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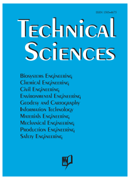

THE EFFECT OF THE EXCESS SLUDGE PRETREATMENT ON BIOGAS PRODUCTIVITY

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Key words: biogas production, sludge pretreatment, activated sludge, aerobic granular sludge, ultrasound disintegration, homogenization.

Abstract

To intensify biogas production during anaerobic stabilization of organic matter in sludge, pretreatment is applied. The effect of pretreatment of excess activated sludge (AS) and excess aerobic granular sludge (GS) on biogas productivity (BP) and composition was investigated. The sludge was pretreated with homogenization (6,500 rpm for 0.5 min ($H_{0.5}$) and 1 min ($H_{1.0}$)) or ultrasound disintegration at 20 kHz (50% amplitude for 2 min ($D_{50\%\_2.0}$) and 4 min ($D_{50\%\_4.0}$), and 100% amplitude for 4 min ($D_{100\%\_4.0}$)). BP of AS of GS without pretreatment was 603.3±5 dm^3/kg TS (793.4±7 dm^3/kg VS); that was 200.6±4 dm^3/kg TS (480.8±6 dm^3/kg VS). With disintegration, the BP of AS increased by 7.8% (650.4±10 dm^3/kg TS) ($D_{50\%\_2.0}$) and 16.1% (700.6±11 dm^3/kg TS) ($D_{100\%\_4.0}$), and that of GS increased by 7.0% (214.0±5 dm^3/kg TS) ($D_{50\%\_2.0}$) and 16.0% (232.8±5 dm^3/kg TS) ($D_{100\%\_4.0}$). With homogenization, BP increased by 2.0-3.0% (AS) and 1.6-3.2% (GS).

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Introduction

Currently, the majority of wastewater treatment plants are operated with activated sludge, which is in the form of flocs. Recent studies that aimed to improve the process of wastewater treatment by modifying the activated sludge have led to development of aerobic granular sludge technology. Aerobic granular sludge, regarded as one of the most promising biotechnologies for municipal wastewater treatment plants, is a specific type of self-immobilized biomass. Granules are densely packed with heterotrophic and autotrophic microorganisms. They have layers with different substrate and aerobic conditions in their structure, providing a broad range of metabolic processes that can occur simultaneously (LIU, TAY 2004). Such a granule structure allows higher resistance to load fluctuations and makes it possible to abandon multi-chamber reactors and secondary settlers, and allows lower energy consumption (SŁAWIŃSKI 2016). Granules have better settling properties than activated sludge; their sludge volumetric index is about 50 cm^3/g MLSS. Granules can be cultured at loadings ranging from 2.5 to 15.0 kg COD/(m^3·d) (CYDZIK-KWIATKOWSKA, ZIELIŃSKA 2011).

Other differences between aerobic granules and activated sludge include the much longer sludge age of granules (BEUN et al. 1999) in comparison with activated sludge (CYDZIK-KWIATKOWSKA et al. 2012) that results in smaller contribution of organic matter to the dry matter content of biomass. In addition, the operational conditions in reactors with granular biomass cause larger amounts of extracellular polymeric substances (EPS) to be produced by granules than by activated sludge (RUSANOWSKA et al. 2019). These properties could make the granules more difficult to degrade than activated sludge.

Biological aerobic and anaerobic (methane fermentation) stabilization are most often conducted methods of sewage sludge stabilization. In the case of methane fermentation, a measurable effect of the process is the biogas yield. To intensify biogas production, a pretreatment step is used. Proper selection of pretreatment methods has an important effect on the efficiency of fermentation and thus on the composition of the biogas. In general, methods of sludge pretreatment can be classified as mechanical and non-mechanical. Mechanical treatment causes grinding or shearing of the solid particles in substrates, resulting in the release of cellular compounds and enlarging the specific surface area of the substrate (CARRERE et al. 2010). The mechanical methods use shear forces or pressure changes, e.g., a mechanical or pressure homogenizer. Non-mechanical treatment can be divided into physical, chemical, biological and mixed methods. Physical methods include, for example, disintegration with ultrasound, thermal treatment using both high (greater than 110°C) and low (lower than 110°C) temperatures or treatment with detergents (ARIUNBAATAR et al. 2014). Thermal treatment is one of the most studied methods and it is used on an industrial scale. Appropriately high temperatures eliminate pathogens, improve dewatering ability and reduce digestate viscosity (VAL DEL RIO et al. 2011). Chemical methods are based on the usage of alkaline solutions, acids or preliminary oxidation (e.g. ozone treatment). They are used to improve the rate of hydrolysis (WANG et al. 2011). Biological methods include the use of single enzymes or their mixtures. Mixed methods include, among others, thermo-chemical treatment, thermo-mechanical treatment or steam explosion with the use of pressure (MONTGOMERY, BOCHMANN 2014). Pretreatment releases organic compounds from microbial cells, thereby increasing the concentration of dissolved organic compounds that are accessible to microorganisms.

Although many studies have investigated the biogas productivity of activated sludge and pretreatment methods for improving this productivity, little research has been done on the biogas productivity of aerobic granular sludge. Therefore, the present study compared the biogas productivity of activated sludge (AS) and aerobic granules (GS). In addition, the effect of pretreatment methods (homogenization or ultrasound disintegration) on the productivity and composition of the biogas produced with both kinds of excess sludge was investigated.

Materials and Methods

Substrates used in the experiment

Two kinds of sludges were used in the study: excess activated sludge (AS), and excess aerobic granular sludge (GS). AS was taken from a mechanical-biological municipal wastewater treatment plant (WWTP) with a maximum capacity of 60,000 m^3/d (north-east of Poland). GS was taken from a laboratory reactor (GSBR) fed with municipal wastewater and operated at the Department of Environmental Biotechnology, UWM in Olsztyn (CYDZIK-KWIATKOWSKA et al. 2017). Characteristics of the sludges are given in Table 1.

Table 1

Characteristics of AS, GS and the inoculum

Indicator		AS	GS	Inoculum
Total solids	%	2.24	8.72	1.52
Moisture	%	97.74	91.28	98.48
Volatile solids	%	1.70	3.64	1.05
	% TS	76.04	41.73	69.11
Ash	%	0.54	5.08	0.47
	% TS	23.96	58.27	30.89

Sludge pretreatment

Homogenization and ultrasound disintegration were used as sludge pretreatment methods. Homogenization was carried out with a T 25 basic ULTRA-TURRAX®, IKA® at 6,500 rpm and at a time of 0.5 min ($H_{0.5}$) and 1 min ($H_{1.0}$). Ultrasound disintegration (20 kHz) was carried out with a Sonics Vibra Cell® at 50% amplitude for 2 min ($D_{50\%\_2.0}$) and 4 min ($D_{50\%\_4.0}$), and 100% amplitude for 4 min ($D_{100\%\_4.0}$).

Experimental design (GP_{21})

To analyze the biogas potential with GP_{21} respirometric test, the following samples of the excess sludges (AS, GS) were prepared:

– AS and GS without pretreatment;

– AS and GS after homogenization at $H_{0.5}$ and $H_{1.0}$;

– AS and GS after ultrasound disintegration ($D_{50\%\_2.0}$, $D_{50\%\_4.0}$ and $D_{100\%\_4.0}$).

The biogas production potential of the sludge was determined during 21 days in triplicate (for each sludge sample) in batch assays in glass bottles (OxiTop® Control AN6/AN12), according to HEERENKLANGE and STEGMANN (2005). 100 g of the inoculum was added to each OxiTop bottle along with a sludge sample. As the inoculum, fermented sludge from the closed mesophilic fermentation chambers in above-mentioned WWTP was used (Tab. 1).

To assure a starting load of ca. 5 g VS/dm^3 (kg VS/m^3), the doses of each sewage sample were calculated, taking into account their contents of total solids and volatile solids. This dosage allowed for complete organic matter biodegradation.

Three bottles with the inoculum were incubated under the same conditions to determine its biogas potential. Finally, the biogas production of the inoculum alone was subtracted from the total production of the sludge and inoculum combined. Before starting measurements, each bottle was flushed with N_2 and the lateral connections of the bottles were sealed with rubber stoppers. The contents of the bottles were manually mixed. Each bottle possessed its own head that measured and recorded pressure changes in the bottle during 21 days of fermentation at 36±1°C in a thermostatic incubator. The pressure changes were caused by formation of biogas during fermentation, and were used to calculate the volume of biogas that was produced based on the ideal gas law.

Analytical methods and calculations

The analysis of TS and VS in sludge samples were measured according to APHA (1992). The percentage of CH_4 and CO_2 in the biogas was measured in the head space of the OxiTop bottles using a GA200+ automatic analyzer (Geotechnical Instruments). Biogas production can be assumed to follow pseudo first-order kinetics and can be described with this equation 1.:

$$C_{t;\mathrm{biogas}} = C_{0;\mathrm{biogas}} \cdot \left(1 - e^{k_{\mathrm{biogas}} \cdot t}\right) \tag{1}$$

where:

$C_{t;\mathrm{biogas}}$ [dm^3/kg TS; dm^3/kg VS] – is the cumulative biogas yield at digestion time t (days),

$C_{0;\mathrm{biogas}}$ [dm^3/kg TS; dm^3/kg VS] – is the maximal biogas yield,

k_{biogas} [d^{-1}] – is the kinetic coefficient of biogas production.

The values of $C_{0;\mathrm{biogas}}$, and k_{biogas} were obtained by non-linear regression analysis with Statistica software, version 10.0 (StatSoft). To determine the fit of the model to the data, the coefficient of determination was calculated (R^2).

Differences between samples were tested for significance by using t test, $p < 0.05$ was considered significant.

Results and Discussion

Figure 1 shows changes in biogas productivity from the sludges without pretreatment during 21-day anaerobic respirometric tests. It was shown that biogas production was explained well by a first-order kinetic model (a high degree of fit between the experimental data and the model; R^2 was 0.97-0.99). The biogas production from AS was much higher (statistically significant, $p<0.05$) (603.3±5 dm^3/kg TS; 793.4±7 dm^3/kg VS) than from GS (200.6±4 dm^3/kg TS;

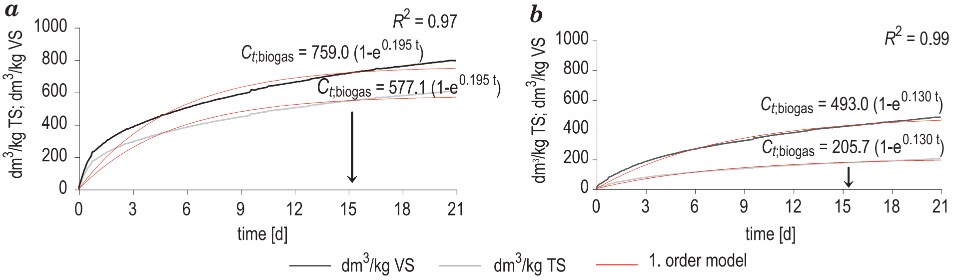

Fig. 1. Biogas productivity of AS (a) and GS (b) without pretreatment; the arrow shows the time after which 90% of the total amount of biogas was produced

480.8±6 dm^3/kg VS). About 90% of the total biogas production was achieved by the 15-16 day of the measurement. After this time, only a small amount of biogas was produced.

Sewage sludge generated during wastewater treatment with activated sludge is characterized by a high content of organic matter of about 70-80% of TS. Organic matter content in the excess activated sludge used in the present study was ca. 76%, and that of the excess granular sludge was ca. 42%. It is assumed that if the organic substrate has a higher content of organic matter (measured as VS in TS) and a greater biodegradability, it can result in higher biogas production. However, not only the content of organic matter but also its composition (e.g. carbohydrate, lipids, protein or fibre content) affects the effectiveness of anaerobic degradation. BERNAT et al. (2017) compared biogas potential of excess activated sludge (VS/TS ratio of ca. 0.76) and aerobic granular sludge (VS/TS ratio of ca. 0.65). The biogas productivity obtained by the authors were 320-410 dm^3/kg TS (ca. 550 dm^3/kg VS) with the excess granular sludge and ca. 830 dm^3/kg TS (1200 dm^3/kg VS) with the excess activated sludge. VAL DEL RIO et al. (2011, 2013) showed that aerobic granular sludge from a pilot plant with SBR fed with the liquid fraction of pig slurry had biogas productivity of 208±51 dm^3 CH_4/kg VS (ca. 350 dm^3 of biogas/kg VS, with the assumption of 60% of methane content). This biogas productivity with granular sludge was lower than that obtained by BERNAT et al. (2017) and that from the present study.

The composition and characteristics of granular sludge differed from those of activated sludge. BERNAT et al. (2017) found high content of lignocellulosic substances (hard-to-biodegrade lignin comprised ca. 54% of fibrous materials) in GS that may have influenced the potential of biogas production. Detailed characteristics of organic matter in the excess sludge used in the present study was not performed, but it could be assumed that the differences in biogas productivity of AS and GS resulted form different composition of organic matter.

In the next step of the experiment, both kinds of sludge were pretreated with homogenization or ultrasound disintegration before measurements of biogas productivity. Changes in the biogas productivity from the sludges after homogenization during 21-day anaerobic respirometric tests are shown in Figure 2. Biogas productivity with AS after 0.5 min of homogenization was 612.9±5 dm^3/kg TS (806.1±7 dm^3/kg VS), and remained on the similar lever of 617.7±7 dm^3/kg TS (812.3±8 dm^3/kg VS) after 1 min of this pretreatment. In comparison to non-pretreated AS, biogas productivity increased by 1.6% and 2.4% (but statistically insignificant, $p > 0.05$), respectively at $H_{0.5}$ and $H_{1.0}$. Biogas productivity with GS was 205.2±5 dm^3/kg TS (491.7±7 dm^3/kg VS) at $H_{0.5}$, and 207.1±5 dm^3/kg TS (496.2±7 dm^3/kg VS) at $H_{1.0}$. These results were comparable to biogas productivity of GS without pretreatment; an increase of only 2-3% was observed (statistically insignificant, $p > 0.05$).

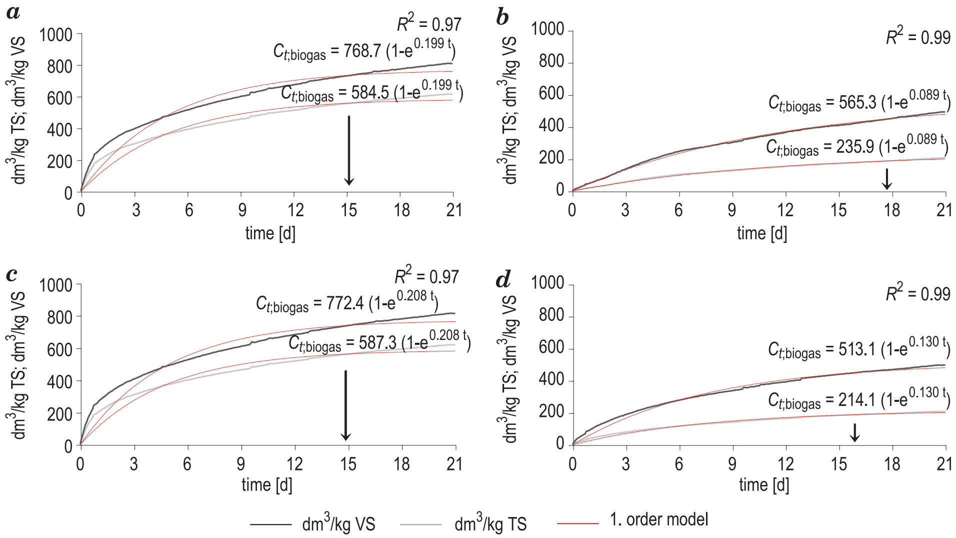

Fig. 2. Biogas productivity of the sludge after homogenization; the arrow shows the time after which 90% of the total amount of biogas was produced: *a* – AS_H$_{0.5}$, *b* – GS_H$_{0,5}$, *c* – AS_H$_{1.0}$, *d* – GS–H$_{1.0}$

After pretreatment with D$_{50\%\_2.0}$, the biogas productivity with AS was 650.4±10 dm^3/kg TS (855.4±12 dm^3/kg VS), and with D$_{50\%\_4.0}$ this productivity increased to 673.1±10 dm^3/kg TS (885.1±12 dm^3/kg VS). With D$_{100\%\_4.0}$, the biogas productivity was the highest – 700.6±11 dm^3/kg TS (921.3±13 dm^3/kg VS). In comparison to AS without pretreatment, there was statistically significant ($p < 0.05$) increase in biogas productivity by 7.8% (D$_{50\%\_2.0}$), 11.6% (D$_{50\%\_4.0}$) and 16.1% (D$_{100\%\_4.0}$). Similar percentage increases (statistically significant ($p < 0.05$) increase in comparison to GS without pretreatment) were obtained when GS was used as a substrate for measurement of biogas productivity, however, the values of biogas productivity were much lower than with AS (214.0±5 dm^3/kg TS; 512.9±7 dm^3/kg VS with D$_{50\%\_2.0}$; 228.2±5 dm^3/kg TS; 546.9±7 dm^3/kg VS with D$_{50\%\_4.0}$ and 232.8±5 dm^3/kg TS; 558.0±7 dm^3/kg VS with D$_{100\%\_4.0}$) (Fig. 3).

Kinetic parameters of the biogas production with both kinds of sludge without pretreatment and after two pretreatment methods are summarized in Table 2 and 3. Kinetic coefficients of biogas production (k_{biogas}) that describe biogas productivity and were determined on the basis of first-model equation were 0.195 d^{-1} and 0.130 d^{-1} for AS and GS, respectively. The rate of biogas productivity (r_{biogas}) with AS was 112.5 dm^3/(kg TS·d) (148.0 dm^3/(kg VS·d)), whereas with GS, r_{biogas} was almost an order of magnitude lower – 26.7 dm^3/(kg TS·d) (64.1 dm^3/(kg VS·d)). In the case of both sludges, homogenization affected neither k_{biogas} nor the methane content in the biogas that was on a similar level as in the case of non-pretreated sludge. Ultrasound disintegration increased

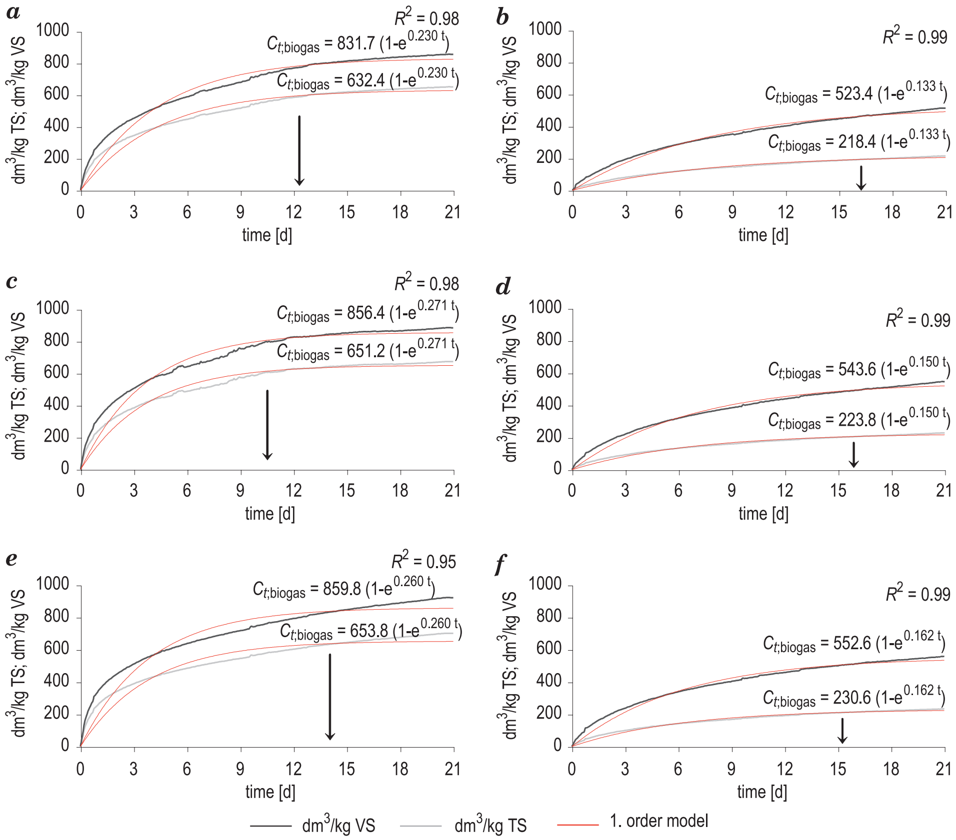

Fig. 3. Biogas productivity of the sludge after disintegration; the arrow shows the time after which 90% of the total amount of biogas was produced: *a* – AS_D$_{50\%\_2.0}$, *b* – GS_D$_{50\%\_2.0}$, *c* – AS_D$_{50\%\_4.0}$, *d* – GS_D$_{50\%\_4.0}$, *e* – AS_D$_{100\%\_4.0}$, *f* – GS_D$_{100\%\_4.0}$

kinetic parameters of the biogas production and methane content more than homogenization. When AS was pretreated by disintegration, k_{biogas} increased in comparison to AS without pretreatment from 0.195 d^{-1} (AS) to 0.230 d^{-1} and to 0.271 d^{-1} (AS after disintegration). Methane content in the biogas also increased and was 64.4% with D$_{50\%\_2.0}$, 65.4% with D$_{50\%\_4.0}$ and 66.6% with D$_{100\%\_4.0}$.

Disintegration of GS caused that k_{biogas} increased in comparison to GS without pretreatment (0.130 d^{-1}), only with D$_{50\%\_4.0}$ (0.153 d^{-1}), and with D$_{100\%\_4.0}$ (0.162 d^{-1}). Methane content in the biogas increased to ca. 62%.

Many studies have investigated the biogas productivity of activated sludge and the effect of pretreatment step on improving this productivity. For example, BOUGRIER et al. (2007) reported that biogas production of activated sludge was ca. 425 dm^3/kg VS which was lower than the values presented by TCHOBANOGLOUS et al. (2003) (500-750 dm^3/kg VS) and obtained in the study of BERNAT

Table 2

Kinetic parameters of biogas production from AS

Excess activated sludge		*WP	$H_{0.5}$	$H_{1.0}$	$D_{50\%\_2.0}$	$D_{50\%\_4.0}$	$D_{100\%\_4}$
Maximal biogas productivity (experimental data)	dm^3/kg TS	603.3±5	612.9±5	617.7±7	650.4±10	673.1±10	700.6±11
	dm^3/kg VS	793.4±7	806.1±7	812.3±8	855.4±12	885.1±12	921.3±13
$C_{t;\text{biogas}}$	dm^3/kg TS	577.1	584.5	587.3	632.4	651.2	653.8
	dm^3/kg VS	759.0	768.3	772.4	831.7	856.4	859.8
r_{biogas}	dm^3/(kg TS·d)	112.5	116.3	122.2	145.5	176.5	170.0
	dm^3/(kg TS·d)	148.0	152.9	160.7	191.3	232.1	223.5
k_{biogas}	d^{-1}	0.195	0.199	0.208	0.230	0.271	0.260
Increase in the biogas productivity**	%	–	1.6	2.4	7.8	11.6	16.1
Methane content	%	62.3±0.5	61.4±0.5	62.4±0.5	64.4±0.5	65.4±0.5	66.6±0.5

* without pretreatment
** comparing to the value without pretreatment

Table 3

Kinetic parameters of biogas production from GS

Excess granular sludge		* WP	$H_{0.5}$	$H_{1.0}$	$D_{50\%\_2.0}$	$D_{50\%\_4.0}$	$D_{100\%\_4}$
Maximal biogas productivity (experimental data)	dm^3/kg TS	200.6±4	205.2±5	207.1±5	214.0±5	228.2±5	232.8±5
	dm^3/kg VS	480.8±6	491.7±7	496.2±7	512.9±7	546.9±7	558.0±7
$C_{t;\text{biogas}}$	dm^3/kg TS	205.7	235.9	214.1	218.4	226.8	230.6
	dm^3/kg VS	493.0	565.3	513.1	523.4	543.6	552.6
r_{biogas}	dm^3/(kg TS·d)	26.7	21,0	27.8	29.0	34.0	37.4
	dm^3/(kg TS·d)	64.1	50.3	66.7	69.6	81.5	89.5
k_{biogas}	d^{-1}	0.130	0.089	0.130	0.133	0.150	0.162
Increase in biogas productivity**	%	–	2.2	3.2	6.7	13.8	16.0
Methane content	%	58.9±0.5	58.6±0.5	59.2±0.5	61.4±0.5	62.5±0.5	62.6±0.5

* without pretreatment
** comparing to the value without pretreatment

et al. (2017) (738-1176 dm^3/kg VS). These differences confirmed that biogas production may be determined by chemical composition of organic matter in the sludges. This composition varies because sludges are generated during treatment of different kind of wastewater (e.g. municipal, landfill leachate, liquid fraction of pig manure), and in different wastewater systems. TOMCZAK-WANDZEL et al. (2011) tested the effect of ultrasound disintegration (200 W UP 200S, 24 kH) at a time of 5 min on biogas productivity of activated sludge. Total volume of produced biogas after pretreatment was 20% higher than with the sludge without pretreatment. In addition, the authors indicated that methane content in the biogas increased by 10%, and this was much higher increase than in the present study.

The information on the biogas productivity of aerobic granular sludge are scarce. Little research has been done on pretreatment step on improving the productivity. VAL DEL RIO et al. (2011) studied thermal pretreatment to treat two different aerobic granular sludges, G1 from a reactor fed with pig manure and G2 from a reactor fed with a synthetic medium that simulates municipal wastewater. Biodegradability of the untreated excess aerobic granular sludge (33% for G1 and 49% for G2) was similar to that obtained for an activated sludge (30-50%). The thermal pretreatment of G1 and G2 enhanced anaerobic digestion respectively by 20% and 14% at 60°C and by 88% and 18% at 170°C, in comparison to the untreated sludge. In others study, VAL DEL RIO et al. (2013) also checked the effect of thermal pretreatment (133°C for 20 min) of aerobic granular sludge from pilot plant SBR, fed with the liquid fraction of pig slurry, on biogas potentials. The authors found that biogas production of the granules after pretreatment was more than 30% higher than that of the granules without pretreatment.

Conclusions

The study showed that biogas productivity from excess activated sludge was much higher than from excess granular sludge. The biogas productivity of AS without pretreatment was ca. 603 dm^3/kg TS (ca. 793 dm^3/kg VS); that of GS was ca. 200 dm^3/kg TS (ca. 480 dm^3/kg VS). Ultrasound disintegration increased sludge digestibility more than homogenization. After pretreatment by ultrasound disintegration there was a noticeable increase in biogas productivity and in methane content with both AS and GS. After disintegration of the sludge, the biogas productivity of AS increased by 7.8–16.1%, and that of GS increased by 7.0–16.0% depending on the parameters of disintegration. However, after homogenization, the biogas productivity increased by 2.0-3.0% (AS) and 1.6-3.2% (GS).

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