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The Changes of Dewaterability of Digested Sludge Conditioned via the Dual Chemical Method

Zmiany podatności na odwadnianie przefermentowanych osadów ściekowych kondycjonowanych z zastosowaniem dualnej metody chemicznej

The dual chemical conditioning is a method based on the sequential preparation of sewage sludge using a coagulant followed by the use of a polyelectrolyte prior to its dewatering. This method is usually used ad hoc at sewage treatment plants, especially in the case of operational problems occurring during dehydration of municipal sludge. In this method the coagulant acts as a particle surface charge neutralizer, whereas the polyelectrolyte is a crosslinking agent enabling the formation of agglomerates. This method can result in noticeable enhancements of sewage sludge dewatering and quality of the separated liquids. But it is not free of disadvantages and the main problem concerns the variable efficiency. The susceptibility to dewatering of digested sewage sludge conditioned via the dual method using the PIX 113 iron coagulant and polyelectrolyte Superfloc C-494 was investigated. The research was divided into two stages, depending on the applied volume dose of the coagulant: 2 cm^3/dm^3 (stage I) and 4 cm^3/dm^3 of sludge (stage II). Two doses of polyelectrolyte were also used. The optimal and reduced dose was 4.8 g/kg dry matter of sludge and 3.7 g/kg d.m. A reduction in the optimal dose by 25% was related to the determination of the possibility of replacing a portion of the polyelectrolyte dose with a coagulant dose. The research was conducted for four combinations: combination A - unprepared sludge; combination B - the sludge conditioned only with the use of the polymer; combination C and D - the sludge prepared using the dual chemical method. In the combination D the used the solution of the coagulant was diluted tenfold. The results showed that the sludge conditioned using the dual method (in combination C and D) have a much greater susceptibility to dehydration compared to the sludge treated using the conventional polymer method. It was observed that the use of a higher dose of the coagulant (stage 2) determines the increased susceptibility to dewatering. The best susceptibility to dewatering was found for sludge conditioned with an undiluted solution of the PIX 113 coagulant at a dose of 0.16 kg/kg d.m., corresponding to 1.9 g Fe^{2+}/kg d.m., in combination with 2.7 g/kg d.m. of the Superfloc C-494 polymer (combination C, stage 2). This allowed for an 85% decrease in the CST value as well as a 96% reduction of the filtration resistivity in comparison to sludge conditioned with the use of the polyelectrolyte only. It was also found that the use of the hybrid methods (combinations C and D) allows for obtaining leachates, characterized by the lowest BOD_5 and COD values. In general, it was found that the key to the dewatering of the tested sludge was the use of a dual chemical method, the dual conditioning method was a secondary factor.

Keywords: **municipal sewage sludge, dewatering, dual chemical conditioning, ferric coagulant, polyelectrolyte**

Introduction

Sewage sludge is an unavoidable by-product of the wastewater treatment process. The produced sludge must be adequately processed and then utilized or disposed. The basic methods of sludge treatment include incineration, composting and landfilling [1]. The elementary principle in sludge management is the reduction of its volume by removing water in the dewatering process. Dewatering of sludge is an essential and costly process among the many subsequent steps of sludge processing [1-4].

The selection of suitable equipment for water removal is only a partial success in the process of sludge dewatering. The dewatering ability of sewage sludge is limited by its content of organic matter, colloidal particles, extracellular polymeric substances (EPS) and its highly compressible nature. The necessary step preceding sludge dewatering is conditioning. The conditioning of sludge involves changing the structure of the sludge and the surface conditions of solid particles. This change leads to the reduction in the interfacial surface and reduces the forces bonding water with the surface of the particles. It is possible to achieve in 3 ways [1]:

- by coagulation or flocculation of sludge solid particles,
- by reducing the compressibility of sludge solids to improve sludge cake filterability (SFR),
- by disintegration of sludge in or der to rupture flocs of cells to release trapped (bound) water molecules from EPS.

The known methods of conditioning can be categorized as physical, chemical or biological methods [1].

The most common are the chemical methods based on the application of in organic coagulants such as iron chloride and, especially, organic polyelectrolytes such as polyacrylamides [5] in general, in organic coagulant activity leads to the neutralization of the surface charge of particles. Polyelectrolyte is a crosslinking factor that facilitates the formation of agglomerates [1, 5, 6].

Currently, the usefulness of a number of chemicals as conditioning agents is tested. Wang et al. [7] studied the impact of cationic surfactants on dewaterability of filtered and centrifuged sludge. To increase the settleability and dewaterability of sludge Liu et al. used a newly prepared ferrate solution (NPAF), containing Fe(VI) and KOH [8]. Alum sludge from water treatment plants can also be considered as a conditioning factor. The residual PACl and in organic matter in the alum sludge act as a chemical conditioner and physical conditioner, respectively [9]. Certain advantages in sludge dewatering and its subsequent disposal could be obtained by using a composite conditioner, the Fenton reagent - lime.

Increasing dewaterability of the sludge can be achieved by means of physical conditioning (physical methods). The use of materials known as skeleton builders mainly including gypsum, lignite, fly ash and others, dominate among the physical methods. The addition of these materials reduce the compressibility of sludge and improve the mechanical strength and permeability of sludge solids during compression [1, 10, 11]. Other physical conditioning techniques include the use of thermal pre-treatment [12], freeze/thaw pre-treatment [13] application of ultrasonic waves [14], or microwaves [15]. These conditioning factors affect the filterability by, among others, changing the viscosity, thixotropic behaviour and mean particle size [16].

The biological methods of conditioning are the least frequently used. These methods involve the use of organism cultures and enzyme preparations. Fundamentally, the biological methods mode of action is the impairment of the flocs gel structure through the hydrolysis of EPS present in the sludge [1]. Murugesan et al. reported that *Acidithiobacillus ferrooxidans* culture can be used as an effective sludge conditioning agent to improve the dewaterability [4]. *A. ferrooxidans* in presence of Fe^{2+} improved the sludge dewaterability by bioacidification, in situ generation of iron-flocculant and modification of EPS. This biogenic flocculant treatment provides additional advantages by increasing the calorific value and improving the leachate quality [2, 4, 17].

In many cases, conditioning methods are based on a combination of new or known physical, chemical or biological conditioning agents. There may be dual or multiple combinations. For example, Wu et al. obtained promising results for sludge dewatering by using rice husk biochar modified by ferric chloride as a conditioning agent [18]. In studies of Shi et al. a composite conditioner consisting of Fe^{2+}-activated sodium persulfate and thermal pretreated phosphogypsum was used. It has been demonstrated that a hybrid treatment consisting of microwave irradiation and acidification (sulfuric acid) or alkalization (sodium hydroxide) might promote sludge dewatering. In order to improve the dewaterability of sludge Lin et al. synthesized a new flocculant by grafting silicon, aluminum and iron onto a starch backbone [19]. The issue of sewage sludge conditioning is still open and current. Many sludge conditioning experiments remain in the realm of laboratory tests. It is possible to use a number of conditioning factors, however it has to be remembered that the dewatered sludge must be characterized by a high energy value or fertilizer properties.

This study presents a dual conditioning method based on the sequential dosage of an in organic coagulant and an organic flocculant. In this method the coagulant acts as a particle surface charge neutralizer, whereas the polyelectrolyte is a cross-linking agent enabling the formation of agglomerates. This method is used ad hoc in several wastewater treatment plants in Poland, especially in the case of operational problems during sludge dewatering. the main disadvantage of this method is its unstable efficiency [20, 21]. Therefore, laboratory studies were undertaken in or der to determine the important variables affecting the effectiveness of the conditioning and dewatering processes.

1. Material and methods

The substrate used in the research was digested sewage sludge, obtained from a municipal wastewater treatment plant (WWTP) in Częstochowa. The inflow of wastewater to the WWTP is about 45 thousand cubic meters per day. The WWTP operates in the UCT technological system. Sludge management at the treatment plant includes, among others, mesophilic anaerobic digestion of mixed sludge (primary and excess sludge) in separate anaerobic digestion chambers. Digested sludge is sequentially stabilized in open anaerobic digestion chambers, after which it is mechanically dewatered and dried [22].

The research was conducted for digested sludge taken from the heat exchanger installation. The collected digested sludge was mixed for 24 hours, at room temperature, and the n subjected to testing. The basic physicochemical properties of the examined sludge are summarized in Table 1.

Table 1. **The physicochemical properties of the tested digested sludge**

Parameter	Minimum	Maximum	Average	Standard deviation
Water content, %	95.37	98.21	96.83	1.28
Dry matter, g/dm^3	24.88	47.31	35.68	8.16
Dry organic matter, % of d.m.	37.16	63.58	49.11	10.64
pH	7.18	7.86		
Alkalinity, mg $CaCO_3$/dm^3	1832	3427	2423	644

As conditioning agents an in organic ferric coagulant Kemipol PIX 113 and a medium-cationic polymer Superfloc C-494 were used. PIX 113 is a ferric coagulant based on iron(III) sulphate with a total iron content of nearly 12%. The working concentration of the polyelectrolyte was 0.1%.

The research was divided in to two stages depending on the applied volume dose of the ferric coagulant. In stage 1, a dose of PIX 113 was 2 cm^3/dm^3 of sludge, while in stage 2 the dose was 4 cm^3/dm^3.

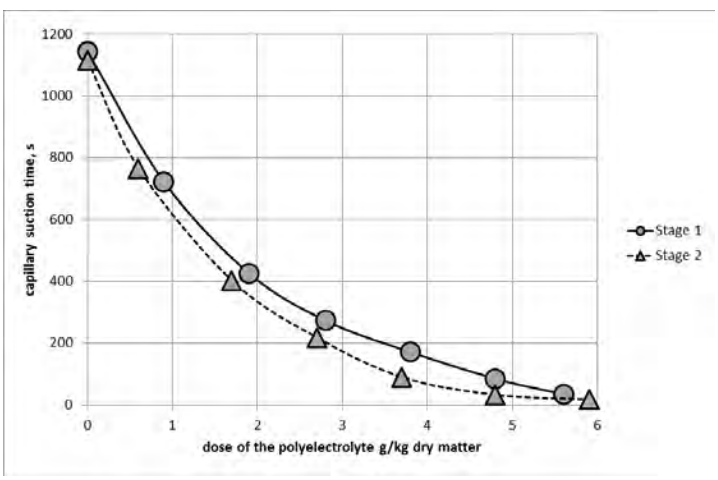

Fig. 1. Effect of polyelectrolyte dose on changes in CST values

The optimal dose of the polyelectrolyte was determined using the capillary suction time test (Fig. 2). The optimal doses of the polyelectrolyte for the first and second stage were 4.8 g/kg d.m. and 3.7 g/kg d.m., respectively, for which CST values of 85 and 89 seconds respectively were noted. For further research, it was decided to use a 25% lower polymer dose of 3.8 g/kg d.m. for stage 1 and 2.7 g/kg d.m. for stage 2.

Lower polyelectrolyte doses used in the study resulted from the addition of the coagulant PIX 113. This allowed for intensification of sludge dewatering by using a dual conditioning method and involved the possibility of replacing the dose of polyelectrolyte with the dose of the coagulant. Due to the varying sludge dry matter content, weight doses were slightly different in the various assays. Table 2 shows the doses of conditioning agents in various stages.

Table 2. **The doses of conditioning agents used in the studies**

Value	PIX 113, kg/kg d.m.		Superfloc C-494, g/kg d.m.	
	Stage 1	Stage 2	Stage 1	Stage 2
Minimum	0.08	0.14	3.6	2.5
Maximum	0.11	0.19	3.9	2.9
Average	0.1	0.16	3.8	2.7
Standard deviation	0.011	0.013	0.18	0.21

The study was conducted for 4 combinations. In Figure 2 the methodology is illustrated.

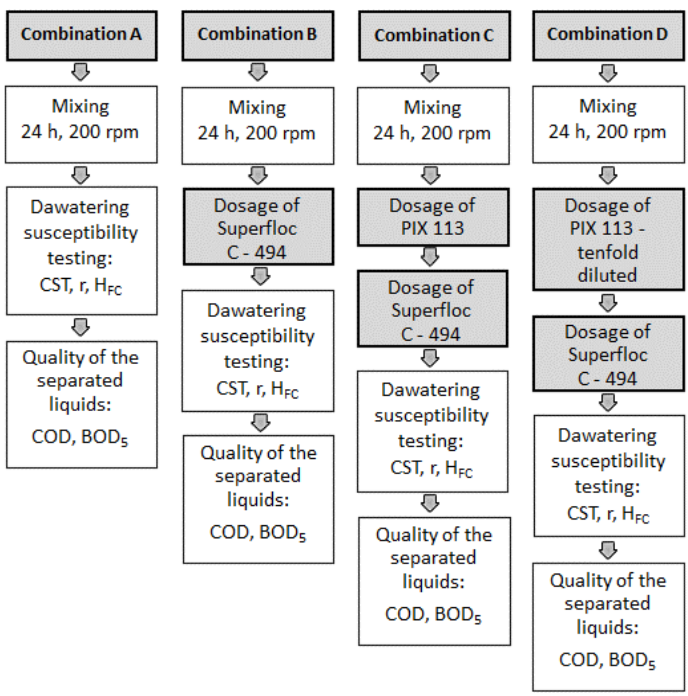

Fig. 2. Research methodology

The determinations were made on the basis of the following methodology:

- Determination of dry matter (DM), water content and water content in filter cake (final hydration in filter cake H_{FC}) were performed according to PN-EN 12880:2004.
- Determination of loss on ignition of dry matter of sludge according to PN-EN 12879:2004.
- Alkalinity and pH determinations were performed in accordance with PN-91/C-04540.05.
- Mixing of the sludge was carried out on a 10 cm diameter paddle stirrer. Before the preparation, samples of 3 liter was stirred for 24 hours at 200 rpm. Subsequently samples of 1 liter were prepared. Each sample was mixed at 400 rpm according to the following methodology: in combination B, the sludge was stirred with the polymer for 20 seconds; in combinations C and D the samples were mixed for 60 seconds with coagulant. After this time, polyelectrolyte was added and stirring continued for 20 seconds.
- Measurement of capillary suction time (CST) was performed according to PN-EN 14701-1:2007. Measured as the transit time of the frontal boundary layer of the filtrate resulting from the suction forces of the used filer paper - Whatman 17.
- Determination of the specific resistance to filtration (r) was carried out according to PN EN 14701-2: 2013-07. 100 ml sample was filtered over a period of 30 minutes at a pressure of 0.05 MPa. As a filtering septum a Whatman 1 paper was used.
- In order to determine the effect of the preparation combinations on the quality of the leachates the sludge samples were centrifuged. It was assumed that the dewatering of sludge surveyed by centrifugation is a more representative way to assess the quality of the leachates. Laboratory vacuum filtration significantly reduces the amount of particulate matter in the filtrate in a manner in dependent of the used conditioning method. The samples were centrifuged for 3 minutes at 3500 rcf. In the decanted liquid five-day biochemical oxygen demand (BOD_5) was defined using a set of the OXI TOP® apparatus via allylthiourea vaccination method in accordance to PN-EN 1899-1: 2002.
- Chemical oxygen demand (COD) was measured via spectrophotometer cuvette tests HACH DR 5000. Prior to the measurement of COD 50 ml samples of leachates were homogenized for 60 seconds using the IKA T10 basic homogenizer.
- In order to compare the two stages of research (stage 1, stage 2) the non-parametric U Mann-Whitney test was used. The aim of the test was to verify the null hypothesis (H_0), which assumes that the susceptibility to dewatering of dually conditioned sludge is not dependent on the dose of the coagulant. Hypothesis verification is based on the values of the test probability (p) and of the accepted significance level alpha ($\alpha = 0.05$). A probability value of less than alpha results in the rejection of the null hypothesis. This means that the values of the studied parameters are significantly different for each stage, and thus

susceptibility to dewatering of dually conditioned sludge is dependent on the dose of the coagulant. When the value of the test probability (p) was smaller than alpha there are no grounds to reject the null hypothesis [23, 24].

2. Results

On the basis of the CST test it was found that dually conditioned sludge showed an increased susceptibility to dewatering in comparison to the sludge conditioned only with the polyelectrolyte. The lowest values of CST were obtained for combination D (Fig. 3). The average CST value of the dually conditioned sludge was 74 s and 42 s respectively for stage 1 and 2 (combination D). For the sludge treated using the conventional polymeric method, the noted CST values were 187 s for stage 1 and 290 s for stage 2. Despite the application of the optimal dose of the polyelectrolyte, there was still a possibility of increasing the susceptibility to dewatering by using a ferric coagulant. Comparing the C and D combinations also showed that there are slight differences between the obtained CST values. This mean that the combination used in the dual conditioning was of secondary importance (undiluted - combination C, or diluted - combination D coagulant), however polyelectrolyte and coagulant doses (stages 1 and 2) were of certain importance.

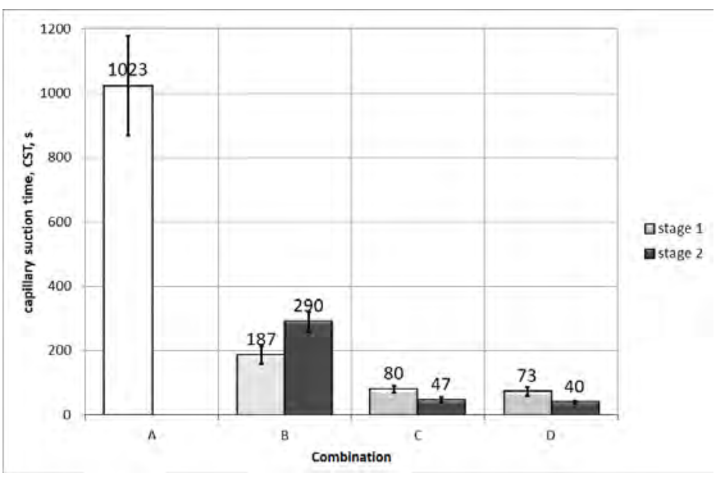

Fig. 3. The influence of the conditioning method on the CST values of sludge

Based on the final hydration of the filter cake (Fig. 4) significant differences in the dehydration ability, depending on the used combination of conditioning, were observed. As a result of the 30 minute vacuum filtration of unconditioned sludge, its hydration was reduced to about 95%. The water content of the filter cake of the sludge conditioned with the polyelectrolyte was close to 90%. A further reduction in sludge hydration was achieved by applying dual conditioning with the use of PIX 113 at a dose of 2 cm^3/dm^3 of sludge (stage 1, combinations C and D). The most significant improvement in filter cake hydration occurred for a volume dose of PIX 113 of 4 cm^3/dm^3 (stage 2, combinations C and D). The resulting hydration of the filter cake was below 80%, which is the basic requirement for mechanically dried sludge. The use of the dual conditioning, in particular for stage 2 for combinations C and D enabled the reduction of hydration by 8 and 12% compared to the effects of dewatering of sludge conditioned only with the polymer.

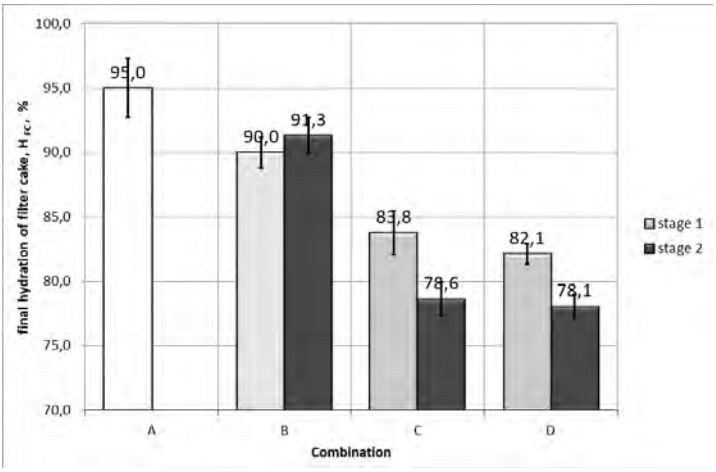

Fig. 4. The effect of the conditioning method on final hydration of filter cake

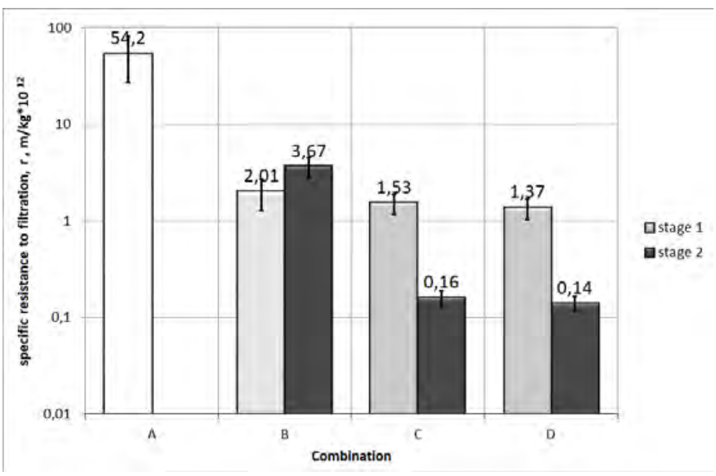

Fig. 5. The influence of the conditioning method on the specific resistance to filtration of tested sludge

The use of dual conditioning methods was also beneficial due to the achieved values of specific resistance to filtration. However, it resulted mainly from the use of a higher PIX dose (stage 2, Fig. 5) in the dual conditioning method. For combinations C and D, the value of the specific resistance of $0.1 \cdot 10^{12}$ m/kg was obtained. The sludge conditioned with the polyelectrolyte, as well as dually conditioned sludge with a dose of 2 cm^3/dm^3 (stage 1), had very similar filterability, with a filtration resistance value in the range of $2 \div 5 \cdot 10^{12}$ m/kg. It should be noted that, as in the case of final hydration, the dual conditioning (combinations C and D) did not significantly affect the final result. An important variable was the dose of the coagulant. Increasing the dose of the coagulant correlates with a decrease in filtration resistance, irrespectively of the use of PIX in the commercial or diluted form.

According to PN-EN14701-2 the sludge can be considered as filterable on industrial scale, when its specific resistance to filtration is lower than $5 \cdot 10^{12}$ m/kg. The results show that in accordance to PN-EN14701-2 the hybrid conditioned sludge as well as the sludge prepared using the conventional polymeric method are filterable on industrial scale. The results show that the dually-conditioned sludge as well as the polymer-conditioned sludge have an operational filterability. However, the application of dual conditioning of sludge will accelerate the filtration process.

According to the research methodology, BOD_5 and COD values were determined in the leachates from the centrifugation of the tested sludge. It was found that the type of conditioning method did not significantly affect the change of BOD_5 values (Fig. 6). In the first stage (combinations B, C and D) the average value of BOD_5 was about 650 mg O_2/dm^3. The results of the second stage showed that the dual conditioning (C and D) reduces the BOD_5 values by about 150 mg O_2/dm^3, in comparison to combination B.

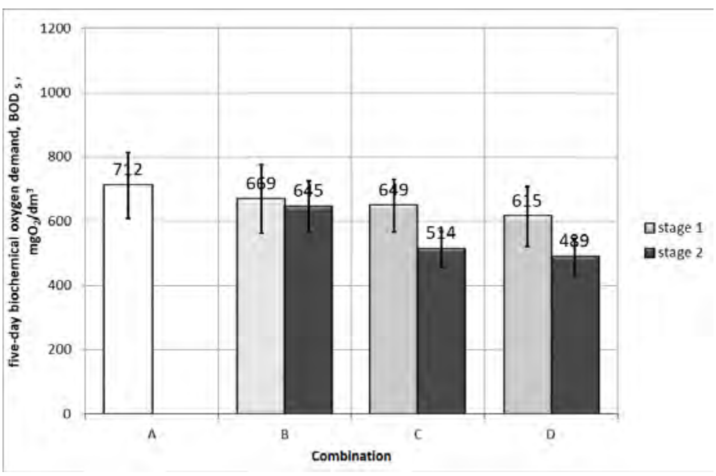

Fig. 6. The influence of the conditioning method on the BOD_5 values in leachates from the dewatering

The chemical oxygen demand of leachates from dewatering of the sludge conditioned with the polyelectrolyte was around $2000 \div 2500$ mg/dm^3 (Fig. 7). The resulting liquids were therefore 3-5 times more concentrated than the raw wastewater. Since leachates from sludge dewatering are recycled to the wastewater treatment process, this means a significant load on the biological treatment. The sludge conditioned using the dual chemical method generated leachate with a COD oscillating around 1770 mg O_2/dm^3 in stage 1, and 1220 mg O_2/dm^3 in the second stage. In general, the use of a dual conditioning method and a high dose of coagulant is the most important aspect in relation to the quality of sludge leachates.

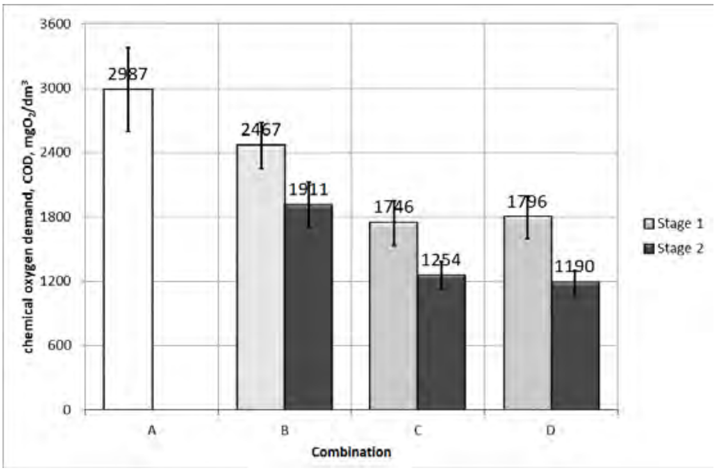

Fig. 7. The influence of the conditioning method on the COD values in leachates from the dewatering

Statistical analysis was also carried out in or der to determine whether the results obtained in both stages of the research were significantly different. The performed U Mann-Whitney test allowed to answer the question, whether susceptibility to dewatering of sludge conditioned by the hybrid chemical method (combinations C and D) depends on the dose of the coagulant (stages 1 and 2). The test probability (p) for the individual parameters is summarized in Table 3.

Table 3. **The results of the statistical analysis U Mann-Whitney test**

	Test probability (p)	
Parameter	Combination C	Combination D
Capillary suction time	0.0092	0.0057
Final hydration	0.5101	0.619
Resistance to filtration	0.00016	0.00021
BOD_5	0.281	0.216
COD	0.00043	0.00065

Based on the test it can be concluded that a higher dose of the coagulant significantly affects most parameters of dually conditioned sludge ($p \leq 0.05$). The exceptions being the final hydration and BOD_5, for which the test probability for combinations C and D was high and exceeded the accepted significance level alpha. Statistically these values were not coagulant dose-dependent. However, due to the fact that the capillary suction time ($p = 0.009$ and 0.05), specific resistance to hydration ($p = 0.00016$ and 0.00021) and COD ($p = 0.00043$ and 0.00065) from a statistical point of view are coagulant dose-dependent.

Conclusions

The use of the dual chemical method of sewage sludge conditioning, based on the dosage of the mineral coagulant and organic polymer significantly affects susceptibility to dewatering of the sludge and the quality of the leachate. It was considered that the most important variable of conditioning was the dose of coagulant. The dilution of the coagulant has no significant impact on the changes of the filtration properties of the sludge and the quality of the leachates.

Analyses show, that by using the PIX 113 coagulant at a dose of 0.16 kg/kg d.m., and the Superfloc C-494 in an amount of 2.7 g/kg d.m. (combination C, stage 2) it is possible to obtain sludge characterised by the best susceptibility to dewatering and quality of leachate. This method of preparation resulted in a decrease of capillary suction time of about 85% compared with sludge conditioned with the polyelectrolyte only. This combination most preferably affected the filtration properties of sludge. The resistance to filtration was 96% lower than that noted for the sludge prepared using only the polymer. The quality of the leachates from sludge dewatering was strongly dependent on the used conditioning combination. The leachate from raw sludge and sludge conditioned only using the polyelectrolyte was of low quality expressed by high chemical oxygen demand. The separated leachates were contaminated by large amounts of unseparated colloids and suspensions. Quality of the leachate was improved by the use of dual conditioning. Higher doses of the coagulant, allowed for a 33% reduction of COD in comparison to the liquids obtained from dewatering of sludge prepared using the polyelectrolyte only.

Acknowledgements

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Streszczenie

Dualne kondycjonowanie osadów ściekowych jest metodą opartą na sekwencyjnym preparowaniu osadów ściekowych koagulantem, a następnie polielektrolitem przed ich dalszym odwadnianiem. Sposób ten jest zazwyczaj stosowany doraźnie na oczyszczalniach ścieków, zwłaszcza w przypadku problemów eksploatacyjnych występujących podczas odwadniania osadów komunalnych. Przyjmuje się, że koagulant pełni funkcję neutralizatora ładunku powierzchniowego cząstek, natomiast polielektrolit traktowany jest jako czynnik sieciujący, umożliwiający łączenie aglomeratów. Metoda ta może przynosić wymierne efekty w zakresie odwodnienia osadów ściekowych oraz jakości odcieków. Nie jest jednak pozbawiona wad, a główny problem dotyczy zmiennej efektywności.

W pracy badano podatność na odwadnianie przefermentowanych osadów komunalnych, dualnie kondycjonowanych z zastosowaniem koagulantu żelazowego PIX 113 oraz polielektrolitu Superfloc C-494. Badania podzielono na dwa etapy, zależnie od stosowanej dawki objętościowej koagulantu: 2 cm^3/dm^3 osadów (etap I) oraz 4 cm^3/dm^3 osadów (etap II). Zastosowano również dwie dawki polielektrolitu. Dawki optymalna i obniżona wynosiły odpowiednio 4,8 g/kg suchej masy osadów oraz 3,7 g/kg s.m. Zmniejszenie dawki optymalnej o 25% wiązało się z określeniem możliwości zastąpienia części dawki polielektrolitu dawką koagulantu. Eksperyment podzielono na 4 kombinacje badawcze: kombinacja A - osady niepreparowane; kombinacja B - osady kondycjonowane konwencjonalną metodą polimeryczną; kombinacje C i D - osady preparowane z zastosowaniem chemicznej dualnej metody kondycjonowania. W kombinacji D dawkowano 10-krotnie rozcieńczony roztwór koagulantu.

Wyniki przeprowadzonych badań potwierdziły, że osady kondycjonowane hybrydowo (kombinacje C i D) wykazują większą podatność na odwadnianie w porównaniu z zastosowaniem konwencjonalnej metody polimerycznej. Zauważono także, że użycie większej dawki koagulantu (etap 2) determinuje zwiększenie podatności na odwadnianie. Najlepsze efekty odnotowano, preparując osady nierozcieńczonym roztworem koagulantu w dawce 0,16 kg/kg s.m., a następnie polimerem w ilości 2,7 g/kg s.m. (kombinacja C, etap 2). Zastosowanie powyższej kombinacji spowodowało spadek czasu ssania kapilarnego o 85% oraz obniżenie oporu właściwego filtracji o 96% w porównaniu z osadami kondycjonowanymi wyłącznie polielektrolitem. Z uwagi na jakość cieczy osadowych osady kondycjonowane hybrydowo (kombinacje C i D) również generowały odcieki o najniższych wartościach BZT_5 i ChZT.

Ogólnie stwierdzono, że kluczowe dla odwadniania badanych osadów było zastosowanie dualnej metody chemicznej, sposób dualnego kondycjonowania był czynnikiem drugorzędnym.

Słowa kluczowe: **komunalne osady ściekowe, odwadnianie osadów ściekowych, dualna chemiczna metoda kondycjonowania, polielektrolit, koagulant żelazowy**