

## Application of an Innovative Ultrasound Disintegrator for Sewage Sludge Conditioning Before Methane Fermentation

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### ABSTRACT

Ultrasonic disintegration is one of the most interesting technologies among all known and described technologies for sewage sludge pre-treatment before the process of methane fermentation. This study was aimed at determining the effects of an innovative ultrasonic string disintegrator used for sewage sludge pre-treatment on the effectiveness of methane fermentation process. In this experiment, we used a device for disintegration of organic substrates, including sewage sludge, with the use ultrasonic waves. Its technical solution is protected by a patent no. P. 391477 – *Device for destruction of tissue and cell structures of organic substrate*. The volume of biogas produced ranged from  $0.194 \pm 0.089$  dm<sup>3</sup>/g o.d.m. at loading of 5.0 g o.d.m./dm<sup>3</sup> and power of 50 W to  $0.315 \pm 0.087$  dm<sup>3</sup>/g o.d.m. at loading of 4.0 g o.d.m./dm<sup>3</sup> and ultrasounds power of 125 W. The study demonstrated a positive effect of sewage sludge sonication on the percentage content of methane in biogas. Sewage sludge exposure to 125 W ultrasounds increased methane content in biogas to  $68.3 \pm 2.5$  % at tank loading of 3.0 g o.d.m./dm<sup>3</sup>.

**Keywords:** sewage sludge, ultrasound disintegrator, methane fermentation, biogas

### INTRODUCTION

Ultrasonic disintegration is one of the most interesting technologies among all known and described technologies for sewage sludge pre-treatment before the process of methane fermentation. The main reasons behind studies on the ultrasonic field and application of installations based on its use in the existing facilities include very wide applicability and technological versatility of the sonication process [Quiroga et al. 2014].

This physical factor may induce deep physicochemical changes in the sonicated sludge that are highly desired from the viewpoint of sewage sludge processing. A special role is ascribed in this case to disintegration of sewage sludge bacteria which are next subjected to the process of methane fermentation. Damage of cell structures

of microorganisms secreted in settling tanks of a wastewater treatment plant enables biodegradation of organic biomass in the process of sludge stabilization with anaerobic methods. Once the cell wall of microorganisms is damaged, cytoplasm and cellular enzymes are released, while the substrates released in this way, in either dissolved or colloidal form, are immediately available for biological degradation by anaerobic bacteria [Negral et al. 2013]. To ensure the maximal release of organic matter from dead cells of microorganisms, disintegration aims to result in particles smaller than 10 μm. In many research works and in many facilities operating in the technical scale, contents of organic substances in the dissolved phase of the super-sludge liquid (expressed as COD, BOD<sub>5</sub>, TOC) were demonstrated to increase immediately after sonication [Negral

et al. 2013, Martínez-Moral et Tena Focused 2013]. This phenomenon is indicative of immediate sonolysis of solid substances contained in the sludge bulk and of the release of the cellular material from dead microorganisms, and finally points to intensification of the hydrolytic stage of the fermentation process. This allows for significant reduction in time needed for anaerobic stabilization of the sludge and for increasing efficiency of producing gaseous metabolites of anaerobic bacteria [Alagöz et al. 2018].

Although the first installations based on the use of ultrasounds for excess sludge pre-treatment via disintegration are already operating in municipal wastewater treatment plants, the acoustic and mechanical disintegration technology of sewage sludge is still in the experimental phase. Studies in progress are focused on both the phenomena occurring in the sonicated sludge and their reference to the effects of disintegration as well as on the search for effective methods and devices for ultrasonic disintegration [Dhar et al. 2012].

This study was aimed at determining the effects of an innovative ultrasonic string disintegrator used for sewage sludge pre-treatment on the effectiveness of methane fermentation process.

## METHODS

### Study design

The experiment was divided into two stages. The first involved analyses of the effect of ultrasounds on sewage sludge parameter significant for methane fermentation, i.e. content of organic carbon in the dissolved form (TOC). The second stage was aimed at determining the course, efficiency and characteristics of the methane fermentation process. The experiment was conducted under laboratory conditions, in 5 series differing in the power of ultrasounds applied to the sewer

age sludge (variant 1 – 0 W, variant 2 – 25 W, variant 3 – 50 W, variant 4 – 100 W, variant 5 – 125 W). In each variant, exposure time to ultrasounds was 600s.

To determine methane fermentation efficiency, model anaerobic reactors (by WTW company) were used in the study. They consisted of reaction tanks with an active volume of 0.5 dm<sup>3</sup> coupled tightly with devices measuring and registering changes in partial pressure in the measuring tank induced by biogas production. In each experimental variant, 200 cm<sup>3</sup> of anaerobic sludge (inoculum) were introduced into the reaction tank which was then filled with the assumed volumes of pre-disintegrated sewage sludge. Anaerobic conditions were provided inside the exploited fermentation tanks by blowing the installation through with nitrogen before measurements to remove atmospheric air from respirometers. The initial organic matter load ranged from 3.0 g o.d.m./dm<sup>3</sup> to 5.0 g o.d.m./dm<sup>3</sup> depending on variant. A complete measuring set was put into a thermostat cabinet with hysteresis not exceeding  $\pm 0.5^{\circ}\text{C}$ . Measurements were conducted at a temperature of 35°C for 20 days, whereas pressure values in the reaction tank were registered every 24 h.

## MATERIALS

Excessive sewage sludge used in the experiment originated from the Municipal Wastewater Treatment Plant “Łyna” in Olsztyn. It is a biomechanical wastewater treatment plant, with the mean flow capacity of 56,135 m<sup>3</sup>/d (max 63,310 m<sup>3</sup>/d). The plant operates based on a three-stage treatment process with biological removal of biogenes. Sludge was obtained from gravity belt thickener intended for sludge thickening before introducing it to fermentation chamber. Characteristics of excess sewage sludge used in the study was provided in Table 1.

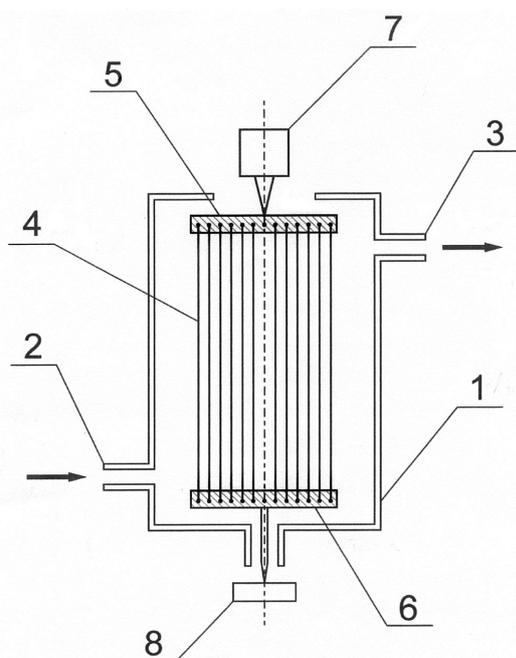
**Table 1.** Characteristics of sewage sludge used in the experiment

Parameter	Unit	Min. value	Max. value	Mean value
Hydration	[%]	97.25	96.92	97.08
Dry matter	[g/dm <sup>3</sup> ]	27.490	28.840	28.165
Mineral substances	[g/dm <sup>3</sup> ]	5.950	7.760	6.855
Volatile substances	[g/dm <sup>3</sup> ]	19.570	20.560	20.065
TOC	[mg/dm <sup>3</sup> ]	2696.3	2949.0	2822.6
Acidity	[pH]	5.78	6.49	6.13
<i>Clostridium perfringens</i>	[CFU/g d. m.]	6.7 × 10 <sup>4</sup>	7.9 × 10 <sup>4</sup>	7.3 × 10 <sup>4</sup>
Coli group bacteria	[MPN/g d. m.]	7.1 × 10 <sup>6</sup>	7.9 × 10 <sup>6</sup>	7.5 × 10 <sup>6</sup>
Fecal coliform bacteria	[MPN/g d. m.]	2.6 × 10 <sup>6</sup>	3.8 × 10 <sup>6</sup>	3.2 × 10 <sup>6</sup>

## Experimental stand

In this experiment, we used a device for disintegration of organic substrates, including sewage sludge, with the use ultrasonic waves. Its technical solution is protected by a patent no. P. 391477 – *Device for destruction of tissue and cell structures of organic substrate*. This device has the form of a cylindrical tank with strings mounted inside that transfer ultrasound vibrations (Figure 1). The flow in the reactor is from bottom up. Sewage sludge flowing through the reactor is exposed to the ultrasounds along the entire active length of the reactors. Ultrasounds cause abrupt condensation and expansion of the medium they act upon. Cavitation “bubbles” are formed which damage the structure of the organic substrate fed to the reactor by their abrupt collapse (implosion).

The device for destruction of tissue and cell structures of the organic substrate consists of a cylindrical tank with an inlet channel (2) and an outlet channel (3) on a side wall, and is equipped in a disintegrating ultrasound generator. Inside the tank (1), strings (4) are fixed to two discs (5 and 6) located in the upper and bottom part of the tank (1). The top disc (5) is connected to the disintegrating ultrasound generator (7), and the bottom disc (6) is connected to the instrument (8) that is used to pull the strings (4).



**Figure 1.** Device for damage of tissue and cell structure of organic substrate with ultrasounds

The device consists of undo elements coupled with each other, namely: main tank made of stainless steel (1–3 mm thick) which is mounted on a 0.2 m high stand and has an inlet in the form of a cylinder ( $\phi 80$  mm, height 50mm). On the bottom of the cylinder there is a disc which fixes  $\phi 50$  strings with thickness of 10 mm. An inlet pipe (50 mm in diameter, 100 mm in length) is mounted on tank side which has a tip allowing to connect it (using a quick connector) with a pressing pipe. From the upper part, the cylinder is connected with a channel (50 mm in diameter and 1200 mm in length). Strings (1 mm in diameter) are mounted in the channel in 0.6 mm spacing, with external strings fixed 3 mm away from channel walls. Three ball valves ( $\phi 10$ mm) for sample collection are mounted inside the channel along its whole length (in equal spacing). Corpus channel is closed with a top cylinder ( $\phi 80$ mm). The top cylinder (50 mm thick) which has a side inlet (made in the same way as the inlet at the bottom) should be mounted so as the inlet tip was in the position of a  $90^\circ$  elbow to be fixed with a quick connector. A disc with fixed strings is mounted in the top part of the cylinder. Its position may be modified to change string tension using one or three turnbuckles. The central part of the disc is coupled with the disintegrating ultrasound generator operating with a frequency of 30 kHz and power from 50 to 300 W, whose casing is coupled with the top cylinder.

## Analytical methods

The scope of conducted analyses included determinations of concentrations of: dry matter (d.m.), organic dry matter (o.d.m.), mineral dry matter (m.d.m.), total carbon (TC), and total organic carbon (TOC), as well as pH measurements. Concentrations of dry matter, organic dry matter and mineral dry matter in sewage sludge were determined with the gravimetric method. Concentrations of TC and TOC were assayed in biomass samples dried at  $105^\circ\text{C}$ . Analyses were carried out with Flash 2000 elementary particle analyzer by Thermo company. Sewage sludge pH was determined by weighing 10 g of a homogenized air-dry sample into a 100-mL beaker, adding 50 mL of distilled water to the beaker and sample mixing, followed by apparatus calibration and pH measurement.

The presence of *Coli* group bacteria and fecal coliforms was analyzed acc. to the Polish Standard PN-EN-ISO 9308-1:2002(U). Samples of sludge were seeded and incubated onto an isolation medium – a lauryl sulfate broth, to determine gas production capability. *Coli* group bacteria were incubated at 37°C whereas fecal coliforms at 44.5°C. The samples determined for the presence of *Salmonella* genus bacteria were proliferated onto Muller-Kauffman medium, and next the bacteria were isolated onto the solid differentiating and selective S-S medium. Biochemical analyses were conducted with API 20E tests by BioMerieux company. Once bacteria from the genus *Salmonella* were detected in the samples, they were subjected to serological tests to confirm their capability for agglutination with the slide method using HM serum by IMMUNOLAB. To confirm the presence of spore forming bacteria from the species *Clostridium perfringens*, sewage sludge was analyzed acc. to the Polish Standard PN-EN-ISO 2646-1:2002. Samples of sludge were seeded and incubated on the Wilson-Blair medium in anaerostats using AnaeroGen sachets by OXOID, to generate anaerobic conditions. Bacteria around which a black precipitate of iron sulfide has developed were counted.

The biogas potential of substrate at different organic compounds loading was tested. The study was carried out in the methane potential analysis tool (AMPTS II Bioprocess Control). This device was used to measure the quantity of biogas produced. Reactors of the volume of 500 mL were connected to the multifunctional agitation system. Mixing in the reactor run for 30 seconds each 10 minutes. Rotating speed was 100 rpm. The 200 mL of digestate was introduced to the reactors. Anaerobic conditions were achieved by continuous flushing of pure nitrogen through the sludge. Methane fermentation was carried out under mesophilic conditions at 37°C for 40 days. The experiments were performed in triplicates.

Samples of biogas were collected from respirometers for qualitative gas analysis using a needle and a gas-proof syringe. Respirometers were equipped in valves with rubber stoppers enabling gas-proof sample collection. A single collected sample contained 20 cm<sup>3</sup> of biogas, the composition and percentage content of which were analyzed with a GC Agilent 7890 A gas chromatograph. In addition, quality of the produced biogas was evaluated with a GMF 430 analyzer by Gas Data company. Biogas was determined for per-

centage contents of: methane CH<sub>4</sub> and carbon dioxide CO<sub>2</sub>. The volume of biogas produced during respirometric analyses was computed based on the equation of perfect gas. The extent of changes in pressure determined inside the measuring chamber allowed calculating the volume of produced biogas expressed per normal conditions.

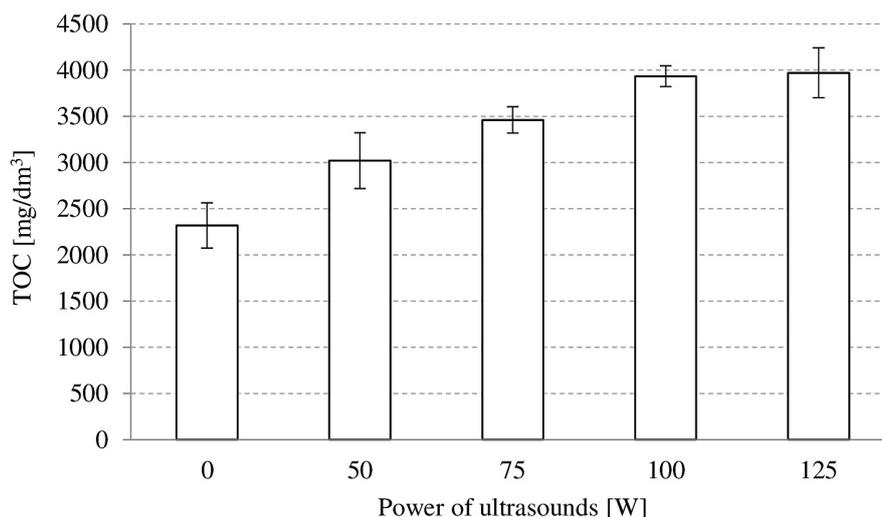
### Statistical analysis

Statistical analysis of the results was made in STATISTICA package 10.0 PL. All physicochemical analyses were carried out in three replications. The hypothesis on distribution of each analyzed variable was verified with the Shapiro-Wilk W test. One-way analysis of variance (ANOVA) was conducted to determine the significance of differences between mean values. Homogeneity of variance in groups was checked with Levene's test, whereas HSD Tukey's test was used to determine the significance of differences between the analyzed variables. Differences were found significant at  $p = 0.05$ .

## RESULTS

The conducted experiment allows concluding that the use of ultrasonic waves contributed to a significant increase ( $p=0,05$ ) in TOC concentration in the effluent after vacuum filtration of the disintegrated sewage sludge. The study showed a direct impact of the applied power of the physical factor on TOC concentration. The highest TOC concentration in the effluent, accounting for 3933±112 mg/dm<sup>3</sup>, was determined in the experimental variant with ultrasound waves having the power of 100 W and was by nearly 40 % higher compared to the non-sonicated sludge. Increasing disintegrator power had no significant effect ( $p=0,05$ ) on the technological effects related to TOC concentration in the effluent, which in this variant equaled to 3970±270 mg/dm<sup>3</sup>. The use of lower powers resulted in a decreased concentration of organic matter in the dissolved phase of the sludge (Figure 2).

It was proved that the volume and composition of biogas produced during respirometric tests was directly dependent on the power of ultrasounds. In turn, efficiency of methane fermentation was not significantly ( $p=0,05$ ) affected by the initial load of organic compounds fed to reactors. Irrespective of the variant of tanks load-



**Figure 2.** TOC concentration in the effluent after vacuum filtration in relation to used power of ultrasounds

ing with organic matter, the volume of produced biogas increased along with an increasing power of ultrasound waves. The volume of biogas produced ranged from  $0.194 \pm 0.089$  dm<sup>3</sup>/g o.d.m. at loading of 5.0 g o.d.m./dm<sup>3</sup> and power of 50 W to  $0.315 \pm 0.087$  dm<sup>3</sup>/g o.d.m. at loading of 4.0 g o.d.m./dm<sup>3</sup> and ultrasounds power of 125 W. The study demonstrated a positive effect of sewage sludge sonication on the percentage content of methane in biogas. Fermentation of non-sonicated sludge allowed producing from  $50.1 \pm 2.1\%$  to  $56.0 \pm 1.8\%$  of methane in biogas depending on respirometric tanks loading with organic compounds. The use of ultrasounds with the power of 125 W increased methane content in biogas to  $68.3 \pm 2.5\%$ , in the variant with organic compounds load of 3.0 g o.d.m./dm<sup>3</sup>. Results of experiments were presented in Tables 2 and 3.

## DISCUSSION

Ultrasonic disintegration aiding the methane fermentation process is mainly aimed at production of biogas as a carrier of renewable energy. The total volume of biogas produced from sonicated excess sludge increases by 30–50% (or even by 100% under laboratory conditions) compared to non-sonicated sludge, which was demonstrated by many authors [Chang et al. 2011]. In our study, the increase in biogas volume was from 12 to 50% depending on the experimental variant.

The first outcome of ultrasonic waves is disintegration and dispersion of flocks with no damage caused to cells of microorganisms – effects of this

type may be monitored with methods of optical or laser microscopy, but also through determination of organic matter content in the dissolved phase. At longer exposure to ultrasound waves or at their higher energy, when cell walls of microorganisms are disrupted and intracellular material is released to the liquid phase, the number of microorganisms decreases [Rochebrochard et al. 2011]. Depending on its origin, the sludge contains microorganisms with various resistance of their cell walls. These walls can be broken only at a high load of energy imposed on a sonicated medium, i.e. either by a long exposure to ultrasounds emission (significantly longer than that needed to damage particle structure of the sludge), or by a short impulse of a very high power [Braguglia et al. 2011, Feng et al. 2009]. Our study demonstrated that the applied disintegrator enabled destruction of cellular structures of the excess sludge and that this phenomenon resulted in increased by 40% TOC concentration in the dissolved phase compared to the non-sonicated sludge.

It was concluded that sewage sludge should be disintegrated with ultrasounds generated at relatively low frequencies, ranging from 16 kHz to 50 kHz, however cavitation excited by acoustic wave intercepted by the sludge requires at least 1.0 W/cm<sup>2</sup> and higher intensity of the acoustic field [Rochebrochard et al. 2013, Grönroos et al. 2005]. For practical applications, especially for estimating costs of the sonication process in wastewater treatment plants, we postulate the calculated index of energy consumption for ultrasound disintegration be at 0.5 kWh/kg d.m. sludge [Wang et al. 2018]. In our study, the frequency of ultrasounds was at 30 kHz.

**Table 2.** Effect conditioning method on the qualitative characteristics of biogas

Parameter		Carbon content in gaseous phase	CO <sub>2</sub> content in gaseous phase	CH <sub>4</sub> content in gaseous phase	CO <sub>2</sub>	CH <sub>4</sub>
Unit		mmol/ g o.d.m.	mmol/ g o.d.m.	mmol/ g o.d.m.	%	%
Power [W]	Load [g o.d.m./dm <sup>3</sup> ]	Value±				
crude	3.0	0.335±0.003	0.147±0.009	0.187±0.016	44.0±1.2	56.0±1.8
	4.0	0.354±0.008	0.170±0.011	0.184±0.019	48.1±1.9	51.9±1.9
	5.0	0.317±0.005	0.158±0.012	0.159±0.015	49.9±2.0	50.1±2.1
50	3.0	0.373±0.011	0.149±0.013	0.224±0.028	39.9±1.8	60.1±1.6
	4.0	0.393±0.009	0.146±0.009	0.247±0.022	39.2±2.1	60.8±1.8
	5.0	0.351±0.008	0.159±0.011	0.192±0.017	45.3±1.9	54.7±1.1
75	3.0	0.373±0.012	0.139±0.020	0.234±0.019	37.2±2.5	62.8±1.6
	4.0	0.393±0.011	0.137±0.015	0.256±0.023	34.9±2.4	65.1±1.8
	5.0	0.351±0.011	0.151±0.016	0.201±0.011	42.9±1.9	57.1±2.0
100	3.0	0.345±0.016	0.160±0.009	0.185±0.017	36.4±3.1	63.6±2.6
	4.0	0.357±0.007	0.155±0.009	0.202±0.009	33.4±3.6	66.6±2.8
	5.0	0.317±0.008	0.153±0.016	0.164±0.018	41.3±1.7	58.7±1.7
125	3.0	0.356±0.005	0.139±0.012	0.217±0.017	37.1±2.9	68.3±2.5
	4.0	0.361±0.008	0.128±0.007	0.233±0.014	35.4±3.1	64.6±2.7
	5.0	0.326±0.009	0.151±0.011	0.155±0.021	40.4±2.9	59.6±1.6

**Table 3.** Effect of conditioning method on the volume of biogas produced during methane fermentation

Parameter		Biogas volume	Biogas volume	Biogas volume
Unit		dm <sup>3</sup> /g d.m.	dm <sup>3</sup> /g o.d.m.	dm <sup>3</sup>
Power [W]	Load [g o.d.m./dm <sup>3</sup> ]	Value		
crude	3.0	0.280±0.059	0.188±0.066	0.414±0.111
	4.0	0.310±0.092	0.208±0.067	0.701±0.201
	5.0	0.260±0.71	0.174±0.045	1.154±0.178
50	3.0	0.330±0.023	0.221±0.055	0.488±0.099
	4.0	0.370±0.040	0.248±0.076	0.836±0.176
	5.0	0.290±0.039	0.194±0.089	1.288±0.199
75	3.0	0.390±0.099	0.261±0.058	0.577±0.166
	4.0	0.420±0.021	0.281±0.044	0.949±0.189
	5.0	0.330±0.043	0.221±0.050	1.465±0.123
100	3.0	0.360±0.036	0.241±0.090	0.533±0.171
	4.0	0.430±0.028	0.288±0.098	0.972±0.132
	5.0	0.340±0.022	0.228±0.121	1.510±0.245
125	3.0	0.420±0.062	0.281±0.090	0.622±0.165
	4.0	0.470±0.031	0.315±0.087	1.062±0.156
	5.0	0.400±0.056	0.268±0.098	1.776±0.233

Changes in the medium induced by the effects of the ultrasonic field may vary and depend not only on the aforementioned acoustic values but also on the physicochemical properties of the medium subjected to sonication: viscosity of liquid, presence of electrolytes and polyelectrolytes, macrostructure and character of the suspension, temperature of the medium, and many others; and therefore may be influenced by many factors acting in the same time [Yaowei, et al. 2018].

The achieved effect of disintegration is largely affected by physical conditions of the process and by parameters of the reactor used for disintegration. Each system will have its own optimal technical conditions of ultrasound disintegration which apart from emission parameters include geometrical conditions of wave propagation: shape and size of the tank, emitter (or emitters) location in the sonicated area and many others [Li et al. 2017].

Ultrasound disintegrators for sewage sludge pre-treatment have so far been produced as individual items by specialized groups in scientific centers or research-and-development units. Their quality and effectiveness are, therefore, largely dependent on the knowledge and skills of their constructors in the fields of acoustics and electronics [de Moortel et al. 2017]. Devices for ultrasound conditioning of sewage sludge before the fermentation process applied so far in municipal wastewater treatment plants represent the type of tubular or tank flow-through reactors. In majority of cases, these are innovative facilities adjusted in their shape, size and power to conditions of a specific installation. Most of devices installed so far have been operating in a frequency range of 20–40 kHz, with feeding electric power ranging from 300 to 2000 W.

## CONCLUSIONS

1. The experiment was conducted with an innovative device for damage of tissue and cell structures of an organic substrate which consisted of a cylindrical tank equipped in a disintegrating ultrasound generator. Its characteristic trait are strings fixed to two discs mounted inside the tank at its bottom and top sides, wherein the top disc is connected with the disintegrating ultrasound generator and the bottom disc – with a device for string tension control. It is an untypical, so far underexploited technical solution.
2. The applied device contributed to a significant increase in TOC concentration in the effluent after vacuum filtration of the disintegrated sewage sludge. The highest TOC concentration in the effluent, accounting for  $3933 \pm 112$  mg/dm<sup>3</sup>, was determined in the experimental variant with ultrasound waves having the power of 100 W and was by nearly 40 % higher compared to the non-sonicated sludge.
3. It was proved that the volume and composition of biogas produced during respirometric tests was directly dependent on the power of ultrasounds. The volume of biogas produced ranged from  $0.194 \pm 0.089$  dm<sup>3</sup>/g o.d.m. at loading of 5.0 g o.d.m./dm<sup>3</sup> and power of 50 W to  $0.315 \pm 0.087$  dm<sup>3</sup>/g o.d.m. at loading of 4.0 g o.d.m./dm<sup>3</sup> and ultrasounds power of 125 W. The study demonstrated a positive effect of sewage sludge sonication on the percentage

content of methane in biogas. Sewage sludge exposure to 125 W ultrasounds increased methane content in biogas to  $68.3 \pm 2.5\%$  at tank loading of 3.0 g o.d.m./dm<sup>3</sup>.

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