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Phosphorus recovery from the wastewater stream—necessity and possibilities

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ABSTRACT

The annual demand of phosphorus (P) for fertilizer use is more than 550,000 t in Germany. Several of the input streams like mineral fertilizer produced from phosphate rock or sewage sludge are problematic due to contamination with heavy metals and organic pollutants and/or supply risks. Phosphorus recovery from the wastewater stream might help to diminish these problems. Several procedures have been developed to recover P from either sewage sludge ash (SSA) or wastewater via precipitation or crystallization as struvite. This study determined the elemental composition and trace metal mass fractions of 252 SSA samples and 17 struvite samples from wastewater treatment plants (WWTP) to see whether there are differences related to the source of the sludge (municipal/industrial) or the stage of the WWTP where the struvite is produced. Results indicated that there is no clear trend of the elemental mass fractions related to the source of the sludge. All struvite samples show very low heavy metal contents, even though the struvite precipitated from the sludge shows slightly elevated heavy metal mass fractions compared to struvite crystallized from sludge liquor. Struvite is a clean and plant available material for fertilizer production from wastewater streams and has some advantages for the wastewater treatment plant. However, since the P recovery rate of struvite processes is low (7–11%) compared to that from SSA processes (90%), recovery from SSA is necessary for a substantial P recovery from the wastewater stream.

Keywords: Phosphorus recovery; Sewage sludge ash; Struvite; Elemental composition; Heavy metal content

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1. Introduction

Phosphorus (P) is essential for all living beings and not replaceable in its functions. It is required for storage and expression of genetic information (DNA, RNA), energy metabolism (ATP-ADP cycle), and bone structure. Since P is ingested via nutrition, it is eventually removed from farmlands and has to be replaced to enable continuous farming. Fig. 1 illustrates the situation in Germany. The annual P intake to farmlands is more than 550,000 t [1]. The five major input streams of P are agricultural fertilizer, especially liquid manure, fermentation residues from biogas production, mineral fertilizer, compost, and sewage sludge. Of those, especially mineral fertilizer and sewage sludge are considered to be critical. Mineral fertilizer is solely produced from phosphate rock [2], a fossil and thus a limited resource. Even though the static range of phosphate rock is calculated to be more than 300 years [3], production and demand are distributed unequally worldwide. The main producers of phosphate rock either have a high domestic demand (USA, China) or are situated in politically unstable regions (Morocco) [3]. Taking into account that Europe has no significant deposits of its own, there is a high import dependency and strong susceptibility to world market price fluctuations. Furthermore, phosphate rock is contaminated with heavy metals, especially Cd, Th, and U that are applied to farmlands via mineral fertilizer and might pose environmental and health hazards. Phosphate rock contains up to 110 mg/kg Cd [4], up to 220 mg/kg Th [5], and up to 660 mg/kg U [5]. This is especially the case for sedimentary phosphate rock that accounts for the vast majority of the known reserves worldwide. As for sewage sludge, there are growing concerns against its direct use for fertilizer purposes. Since the sludge is the pollutant sink of wastewater treatment, it contains inorganic and organic pollutants, especially emerging pollutants of concern (EMPOC) [6,7]. Furthermore, the bioavailability of P in sewage sludge is often debated, especially after P elimination in the wastewater treatment plant (WWTP) with ferric salts that are used frequently [8]. This treatment might lead to low bioavailable iron phosphates. Thus, additional P sources are needed in order to reduce these problems. One possible source for P is the wastewater stream that contains about 70,515 t of P [1]. Until now, 15,315 t/a P are reused in the form of sewage sludge. Thus, the additional P recovery potential from wastewater is 55,200 t/a, as stated in Fig. 1. P precipitation is mandatory in Germany for WWTP with more than 10,000 inhabitant equivalents [9]. Since the wastewater from more than 91% of the population is treated in such WWTPs, the majority of P in the wastewater finally ends up in the sewage sludge [9].

Reuse of P in sewage sludge ash (SSA) might be an option to avoid the above-mentioned problems with direct utilization of sewage sludge for fertilizer

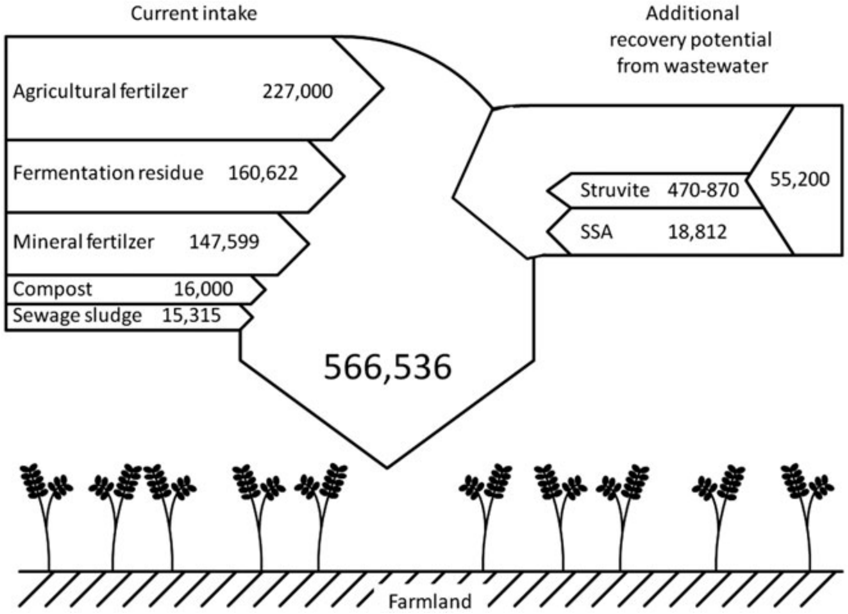

Fig. 1. Phosphorus input streams to German farmlands and phosphorus recovery potential from the wastewater stream (all values in t/a; with data from [1,10,11]).

purposes. SSA shows a high mean concentration of P of 9% in ashes from municipal sludge with peak values of 13% [10]. On the other hand, the concentration of heavy metals is also increased due to the incineration. Thus, an additional treatment of SSA prior to its use as a fertilizer might be required, depending on the actual composition of the SSA [10,12]. In a recent survey of German SSA that took into account only ashes from monoincineration plants, a considerable P recovery potential of up to 19,000 t/a was found [12]. This number might be increased if the sewage sludge that is presently applied to farmlands is incinerated instead and coincineration of sewage sludge is reduced in favor of monoincineration. Another option for P recovery from wastewater is the precipitation as struvite. However, for high P recovery rate and economically feasible production, this process requires a WWTP that applies biological P elimination. In addition to the potential revenue from sale of struvite as a fertilizer, struvite is known to cause problems in WWTP due to uncontrolled precipitation [13]. Thus, precipitation on purpose in a controlled way is an option to overcome these problems [14]. This process was optimized on technical scale [15] and also proposed for P removal from landfill leachate [16]. Despite the obvious benefits, the amount of P recovered by this process is currently marginal [11].

In general, P recovery is possible during all process steps of wastewater treatment, as shown in Fig. 2. However, the concentration of P in these various process steps differs significantly. This influences both the amount of material that needs to be handled during the recovery process as well as the maximum possible P recovery rate [17]. For instance, the SSA account only for 0.015% of the relative flow of the effluent and is thus much easier to handle. Furthermore, the P recovery rate from SSA is 90% or higher, whereas from the effluent, it is only up to 55%. From that point of view, P recovery from SSA is a promising option. P recovery via struvite is usually done either before the dewatering step as precipitation from the sludge or after the dewatering step as crystallization from the sludge liquor. One example for the first case is a process that was developed to avoid incrustations of struvite in pipes, valves, etc. and is aimed to precipitate struvite on purpose instead [18]. This is achieved by adding $MgCl_2$ and adjusting the pH by stripping the sludge with air to remove CO_2. The process results in struvite that is marketable as fertilizer and furthermore improves the following dewatering step. However, the overall P recovery is limited to 7% in total. Struvite crystallization from the sludge liquor after the dewatering is performed similarly by adding $MgCl_2$. In this case, the pH is adjusted by adding NaOH [19]. The P recovery rate of this process is up to 11% of the total P content in the wastewater. However, these recovery rates fluctuate due to changing conditions like weather, efficiency of biological P removal, P load and concentration. Thus, only a range of P recovery rates can be given for these processes. Regardless of where the water is taken from to precipitate struvite, $MgCl_2$ supplement is necessary. The primary chemical requirement cost for a struvite crystallization process is usually due to this magnesium demand. One cheap and environmentally friendly source of magnesium is through the reuse of desalination plant reject water [20]. Desalination plants produce

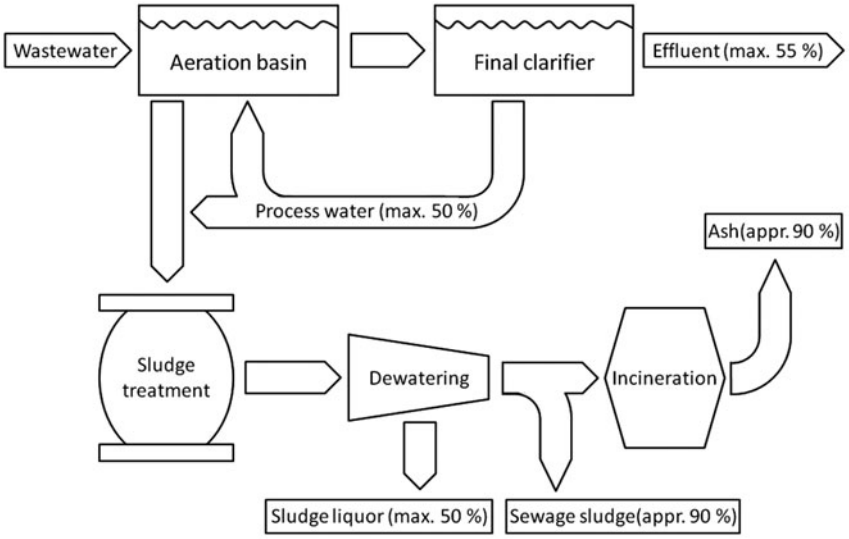

Fig. 2. Possible starting points for phosphorus recovery during wastewater treatment including the respective maximum possible phosphorus recovery rates (according to [17]).

considerable amounts of those as by-product that is normally discharged back to the ocean. However, using the reject, water can provide the possibility of combining two waste sources for resource recovery. This reuse for magnesium supplement can be important for places like the Middle East where the desalination process is extensively used. With magnesium costs around USD 3.5/kg, there is great potential to reduce chemical cost for P recovery depending on the size of the WWTP.

Several processes for struvite recovery have been developed and the composition of SSA was analyzed thoroughly [10,21]. However, very little is known about possible differences in SSA composition and trace element mass fractions related to the source of the sludge (purely municipal, industrial, or mixed). Also, there is only limited information whether there are differences in the resulting struvites depending on the respective method and stage of the WWTP where it is produced. Information on whether the elemental composition of SSA is affected by the source of the sludge is crucial for their intended use as fertilizer. The same applies to the type of the struvite production. This study determined the respective elemental mass fraction in SSA and struvite and discusses them is this respect. The results obtained were compared to the limit values as stipulated by the German fertilizer ordinance to determine whether additional treatment is necessary to reduce the heavy metal content prior to fertilizer production. Phosphorus plant availability is another crucial parameter for the use of secondary raw materials for fertilizer purposes. Discussion on this topic for SSA can be found in [12].

2. Materials and methods

2.1. Sample acquisition

In this study, 252 samples of SSA were obtained. 163 of the samples were from 15 monoincineration facilities that utilize solely municipal sewage sludge, 20 from two plants that use only industrial sludge, and 69 samples were from seven facilities that combust sludge from mixed sources [10]. Independent of these, we received 17 struvite samples from seven other European WWTPs, six samples from six facilities that utilize struvite precipitation, and 11 samples from one plant that uses struvite crystallization. Approximately, 1 kg of each sample was collected.

2.2. Sample preparation

The samples were air-dried, divided representatively by dividing cross and grinded with a tungsten carbide vibratory disc mill. The samples were dissolved with microwave-assisted digestion (according to [22]). Approximately, 0.1 g of milled SSA was weighed in on 0.1 mg, mixed with 4 mL of concentrated nitric acid (HNO_3), 1.5 mL perchlorid acid ($HClO_4$), and 0.5 mL hydrofluoric acid (HF). Excess HF was complexed with 2.5 mL cold saturated boric acid (HBO_3). The solution was filled up with doubly distilled water to 50 mL. All reagents were purchased in analytical grade, all acids from AppliChem GmbH, Darmstadt and $LiNO_3$ (for the elemental analysis, see below) from Merck KGaA, Darmstadt. HF/$HClO_4$ digestion was chosen to achieve a complete as possible solubilization of the sample [10].

2.3. Elemental analysis

The concentrations of 18 elements were determined in 252 SSA and 17 struvite samples by means of inductively coupled plasma optical emission spectroscopy (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS), respectively. Solutions for ICP-OES measurements were spiked with 10 g/L $LiNO_3$ to even out matrix effects. ICP-OES measurements were performed from undiluted solutions according to the respective standard [23] with a Thermo Scientific IRIS Intrepid II and a Thermo Scientific iCAP 7400. ICP-MS measurements [24] were performed from tenfold dilutions with a Thermo Scientific iCAP Qc. Details on the analytical procedure, including the measurement wavelength and isotope for each element, quality control, and the limits of quantification (LOQ) can be found in our recent publications [10,25]. The LOQ refer to the actual solid matter amount, that is, the dilution due to sample preparation is already accounted for (1:500 for ICP-OES; 1:5,000 for ICP-MS). Due to the large number of results, only minimum, maximum, mean, and median values for selected elements determined in SSA and struvite samples have been presented in this paper. Detailed information on the mass fractions of 57 elements in all 252 single samples can be found in the respective publications [10,25].

3. Results and discussion

Table 1 shows the minimum, maximum, mean, and median main elemental mass fractions in the tested SSA samples, distinguished between the different sources of the incinerated sludge (municipal, industrial, or mixed). Furthermore, the respective results for struvite samples are listed, depending on where the struvite was obtained, either precipitated

Table 1
Main elemental mass fractions in SSAs and struvite products (%)

Material	Al	Ca	Fe	K	Mg	Na	P	Si
SSA municipal								
Min	2.2	6.1	1.8	0.3	0.3	0.2	3.6	4.4
Max	20.2	17.7	20.3	1.7	3.9	1.1	13.1	23.7
Mean	5.5	10.0	10.6	1.0	1.3	0.6	9.0	12.3
Median	5.3	9.8	10.2	1.0	1.2	0.5	9.1	12.1
SSA mun./ind.								
Min	1.9	8.0	4.6	0.5	0.4	0.2	2.8	2.4
Max	9.4	37.8	14.2	1.3	3.3	1.8	7.5	21.8
Mean	5.1	17.8	8.9	0.8	1.5	0.9	4.9	12.7
Median	4.9	13.2	9.0	0.7	1.6	0.9	4.8	13.4
SSA industrial								
Min	0.7	27.9	4.5	<0.006	1.1	0.4	1.5	4.3
Max	5.1	35.5	12.2	0.68	3.5	2.6	3.8	23.7
Mean	2.4	31.2	8.0	0.18	1.6	1.1	2.3	8.6
Median	2.5	31.2	8.0	0.05	1.5	0.7	2.3	7.0
Struvite (precipitation)								
Min	0.04	0.08	0.03	0.14	8.91	0.06	13.2	1.36
Max	0.39	0.72	3.17	0.31	12.6	0.09	16.5	10.8
Mean	0.22	0.52	0.92	0.19	10.7	0.08	14.7	6.26
Median	0.21	0.60	0.55	0.18	10.4	0.08	14.5	5.93
Struvite (crystallization)								
Min	0.03	0.06	0.02	0.10	9.50	0.05	12.1	0.74
Max	0.47	0.46	0.08	0.40	16.9	0.86	18.8	12.3
Mean	0.14	0.14	0.04	0.20	11.5	0.25	14.3	3.68
Median	0.04	0.07	0.04	0.15	9.81	0.09	12.5	1.41

from the sludge or crystallized from sludge liquor. The main constituents of SSA are calcium (Ca) with median values ranging from 9.8 to 31.2%, silicon (Si) (7.0–13.4%), iron (Fe) (8.0–10.6%), phosphorus (P) (2.3–9.1%), and aluminum (Al) (2.4–5.5%). Potassium (K), magnesium (Mg), and sodium (Na) are around or below 1% in all SSA samples. Even though the single values cover a broad range, the mean and median values are comparable in most cases, indicating evenly distributed single values with only single outliers. The exception is Ca in SSA from mixed municipal and industrial sources where the mean value (17.8%) is significantly higher than the median (13.2%). This can be attributed to SSA from two facilities that show Ca contents higher than 30%. This can be explained by operation procedures in the respective facilities, namely additional wastewater treatment with lime and/or gypsum residues from flue gas cleaning. Similar considerations apply to the two incineration pants for industrial sludge, which exhibit Ca concentrations around 30%. Single higher values of Al (up to 20.2%) and Fe (up to 20.3%) correlate with the P precipitation method that is applied in the respective WWTP, since chemical P elimination is usually done with either aluminum or ferric salts [8]. High Si values up to 23.7% are presumably due to residual sand and split from the WWTP [26] and/or coal that is used in some incineration facilities for auxiliary firing and that contains considerable amounts of SiO_2 [27]. The element concentrations in SSA differ significantly for some elements depending on whether the original sludge arises mainly from municipal or industrial sources. However, as described previously, the majority of these differences can be attributed to different operating procedures in wastewater treatment and sludge incineration and are not due to the source of the sludge. Only the P concentrations show a clear correlation with the source of the sludge. SSA from municipal sludge has the highest concentration with a median of 9.1%, whereas SSA from mixed sources has 4.8% and that from purely industrial sludge only 2.3%. This is to be expected, since the majority of P in the wastewater stream comes from human excreta and dishwashing agents, the latter accounting for approximately 18% of P in wastewater [1].

The main constituents of both types of struvite—aside from ammonium, which was not measured in the course of this project—are Mg (median 9.81–10.4%) and P (12.5–14.5%) in almost equimolar ratio as can be expected for magnesium ammonium phosphate. The Si concentrations vary between 1.4 and 5.9% median, whereas Al, Ca, Fe, K, and Na are all well below 1%. Concerning the single values, the differences in Mg and P mass fraction are higher for the sludge liquor-derived struvite (9.50–16.9% Mg; 12.1–18.8% P) than for struvite precipitated from the sludge (8.91–12.6% Mg, 13.2–16.5% P), resulting in greater differences between mean value and median. This indicates greater inconsistencies in the former struvite type. The single values for Si cover broad ranges in both cases (1.36–10.8 and 0.74–12.3%, respectively). In both cases, the median values are considerable lower than the mean values, indicating several upwards outliers. Again, the Si content is probably due to sand and split from the wastewater treatment [26] and the differences due to varying operational states, especially rain events and dry periods. Struvite precipitated from the sludge contains significantly more Si than that crystallized from the sludge liquor (5.93 vs. 1.41%). This is presumably due to the different stages of the WWTP in which the struvite is produced. The sludge liquor remains after dewatering and is supposed to contain less solid components like sand or hardly soluble inorganic salts than the sludge before the dewatering. Thus, struvite crystallized from the liquor contains less Si than that precipitated from the sludge. This is in accordance with the mass fractions of the other elements. K and Na mass fractions are very similar for both types of struvite, which is to be expected since most of their compounds show very good water solubility. Thus, there should be no observable influence of the process step. On the other hand, Al, Ca, and Fe mass fractions in the sludge-based products are 4–14 times higher than in the struvite that was precipitated from the liquor. This is presumably due to insoluble salts like for instance phosphates that remain in the sludge and are incorporated in the resulting struvite.

Table 2 shows the limit values for trace elements in fertilizer as stipulated in the German fertilizer ordinance [28] and the respective mass fractions measured in the SSA and struvite samples. Additionally, Th and U values are also stated, albeit they are not regulated in the ordinance. The most abundant trace metals found in SSA are Zn (median values: 1,132–2,716 mg/kg), Cu (300–832 mg/kg), and Cr (156–817 mg/kg), followed by Pb (75.0–192 mg/kg) and Ni (70.7–130 mg/kg). The single values for all tested trace elements vary over a broad range. However, the mean and median values of As (in SSA from industrial and mixed sludge), Cd, Hg, Th, and U are comparable, indicating evenly distributed single values. Mean and median values differ significantly for the other elements, in particular Cu (mixed sources; mean: 942 mg/kg, median: 688 mg/kg), Zn (mixed; 2,192 vs. 1,637 mg/kg), Pb (mixed 192 vs. 144 mg/kg), Cr (industrial; 642 vs. 817 mg/kg), and Zn (industrial; 1,780 vs. 1,132 mg/kg). This shows that there are considerable upward outliers exceeding the respective mean values significantly, as for Cu (3,467 mg/kg), Zn (5,515 mg/kg), Pb (1,112 mg/kg), and Cr (1,502 mg/kg). The same applies to As (97.0 mg/kg), Cd (80.3 mg/kg), and Hg (3.6 mg/kg). According to the operators of the incineration plants, the high values can be attributed to industrial processes in the catchment area of the WWTP that produced the respective sludge [10]. Although there are differences in trace elemental mass fractions relating to the source of the incinerated sludge, there is no clear trend observable. This is presumably due to the fact that industrial dischargers can be found in municipal areas also. Thus, even though a SSA is classified as derived solely from municipal sewage sludge, residues from industrial processes are possible that could lead to high mass fractions of the respective elements. Hg mass fractions are below 1 mg/kg with the exception of SSA from mixed sources (median 1.5 mg/kg). These SSA originate partially from large incineration plants that use coal for auxiliary firing. Since coal contains considerable amounts of Hg [29] this is the likely cause for the elevated Hg contents.

All median mass fractions are well below the respective limit and labeling values of the German fertilizer ordinance [28] with the exception of Cr in SSA from industrial sludge (817 mg/kg). However, it is obvious that SSA from several incineration plants exceed these values, in some cases significantly. Thus, an additional treatment of SSA is necessary to reduce the heavy metal content prior to possible use in fertilizer production. Several procedures have been developed to achieve this, by the majority wet-chemical or thermochemical processes [12]. The median values of U (2.4–5.9 mg/kg) and Th (4.8–9.3) in SSA are generally low and up to several orders of magnitude lower than the respective mass fractions in phosphate rock (U up to 660 mg/kg, Th up to 220 mg/kg [5]). The same applies to Cd that also shows significantly lower concentrations in SSA (median 0.5–3.1 mg/kg) than in phosphate rock (up to 110 mg/kg [4]). Thus, the use of SSA for fertilizer production as supplement or

Table 2
Trace elemental mass fractions in SSAs and struvite products (mg/kg)

Material	Cr	Ni	Cu	Zn	As	Cd	Hg	Pb	Th	U
Limit values fertilizer ordinance	300[a]	80	900	5,000	40	50[b]	1	150	n/a	n/a
SSA municipal										
Min	79.2	8.2	225	844	4.4	0.02	0.1	54.2	1.0	2.2
Max	1,088	272	2,477	4,930	124	14.1	3.6	363	19.2	25.5
Mean	206	79.0	980	2,773	18.3	2.7	0.7	145	5.2	7.0
Median	156	70.7	832	2,716	13.4	2.7	0.5	118	5.1	5.9
SSA mun./ind.										
Min	58	38.6	287	588	6.2	0.9	0.1	22.4	1.7	2.7
Max	773	1,356	3,467	5,515	97.0	80.3	3.0	1,112	8.0	6.1
Mean	300	184	942	2,192	17.0	5.5	1.4	192	4.6	3.9
Median	205	130	688	1,637	15.1	3.1	1.5	144	4.8	3.7
SSA industrial										
Min	85.0	46.3	162	552	4.2	0.3	0.1	<3.5	1.2	1.58
Max	1,502	484	523	4,101	26.8	3.8	0.6	144	19.3	5.0
Mean	642	117	304	1,780	12.3	0.8	0.4	65.5	7.4	2.6
Median	817	86.2	300	1,132	11.9	0.5	0.4	75.0	9.3	2.4
Struvite (precipitation)										
Min	4.6	<2.7	4.1	<59	<0.58	<0.10	<0.10	<3.5	<0.15	<0.16
Max	31.4	18.7	53.8	118	3.54	<0.10	1.98	36.5	0.36	0.17
Mean	15.7	8.99	32.1	74.3	1.50	<0.10	0.84	21.1	0.20	0.16
Median	13.2	8.36	31.1	64.9	0.58	<0.10	0.67	22.7	0.17	0.16
Struvite (crystallization)										
Min	2.9	<2.7	4.6	<59	<0.58	<0.10	<0.10	<3.5	<0.15	<0.16
Max	8.75	4.9	14.4	<59	1.23	<0.10	2.39	29.6	0.22	0.18
Mean	5.39	2.9	7.77	<59	0.63	<0.10	0.33	9.0	0.16	0.16
Median	4.43	2.7	7.51	<59	0.58	<0.10	0.11	3.5	0.15	0.16

[a] Labeling value.

[b] Calculated as mg/kg P_2O_5.

replacement of phosphate rock could reduce the Cd, Th, and U intake to farmlands.

Both types of struvite tested in this work show very low trace element mass fractions, many even below the respective limit of quantitation (LOQ). However, struvite precipitated from the sludge shows higher mass fractions, up to sixfold excess compared to the struvite crystallized from sludge liquor. This was already observed for the main elements and is presumably due to the same reason. Both types of struvite are produced at different stages of the WWTP and it can be assumed that struvite crystallized from the liquor is less contaminated than that precipitated from the sludge. Anyway, both types of struvite are well below all limit and labeling values of the fertilizer ordinance and have U and Th mass fractions that are even lower than in SSA (median values around 0.16 mg/kg). The struvite is perfectly suited for fertilizer use from that point of view. Furthermore, these processes reduce maintenance and operating costs of wastewater treatment by means of avoided incrustations and improved dewatering. However, since the P recovery rate of the struvite precipitation and crystallization methods is low (7–11% of the overall P content in the sludge [18,19]), the majority of P remains in the sludge. On the other hand, P recovery from SSA allows for P recovery rates of up to 90% [17] but requires additional treatment to reduce heavy metal contents. Thus, P recovery from the sludge or from SSA is inevitable to achieve an as complete as possible P recovery from the wastewater stream.

4. Conclusions

This study tested monoincinerated SSAs from varying sources (municipal, industrial, and mixed sludge) and struvite (precipitated from sludge and crystallized from sludge liquor) for their main elemental composition and trace element mass fractions. All samples show considerable amounts of phosphorus (P) and have a significant potential for P recovery from the wastewater stream. Median values of SSA trace element mass fractions are below the limit values of the respective fertilizer ordinance but there are numerous single samples that exceed these values. Thus, an additional treatment to reduce the heavy metal content is necessary before SSA can be used for fertilizer production. There is no clear trend of the elemental mass fractions related to the source of the sludge. All struvite samples show very low heavy metal contents, even though the struvite precipitated from the sludge shows slightly elevated mass fractions compared to struvite crystallized from sludge liquor. Since the P recovery rate of struvite is low compared to that from SSA (7–11% vs. 90%), P recovery from SSA is necessary for an extensive P recovery from the wastewater stream.

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