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The effect of selected methods of conditioning of digested sewage sludge on the content of organic and biogenic compounds in sludge liquids

Beata Bień[a],*, Jurand D. Bień[a], Bartłomiej Macherzyński[b]

[a]*Czestochowa University of Technology, Faculty of Infrastructure and Environment, 42-200 Czestochowa, Poland, emails: beata.bien@pcz.pl (B. Bień), jurand.bien@pcz.pl (J.D. Bień)*
[b]*Faculty of Biology and Environmental Science, Cardinal Stefan Wyszynski University in Warsaw, Wóycickiego 1/3 Str., 01-938 Warsaw, Poland, Warsaw, Poland, email: b.macherzynski@uksw.edu.pl*

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ABSTRACT

A new approach to the problem of sludge liquids in wastewater treatment plants involves the use of technologies designed not only for increased removal of nitrogen and phosphorus from leachate, but primarily for the recovery of nutrients. Due to the high content of nitrogen and phosphorus in sludge liquids separated from digested sludge, they were found to be a potential source for the recovery of biogenic compounds. The selection of appropriate chemical and physical agents for conditioning of digested sludge is one of the factors that have a significant impact on the quality of sludge liquids. The article presents the effect of conditioning methods (ultrasonic field, coagulant PIX 123, polyelectrolyte Zetag 8160) on the content of organic and nutrient compounds in sludge liquids. The raw sludge liquids were characterized by very high phosphate concentrations: 122.4 mg·PO_4^{3-}/L, ammonium nitrogen: 1,718 mg·N–NH_4^+/L) and organic compounds determined as chemical oxygen demand (COD) 2,240 mg·O_2/L. The aim of the study was to determine the effect of selected methods of conditioning digested sewage sludge on the quality of sludge liquids obtained after the sludge dewatering process. The scope of the study included measurement of pH, phosphate, ammonium nitrogen and COD concentration in sludge liquids. The highest amount of phosphate was recorded in sludge liquids separated from sonicated sludge (A = 45.75 µm, t = 60 s). It was 355.5 mg·PO_4^{-3}/L (an increase of 190.4% was noted compared to phosphate concentration in raw liquids).

Keywords: Sludge liquids; Sludge conditioning; Biogenic compounds; Nitrogen; Phosphorus

1. Introduction

In wastewater treatment plants (WWTPs) sludge conditioning is used as a pretreatment process prior to sludge thickening and dewatering in order to promote the separation of sludge into solid and liquid phases [1–5]. Modification of the sludge structure ensures rapid and effective removal of the water contained in the sludge. In daily practice at WWTPs chemical conditioning, involving the addition of precipitating and coagulating agents to the sludge, is commonly applied [6–10]. Many researchers also address the mechanical conditioning, involving the addition of substances that change the structure of the sludge (e.g., ash or biocarbon) [5,11–15]. Other treatments are also used, primarily using electromagnetic and ultrasonic fields [16–19]. The ultrasonic field, due to its highly complex interaction in solid–liquid systems, can induce profound physical and chemical changes in sonicated sludge. Particularly important is the disintegration of microorganism cells in excess sludge before anaerobic digestion [20,21]. Solid phase particles are fragmented and the cell membranes of microorganisms are destroyed with the release of the cell interior into the sludge

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* Corresponding author.

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liquid during sonification. However, the disadvantages resulting from the use of disintegration are problems associated with the release of nutrients from the digested sludge into the leachate during sludge dewatering. In counterbalance, chemical conditioning improves the structure and properties of sludge which also prevents excessive return of nitrogen and phosphorus compounds found in leachate to the wastewater line at WWTP. To date, the goal of wastewater treatment has been to remove nutrients with organic compounds, since their release into the environment causes eutrophication of water bodies [22,23]. Nowadays, due to the increasing demand for macronutrients such as nitrogen and phosphorus there is a change in the ways sludge liquids are disposed of. It involves the recovery of raw materials found in wastewater, as well as in sludge liquids [24–29]. Nutrient recovery is an important component of a closed-loop economy and contributes significantly to sustainability goals. Unfortunately, commonly used nitrogen and phosphorus removal processes do not offer the possibility of recovering nutrient compounds, but convert them into other forms that have minimal environmental impact.

A new approach to the problem of sludge liquids involves the use of technologies designed not only for increased removal of nitrogen and phosphorus from leachate, but primarily for nutrient recovery [30,31]. Al-Juboori et al. [24] conducted research on the use of a membrane system designed to recover nitrogen and phosphorus in the form of ammonium salts. Tests were conducted for sludge liquid taken from an anaerobic digester. The ammonium salts produced were of high purity and nutrient content was comparable to fertilizers. Podstawczyk et al. [32] presents the possibility of removing and recovering nutrients from liquids after dewatering of digested sewage sludge using a novel two-step method involving an adsorption system and a membrane system. The use of the membrane system led to a 98.9% ± 0.1% reduction in the concentration of ammonium in the feed and the production of an almost threefold concentrated pure ammonium solution. In contrast, the efficiency of orthophosphate removal from pre-treated sludge liquids was 92.4% ± 0.1%.

Modern wastewater treatment technologies associated with biological removal of nutrients are a source of increasing amounts of excess sludge [31,33]. Excess sludge constitutes the main mass of sewage sludge. Many technologies for sewage sludge treatment and stabilization are based on processes of anaerobic decomposition of organic compounds [34–37]. The use of anaerobic digestion of sewage sludge as well as the thickening and dewatering of digested sludge in wastewater treatment plants leads to the generation of sludge liquid streams with high concentrations of nitrogen and phosphorus [38–40]. In addition to the released nitrogen and phosphorus, the leachate introduces significant pollutant loads in the form of organic suspended solids, which are difficult to biologically decompose. The use of PIX coagulant for sludge conditioning helps achieve a clean, suspended solids-free filtrate. Due to the binding of orthophosphates by Fe(III) ions present in PIX, very high reduction of phosphorus in the leachate is achieved, which generally makes it possible to significantly reduce the consumption of the coagulant previously used in the wastewater treatment line. Thus, the concentration of contaminants in sludge liquids can decrease after the application of appropriate polyelectrolytes and coagulants in the sludge conditioning process prior to dewatering. Sludge liquids separated from digested sludge can be a potential source of nutrient recovery due to their high content [27,32,41], which at the same time will reduce their harmful effects on the environment. With a view to a sustainable wastewater treatment system, which should aim to recover nutrients from liquid streams as much as possible, such activities should be carried out that will affect the quantity and quality of sludge liquids. A process that has a significant impact on the quality of sludge liquids is conditioning. The use of some conditioning methods increasing the content of biogenic compounds in sludge liquids involves some limitations, but much more benefits could be achieved (Table 1).

The article presents the results of a study on the effect of selected sludge conditioning methods on the content of biogenic and organic compounds in sludge liquids generated during sludge dewatering process.

2. Materials and methodology

2.1. Study materials

Digested sludge from a municipal wastewater treatment plant (PE > 100,000), which is a mechanical-biological treatment plant, was used for the study. The sludge management sequence includes the following unit processes:

- thickening of primary sludge in gravity thickeners,
- thickening of excess sludge in mechanical thickeners,
- mesophilic fermentation of mixed sludge,
- stabilization and thickening of digested sludge in open digesters,
- mechanical dewatering of digested sludge on belt presses,
- drying of dewatered sludge.

Table 1
Benefits and limitations of using selected conditioning methods that increase the content of biogenic compounds in sludge liquids

Benefits	Limitations
Sludge liquids are a potential source of biogenic compounds [27,32,38–40]	Need to upgrade the sludge line at WWTP [28,40,42,44]
Increasing demand for macronutrients such as nitrogen and phosphorus [23,27,30,42]	Costs of sludge sonification [18,20,47]
Recovery of biogenic compounds [24,27,30,31,40,42,43,58]	
No need to pretreat sludge liquids before they enter the wastewater line [28,38,44,48]	
Sonification is a simple method of sludge conditioning [17,18,20,45,46]	

Sludge was taken at a mechanical dewatering station from a pipeline that transports sludge from open digesters to belt presses. The sludge was stored at 4°C and warmed to room temperature before the experiments.

The following chemicals were used for sludge preparation: cationic polyelectrolyte Zetag 8160 and inorganic coagulant PIX 123 at doses: 4.0, 5.0, 6.0, and 7.0 mg/g DM. These doses were within the range of optimal doses. The characteristics of the coagulants are shown in Table 2.

2.2. Research plan

The research was conducted according to the plan shown in Fig. 1. Initially, the sludge liquids from digested and unprepared sludge were tested. Next, the digested sludge was chemically pretreated prior to the dewatering. Selected chemicals were added to the digested sludge:

- 0.1% solution of Zetag 8160,
- 10% solution of PIX 123.

in appropriate doses. Digested sludge was mixed with selected chemicals on a magnetic stirrer type MMS-3000N from Biosan. After the chemicals were added to the sludge, rapid stirring was carried out for 60 s (200 rpm), followed by slow stirring for 300 s (30 rpm). Then, the digested sludge was dewatered by centrifugation within 300 s, and with the speed of 5,000 rpm. The separated sludge liquids were then analyzed.

In the next test cycle, the digested sludge was sonicated using a Sonics VC750 microprocessor-based ultrasonic disintegrator at the beginning. Sludge samples with a volume of 0.4 L were sonicated for a period of 60 s. The variable parameter was the amplitude of the wave. It was 15.25, 30.50, and 45.75 µm, respectively. The same chemicals were then added to the sludge as before. Then the samples were dewatered under the same conditions to achieve sludge liquids. The following parameters were tested in sludge liquids: pH - by potentiometric method (pH-meter CP401 - by Elmetron), COD (chemical oxygen demand) by abbreviated dichromate method (PN-ISO 6060:2006 [52]), ammonium nitrogen and phosphate PO_4^{-3} by spectrophotometric method (Spectrophotometer JENWAY 6300). Each test was carried out in triplicate, and the results are given as an average value. Table 3 shows the symbols used in the graphs for sludge liquids identification.

3. Results

Raw sludge liquids from digested, unprepared sludge were characterized by very high concentration of ammonium nitrogen: 1,718 mg·N–NH_4^+/L, phosphate 122.4 mg·-PO_4^{3-}/L and organic compounds determined as COD: 2,240 mg·O_2/L. The characteristics of sludge liquids are given in Table 4.

The results of sludge liquids obtained from the sludge conditioned by different methods are shown in Table 5. A decrease in the pH value was noted in the sludge liquids

Table 2
Characteristics of the chemical agents used in the study

Chemical agent	Properties
PIX 123	Iron coagulant, iron(VI) sulfate, iron(III) sulfate solution in water with a dark brown color, no odor, the content of total iron Fe is 12.6% ± 0.3%, while iron ions Fe^{+2} is max 0.7% [49,50]
Zetag 8160	Synthetic polyacrylamide with a high molecular weight. It is supplied as a free-flowing, white powder. Zetag 8160 is of medium-high cationic charge. The coagulant is manufactured by Ciba Specialty Chemicals, Canada [51]

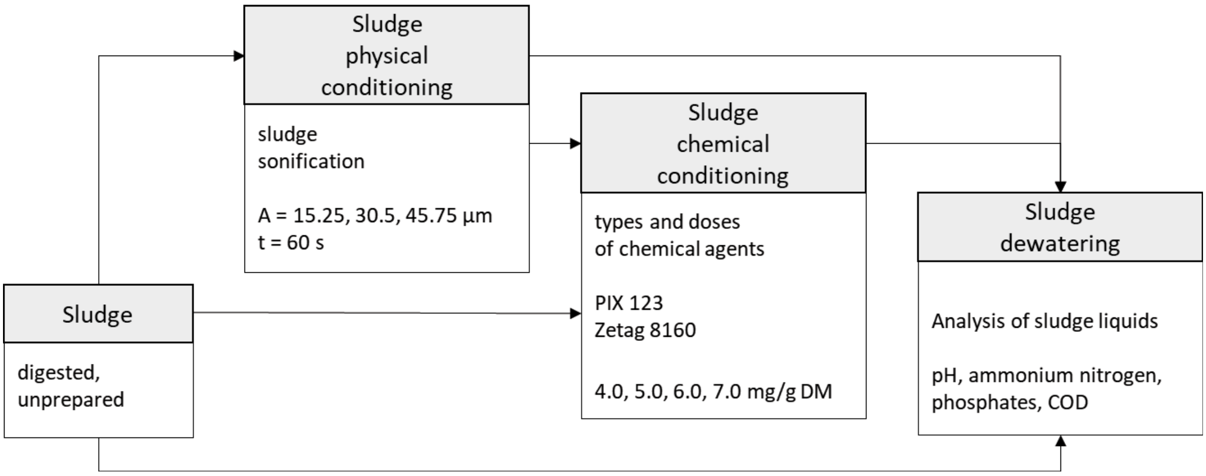

Fig. 1. Scheme of the study.

Table 3
Symbols assigned to sludge liquids obtained after dewatering of prepared sludge

No.	Symbols	Description of test conditions
1.	CPO	Sludge liquids from digested, unprepared sludge
2.	CNO1 CNO2 CNO3	Sludge liquids from digested and sonicated sludge. Sonication time $t = 60$ s, wave amplitude: $A = 15.25$ µm (1); $A = 30.5$ µm (2); $A = 45.75$ µm (3)
3.	CPO+PIX123 CPO+Zetag 8160	Sludge liquids from digested sludge to which the following agents were added: PIX 123, Zetag 8160 at the selected dose (4.0, 5.0, 6.0, and 7.0 mg/g DM)
4.	CNO1+PIX123 CNO1+Zetag 8160	Sludge liquids from digested and sonicated sludge ($t = 60$ s, $A = 15.25$ µm) to which the following agents were added: PIX 123, Zetag 8160 at the selected dose (4.0, 5.0, 6.0, and 7.0 mg/g DM)
5.	CNO2+PIX123 CNO2+Zetag 8160	Sludge liquids from digested and sonicated sludge ($t = 60$ s, $A = 30.5$ µm) to which the following agents were added: PIX 123, Zetag 8160 at the selected dose (4.0, 5.0, 6.0, and 7.0 mg/g DM)
6.	CNO3+PIX123 CNO3+Zetag 8160	Sludge liquids from digested and sonicated sludge ($t = 60$ s, $A = 45.75$ µm) to which the following agents were added: PIX 123, Zetag 8160 at the selected dose (4.0, 5.0, 6.0, and 7.0 mg/g DM)

Table 4
Characteristics of sludge liquids obtained from digested, unprepared sludge

Indicator	Value
pH	7.32
Total suspended solids (TSS), mg/L	1,142
Chemical oxygen demand (COD), mg·O$_2$/L	2,240
Ammonium nitrogen, mg·N–NH$_4^+$/L	1,718
Phosphates, mg·PO$_4^{-3}$/L	122.4
Phosphorus, mgP–PO$_4^{-3}$/L	40

separated from sludge prepared with PIX 123. The pH value decreased with the increasing coagulant dose. The lowest value of pH was obtained for a dose of 7.0 mg/g DM and was 5.28. It decreased by 27.9% compared to pH of raw sludge liquids. On the other hand, the pH of liquids obtained from the sludge conditioned with Zetag 8160 remained within a small range of 7.40–7.46. Similar correlations were obtained for sludge liquids where sludge was first sonicated before chemical agents were added. In the case of phosphate, a decrease in the amount was observed in all sludge liquids. The lowest value was recorded for the samples, where PIX 123 was applied. For liquids from non-sonicated sludge the decrease was 97%, when a dose of PIX was 7.0 mg/g DM. For sonicated sludge the decrease was 97.2%, 95.9%, 97.9%, respectively for the wave amplitude of 15.25, 30.5, and 45.75 µm (Figs. 2 and 3). The coagulant bound and retained phosphates in sludge according to the dose applied. A decrease in both phosphates and ammonium nitrogen concentration was observed in all samples. The lowest value of ammonium nitrogen was obtained for liquids separated from non-sonicated sludge with the addition of Zetag 8160 at a dose of 7.0 mg/g DM. It was 824.1 mg·N–NH$_4^+$/L (a reduction of 52% compared to the amount of ammonium nitrogen in the raw sludge liquids). The lowest contents of ammonium nitrogen in the liquids from sonicated and prepared with chemical agents sludge were obtained for a dose of 7.0 mg/g DM both for PIX 123 as well as Zetag 8160 (Figs. 4 and 5). COD in sludge liquids, obtained from chemically conditioned sludge, decreased with increasing dose of chemical agents. The smallest COD value (240 mg·O$_2$/L) was obtained for liquids obtained from non-sonicated sludge, conditioned with PIX 123 at a dose of 7.0 mg/g DM. In this case reduction of COD concentration was 89.3%. In the case of sludge liquids obtained from sonicated sludge with the addition of PIX 123 at a dose of 7.0 mg/g DM the reduction was higher than 90% (Figs. 6 and 7). Based on the data in Table 5, it can be concluded that PIX 123 gives better results than Zetag 8160 in terms of contaminants reduction.

As can be seen from the correlations shown in Figs. 2 and 3 phosphate concentration in sludge liquids from sonicated sludge increased with the increase of ultrasonic wave amplitude, but it decreased with an increase of chemicals dose. The highest concentration of phosphate in the sludge liquids was 355.5 mg·PO$_4^{-3}$/L when sludge was sonicated with an amplitude of 45.75 µm.

Sonification of sludge resulted in a decrease in the concentration of ammonium nitrogen in sludge liquids. While for sludge liquids from non-sonicated sludge the concentration of ammonium nitrogen was 1718 mg·N–NH$_4^+$/L, its concentration in sludge liquids from sonicated with the wave amplitude of 15.25, 30.5, and 45.75 µm was 853, 1,354 and 600 mg·N–NH$_4^+$/L, respectively (Figs. 4 and 5). As the dose of chemicals increased the concentration of ammonium nitrogen in sludge liquids also decreased.

The concentration of organic compounds, determined as COD, in the sludge liquids obtained from sonicated sludge increased with the increase of amplitude of ultrasonic wave applied. While for sludge liquids from non-sonicated sludge the concentration of COD was 2,240 mg·O$_2$/L, the concentration of COD in sludge liquids from sludge sonicated with and amplitude of 15.25, 30.5, and 45.75 µm was 3,679.2; 3,960 and 5,260 mg·O$_2$/L, respectively (Figs. 6 and 7). As the dose of chemicals increased the concentration of COD in sludge liquids decreased. The observed increase in COD during sonication might be due to the release of extracellular polymeric substances during floc disintegration and the release of cell components during cell lysis. Similar relationship was obtained by Zawieja and Wolny [53]. They observed

Table 5
Analysis of sludge liquids obtained from sludge conditioned by physical and chemical methods

Indicator	Dose	pH	Phosphates	Phosphorus	Ammonium nitrogen	Chemical oxygen demand
Unit	mg/g DM	–	mg·PO_4^{-3}/L	mgP–PO_4^{-3}/L	mg·N–NH_4^+/L	mg·O_2/L
Sludge liquids separated from non-sonicated, digested sludge conditioned with chemicals						
PIX 123	4.0	6.41	9.2	3.0	1,390.0	750.0
	5.0	6.02	7.8	2.5	1,125.6	480.0
	6.0	5.67	6.4	2.1	967.4	270.0
	7.0	5.28	3.6	1.2	942.0	240.0
Zetag 8160	4.0	7.40	61.3	20.0	1,338.3	2,280
	5.0	7.42	54.1	17.7	946.1	1,980
	6.0	7.43	48.5	15.8	898.3	1,910
	7.0	7.46	37.4	12.2	824.1	1,750
Sludge liquids separated from sonicated (*A* = 15.25 μm; *t* = 60 s) and conditioned with chemicals sludge						
PIX 123	–	7.8	215.2	70.3	853.0	3,679.2
	4.0	6.15	21.4	7.0	683.0	1,182.2
	5.0	5.88	10.3	3.4	540.0	502.4
	6.0	5.58	8.1	2.6	462.0	382.8
	7.0	5.20	6.0	2.0	273.0	312.3
Zetag 8160	4.0	7.76	118.2	38.6	626.0	3,489.0
	5.0	7.78	114.5	37.4	416.0	2,910.3
	6.0	7.79	100.4	32.8	378.0	2,797.2
	7.0	7.81	87.2	28.5	312.0	2,588.5
Sludge liquids separated from sonicated (*A* = 30.5 μm; *t* = 60 s) and conditioned with chemicals sludge						
PIX 123	–	7.86	244.5	79.9	1,354.0	3,960.0
	4.0	6.47	56.0	18.3	1,168.2	880.0
	5.0	6.1	17.0	5.5	1,078.6	530.0
	6.0	5.78	14.6	4.8	1,053.2	510.0
	7.0	5.6	10.1	3.3	974.4	370.0
Zetag 8160	4.0	7.87	170.2	55.6	1,224.0	2,900.0
	5.0	7.89	143.8	47.0	1,204.0	2,820.0
	6.0	7.90	137.1	44.8	872.3	2,710.0
	7.0	7.91	122.0	39.9	822.4	2,460.0
Sludge liquids separated from sonicated (*A* = 45.75 μm; *t* = 60 s) and conditioned with chemicals sludge						
PIX 123	–	7.88	355.5	116.2	600.0	5,260.0
	4.0	6.24	26.2	8.6	258.0	1,084.0
	5.0	6.01	19.1	6.2	236.0	816.0
	6.0	5.62	10.4	3.4	204.0	593.0
	7.0	5.30	7.3	2.4	162.0	425.0
Zetag 8160	4.0	7.88	374.5	122.4	586.0	5,206.0
	5.0	7.89	302.2	98.7	494.0	5,072.0
	6.0	7.91	271.4	88.7	314.0	4,886.0
	7.0	7.93	256.7	83.9	293.0	4,620.0

that with an increase in the power of ultrasonic field an increase in the degree of liquefaction of sludge particles, expressed by COD, took place. The results of Zhang et al. [54] also showed that sonification effectively degrades and deactivates sludge. The concentration of COD and the degree of sludge mass reduction increased with increasing sonification time and ultrasonic field power density.

The problem of return loads from the sludge line to the wastewater line at WWTPs is increasingly recognized and addressed in the literature [55–58]. Considering the amount of phosphorus and nitrogen contained in sludge liquids, it is necessary to consider the possibility of changes in disposal methods. Recovery of biogenic substances is the best option and fits into a closed-loop economy. The presented studies

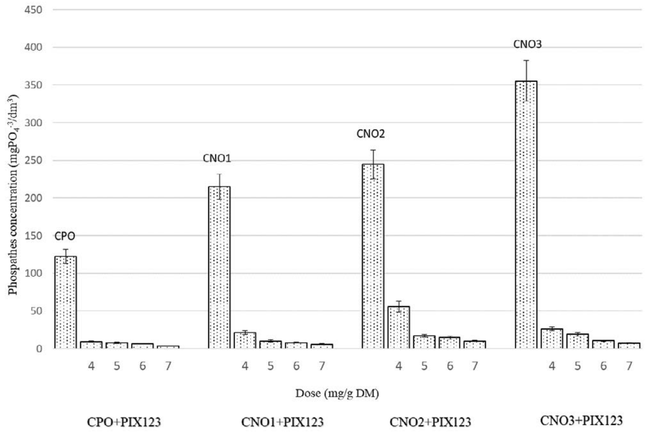

Fig. 2. Changes in phosphate concentration in sludge liquids obtained from digested, sonicated sludge and conditioned with coagulant PIX 123.

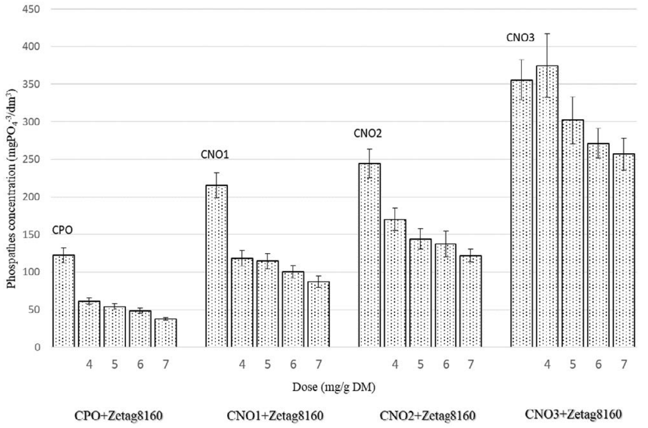

Fig. 3. Changes in phosphate concentration in sludge liquids obtained from digested, sonicated sludge and conditioned with polyelectrolyte Zetag 8160.

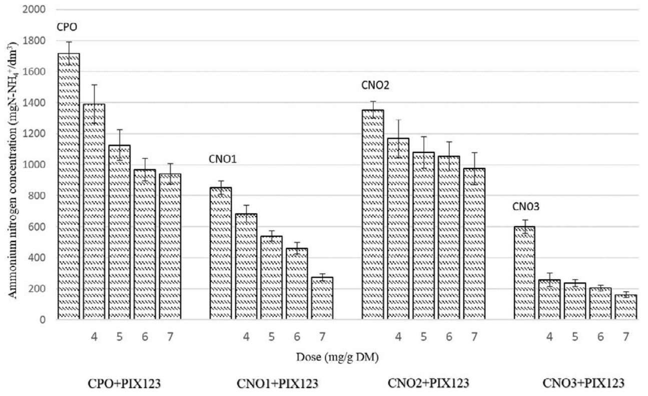

Fig. 4. Changes in ammonium nitrogen concentration in sludge liquids obtained from digested, sonicated sludge and conditioned with coagulant PIX 123.

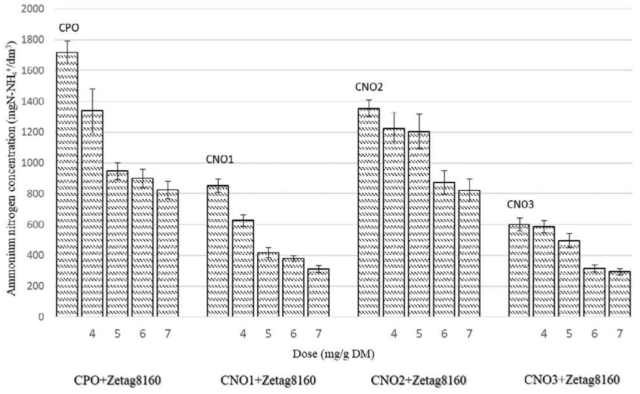

Fig. 5. Changes in ammonium nitrogen concentration in sludge liquids obtained from digested, sonicated sludge and conditioned with polyelectrolyte Zetag 8160.

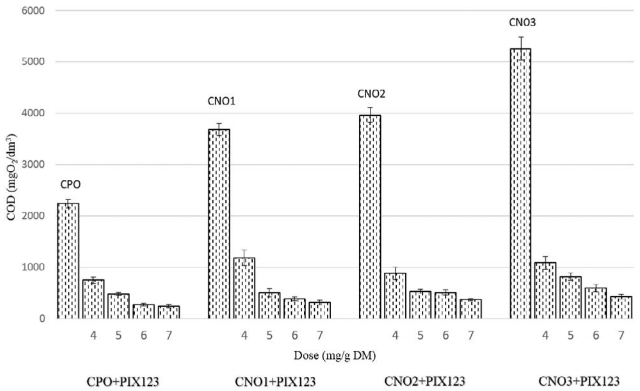

Fig. 6. Changes in chemical oxygen demand concentration in sludge liquids obtained from digested, sonicated sludge and conditioned with coagulant PIX 123.

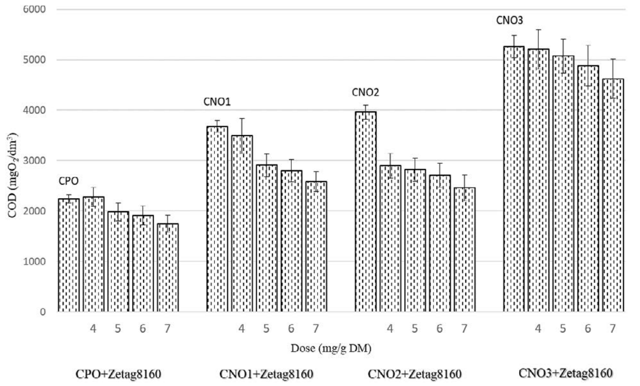

Fig. 7. Changes in chemical oxygen demand concentration in sludge liquids obtained from digested, sonicated sludge and conditioned with polyelectrolyte Zetag 8160.

showed that it is possible to influence the amount of biogenic compounds in sludge liquids through the process of sludge conditioning.

4. Conclusions

- Physical sludge conditioning by sludge sonification increases the phosphate and organic compounds concentration in sludge liquids. An increase in the amplitude of the ultrasonic wave causes an increase in the concentration of these compounds. However, the introduction of chemicals for sludge conditioning results in a decrease in the content of phosphate and organic compounds in sludge liquids as the doses of chemicals increases.
- The highest concentration of phosphates in samples was obtained in sludge liquids obtained from sonicated sludge with A = 45.75 μm. It was 355.5 $\text{mg}\cdot\text{PO}_4^{3-}/\text{L}$. The lowest concentration of phosphates was noted in sludge liquids obtained from non-sonicated sludge but chemically conditioned with PIX 123. The value of 3.6 $\text{mg}\cdot\text{PO}_4^{3-}/\text{L}$ was achieved when a dose of 7.0 mg/g DM was applied in sludge conditioning. In general, phosphates concentration in sludge liquids decreases as the dose of chemicals added to sludge increases.
- The highest COD concentration in sludge liquids was 5,260 $\text{mg}\cdot\text{O}_2/\text{L}$. It was obtained for liquids from sonicated sludge (A = 45.75 μm, t = 60 s). The COD concentration was significantly higher than the COD concentration in raw sludge liquids. Conditioning of sludge with chemicals had an effect on the reducing in COD concentration, which decreases as the chemical dose used for sludge conditioning increases. The lowest COD concentration was achieved in sludge liquids obtained from non-sonicated sludge but chemically conditioned with PIX 123 at a dose of 7.0 mg/g DM.
- The concentration of ammonium nitrogen in sludge liquids, obtained from sonicated sludge, decreases with increasing amplitude of ultrasonic wave. The highest concentration of ammonium nitrogen was noted in raw sludge liquids. In contrast, the lowest content of ammonium nitrogen (162 $\text{mg}\cdot\text{N–NH}_4^+/\text{L}$) in sludge liquids was obtained when sludge was sonicated with an amplitude of 45.75 μm and PIX 123 was added at a dose of 7.0 mg/g DM.
- The recovery of phosphorus and nitrogen from sludge liquids is gaining a lot of interest due to the potential for high-quality products. Some sludge conditioning methods, for example, sonification, affect the nutrient content of sludge liquids so this process is an important element in sludge management prior to nutrients recovery technologies.

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