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CO-DIGESTION OF SEWAGE SLUDGE AND GLYCEROL FRACTION MIXTURE FACILITATED BY MICROWAVE PRETREATMENT

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Abstract: In the present study, the concept of integrated technological co-digestion system that ensures high effectiveness of glycerine fraction and sewage sludge was developed. The addition of glycerol fraction to sewage sludge influenced positively the degree of organic matter biodegradation and the quantity and quality of biogas produced. Introducing sewage sludge after effective microwave disintegration into feedstock allowed to further improve the effectiveness of the co-digestion process analysed. The co-digestion mixtures performed in conditions ensuring high effectiveness and an appropriate digestion stability (hydraulic retention time (HRT) = 20 days) allowed to increase the methane production by 18–23% and methane yield up to 10% compared to the samples of glycerine and untreated sludge digested in optimal conditions (HRT = 22–24 days). However, it should also be taken into consideration that initial sludge pre-treatment allowed to decrease the HRT value by at least 2 days.

Keywords: co-digestion, glycerol fraction, biodiesel, microwave pre-treatment, sewage sludge

Introduction

The scarcity of fossil fuels, growing emissions of combustion-generated pollutants, and their increasing costs, have made alternative fuel sources more attractive. Biodiesel (fatty acid methyl esters) produced by the process of transesterification of vegetable oils

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or animal fats with methanol are potential substitutes for petroleum-based diesel fuels. Compared to conventional diesel, biodiesel is biodegradable, renewable, non-toxic, and has low pollutant emissions (especially SO_x) [1]. However, biodiesel production generates by-products; including oilcake resulting from the extraction of oil and glycerol fraction generated at the stage of esterification of oil. During the processing of 1000 kg of rape, cake of about 650 kg and about 100 kg glycerol phase are obtained [2]. Oil cakes are mostly utilized as a component of animal feed. Whilst, the research is carried out in the direction of their usage as feedstock in the process of pyrolysis, for the bio-oil and bio-char generation. These substances can be used as substrates for the production of enzymes, antibiotics, etc. [3]. The glycerol fraction, the second by-product, can be assigned to one of three categories based on its purity. Raw glycerol contains up to 63.3% of glycerol, up to 26.8% of water, and approximately 26.7% of methanol. In addition, it contains organic substances (FAME) and potassium and sodium salts. Glycerol of average purity (technical glycerol) contains approximately 80% of glycerol and much lower percentages of water and methanol. Glycerol of the highest purity ($> 99.8\%$) is received in glycerol distillation plants. In the largest biodiesel production plants, methanol is evaporated from the glycerol fraction, and the free fatty acids are purified away, producing technical glycerol of $\sim 80\%$ purity. If technical glycerol is collected and purified up to 99.8%, this generates additional costs connected with energy and materials. In principle, the demand for it is limited because of a large surplus in the market. Thus, new possible uses of technical, *ie* non-purified glycerol are highly desirable [4]. One of the most attractive ones is to use the liquid fuel in various combustion plants. However, glycerol has quite a high auto-ignition temperature (370°C) compared to other liquid fuels. Moreover, when glycerol is heated above 280°C , toxic acrolein is produced [5, 6]. As glycerol is highly flammable and combusts at a higher temperature, a high temperature should be maintained in glycerol combustion processes. Other options include the production of fuel emulsions using raw or technical glycerol. However, the use of this fuel in boilers is currently limited because of its high sulphur content [5, 6]. Another possible applications of glycerol include: pharmaceuticals, cosmetics and plastics production. For example, 1,3-propanediol is produced microbiologically from glycerol. The latter product can substitute toxic ethylene glycol used for polyesters production [7]. However, in order to obtain high quality of glycerol, – depending on the presence of impurities therein, it is necessary to highly pretreat raw glycerol fraction after biodiesel production, which is not always economically justified. In practice, glycerol fraction similarly to rapeseed cakes constitutes very frequently the component of animal feed. Considering its high content of easily biodegradable ingredients (80–90%), anaerobic digestion of glycerol for biogas production constitutes an interesting alternative. However, glycerol direct utilization as the only substrate in the fermentation processes is very limited due to lack of nitrogen, which is necessary to ensure the sufficient activity of microorganisms involved in the biochemical processes. The problem can be solved by glycerol co-digestion with other co-substrates, *eg* waste sewage sludge [8]. However, due to the complex structure of waste activated sludge (WAS), it is difficult to achieve high effectiveness of the sludge undergoing biodegradation in anaerobic conditions. The

problem can be solved by the application of effective sludge pre-treatment, *eg* by microwave irradiation, – which is considered now as very novel method. The sludge contains a high proportion of water and thus easily absorbs microwave irradiation and the mechanism of microwave irradiation includes “thermal” and so-called “non-thermal” effects. The positive effects of sludge pre-treatment by means of microwave irradiation have recently been widely reported in the literature [9–12]. However, the cases mainly focus on the determination of optimal pre-treatment conditions and/or assessment of its advantages over traditional thermal pre-treatment method. The research aimed at subsequent anaerobic digestion of treated sludge together with other co-substrates is very scarce. What is more, according to our knowledge, there is no reports on enhancement of continuous co-digestion process (surplus activated sludge and non-purified glycerol fraction), by the microwave irradiation. The aim of the study was to develop the concept of integrated technological system that ensures high effectiveness of waste glycerol (technical glycerol) and sludge anaerobic co-digestion. The present study is focused on the assessment of microwave irradiation as the effective means of enhancing the anaerobic digestion (AD) effectiveness of the analysed co-digestion mixtures. The anaerobic digestion processes encompassed batch and continuous modes. The purpose of the former process was to determine the optimal proportion of waste glycerol, which can be added to the mesophilic digestion of sludge without significant deterioration of the stability of the process. Whilst, the latter process (continuous mode) allowed to determine the most appropriate parameters of the process, *ie* organic loading of the bioreactor and hydraulic retention time (HRT) of the optimal co-digested mixture.

Materials and methods

Feedstock of anaerobic digestion

Samples of surplus activated sludge (SAS) were taken from an Enhanced Biological Nutrient Removal (EBNR) full-scale municipal sewage treatment plant in Silesia region; Poland. The treatment plant was designed for nutrients removal. With the intention of phosphorous and nitrogen compounds removal, anaerobic, anoxic and aerobic sectors have been distinguished. The plant was designed for a flow of 120 000 m³/day. At present the amount of treated wastewater is about 90 000 m³/day. Solid retention time (SRT) is about 14 days and concentration of mixed liquid suspended solid (MLSS) 4320–4640 mg/dm³. Surplus activated sludge samples were collected immediately after the compaction process (using a belt compactors) (Table 1). The by-product after biodiesel production (technical glycerol) named so “glycerol fraction” in this paper, was used as a co-substrate of anaerobic digestion. The glycerin was obtained from a local biorafinery (Silesian province, Poland). The characteristics of the glycerol fraction is shown in Table 1. Co-digestion trials were inoculated using digested sludge; from the same plant which provided samples of surplus activated sludge (Table 1).

Table 1

Characteristics of co-substrates and inoculum used for anaerobic digestion

Indicator	Unit	Untreated sludge ^a	Pretreated sludge ^b	Digested sludge	Glycerine fraction
pH	[-]	6.4 (0.4)*	6.2 (0.2)	7.3 (0.2)	5.2 (0.3)
Total solids (TS)	[g/dm ³]	51.6 (3.4)	52.9 (2.5)	34.3 (0.4)	783 (7)
Volatile solids (VS)	[g/dm ³]	35.1 (2.7)	36.2 (2.1)	19.3 (0.3)	770 (7)
Soluble chemical oxygen demand (SCOD)	[g/dm ³]	0.27 (0.10)	6.10 (0.45)	1.64 (0.21)	—
C _{org}	[% TSS]	33.0 (0.5)	33.5 (0.4)	31.0 (0.9)	46.7 (0.7)
Total Kjeldahl nitrogen (TKN)	[% TSS]	5.01 (0.36)	4.89 (0.25)	3.27 (0.03)	0.61 (0.01)
NH ₄ ⁺	[mg/dm ³]	15.8 (5.9)	115 (10)	1650 (110)	254 (25)
PO ₄ ³⁻	[mg/dm ³]	144 (35)	695 (25)	240 (60)	—
Volatile fatty acids (VFA)	[mg/dm ³]	83 (20)	125 (25)	870 (26)	—
Alkalinity	[mg CaCO ₃ /dm ³]	1161 (104)	1085 (95)	5845 (346)	—

* () – standard deviation, ^a surplus activated sludge without pretreatment, ^b surplus activated sludge after effective pre-treatment by microwave irradiation [13].

Microwave sludge pre-treatment

The most appropriate technological conditions of sewage sludge pretreatment (exposure time, sludge final temperature, microwave power) as factors ensuring an efficient sludge lysis, has been established previously. The process of sewage sludge disintegration by the microwave irradiation led to the effective destruction of flocks, which was confirmed by an increase in the organic content (SCOD, soluble proteins), mineral substances (NH₄⁺, PO₄³⁻) in the liquid sludge phase as well as an increase in biogas production [13]. Based on our previous studies on microwave excessive sludge disintegration (SASMD), the sludge pretreated exposed to the action of microwaves for 2.0 minutes and resulting in the sludge temperature of about 70°C, – was used in the present study. There was no influence of microwave power applied in the range of 700–1200 W – on the effectiveness of the sludge pretreatment. The microwave power of 1200 W was used in the present study, as it turned out to be the less energy demanding [13, 14].

Anaerobic digestion in batch and continuous mode

Anaerobic digestion (co-digestion) in mesophilic conditions (36 ± 1)°C was performed under batch and continuous conditions. The first of these processes was conducted in order to determine the most favorable proportion of technical glycerol in the co-digestion mixtures (glycerol + sewage sludge); providing a high efficiency and stability of the anaerobic digestion. The content of waste glycerine in co-digestion mixtures amounted to between 1–5% (by weight). During the continuous co-digestion process, the influence of the hydraulic retention time (HRT) of the co-digestion (the most favorable) mixture on the effectiveness and stability of the process was analysed.

The batch tests were performed in 1 dm³ bottles (0.5 dm³ of working volume). The co-digestion mixtures were inoculated with fermented sludge. The proportion of inoculum amounted to 50% of total working volume. Digestion mixtures were enriched with nutrients [15]. Mixtures containing only nutrients and inoculum were used as controls. Assays were performed at (36 ± 1)°C for a period of 25 days. The amount of methane produced was expressed per unit of initial organic matter – volatile solids (VS) – in the feedstock. Methane produced by inocula (control samples) was subtracted from the amount of methane produced in all tested assays. The digestion process in semi-continuous conditions was conducted in two bioreactors with a working volume of 3 dm³. The bioreactors were operated at the following hydraulic retention times (HRT): 16, 18, 20, 22 and 24 days. The bioreactors were kept at a constant temperature of 36°C and their content was mixed periodically – 5 min. in every 2–3 h. Bioreactors were maintained for at least three HRTs before decreasing of HRT value. The kit for measuring the amount of biogas generated included: calibrated cylinder filled with 5% NaCl solution and expansion tanks. The first reservoir allow reading of the amount of biogas produced. The volume of biogas produced was adjusted to standard temperature (0°C) and pressure $1 \cdot 10^5$ Pa (1 atm) and expressed as daily methane production [$\text{dm}^3/(\text{dm}^3 \cdot \text{day})$] and methane yield [m^3/kg of VS_{added}].

Analytical methods

Total solids (TS), volatile solids (VS), ammonia (NH₄⁺), total Kjeldahl nitrogen (TKN), total organic carbon (TOC), phosphates (PO₄³⁻), total VFA and total alkalinity (TA) were determined in triplicate according to the standard methods [16]. The methane content in the biogas produced was determined using a gas analyzer Gas Data GFM 416.

Results and discussion

Anaerobic co-digestion in batch mode

The proper course of the fermentation process depends on a number of technological parameters, while, main objectives of the anaerobic processes, in controlled conditions, are focused on: intensification of organic matter mineralization and biogas production. Co-digestion of sewage sludge together with other organic wastes is one of the option

allowing to increase the efficiency of sludge anaerobic digestion [8]. The characteristics of the co-digestion mixtures before and after digestion is presented in Table 2.

Table 2

Characteristics of co-digestion mixtures before and after fermentation

Co-digested mixtures [%]	Before digestion			After digestion		
	pH [-]	VS [%]	C/N [-]	pH [-]	VS [%]	R _{VS} [%]
SAS (100%)	7.2	2.72 (0.14)	7.73	7.1	1.59 (0.06)	41.6 (1.0)
GF (1%) + SAS (99%)	7.2	3.46 (0.13)	18.5	7.1	1.74 (0.05)	50.0 (1.6)
GF (2%) + SAS (98%)	7.1	4.19 (0.13)	26.5	7.2	1.95 (0.10)	53.5 (1.0)
GF (3%) + SAS (97%)	7.1	4.93 (0.12)	32.7	7.2	2.19 (0.10)	55.5 (1.0)
GF (4%) + SAS (96%)	7.1	5.66 (0.12)	37.7	6.2	2.42 (0.11)	57.3 (1.1)
GF (5%) + SAS (95%)	7.0	6.40 (0.11)	41.7	6.1	2.55 (0.13)	60.2 (1.3)

* () – standard deviation; SAS – surplus activated sludge, GF – glycerine fraction, R_{VS} – organic matter removal.

Firstly, the influence of waste glycerol as a co-substrate on the organic matter removal was analysed. The digestion assay including the only sewage sludge exhibited the organic matter removal of 42%. Gradual increase of waste glycerine in the co-digested feedstock had a positive impact on the degree of organic matter removal. The degrees of organic matter removal amounted to 50–60% (Table 2), which can be attributed to the improvement of C/N ratio in the feedstock, – due to addition of co-substrate of high C/N ratio (Table 1), which also finds confirmation in previous reports [17, 18].

Secondly, the influence of glycerol addition as a co-substrate on the biogas production, was analysed. The average content of methane in the biogas produced was 57% (56–59%) and 66% (64–67%) in case of assays containing only sewage sludge and co-digestion mixtures, respectively (data not shown). Results presented as cumulative methane production are presented in Fig. 1. Even a small addition of waste glycerol (1%) to sewage sludge led to a significant increase of the total biogas production; *ie* 88%. A further increase in the proportion of glycerol in the feedstock undergoing fermentation led to a further increase of the amount of methane generated. The highest amount of methane produced (4.67 dm³) was recorded for the mixture containing 3% of waste glycerol. The latter mixture allowed to receive more than a 4-fold increase of methane production in relation to the sample containing the sludge exclusively. The highest proportions of glycerol (4–5%) caused a significant decrease in methane production (Fig. 1). Fountoulakis et al [19] found that the most favorable proportion of glycerol in co-digestion mixtures, including primary and secondary sludge, amounted to

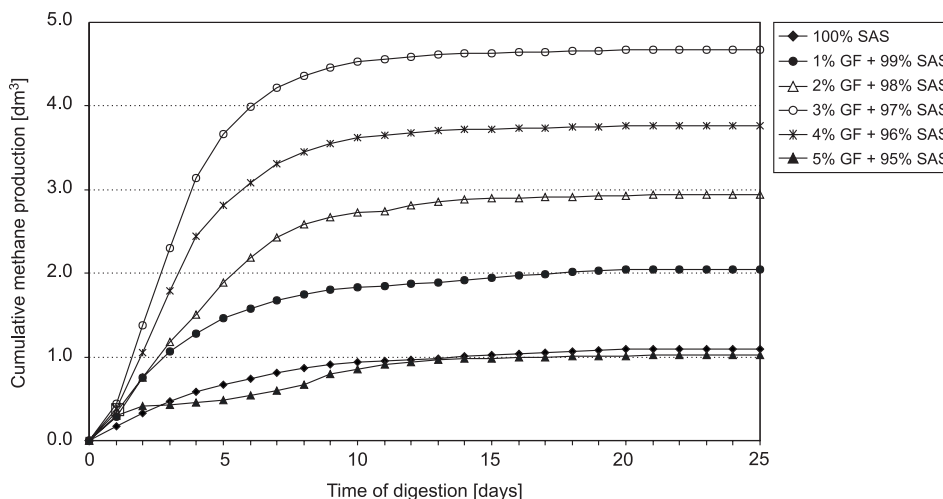


Fig. 1. The effect of glycerol addition to sewage sludge on the value of cumulative methane production

1% (by volume). This share has led to an increase in daily biogas production by over 100%.

Besides, glycerine fraction from biodiesel production, industrial waste with a high content of biodegradable components (*eg* wastes from meat processing or residues from fat separators) turned out to be an appropriate sewage sludge co-substrates [8, 20]. Wastes from this industry pose a threat to the environment, and their disposal is problematic for meat plants. According to research conducted by Lust and Luostarinen [21], the addition of 12.5–25.0% (by volume) of wastes from the meat industry (content and digestive systems, flotation sludge from wastewater treatment and fatty deposits) increased the methane production by 20%. Another study conducted by the same group of authors [22] revealed that the most preferred proportion of fatty wastes added to sewage sludge – amounted to 10% by volume, which was equivalent to 46% VS of the feedstock. In such conditions, more than 100% increase in daily and nearly 70% of the biogas yield were recorded. The addition of stillage (15% by volume) to sewage sludge also led to an 40% increase of biogas production.

Finally, the impact of feedstock enrichment on the anaerobic digestion stability was taken into account. Volatile fatty acids (VFA) accumulation leads to a significant decrease in pH value and thereby can inhibit the methanogenic phase of anaerobic digestion [23]. In the case of sewage sludge fermentation and co-digestion mixtures containing up to 3% of glycerol, there was no significant accumulation of VFA observed and the pH value was at a level ensuring proper course of anaerobic digestion (pH = 7.1–7.3) (Table 2 and 3). For mixtures including in its composition more than 3% of glycerol, concentrations of VFAs (3545–4248 mg/dm³) and the ratio of VFA to alkalinity (0.92–1.31) increased above the value indicating the instability of the methanogenesis process (VFA > 2000 mg/dm³; VFA/Alkalinity > 0.3–0.4) (Table 3) [8, 24, 25]. This was probably strictly connected with a decrease of pH value (6.1–6.2)

Table 3

Stability indicators of the batch co-digestion tests

Co-digested mixtures [%]	Indicator			
	VFA [mg/dm ³]	Alkalinity [mgCaCO ₃ /dm ³]	VFA/TA [-]	NH ₄ ⁺ [mg/dm ³]
SAS (100%)	429 (14)	4081 (24)	0.11	1420 (70)
GF (1%) + SAS (99%)	754 (14)	4610 (17)	0.16	1454 (85)
GF (2%) + SAS (98%)	909 (16)	4773 (67)	0.19	1398 (90)
GF (3%) + SAS (97%)	943 (10)	4825 (56)	0.20	1405 (85)
GF (4%) + SAS (96%)	3545 (110)	3853 (65)	0.92	1338 (90)
GF (5%) + SAS (95%)	4248 (220)	3233 (20)	1.31	1298 (82)

* () – standard deviation; SAS – surplus activated sludge, GF – glycerine fraction; VFA/TA – total volatile fatty acids to total alkalinity.

(Table 2). Although, ammonia released may counteract the decrease of pH values, excessive amounts of ammonia-nitrogen may also inhibit the methanogenic bacteria, and consequently lead to a decrease or even inhibit the production of biogas. The concentration of ammonia nitrogen greater than 3 g/dm³ is generally considered as threshold value; above which the activity of methanogens is negatively affected [1, 8]. However, the concentration of ammonia-nitrogen in all analysed co-digestion mixtures did not exceed the above level (Table 3), thus the ammonia-nitrogen was not responsible for the deterioration of process stability of mixtures including more than 3% of glycerol. The stability indicators are presented in Table 3. Similar situation was observed in other studies. Increasing the excessive co-substrate content influenced the stability of the process in a negative way, *eg* stillage above 50% (by weight) resulted in VFA accumulation (4560–10 200 mg CH₃COOH/dm³). A similar situation was observed after the addition of glycerol waste > 1% vol., which led to a high content of organic matter in post-digestion liquors and decrease of pH values (pH~5) due to accumulation of VFA [19].

Continuous anaerobic digestion of waste glycerine and sewage sludge

In this study, the mixture providing the highest effectiveness in batch co-digestion tests (3% of glycerine fraction + 97% of sludge) and an appropriate stability of the process was tested in continuous conditions. The sewage sludge was additionally treated with microwave irradiation with the aim to enhance the effectiveness of the overall co-digestion process; under the conditions determined during our previous studies [13, 14].

For all analysed co-digestion mixtures of sewage sludge (untreated) and glycerine fraction, reducing redox potential was obtained. These mixtures, with the exception of the shortest of HRTs (16–18 days), were characterized by the values of ORP in a range between –370 mV to –446 mV. The ORP values (–155 mV to –170 mV) recorded for

the HRT in range 16–18 days, were above values considered to be favorable for methane fermentation process (–300 mV) (Table 4) [26]. Mixtures including pretreated sludge instead of untreated sludge exhibited the ORP value between –366 do –435 mV (Table 4); which is in the favorable range for anaerobic digestion. Similarly to the results of ORP, the mixture containing untreated (microwaves) sludge exhibited some disturbance of pH values when digested for the HRT in the range of 16–18 days. The values indicated a significant decrease in pH value (pH 6.2–6.4), which was accompanied by increased concentrations of VFAs (4053–5445 mg CH₃COOH/dm³) (Table 4). For all others retention times tested, pH values fluctuated between 7.1–7.6; *ie* the range most commonly reported for the proper course of anaerobic digestion, *eg* [8, 27].

Table 4

Characteristics of digested mixtures (glycerol + untreated sludge/pretreated sludge) related to different values of HRTs

Parameter	Co-digestion mixture 97% SAS + 3% GF					Co-digestion mixture 97% SASMD + 3% GF				
	HRT [day]									
	16	18	20	22	24	16	18	20	22	24
pH [-]	6.2 (0.1)*	6.4 (0.1)	7.3 (0.2)	7.4 (0.2)	7.5 (0.1)	7.1 (0.1)	7.3 (0.2)	7.5 (0.2)	7.6 (0.1)	7.6 (0.1)
ORP [mV]	–155 (41)	–170 (31)	–420 (25)	–446 (30)	–440 (20)	–366 (30)	–430 (20)	–425 (15)	–426 (20)	–433 (25)
VS [%]	25.8 (1.3)	22.7 (1.1)	20.4 (0.5)	19.7 (1.1)	19.5 (1.1)	22.2 (1.5)	19.1 (0.7)	18.0 (1.2)	18.4 (1.2)	18.7 (0.9)
VFA [mg/dm ³]	5445 (256)	4053 (285)	1225 (65)	1155 (55)	1090 (59)	2555 (166)	2256 (60)	1230 (52)	1180 (52)	1185 (55)
Alkalinity [mg CaCO ₃ /dm ³]	5855 (350)	6235 (280)	6419 (310)	6554 (225)	6550 (360)	6255 (250)	6565 (260)	6690 (305)	6960 (315)	7015 (240)

* () – standard deviation, SAS – surplus activated sludge without pre-treatment, SASMD – SAS after microwave disintegration, GF – glycerine fraction.

Organic matter removal

The most favorable conditions of anaerobic co-digestion (untreated sludge + glycerine fraction) were established. The highest values of organic matter removal (65–66%) were obtained for the HRT of 22–24 days, which corresponded with organic loading of the bioreactor at the level of 2.4–2.6 kg VS/(m³ · day). Figure 2 shows the effect of feedstock retention time in the bioreactor on the degree of organic matter removal. Mixtures containing the same amount of sludge, which was not subjected to the action of microwaves and digested for less than 20 days, resulted in a significant decrease in organic matter removal (56–60%). Under these conditions, a significant increase of the dissolved organic compounds has been observed (COD = 3845–9345 mg O₂/dm³; data not shown). Modification of co-digestion mixtures by introducing pre-treated sludge caused only a slight increase of the organic matter removal, *ie* 67–69% (Fig. 2).

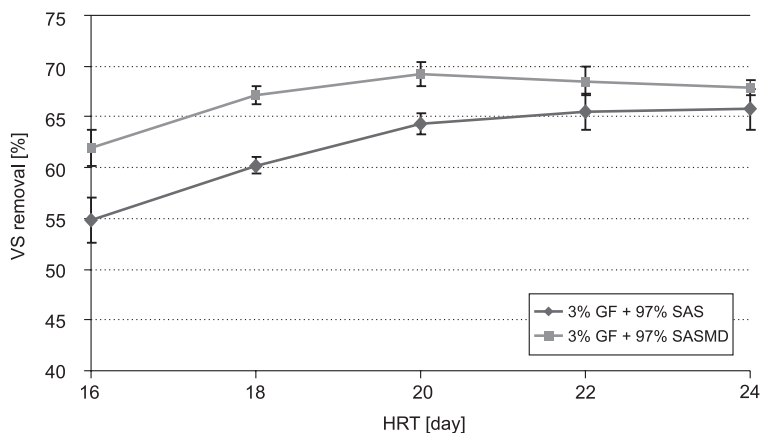


Fig. 2. Impact of HRTs and pre-treatment of feedstock (sewage sludge as a co-substrate) on degree of organic matter removal (SAS – surplus activated sludge without pre-treatment, SASMD – SAS after microwave disintegration, GF – glycerine fraction)

However, it is worth noting that the pre-treatment disintegration of the sludge being a part of co-digestion feedstock enabled to achieve these degrees of organic matter removal (67%) for much shorter retention times, *ie* 18 days. The latter value corresponds with the organic loading of the bioreactor at the level of 3.23 kg VS/(m³ · day).

Biogas production

Secondly, the impact of different values of HRTs on the biogas production was taken into account. The amount of methane produced expressed as its daily methane production and methane yield are presented in Fig. 3 and 4 respectively. The highest values achieved during co-digestion of mixtures including untreated sludge amounting to 2.24 m³/(m³ · day) (methane production) and 0.29 m³/kg VS (methane yield) were recorded for the HRT of 22 days. Further increase in HRT values led to a decrease of methane production indices. Decreasing the HRT value to 18–20 days also allowed to generate lower methane volumes, *ie* 1.51–1.76 (m³/(m³ · day)) and 0.16–0.21 m³/kg VS. Whilst, during the process conducted for the lowest HRT, *ie* 16 days; a clear inhibition of the biogas (methane) production was noticed (0.96 m³/(m³ · day); 0.09 m³/kg VS (Fig. 3 and 4); and these values are in the range obtained during the fermentation of sludge without the addition of a co-substrate reported previously [13]. This inhibition was probably caused by the accumulation of VFA (> 5445 mg CH₃COOH/dm³) and higher organic matter loading of bioreactor's chamber, *ie* 3.57 kg VS/(m³ · day) (Table 4).

Implementing the sludge pre-treated by microwaves influenced positively the methane production process. The highest values of methane production were recorded for the HRT of 18–22 days and amounted to about 2.60–2.70 m³/(m³ · day) (methane production) and 0.28–0.33 m³/kg VS (methane yield) (Fig. 3 and 4). These values

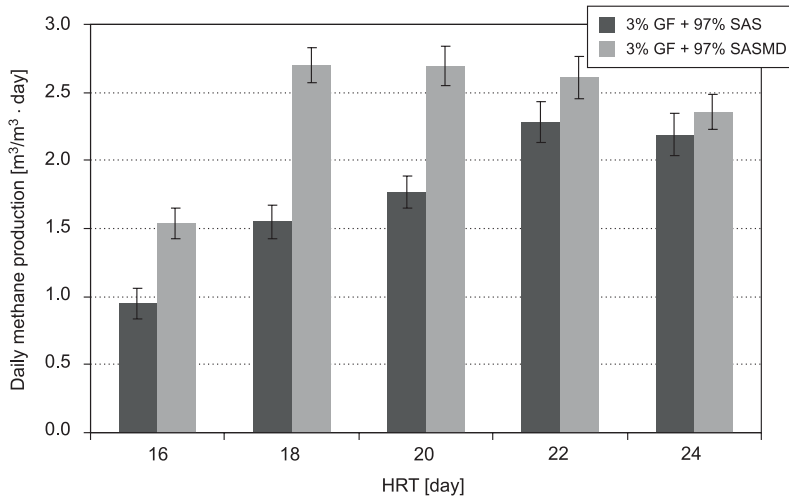


Fig. 3. Influence of hydraulic retention time of feedstock (glycerine fraction + untreated sludge; glycerine fraction + pretreated sludge) on daily methane production (SAS – surplus activated sludge, SASMD – SAS after microwave disintegration, GF – glycerine fraction)

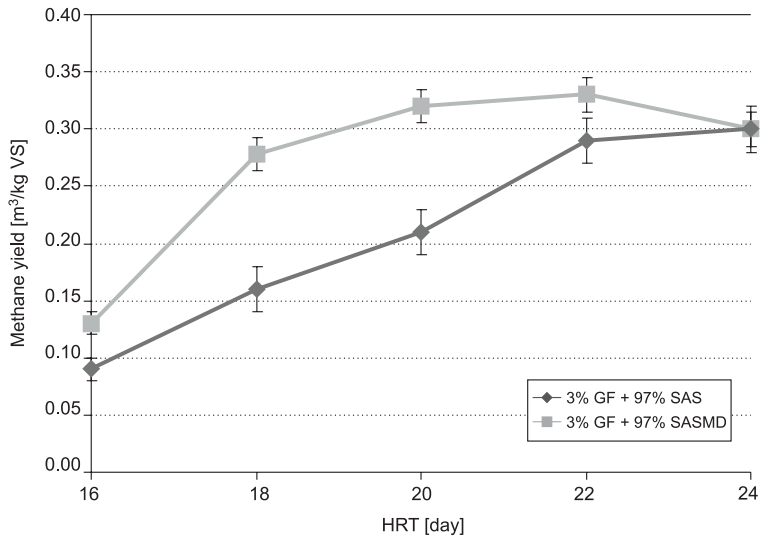


Fig. 4. Influence of hydraulic retention time of feedstock (glycerine fraction + untreated sludge; glycerine fraction + pretreated sludge) on methane yields (SAS – surplus activated sludge, SASMD – SAS after microwave disintegration, GF – glycerine fraction)

represent 52–74% (HRT = 18–20 days) and 14–15% (HRT = 22 days) increase of methane indices related to equivalent values obtained for mixture without pre-treated sludge. It is also worth mentioning that sludge pre-treatment allowed to achieve a high

methane production for shorter values of HRTs; compared to co-digestion mixtures containing the sludge without previous treatment.

Stability indicators of co-digestion

Finally, the influence of sludge microwave pretreatment on the stability of anaerobic digestion was taken into consideration. It was established that mixtures of glycerine and both types of sludge (untreated/pretreated) exhibited stable conditions for the HRT of > 18 days (VFA = 1090–1230 mg CH₃COOH/dm³; VFA/Alkalinity = 0.16–0.19) (Table 4). These values of stability indices are in the range ensuring the stable course of the digestion processes [24, 25]. In case of lower HRTs values, a significant accumulation of VFA (2255–5445 mg CH₃COOH/dm³) and increased the ratio of VFA/Alkalinity (0.41–0.93) were recorded. This is especially evident for the mixtures including glycerin fraction and sludge without pretreatment (Table 4).

Conclusions

The addition of glycerine fraction to sewage sludge turned out to be an effective way of increasing the mesophilic digestion of sewage sludge. Introducing sewage sludge after effective microwave disintegration into feedstock allowed to further improve the effectiveness of the co-digestion process analysed. The most favorable value of the biomass retention time (HRT) amounted 20 days; which allowed to achieve about 18–23% higher value of methane production and up to 10% higher methane yield compared to the values recorded in the conditions of the most appropriate digestion of glycerine and sludge without pre-treatment (HRT 22–24 days).

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References

- [1] Chen H, Peng B, Wang D, Wang J. *Front Chem Eng China*. 2007;1:11-15. DOI: 10.1007/s11705-007-0003-y.
- [2] Pińkowska H., Wolak P., Oliveros E. *Biomass Bioenerg.* 2014;64:50-61. DOI: 10.1016/j.biombioe.2014.03.028.
- [3] Özçimen D, Karaosmanoglu F. *Renew Energy*. 2004;29:779-787. DOI:10.1016/j.renene.2003.09.006.
- [4] Van Gerpen J. *Biodiesel Production Technology*. NREL/SR-510-36244, Golden, Colorado. <http://www.osti.gov/bridge> (2004 05).
- [5] DIPPRProject 801 Full version evaluated standard thermophysical property values. Design Institute for Physical Properties, Department of Chemical Engineering, Brogham Young University, Provo, Utah. 2005. <https://www.aiche.org/dippr/projects/801>
- [6] EPA/635/R-03/003. Toxicological review of acrolein. CAS No. 107-02-8. 2003. https://cfpub.epa.gov/ncea/iris/iris_documents/documents/toxreviews/0364tr.pdf
- [7] López JAS, Santos MAM, Pérez AFC, Martín AM. *Bioresour. Technol.* 2009;100:5609-5615. DOI: 10.1016/j.biortech.2009.06.017.

- [8] Mata-Alvarez J., Macé S., Lladrés P. *Bioresour Technol.* 2000; 74: 3-16.
DOI: 10.1016/S0960-8524(00)00023-7.
- [9] Ahn J-H, Shin SG, Hwang S. *Chem Eng J.* 2009;153(1-3):145-150. DOI: 10.1016/j.cej.2009.06.032.
- [10] Eskicioglu C, Kennedy KJ, Droste RL. *Desalination.* 2009;248(1-3):279-285.
DOI: 10.1016/j.desal.2008.05.066.
- [11] Doğan I, Sanin FD. *Water Res.* 2009;43(8):2139-2148. DOI: 10.1016/j.watres.2009.02.023.
- [12] Eskicioglu C, Kennedy KJ, Droste RL. *Water Res.* 2006;40(20):3725-3736.
DOI: 10.1016/j.watres.2006.08.017.
- [13] Bohdziewicz J, Kuglarz M, Grübel K. *Ecol Chem Eng S.* 2014;21(3):447-464.
DOI: 10.2478/eces-2014-0033.
- [14] Kuglarz M, Karakashev D, Angelidaki J. *Bioresour Technol.* 2013;134:290-297.
DOI: 10.1016/j.biortech.2013.02.001.
- [15] Angelidaki I, Alves M, Bolzonella D, Borzacconi L, Campos L, Guwy A, et al. *Water Sci Technol.* 2009;59:927-934. DOI: 10.2166/wst.2009.040.
- [16] APHA, 1995. *Standard Methods for the Examination of Water and Wastewater*, 19th ed. American Public Health Association, New York, USA.
- [17] Ward AJ, Hobbs P, Holliman PJ, Jones DL. *Bioresour Technol.* 2008;99:7928-7940.
DOI: 10.1016/j.biortech.2008.02.044.
- [18] Panichnumsin P, Nopharatana A, Ahring B, Chairasert P. *Biomass Bioener.* 2010;34:1117-1124.
DOI: 10.1016/j.biombioe.2010.02.018.
- [19] Fountoulakis MS, Petousi I, Manios T. *Waste Manage.* 2010;30:1849-1853.
DOI:10.1016/j.wasman.2010.04.011.
- [20] Alatríste-Mondragón F, Samar P, Cox HHJ, Ahring BK, Iranpour R. *Water Environ Res.* 2006;78(6):607-635. DOI: 10.2175/106143006X111673.
- [21] Lust S, Luostarinen S. *Bioresour Technol.* 2010;101:2657-2664. DOI: 10.1016/j.biortech.2009.10.071.
- [22] Luostarinen S, Luste S, Sillanpää M. *Bioresour Technol.* 2009;100(1):79-85.
DOI: 10.1016/j.biortech.2008.06.029.
- [23] Monou M, Kythreotou N, Fatta D, Smith SR. *Waste Manage.* 2009;29(2):712-20.
DOI: 10.1016/j.wasman.2008.06.013.
- [24] Boe K. *Online monitoring and control of the biogas process (Ph.D. Thesis)*. Copenhagen: Technical University of Denmark, 2006.
<https://pdfs.semanticscholar.org/255d/7714f4748cc82e0e0cee89f51893f8651c77.pdf>.
- [25] Chen Y, Cheng JJ, Creamer KS. *Bioresour Technol.* 2008;10(99):4044-4064.
DOI: 10.1016/j.biortech.2007.01.057
- [26] Miyamoto K. *Renewable biological systems for alternative sustainable energy production*. (Chapter 4). Food and Agriculture Organization of the United Nations. 1997. Rome.
[http://www.fao.org/docrep/w7241e/w7241e0f.htm#chapter4methane production](http://www.fao.org/docrep/w7241e/w7241e0f.htm#chapter4methane%20production)
- [27] Liu C-F, Yuan X-Z, Zeng G-M, Li W-W, Li J. *Bioresour Technol.* 2008;99(4):882-888.
DOI: 10.1016/j.biortech.2007.01.013.

KOFERMENTACJA MIESZANINY OSADÓW ŚCIEKOWYCH I FRAKCJI GLICERYNOWEJ WSPOMAGANA PROMIENIOWANIEM MIKROFALOWYM

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Abstrakt: Celem prowadzonych badań było opracowanie koncepcji zintegrowanego układu technologicznego, który zapewniłby wysoką efektywność kofermentacji produktów ubocznych pochodzących z wytwarzania biodiesla i osadów ściekowych. Dodatek frakcji glicerynowej do osadów ściekowych wpłynął korzystnie na stopień biodegradacji materii organicznej oraz na ilość i jakość wydzielanego biogazu. Zastosowanie jako

wsadu bioreaktora osadów ściekowych po dezintegracji mikrofalowej pozwoliło na dalszą poprawę efektywności analizowanego procesu kofermentacji. W odniesieniu do najkorzystniejszych warunków fermentacji mieszaniny osadów ściekowych nie poddawanych działaniu promieniowania mikrofalowego oraz frakcji glicerynowej (HRT = 22–24 dni) mieszanina kofermentacyjna zawierająca w swym składzie osady po dezintegracji mikrofalowej (HRT = 20 dni) wygenerowała 18–23% oraz do 10% więcej metanu w przeliczeniu odpowiednio na dobę oraz jednostkową produkcję metanu. Ponadto, zastosowanie wstępnej obróbki mikrofalowej pozwoliło na skrócenie czasu zatrzymania mieszaniny kofermentacyjnej w komorze bioreaktora o co najmniej 2 dni.

Słowa kluczowe: kofermentacja, frakcja glicerynowa, biodiesel, osady ściekowe, promieniowanie mikrofalowe