, and Ash B, which had a higher and more favourable ratio of 1. As the output gas composition was not measured, the amount of phosphorus that evaporated (as product) is obtained from the difference between the phosphorus in the input and the phosphorus remaining in the slag and alloy at the end of the experiment.

A yellow residue was formed at the bottom of the tube furnace. The collected quantity was insufficient for detailed evaluation, but a qualitative evaluation with XRF indicated that it was phosphorus-rich with minor amounts of volatile elements Na, K, Zn, Sn, and S. This corroborates that phosphorus evaporated during the experiment and then condensed at the cold surface in the furnace together with the other volatile components. More details on the phosphorus product can be expected when larger amounts are produced in the pilot plant.

Table 4 and Table 5 show the comparison between the experimental analysis and the calculation results (FactSage) at carbon saturation (12% C) of Sample 2. Sample 2 is close to the expected operating parameters of the FlashPhos process and therefore provides insight into the suitability of the industrial scale application. Good alignment is found between the analysis and the calculation results. The Si and P calculated percentage differ from the measured results by +4.2% and -6.3%, respectively. This is due to excess carbon present reducing more Si and increasing the P activity. It is clear that this point is not reached in the experiment despite the abundance of carbon from the crucible. This could be due to basicity or incomplete melting.

A mass balance was conducted for the various experiments, calculating the weight of metal and slag based on the Fe and Ca input and their respective fractions. The phosphorus balance, presented in Table 6 suggests that phosphorus recovery and evaporation into the gas phase remain unaffected by basicity, regardless of the ash type. However, this contradicts the calculated results. Experimental data indicate that temperature plays a significant role in phosphorus recovery, with higher temperatures enhancing the process. Ash B demonstrated greater phosphorus recovery, primarily due to its higher P_2O_5:Fe_2O_3 ratio—an observation that aligns with thermodynamic simulations (Table 5). Furthermore, the findings suggest that this ratio has a more substantial impact on efficiency than an increase in operational temperature.

Table 4

ICP-MS and FactSage comparison of metal and slag phase at 1600 °C with basicity of 1 (Sample 2)

wt%	ICP-MS	FactSage (12% C addition
Metal		
Fe	73.0	76.8
P	21.2	14.9
Si	0,4	4.6
C	Not analysed	2.3
Ti	0.9	0.1
Cu	0.9	0.4
Mn	0.3	0.6
Ca	0	–
Al	–	0.1
Other	–	0.1
Slag		
SiO_2	39.3	28.2
CaO	34.4	31.1
Al_2O_3	19.5	34.6
MgO	4.1	3.2
K_2O	0.3	1.5
Na_2O	0.3	0.7
TiO_2	0.4	0.08
SO_3	0.2	–
Other	–	0.4

From these lab-scale experiments, an analysis of the minor elements' distribution was also conducted. Due to the low concentration of the elements present and the quantification of volatilisation by difference in the mass balance, these results are considered to be qualitative only. Larger samples in the pilot scale plant will enable better evaluation of the minor elements. At this stage it can be confirmed that the Cd, As, Sn, and Zn vaporise along with the P, in line with the thermodynamic calculations. The conclusions made on the elemental distribution remain similar as in the calculation section (Figure 6 and Figure 7). Differences are seen for Co, As, and Sn, but as their mass balance does not match (more output than input) further analysis and experiments on a larger scale are needed.

Conclusion

The study confirmed the feasibility of producing a phosphorus product with the FlashPhos process. For two selected sludges, a phosphorus recovery of 28% and 73% can be achieved. based on thermodynamic calculations corresponding with a 72% and 27% of the phosphorus to the metal phase. The main parameter influencing this recovery is the P_2O_5:Fe_2O_3 ratio in the sludge. Tube furnace experiments achieved a phosphorus recovery of 40% and 75% for these sludges, slightly higher than predicted by the thermodynamic assessment. Good alignment is found between the alloy and slag composition of the tube furnace experiments and FactSage calculations. The primary heavy metals that co-evaporate with phosphorus include Zn, Pb, As, Sb, and Sn. Additional processing steps are necessary to remove these impurities and obtain a pure P_4 product. The metal alloy consists of mostly Fe and P with smaller amounts of other components such as Mo, Cr, Cu, Mn, Sn, Sb, and V. The final slag consists of CaO, SiO_2, MgO, and Al_2O_3 with virtually complete removal of P and heavy metals.

CRediT author statement

AK: Conceptualisation, methodology, investigation, validation, formal analysis, visualisation, writing – original draft preparation, writing – review and editing preparation.
DM: Methodology, investigation, validation, formal analysis, visualisation.
YC: Methodology, investigation, validation, formal analysis, visualisation.
EN: Conceptualisation, writing – review and editing preparation, supervision
SA: Conceptualisation, writing – review and editing preparation, supervision, project administration.

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Table 5

Mass balance of phosphorus recovery from tube furnace experiments

Sample	Sludge	Temperature	Basicity	P input	P to metal	P to slag	P evaporated (by difference)	
		°C	CaO/SiO_2	g	g	g	g	%
1	A	1 600	1.2	2.84	1.51	0.07	1.25	44
2	A	1 600	1.0	2.84	1.66	0.02	1.15	40
3	A	1 500	1.0	2.84	1.86	0.19	0.79	27
4	A	1 600	0.8	2.84	1.65	0.03	1.16	41
5	A	1 500	0.8	2.84	1.86	0.31	0.67	24
6	A	1 600	0.54	2.84	1.57	0.06	1.21	43
7	B	1 600	1.0	4.89	1.16	0.02	3.72	76
8	B	1 600	0.6	4.89	24	0.06	3.67	75

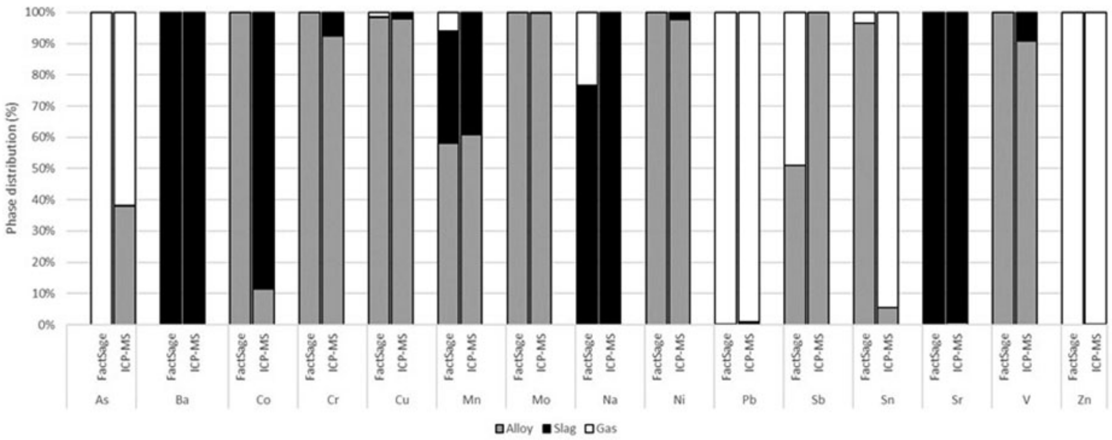

Figure 6—Comparison between FactSage calculations and ICP-MS analysis of the distribution of the minor elements for the ash A

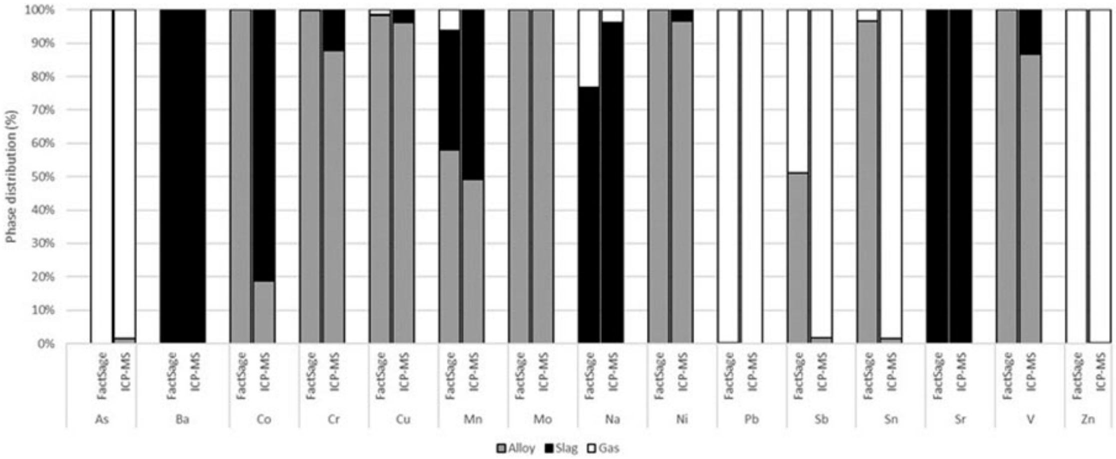

Figure 7—Comparison between FactSage calculations and ICP-MS analysis of the distribution of the minor elements for the ash B

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