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Jolanta BOHDZIEWICZ[1], Mariusz KUGLARZ[2*] and Klaudiusz GRŰBEL[2]

INFLUENCE OF MICROWAVE PRE-TREATMENT ON THE DIGESTION AND HIGIENISATION OF WASTE ACTIVATED SLUDGE

WPŁYW DEZINTEGRACJI MIKROFALOWEJ NA PROCES FERMENTACJI ORAZ HIGIENIZACJI NADMIERNYCH OSADÓW ŚCIEKOWYCH

Abstract: The article presents the results of determining the most appropriate conditions of microwave sludge pre-treatment (500-1200 W), prior to its anaerobic digestion in a continuous mode. The assessment of the pre-treatment conditions (microwave power, sludge temperature after pre-treatment) was based on: the release of organic (COD, protein) and inorganic (NH_4^+, PO_4^{3-}) substances into liquid, the quantity of methane produced, sludge higienisation and the susceptibility of the pre-treated sludge to dewatering. The power of the microwaves applied did not play significant role on the pre-treatment effectiveness. Taking into account the fact that sludge pre-treatment by microwave irradiation requires the delivery of energy, the pre-treatment by microwaves of higher power (1200 W) and resulting in sludge temperature of 70°C was recommended for further experiments. Sludge pre-treatment by means of microwave irradiation as a pre-treatment step influenced the effectiveness of the subsequent anaerobic digestion, conducted in continuous conditions, in a positive way. The largest amount of biogas was obtained for HRT in the range of 15-20 days. As compared to the sludge which did not undergo pre-treatment, daily biogas production and biogas yield increased by 18-41% and 13-35% respectively. The combination of microwave pre-treatment and mesophilic anaerobic digestion ensured the elimination of pathogens (*Salmonella* spp., *Escherichia coli*).

Keywords: microwave irradiation, sludge pre-treatment, anaerobic digestion, secondary sludge

Introduction

The main purpose of the sludge anaerobic digestion is the conversion of strongly hydrated, odorous and environment-unfriendly raw sludge into a well-dewatering, digested sludge. Additionally, the process allows producing renewable energy in the form biogas [1, 2]. The main ingredients of waste activated sludge (WAS) are organic and inorganic substances attached to microbial cells. Due to the complex structure, agglomerated by extracellular polymeric substances (EPS) as well as cations determining the flocs integrity

[1] Institute of Water and Wastewater Engineering, Faculty of Energy and Environmental Engineering, Silesian University of Technology, ul. Konarskiego 18, 44-100 Gliwice, Poland
[2] Institute of Environmental Protection and Engineering, Faculty of Materials and Environmental Sciences, University of Bielsko-Biala, ul. Willowa 2, 43-309 Bielsko-Biala, Poland
[*]Corresponding author: mkuglarz@ath.bielsko.pl

and strength, the secondary sludge is difficult to undergo biodegradation in anaerobic conditions. Accordingly, the main purpose of its disintegration is to disrupt cell membranes and, thus lysis microbial cells. Both lead to a release of polymeric substances in a soluble form into liquid phase [3, 4].

The application of microwave irradiation to facilitate the process of sludge anaerobic digestion is a relatively novel method and has recently gained particular interest. Microwaves are a type of electromagnetic waves within the range between 0.3-300 GHz, and the wave length of 0.01-1 m [4, 5]. Due to the high content of water, sludge easily absorbs microwave irradiation. The mechanism of microwave irradiation includes “thermal” and so-called “non-thermal” effects. The former is strictly connected with heat generation by chemical particles which are dipoles. They spin and heat neighbouring particles and, thus pass the energy gained onto them. The latter effects are not strictly connected with the temperature increase. They are probably caused by polarized parts of macromolecules aligning with the poles of the electromagnetic, which may be responsible for breakage of hydrogen bonds, leading to denaturation and microorganism death [6]. The positive effects of sludge pre-treatment by means of microwave irradiation have recently been reported in the literature [7-10]. However, the cases mainly focuses on the influence of microwave irradiation on the pre-treatment and do not contain research aimed at subsequent anaerobic digestion of treated sludge in continuous conditions. The present study is focused on the assessment of microwave irradiation at the power range of 500-1200 W as a pre-treatment step before sludge anaerobic digestion in continuous conditions. The assessment of the sludge pre-treatment conditions was based on the release of organic (COD, protein) and inorganic (NH_4^+, PO_4^{3-}) substances, the quantity and quality of biogas produced and the susceptibility of the pre-treated sludge to dewatering. Additionally, the influence of microwave pre-treatment and/or anaerobic digestion on biological quality of the sludge treated was taken into account.

Materials and methods

Characteristics of the sludge

The sludge (WAS) was taken after thickening from a full scale municipal treatment plant, based on Enhanced Biological Nutrients Removal (EBNR), operated on the activated sludge method. The plant treats domestic as well as industrial wastewater. The latter fraction constitutes up to 10% of the total influent and is pre-treated before mixing with domestic wastewater. Table 1 presents the physical and chemical properties of the sludge, used as a research material.

Description of the pre-treatment

Microwave treatment was performed using a laboratory unit equipped with magnetrons of the following powers: 500, 800, 1000 and 1200 W. The disintegration was performed in closed vessels and the volume of sludge treated in one run amounted to 1 dm^3. The temperature of the sludge was gradually increased by 10°C, - within the range of 20°C and ~100°C. As long as the samples reached the fixed temperature, they were cooled down to about 10°C. The temperature of the sludge treated was taken by a probe installed inside the pre-treatment unit. Table 2 presents microwave exposure times required to obtain the particular temperatures of the sludge pre-treated.

Table 1

Characteristics of the secondary sludge

Indicator	Unit	Sewage sludge	
		Range of values	Average value
pH	-	5.9-6.6	6.3 (0.3)*
TS	[%]	5.17-5.41	5.27 (0.17)
VS	[%]	3.63-3.83	3.71 (0.16)
VFA	[mg/dm^3]	580-795	655 (168)
TKN	[% TS]	4.88-5.01	4.96 (0.11)
NH_4^+	[mg/dm^3]	11.4-19.8	15.8 (5.9)
PO_4^{3-}	[mg/dm^3]	63.2-80.8	75.5 (12.6)
SCOD	[mg/dm^3]	102-430	247 (122)
Protein	[mg/dm^3]	115-132	122 (12)
TCOD	[mg/dm^3]	20600-23500	22520 (1890)

*() - standard deviations

Table 2

Microwave exposure times

Temperature [°C]	Microwave power			
	500 W	800 W	1000 W	1200 W
	Reaction time of microwaves [s]:			
30°	112 (6)*	69 (4)	52 (4)	40 (3)
40°	202 (5)	105 (5)	70 (3)	55 (3)
50°	310 (6)	150 (6)	115 (4)	72 (3)
60°	425 (8)	212 (5)	152 (5)	95 (3)
70°	485 (9)	268 (6)	185 (6)	122 (3)
80°	550 (8)	310 (7)	215 (5)	148 (4)
90°	610 (9)	340 (6)	235 (5)	168 (4)
~100°	630 (8)	350 (5)	260 (4)	185 (4)

*() - standard deviation

The degree of sludge disintegration (DD_{COD}) was determined according to equation (1) [11, 12]:

$$DD_{COD} = \frac{SCOD_D - SCOD_O}{TCOD - SCOD_O} \cdot 100\% \qquad (1)$$

where: $SCOD_D$ - concentration of organic matter (*COD*) after pre-treatment, measured in the liquid phase [mg O_2/dm^3], $SCOD_0$ - concentration of organic matter (*COD*) in a raw sample, measured in the liquid phase of sludge [mg O_2/dm^3], *TCOD* - total concentration of organic matter (*COD*) after chemical disintegration by means of 1M NaOH [mg O_2/dm^3].

The process of chemical disintegration was conducted for 10 min at 90°C. The ratio of NaOH to sludge amounted to 1:1 by volume.

Energy demand of the pre-treatment

The energy demand of the pre-treatment was calculated taking into account the power of the microwaves as well as the exposure time applied for achieving particular sludge temperatures. Energy consumption $E_{COD/Protein}$ [kJ/g of SCOD or protein released] was calculated according to the formula (2):

$$E_{COD/\mathrm{Protein}} = \frac{P_D \cdot t_D}{V} \tag{2}$$

where P_D - is the power of microwaves [kW], t_D - is the exposure time of microwaves [s], V - is the organic matter released per kg of sludge treated, expressed as COD or proteins [g].

Biogas potential of sludge pre-treated

Methane potential of raw and pre-treated sludge by microwave irradiation was conducted in bottles with working volume of 500 ml in static conditions. Sludge (250 cm^3) and methanogenic inocula (250 cm^3) were added to the bottles. Digested sludge (Silesian district, Poland) taken form continuous mesophilic process was used as inocula. The assays were undergoing digestion at 37°C, until no significant amounts of CH_4 were produced (approx. 25-30 days). The amount of methane produced was expressed per unit of VS added. The biogas produced by inocula was subtracted from all tested trials [13].

Continuous anaerobic digestion

The digestion process was conducted in two bioreactor with a working volume of 3 dm^3, the first was fed with raw sludge and the other with pre-treated sludge by microwaves in conditions considered as the most appropriate. The process was carried out at the following hydraulic retention times (HRT): 10, 12, 15, 17, 20, 25 and 30 days. The digesters were kept at a constant temperature of 36ºC (±0.5) and their contents were mixed periodically - 5 minutes in every 3 hours. The applied range of HRT corresponds to the OLR (Organic Loading Rate) value of between 1.18 and 2.35 kg VS/($m^3 \cdot$ d). Both bioreactors were operated at a constant HRT until the fluctuation of biogas production was lower than ±10%. The reactors were started at a hydraulic retention time of 30 days and then the value of HRT was subsequently decreased. Bioreactors were maintained for at least three HRTs before decreasing HRT. The biogas produced was measured by water displacement method and stored in a plexus tube containing 5% NaOH solution. The recorded amounts of biogas were adjusted to the volume at standard temperature (0°C) and pressure (1 atm). Biogas samples were analysed for CH_4 by means of portable meter. The amount of biogas produced was expressed as daily production [dm^3/($dm^3 \cdot$ d)] and biogas yield [m^3/kg of VS_{added}].

IR spectroscopy analysis

The influence of microwave irradiation on the effectiveness of sludge pre-treatment was confirmed by IR spectroscopy analysis. Changes in the structure of organic matter after pre-treatment were assessed by the application of Nicolet Magna-IR 860 spectrophotometer. The sludge samples were centrifuged (4000 rpm/min) and filtrated (0.45 μm). Residues after water evaporation and carrier addition (KBr) were compressed by pressure (1000 MPa). The sample containing only KBr was used as a control. Analyses were conducted for the spectrum range of between 1000 and 4000 cm^{-1}. IR analyses were conducted for the raw as well as pre-treated sludge, in conditions of sludge temperature considered as the most appropriate (70°C).

Analytical methods

The scope of the analyses conducted encompassed: pH value measurement and determinations of total solids (TS), volatile solids (VS), chemical oxygen demand (COD), total oxygen demand (TCOD), soluble protein, ammonia (NH_4^+) and total Kjeldahl (TKN) nitrogen as well as, phosphates (PO_4^{3-}) [14]. The susceptibility of the sludge to dewatering was based on the capillary suction time (CST). Microbiological studies included indication of: total number of bacteria in the sludge, the number of *Escherichia coli*, *Salmonella* spp. and *Clostridium perfringens*. The number of colony forming units (CFU) of bacteria in the sludge samples was determined by platelet grated method on selective media: blood agar, Mac Conkey. Before determination of *Clostridium perfringens*, dilutions of sludge samples were heated in water-bath at 80°C for 15 min to kill vegetative bacteria [15]. All analyses were based on 4 replications and the results were presented as average values. The error bars in figures and parentheses in tables represent standard deviations.

Results and discussion

Sludge microwave pre-treatment

Firstly, the study focused on effectiveness of sludge pre-treatment by microwave irradiation. The assessments of various levels of microwave power as well as the temperature of the treated sludge were taken into consideration.

Organic matter release

As the temperature of the sludge pre-treated increased, the concentration of the organic matter, expressed as CODs increased. Figure 1 presents influence of the microwave irradiation on COD release and degree of disintegration. Although the highest values of COD concentration were recorded for the sludge temperature of 90°C (7455-7665 mg O_2/dm^3), it should be noted that increasing temperature of sludge treated above 80°C did not have a significant influence on the amount of organic matter released. When the temperature of the sludge increased to ~100°C, pre-treatment methods applied were not recorded to have a positive impact on COD release. An intensive boiling of the sludge was observed, which might have led to a partial release of organic substances into the atmosphere. As a result, a slight decrease in COD concentration was observed.

One of the most frequently indicator used for the assessment and comparison of disintegration effects, caused by varied process conditions, is the degree of disintegration [10, 16]. Taking into account the fact that the parameter reflects the release of soluble organic substances (COD), it was not surprising that its value increased as the temperature of the sludge pre-treated increased. The highest values after microwave pre-treatment reached the level of 30-32% and corresponded with the sludge temperature of 80-90°C (Fig. 1).

The power of the microwave irradiation applied did not play a significant role in the effectiveness of pre-treatment processes. However, slightly higher values of COD concentration were recorded in case of sludge pre-treated by microwaves of the lower power, *ie* 500-800 W, which was accounted by the fact that the sludge was exposed to longer microwaves action in order to achieve fixed temperature ranges. This is in agreement with previous studies [16, 17].

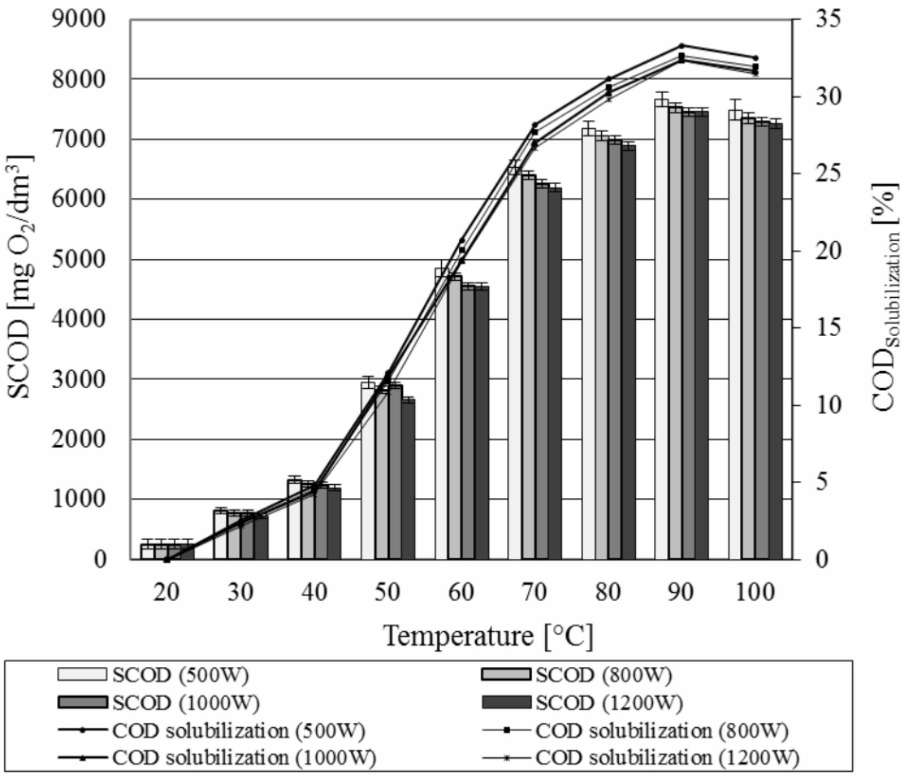

Fig. 1. The influence of the microwave irradiation on COD release and degree of disintegration

An effective sludge pre-treatment can also be proved by extracellular polymeric substances increase (EPS) in liquid sludge phase [7, 18]. In this study, the influence of microwave irradiation on the amount of soluble protein released was observed (Fig. 2). As long as the sludge reaches the temperature of about 60°C, there was almost a linear correlation between the concentration of protein released and the temperature of the sludge subjected to action of microwaves. An increase of temperature in the range of 60-70°C caused further release of proteins and the highest values were observed, *ie* 2708-2762 mg/dm^3. Further pre-treatment did not play a positive influence on the protein release, its concentration decreased by 2-3%. Under conditions of high temperature, denaturation, aggregation and precipitation of dissolved protein occur, which are believed to be associated with changes in their degrees of hydration and solubility. Decrease in biopolymers content at temperature of ~80°C is frequently observed [6, 19, 20].

Under conditions of high temperature, reactions between nitrogen-containing compounds such as amino acids, proteins and dissolved sugars, which are so-called Maillard reactions, - are leading to their polymerization and re-bound in the form of macromolecular biopolymer compounds [6, 7]. In case of the pre-treatment leading to an increase in sludge temperature above 80°C, these phenomena may be responsible for a slight decrease in proteins.

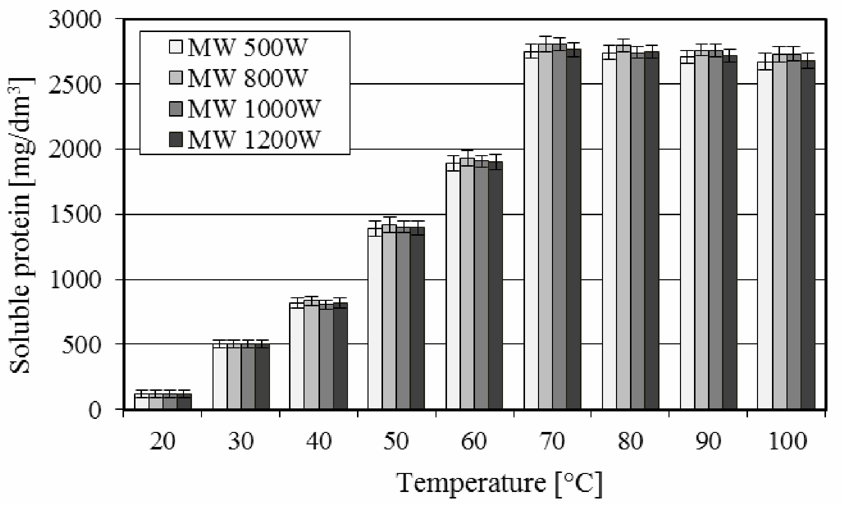

Fig. 2. The influence of the microwave irradiation on protein release

Structure of organic compounds after pre-treatment

The study presents identification of selected functional groups of compounds released during the pre-treatment into sludge liquid by means of IR technique (Fig. 3).

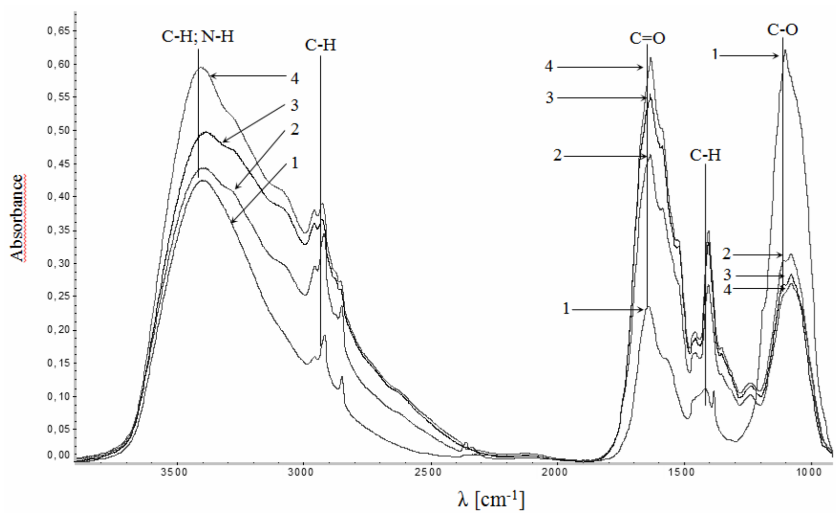

Fig. 3. Changes in organic matter structure after sludge pre-treatment at 60°C (1 - raw sludge, 2 - sludge pre-treated at 500 W, 3 - sludge pre-treated at 800 W, 4 - sludge pre-treated at 1200 W; as an example)

There was a clear difference in the absorbance between the untreated sludge and those pre-treated by microwave irradiation. Surface area increase corresponding to the peak at a wavelength of about 3350 cm^{-1} shows the release of compounds containing in its structure O-H and N-H bonds. The biggest impact on changing concentrations of these oscillators had a pre-treatment by means of microwave power of 1200 W.

The sludge after disintegration with microwaves of lower power (500-1000 W) exhibited a significantly lower absorbance value. A significant increase in absorbance was also obtained for the C-H bonds (λ = 2900 cm^{-1}). However, there was no significant effect of microwave power on the value of absorbance. Moreover, the increase in areas at wavelengths of 1700 and 1800 cm^{-1} indicates increase of C=O bonds, which are present in carboxylic acids, esters and amides. For this wave length, a significant increase in absorbance was observed after applying all microwave powers. However, the higher the power of microwaves, the higher the value of absorbance. There were no beneficial effects of applied pre-treatment methods on absorbance at a wavelength of 1100 cm^{-1}, corresponding to release of compounds containing the C-O, P-O and S-O bonds.

Inorganic matter release

Destruction of cell membranes during pre-treatment leads to the release of enzymes located in protoplasma, which are responsible for hydrolytic decomposition of nitrogen and phosphorus compounds [18].

a)

b)

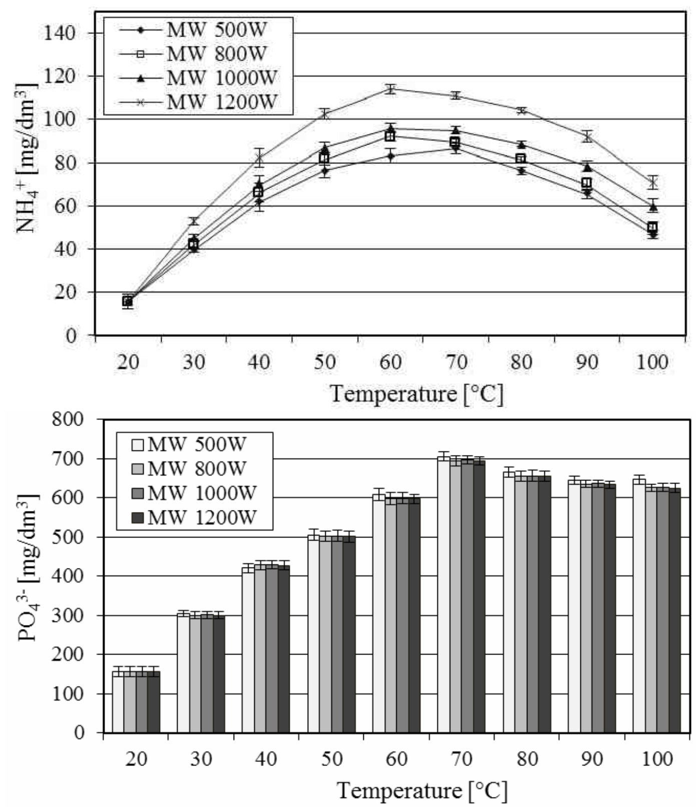

Fig. 4. The influence of the sludge pre-treatment on the ammonia-nitrogen (a) and phosphates (b) release

For all microwaves powers tested, the highest release of NH_4^+ and PO_4^{3-} was observed for the sludge temperature of 60-70°C (Fig. 4). The lower the power of the microwaves, the higher the concentration of NH_4^+ released, which was associated with longer exposures times of the disintegrating agent in order to reach the particular temperatures of the sludge treated. Furthermore, the increase of temperature above 70°C caused a significant decrease in ammonia and phosphates concentration. This is in agreement with previous studies [6, 21].

Biogas potential

The sludge solubilization caused by an effective destruction influences the amount of biogas produced. As the temperature of the sludge pre-treated increased, the amount of methane produced increased (Fig. 5).

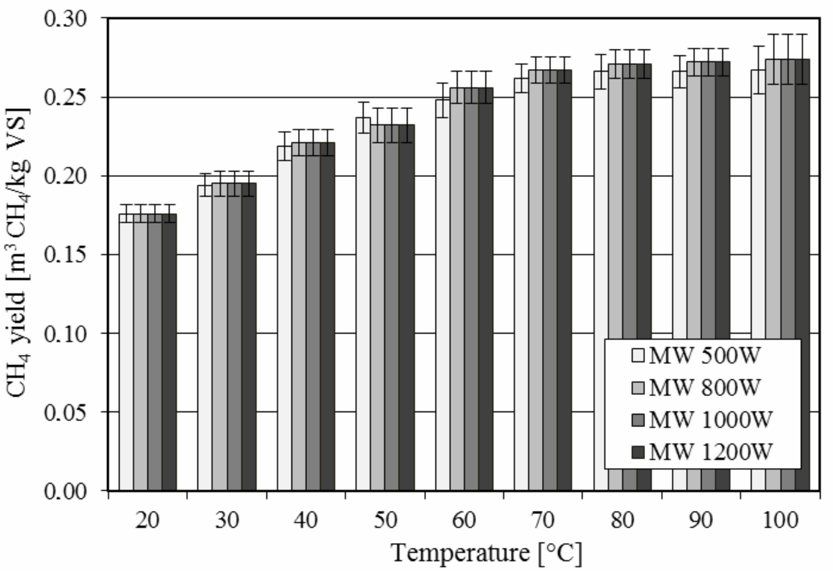

Fig. 5. The influence of the sludge pre-treatment on methane yield

The highest methane yields amounted to 0.262-0.274 m^3 CH_4/kg VS and were achieved after pre-treatment, resulting in sludge temperature of 70-100ºC. These values constitute 49-56% increase compared to untreated sludge. What is more, power of microwaves applied did not affect significantly the methane potential, which is in agreement with previous studies [21, 22].

Higienisation and dewatering

The influence of microwave irradiation on the biological quality and sludge susceptibility to dewatering was taken into account. Regardless of the power of the microwaves applied, the overall number of bacteria was decreasing along with the increase of sludge temperature (Table 3). The sludge pre-treated at 70°C did not contain *Escherichia* *coli*, while, the sludge pre-treated at 80°C did not contain both *Escherichia coli* and *Salmonella spp.* To demonstrate the higienisation effect of thermal pre-treatments, including microwave irradiation, the presence of heat-resistant microorganisms (sporulating sulfite reducing anaerobic bacteria (SRC)), *eg Clostridium perfingens*, may be used as indicators [23, 24]. The sludge pre-treatment resulting in the sludge pre-treatment of ≤ 70°C did not have a significant effect on *Clostridium perfringens* content. Application of the microwave irradiation at 80-90°C and ~100°C resulted in the 22-25% and 57% decrease of heat-resistant microorganisms (*Clostridium perfringens*), respectively.

Table 3

Impact of microwave irradiation on microbiological and dewatering sludge properties

Parameter	Raw sludge	Pre-treated sludge					
		≤ 50°C	**60°C**	**70°C**	**80°C**	**90°C**	**~100°C**
Microbiological indicators							
Overall number of bacteria [log cfu/g dry weight]	8.93 (0.4)*	8.30 (0.2)	7.83 (0.3)	7.38 (0.2)	6.95 (0.3)	5.95 (0.3)	5.27 (0.2)
Escherichia coli [log cfu/g dry weight]	6.41 (0.3)	5.79 (0.2)	4.66 (0.3)	-	-	-	-
Salmonella spp. [MPN lower limit of 95% CI - MPN upper limit of 95% CI]]MPN/g wet weight]	11 [2.6 - 4.7]	2.1 [0.61-7.6]	1.5 [0.42-5.4]	0.092 [0.023-0.37]	-	-	-
Clostridium perfringens [log cfu/g dry weight]	7.71(0.6)	7.61 (0.2)	7.30 (0.3)	6.65 (0.3)	6.04 (0.2)	5.77 (0.2)	3.30 (0.3)
Susceptibility to dewatering							
CSK (s)	40 (7)	32 (4)	36 (5)	50 (5)	54 (6)	62 (5)	58 (5)

*() - standard deviation

Furthermore, the impact of pre-treatment on the sludge susceptibility to dewatering was analysed. Sludge dewatering has a significant impact on further sludge treatment as well as capacity of containers used for digested sludge storage [25, 26]. The microwave pre-treatment resulting in an increase in sludge temperature ≤ 60°C influenced the sludge filtration properties in a positive way (Table 3). Furthermore, a slight deterioration of the sludge susceptibility to dewatering was observed. The CST value of the sludge pre-treated at ≥ 70°C amounted 50-62 s, which is about 25-45% higher than the value obtained for raw sludge (CST = 70 s). This deterioration of the sludge dewaterability might have been caused by increased concentration of organic and inorganic substances, release to sludge liquor during pre-treatment. As in case of previous parameters analysed, the power of the microwave irradiation did not have a significant influence on the sludge dewatering properties.

Energy demand of the pre-treatment

The higher the power of the microwaves applied, the lower the energy consumption during organic matter release. The lowest energy demands were achieved after treatment by means of microwaves of the highest power (1200 W) and the temperature of the sludge treated of 70°C and the values amounted to 23.6 and 53.0 kJ/g, expressed as COD and protein respectively. For the same temperature (70°C), the energy demand of processes conducted at lower powers (500-1000 W) turned out to be more energy demanding. In this case, about 25-57% and 25-66% higher values of the parameters were achieved, expressed in terms of COD and protein respectively. Increasing the temperature of sludge pre-treated above 70°C caused an increased in energy demand, which was ascribed to longer microwaves exposure times (Table 4).

Table 4

Energy consumption during organic matter release

T [°C]	Microwave 500 W		Microwave 800 W		Microwave 1000 W		Microwave 1200 W	
	$E_{(COD)}$	$E_{(Protein)}$	$E_{(COD)}$	$E_{(Protein)}$	$E_{(COD)}$	$E_{(Protein)}$	$E_{(COD)}$	$E_{(Protein)}$
	[kJ/g]		[kJ/g]		[kJ/g]			
30	69.6 (4.4)*	112 (5.6)	71.5 (3.7)	108 (5.3)	67.1 (3.5)	103 (6.1)	66.5 (4.0)	95.3 (5.7)
40	74.6 (3.4)	124 (4.6)	67.1 (5.1)	101 (3.6)	55.5 (1.8)	88.0 (2.4)	55.6 (3.0)	80.1 (3.3)
50	52.5 (1.6)	112 (4.7)	45.4 (1.1)	90.3 (3.1)	39.7 (0.8)	82.0 (2.9)	32.5 (0.8)	61.9 (2.4)
60	43.6 (1.0)	112 (4.6)	36.0 (0.5)	87.9 (2.2)	33.1 (0.5)	80.3 (2.1)	25.1 (0.3)	59.9 (1.6)
70	37.0 (0.8)	88.0 (1.8)	33.5 (0.4)	76.3 (1.8)	29.6 (0.4)	66.2 (1.0)	23.6 (0.3)	53.0 (1.3)
80	38.4 (0.8)	100 (1.7)	35.2 (0.4)	88.7 (2.1)	31.0 (0.3)	78.8 (0.7)	25.8 (0.4)	64.6 (1.0
90	39.7 (0.6)	113 (1.6)	36.1 (0.4)	98.5 (1.6)	31.4 (0.2)	85.5 (1.6)	27.1 (0.3)	74.1 (0.6)
100	41.9 (0.9)	118 (3.8)	37.9 (0.3)	103 (3.3)	35.7 (0.3)	95.5 (0.6)	30.6 (0.4)	82.8 (1.6)

*() - standard deviation

The power of the microwaves applied in this study did not play significant role on the pre-treatment effectiveness. For all the analyzed values of power, comparable amounts of organic substances (COD, protein) were released. The most appropriate values were recorded for the sludge temperature at the level ≥ 70°C. Taking into account the fact that sludge pre-treatment by microwave irradiation requires the delivery of energy, the pre-treatment by microwaves of higher power (1200 W) and resulting in sludge temperature of 70°C was recommended for further experiments.

Anaerobic digestion in continuous mode

As the sludge was undergoing anaerobic digestion, the influence of particular HRT values on the degree of organic matter reduction, biogas production, process stability, higienization, susceptibility to dewatering and characteristics of post-digestion waters was discussed.

Organic matter removal

Firstly, determination of the organic matter removal was started by the application of the shortest HRT value, *ie* 10 days, which was tantamount to 3.72-3.89 kg VS/(m^3·d) of bioreactor organic loading rate (OLR). Under those conditions, VS removal amounted to 27% and 34% for the sludge without pre-treatment and pre-treated sludge respectively. Furthermore, the value of HRT was gradually extended and the influence of sludge pre-treatment on the value of VS removal was analysed. The highest values of VS removal, *ie* 38-40% (untreated sludge) and 49-50% (pre-treated sludge) were achieved when the HRT was extended to 20-30 days, which was associated with an OLR value of 1.24-1.86 kg d.m.s./(m^3·d) (untreated sludge) and 1.30-1.95 kg d.m.s./(m^3·d) (pre-treated sludge). Taking into account the concept of technical stabilization boundary, established for mesophilic conditions at the level of 38-40% [27] of volatile solids removal, it was stipulated that the above condition was met for HRT value above 12 days (pre-treated sludge) and 20 days (sludge without pre-treatment). Figure 6 shows the influence of sludge pre-treatment on the degree of organic matter removal.

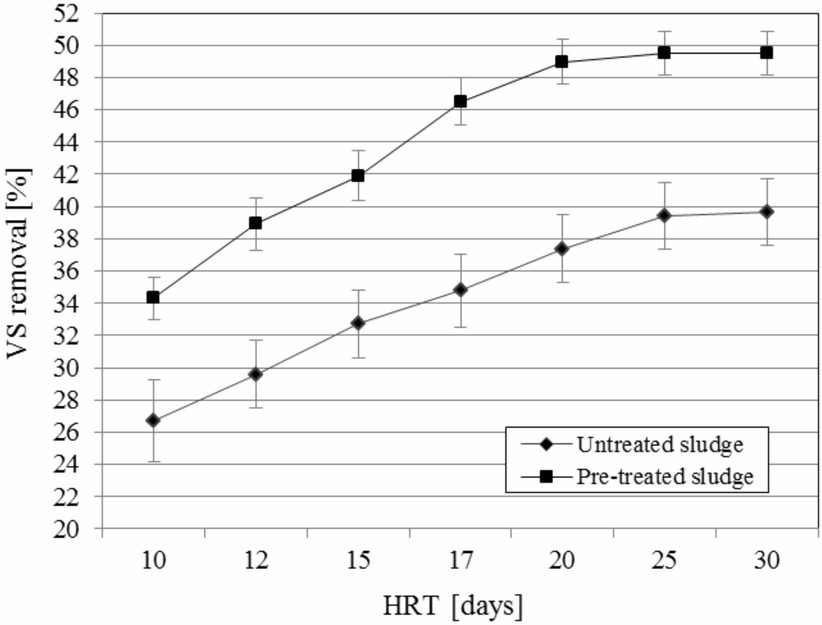

Fig. 6. Influence of sludge pre-treatment on the degree of organic matter removal

Biogas production

Secondly, the biogas production as well as methane content in the biogas generated was recorded.

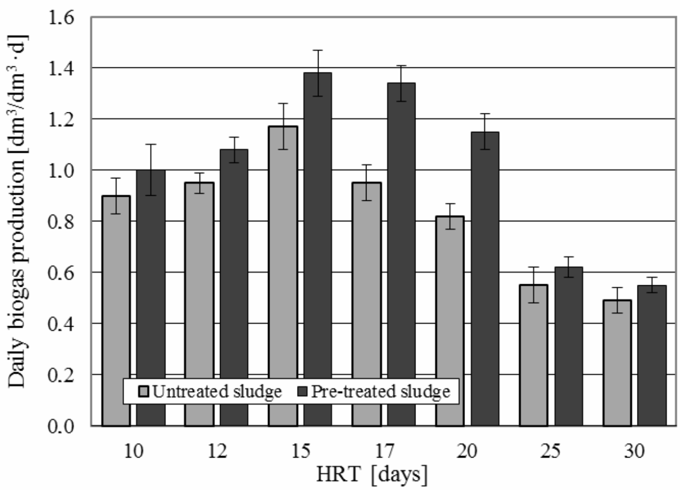

Fig. 7. Influence of sludge pre-treatment on the daily biogas production

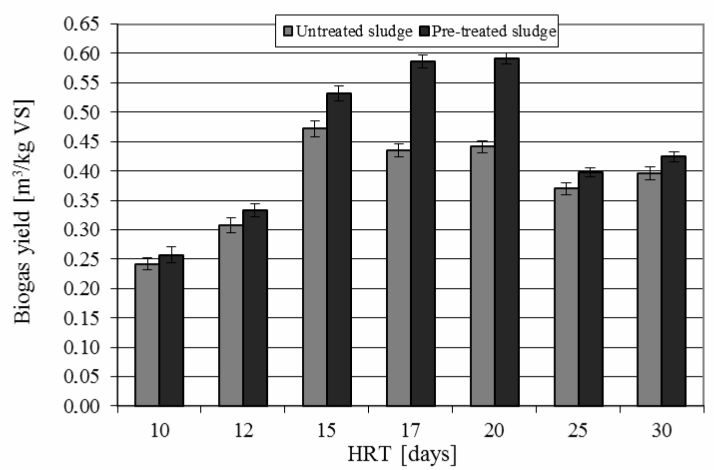

Fig. 8. Influence of sludge pre-treatment on biogas yield

Figures 7 and 8 show the amount of biogas produced for various HRT, expressed in terms of daily production and biogas yield, respectively. For the whole range of HRT analysed, the sludge after microwave pre-treatment generated more biogas, as compared to untreated sludge.

The largest amount of biogas produced, *ie* 0.82-1.17 $dm^3/(dm^3 \cdot d)$ (untreated sludge) and 1.15-1.38 $dm^3/(dm^3 \cdot d)$ (pre-treated sludge) was obtained for HRT in the range of 15-20 days. With respect to untreated sample, the biogas production increased by 18-41%. Under those conditions, biogas yield increased by 13-35%. Further extension of the HRT value (> 20 days) as well as applying the shortest retention times (< 15 days) resulted in a much smaller amount of biogas produced. Moreover, there was no longer apparent effect of microwave exposure on the amount of biogas produced. Sludge pre-treatment by microwave irradiation did not affect the content of CH_4 in biogas generated. For both types of sludge, concentration of CH_4 ranged between 54 and 57% (data not shown).

Stability of anaerobic digestion

Discussing the overall stability of the anaerobic digestion, a primary indicator which influences the biological conversion is pH value. The use of the indicator is based on the fact that pH drop is commonly related to the accumulation of VFAs, which consumes buffer capacity [28, 29]. However, a significant decrease of pH value does not take place until the process collapses and the acidic phase of the process dominates. Taking into account the above facts, a more reliable stability indicator seems to be a volatile fatty acids to total alkalinity (VFA/TA) ratio. If the latter exceeds the threshold of 0.3-0.4, it is believed to have an inhibitive effect on biogas production or can even lead to the collapse of the process in a short term [30, 31]. It can be pointed out that the process exhibited stable properties for the HRT value above 10 days. For the lowest value of HRT (10 days), a significant accumulation of VFA (2135-3944 mg CH_3COOH/dm^3) as well as increased value of the VFA/TA ratio (0.47-0.67) were recorded (Fig. 9).

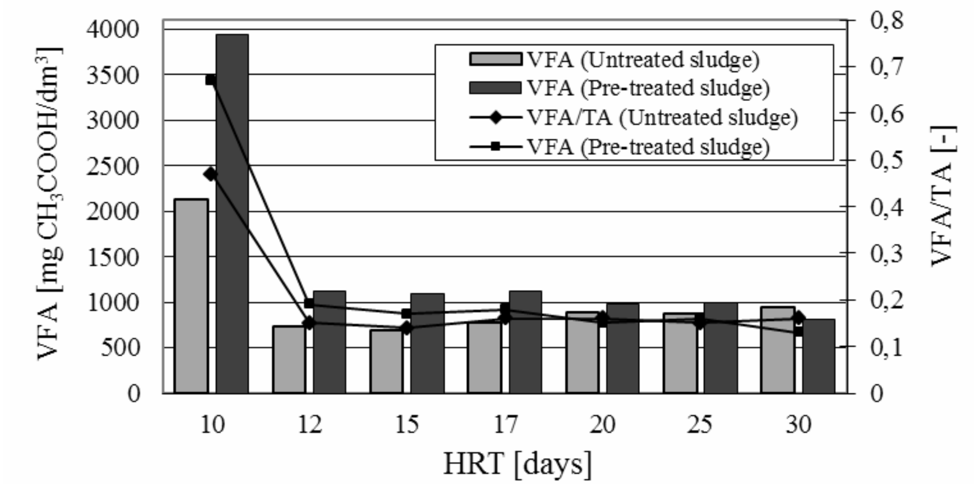

Fig. 9. Influence of sludge pre-treatment on the stability of anaerobic digestion

Sludge dewatering and characteristics of post-digestion waters

During anaerobic digestion about 80% of organic matter, which undergoes biodegradation, is converted into biogas, while further 10% is converted into complex organic substances and the remaining part passes to the post-digestion effluent and is responsible for high COD concentration. What is more, high concentrations of biogenic substances in post-digestion liquids are the results of hydrolytic decomposition of organic nitrogen and phosphorus compounds, and thus in turn cause both ammonia and phosphates release into a post-digestion effluent [32, 33]. The application of pre-treatment before subsequent anaerobic digestion causes further increase of organic and inorganic substances in post-digestion waters. In our studies, the application of microwave irradiation as a pre-treatment step led to about 10% increase of COD concentration in post-digestion waters, compared to waters coming from the process without sludge pre-treatment. In these conditions, the concentration of ammonia-nitrogen and phosphates increased by 6-14% and 4-10% respectively. An increased concentration of organic and inorganic substances in post-digestion waters undergoing pre-treatment influenced also the sludge susceptibility to dewatering in a negative way (Table 5). Such liquors exhibited a negative impact on the wastewater treatment processes, especially the denitrification [34, 35]. Nowadays, the post-digestion liquors are more and more frequently treated separately with the application of biological as well as physical and chemical methods. In our previous studies, the method of liquors treatment - based on struvite precipitation and a subsequent reverse osmosis - has been developed. Depending on the process conditions, the method allows to significantly pre-treat the post-digestion waters before directing them back to the wastewater treatment facility or decrease contaminants' concentration below discharge limits, allowing direct release of permeate to a natural receiver [36, 37].

Table 5

Sludge susceptibility to dewatering and characteristics of post-digestion liquors

Indicator	**HRT [days]**						
	10	**12**	**15**	**17**	**20**	**25**	**30**
Sludge without pre-treatment							
NH_4^+ [mg/dm^3]	1679 (66)*	1680 (25)	1623 (45)	1530 (45)	1553 (51)	1590 (41)	1586 (39)
PO_4^{3-} [mg/dm^3]	630 (19)	610 (30)	565 (18)	520 (17)	585 (20)	576 (34)	545 (27)
SCOD [mg O_2/dm^3]	2250 (291)	2398 (209)	1779 (78)	1717 (110)	1807 (55)	1820 (89)	1684 (32)
CSK [s]	156 (9)	115 (7)	89 (6)	67 (5)	68 (5)	75 (6)	80 (6)
Pre-treated sludge							
NH_4^+ [mg/dm^3]	1775 (30)	1830 (30)	1825 (25)	1736 (43)	1789 (45)	1665 (35)	1628 (41)
PO_4^{3-} [mg/dm^3]	652 (15)	662 (10)	610 (12)	573 (31)	635 (11)	598 (20)	556 (22)
SCOD [mg O_2/dm^3]	2450 (151)	2118 (136)	1942 (78)	1788 (92)	1787 (95)	1812 (115)	1806 (105)
CSK [s]	256 (10)	196 (9)	180 (8)	172 (8)	162 (7)	170 (7)	175 (8)

()* standard deviation

Microbiological quality of digested sludge

Finally, in order to estimate the microbiological risk of sludge usage, *ie* in agriculture, the microbiological quality of the sludge was taken into account. Moreover, as it is commonly known that the process of anaerobic digestion conducted in mesophilic conditions usually does not ensure their complete elimination. In this study, the influence of anaerobic digestion, conducted in mesophilic conditions, as well as combination of microwave pre-treatment and anaerobic digestion was analysed. Mean values of biological indicators are shown in Table 6. On the one hand, the overall number of bacteria decreased by about 17% after anaerobic digestion and additional pre-treatment compared to sludge (dual method), which underwent only mesophilic digestion. On the one hand, the sludge treated by dual method contained no *Escherichia coli* and *Salmonella* spp. whilst, for the sludge after mesophilic digestion without pre-treatment - *Escherichia coli* was still detected. Positive effect of microwave irradiation on the sludge higienization was also confirmed by Dańczuk and Łomotowski [38].

Table 6

Impact of microwave irradiation on sludge microbiological quality

Microbiological parameters	**Digested sludge without pre-treatment**	**Digested sludge after microwave pre-treatment**
Overall number of bacteria [log CFU/g dry weight]	4.33 [0.2]	3.57 [0.3]
Escherichia coli [log CFU/g dry weight]	2.55 [0.2]	0.00
Salmonella spp. [MPN/g wet weight]	2.1 [0.59-7.1]	< 0.03

Conclusions

Microwave pre-treatment by means of microwave irradiation resulted in sludge flocs fragmentation and lysis of bacterial cells, leading to the release of organic and inorganic substances into the liquid sludge phase. The degree of sludge destruction was also confirmed by IR spectroscopy analysis, which showed changes in the type of chemical bonds and groups (*ie* O-H, C-H, N-H, C=O). It was established that the power of microwaves did not have a significant influence on the effectiveness of the process. The highest values of organic matter release were recorded in conditions resulting in sludge temperature of ≥ 70°C. Taking into account the fact that sludge pre-treatment by microwave irradiation requires the delivery of energy, the pre-treatment by microwaves of the highest power (1200 W) was recommended for further experiments, which turned out to be less energy demanding, as compared to the power of 500-1000 W.

Sludge pre-treatment by means of microwave irradiation influenced the effectiveness of subsequent anaerobic digestion, conducted in continuous conditions, in a positive way. The largest amount of biogas was obtained for HRT in the range of 15-20 days. As compared to the sludge which did not undergo pre-treatment, daily biogas production increased by 18-41%. Under those conditions, biogas yield increased by 13-35%. Additionally, pre-treatment of sewage sludge before undergoing the process of anaerobic digestion has a positive effect on the sludge hygienisation. The results showed that mesophilic fermentation process itself did not ensure the required quality of digested sludge. Whilst, the combination of microwave pre-treatment and mesophilic process of anaerobic digestion ensured almost complete elimination of pathogens (*Salmonella* spp., *Escherichia coli*).

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WPŁYW DEZINTEGRACJI MIKROFALOWEJ NA PROCES FERMENTACJI ORAZ HIGIENIZACJI NADMIERNYCH OSADÓW ŚCIEKOWYCH

[1] Instytut Inżynierii Wody i Ścieków, Wydział Inżynierii Środowiska i Energetyki, Politechnika Śląska
[2] Instytut Ochrony i Inżynierii Środowiska, Wydział Nauk o Materiałach i Środowisku
Akademia Techniczno-Humanistyczna w Bielsku-Białej

Abstrakt: Celem badań przedstawionych w artykule było wyznaczenie najkorzystniejszych warunków prowadzenia procesu dezintegracji mikrofalowej (500-1200 W) osadów ściekowych przed ich przeróbką w warunkach beztlenowych (warunki ciągłe). Oceny efektywności procesu mikrofalowej dezintegracji osadów (moc mikrofal, temperatura) dokonano, kierując się stężeniem związków organicznych (ChZT, białka rozpuszczalne) uwolnionych do cieczy nadosadowych, wskaźnikami jakościowymi produkowanego biogazu, stopniem higienizacji oraz podatnością osadów przefermentowanych na odwadnianie. Na podstawie przeprowadzonych badań ustalono, iż moc zastosowanego promieniowania mikrofalowego nie wpłynęła w znaczący sposób na efektywność procesu dezintegracji. Biorąc pod uwagę efektywność oraz zapotrzebowanie energetyczne, ustalono, iż najkorzystniejsze warunki prowadzenia procesu dezintegracji mikrofalowej zapewnia zastosowanie mikrofal o mocy 1200 W oraz temperatury osadów wynoszącej 70°C. Dezintegracja mikrofalowa osadów ściekowych za pomocą promieniowania mikrofalowego wpłynęła korzystnie na efektywność procesu fermentacji metanowej prowadzonego w warunkach ciągłych. Najkorzystniejsze wskaźniki produkowanego biogazu uzyskano dla HRT wynoszącego 15-20 dni. W tych warunkach dobowa i jednostkowa produkcja biogazu wzrosły odpowiednio o 18-41% oraz 13-35%. Zastosowanie dezintegracji mikrofalowej przed procesem mezofilowej fermentacji zapewniło całkowitą eliminację mikroorganizmów patogennych (*Salmonella* spp., *Escherichia coli*).

Słowa kluczowe: promieniowanie mikrofalowe, dezintegracja osadów, fermentacja metanowa, osady nadmierne