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Preliminary assessment of phosphorus mobility in sewage sludge for their potential leaching from soil

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ABSTRACT

Sewage sludge is rich in organic matter, nitrogen, phosphorus and other macro and micronutrients, making it a useful raw material for agricultural use. The natural management of sewage sludge, including the use of its fertilizing properties, is characterized by low costs, but it cannot be used without restrictions. It is important that sludge is produced throughout the year, while the possibility of its application occurs during a specific time of the growing season. Poor fertilizer management could result in the release of excess nitrogen and phosphorus from fertilizers and eutrophication of aquatic ecosystems. The study attempted to assess the mobility of phosphorus in various sewage sludges in terms of their potential leaching when used in nature for fertilizer purposes. The method of special analysis according to Golterman, which is originally applied to soils, was used for this purpose. This method makes it possible to determine the content of various forms of phosphorus. This is a novel attempt to assess the potential leaching of mobile forms of phosphorus from sewage sludge, as such analyses are not usually used in the case of potential use of sludge for natural purposes. The study attempted to evaluate the mobility of phosphorus in various sewage sludge in terms of its potential leaching when used in nature for fertilizer purposes. Comparing pre-sludge, activated sludge and surplus sludge and considering its potential use for natural purposes, it seems that surplus sludge containing the most mobile forms of phosphorus (63%) at this time is the most valuable sewage sludge of those analyzed. Obviously, the necessary treatment to meet sanitary requirements when using the sludge for natural purposes is hygienization, which caused an 18% reduction in biodegradable (mobile) forms of phosphorus. The most readily available form of phosphorus is the Ca-EDTA fraction, the proportion of which has been reduced sequentially in the sludge generated at the wastewater treatment plant (preliminary, activated and surplus). This fraction is the most labile and is identified with phosphorus readily released into the soil solution. The low amount of analysis makes it necessary to continue research with a special focus on the analysis of sludge after it has been applied to the soil.

Keywords: Phosphorus mobility; Sewage sludge; Hygienization

1. Introduction

The concentration of phosphorus in the soil ranges from 50 to 3,000 mg P/kg d.m. Typically, the concentration of this element decreases with the depth of the soil profile. Phosphorus compounds in the soil exist in organic and mineral forms. The content of organic phosphorus compounds in the soil depends on the type of soil and ranges from 15% to 80% of total phosphorus [1]. The main organic forms of phosphorus in soil are inositol phosphates (10%–50%), phospholipids (1%–5%) and nucleic acids (0.2%–2.5%) [2]. They also occur in the form of organic phosphoric acid

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esters. In a hydrolysis reaction involving phosphatase, the mineral form of phosphorus that is orthophosphates (V) is released. The above mineralization process depends on moisture, temperature, reaction and soil type, as well as on the ratio of organic carbon to phosphorus in the soil [3]. The smaller this ratio is, the greater the activation of phosphorus from organic compounds. Organic forms of phosphorus are converted into inorganic soluble forms H_3PO_4 (phosphoric acid), $H_2PO_4^-$, HPO_4^{2-}, PO_4^{3-} (phosphate), through mineralization. Inorganic soluble forms can be incorporated into the biomass of organisms or be immobilized by precipitation or adsorption [4]. Phosphate precipitation depends primarily on the pH and moisture content of the soil. In acidic or alkaline soils, phosphate fixation occurs in the form of insoluble salts. In acidic environments, precipitation of either aluminum or iron phosphate occurs [5]. Precipitated phosphates can crystallize to form forms such as variscite ($AlPO_4·2H_2O$) and strengite ($FePO_4·2H_2O$). This is the aging process of phosphates. The tendency of phosphates to form stable minerals is due to the fact that the electronegativity of oxygen in the phosphate ion is much greater than that of phosphorus. This causes a strong attraction of phosphate ions and cations in the crystal structure of the formed mineral [6]. Sewage sludge is produced as a by-product of wastewater treatment plants. Worldwide production is estimated at 45 million dry tons/y. Sewage sludge disposal reaches up to 60% of the total operating costs of a wastewater treatment plant, making the process problematic and expensive [7]. Sewage sludge is rich in organic matter, nitrogen, phosphorus and other macro and micronutrients, making it a useful raw material for agricultural use. On average, dry sewage sludge contains 50%–70% organic matter and 30%–50% minerals [8]. The physicochemical and biological properties of agricultural soils that are supplemented with organic matter-rich sludge can be significantly improved. The use of sewage sludge in combination with mineral fertilizers has a positive effect on yield growth and soil microbial activity. Taking into account the priorities of waste management, which put a premium on material recovery, the reuse of sludge in agriculture is a potentially optimal solution, due to its valuable properties: organic carbon, nitrogen content [9]. A valuable resource is phosphorus due to the ongoing depletion of mines. The introduction of components accumulated in sewage sludge into the soil is not only economically appropriate, but also necessary for the preservation and restoration of ecological balance. The mineral and organic composition of sludge from municipal wastewater treatment plants is similar to soil organic matter, known as humus [10]. However, in order to practically assess the role of sludge as a secondary source of nutrients, it is necessary to estimate the content of bioavailable phosphorus in sludge, defined as the sum of immediately available phosphorus and that which can be transformed to available forms as a result of natural processes occurring in sludge [11].

The proportion of bioavailable phosphorus compounds can be determined using speciation analysis, an analytical procedure that quantifies the chemical forms in which an analyte occurs. One procedure for speciation analysis is the Golterman method [12]. This method involves the use of chelate reagents Na-EDTA (edetate disodium), Ca-EDTA, and sulfuric acid and sodium hydroxide solutions in the analysis. The creator of the method in question detailed that the speciation form with the greatest biological availability is the phosphorus adsorbed on the surface of sludge particles, that is, the Ca-EDTA fraction and the Na-EDTA fraction. Decisive factors in the use of sewage sludge for natural purposes are the heavy metal content of the sludge and its hygienic and sanitary condition. Therefore, stabilization combined with sludge hygienization is often used, which involves the effective destruction of pathogenic microorganisms, parasites and spore forms by means of strongly alkalizing or acidifying substances [13]. Alkaline hygienization also immobilizes heavy metals until the pH remains high. This is usually done with quicklime (CaO) or in the form of hydrated lime ($Ca(OH)_2$) [14].

From the point of view of the natural use of sludge, liming makes it possible to immobilize the mobile forms of heavy metals and reduce the potential sanitary harmfulness of the sludge [14]. However, it should be mentioned that the addition of lime to sewage sludge can also affect the shares of mobile forms of nutrients, such as phosphorus. According to Wysokiński and Kalembasa [15], hygienization of sewage sludge by the addition of CaO leads to a decrease in the proportion of phosphorus in soluble forms (water-extractable and CH_3COONa), which can lead to a decrease in the availability of this element for plants in the soil The assimilability of nutrients for plants introduced into the soil with sludge depends, however, both on the forms in which they occur in the sludge and on soil conditions. The environmental management of sewage sludge cannot be applied without restrictions. It is important that sludge is produced throughout the whole year, while the possibility of its application occurs at a certain time of the growing season. The release of excess nitrogen and phosphorus from fertilizers used in agricultural production causes eutrophication of aquatic ecosystems [16]. Although the exchange of substances between bottom sludge and water is the main component of the circulation of matter in aquatic ecosystems, the introduction of phosphorus into the circulation in different amounts, at different times, can lead to an imbalance of the aquatic ecosystem and eutrophication. Changes in the amount of phosphorus in the aquatic environment can be caused by the consumption of this element by algae [17,18]. It should be mentioned that maintaining the balance of phosphorus in the ecosystem is one of the priority issues of environmental protection. In view of this, efforts are being made to minimize external loads entering inland waters.

Currently, there is a rich resource of publications on the speciation analysis of phosphorus by extraction methods [19–22]. These methods were originally used for the analysis of speciation forms in soil and bottom sludge [23–26], but since 2009 there have been publications in which these procedures have been adopted for the analysis of sewage sludge. At that time, the effects of precipitating agents (PIX and PAX), ultrasound and temperature on phosphorus speciation in sewage sludge were analyzed [27–29]. There were also publications on the effect of sludge hygienization on phosphorus speciation [15]. However, there are no literature reports on the speciation of phosphorus in all types of sewage sludge from a single facility, that is, from pre-sludge to hygienized sludge. The compilation of such results allows a comparative assessment of sewage sludge in terms of its phosphorus mobility and suitability for natural purposes. It should be noted that phosphorus fractionation methods are originally chemical analyses used to study soil while a number of fractionation methods have been adapted for sewage sludge analysis. Thus, presenting the results of phosphorus fractionation using the Golterman method is an attempt to evaluate the mobility of phosphorus in different types of sewage sludge.

This study attempts to evaluate the mobility of phosphorus in various sewage sludges in terms of their potential leaching when used for fertilizer purposes.

The purpose of the research undertaken in this study was to investigate the potential release of phosphorus from sewage sludge by performing speciation analysis in sewage sludge.

Speciation analysis allows us to study the processes involved in the release of phosphorus in sewage sludge. We assume that in the case of varying technological conditions prevailing in the reactors of wastewater treatment plants and depending on the changing physical and chemical conditions caused by the addition of calcium oxide to excess sludge in the hygienization process, the proportions of phosphorus speciation forms change.

2. Material and research methods

The research was carried out on the sewage sludge from a little wastewater treatment plant (3000 PE), where the mechanical and biological treatment is used. The biological treatment in this wastewater treatment plant consists in the method of the three-phase activated sludge (with separated zones: anaerobic, anoxic and aerobic). The sewage sludge was collected in March 2020 when the temperature of sludge was 13°C. The samples were collected from preliminary, active, excess sludge and excess sludge after a 24-h-long process of hygienisation. The process of hygienisation was carried out by adding calcium oxide to the dewatered sludge which resulted in pH amounting to 12 and after another 22 h the pH-value amounted to about 11. The sewage sludge was submitted to hygienisation with a dose of 0.2 kg CaO/kg d·m. This selected dose allowed for effective hygienisation of the tested material [30]. The initial pH of the excess sludge was 6.8. In the sludge collected in three series of samples, particular fractions of phosphorus were marked using the pattern suggested by Golterman (Fig. 1) [12]. The first stage was the extraction with the solution Ca-EDTA (edetate calcium disodium) which took 4 h. In the second stage the samples had been extracted for 18 h by means of solution Na-EDTA (edetate disodium). The next step was the extraction of the sample for 2 h with the solution H_2SO_4 (sulphuric acid (VI)). The last stage also took 2 h. In this stage the samples were extracted with the solution NaOH (sodium hydroxide). After each stage of extraction the samples were filtered and after filtration the sludge was treated with the next extraction reagent. In the obtained filtrate the concentration of the total phosphorus was marked. The marking of phosphorus

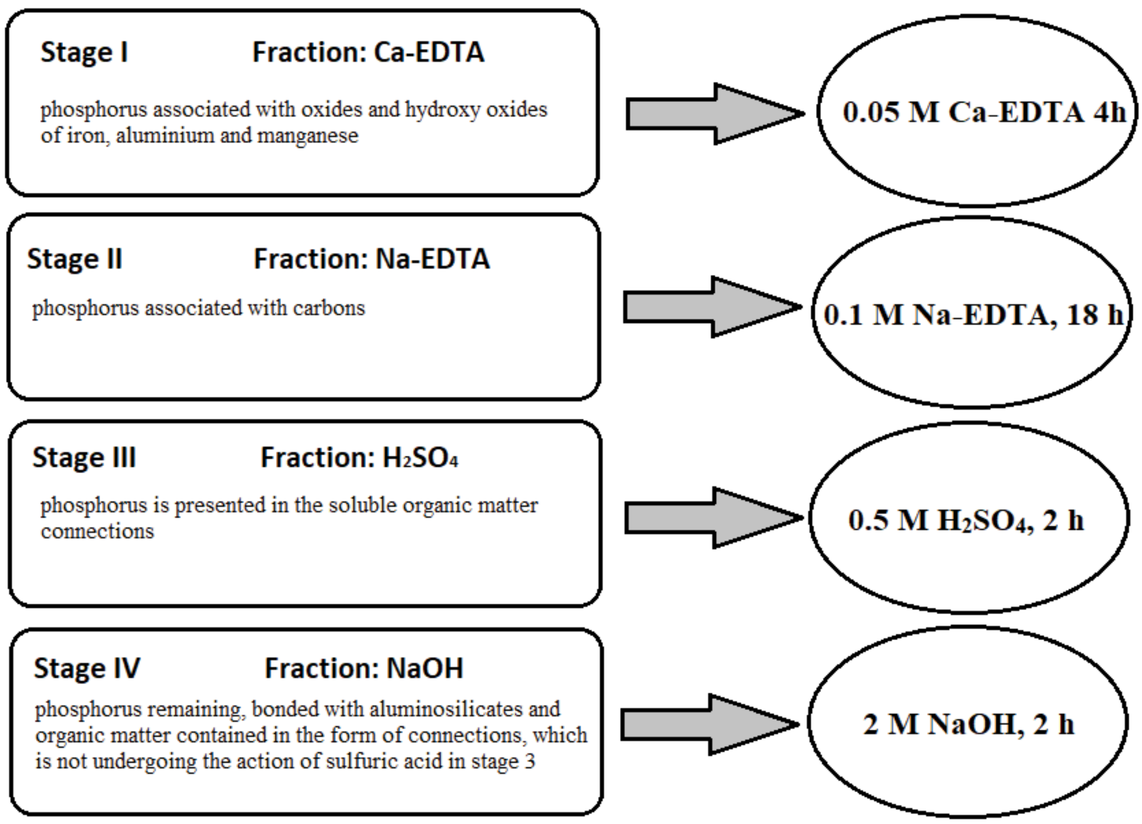

Fig. 1. Phosphorus sequential extraction scheme according to Golterman [12].

was carried out using the spectrophotometer UV-VIS from PerkinElmer Company (Waltham, Massachusetts, U.S.). The measurement was led according to the procedure of marking of orthophosphates using phosphoric molybdenum blue and the marking of total phosphorus after the previous oxidising of the sample with potassium persulfate [31].

3. Results and discussion

The obtained results of the phosphorus speciation carried out on the samples of the primary sludge, active in the excess and also after the hygienization process (Figs. 2–5) show the variability of the proportion of phosphorus forms. In the case of the ascending sludge, the dominant phosphorus fraction was the NaOH fraction, that is, phosphorus contained in organic matter and also bound to aluminosilicates. The fewest forms of phosphorus were bound to veglates, that is, the Na-EDTA fraction.

The next sludge analyzed was activated sludge (Fig. 3). It was observed that the amount of phosphorus in the organic fraction, called the NaOH fraction, varied compared to that in the primary sludge, and the mobile phosphorus fraction, the Na-EDTA fraction, increased.

In the case of excess sludge (Fig. 4), the dommifying form of phosphorus was the Na-EDTA fraction and accounted for as much as 42% of the total phosphorus contained in the sludge. Thus, one can observe changes in the forms of phosphorus going in the direction of mineralization of organic forms in the process line of the wastewater treatment plant. The high content of the Na-EDTA fraction in excess sludge is the result of sedimentation of phosphonates and their adsorption on carbonates. Phosphorus contained in the Na-EDTA fraction is mobile phosphorus otherwise mobile. Mobile forms of phosphorus are compounds soluble in weak acids, such as freshly precipitated amorphous aluminum and iron phosphates, calcium hydrogen phosphates, and phosphorus

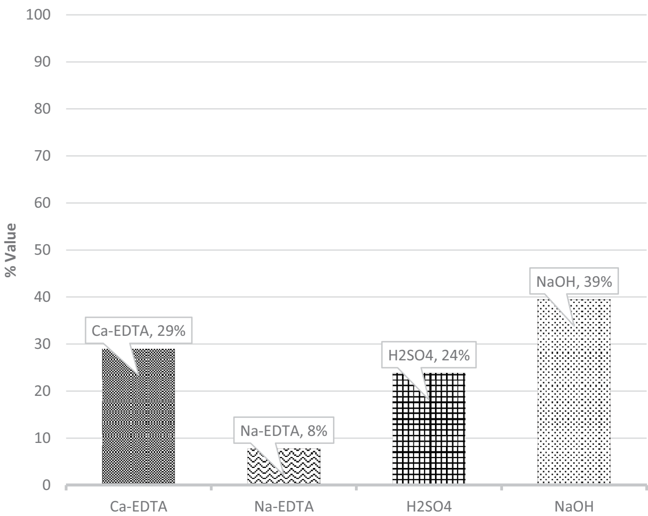

Fig. 2. Proportion of speciation forms of phosphorus in primary sludge.

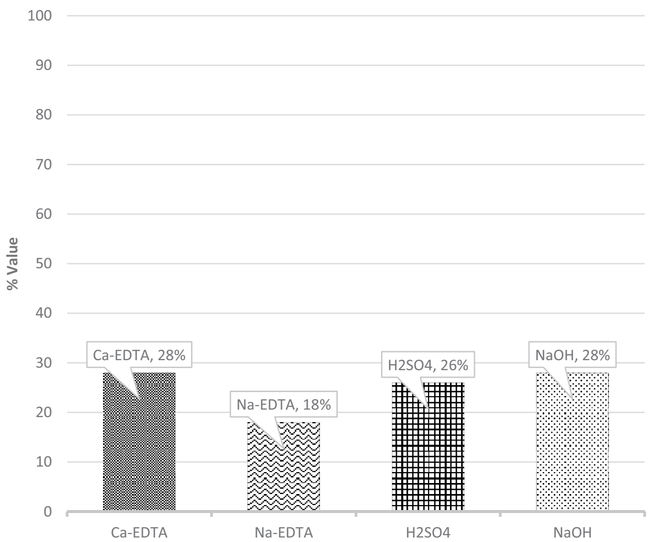

Fig. 3. Proportion of speciation forms of phosphorus in activated sludge.

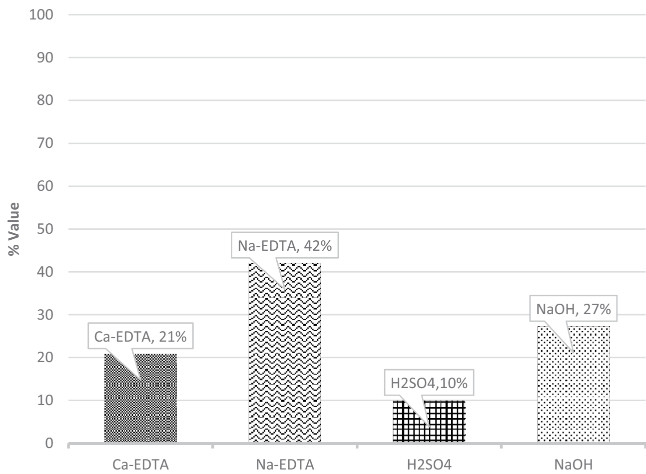

Fig. 4. Proportion of speciation forms of phosphorus in excess sludge.

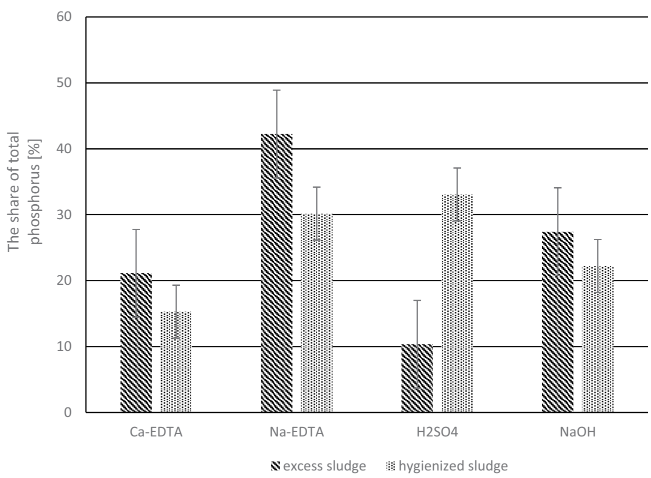

Fig. 5. Proportions of speciation forms of phosphorus in excess sludge and after the hygienization process.

compounds forming sorption complexes with, for example, aluminum and iron oxides [32].

The obtained results of the carried out speciation on the samples of excess sludge and after the process of hygienisation (Fig. 5) show the share reduction of fraction containing mobile and bioavailable phosphorus, that is the fraction obtained after extraction with chelating reagents in proportion to the fractions containing organic phosphorus after exposing the dewatered sludge to the process of hygienisation. The total share of these fractions (Ca-EDTA and Na-EDTA) in the case of total phosphorus after dewatering amounted to about 67%, whereas after the process of hygienisation to 45%. The causes of the reduction of share of the bioavailable forms of phosphorus in the hygienised sludge should be analysed against the background of a sudden increase of pH-value up to 12. The strong alkaline reaction of the sludge caused the precipitation of sparingly soluble calcium phosphates. Similar results were obtained by Wysokiński and Kalembasa [15], who found that the hygienization of sewage sludge by adding CaO leads to a reduction in the proportion of phosphorus in soluble forms (extracted with water and CH_3COONa). The reduction of the share of phosphorus in the process of hygienisation happened also in case of fraction NaOH in which phosphorus is contained in the organic matter. The cause of reduction of shares of this fraction may be the decomposition of the organic matter under the influence of high temperature which was noticed during the process of hygienisation. The sequential analysis also showed a significant increase of the share of the fraction H_2SO_4 which can be explained by thermal decomposition of organic structures and dissolving freely soluble links of phosphorus with organic matter.

There is a paucity of literature reports on the effect of hygienization on phosphorus speciation in sewage sludge. However, there are reports on the effect of soil liming on phosphorus speciation [33–35]. Phosphorus mobility from soil is strongly dependent on soil pH [36]. A high concentration of active (mobile) phosphorus as a consequence of weaker phosphorus binding occurs in soils with a pH ranging from 5.5–7.0. In acidic or alkaline soils, phosphate binding occurs in the form of insoluble salts. In acidic environments, precipitation of aluminum or iron phosphate occurs according to the following reaction:

$$Al^{3+} + H_2PO_4^- + 2H_2O \rightarrow Al(OH)_2 H_2PO_4 + 2\ H^+ \quad (1)$$

In contrast, in alkaline environments, phosphates react with calcium:

$$3HPO_4^{2-} + 5CaCO_3 + 2H_2O \rightarrow Ca_5(PO_4)_3OH + 5HCO_3 \quad (2)$$

A strongly alkaline environment results in the formation of hard-to-move forms of phosphorus. Literature reports describe changes in the amount of mobile forms of phosphorus, after lime is applied to acidic soils to increase soil pH and neutralize the reaction [33–35]. There are significant amounts of bioavailable forms of phosphorus in neutral pH. In contrast, our research looks at other pH ranges, where there is a reduction in available forms of phosphorus due to precipitation of the hard-to-access forms of this element at pH around 12.

Fig. 6 shows the shares of mobile forms of phosphorus (sum of Ca-EDTA and Na-EDTA fractions) in the four analyzed sewage sludge. It was observed that with the degree of mineralization of sewage sludge in the process line of the wastewater treatment plant, the share of mobile forms of phosphorus increases and after the hygienization process, the share of mobile forms decreases.

The sewage sludge was taken in March, when fertilization of agricultural fields in Poland is applied. The temperature of the sludge at the time was 13°C. This is of particular importance in the case of phosphorus speciation, as the amount of biodegradable forms of phosphorus increases as the sludge temperature increases [37]. Comparing pre-sludge, activated sludge and surplus sludge, and considering its potential use for natural purposes, it seems that surplus sludge containing the most mobile forms of phosphorus at that time is the most valuable sludge of those analyzed. Obviously, the necessary treatment to meet sanitary requirements in the case of using the sludge for natural purposes is hygienization, which reduces the amount of biodegradable (mobile) forms of phosphorus. In the soil solution, phosphorus can be either volatilized to forms that are difficult to access or dissolved into weak acids that are produced by soil-dwelling bacteria. Therefore, in order to learn about potential transformations of phosphorus forms, special analyses of sludge used as fertilizer should be carried out. The most readily available form of phosphorus is the Ca-EDTA fraction, the proportion of which has decreased in the technological course of wastewater treatment plants (Figs. 2–4). This fraction is the most labile and is identified with phosphorus readily released into the soil solution. This knowledge is important in the case of over-fertilization of soils and leaching of phosphorus, which in turn can contribute to eutrophication of waters. Given the range of information that can be obtained by performing phosphorus speciation analysis, it would be advisable to consider performing such analyses as necessary when using sewage sludge for natural purposes. This work presents preliminary results. The small number of analyses makes it necessary to continue the research. Nevertheless, the research conducted may find application in environmental protection, especially in the protection of inland waters, preventing eutrophication.

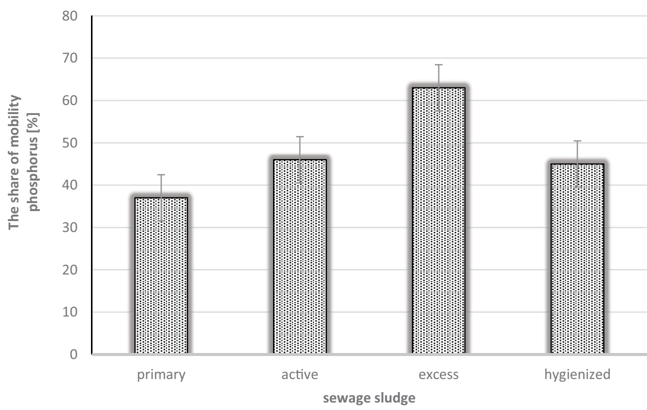

Fig. 6. Contribution of mobile forms of phosphorus in sludge: primary, activated, excess and after hygienization process.

4. Conclusion

This paper presents the results of a study of the proportion of bioavailable phosphorus fractions in sludge generated at a wastewater treatment plant, that is, pre-sludge, activated sludge and excess sludge, as well as hygienized sludge.

The results of the study allow the following conclusions:

- Depending on the degree of biological mineralization, the sewage sludge studied differed in the proportions of the various speciation forms of phosphorus, which may indicate that each sludge has an individual chemical composition. In the case of the initial sludge, the dominant fraction of phosphorus was the NaOH fraction, that is, phosphorus contained in organic matter and also bound to aluminosilicates. The proportion of the NaOH fraction was lower in activated sludge and in excess sludge, which may be related to the mineralization of the sludge.
- Comparing the variation of phosphorus forms after the lime hygienization process, it is possible to observe a decrease in the share of phosphorus also for the NaOH fraction, in which phosphorus is contained in organic matter. The reason for the decrease in the share of this fraction may be the decomposition of organic matter under the influence of high temperature. Sequential analysis also showed a significant increase in the share of the H_2SO_4 fraction, which can be explained by thermal decomposition of organic structures and dissolution of freely soluble phosphorus bonds with organic matter. Hygienization of the excess sludge also resulted in a decrease in the amount of biodegradable (mobile) forms of phosphorus.
- The most readily available form of phosphorus is the Ca-EDTA fraction, the proportion of which has been reduced in the technological sequence of the wastewater treatment plant. This fraction is the most labile and is identified with phosphorus easily released into the soil solution. This knowledge is important in the case of overfertilization of soils and leaching of phosphorus, which in turn can contribute to eutrophication of waters. Therefore, sewage sludge used for natural purposes should be subjected to phosphorus mobility analysis, since the determination of only total phosphorus in sludge gives incomplete information about its fertilizer values.

This work presents preliminary results. The small number of analyses makes it necessary to continue the research with a special focus on the analysis of sewage sludge after its application to the soil.

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