



Characteristics of Excess Sludge Subjected to Disintegration

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1. Introduction

Manuscripts Human functioning in the sphere of existentially – economic risk of generating ever-increasing amounts of sewage and sludge that need to undergo extensive purification processes, followed by neutralization. Issues related to the economy of waste is regulated primarily by the Act of 24.12.2012 r. on waste, which determines the main directions for waste disposal. Raw sludge generated in sewage treatment pose a serious problem to its nuisance. This material is biologically dangerous, easily rotting, emitting unpleasant odors and having pathogenic microorganisms.

Numerous studies indicate that sludge due to the presence of heavy metals and sanitary condition can pose a serious threat to the environment (Dąbrowska 2013, Jacewicz i in. 2015, Ociepa i in. 2013, Pachura i in. 2015). Dedicated in the process of mechanical-biological treatment of sewage sludge must be processed and disposed of. These processes should lead to the maximum economically justified to reduce the weight and volume of sewage sludge and deprivation impact on the environment (Piecuch i in. 2013). Commonly used process of stabilization of sewage sludge over a hundred years is mesophilic digestion. The efficiency of the anaerobic decomposition of sewage sludge is associated primarily with the problem of free time multiplication of bacteria running this process. Particularly sensitive to environmental conditions are methanogenic bacteria. The process of anaerobic stabilization requires no costly aeration and produced biogas is a valuable energy source

(Rozińska & Dąbrowska 2014, Wójtowicz 2014). The need to continuously monitor process conditions and high investment costs for the construction of reservoirs, whether checking apparatus thereby making biogas costly and technologically challenging for most wastewater. Therefore it conducted a series of studies leading to an increase in the efficiency of methane fermentation process by subjecting the sludge disintegration. Produced during anaerobic stabilization of sludge disintegrated high calorific biogas can be used to generate electricity, or be used as a raw material for heating purposes in the same plant. The use of biogas allows for partial reimbursement invested in modern technologies to safely dispose of waste sludge. Sludge disintegration is known to conditioning processes. Its main aim is to release the cell components for faster and more intense stabilization. This is achieved by destroying the structure of the sludge by the action of external forces on it. Consequently, there is a change in the structure of the precipitate and inactivation of microorganisms contained therein. The released are also the organic components inside the cells, that lose their cell membranes upon contact with conditioning agent (Rozińska & Dąbrowska 2014, Wójtowicz 2014). Chemical methods of conditioning of sludge due to the type of reagent added to the sludge divided into methods of chemical acid hydrolysis or alkaline. Exposure to excess sludge of acid or base in a suitable dose leads to a significant change in the pH value (Eastman & Ferguson 1981). By employing a chemical disintegration of the pellet is prepared for anaerobic biodegradation, resulting in shortened biological hydrolysis (Appels I in. 2011). Phase hydrolysis occurs spontaneously and is-limiting methane fermentation process (Perkowski & Zarzycki 2005). There are many methods to assess the degree of excess sludge disintegration, however, describe this process, the number of absolute right for all of these methods is not a simple task. The degree of disintegration depends on many factors such as the composition of the precipitate, the amount and type of energy required to obtain the effect desired, as a function of inter alia polymer content in the sludge, sludge concentration, biological indicators and physico-chemical properties (Eastman & Ferguson 1981).

A method was developed based on measurement of a comparative study, using as a starting reference preparation scheduled sludge level by using an alkaline hydrolysis – CODa (Eastman & Ferguson 1981). This method can be expressed by the formula:

$$DD_{COD} = \frac{(COD_D - COD_0)}{(COD_a - COD_0)} * 100, \% \quad (1)$$

where:

DD_{COD} – the degree of disintegration, %,

COD_D – COD of the liquid supernatant after the disintegration, mg O₂/l,

COD_0 – initial value of COD in the supernatant liquid, mg O₂/l,

COD_a – COD reference sample pellet subjected to chemical hydrolysis with 1-NaOH molar ratio of 1:1, at a temperature of 90°C for a period of 10 minutes, mg O₂/l (Zielewicz 2007).

The aim of this study was to determine the effect of chemical disintegration of excess sludge to increase the concentration of organic substances in the supernatant liquid and the value of the degree of disintegration of modified sludge.

2. Experimental part

For the studies used excess sludge, which was taken from the Central Wastewater Treatment Plant in Czestochowa Warta PSW, which was designed initially as a classical mechanical-biological treatment plant (<http://www.wartasa.eu/10/06/2014>).

Table 1. General characteristics of the research substrate

Tabela 1. Ogólna charakterystyka substratów badań

Indicator	Dry mass	Organic dry mass	VFAs	COD	Kjeldahl nitrogen	Ammonia nitrogen
Unit	g/l	g/l	mg CH ₃ COOH/l	mg O ₂ /l	mg N/l	mg N-NH ₄ /l
Excess sludge	13,43 ±0.16 ^S	9,06 ±0.06 ^S	103 ±3 ^S	133 ±5 ^S	92 ±4 ^S	58±2 ^S

^S - standard deviation

The following physico-chemical indications as: dry mass, organic dry mass, volatile fatty acids, chemical oxygen demand was made (EN-12879, ISO 7027, PN-75/C-04616/04). The degree of sludge disintegration was determined according to the formula (1). To evaluate the chemical disintegration methods in the excess sludge conditioning process is carried out with water solution of 10% peracetic acid. During the test

reagent was applied in a dose of from 0.2 to 4.0 ml/l of sludge, and three times the data doses brought into contact with excess sludge considered: $t = 1, 8, 16$ h. The process of disintegration of chemical consisted of adding to test samples of excess sludge predetermined dose of reagent, exact mixing, the sample must be tightly protected from air and leaving for a certain time at room temperature. The volume of each sample was 0.5 l.

3. Results and discussion

3.1. The term of most favorable conditions for the disintegration of excess sludge by chemical method

The most preferred dose of the water solution of 10% peracetic acid and the time of its contact with the excess sludge is selected on the basis of carrying out three test cycles. During the study, the following reagent dose: 0.2-4.0 ml/l of sludge was applied. The time of pretreatment was 1, 8 and 16 h. Selection of the most favorable parameters of disintegration was made on the basis of changes in the value of COD and VFAs in the supernatant liquid of sludge. With increasing dose of reagent decreased pH. Table 2 shows changes in the value of COD, VFAs and pH after a one-hour process of chemical disintegration.

Table 2. Changes in the VFA, COD, pH of the supernatant liquid of excess sludge undergoing chemical method of pretreatment for 1 h

Tabela 2. Zmiany LKT, ChZT, pH w cieczy osadowej chemicznie dezintegrowanych osadów nadmiernych przez 1 h

Dose of reagent, ml/l of sludge	VFAs, mg CH ₃ COOH/l	COD, mg O ₂ /l	pH
Sample 0	103 ±3 ^s	133 ±5 ^s	7.43
0.2	240 ±5 ^s	231 ±3 ^s	7.01
0.4	565 ±4 ^s	685 ±6 ^{ss}	6.26
0.8	1028 ±7 ^s	1200 ±4 ^s	5.44
1.2	1457 ±5 ^s	1625 ±3 ^s	4.98
1.6	1954 ±3 ^s	2015 ±6 ^s	4.77
2.0	2365 ±7 ^s	2250 ±4 ^s	4.53
4.0	4560 ±6 ^s	4850 ±5 ^s	4.11

^s - standard deviation

The increase in the chemical oxygen demand and volatile fatty acid concentrations had already occurred at the lowest dose of the applied reagent. At each subsequent dose of COD and VFAs gradually increased reaching for a dose of 5.0 ml/l respectively deposits 4850 mg O₂/l and 4560 mg CH₃COOH/l. The dose of 5.0 ml/l of sewage sludge liquor pH was 4.11. This value requires adjusting to the optimum value for the process of anaerobic stabilization. Table 3 shows the change in the value of COD, VFA and pH after a eight-hour process of chemical disintegration.

Table 3. Changes in the VFA, COD, pH of the supernatant liquid of excess sludge undergoing chemical method of pretreatment for 8h

Tabela 3. Zmiany LKT, ChZT, pH w cieczy osadowej chemicznie dezintegrowanych osadów nadmiernych przez 8 h

Dose of reagent, ml/l of sludge	VFAs, mg CH ₃ COOH/l	COD, mg O ₂ /l	pH
Sample 0	103 ±3 ^s	133 ±5 ^s	7,43
0.2	222 ±3 ^s	278 ±4 ^s	7.18
0.4	565 ±6 ^s	711 ±7 ^s	6.72
0.8	1062 ±4 ^s	1342 ±6 ^s	6.12
1.2	1662 ±7 ^s	1787 ±8 ^s	5.00
1.6	2022 ±5 ^s	2170 ±5 ^s	4.71
2.0	2314 ±2 ^s	2421 ±7 ^s	4.57
4.0	4645 ±4 ^s	5012 ±8 ^s	4.10

^s - standard deviation

In the case of eight – hour period of preparing after addition of 0.2 ml/l sludge there was an increase of COD and concentration of VFA in the supernatant liquid of sludge. For a dose of 4.0 ml/l of sludge, the following values of COD and VFAs equal 5012 mg O₂/l and 4645 mg CH₃COOH/l was obtained. However, as in the case of a two-hour disintegration at the highest dose of reagent pH was 4.10, which is quite disadvantageous in terms of carrying out the anaerobic stabilization. Table 4 shows the changes of COD, VFAs concentration and pH after a sixteen-hour chemical disintegration process.

Table 4. Changes in the VFA, COD, pH of the supernatant liquid of excess sludge undergoing chemical method of pretreatment for 16h

Tabela 4. Zmiany LKT, ChZT, pH w cieczy osadowej chemicznie dezintegrowanych osadów nadmiernych przez 16 h

Dose of reagent, ml/l of sludge	VFAs, mg CH ₃ COOH/l	COD, mg O ₂ /l	pH
Sample 0	103 ±3 ^s	133 ±5 ^s	7.43
0.2	257 ±7 ^s	304 ±3 ^s	7.33
0.4	582 ±5 ^s	769 ±8 ^s	6.91
0.8	1097 ±2 ^s	1497 ±4 ^s	6.55
1.2	1542 ±6 ^s	1901 ±8 ^s	5.13
1.6	2040 ±4 ^s	2210 ±7 ^s	4.78
2.0	2348 ±7 ^s	2507 ±4 ^s	4.60
4.0	4700 ±6 ^s	4937 ±5 ^s	4.14

^s - standard deviation

As with the previous two test cycles, there was an increase of COD and concentration of VFAs already at the lowest dose of reagent added to sludge. For the highest dose of 4.0 ml/l of sludge the volatile fatty acids concentration and chemical oxygen demand value was 4937 mg CH₃COOH/l and 4 700 mg O₂/l, at a pH value of 4.14. Given the need to correct the pH of the disintegration process of preparation and adjustment to the optimum value for methane fermentation process the most beneficial dose was 0.8 ml/l of sludge and twelve-hour preparation time. The pH was 6.55, while the COD value and VFAs concentration was 1497 mg O₂/l and 1097 mg CH₃COOH/l.

3.2. Determination of excess sludge disintegration

The degree of sludge disintegration subjected to chemical disintegration calculated based on the value of the dissolved COD marked in the supernatant liquid of sludge. Figures 1, 2, 3 show the value of the degree of disintegration in percent obtained with the chemically modified excess sludge.

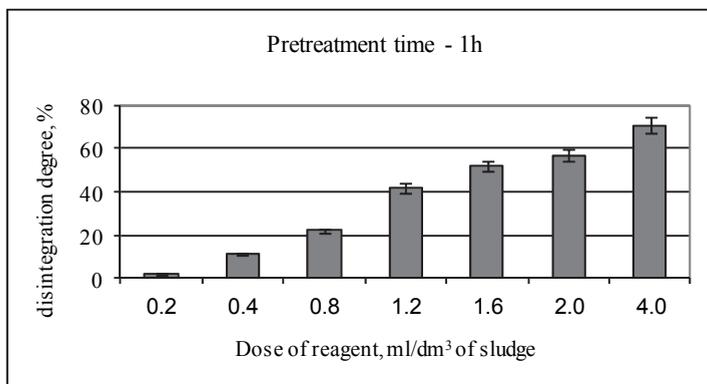


Fig. 1. Changes in the degree of chemically disintegrated sludge by 1 hours depending on the dose of reagent

Rys. 1. Zmiany stopnia dezintegracji chemicznie modyfikowanych osadów przez 1 h w zależności od dawki reagenta

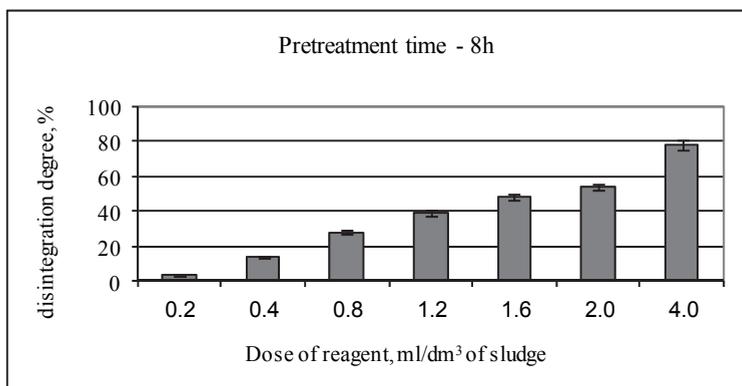


Fig. 2. Changes in the degree of chemically disintegrated sludge by 8 hours depending on the dose of reagent

Rys. 2. Zmiany stopnia dezintegracji chemicznie modyfikowanych osadów przez 8 h w zależności od dawki reagenta

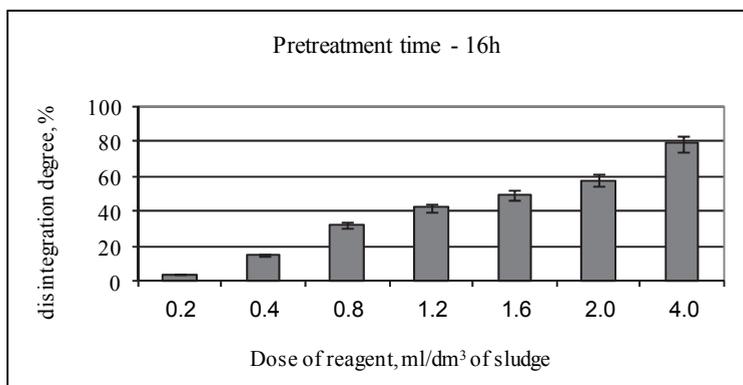


Fig. 3. Changes in the disintegration degree of chemically disintegrated sludge by 18 hours depending on the dose of reagent

Rys. 3. Zmiany stopnia dezintegracji chemicznie modyfikowanych osadów przez 16 h w zależności od dawki reagenta

The highest degree of disintegration achieved with the selected conditioning, ie. the time $t = 1, 8, 16$ hours and the dose of reagent 4.0 ml/l of sludge. For a dose of 0.8 ml/l of sludge and time of preparation 18 hours to give the degree of disintegration amounting to 32%. Disintegration by dose of 0.8 ml/l of sludge for 16 h ($\text{pH} = 6.55$), due to the technological conditions carried out in the next stage of research methane fermentation process was considered the optimal for carrying out the process of anaerobic stabilization.

3.3. Conducting the process of 25-daily anaerobic stabilization of excess sludge

In the first 25 daily anaerobic stabilization of excess sludge was inoculated with non-conditioned sludge digested in a volume ratio of 10:1. Conducting untreated methane fermentation of excess sludge was aimed at getting results by reference to values obtained after the fermentation methane deposits chemically disintegrated. Therefore, it was possible to determine the effectiveness of chemical disintegration, as a method of supporting the process of anaerobic stabilization. Table 5 shows the basic physico-chemical parameters made during anaerobic stabilization of non-prepared excess sludge and chemically disintegrated by reagent dose of 0.8 ml/l of sludge for 16 h.

Table 5. Selected physico-chemical parameters of excess sludge during the 25-daily methane fermentation

Tabela 5. Wybrane parametry fizyczno-chemiczne osadów nadmiernych podczas 25-dobowej fermentacji metanowej

The sort of sludge Time of fermentation, d	Nonprepared excess sludge		Chemical prepared excess sludge	
	0	25	0	25
Indicator/unit				
Dry mass, g/l	13.43±0.16 ^S	9.51±0.08 ^S	12.19±0.18 ^S	7.71±0.12 ^S
Organic dry mass, g/l	9.06±0.06 ^S	5.80±0.14 ^S	9.89±0.17 ^S	3.80±0.15 ^S
Mineral dry mass, g/l	4.37±0.08 ^S	3.71±0.12 ^S	8.30±0.05 ^S	3.90±0.15 ^S
COD, mg O ₂ /l	133±5 ^S	321±3 ^S	505±6 ^S	570±4 ^S
VFAs, mg CH ₃ COOH/l	103±2 ^S	171±4 ^S	686±3 ^S	342±2 ^S
Kjeldahl nitrogen, mg N/l	92±4 ^S	445±5 ^S	87±3 ^S	454±7 ^S
Ammonia nitrogen, mg N-NH ₄ /l	58±2 ^S	422±4 ^S	110,6±3 ^S	590±6 ^S
pH	7.62	7.43	6.55	6.34

^S - standard deviation

After the anaerobic stabilization of untreated excess sludge obtained COD value and the concentration of VFAs was respectively 321 mg O₂/l and 171 mg CH₃COOH/l. In the case of chemically disintegrated sludge by dose of 0.8 ml/l of sludge COD value and the concentration of VFAs was respectively 570 mg O₂/l 342 and mg CH₃COOH/l. The degree of fermentation for unmodified and chemically modified sludge was 36 and 62%. The pH was 7.43 and 6.34, respectively. The total production of biogas from untreated excess sludge after 25 days of anaerobic stabilization process was 3.95 l, from chemically disintegrated sewage sludge by dose of 0.8 ml/l of sludge 23.32 l. Figure 4 and 5 show changes in the content in % by volume of methane in the generated biogas.

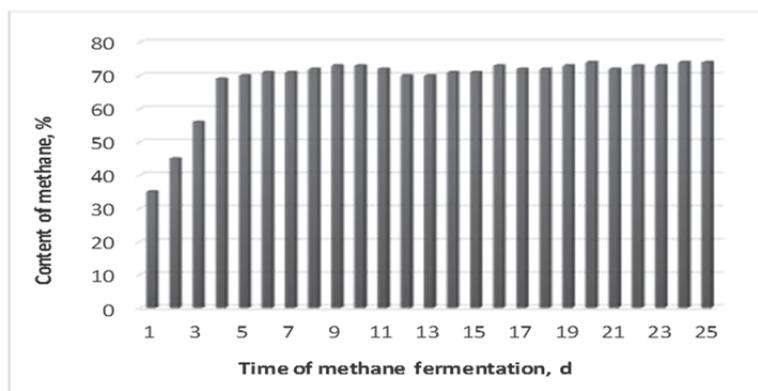


Fig. 4. Changes in the content of methane in the biogas during anaerobic stabilization of untreated excess sludge

Rys. 4. Zmiany zawartości metanu w biogazie podczas stabilizacji beztlenowej niepreparowanych osadów nadmiernych

During the initial days of the process in the analyzed biogas methane content was approx. 35%. This value increased until the sixth day, when it amounted to approx. 71% CH₄ content in the sample. After this period the volume content of methane in the biogas stabilized and amounted over approx. 70 percent.

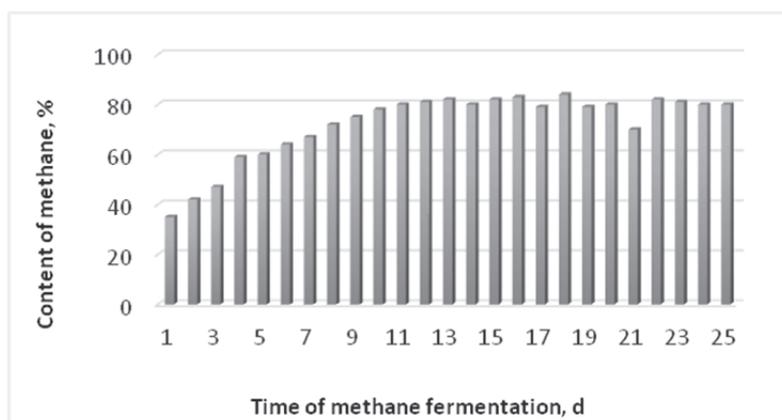


Fig. 5. Changes in the content of methane in the biogas during anaerobic stabilization of chemically disintegrated excess sludge

Rys. 5. Zmiany zawartości metanu w biogazie podczas stabilizacji beztlenowej chemicznie dezintegrowanych osadów nadmiernych

In the first day of the stabilization process methane content in biogas it was at approx. 35%. With each day, this value increased up to eleventh days in which the methane content was approx. 80% and remained at this level until the end of the process.

4. Conclusions

Based on the results of research on the effects of chemical disintegration of excess sludge to increase the concentration of organic substances in the form of dissolved following conclusions were formulated:

- Submission of sludge disintegration by chemical method resulted in an increase of the degree of disintegration of modified sludge and correlates with the increase in concentration of VFAs.
- The most favorable conditions of the chemical disintegration was reagent dose equal 0.8 ml/l and time 18 hours to give a value of COD and VFA concentration 1497 mg O₂/l and 1097 mg CH₃COOH/l and pH = 6.55.
- As a result of untreated excess sludge to undergo 25-daily anaerobic stabilization was achieved biogas yield of 0.12 l/g VSS., and disintegrated chemically 0.53 l/g VSS. The degree of mineralization was respectively 25 and 62%.
- The results found that the methane content in biogas produced from untreated sewage hovered at around 70%, while undergoing chemical disintegration of approximately 80%.

Acknowledgements

The research was funded by the project No. BS-PB-401/303/12.

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Charakterystyka osadów nadmiernych poddanych dezintegracji

Streszczenie

W wyniku procesu dezintegracji następuje rozdrobnienie cząstek stałych osadów oraz inaktywacji mikroorganizmów zawartych w osadach nadmiernych. Zniszczenie komórek mikroorganizmów prowadzi do uwalniania

enzymów i substancji wewnątrzkomórkowych do cieczy nadosadowej. Zwiększenie stężenia substancji organicznych w postaci rozpuszczonej wpływa na intensyfikację fermentacji metanowej. Celem przeprowadzonych badań była charakterystyka osadów nadmiarnych poddanych chemicznej dezintegracji. W trakcie badań zastosowano 10% wodny roztwór kwasu nadoctowego. Wyboru optymalnych warunków preparowania dokonano w oparciu o wybrane wskaźników takie jak: lotne kwasy tłuszczowe (LKT) i chemiczne zapotrzebowanie na tlen (ChZT). Wartość stopnia dezintegracji osadów była kryterium oceny skuteczności zastosowanych sposobów modyfikacji. W wyniku modyfikacji odnotowano wzrost wartości badanych wskaźników, który odzwierciedlał zachodzące zmiany struktury preparowanych osadów. Za najkorzystniejsze warunki chemicznej dezintegracji uznano dawkę reagenta 0,8 ml/dm³ i czas 18 godzin, otrzymując wartość ChZT wynoszącą 1497 mg O₂/dm³ i stężenie LKT 1097 mg CH₃COOH/dm³ przy pH = 6,55.

Abstract

Disintegration is the fragmentation of the solid phase of sludge and inactivation of sludge microorganisms. The destruction of microbial cells results in the release of substrates and enzymes to liquid sludge. The increased concentration of organic substances in the form of dissolved affects the intensification of methane fermentation process. The aim of the study was to characterize the excess sludge subjects chemical disintegration. During the research water solution of 10% peracetic acid was used. Selection of optimal conditions of preparation were made based on selected performance indicators such as volatile fatty acids (VFAs) and the chemical oxygen demand (COD). The value of the degree of sludge disintegration was a criterion for assessing the effectiveness of the modification methods. As a result, occurring in modified sludge lysis process achieved an increase in the value of the examined indicators, which was directly reflected in the change of image structure of the prepared sludge. The most favorable conditions for the chemical disintegration was reagent dose of 0.8 ml/l and time 18 hours to give a value of COD 1497 mg O₂/l and concentration of VFAs 1097 mg CH₃COOH/l and pH = 6.55.

Słowa kluczowe:

dezintegracja, kwas nadoctowy

Keywords:

disintegration, peracetic acid