

Ultrasonic Disintegration of Sewage Sludge to Increase Biogas Generation

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The value of methane fermentation in sewage sludge treatment is well established. It causes volume reduction, improves stability, destroys pathogens, and increases the amount of biogas obtained, which can be used as an energy source. Ultrasound treatment destroys cell membrane continuity, increasing the bioavailability of the soluble fraction of organic matter, which contributes to the intensification of hydrolysis. This paper presents the investigations concerning the influence of ultrasonic pretreatment of sewage sludge on the effectiveness of 25-day stabilization process. In the first series untreated sludge was used, and for the second and third series the following treatment parameters were applied: ultrasonic field amplitude 39.25 μm and sonification time 5 or 10 minutes. During the investigation unitary biogas production of 9.8 L g^{-1} VSS and 6.29 L g^{-1} VSS was obtained, for sonication time 10 and 5 minutes, respectively. Biogas generation from unconditioned and stabilized sludge was 4.43 L g^{-1} VSS.

Key words:

Excess sludge, ultrasonic field (UF), disintegration, anaerobic stabilization, biogas

Introduction

Renewable energy will play a key role in the future in limitation of greenhouse gas emissions, with particular focus on carbon dioxide. Biomass and waste are considered among the dominant renewable energy sources. Due to their availability on the energy resources market, it has potential to ensure permanent energy generation.^{1–3}

According to the International Energy Agency (IEA), fossil fuels accounted for 81 % of primary energy supply in 2007, whereas renewable energy sources were merely 13 %. Although particular interest is paid to technological and economic aspects of development and implementation of renewable energy sources, fossil fuels remain a dominant energy carrier all over the world, with its 77 % share in the world energy balance in 2007–2030.^{4,5}

The very fast increase in sludge production, mainly due to the implementation of the European Council Directive 91/271/EEC concerning urban wastewater treatment, requires new strategies in sludge management to cope with this critical issue in an economically and environmentally fully acceptable manner.⁶

Anaerobic stabilization, which is the biochemical process of organic matter conversion, is widely applied in wastewater treatment. The origins of the industrial use of fermentation date back to the first

half of the 20th century. Methane fermentation is a chemical conversion of organic waste into inorganic compounds that occurs in the microbial cells present in water environment, which means that biomass sources that contain high levels of water can be converted without using initial physical processing.^{7,8} The use of methane fermentation as a method for processing sludge, the waste product of wastewater treatment, is a highly beneficial solution. It limits the direct negative effect of sewage sludge on the environment and provides a good source of biogas.

Biogas, which contains 65 % CH_4 , 35 % CO_2 and trace amounts of H_2S , H_2 i N_2 , is an effective and environmentally friendly energy source. Its combustion is associated with low-level emissions of harmful pollutants.⁹

Disintegration of sewage sludge involves destruction of the sludge structure using external forces that affect their physical and chemical parameters and change the character of the structure. The use of an ultrasonic field on a medium would cause mechanical and sonochemical effects.^{10–13}

During the first phase of ultrasonic field conditioning, according to Tiehm et al.^{14,15} depending on the amount of supplied energy and the duration of sonification, floccular structures undergo disintegration. At this stage of conditioning, no destruction of microorganism cell walls was observed. The destruction of the cell walls of activated sludge micro-

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organisms occurs along with the transformation of insoluble organic substances to their soluble forms during the second phase of sonification.

Acute conditions (high temperature and pressure) in the area of cavitation explain the changes in physical and chemical properties in the sonicated systems.

When ultrasound pressure waves propagate through water, gas and vapor bubbles are generated and collapse violently and rapidly, which leads to high shear forces and micro-regions of extreme conditions with estimated temperatures as high as 5000 K and pressure up to 1×10^8 Pa.¹⁶ According to research conducted by Christi et al.¹⁷ high-power ultrasound induces cavitation, generation of free radicals, and other mechanical and chemical effects.

Ultrasonic conditioning of excess sludge results in the disintegration of its structure, which, apart from the dispersion of the sludge solid phase, also damages the cell membranes of the microorganisms and releases the substrate contained in these cells. This process facilitates the further biochemical decomposition of organic matter. The degree of sludge structure destruction depends on the method used, energy supplied, and the sludge's physical and chemical properties.^{18–21}

The effect of ultrasound frequency, specific energy input, and theoretical resonant cavitation bubble size on the degree of sludge disintegration has already been studied. In the studies of Pilli et al., the degree of disintegration decreased gradually with increase in frequency ($DD_{\text{COD}} = 80\%$ at 41 kHz and $DD_{\text{COD}} = 7\%$ at 3217 kHz).²²

Moreover, the input power of the disrupter P (J s^{-1}) and volume of the treated sludge VS (l) are also important factors, which was shown by Onyeché et al.²³

SCOD increases with increase in specific energy input. The rate of solubilisation is higher between specific energy of 0–8000 kJ kg^{-1} TS, beyond which the increase in specific energy reduces the rate of total solid solubilisation.²⁴

The mean particle size reduction also increases with increase in sonication densities. At densities of 0.52 W mL^{-1} , the mean particle is reduced from 51 μm to 15 μm , and from 51 μm to 19 μm at 0.33 W

mL^{-1} , respectively. Low power level has no effect on floc size reduction by sonication. With increase in power level, the floc size reduction increases with increase in ultrasonication density and sonication time.²²

Disintegration causes an increase in the amount of organic matter in soluble form, which afterwards could be converted into biogas. Destruction of microorganisms in activated sludge, which is resistant to anaerobic conditions, accelerates and intensifies sludge fermentation in fermentation chambers. After the process of anaerobic fermentation, sewage sludge contains less organic matter.^{25–27}

The aim of this study was to determine the effect of the initial processing of excess sludge exposed to an ultrasound field on the process of anaerobic stabilization and the efficiency of biogas production.

Material and methodology

The basic substrate in this study was excess sludge (90 %) and fermented sludge (10 %) which represented the *inoculum*. The sludge was sampled from a wastewater treatment plant. The general characteristics of raw excess sludge are presented in Table 1.

The criterion for the physical, chemical, and biological changes occurring in the sludge after the use of physical conditioning methods at the first stage of the study was changes in SCOD. Then, based on the above parameter, the most favourable parameters of excess sludge conditioning with ultrasound field (frequency and amplitude) were determined.

In order to determine the degree of disintegration, according to Formula 1,²⁸ the sludge was conditioned by means of 1-mol solution of NaOH for 10 minutes at temperature of 90 °C, with unchanged volumetric proportion of the sludge and the solution (1:1). According to the methodology, for the alkaline conditioned excess sludge, the authors obtained chemical oxygen demand of $2.35 \text{ kg O}_2 \text{ L}^{-1}$.

Table 1 – General characteristics of raw excess sludge

Parameter sludge sort	Hydration	Dry mass (TS)	Organic dry mass (VSS)	Mineral dry mass	Soluble chemical oxygen demand (SCOD)
	%	g L^{-1}	g L^{-1}	g L^{-1}	$\text{kg O}_2 \text{ L}^{-1}$
Excess sludge	98.75	7.71–15.52	5.51–10.53	2.2–4.69	0.070–0.120
Digested sludge	97.81	16.12–20.28	9.64–12.37	6.48–7.91	1.070–1.100

The degree of disintegration was obtained from the following formula:²⁰

$$DD_{\text{COD}} = (\text{SCOD}_1 - \text{SCOD}_2) / (\text{SCOD}_3 - \text{SCOD}_2) \cdot 100 \quad (1)$$

where:

DD_{COD} – disintegration degree, %;

SCOD_1 – SCOD level in the sludge conditioned with ultrasounds, $\text{kgO}_2 \text{ L}^{-1}$;

SCOD_2 – SCOD level in the raw sludge, $\text{kgO}_2 \text{ L}^{-1}$;

SCOD_3 – SCOD level in the sludge conditioned chemically 1-mol NaOH with ratio 1:1, temp. 90 °C for 10 minutes, $\text{kgO}_2 \text{ L}^{-1}$.

Biogas yield was obtained from formula (2) according to:²⁰

$$\text{b.y.} = V / \Delta \text{VSS} \quad (2)$$

b.y – biogas yield, $\text{L g}^{-1} \text{ VSS}$

V – total biogas volume released throughout the fermentation process, L

ΔVSS – decrement of dry organic mass in the sludge, g VSS

The choice of the ultrasonic field conditioning parameters was controlled by a VCX – 1500 ultrasound disintegrator with automated tuning (SONICS, USA). The maximum power output of this device is 1500 W, whereas the frequency of vibration of the ultrasound field is 20 kHz. The sonotrode was immersed into the 10-centimeter graduated cylinder filled with the excess sludge studied to 5-cm level. The volume of the conditioned sample was 0.5 L.

The main principle of the above equipment consists of the active effect of ultrasonic field on the sludge. The power converter of the disintegrator converts the supplied electrical energy into mechanical vibrations. The mechanical vibrations are transferred in the form of linear waves to the working tip made of titanium. The sludge prepared according to the above procedure for anaerobic stabilization process, was cooled down to 37 °C, and then inoculated with fermented sludge to the volume of 10 % of total volume. The obtained mixtures of sludge were then mixed and placed into the fermentation chamber with active volume of 10 L, using the process of 25-day stabilization. The system was equipped in an installation that maintained constant process temperature and equipment that ensured optimum mixing rate, which was constant and amounted to 170 rpm for all the processes, and an installation for biogas sampling. A water jacket was installed outside in order to heat the sludge in the chamber. The temperature of 37 °C was ensured by a platinum electrode placed inside the chamber. Biogas was captured by a system, which included a 5 L cylinder, filled with saturated solution of sodium chlo-

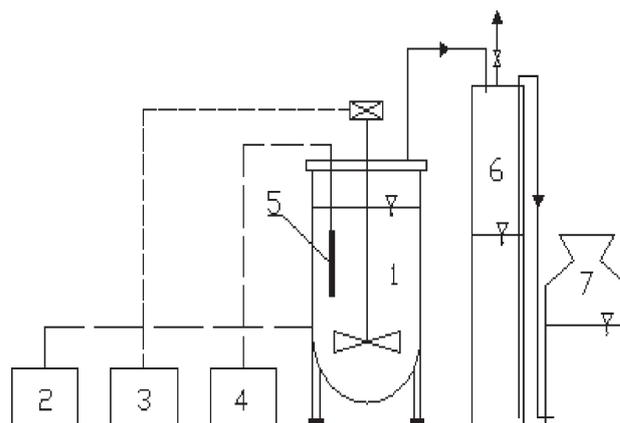


Fig. 1 – Fermentation chamber Applicon (USA)
1. fermentation chamber, 2. heat exchanger, 3. mixing system, 4. temperature control system and pH changes, 5. probe, 6. biogas tank, 7. expansion tank

ride and a compensatory 5 L gas cylinder. The process of fermentation was controlled everyday based on the amount of biogas produced (Fig. 1).

Both before (day 0) and after the process of fermentation (day 25), the dry mass, dry organic mass, and dry mineral mass by the direct gravimetric method according to the PN-EN-12879 standard were determined. The level of chemical oxygen demand was determined by means of dichromate method using HACH 2100N IS. In order to separate the sludge liquor of the conditioned and raw excess sludge, the authors used a centrifuge (Centrifuge 5810).

The Kruskal-Wallis statistics were used to compare the changes of COD of the supernatant, as well as daily and total biogas production during anaerobic stabilization of sludge depending on disintegration conditions described as Mixture I, II, and III. The difference was significant for the p value < 0.05 .

The research results and discussion

Initial sludge treatment before the process of methane fermentation was aimed at disintegration of sludge particles, destruction of microorganisms in excess sludge and release of the organic compounds and enzymes. Based on the results obtained during pilot investigations, with regard to the changes in SCOD levels in sludge liquor disintegrated with ultrasound field (Table 2), the most favourable amplitude and conditioning time was determined. The highest level of SCOD for the amplitude of 39.25 μm , was obtained for the time of 600 s. For the analysed amplitude and the above sonication time, a SCOD level of 3.912 $\text{kg O}_2 \text{ L}^{-1}$ was observed, whereas in the case of non-conditioned sludge, this value was 0.185 $\text{kg O}_2 \text{ L}^{-1}$.

Table 2 – Changes in SCOD values marked for excess sludge conditioned by ultrasonic field

Exposition time	Amplitude of vibration	39.25 μm
		SCOD, kg O ₂ /L
Sample 0		0.185
60s		1.098
120s		1.210
180s		1.580
240s		1.762
300s		2.034
360s		2.285
420s		2.612
480s		2.885
540s		3.275
600s		3.912

The analysis revealed the most favourable time of sludge preparation (10 minutes) and vibration amplitude of 100 % (39.25 μm). In order to compare the results obtained for the same amplitude of vibrations, the time of 5 minutes was adopted for another cycle of experiments. For the above sonication times and the adopted amplitude of vibrations, a process of anaerobic stabilization was carried out. The ultrasonic density for the vibration amplitude of 39.25 μm and times of disintegration of 5 and 10 minutes equaled 0.43 W mL⁻¹. Pilli et al.²² evaluated the effect of ultrasonic density on floc disintegration and found that, for example, the floc size is reduced from 94 μm to less than 3 μm with a sonication density of 0.22 W mL⁻¹ and 0.44 W mL⁻¹, respectively.

Table 3 – The value of the disintegration degree DD for a given ultrasonic field amplitude and the testing time of sonification

Amplitude of vibration	Exposition time, min	Disintegration degree DD _{COD} , %
Sample 0	0	–
39.25 μm	1	26.68
	2	29.61
	3	39.31
	4	44.08
	5	51.21
	6	57.79
	7	66.36
	8	73.52
	9	83.74
	10	100.44

The effectiveness of the ultrasonic field conditioning process was determined based on equation (1) i.e. the increase in the degree of disintegration (Table 3). The highest value of the fraction of disintegration of excess sludge (100.44 %) was found for the time of 10 minutes and the amplitude of 39.25 μm . In the case of sonication time of 5 minutes, the fraction of disintegration was 51.21 %.

At the next stage of the investigations, after determining the degree of disintegration, the process of 25-day anaerobic stabilization of excess sludge was carried out for the vibration amplitude of 39.25 μm and sonication time of excess sludge of 5 and 10 minutes.

Changes in sludge parameters after physical conditioning are presented in Table 4.

Table 4 – Set of results of physically conditioned sludge undergoing 25-days anaerobic stabilization process

Time of methane fermentation, d	Parameter	Unit	Mixture I (raw sludge)	Mixture II (ultrasonic field: A = 39.25 μm , t = 5 min.)	Mixture III (ultrasonic field: A = 39.25 μm , t = 10 min.)
0	Dry mass	g L ⁻¹	9.87	15.52	13.43
	Organic dry mass	g L ⁻¹	6.46	10.54	9.45
	SCOD	kgO ₂ L ⁻¹	0.133	2.600	4.250
10	Dry mass	g L ⁻¹	11.33	11.01	7.23
	Organic dry mass	g L ⁻¹	6.99	6.42	4.29
	SCOD	kgO ₂ L ⁻¹	0.459	1.094	0.722
25	Dry mass	g L ⁻¹	7.16	10.7	8.51
	Organic dry mass	g L ⁻¹	4.18	6.05	4.8
	SCOD	kgO ₂ L ⁻¹	0.321	0.602	0.432

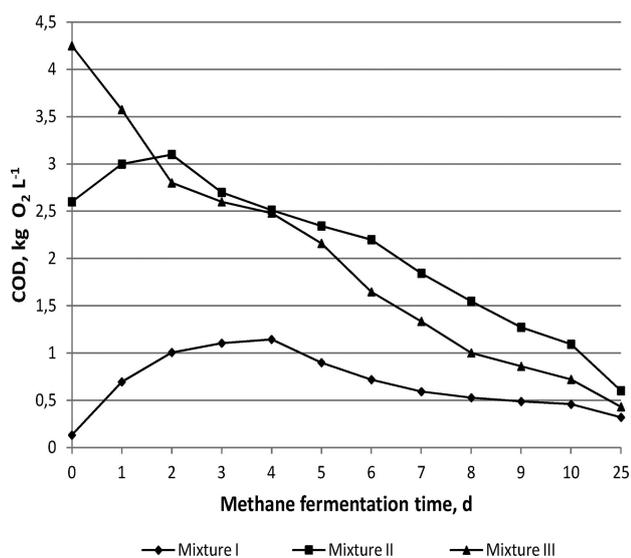


Fig. 2 – Changes of SCOD levels during the process of anaerobic stabilization

The highest value of SCOD ($4.25 \text{ kg O}_2 \text{ L}^{-1}$) was observed before the process of anaerobic stabilization after conditioning with ultrasonic field (amplitude of 100 % and sonication time of 10 minutes - Mixture III). The highest level of SCOD was nearly 32 times higher compared to the non-conditioned excess sludge (Mixture I, $0.133 \text{ kg O}_2 \text{ L}^{-1}$). On the 10th day of methane fermentation, the value of SCOD for the sludge conditioned with ultrasonic field for 5 and 10 minutes was, respectively $1.094 \text{ kg O}_2 \text{ L}^{-1}$ and $0.722 \text{ kg O}_2 \text{ L}^{-1}$. Fig. 2 presents SCOD levels recorded on each day of the process of anaerobic stabilization.

After the process of anaerobic stabilization in the fermentation chamber (25 days), the SCOD level decreased to $0.602 \text{ kg O}_2 \text{ L}^{-1}$ and $0.432 \text{ kg O}_2 \text{ L}^{-1}$, respectively, for the sludge exposed to active ultra-

sound field for 5 and 10 minutes and the amplitude 100 %.

In a study by Cesaro et al., after 25 days of digestion, SCOD removal was higher than 60 % in the partially sonicated mixture, while it was lower than 50 % in the untreated mixture.²⁹ Moreover Braguglia et al.³⁰ found 74 % SCOD removal in digester filled with sonicated sludge.

Critical importance in terms of the economics of the anaerobic stabilization process is also emphasized by the amount of biogas that can be converted into electricity or used for heating fermentation chambers. This causes a considerable reduction in the costs of maintenance and operation of wastewater treatment plants. The highest total biogas production after the anaerobic stabilization process in the fermentation chamber (25 days), which reached 28.69 L, was recorded for the excess sludge conditioned with ultrasound field for 10 minutes and the amplitude of vibrations $39.25 \mu\text{m}$ (Mixture III). In the case of the sludge conditioned with ultrasound field for 5 minutes (Mixture II), the total value of biogas produced was 22.35 L, whereas this value for non-prepared sludge was 10.92 L. Daily biogas production is compared in Fig. 3.

The highest daily biogas production was observed on the 9th day of the process for the sludge conditioned for 5 and 10 minutes. The maximum value of daily biogas production for these times amounted to, respectively, 3 L d^{-1} and 2.92 L d^{-1} . In the case of non-conditioned sludge, the highest daily biogas production value was 1.14 L d^{-1} . According to Braguglia et al.,³⁰ in the case of anaerobic stabilization process of ultrasound pretreated sludge, 26 % more biogas was obtained than with untreated sludge.

Total biogas production is presented in Fig. 4.

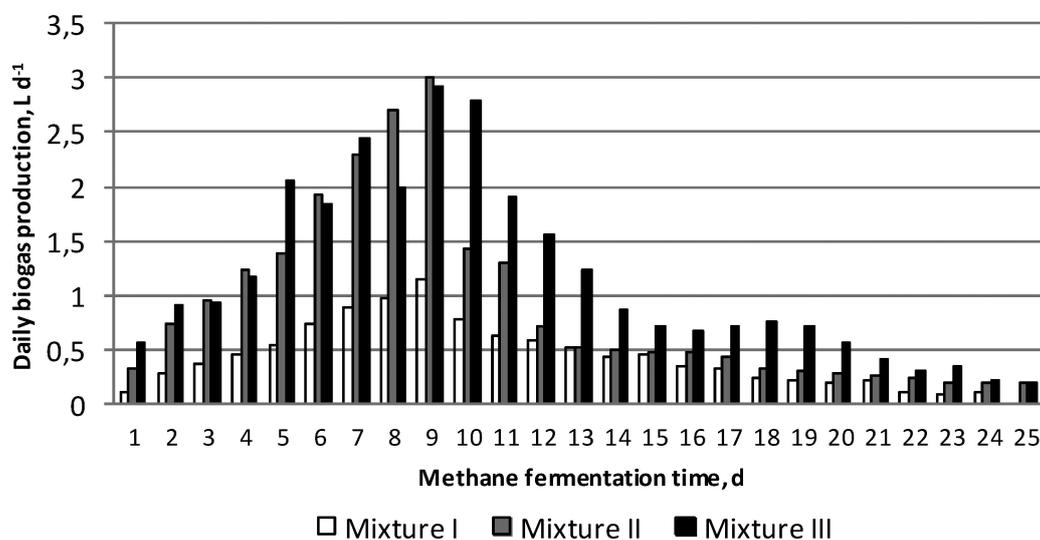


Fig. 3 – Daily biogas production during anaerobic stabilization process

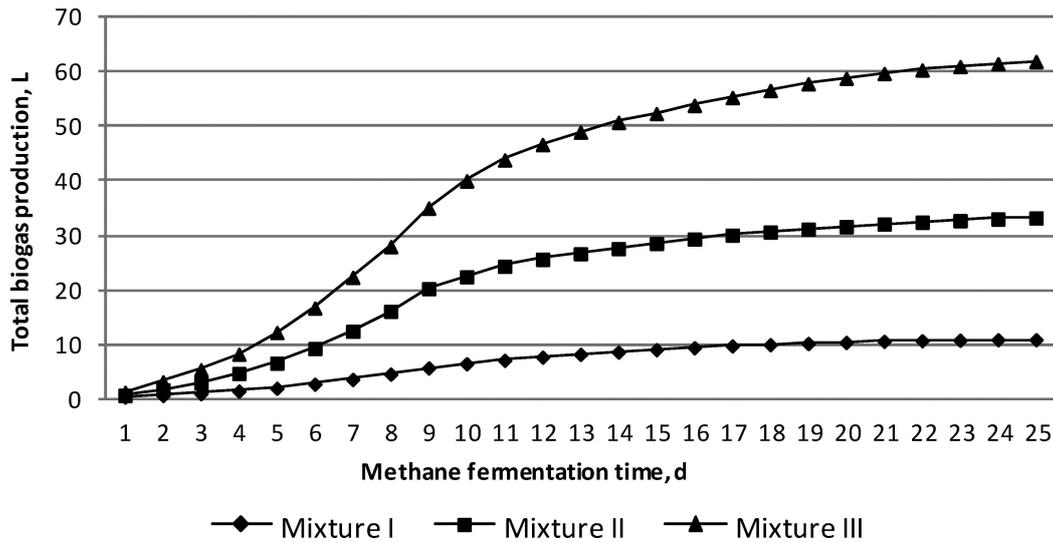


Fig. 4 – Total biogas production during anaerobic stabilization process

The highest biogas yield (b.y.), calculated according to formula (2), which was $9.63 \text{ L g}^{-1} \text{ VSS}$, was obtained for excess sludge conditioned with the amplitude of $39.25 \mu\text{m}$ for 10 minutes. High value of biogas yield ($6.29 \text{ L g}^{-1} \text{ VSS}$) was also observed for sonication time of 5 minutes. The biogas yield value for excess sludge was $5.15 \text{ L g}^{-1} \text{ VSS}$. The biogas yield value for non-prepared excess sludge was $3.20 \text{ L g}^{-1} \text{ VSS}$.

There was a significant difference in SCOD of the obtained supernatant between Mixture I and Mixture II ($p < 0.001$) and III ($p = 0.003$). The SCOD of Mixture II and III did not differ significantly ($p = 1$), which shows that the parameters of disintegration do not influence the quality of obtained supernatant. The efficiency of fermentation measured by daily biogas production was significantly higher only for Mixture III compared to Mixture I ($p < 0.001$). When the total biogas production is considered, the difference compared to Mixture I was significant for both Mixtures II ($p = 0.01$) and III ($p < 0.001$).

The analysis shows that ultrasonic disintegration conditions applied both for Mixture II and III lead to higher efficiency of methane fermentation and better supernatant quality in comparison to unconditioned sludge (Mixture I).

Conclusions

Conditioning of excess sludge with ultrasonic field before the process of anaerobic stabilization leads to an increase in the disintegration degree, which is expressed by the increase in concentration of organic matter in water expressed in SCOD levels.

In the case of exposure of the excess sludge to 600 s of sonication before the stabilization process, the maximum SCOD level was obtained and, in the following days of the process, a rapid decline in this parameter was observed. Furthermore, for the sludge conditioned with ultrasound field of 300 s, a decrease in SCOD level was recorded from the second day of the process. For excess sludge without physical processing, the decline in the parameter was observed on the fifth day of the process. The increase in sludge sample temperature was observed as the result of cavitation phenomenon. In the entire volume of disintegrated sludge sample, the diffusion and heat convection effect was noticed. As the result of sonochemical reactions, the decomposition of hydrolyzed substances to volatile fatty acids was obtained. The increase of acoustic power influenced the intensity of disintegration and accompanying temperature growth. Increase in temperature of disintegrated raw excess sludge aided the sonolysis process of the sludge organic substances. The effectiveness of conditioning process depends on the ultrasonic vibrations and the accompanying temperature increase.

The analysis of the results obtained in the present study leads to the following conclusions:

- The experiments carried out using the high-power ultrasonic field, where high SCOD values, substantial level of excess sludge disintegration and improved biogas production were recorded, provide evidence of the effectiveness of this method in obtaining the best possible effects of anaerobic sludge stabilization.

- For both times of conditioning, 300 and 600 s, a considerable degree of disintegration of excess sludge 51.21 % and 100.44 %, respectively, was noticed.

– The use of conditioning before the process of anaerobic stabilization facilitates the operation of separated fermentation chambers, which results in intensified biogas production during the process of methane fermentation and greater organic dry mass reduction. In the case of ultrasonic disintegrated excess sludge, biogas yield of 5.15 L g⁻¹ VSS and 9.63 L g⁻¹ VSS was obtained, while digested degree was 43 % and 64 %. Moreover, in the case of raw excess sludge, biogas yield and digested degree of 3.20 L g⁻¹ VSS and 35 % respectively was noticed.

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List of symbols

- UF – Ultrasonic Field
 DD_{COD} – disintegration degree, %
 SCOD – soluble chemical oxygen demand, kg O₂ L⁻¹
 VSS – volatile suspended solid, kg L⁻¹
 b.y. – biogas yield, L g⁻¹ VSS
 V – total of biogas volume released throughout the process of fermentation, L
 ΔVSS – decrement of dry organic mass in the sludge, g VSS

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