



# Article Conditioning of Sewage Sludge with Physical, Chemical and Dual Methods to Improve Sewage Sludge Dewatering

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Abstract: The paper presents the impact of different methods of sewage sludge conditioning on the improvement of sludge dewatering during pressure filtration processes. The following conditioning methods were tested for sludge preparation: sonication, addition of organic and inorganic chemicals (Zetag 8180, PIX 113 and the combined action of both substances). The research covered: physical and chemical analysis of sewage sludge, measurement of capillary suction time as an indicator of sludge dewaterability, some technical parameters of sludge pressure filtration process and the analysis of filtrate to assess the degree of contamination. The results of the research showed that the final water content of the prepared sludge decreased, while the specific filtration resistance increased. Among the tested methods the best results of sludge dewatering effects were obtained for sonicated sludge and its preparation with inorganic coagulant PIX 113. The combined effect of sonication with the addition of chemicals Zetag 8180 and PIX113 to sludge allowed for the reduction of organic substances, ammonium nitrogen and phosphates in filtrate after sludge dewatering.

Keywords: sewage sludge; conditioning; dewatering; pressure filtration

## 1. Introduction

Sewage sludge is a multi-component mixture consisting mainly of 95% water, organic compounds, microorganisms and colloids. The high water content in sludge contributes to the high costs of further sludge treatment. Moreover, due to sludge properties, it may pose problems related to its final disposal [1–5]. An important element of the sludge treatment process in sewage treatment plants is sludge dewatering. This action is to reduce the hydration and volume of the sludge, and facilitate further sludge processing, ensure economic viability during final sludge disposal [6–10]. Depending on the susceptibility of sewage sludge to mechanical dewatering, the sludge hydration may vary from 95–99% to 65–85% [11,12]. Without modification of the sewage sludge structure (sludge conditioning) even properly designed and operated mechanical dewatering devices do not guarantee high efficiency [13,14].

There are many ways to modify sludge structure in the conditioning process; however, the most common method, currently used at many wastewater treatment plants (WWTPs), is the use of inorganic and organic coagulants [5,15–19]. A model algorithm, which would use the properties of polyelectrolytes or other chemical agents to calculate the most advantageous dose allowing for the best dewatering effects, has not been developed yet [2]. To reduce the consumption of chemical reagents, sludge conditioning processes are modified, and also new methods of sludge preparation prior to dewatering are investigated, such as application of physical conditioners [20–25]. Many scientific reports confirm the usefulness of various structure-forming materials, such as: cement, lime, gypsum, hard coal ashes, biomass ashes, brown coal, wheat chips and bran, rice biochar, rice husk and nut shells [26–35]. Within the group of physical conditioners inorganic coagulants may also modify the sludge structure and reduce its compressibility. This is particularly advantageous when high-pressure dewatering methods (e.g., filter presses) are used because the



Citation: Bień, B.; Bień, J.D. Conditioning of Sewage Sludge with Physical, Chemical and Dual Methods to Improve Sewage Sludge Dewatering. *Energies* **2021**, *14*, 5079. https://doi.org/10.3390/en14165079

Academic Editor: Agnieszka Cydzik-Kwiatkowska

Received: 27 July 2021 Accepted: 16 August 2021 Published: 18 August 2021

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**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). porosity of the sludge cake must be kept under high pressure [36,37]. For better sludge dewatering effects, the physical conditioners are combined with chemicals. Wójcik et al. [11] found the use of polyelectrolyte in combination with biomass ash to be the most effective. The double conditioning method did not significantly improve the sludge dewatering compared to the use of polyelectrolyte only, but it reduced the polyacrylamide dose by half and in result the sludge dewatering cost was lowered by up to 30%. On the other hand, Zhu et al. [38] used combined sludge conditioning with the use of ultrasounds, traditional cationic polyacrylamide and rice husk to improve the sludge dewatering efficiency. Optimal dehydration of sludge was obtained under the following conditions: sonication was performed with ultrasounds of 22 kHz, the power of 0.3 W/mL and for a time of 12 s, the dose of rice husk and polyacrylamide was 50.0% by weight and  $20 \text{ mg/dm}^3$  respectively. As a result the lowest sludge filter cake hydration of 62.22% was achieved. Cao et al. [39] used an aluminum salt coagulant for sludge conditioning and investigated the mechanisms of interaction between sewage sludge particles and aluminum salt coagulants of different hydroxy-aluminum speciation. They found that medium and high polymerization of aluminum salt coagulant performed better than monomeric aluminum and oligomeric aluminum in reducing the specific filtration resistance and compressibility of sludge. In addition, polymeric aluminum salt coagulants with a medium and high polymerization state showed better compression properties of the structure of extracellular polymeric substances. It improves the filtration efficiency of sewage sludge. Xu et al. [40] showed that the combination of methanol and inorganic coagulants (PAC or FeCl<sub>3</sub>) improved the sludge dewatering process, as well as the dryness of the sludge cake. Masihi et al. [41] used acid-modified bentonite to improve the conditioning and dewatering processes of anaerobically digested sludge. Its action has been compared with inorganic salts, incl.  $FeCl_3$ ,  $AlCl_3$ ,  $Al_2(SO_4)_3$  and  $Fe_2(SO_4)_3$ . Modified bentonite reduced the specific filtration resistance, capillary suction time and sludge filtration time by 95.8%, 90.4% and 80.8%, respectively. The compressibility of the sludge has decreased also significantly. Conditioning of sludge with modified bentonite resulted in a significant increase in the particle size of the sludge and formation of denser and stronger sludge flocs. Parker et al. [42] showed that the chemical structure of the polymer influences the ability to dewater the sewage sludge. Conditioning and dewatering of sewage sludge is a complex process. The cited studies indicate many different solutions to increase the dewatering capacity of sludge, which may be an alternative or supplementary to the chemical method used at WWTPs.

During sludge thickening and dewatering the filtrate is separated in the process, and it is usually highly contaminated [43,44]. Filtrate is most often returned to the sewage treatment process line without any treatment. The amount of sludge filtrate is varied and most often amounts to 0.4–12% of the wastewater volume flowing into WWTP [45,46], although some literature sources give about 20% [47]. Moreover, the high variability in the filtrate inflow and the uneven inflow of the pollutant load reduces the effectiveness of individual devices, which negatively affects the total effectiveness of biological treatment at WWTPs [48,49]. In general, the quality of sludge filtrate is very variable. It depends on the technological system of wastewater treatment and the sewage sludge processing (especially the method of sludge stabilization and dewatering) [49–53]. The problem of back-loads from sludge treatment is more and more often noticed [54–57].

The aim of the research, in which independent and combined methods of sewage sludge conditioning applied, was to assess the impact on the effectiveness of the pressure filtration process and the quality of the obtained sludge filtrate. Table 1 presents the advantages of chemical and combined (chemical and physical) methods applied in sewage sludge conditioning.

	Independent (Chemical) Methods	Co	mbined (Chemical and Physical) Methods
•	a large selection of various types of polyelectrolytes	•	the reduction of sewage sludge compressibility
•	the increase in the amount of dry mass in the dewatered sludge	•	the increase in the amount of dry mass in the dewatered sludge
•	the destruction of the colloidal system	•	the limitation of chemical compounds introduced into the environment
•	the most commonly used in practice	•	the reduction in cost of chemical reagents

Table 1. Advantages of independent and combined methods of sewage sludge conditioning.

The article presents the results of tests with the use of polyelectrolyte Zetag 8180, inorganic coagulant PIX 113 and an ultrasonic field as a medium favorable for the sludge conditioning due to the simplicity of its implementation at WWTPs and the lack of secondary contamination.

#### 2. Materials and Methods

A digested sewage sludge (DSS) from a municipal wastewater treatment plant with a capacity of 40,000 m<sup>3</sup>·d<sup>-1</sup> was used for the study. The sludge was collected at a mechanical dewatering station before dewatering. To ensure the appropriate process conditions, samples of sludge were stored in the fridge at 4 °C and warmed up to room temperature prior to tests. Parameters of sludge: pH = 7.98, specific filtration resistance (SRF):  $2.52 \times 10^{13}$  m/kg, hydration 97.8%, color: gray-black, odor: earthy. Capillary suction time (CSK) was 4291 s, the digested sludge is difficult to dewater. To improve sludge dewaterability the following chemical reagents were used: cationic polyelectrolyte Zetag 8180 and inorganic coagulant PIX 113. Zetag 8180 is an acrylamide polymer and a quaternary cationic monomer. It is supplied as a white powder manufactured by Brenntag NV [58,59]. An inorganic coagulant PIX 113 is classified as a structure-forming substance in sludge conditioning. PIX 113 is an iron coagulant, iron (III) sulphate (VI), an aqueous solution of iron (III) sulphate (VI) with a dark brown color, no odor, the content of total iron Fe is  $11.8 \pm 0.4\%$ , while iron ions Fe<sup>+2</sup> are  $0.4 \pm 0.3\%$  [60,61]. PIX 113 is produced by Kemipol. Before dewatering, sludge samples were also disintegrated. Disintegration was carried out with Sonics VC750 microprocessor ultrasonic disintegrator at a frequency of 20 kHz. The sonication of sludge samples was carried out for the time of 60 s and two different amplitudes were applied:  $A_1 = 15.25 \ \mu m$  and  $A_2 = 45.75 \ \mu m$ . In the case of  $A_1$  amplitude, the energy introduced into the medium was 824 J  $\pm$  25, while for A2: 4419 J  $\pm$  65. The sludge samples were sonicated under static conditions in a constant sample volume of 400 mL. Next, 0.1% Zetag 8180 polyelectrolyte solution, 10% PIX 113 coagulant solution or a combination of both chemicals were added. Samples were mixed using a Biosan MMS-3000N magnetic stirrer according to the following scheme. After adding chemical reagents to the sonicated sludge, rapid mixing was performed for 60 s (200 rpm) to mix the sample thoroughly, then a slow mixing for 300 s (30 rpm) was applied. In the case when both chemical reagents were used for sludge conditioning, PIX 113 was used first with the same mixing procedure. Zetag 8180 polyelectrolyte was then applied as a second, and after 120 s of contact, the sample volume was mixed again for 120 s (120 rpm). The research was conducted in two stages (Table 2).

In the first stage, the sludge samples were sonicated within the time of 60 s with an amplitude  $A_1 = 15.25 \mu m$ , and Zetag 8180 or PIX 113 were added in a given doses. When both reagents were used, PIX 113 was given at a constant dose of 1.0 mg/g DM, while Zetag 8180 was given with variable doses. PIX 113 costs more than polyelectrolyte, so the economic aspect was taken into account as well as a good dewatering effect. In the second stage, the amplitude of the ultrasonic field was increased to  $A_2 = 45.75 \mu m$ .

No.	I Phase Sonication: $A_1 = 15.25 \ \mu m$ , t = 60 s			II Phase Sonication: $A_2 = 45.75 \ \mu m$ , t = 60 s		
	Zetag 8180, mg/g DM	PIX 113, mg/g DM	PIX 113 + Zetag 8180, mg/g DM	Zetag 8180, mg/g DM	PIX 113, mg/g DM	PIX 113 + Zetag 8180, mg/g DM
1.	4.0	4.0	1.0 + 4.0	4.0	4.0	1.0 + 4.0
2.	5.0	5.0	1.0 + 5.0	5.0	5.0	1.0 + 5.0
3.	6.0	6.0	1.0 + 6.0	6.0	6.0	1.0 + 6.0
4.	7.0	7.0	1.0 + 7.0	7.0	7.0	1.0 + 7.0

Table 2. Research stages.

After sludge conditioning, 100 mL of the sludge samples were poured into the cylinder with ET 18II polyester fabric filter, and the pressure filtration process was carried out at a pressure of 0.5 MPa. Sludge dewaterability was measured by a capillary suction time (CST) and a specific filtration resistance (SRF). The capillary suction time measurement was based on the Baskerville and Galle methodology, which measures the passage of the head filtrate layer due to a suction force of the Whatman filter paper [62]. The specific filtration resistance was determined based on the PN-EN 14701-2:2013 standard [63]. This method follows the flow of a fluid through a porous medium in accordance with Darcy's law.

For sludge filtrate, the following parameters were determined: pH—potentiometric method (pH-meter CP401—by Elmetron), total suspension (TS) by gravimetric method, chemical oxygen demand (COD) by the abbreviated dichromate method based on the PN-ISO 6060:2006 [64], ammonium nitrogen and phosphates  $PO_4^{-3}$  by spectrophotometry (Spectrophotometer JENWAY 6300).

Each test was repeated three times and the result is given as an average with a standard deviation. Table 3 presents the symbols assigned to each test.

No.	Symbols	Explanation
1.	ON	sonicated sludge
2.	ON + Zetag 8180	sonicated sludge + Zetag 8180 at a selected dose (e.g., 4, 5, 6, 7 mg/g DM)
3.	ON + PIX 113	sonicated sludge + PIX 113 at a selected dose (e.g., 4, 5, 6, 7 mg/g DM)
4.	ON + PIX 113(1) + Zetag 8180	sonicated sludge + PIX 113 at a constant dose of 1.0 mg/g DM + Zetag 8180 at a selected dose (e.g., 4, 5, 6, 7 mg/g DM)

 Table 3. Symbols assigned to tests carried out during the research.

## 3. Results

The conditioning of the sonicated sludge with Zetag 8180 and PIX 113 independently or using both chemical reagents simultaneously showed an improvement in sludge dewatering, which is confirmed by the results of the capillary suction time (CST) test. The decrease in CST of the sonicated sludge ( $A_1 = 15.25 \mu m$ ) prepared with Zetag 8180, PIX 113 and with both reagents together was respectively: 82.7%; 98.5% and 93.4% (Figure 1). The smallest CST value was obtained for the sonicated sludge prepared with PIX 113 at the highest dose of 7.0 mg/g DM. On the other hand, for sludge sonicated with  $A_2 = 45.75 \mu m$ , the reduction in CST for selected chemicals was respectively: 52.6%; 97.3% and 80.6% (Figure 2). The smallest CST occurred again when sonicated sludge was prepared with PIX 113 at a highest tested dose of 7.0 mg/g DM.







Figure 2. Effect of the dose of selected chemicals on CST of sonicated sludge ( $A_2 = 45.75 \mu m$ ).

A pressure filtration was selected as a mechanical dewatering process. Research on the process of pressure filtration of sewage sludge was carried out for sonicated sludge prepared with selected combinations of chemical reagents. During the tests the greatest attention was focused on specific filtration resistance (SFR) and the effect of sludge dewatering. Figures 3 and 4 presents results of specific filtration resistance achieved for individual methods of sludge conditioning. The analysis of the results presented in Figure 3 shows that the highest specific filtration resistance ( $6.67 \times 10^{13} \text{ m/kg}$ ) was obtained for the sludge conditioned with the combination of PIX 113 and Zetag 8180 at a dose of 7.0 mg/g DM. In general, the specific filtration resistance increased with the increase of the dose of chemicals used. A similar tendency was observed in Figure 4 for sludge sonicated at A<sub>2</sub> = 45.75 µm. In this case the highest specific filtration resistance ( $5.78 \times 10^{13} \text{ m/kg}$ ) was obtained for the

sludge conditioned with PIX 113 at the dose of 7.0 mg/g DM. The factor determining the increase of SFR for each combination of sonicated sludge was the dose and type of chemical reagent used. The highest values of specific resistance were obtained for the inorganic coagulant PIX 113. The lowest value of the filtration resistance was obtained for samples of sludge that were only sonicated. For a higher ultrasonic field amplitude, a smaller value of SFR was obtained.



**Figure 3.** Effect of the dose of selected chemicals on SFR of sonicated sludge ( $A_1 = 15.25 \mu m$ ).



Figure 4. Effect of the dose of selected chemicals on SFR of sonicated sludge ( $A_2 = 45.75 \mu m$ ).

The effects of dewatering of the sonicated sludge prepared with chemical reagents: Zetag 8180, PIX 113 and combined ones are shown in Figures 5 and 6.



**Figure 5.** Effect of the dose of selected chemicals on the final hydration of prepared sludge  $(A_1 = 15.25 \ \mu m)$ .



**Figure 6.** Effect of the dose of selected chemicals on the final water content of prepared sludge  $(A_2 = 45.75 \ \mu m)$ .

The water content of sewage sludge sonicated with  $A_1 = 15.25 \mu m$  was 86%. It was found to be the highest water content. The final water content for sludge conditioned with chemicals was 81.5–86% (Figure 5). The lowest value of the final water content (81.5%) was obtained for the sludge with the addition of PIX 113 at a dose of 7.0 mg/g DM, and the highest was for the sludge prepared with Zetag 8180 at the dose of 4.0 mg/g DM. The final water content of sludge sonicated with  $A_2 = 45.75 \mu m$  was 85% (Figure 6). For other cases, it was found that the final water content was in the range of 80.5–85%. The data presented in Figure 6 shows that the final water content of sonicated and chemically conditioned sludge was slightly lower than the values achieved for sonicated sludge only. With the increase in the dose of chemicals added to the sonicated sludge the value of the final water content decreased slightly in almost every case. The greatest drop in final water content was observed for the sludge prepared with PIX 113 at the dose of 7.0 mg/g DM—it was 80.5%. In general, better dewatering effects were observed when chemical reagents were used at a higher dose. The best results of final water content in sonicated sludge were achieved when an inorganic coagulant was used separately.

The filtrate after dewatering of sonicated sludge ( $A_1 = 15.25 \ \mu m$ ) was characterized by the following parameters: pH = 7.8; concentration of ammoniacal nitrogen—853 mgN-NH<sub>4</sub><sup>+</sup>/dm<sup>3</sup>, phosphates—215.2 mgPO<sub>4</sub><sup>3–</sup>/dm<sup>3</sup> and organic compounds marked as COD— 3679.2 mgO<sub>2</sub>/dm<sup>3</sup>. Conditioning of sludge with PIX 113 caused a decrease in pH (Figure 7) along with an increase dose of coagulant. When polyelectrolyte Zetag 8180 was used, an increase in pH was observed.



**Figure 7.** pH changes in filtrate after sludge dewatering. Sludge sonicated ( $A_1 = 15.25 \mu m$ ) and prepared with selected chemical reagents.

The concentration of organic compounds expressed as COD in filtrate after dewatering of prepared sludge is given in Figure 8. The highest value of COD was observed for sonicated sludge. Conditioning of sludge with chemical reagents caused a decrease in COD. The best quality of filtrate after dewatering was obtained for sludge prepared with PIX 113. The highest efficiency of organic compound removal was achieved when PIX 113 was used in a dose of 7.0 mg/g DM. Then the lowest COD value was 386.4 mgO<sub>2</sub>/dm<sup>3</sup>, and COD was reduced by 89.5%. Conditioning of sludge with chemical reagents caused a decrease in phosphates and ammonium nitrogen in filtrate after dewatering (Figures 9 and 10). The lowest phosphate value of 6.2 mg P-PO<sub>4</sub><sup>3-</sup>/dm<sup>3</sup> was observed for the same sample, when PIX 113 was used at the dose of 7.0 mg/g DM. The phosphate concentration was reduced then by 97.1%. On the other hand, the lowest value of ammoniacal nitrogen was obtained in filtrate for the samples of sludge prepared with Zetag 8180. The lowest concentration of ammoniacal nitrogen (206 mg N-NH<sub>4</sub><sup>+</sup>/dm<sup>3</sup>) was observed at the dose of 7.0 mg/g DM. The reduction of N-NH<sub>4</sub><sup>+</sup>/dm<sup>3</sup> was 75.8%.



**Figure 8.** COD changes in filtrate after sludge dewatering. Sludge sonicated ( $A_1 = 15.25 \mu m$ ) and prepared with selected chemical reagents.



**Figure 9.** Phosphate changes in filtrate after sludge dewatering. Sludge sonicated ( $A_1 = 15.25 \mu m$ ) and prepared with selected chemical reagents.



**Figure 10.** Ammoniacal nitrogen changes in filtrate after sludge dewatering. Sludge sonicated ( $A_1 = 15.25 \mu m$ ) and prepared with selected chemical reagents.

### 4. Discussion

The effectiveness of sludge conditioning with chemicals is related to the change of sludge structure and to increase the spacing between solid particles in sludge [65]. As a result, more water may be released from the sludge. The use of physical conditioners can only improve the strength and permeability of sludge. Methods using both chemical and physical substances may show a better efficiency in sludge dewatering [66]. The achieved results showed the reduction of the capillary suction time parameter when sonicated sludge was prepared with selected chemicals. The sonicated sludge was mixed with organic or inorganic chemicals so the solid phase of sludge, in combination with a finer and colloidal turbid substance, coagulates, forms flocs and dehydrates. Based on the CST test, it was found that the sonicated sludge prepared with PIX 113 showed the best properties for dewatering. The final water content of raw sludge after pressure filtration was 80%. The sonication of the sludge increased the value of final water content in both cases (A<sub>1</sub> = 15.25  $\mu$ m, A<sub>2</sub> = 45.75  $\mu$ m), increasing the amplitude of the ultrasonic wave, i.e., the energy with the sludge was applied during sonication only slightly improved the sludge final water content after pressure filtration. This is related to the low permeability of sonicated sludge which can easily deform under pressure. As a result, the removal of water from such sludge is difficult, which was also reported in [28]. The disintegration of sludge particles during sonication prior to sludge chemical conditioning improved the sludge processing. The sludge flocs formed a denser cake filter during pressure filtration. The specific properties of the chemicals used for sludge conditioning destabilized the sludge colloidal systems. Inorganic coagulant PIX 113 improved the aggregation of sludge flocs and the best results of final water content in sonicated sludge were achieved. The lowest final water content in the filter cake was observed for the sludge prepared with PIX 113 at the dose of 7.0 mg/g DM, showing the synergy between sonication and using a chemical reagent. The smaller sludge particles formed during sonication were then combined into larger particles by the effect of flocculation.

Sludge conditioning prior to dewatering aims to achieve a high concentration of dry matter in sludge and high-quality filtrate. This is to be facilitated by various physical and chemical methods changing the physico-chemical properties of sludge. The applied methods for sludge preparation before its dewatering resulted in a reduction of pollutants in the filtrate in comparison to filtrate achieved from sonicated sludge [44,67]. The best effect of removing organic compounds and phosphates was obtained for filtrate from sonicated sludge prepared with PIX 113, respectively—89.5%; 97.1%. The best results for ammonia nitrogen reduction was observed when sonicated sludge was prepared with Zetag 8180 (75.8%). However, Zetag 8180 was the least effective chemical reagent in reducing the impurities in the filtrate after sludge pressure filtration.

#### 5. Conclusions

The use of polyelectrolytes in sludge conditioning and dewatering was a common solution in terms of the efficiency of sludge pressure filtration, but was not sufficient. Various methods are currently developed to improve sludge dewatering and obtain a high-quality filtrate. In the study, physical and chemical methods were used. The digested sludge was initially exposed to the ultrasonic field for 60 s. Two amplitudes of ultrasonic wave were used (A<sub>1</sub> = 15.25; A<sub>2</sub> = 45.75  $\mu$ m) to investigate the effect of wave power on sludge fragmentation and its further dewatering behavior. Then sludge was conditioned with selected organic and inorganic chemicals. Sonication of sludge increased the capillary suction time. However, the combination of sonication and chemical application reduced the capillary suction time. The best effect of reducing the capillary suction time (CST = 73 s) was observed for sludge sonicated with  $A_1 = 15.25 \ \mu m$  and prepared with PIX 113 at a dose of 7.0 mg/g DM, then for PIX 113(1.0) + Zetag 8180 at a dose of 7.0 mg/g DM-313 s and Zetag 8180 at a dose of 7.0 mg/g DM-824 s. Chemicals dosing to the sonicated sludge with  $A_2 = 45.75 \ \mu m$  allowed us to achieve better dewatering effects to the sonicated sludge with  $A_1 = 15.25 \mu m$ . However, the difference in final water content was not significant. The greatest decrease in final water content was found for the sonicated sludge with  $A_2 = 45.75 \,\mu\text{m}$  and was prepared with inorganic coagulant PIX 113 at the dose of 7.0 mg/g DM. The reduction was 80.5%. Then, the final water content was 81% for Zetag 8180 at a dose of 7.0 mg/g DM, and 82% for PIX 113(1.0) + Zetag 8180 at a dose of 7.0 mg/g DM. The final water content parameter was more appropriate for the evaluation of the sludge dewatering than the specific filtration resistance parameter. The combination of different methods in sludge conditioning reduced the contamination in filtrate. The most effective at obtaining the highest quality of filtrate was the combination of sonication together with the application of inorganic coagulant PIX 113 for sludge conditioning prior to dewatering. The type of sludge conditioning method should be selected experimentally at WWTP, depending on the type of sewage sludge. Incorrect selection may result in increased operating costs, and may also cause deterioration of the sludge dewatering effect.

**Author Contributions:** Conceptualization, B.B.; methodology, B.B.; software, J.D.B.; validation, B.B. and J.D.B.; formal analysis, B.B. and J.D.B.; investigation, B.B.; resources, B.B.; data curation, J.D.B.; writing—original draft preparation, B.B.; writing—review and editing, J.D.B.; visualization, J.D.B.; supervision, B.B. Both authors have read and agreed to the published version of the manuscript.

**Funding:** The scientific research was funded by the statute subvention of Czestochowa University of Technology, Faculty of Infrastructure and Environment.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Exclude this statement.

**Conflicts of Interest:** The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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