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ASSESMENT OF TOTAL PHOSPHORUS CONTENT AND ITS FRACTIONS IN MUNICAPAL SEWAGE SLUDGE

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Abstract

Phosphorus (P) is necessary for the proper functioning of organisms and is widely used in many fields of human activity, however, the ores from which it is obtained are not inexhaustible. One of the unconventional sources of phosphorus are sewage sludge. However, the cost-effective recovery of phosphorus from this source depends on the total P concentration and the chemical form in which P is bound. The aim of this study was to evaluate the content of phosphorus and its fractions in municipal sewage sludge generated at different stages of wastewater treatment in the plant operating on the basis of activated sludge technology. Three types of the sludge: excess sludge, anaerobically digested dewatered sludge, and sludge after drying process were used in the study. The content of total phosphorus and its fractions were determined using the Chang-Jackson sequential extraction method with subsequent modifications by Peter-sen and Corey. The total phosphorus content in the examined sludge ranged from 50-70g/kg d.m. The highest amount of phosphorus (over 60%) was concentrated in the fraction bound in aluminium phosphate (fraction II). The distribution of the fractions in the studied sludge was arranged in the following order: P-Al>P-red.>P-Fe>P-lab.>P-Ca>P-ocl. There were no significant differences in the distribution of phosphorus in particular fractions depending on the sludge type, while the time of sludge sampling significantly affected the proportion of phosphorus in fraction II, which is considered to be an important source of plant-available phosphorus.

Keywords: Phosphorus recovery; Phosphorus fractions; Sewage sludge, Natural resource conservation

Introduction

The development of civilization brings whole range of threats to the environment. Many pollutants migrate into natural ecosystems and agroecosystems [1, 2]. On the other hand, the high demand for raw materials used in many industries leads to the depletion of their resources. One of the chemical elements which finds numerous industrial applications, and is introduced to the environment in large amounts, is phosphorus. Environmental pollution with compounds including phosphorus is still increasing [3]. This element enters the environment mainly from municipal wastewater and runoff from farmland. It primarily affects the process of eutrophication of surface waters [4]. This process leads to a excessive growth of algae biomass, which increases water turbidity and decrease in light transmission through the water body, leading to death of photophilic aquatic organisms. Additionally, an intense decomposition of high amount of organic matter which are sedimented on the bottom of the reservoir leads to oxygen depletion, which generally cause deterioration of water quality, i.a. related to the

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emission of odour compounds, such as hydrogen sulfide. These changes contribute to the death of large aquatic organisms such as fish. The limiting concentration of phosphorus, above which intensive algal growth is observed, is 0.1mg $P\text{-}PO_4/dm^3$.

Although the excessive concentration of phosphorus is a real problem, this element plays a key role in the environment, as an essential nutrient for all the organisms. It is a basic component of proteins, and compounds building the vertebrate skeletons, and takes part in the metabolism of sugars and fats. In the form of phosphates, it is a component of biological molecules: nucleic acids DNA and RNA and compounds transferring energy at the molecular level, such as ATP. It is essential in the regulation of biochemical reactions in the cells (phosphorylation). Phospholipids, in which phosphorus is found, are an essential structural component of cell membranes. Human and animal bones are largely composed of tricalcium phosphate, which causes their rigidity. For example, human body contains 1kg of phosphorus, 75% of which is found in bones and teeth. Phosphates also act as buffers in the blood and urine, allowing the pH to remain constant. Phosphorus deficiency in humans causes dysfunction of the central and peripheral nervous systems and abnormal blood cell function. Daily human consumption and excretion of phosphorus ranges from 1 to 3g [5]. Phosphorus also affects the development of plant roots, maturation and development of seeds. Concentraion of phosphorus in plant-available forms in soil ranges from 0.0002 to 0.008% P and this amount is insufficient to cover their nutrient requirements, which creates the need for soil fertilization with this nutrient. For example, about 150 thousand tons of phosphorus derived from non-renewable sources enters the food chain in Poland, each year. About 11 thousand tons are discharged into the Baltic Sea. The biggest amount of phosphorus is introduced to the environment with mineral fertilizers, and then with fodder, where phosphorus compounds are introduced as mineral additives. The largest amount of phosphorus in the food chain is concentrated in soil, followed by waste [6].

The main sources of phosphorus, which are used for production of phosphate fertilizers, are deposits of volcanic (magmatic) origin, whose main mineral is fluoroapatite, and deposits of sedimentary origin, which were formed by precipitation of calcium phosphate from sea water, in which the main mineral is francolite. Most of the world's identified phosphate reserves come from Morocco, the Sahara, China, the United States and Russia. In Europe, small amounts of phosphate are mined in Finland. The demand for phosphate raw materials in Poland is covered by imported raw materials. The American Deposit Classification Service estimates that world recoverable phosphate reserves are about 50-65 billion tons. These resources in the current mining conditions should be sufficient for about 300 years but taking into account the current economic and technical conditions, the amount of available phoshates is about 18 billion tons, and it will be exhausted after about 130 years. Approximately 90% of mined minerals are processed into phosphate fertilizers, the consumption of which will grow at a rate of 1.5-3.6% per year [7, 8]. Limitation in phosphorus sourcing will lead to reduced food production on the global scale [8-10]. Data published by the International Fertilizer Development Center (IFDC) demonstrate that exploiting the phosphate reserve base with new advanced technologies, will result in a significant increase in the cost of extraction [11]

Over the past few decades, the world population has tripled while arable land has increased by 12%, and arable land per capita has declined from nearly 0.5ha in 1950 to 0.2ha. Due to the fact that about 50% of crop growth is determined by mineral fertilization, the demand for fertilizers and their use, according to the Food and Agriculture Organization of the United Nations, will increase due to the expected increase in population to 9 billion in 2050. Currently, population growth is observed mainly in developing countries, and in these areas the demand for fertilizers, including phosphate fertilizers, will significantly increase. The main reasons for this increase in demand is growing production of livestock and biofuels. Apart from the decreasing quantity of available phosphate rock reserves, their quality is the second important factor that limits their use for production of phosphate fertilizers. These raw materials contain trace elements, including cadmium, lead, chromium and uranium. Limited resources of raw materials for production of phoshorus fertilizers, and their low quality, force to seek the other sources of this element, such as organic waste, including sewage sludge. Municipal sewage sludge can become a secondary source of phosphorus. There is much more of this nutrient in sludge than in natural fertilizers and the average content is about 3% DM [12].

Phosphorus in municipal wastewater comes from three primary sources: (1) human physiological functions - 30-50%, (2) detergents - 50-70%, and (3) industry - 2-20%. The average concentration of total phosphorus in urban wastewater in Poland is 9-10mg P/dm^3, of which 50-80% are orthophosphates [13]. In rural wastewater, the concentration of phosphorus, as a result of lower water consumption, may be higher, usually it ranged from 10 to 20mg P/dm^3, although it can reach up to 25.8mg P/dm^3 [14]. As a result of the wastewater treatment process using activated sludge, phosphorus from wastewater passes to sewage sludge [15-17]. In addition, the coagulation process which is often used to precipitate phosphorus from wastewater to decreasing the risk of eutrophication of the water body, which is the receiver of the treated wastewater, also increases the phosphorus concentration in sewage sludge. The amount of sewage sludge produced in the world is still increasing. For example, in Poland the amount of sewage sludge generated in municipal sewage treatment plants increased by 62% when compared the years 2000 and 2018 [18]. Taking into account the high phosphorus content in the sludge, this can be a significant source of phosphorus for crop plants. It turns out that the assimilability of phosphorus from properly processed sludge can be higher than from NPK fertilizers. Therefore, more and more attention is paid to treating sewage sludge as one of the most important sources of recovery of phosphorus and its compounds. Especially that sludge is quite a troublesome waste when it comes to agricultural use due to the content of heavy metals, organic pollutants and biological hazard related to presence of parasite eggs [19, 20]. Becouse of high moisture content, dewatering and transportation of sludge are energy-consuming and costly processes.

A necessary condition for use of the sludge as a phosphorus resource is maintaining strict control procedures which is associated with costs. However, the important advantage of such solution is the possibility of providing agriculture with phosphorus derived from renewable source. Regardless of cost, such a production of phosphorus can stabilize and limit the impact of the instability of global phosphates prices on the products made from them. It should be considered as a priority to prefer methods limiting the use of phosphorus from primary sources in favour of recovery of phosphorus from waste derived organic matter, such as human and animal faeces, sewage, sludge, green waste or ashes produced from them. This solution should be seen as a commercial opportunity and an environmental benefit, rather than an onerous and costly investment [21]. Before suitable methods for phosphorus recovery can be developed on the technical scale, it is necessary to know in which fractions phosphorus in sludge occurs. Knowledge of the occurrence of fractions of this important nutrient for living organisms contained in sewage sludge is important to determine its bioavailability.

The aim of the study is to evaluate the content of total phosphorus and the particular chemical forms of this element in the municipal sewage sludge produced at different stages of sludge processing in a treatment plant, in order to assess the possibilty of phosphorus recovery from sludge in a wider scale.

Experimental section

Materials

The study was conducted between 2019 and 2020, on the samples of excess sludge, anaerobically digested dewatered mixed (primary and excess) sludge, and the sludge after drying process, which were collected in the municipal wastewater treatment plant (WWTP) in

Białystok (Poland). Six samples of each type of sludge were taken out (to obtain representative sample) in four terms: corresponding to spring, summer, autumn and winter.

The wastewater treatment plant in Bialystok was put into operation in 1994 (although construction began in the 1970s). Activated sludge technology with removal of organic compounds is used there. Twenty years ago the plant was modernized to increase the reduction of biogenic compounds. Its capacity is 100,000m^3/day. The WWTP operates as an important element of the Green Lungs of Poland program. Sewage treatment processes are divided into three technological units: mechanical, biological and sludge treatment ones. The treatment plant receives wastewater from the city of Bialystok as well as from nearby communities. The treated wastewater flows through the control and measurement station and is discharged into the receiving body, which is the Biala River.

Raw sludge separated in the primary settling tanks is pumped out cyclically by using three pumps with the capacity of 100m^3/h each (pumping station I°). Next it is directed to thickeners that are designed to reduce the volume of primary sludge by separating and draining off excess sludge water. The activated sludge separated in the secondary settling tank is partially returned through the pumping station to the beginning of the treatment system (recycled activated sludge). The rest of the sludge is discharged from the system as excess sludge and is directed to the station of mechanical thickening of excess sludge, and from there to the digesters. The pumping station II° is equipped with two sludge pumps with a capacity of 20-100m^3/h each. The quantity of thickened sludge varies from 1000-1600m^3/d.

After thickening, both the primary and excess sludges are pumped into four anaerobic digesters (dimenstions of digester: diameter 20m, total height 32.56m, height of cylindrical part 20.5m, height of upper conical part 3.98m, and height of lower conical part 5.48m). Each of digester has a total capacity of 7700m^3, and the active capacity of 7300m^3. The sludge is digested at 35-37˚C at hydraulic retention time of 25-30 days. The sludge is mixed by using mechanical tubular Halberg mixers with a capacity of 3000m^3/h, and rotation rate up to 490rpm.

The digested sludge is transported to 2 retention tanks with capacity of 600m^3 each. The sludge is dewatered and its volume is reduced. The digested sludge undergoes a drying process (to obtain dry matter conent 88-90%) which makes possible its further use as fertilizer or fuel.

Methods

The content of total phosphorus and its fractions were deteremined after the samples drying, by using the Chang-Jackson sequential extraction method with later modifications by Petersen and Corey [20, 22].

The following fractions were separated:

-P-lab. (labile): labile phosphorus, fraction of phosphorus compounds readily soluble, extracted with 0.1 M NH_4Cl solution (Fraction I),

-P-Al: phosphorus in aluminum phosphates, extracted with 0.5 M NH_4F solution (Fraction II),

-P-Fe: phosphorus in iron phosphates, extracted with 0.1 M NaOH solution (Fraction III),

-P-red. (reduced): extracted with a 0.3 M solution of sodium citrate and sodium dithionite (Fraction IV),

-P-ocl. (occluded): fraction of occluded phosphates adsorbed on the surface of mineral particles, extracted with 0.1 M NaOH solution (Fraction V),

-P-Ca: phosphorus in calcium phosphates, extracted with 0.25 M H_2SO_4 (Fraction VI).

Some modifications were made in the determination of the above fractions. The pH value of the ammonium fluoride solution used for the extraction of aluminum phosphates was raised from pH = 7.0 to 8.5, because at a lower pH value the extract contained, in addition to the expected P-Al fraction, significant amounts of iron-bound phosphorus; raising the pH value to 8.5 allows the selective determination of the mentioned forms [23]. Instead of a 17-hour shaking of organic materials with extraction solution, a 5-hour shaking and a repeated 3-hour shaking on the following day were introduced for the determination of iron phosphates [23]. In the solutions thus prepared, the phosphorus content of each fraction, as well as the total phosphorus content, was determined by ICP-MS Determinations were performed in triplicate and results are given as means. Total phosphorus was given as averages of six for each sediment and term. The percentages of the phosphorus fractions in the total phosphorus content were calculated.

Results and discussion

The total content of phosphorus in the examined sewage sludge varied depending on the sampling term (Table 1). The significantly highest amount of phosphorus was found in the dried granular sludge and the smallest in the excess sludge. The content of total phosphorus in the determined sludges was much higher than in the materials studied by *Kuziemska and Kalembasa* [24].

Table 1. Total phosphorus content in the studied sludges [g/kg^{-1} d.m.]

Type of sludge	Spring (first intake)	Summer (second intake)	Autumn (third intake)	Winter (fourth intake)	Mean ± SD
excess	47.84	61.00	59.96	34.06	50.71 ± 10.91
digested and dewatered	49.41	67.5	63.84	54.70	58.86 ± 7.17
dried	76.02	77.96	59.57	57.36	70.90 ± 9.32

The distribution of phosphorus in the different fractions of the examined sludges is presented in Figures 1-3. The largest amount of phosphorus in all the examined sludges was found in the fraction II, i.e. aluminium phosphates. In the excess sludge, the contribution of fraction II in total phosphorus content ranged from 63.33 to 70.72% depending on the sampling term. Similar results were obtained by *Kuziemska and Kalembasa* [24], who examined sludges from two treatment plants, using the same method.

Xu et al. [25] have found the highest share of phosphorus in raw sewage sludge in fraction extracted with NaOH solution. It ranged from 67.1 to 81.7%. But they used the other sequential extraction method, which fractioned P into five fractions: NH_4Cl-P, BD-P, NaOH-P, HCl-P and Res-P, using 1.0mol/L NH_4Cl, 0.11mol/L $Na_2S_2O_4/NaHCO_3$, 1.0mol/L NaOH and 0.5mol/L HCl, respectively. They consider the NaOH-P fraction to be available to plants for a longer time than that extracted with water. The share of the fraction III extracted with 0.1M NaOH in our study was significanly lower (Fig. 1). *Bezak-Mazur and Stoinska* [26], using the Golterman method for determining phosphorus fractions, found that phosphorus in the primary and activated sludge occurred mainly in organic forms (fraction extracted with 0.5M H_2SO_4 and 2M NaOH), and only in the excess sludge these compounds underwent mineralization and occured in mobile forms (fractions extracted with 0.1 M Na-EDTA and 0.05M Ca-EDTA).

Similar statements were made by *Czechowska-Kosacka* [27]. This statement corresponds to the results of conducted research in which phosphorus in the excess sludge is presented mainly in the mobile form of fraction II (aluminium phosphates). This is important for the recovery of phosphorus from sludge and the possibility of using it in agriculture, without incurring costs for drying or thermal processing. Most of the studies conducted in recent years on phosphorus speciation in sewage sludge concern thermally transformed sludge [28-30]. The results of these studies indicate a reduction in the proportion of the plant-available fraction in favor of the HCl-extracted inaccessible fraction due to thermal conversion of the sludge. *Steckenmeesser et al*. [30] stated that during pyrolysis condensed phosphates (di-, tri-, poly-, and metaphosphates) are formed from primary and secondary phosphates (orthophosphates) and organic polyphosphates through condensation and polymerization reactions. Plants take up phosphorus mostly in the form of $H_2PO_4^-$ and HPO_4^{2-} and in these forms it is brought into the soil with mineral fertilizers. Phosphorus after thermal treatment of sludge is found in the fractions that are less available to plants and it takes some time and need the appropriate conditions, including soil pH, to become available [31].

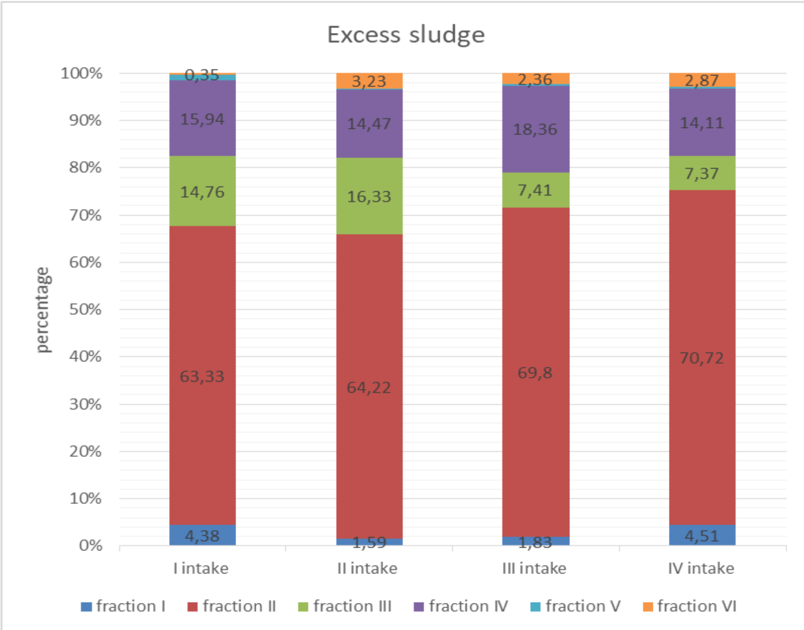

Fig. 1. Percentage share of phosphorus fraction in excess sludge

The differences were found in the proportion of particular phosphorus fractions depending on the sampling term (Figs. 1-3). The labile phosphorus fraction varied more with sampling date. The proportion of this fraction was low and did not exceed 5% in all the examined sludge. The proportion of the fraction III (iron-bound phosphates), also varied with the sampling time and in the case of excess sludge it was more than half as high for the sludges collected in autumn and winter then in spring and summer. However, further studies are needed to determine whether this variability can be explained by the seasonal changes in sewage sludge composition.

The distribution of the reduced phosphorus fraction IV in the sludge collected at different times was similar. Only a slightly higher proportion of this fraction was observed in sludge collected in the autumn. The proportion of the fraction V (adsorbed on mineral particles) was negligible, and the share of fraction VI (bound to calcium) was also small. It did not exceed 4% of total phosphorus amonut. The share of phosphorus in the anaerobically digested mixed sludge after dewatering was also the highest in the fraction II, but in comparison to the raw excess sludge it was on average 3% lower, regardless of the sampling term. The distribution of different forms of phosphorus was similar in the sludge taken in each sampling term. However, the avearge contribution of the fraction I (labile phosphorus) was slightly higher than in the excess sludge. It amounted to over 5%. The share of fraction III was similar when compared with excess sludge, while the share of fraction IV was significanly higher in dewatered digested sludge. The phosphorus fraction IV was comparable in the dewatered and excess sludge, but its distribution depending on sampling time was slightly different. Significantly more of this fraction was found in the sludge collected on the second and fourth terms than on the first and third intakes. Similary to the excess sludge, the share of the fraction V in the mixed digested sludge did not exceed 1%, and it was not depended on the sampling term. Also, the share of fraction VI was similar in all samples of sludge and did not exceed 3%.

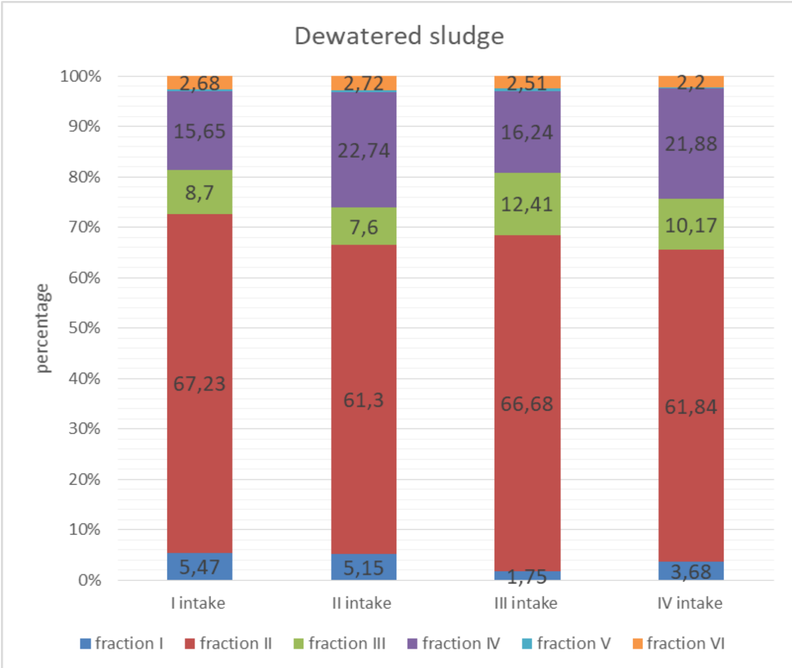

Fig. 2. Percentage share of phosphorus fraction in dewatered mixed sludge after fermentation

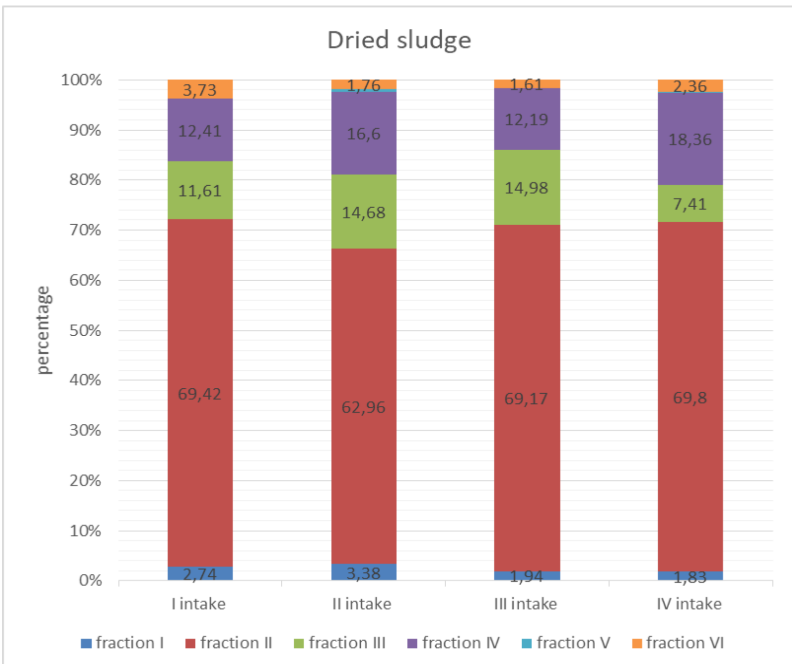

Fig. 3. Percentage share of phosphorus fraction in dried granulated sludge

The dried, granular sludge was also dominated by the fraction II of phosphorus in the form of aluminium phosphates and differed on average by only 1-2% with respect to the excess and dewatered sludge. In this sludge a lower share of this fraction was found only for the second term and in the other terms it was higher and similar to each other. The proportion of labile phosphorus (fraction I) ranged from 1.83-3.38%, and its average value was lower when compared with both the types of previously described sludge.

The share of phosphorus fraction III in dry sludges was in range of 7.41- 14.98% and it was comparable to the two other types of the sludge. The average contribution of fraction IV in dried sludge was similar to that found in case of fraction III. This similarity was not observed in the digested sludge before drying process. Phosphorus in fraction V was present in minimal amounts in the samples taken out in all the terms. There was also little phosphorus in fraction VI, and the highest proportion (3.73%) was found in the samples collected in the spring. It was the higest value compared to all the examined samples of sludge. During drying process in temperature of about 90°C, the phosphorus from the organic compounds is converted to the mineral ones. The authors emphasize that at high drying temperatures phosphorus transforms into compounds unavailable to plants [32, 33].

For all the examined sludge, regardless of their types, the proportion of phosphorus in each fraction can be arranged in the following order: P-Al>P>red.>P-Fe>P-lab.>P-Ca>P-ocl. In the study by *Kuziemska and Kalembasa* (2007), the order of fractional contributions was slightly different and was as follows: P-Al>P-lab.>P-red.>P-Fe>P-Ca>P-ocl.

The occurrence of individual phosphorus fractions in sludge is influenced by the type of coagulant used for phosphorus precipitation in WWTPs. The use of aluminium (III) salts contributes to an increase in mobile forms of phosphorus in the sludge, as highlighted by *Bezak-Mazur and Mazur* [34]. *Gutierrez et al*. [35] demonstrated the effect of treatment processes on phosphorus speciation in municipal wastewater sludge. In the treatment plant where sludge samples were taken, aluminium (III) sulphate was used for coagulation and the proportion of mobile fraction II is relatively high and in the tested sludge was more than 60%.

The occurrence of phosphorus fractions is influenced not only by the type and dose of coagulant but also by the place where it is used during wastewater treatment [36, 37]. Bezak-*Mazur and Mazur* [38] in a laboratory study found that phosphorus speciation was most influenced by sludge aeration and to a lesser extent by temperature. According to our observation the term of sampling and thus the effect of ambient temperature on the share of particular phosphorus forms was not important.

The study of the speciation of phosphorus compounds allows for a better understanding of the distribution of this component in its total content and the mechanism of transformation between fractions as a result of the treatment of biowaste such as sewage sludge. This allows designing techniques to recover this valuable biogen. The selection of P recovery technology should take into account the type of wastewater treatment process and the occurring phosphorus speciation. The conducted studies allow to conclude that recovery of phosphorus from municipal sewage sludge at the level of 60-70% (because such level was fraction II in which phosphates were bound with aluminium) is a promising direction for phosphorus recovery from burdensome waste such as sludge.

Taking the Poland as an example, where 048.7 thousand tons of s.m. including 574.5 thousand tons of municipal sewage sludge were produced in 2019 [39], and assuming the phosphorus content of 3%, it can be calculated that all the sludge is a potencial source of 31.5 thousand tons phosphorus, and ithe municipal sewage sludge can deliver 17.5 thousand tons. Only the extraction of second fraction from municpal sewage sludge may provide 10-12.2

thousand tons of phosphorus available for plants. According to the statistical data [39] in Poland 6191 thousand tons d.m. of sludge was stored at the sewage treatment plants, which is six time higher than the amount of sludge produced annually. The use of phosphorus from this type of waste is in line with the principles of a closed loop economy. Recovery of phosphorus from various wastes contributes to protection of non-renewable resources, which are raw materials for production of phosphate fertilizers.

Recovery of phosphorus from sewage sludge should be preceded by recognition of its fraction in the total phosphorus. The applied method for phosphorus speciation is less frequent than the Golterman method and includes as many as six phosphorus fractions, thus it may be considered more accurate in terms of identification of speciation of this indispensable for living organism's component.

Conclusions

The study showed that the dominant phosphorus fraction in the sludge, irrespective of the type of sludge studied, was the fraction of phosphates with aluminium which is available for plants and its share was 60-70%. The remaining fractions constituted 30-40% of the total phosphorus, of which the labile fraction did not exceed 5%. These results showed that selecting an appropriate technology, it is possible to recover phosphorus from sewage sludge for fertilizing purpose. Phosphorus recovery from sludge with high moisture content is an alternative to the methods, in which thermally converted sludge are used and which consume significant amount of energy. For this reason, application of wet sludge has positive impact on the climate protection and the global environment. Phosphorus recovered from sludge can reduce the amount of mined raw materials for mineral fertilizer production not only on a national, but also on the global scale. Raw materials for the production of phosphate fertilizers are non-renewable resources that should be protected and their extraction should be limited. It can be achieved at least to some extent by recovering phosphorus from waste, including sludge. Our study showed that each type of the examined sewage sludge can be considered as the direct source of phosphorus for plants from the sludge, because this element is bound mainly in a aluminium phospahtes. The share of selected phosphorus fractions differed slighlty depending on the term of sludge collection. However, further studies are needed to determine whether this variability can be caused by the seasonal changes in sewage sludge composition.

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