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Impact of Ultrasonic Pretreatment on the Anaerobic Fermentation of Dairy Waste Activated Sludge

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1. Introduction

Anaerobic digestion is the most commonly applied process for the stabilization of sewage sludge. Anaerobic digestion is generally considered to be an economic and environmentally friendly technology for the treatment of different organic waste. Indeed, the biological degradation process of organic matter, under anoxic conditions, led to the production of methane, which can be used as a renewable energy (Appels et al. 2011, Cesaro et al. 2012, Braguglia et al. 2012, Abe et al. 2011, Sadecka & Suchowska-Kisielewicz 2016). However, organic matters hydrolysis presents the rate-limiting step in sludge anaerobic digestion process (Hu et al. 2011, Galipoli & Braguglia 2012). Therefore, physical, chemical, biological process or their combination can be used to improve anaerobic treatment efficiency. Additionally, different types of pre-treatment methods can improve the degradation and thus result in higher methane production and a more stabilized end-product (Luste et al. 2012, Erden & Filibeli 2010, Xu et al. 2011, Braguglia et al. 2011, Carrere et al. 2010). Ultrasound processes are a potential option for promoting organic matter solubilisation, thus improving anaerobic digestion yields (Cesaro et al. 2012, Zawieja et al. 2015).

Ultrasound can be classified according to the frequency level into: (1) high frequency and low power (2-10 MHz range) ultrasound, also termed extended range or diagnostic ultrasound, which is used in medical imaging and chemical analyses; and (2) low frequency and high power (20-100 kHz) ultrasound, the conventional type of ultrasound, which is used for cleaning and welding and also for sonochemistry (Rokhina et al. 2009, Braguglia et al. 2012a). The ultrasonic power is the key parameter in sludge disintegration. The sludge type is very important to decide about the operational parameters (Kidak et al. 2009).

Ultrasound generated high acoustic energy, and when this energy is applied to a liquid system, it is possible to generate physical and chemical reactions that can significantly modify the character of dissolved and particulate substances present in the liquid. These reactions result from the generation and collapse of cavitation bubbles produced under this acoustic condition. While ultrasound shows great potential in environmental engineering, a number of scientific and technical questions exist, which include the influence of frequency, dissolved gases, and suspended solids on cavitation; optimal reactor design; economy, reliability, and life expectation of ultrasound equipment (Show et al. 2010). The ultrasound technique is well known for disrupting sludge floc and lysed biological cells, which can lead to solubilisation of organic matter, reduction of particle size and inactivation of sludge microorganisms (Huan et al. 2009, Wang et al. 2006). The mechanism of sludge destruction by ultrasound is divided into three stages. The first stage is the flocs loosing, where particle size decreases and extracellular materials are escaped from the surface of the flocs. The second stage is the cell breakage, where the intracellular organic materials inside the cells are set free, but most of them were macromolecular compounds. In the last stage, the macromolecular compounds were degraded into short chain organic micromolecular compounds (Chang et al. 2001, Khanal et al. 2007, Feng et al. 2009).

This research examines the effectiveness of ultrasound pretreatment on waste activated sludge (excess sludge)(WAS) disintegration at different specific energies and sonication time durations in the anaerobic digestibility of control, full stream sonicated WAS. Determine the effect the ultrasonic field on the efficiency anaerobic digestion of sewage sludge from dairy industry, that is increase in biogas production and the degree reduction of digested.

2. Methods

2.1. Materials

Sewage sludge was obtained from mechanical-biological treatment plant located in the dairy cooperative "Włoszczowa". Substrate for the study was collected from recycled sludge inflow stub pipe to the circulation chamber sludge. The substrate was thickened by gravitational method to reach VS (volatile solid) concentration of approximately 16 g L^{-1}. The characteristic of the substrates and *inoculum* are shown in Table 1.

Table 1. Characteristics of raw substrates and *inoculum* mixtures used in the study
Tabela 1. Charakterystyka substratów badań: *inoculum*, mieszanin użytych w badaniach

Parameter	Unit	Substrates		
		Sewage sludge	Thickened sewage sludge	*Inoculum*
TS	[g L^{-1}]	13.8	23.8	18.6
VS	[g L^{-1}]	8.5	16.5	11.7

2.2. Sonication (ultrasonic disintegration)

Sonication was carried out with a low-frequency (20 kHz) ultrasonic processor VC750 (Sonics, USA), with a 19 mm titanium tip. Sludge samples with a volume of one liter was sonicated at ambient temperature. The quantity of the amplitude is set as a percentage of maximum amplitude and kept constant by generator. Four different amplitudes: A_1 = 24.4 µm (40%), A_1 = 36.6 µm (60%), A_3 = 48.8 µm (80%) and A_4 = 61.0 µm (100%), were tested in the study. Sludge sonication times were 18, 360, 540, 720, 900, 1080, 1200 s. The amount of acoustic energy that was being delivered to the probe was showed in Table 2. The acoustic energy (E) delivered to the sample was adjusted by varying the input amplitude (A) and sonication time (t_S). The acoustic energy was calculated by the integrated circuit with the Sonic VC750 device. The producer did not provide data on the efficiency of the transformation of electricity to acoustic energy. An external electrical power consumption counter was not used.

Table 2. Characteristics of sonication parameters
Tabela 2. Charakterystyka parametrów nadźwiękawiania

Time	Amplitude	Acoustic energy	Amplitude	Acoustic energy
S	µm	kJ	µm	kJ
180 (3 min)	24.4	7.0	36.6	10.3
360 (6 min)		13.1		16.8
540 (9 min)		19.2		28.9
720 (12 min)		25.4		41.1
900 (15 min)		31.5		53.2
1080 (18 min)		37.6		65.4
1260 (21 min)		41.7		73.4
180 (3 min)	48.8	13.8	61.0	20.1
360(6 min)		31.8		46.7
540 (9 min)		52.1		73.3
720 (12 min)		72.4		99.9
900 (15 min)		92.8		126.6
1080 (18 min)		113.1		153.2
1260 (21 min)		126.7		170.9

2.3. Experimental setup

The study was performed in three stages (Figure 1). During the first stage, the effect of ultrasonic disintegration was characterized with ratio of total organic carbon concentrations (TOC) and acoustic energy as well as ratio of total carbon concentrations (TC) and acoustic energy (Table 2). The second stage was conducted in order to evaluate the effect of selected pre-treatment US parameters of sewage sludge on potential biogas production. Experiment was determined with the use of Oxi-Top Control AN6 measuring system. This system consists of glass vessels, measuring pressure heads, a controller, and shaking plateau. Pressure heads measure pressure variations in digestion vessels. The tests were performed in 12 vessels with continuous stirring at 37°C. Batch tests were carried out for 21 days. Based on the results of the first and second stage of the studies the best ultrasonic field parameters were determined, which were used in third stage of research. In this part of study, digestion was carried out in 5.5 L glass reactor at 37°C (Fig. 1). Reactor was operated in draw-and fill mode (on a daily basis) with retention time of 10 days. The adaptation of the digester biomass in the reactor was achieved after 30 days. Initially reactor experiment was performed with sewage sludge without pretreatment (control). Subsequently, reactor was fed with sewage sludge pretreated at 36.6, 48.8. and 61 µm for 21, 18 and 15 respectively.

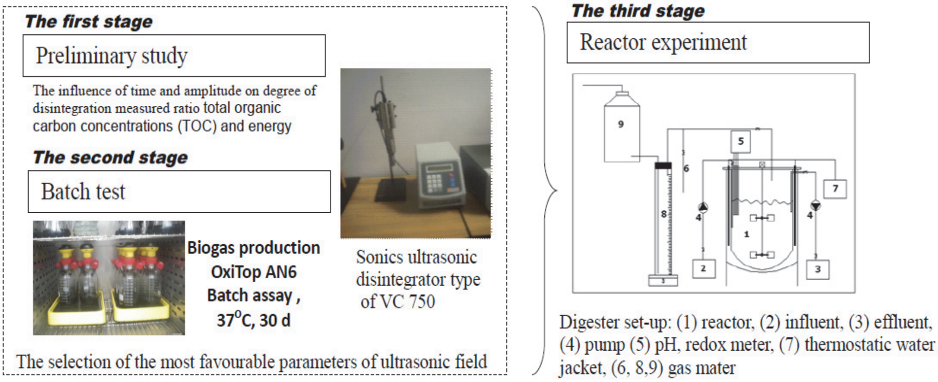

Fig. 1. Experimental setup
Rys. 1. Schemat doświadczenia

2.4. Analyses and calculations

Total carbon (TC) and total organic carbon (TOC) were measured by multi N/C analyzer (Analytik Jena UK). All measurements were done on soluble fractions of sludge. The soluble fraction was defined as the fraction resulting from the centrifugation and filtration. The samples were centrifuged at 10.000 rcf (relative centrifugal force) for 15 min. The supernatant was filtrated through a cellulose nitrate membrane (0.45 µm pore size). Total solids (TS), volatile solids (VS), pH (pH meter Cole Parmer Model No. 59002-00), alkalinity, chemical oxygen demand (COD), ammonium nitrogen and volatile fatty acids (VFAs) (steam distillation – BÜCHI K-355) were determined according to standard methods (APHA 1999). Biogas production was measured daily by the water displacement method and biogas composition (methane, carbon dioxide, ballast and oxygen) was analyzed by a portable gas analyzer (GA 2000, Geotechnical Instruments (UK) Ltd.). All results was calculated at standard temperature and pressure.

3. Results

In the first stage, the selection of the most favorable sonication parameters of sewage sludge was selected. For this purpose, the concentration of total carbon (TC) and applied acoustic energy (E) ratio (TC/E)was used (Fig. 2). Moreover the TOC/E ratio was calculated (Fig. 3). It was found that, the highest value of TC/E and TOC/E ratios was observed for the highest tested amplitude (61.0 μm), and the lowest for the amplitude of 24.4 μm. This is evident by the fact that the maximum acoustic energy introduced into the sludge as a result of propagation of the ultrasonic wave at amplitude 24.4 μm was 41.7 kJ. However, in case of amplitude 61.0 μm application it was 170.9 kJ. At this stage the criterion for selection of the most favorable conditions for sonication was the maximum value of the TC/E and TOC/E ratios. Based on these results, it was decided that further research will be conducted using three amplitudes, i.e. 36.6 μm, 48.8 μm, 61.0 μm, respectively, and exposure times: 15, 18 and 21 minutes.

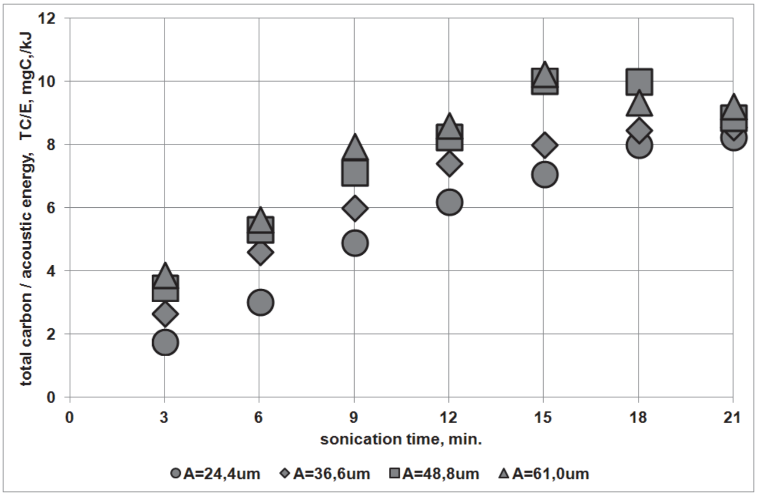

Fig. 2. Total carbon concentrations (TC) and acoustic energy (E) ratio as a function of sonication amplitude and time
Rys. 2. Całkowity stosunek stężenia węgla (TC) i energii akustycznej (E) w funkcji amplitudy nadźwiękawiania i czasu

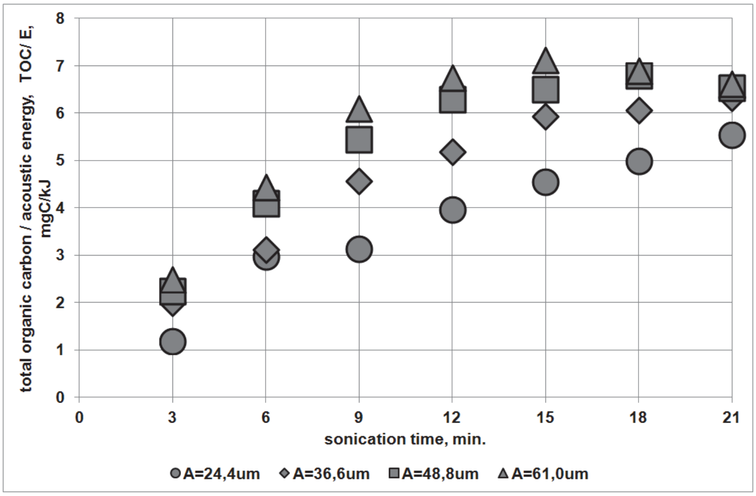

Fig. 3. Total organic carbon concentrations (TC) and acoustic energy (E) ratio as a function of sonication amplitude and time

Rys. 3. Całkowity stosunek węgla organicznego (TC) i energii akustycznej (E) w funkcji amplitudy nadźwiękawiania i czasu

3.1. Bach test

Figure 4 shows the change of biogas generation during anaerobic digestion carried out with OxiTop AN6. It was found, that only below listed sonication parameters allowed to increase the efficiency of biogas production in comparison to the control (non sonicated sludge). These were propagation of ultrasonic wave with amplitude A = 36.6 μm at 21 minutes and also combinations 48.8 μm/18 minutes and 61.0 μm/15 minutes. The composition of biogas for each combination was presented in Figure 5. Compared to the control only sludge sonication with amplitude of 61 μm (regardless of the sonication time) allowed minimal increase in methane content in biogas. Specified combinations of sonication parameters were decided to test in the continuous fermentation process (third stage).

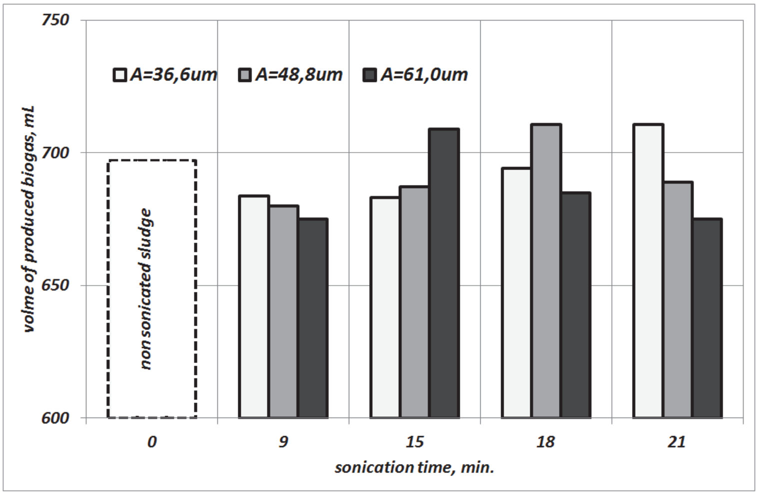

Fig. 4. The volume of biogas generated during the fermentation in set OxiTop AN6

Rys. 4. Objętość biogazu wytworzonego podczas fermentacji w zestawie OxiTop AN6

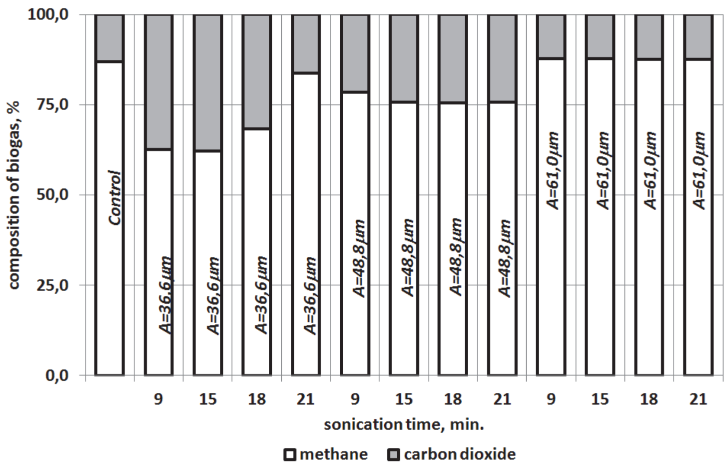

Fig. 5. The composition of biogas generated during the fermentation in set OxiTop AN6

Rys. 5. Skład biogazu wytworzonego podczas fermentacji w zestawie OxiTop AN6

3.2. Reactor experiment

The dynamic changes of the volatile fatty acids concentration during anaerobic digestion are shown in the Table 3. In the initial period (sludge not conditioned by US) the VFA concentrations ranged from 994 to 1.222 mg L^{-1}. From the day 50 the reactor was feed with US conditioned sludge (36.6 μm/21 minut). At the beginning of the experiment the increase of VFA (1428 mg L^{-1}) was observed, then the concentration of volatile fatty acids decreased to the level of 1120 mg L^{-1}. For the amplitude of 48.8 μm/18 min, the increase of organic loading rate cased the increase of VFA concentration in the reactor. For the last applied US field amplitude the concentration of VFA decreased to the level of 1,394 mg L^{-1}.

During the first period (without US conditioning) the total alkalinity changed from 3600 to 3900 mg L^{-1} . However, in case of the use of sonication to pretreat sewage sludge a steady growth of that index values was observed. The highest alkalinity (910 mg L^{-1}) was observed for amplitude of 48.8 μm and 18 minutes of exposure time. During the process, the VFA/ alkalinity ratio ranged from 0.25 to 0.36. However, it did not exceed the value recognized in the literature as alarming.

The highest ratio value was recorded in the 15 and 60 day trial. The highest ratio was recorded on the fifth day, the lowest in 10 days after the introduction of a new charge to the reactor. The pH of 7.33 to 7.61 was observed within the experience.

For the control the ammonium nitrogen concentration varied from 489 to 694 mg L^{-1}. After changing the conditions of the sewage sludge conditioning the $N\text{-}NH_4$ concentration increase of 62 mg L^{-1} (36.6 μm/21 min), 247 mg L^{-1} (48.8 μm/18 min) and 78 mg L^{-1} (36.6 μm/21min). After application of ultrasonic disintegration the chemical oxidation demand (COD) increased from 698 to 1430 mg L^{-1}. In the last day of the process (61 μm/15 min) the concentration of COD stood at 1458 mg L^{-1}.

Figure 5 shows the daily biogas production during the digestion process. In the first stage of the study (fermentation without US) a steady increase in biogas volume produced per day was observed. During this period the highest biogas production of 2.84 L was observed in the day 41 of experiment.

Table 3. Characteristics of the digested material during the fermentation
Tabela 3. Charakterystyka osadów przefermentowanych podczas procesu fermentacji

Pretreatment Amplitude	Day	VFA (mg L^{-1})	VFA/ Alkalinity	pH	$N\text{-}NH_4$ (mg L^{-1})	COD (mg L^{-1})	VS (g L^{-1})
–	1	977±247	0.25	7.61	694±6	385±55	8.96±0.20
	5	994±34	0.27	7.54	586±3	629±11	9.87±0.24
	10	1097±34	0.31	7.42	577±10	642±38	9.95±0.11
	15	1257±39	0.36	7.43	553±9	611±39	9.63±0.28
	20	1080±17	0.32	7.42	489±14	724±30	9.13±0.12
	25	1206±36	0.35	7.36	530±9	540±15	9.61±0.05
	30	977±45	0.27	7.4	547±3	840±40	10.37±0.10
	35	1223±71	0.33	7.37	579±3	760±1	11.86±0.23
	40	1177±50	0.30	7.38	657±12	964±19	12.92±0.09
	45	1017±49	0.26	7.33	669±3	698±37	12.95±0.09
36.6 µm	50	1429±36	0.35	7.48	735±9	923±29	11.50±0.08
	55	1120±20	0.27	7.54	732±3	1431±21	21.94±0.31
48.8 µm	60	1714±34	0.36	7.54	930±6	1278±82	11.64±0.12
	65	1703±20	0.34	7.61	978±3	1313±76	12.39±0.13
61 µm	70	1577±34	0.30	7.67	1036±6	1381±79	11.61±0.03
	75	1394±52	0.26	7.61	1057±6	1459±126	10.88±0.31

After the application the ultrasound for sewage sludge pretreatment with amplitude of 48.8 μm and sonication time of 18 min an increase in the daily production of biogas compared to controls by 52% was observed. For all other amplitudes large fluctuations in the value of the measured parameter was observed. In their case, compared to controls it has been an increase in volume of gas produced by the fermentation of 16% (A = 36.6 μm) and 45% (A = 61.0 μm). The use of ultrasound has not significantly changed the composition of the biogas produced in the process. During the fermentation process, the methane content in the biogas about 69%.

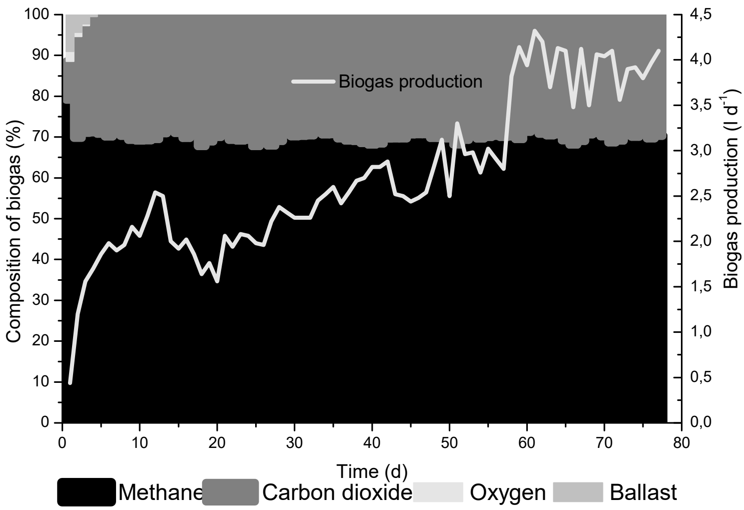

Fig. 5. Daily biogas production and its composition observed during study
Rys. 5. Dzienna produkcja biogazu i jego skład obserwowany podczas badań

Figure 6 shown biogas yield and OLR (organic loading rate) in the semi-continuous lab-scale reactor experiment. During the period when sludge was not conditioned by US organic loading rate remained at a level of 1.7 g L^{-1} d^{-1}. After starting the ultrasound sludge pretreatment, for the first tested amplitudes (36.6 μm), a low (4%) reduction in the value of the parameter was observed. OLR was higher for all other amplitudes, 1.8 g L^{-1} d^{-1} for A = 48.8 μm and 1.9 for A = 61.0 μm. Changes of OLR correlated with the production of biogas and specific biogas yield (Y). The value of this parameter for no prepared sludge ranged from 0.1

to 0.16 $LgVS^{-1}$. While the average value of Y for sludge conditioned by US field with an amplitude of 36.6 µm, 48.8 µm and 61.0 µm was 0.18, 0.22 and 0.21 respectively. It means that sonication of sludge at A = 48.8 µm/18 min causes a significant increase in the rate of biogas yield.

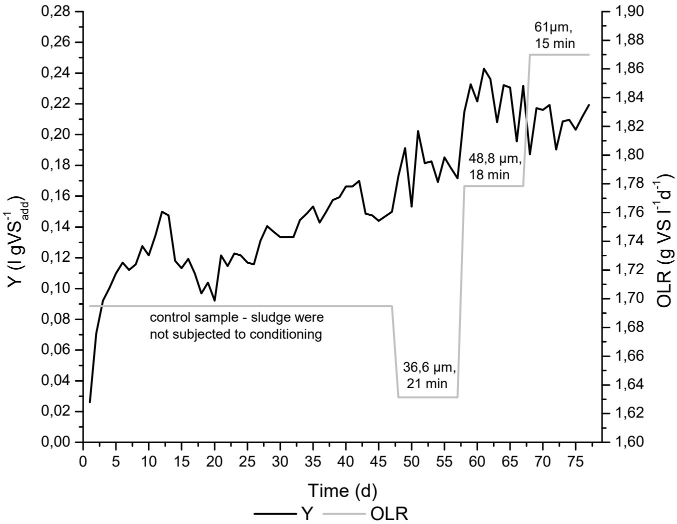

Fig. 6. Biogas yield and OLR in the semi-continuous lab-scale reactor experiment
Rys. 6. Wydajność biogazu i OLR w eksperymencie z reaktorem półciągłym w skali laboratoryjnej

Figure 7 shown the effect of sonication on composition of biogas, biogas yield and VS removal. During the fermentation of sewage sludge average methane content in the gas fermentation was at the level 70% and ultrasound conditioning of sewage sludge did not affected the biogas composition. The same situation was observed for the fermentation efficiency basis on VS removal. Only in the case of the highest amplitude VS removal was 2% higher as compare as the control test.

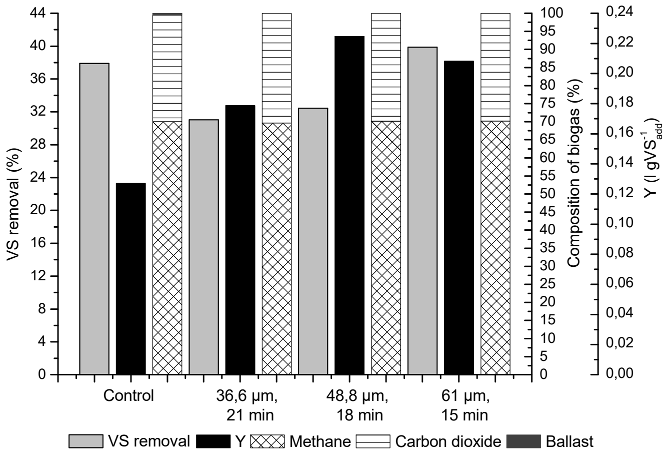

Fig. 7. Effect of sonication on composition of biogas, biogas yield and VS removal
Rys. 7. Wpływ nadźwiękawiania na skład biogazu, wydajność biogazu I usuwanie VS

4. Conclusions

The main objective of this study was to determine the most favorable US field parameters for preparation of sewage sludge generated in the dairy industry. Moreover the effect of sewage sludge sonication (for selected parameters) on the anaerobic digestion effects was determined.

Study of ultrasound pretreatment before anaerobic digestion showed that:

- ultrasound field had a positive impact on the effects of biogas production rate. Compared to the control reactor, during fermentation of dairy sludge conditioned with US field with amplitude 48.8 microns 52% higher biogas production was observed;
- UD field sludge conditioning did not significantly influence the extent and composition of biogas and VS removal (average methane content fluctuated in the range of 70%).

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Wpływ kondycjonowania polem ultradźwiękowym na fermentację metanową osadów ściekowych z przemysłu mleczarskiego

Streszczenie

W niniejszym artykule określono wpływ preparowania przemysłowych osadów ściekowych polem ultradźwiękowym na efekt fermentacji metanowej. Najkorzystniejsze parametry nadźwiękawiania dla preparowanych osadów, ustalono na podstawie zmian TOC w wodzie nadosadowej oraz efektów fermentacji metanowej w układzie ciągłym, wyrażonej wzrostem produkcji biogazu oraz ubytku suchej masy organicznej. Na podstawie uzyskanych wyników stwierdzono, że optymalne parametry nadźwiękawiania to czas ekspozycji wynoszący 18 minut i amplituda drgań 48.8 μm. Kondycjonowanie polem UD pozytywnie wpłynęło na efekty procesu, wyrażone współczynnikiem produkcji biogazu. W porównaniu do próby kontrolnej w osadach kondycjonowanych polem UD o amplitudzie 36.6 μm, 48.8 μm i 61.0 μm odnotowano zwiększenie ww. parametru odpowiednio o 38, 73 i 60%.

Abstract

In this paper, we focused on the effects of ultrasound (US) irradiation at different parameters on solubilization, biodegradation and anaerobic fermentation of sludge from the dairy industry. The changes of TOC in soluble fraction of sludge, the biogas yield, and the methane content in the biogas were used as control parameters for evaluating the effects of the US pretreatment. The optimal sonication parameters were found to be an exposure time of 18 min and ultrasound wave amplitude of 48,8 μm. The UD field conditioning positively influenced the process effects measured by the biogas production coefficient. In comparison to the control sample in sediments conditioned with a UD field with an amplitude of 36.6 μm, 48.8 μm and 61.0 μm, an increase was noted in the above-mentioned parameter by 38, 73 and 60%, respectively.

Słowa kluczowe:
biogaz, fermentacja metanowa, ultradźwięki

Keywords:
biogas, anaerobic fermentation, ultrasound