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Recovery of phosphorus as struvite from sewage sludge and sewage sludge ash

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ABSTRACT

The growing demand for phosphate fertilizers, with the simultaneous high exploitation of phosphate rock—a raw material for the production of fertilizers—has contributed to the search for alternative sources of this element. Sewage sludge contains huge reserves of unused phosphorus. Around 90% of phosphorus compounds that are disposed in sewage treatment plants are contained in sewage sludge. Phosphorus compounds can be recovered directly from sewage sludge or, after thermal processing, from ashes or slag. The use of sewage sludge in agriculture is limited by the content of heavy metals and polycyclic aromatic hydrocarbons, which can migrate throughout the food chain. In the presented work, an attempt was made to recover phosphorus in the form of struvite from sewage sludge and sewage sludge ashes. For this purpose, the optimal concentration of ions necessary to precipitate struvite was determined for which the highest mass of precipitates was obtained. The analysis of the obtained sediments was carried out using the X-ray spectra analysis. The highest mass (6.70 g) was obtained for the ion configuration of Mg^{2+}, 40mg/L, and NH^{4+}, 300mg/L. Also, at this ion configuration, the highest percentage recovery of phosphorus of 19% was obtained from sewage sludge. The highest amount of recovered phosphorus of 54% was obtained after 2 h of extraction with 0.4 mol/L solution from ashes.

Keywords: Phosphorus; Sewage sludge; Heavy metals

1. Introduction

Phosphorus (P) is one of the basic elements needed for all living organisms and food production [1,2]. Phosphorus used for fertilizers exists in the form of P_2O_5. It is estimated that 6,700 million tons of phosphate rock exist as P_2O_5 (with phosphorus concentrations ranging from 28% to 39%) in reserves that could be economically mined, and about 80% of phosphate produced is currently used for fertilizers [3]. However, it is also estimated that more than a half of all phosphate ore deposits will have been depleted in the next 60–70 years [4–6]. In addition, the environment is being degraded in areas where phosphorus is extracted in large quantities. Therefore, the methods of effective recovery of phosphorus from waste, such as municipal wastewater, sewage sludge, or waste from the meat industry should be identified. There are large reserves of unused phosphorus deposited in sewage treatment plants. About 90% of phosphorus compounds reaching the sewage treatment plant are stored in the sludge. Phosphorus compounds can be recovered directly from sewage sludge or, after thermal treatment, from ash or slag [7].

Total phosphorus content in dry matter of sewage sludge or ash from sewage sludge ranges, depending on different sources, from 2.62% to 13.4% (Table 1). Higher phosphorus contents can be observed for more compacted sewage sludge and ashes.

Table 2 presents a percentage content of phosphate ions in ashes over the temperature range of 600°C–950°C, with combustion time of 3 h. It can be observed that the increase in temperature leads also to the increase in the content (%) of PO_4^{3-} ions. Therefore, it can be concluded that ashes

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Table 1
Content of total phosphorus in sewage sludge

Type of sewage sludge	Phosphorus content percentage in dry matter	References
Dewatered sewage sludge	3.42	N.Y. Acelasny et al. [8]
Dried sewage sludge	5.1–7.6	M. Atienzae-Martınez et al. [9]
Ashes from sewage sludge	8.88	B.K. Biswas et al. [10]
Ashes from sewage sludge	12.9–13.4	P. Guedes et al. [11]
Raw sewage sludge	4.65	K. Guney et al. [12]
Ashes from sewage sludge	8.0–11.0	S. Petzet et al. [13]
Ashes from sewage sludge	3.3–6.0	H. Xu et al. [14]
Dewatered sewage sludge	2.62	W. Shi et al. [15]

Table 2
Content (%) of phosphate ions in ash at increasing temperature (°C) [17]

Ash incineration temperature, °C	Ash mass for mineralization, g	Content in ash, %	
		PO_4^{3-}	Fe^{3+}
600	1.002	21.4	16.9
850	1.003	21.9	14.5
930	1.002	23.0	17.0
950	1.005	23.9	18.4

represent a valuable phosphorus-rich source and are much more attractive for agricultural applications as, for example, fertilizers [16].

Difficulties in the processes of recovery of phosphorus compounds from communal sewage sludge can be connected with the content of microcontaminants in the form of heavy metals. The content of various microcontaminants in sewage sludge is also affected by the processing methods, such as dewatering and conditioning methods [18–22].

Most of the methods of phosphorus recovery are based on solving the substrate in the sulfuric acid, in which heavy metals are also solved, consequently forming salts of phosphorus compounds and reducing the efficiency of phosphorus recovery. The increase in the effectiveness of the methods used to recover phosphorus should be also focused on the removal of heavy metals [23].

The literature data show that it is substantially advantageous to use the methods to recover phosphorus from ashes or slag from sewage sludge due to higher concentration. Ash that is generated after combustion of sewage sludge is composed mainly of Si, Fe, Al, Ca, and P, with substantial content of heavy metals, which makes it impossible for agricultural applications [24]. Ash from sewage sludge is an interesting and rich source of phosphorus (content of phosphorus in ash can reach even 11%–13.4%), whereas there are more than 10 applicable and economically justified methods of phosphorus extraction [25,26]. One of the simplest and cheapest methods is reaction with phosphoric acid. This yields a fertilizer which, however, remains to show above-normal levels of heavy metals [27,28]. Depending on the method, it is estimated that phosphorus recovery ranges from 80% to 90% [8,14,12].

Compared with traditional fertilizers, struvite ($MgNH_4PO_4{\cdot}6H_2O$) is characterized by a high P_2O_5 content (58%). Struvite-based phosphate compounds are released to the soil solution slowly, which makes this mineral a valuable product and safe for plants. Another important fact is that struvite crystals contain two important macroelements necessary for the growth and development of plants: phosphorus and nitrogen. This mineral is characterized by low solubility, that is, it is the slow-acting fertilizer, solves very slow in the soil and the plant can stimulate the intensity of collecting the elements by itself. The biggest natural source of struvite is decomposed organic materials. It is the mineral with crystalline structure, discovered in the middle of the 19th century [29], and its synthesis is presented by the following chemical equation [30]:

$$Mg^{2+} + NH_4^+ + PO_4^{3-} \rightarrow (NH_4)Mg[PO_4] \cdot 6H_2O \tag{1}$$

As results from this equation, generation of struvite requires mole ratio as follows [31]:

$$1\,(Mg^{2+}) : 1\,(NH_4^+\,) : 1\,(PO_4^{3-}) \tag{2}$$

Currently, extraction of struvite remains cost-effective. However, natural sources of phosphorus are being gradually depleted, which stimulates explorations of alternative sources of phosphorus and opportunities to use struvite as a fertilizer. Due to the depleting sources of apatites and phosphorites used for phosphorus fertilizers, some EU countries such as Sweden, Switzerland, the Netherlands, or Denmark are planning to introduce new regulations concerning recycling of phosphorus from various sources, especially secondary. The natural source of struvite is decomposed organic materials. Struvite can be precipitated in sewage sludge after the processes of anaerobic fermentation in liquid sludge from animal farming and the sludge from biological sewage treatment [28].

The aim of the study was to analyze on the effectiveness of the recovery of phosphorus compounds in the form of struvite from fermented sewage sludge and ashes of fermented sewage sludge. An optimal concentration of selected ions (Mg^{2+}, NH_4^+) at which maximal sludge mass was precipitated was determined in the study. The X-ray spectra analysis was used to determine the content of sludge generated in the laboratory environment, which allowed for determination of the surface content.

2. Experimental part

2.1. Methodology

2.1.1. Test stand and experimental procedure

The experiment was composed of two stages: struvite precipitation in supernatant liquor of fermented sewage sludge and struvite precipitation from ashes of fermented sewage sludge. Variable ion concentrations were used in the first stage of the examinations: Mg^{2+} in the form of the solution of $MgSO_4{\cdot}6H_2O$ (10, 20, 30, 40, 50, 60 mg/L) and variable concentrations of NH_4^+ ions in the form of NH_4Cl (200, 300, 400, 500 mg/L), prepared based on the distilled water.

At the second stage of the examinations, ashes were prepared in the laboratory environment by ignition of fermented sewage sludge at temperature of 850°C for 3 h. The next step was extraction of the phosphorus compounds: 0.5 g of ashes were sampled and ground in the mortar and, with addition of 75 cm^3 of hydrochloric acid, shaken in order to extract the phosphorus compounds. The molarity of the hydrochloric acid was 0.2, 0.4, and 0.8 M, with shaking times of 1, 2, and 4 h, respectively. Struvite precipitation was performed in the supernatant liquor. Ion concentrations were as follows: Mg^{2+} in the form of $MgSO_4{\cdot}6H_2O$ solution, 15 mg/L, and NH_4^+ ions in the form of NH_4Cl, 500 mg/L. Conditions of the reaction during the first and the second stage of the examinations were the same: temperature of 20°C–22°C, pH was set at equal level of 9.5. The constant pH used in the experiment was determined based on the literature data which demonstrate that at this level, struvite residue can be observed for almost any concentration of ammonium and phosphate ions if adequate amount of magnesium ions is ensured.

2.2. Substrate

The substrate for the study was fermented sewage sludge and ashes from fermented sewage sludge. The sludge was sampled from municipal wastewater treatment plant with a capacity of about 45.000 m^3/d (31h4 835 PE), which is a mechanical and biological treatment plant with increased biogen removal. Selected samples were subjected to analysis and technological research on the day of collection. All determinations were made using a three-point repetition.

2.2.1. Physical and chemical analyses

The following physicochemical determinations were made:

- dry matter [32],
- ammonium nitrogen [33],
- total phosphorus [34],
- phosphates, bioavailable phosphorus [35],
- organic carbon [34],
- heavy metals [36],
- determination of pH [37].

2.2.2. Examination of the surface composition of the sludge

Bruker D8 Advance X-ray Diffractometer (XRD) was used to examine the precipitated sludge. The X-ray photoelectron spectroscopy (XPS) methodology was employed. The methodology uses the effects of interactions of electrons, photons, ions, or neutral particles with atoms of the examined specimen. XPS analyzes the electrons that escape from the material being analyzed due to irradiation with monochromatic soft X-ray radiation (<8 keV). Kinetic energy of photoelectrons allows for identification of elements and analysis of their binding. Furthermore, photoelectron intensity allows for the evaluation of concentrations of elements and the analysis of contribution of various bindings. All analysis was performed in triplicates. The results are presented in the tables and figures as the arithmetic means with standard deviation.

3. Results and discussion

3.1. Characteristics of the substrate

Sewage sludge was characterized by hydration at the level of 97%, whereas the content of organic substance in dry matter was 1.06%. Table 3 presents a general characterization of fermented sewage sludge used in the study.

The values obtained for heavy metals do not exceed permissible standards provided in the Ordinance of the Minister of Environment as of February 6, 2015, on municipal sewage sludge. The results of the analysis of the content of heavy metal in sewage sludge are shown in Table 4.

In the case of ashes, the analyses were made with regard to the content of various forms of phosphorus. The value

Table 3
Physical and chemical parameters of fermented sewage sludge

Factor	Value	Standard deviation
Dry matter, %	2.74	±0.03
Loss on ignition, %	1.06	±0.01
Residue on ignition, %	1.68	±0.02
Water content, %	97	±0.7
Ammonium nitrogen, mg NH_4/L	760	±3.5
Total phosphorus, mg P/L	17.52	±0.4
Phosphates, mg PO_4^{3-}/L	53.72	±0.7
Organic carbon, mg/L	242.47	±4.4
pH	7.8	±0.07

Table 4
Content of heavy metals in fermented sewage sludge

Heavy metal	Value/standard deviation
Cd (cadmium), mg/kg d.m.	0.18±0.20
Cr (chromium), mg/kg d.m.	5.88±0.58
Cu (copper), mg/kg d.m.	4.54±0.17
Ni (nickel), mg/kg d.m.	4.59±0.19
Pb (lead), mg/kg d.m.	2.19±2.26
Zn (zinc), mg/kg d.m.	27.9±2.43

of total phosphorus for ashes was 24.08 mg P/L, including phosphates 73.83 mg PO_4/L and available phosphorus 5.22 mg P/L (Table 5). Analysis of heavy metal content in ashes is presented in Table 6. The contents of chromium, nickel, and zinc were exceeded with respect to the standards provided by the Ordinance of the Minister of Environment as of February, 6, 2015 on municipal sewage sludge. The values obtained for cadmium, copper, and lead are within the standard. The ashes cannot be used in agriculture, for soil reclamation of agricultural and non-agricultural areas and production of compost for growing plants not designed for consumption and fodder production [38]. The results of physical and chemical analysis for ashes of fermented sludge are contained in Tables 5 and 6.

3.2. Struvite recovery from the supernatant liquor from fermented sewage sludge

Fig. 1 presents the mass of precipitated sludge in the liquor of fermented sludge. The highest mass (g) is obtained for NH_4Cl concentration of 300 mg/L for each case from $MgSO_4{\cdot}6H_2O$. The highest sludge mass (6.70 g) was obtained only for the concentration of $MgSO_4{\cdot}6H_2O$ of 40 mg/L.

In Fig. 2, the average percentage recovery of phosphorus from the supernatant water is presented. The highest recovery values of phosphorus compounds were recorded at the concentration of 40 mg Mg^{2+}/L ions at any concentration of NH_4^+ ions. For a concentration of 300 mg NH_4^+/L, the highest yield of 19% was obtained. The lowest value of 7% was obtained for 20 Mg^{2+} mg/L and 400 mg NH_4^+/L concentrations. The low efficiency of recovery of phosphorus compounds obtained may be associated with impurities in the form of, inter alia, heavy metals.

Table 5
Physical and chemical analyses of ashes from fermented sewage sludge

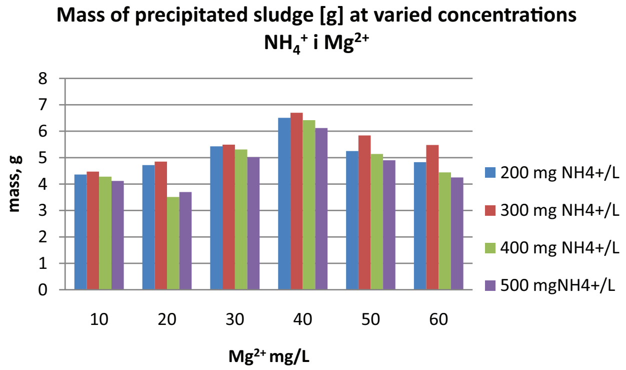
Factor	Ashes from fermented sewage sludge/standard deviation
Total phosphorus, mg P/L	24.08±0.6
Phosphates, mg PO_4/L	73.83±0.5
Available phosphorus, mg P/L	5.22±0.4

Table 6
Content of heavy metals in ashes from fermented sewage sludge

Heavy metal	Value/standard deviation
Cd (cadmium), mg/kg d.m.	10.7±2.80
Cr (chromium), mg/kg d.m.	927.9±145
Cu (copper), mg/kg d.m.	675.4±160
Ni (nickel), mg/kg d.m.	490.9±72.6
Pb (lead), mg/kg d.m.	363.3±198
Zn (zinc), mg/kg d.m.	5228.9±884

3.3. Struvite recovery from the ashes of fermented sewage sludge

Fig. 3 presents the results for the precipitated sludge mass. The highest mass (6.524 g) was obtained for the concentration of HCl of 0.4 mol/L and after 1 h of shaking. The lowest mass (5.683 g) was reached for the concentration of HCl of 0.8 mol/L and after 4 h of shaking. It can be concluded based on the results that longer the shaking time, lower the mass of the precipitated sludge that can be obtained. Furthermore, considering the concentration of hydrochloric acid, the highest mass was obtained for the concentration of 0.4 mol/L.



Mass of precipitated sludge [g] at varied concentrations NH_4^+ i Mg^{2+}

Fig. 1. Mass of precipitated sludge (g) at varied concentrations NH_4^+ i Mg^{2+}.

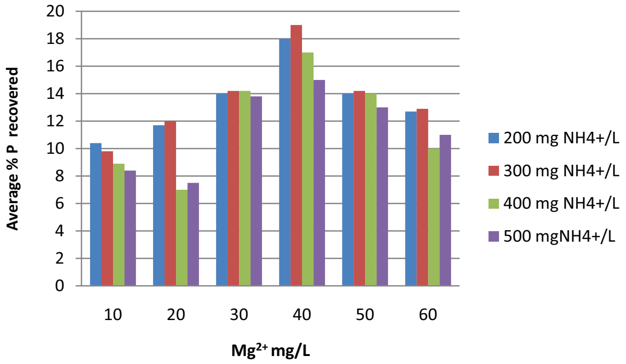


Fig. 2. Amount of phosphorus recovered (%) at varied concentrations NH_4^+ i Mg^{2+}.

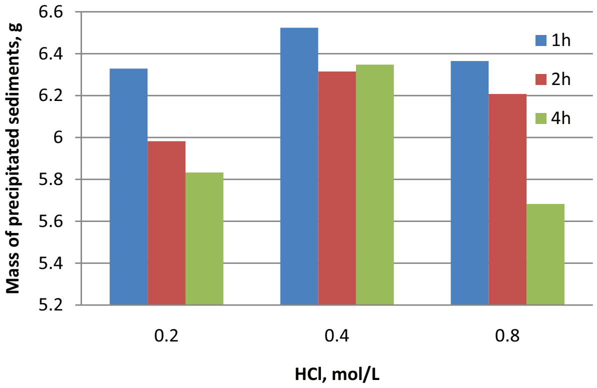


Fig. 3. Precipitated sludge mass (g) in ashes.

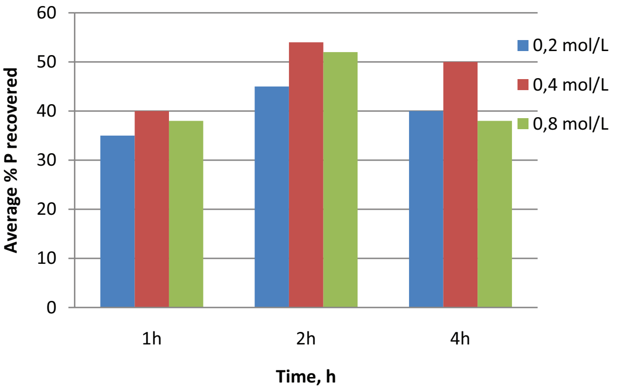


Fig. 4. Amount of phosphorus recovered (%) at different residence times (h).

Fig. 4 shows the results obtained from the leaching of phosphorus from sewage sludge at different residence times. An increase in retention time increased the amount of phosphorus recovered. The highest amount of P recovered was 54% at 2 h and it decreased thereafter. A possible reason would be that there might be other reactions taking place or processes such as crystallization and precipitation of insoluble compounds such as iron phosphates thus forming chemical bonds which are harder to break [39]. High concentrations of heavy metals in the ash can cause pollution thus reducing the degree of phosphorus release [40].

3.4. Examination of the surface composition of the sludge

The structure of the collected precipitants was evaluated using XRD. The results of the analysis of the surface composition of samples from laboratory tests also revealed a multitude of types of compounds precipitated in sediments. The composition of ash samples is identical, and the difference in composition is characterized by precipitated deposits from fermented sewage sludge. The result of the analysis indicates that in the ash sludge is a mixture of struvite, its amorphous forms, but also calcium compounds and trace amounts of heavy metals. These are $CaCO_3$ and $Ca_3(PO)_2$. The obtained results are in line with the results of a study by Crutchik and Garrido [41], who found that in certain sediments under certain conditions, crystalline struvite was precipitated, and in other cases, decahydrate crystals of magnesium phosphate and amorphous calcium and magnesium phosphates. A greater diversity of composition was characterized by precipitated sludges from the supernatant water of fermented sewage sludge. XPS analysis showed the participation of calcium compounds in sediments precipitated, however, the percentage of atomic connections to which struvite was classified is small and amounts to less than 4%, for ashes for sewage sludge, respectively, 0.5%. This analysis did not give an unambiguous answer as to the composition of the precipitates. Obtained spectra allowed to state that sediments precipitating in wastewater treatment plants commonly called the struvite term are in fact a mixture of various compounds composed of ions found in solutions [42]. Taking into account the fact that the composition of the sedimentary liquid is very diverse and varies over time, sediments form with a significantly different composition. In Hermassi et al.'s [43] research on phosphate recovery and stabilization from urban sewage sludge anaerobic digestates (2018), XRD analysis of the powdered mixture did not detect any phosphate mineral phase, because of phosphate minerals precipitated are below of the limit of detection [43]. Thoroughly tested, the influence of various physicochemical process parameters on kinetics and growth mechanism of struvite crystallisation [44,45], in many cases, cannot be confirmed by X-ray analysis. In sewage sludge, P is associated in the form of microbial cell structures, which reduces the efficiency of P recovery. In order to increase the recovery efficiency of P, it is necessary to destroy organic matter [46].

Studies carried out by Xu et al. [14] confirmed the significant efficiency of struvite recovery (97%) in the course of sequential extraction: first heavy metals followed by precipitation of struvite.

The low recovery efficiency of phosphorus compounds in the form of struvite from sewage sludge can thus be connected on the one hand with the presence of phosphorus compounds in the organic form (cellular structures) and on the other hand with the presence of metals and other compounds. It seems that in the case of ashes, the problem with the efficiency of phosphorus recovery concerns the presence of micro-pollutants.

4. Conclusions

The study attempted to precipitate phosphorus compounds, including struvite from fermented sewage sludge and ashes obtained by drying and incineration of fermented sewage sludge. The study attempted to recover phosphorus compounds in the form of struvite. Presence of heavy metals in ashes and sewage sludge leads to the contamination of samples, thus affecting the quality and limiting their further use as fertilizers. The findings of the study lead to the following conclusions:

1. During precipitation of sludge from supernatant liquor of fermented sewage sludge, the highest mass (6.70 g) was obtained for ion configuration of Mg^{2+}, 40 mg/L, and NH_4^+, 300 mg/L. Also at this ion configuration, the highest percentage recovery of phosphorus of 19% was obtained.
2. In the case of sludge precipitation from ashes, the highest mass (6.524 g) was obtained after 1 h of shaking in 0.4 mol/L solution of HCl. However, the highest amount of recovered phosphorus of 54% was obtained after 2 h of extraction with 0.4 mol/L solution.
3. Based on the results of the XPS analysis of the sediment structure, the efficiency of phosphorus recovery from sewage sludge was found to be lower than from sewage sludge ashes. The obtained content of phosphorus compounds in the form of struvite for pit hole deposits was 0.5% and for ashes 4%, respectively.
4. The low efficiency of the recovery of phosphorus in the form of struvite from sewage sludge and their ashes confirmed in the studies confirms the negative impact of heavy metals. Therefore, the necessary preliminary step in the phosphorus recovery procedure is first the removal of heavy metals, often sequential and then the recovery of phosphorus.

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