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Original article

Possibilities of Obtaining Biogas from Manure and Amaranth

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Introduction. The use of biomass allows increasing the rate of biogas formation and its specific yield. This work aims to study the kinetics of methanogenesis and determine the optimal duration of digestion and organic load, which are the main indicators of the technological process of biogas formation.

Materials and Methods. The substrate (dairy manure, biomass of amaranth) was the study object. Experimental studies were carried out using a laboratory biogas plant. The computer program (certificate No. 2018662045) was used to obtain modified Gompertz models describing the kinetics of biogas formation. Based on the obtained data, the hydraulic retention time and organic loading rate (the key parameters in the design of biogas plants were determined).

Results. The paper presents the experimental studies results of the biogas formation kinetics when using dry amaranth biomass. The Gompertz mathematical models were obtained. Methane-tank control parameters (hydraulic retention time and organic loading rate) were obtained for anaerobic digestion of a new substrate.

Discussion and Conclusion. The use of new co-substrate *Amaranthus retroflexus* L. allowed increasing the specific biogas yield from dairy manure by 52.2 % and the ultrasonic pre-treatment in combination with the herbal supplement by 89.1 %. The optimal hydraulic retention time value was 10 days and organic loading rate was 4.1 kg of volatile solids per m^3 of digester per day.

Keywords: biogas, co-digestion, dairy manure, biomass, hydraulic retention time, amaranth

Conflict of interest: The authors declare no conflict of interest.

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Оригинальная статья

Возможности получения биогаза из навоза и амаранта

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Введение. Использование биомассы позволяет увеличивать скорость образования биогаза и его удельный выход. Целью данной работы является исследование кинетики метаногенеза и определение оптимальной продолжительности сбраживания и органической нагрузки как главных показателей технологического процесса образования биогаза.
Материалы и методы. Объектом исследования являлся субстрат (коровий навоз, биомасса амаранта). Экспериментальные исследования проводились на лабораторной биогазовой установке. Для получения модифицированных моделей Гомперца, описывающих кинетику образования биогаза, использовалась программа для ЭВМ (свидетельство № 2018662045). На основе полученных данных определялись время пребывания субстрата в метантенке и доза загрузки (ключевые параметры при проектировании биогазовых установок).
Результаты исследования. В работе представлены результаты экспериментальных исследований кинетики образования биогаза при использовании сухой биомассы амаранта. Получены математические модели Гомперца. Найдены показатели для контроля метантенка (время пребывания субстрата в метантенке и доза загрузки) для анаэробного сбраживания нового субстрата.
Обсуждение и заключение. Использование нового дополнительного субстрата *Amaranthus retroflexus* L. позволило увеличить удельный выход биогаза из коровьего навоза на 52,2 %, а предварительная обработка ультразвуком, в сочетании с травяной добавкой, – на 89,1 %. Оптимальное значение времени пребывания субстрата в метантенке составило 10 дней, доза загрузки – 4,1 кг органического сухого вещества на 1 м^3 аппарата в день.

Ключевые слова: биогаз, метановое сбраживание, коровий навоз, биомасса, время пребывания субстрата, амарант

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Introduction

Sustainable development requires a systematic approach to solving the problem of organic waste recycling. At the moment, many technologies have been developed to reduce environmental pollution. However, in the application of methods for organic waste recycling, Russia has not yet reached the modern world level. Up to 250 million tons of organic waste is accumulated annually, a significant part of which decomposes in an open environment, posing a serious threat to nature and humans.

The use of improved technologies and also the joining of various technologies for organic waste recycling contribute to the development of a "circular economy" and an increase in the efficiency of resource use [1–3].

At present, a combined technology, including anaerobic digestion (AD) and pyrolysis (Py), is of particular interest [4; 5].

It allows implementing a full cycle of organic waste recycling.

Three types of process integration are known [6]:

– AD-Py. Anaerobic digestate is used for pyrolysis as a valuable feedstock material for energy and biochar production;

– Py-AD. Pyrolysis products such as biochar, gas, aqueous phase can be suitable feedstock or effective additives for the AD process;

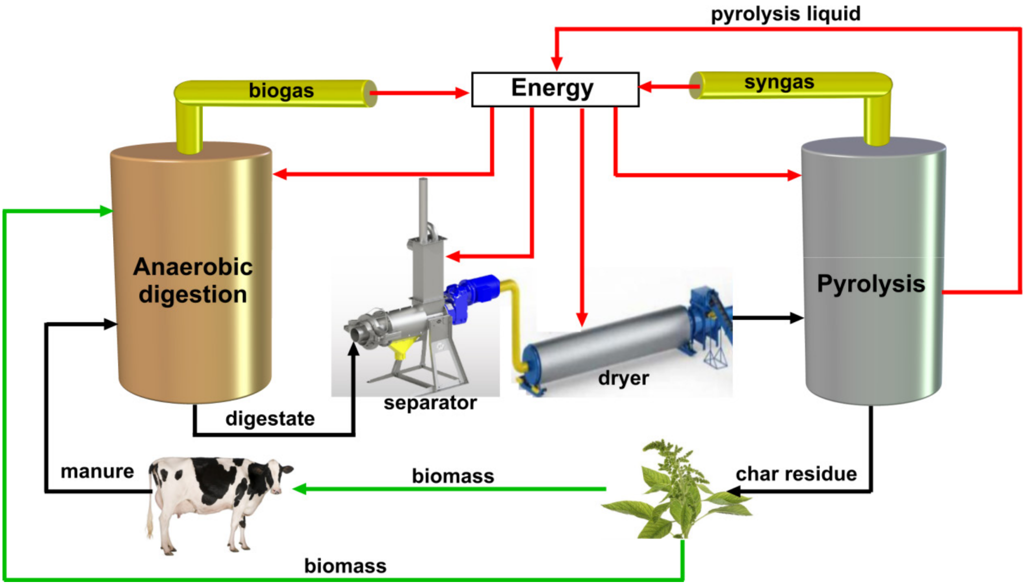
– AD-Py-AD combines the two previous methods.

Figure 1 shows the AD-Py technology combining biological and thermochemical processes. Livestock waste and plant biomass are sent for anaerobic digestion. The resulting biogas is used for energy production. The effluent is separated and dried, followed by thermochemical processing [3]. Synthesis gas and pyrolysis liquid are used as energy resources. Char residue is a good soil additive used to increase biomass yields [7]. The biomass is then used as feedstuff in livestock, and its waste is again sent for anaerobic digestion.

Such technologies are poorly studied since they include two processes: methanogenesis and thermochemical processing [8; 9]. This paper presents the experimental studies results of the first key process – anaerobic digestion of dairy manure and dry biomass of the weed plant *Amaranthus retroflexus* L. [10]. The description of the second process – the thermochemical processing of the effluent – is presented in another publication of the authors [10].

Several parameters influence the performance and biogas production for anaerobic digestion, but especially the organic loading rate (OLR) [11–13] and the hydraulic retention time (HRT) [14; 15]. Reactor control is based on OLR and HRT values [16]. The Modified Gompertz model is often used in practical applications to optimize process parameters for improving the design of the methane tank and the entire technology as a whole [15].

Some experimental studies were carried out in the laboratory of energy systems and technologies of the Institute of



F i g. 1. The AD-Py technology

Power Engineering and Advanced Technologies of the Kazan Scientific Center of the Russian Academy of Sciences to obtain a modified Gompertz model, reactor control indicators (HRT and OLR) for anaerobic digestion of a new substrate.

The aim of this study is the applicability of co-digestion of manure and amaranth biomass for improving methane production at the mesophilic temperature.

Literature Review

Anaerobic digestion of biomass (organic agricultural and domestic wastes) has a special place in energetics. It allows you to obtain biogas containing about 70 % methane, and disinfected organic fertilizers. Biomass utilization is extremely important in agriculture, where a large amount of fuel is spent on various technological needs, and the need for high-quality fertilizers is continuously increasing [17].

The total cow number in Russia is 8.3 million animal units. Thus annually more than 166.7 million tons of manure is accumulated in the region near livestock farms and poses a serious environmental threat. Besides, there is a tendency to increase the size of farms and reduce their total number. For example, in the Republic of Tatarstan (region of Russia), there are 11 megafarms, that are the largest in Europe.

Biogas production from dairy manure is unprofitable because of the low specific biogas yield [18]. Some researchers propose co-digestion to eliminate this problem [18–20]. The most popular co-substrates at biogas plants are maize, wheat straw, and grass [21–24].

Combined treatment of several substrates under AD can increase the efficiency of biogas production. The synergistic effect is achieved by the fact that the necessary microelements and nutrients contained in substrates in different quantities reach their optimal values under the correct combination [25]. At co-digestion, it is also possible to regulate the C/N ratio, which promotes better biological decomposition of organic waste and, accordingly, increases the biogas yield [26; 27].

Biogas is a promising renewable energy source that is why the search for suitable substrates is at the center of attention. During the period from 2009 to 2018, biogas production in the world doubled and continues to grow [28]. In European countries, 70 % of substrates for biogas production come from the agro-industrial complex and include manure and crop waste[1].

Plants of the Amaranth family are promising co-substrates for a significant increase in the rate of methanogenesis and the amount of produced biogas [29]. Thus, in earlier studies, it was proved that the biomass of plants from the *Amaranthaceous* family is a co-substrate for anaerobic digestion. But since cultivar amaranth is an expensive raw material, it was necessary to continue the search for affordable and cost-effective methanogenesis stimulating agents. In the present work, an experiment with biomass of *Amaranthus retroflexus* L. the closest wild-growing relative of amaranth was conducted.

Materials and Methods

The substrate (dairy manure, biomass of amaranth) was the object of the study. It was stored for two days in a refrigerator at 4 °C.

Amaranthus retroflexus L. and *Amaranthus cruentus* L. were gathered in the dissemination phase at the field in the Republic of Tatarstan (Russia).

The co-digestion process was studied in the laboratory experimental setup consisting of an LB-162 water bath,

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[1] Calderon C., Colla M., Jossart J.-M., et al. Statistical Report: Biogas. 2019. 23 p. Available at: https://www.europeanbiogas.eu/wp-content/uploads/2020/01/EBA-AR-2019-digital-version.pdf (accessed 22.03.2021). (In Eng.)

0.5 l anaerobic digesters, plastic containers, measuring cylinders, a system of rubber hoses, ultrasonic technological apparatus of the Series "Wave" UZTA-0.2/22 Ohm (Fig. 2).

The experiments were carried out in three repetitions; a thermostatic water bath maintained a mesophilic temperature (37 °C).

The volume of produced biogas was determined daily [29]. The composition of the gas was determined every 7 days in two repetitions by gas-liquid chromatography. Biogas was sampled using a 1 000 µl gas-tight syringe. Khrom 5 gas chromatograph (Austria), Porapak Q column (2.4 m long), thermal conductivity detector and gas carrier. He were used for this purpose.

The following substrates compositions were used in the experiments (table 1).

Pre-treatment was carried out for 4 minutes using the ultrasonic device with the power of 80 W at an oscillation frequency of 22 kHz and an exposure intensity of at least 10 W/cm^2.

The experiments were considered complete "when the daily biogas yield was less than 1 % of the cumulative gas yield for three days" [30]. The experiment lasted 55 days. For sample No. 1 (control), the digestion period was 37 days. For other samples, it was 55 days. The analysis of biogas yield kinetics was normalized by pressure (P = 101.3 kPa) and temperature (T = 0 °C).

Elemental analysis of the studied samples was carried out using the

F i g. 2. Experimental set-up

T a b l e 1

Composition of samples for experiments

Sample	Dairy manure mass, g	Biomass additive	Biomass / Manure ratio	Total volatile solids, g VS·L^{-1}	Ultrasonic pre-treatment
No. 1	80	–	–	40.4	no
No. 2	48	*Amaranthus retroflexus* L.	1:8	40.9	no
No. 3	48	*Amaranthus retroflexus* L.	1:8	40.9	yes
No. 4	48	*Amaranthus cruentus* L.	1:8	40.9	yes

Euro EA 3000 analyzer (analysis conditions: column temperature 115 °C, furnace temperature 850 °C).

The content of macro- and microelements was studied using the EDX-800HS2 energy-dispersive fluorescence X-ray spectrometer manufactured by Shimadzu (Japan) by a semi-quantitative method [10].

A modified Gompertz model was used by many authors to describe the kinetics of gas formation in batch anaerobic digesters from various organic substrates [21]. The modified Gompertz equation has the following form:

$$F(l) = W\exp\left(\exp\left(\frac{R_{\max}\cdot e}{W}(\alpha - l) + 1\right)\right), \quad (1)$$

where $F(l)$ – cumulative specific gas production at a time l days, liters per kilogram of volatile solids (L/kg VS); W – the gas production potential (L/kg VS); $R_{\max}$ – maximum gas production rate, L/kg VS·day; α – lag phase period, day [31].

Results

The main characteristics of dairy manure were: volatile solids (VS) (the percentage of VS content from total solids content), 75.03 ± 4.68 %; total solids (TS), 16.82 ± 1.45 %. Its principal characteristics of amaranth dry biomass were: VS = = 75.97 ± 0.6 % (the percentage of VS content from TS content); TS = 89.55 ± 0.3 %. Table 2 presents the results.

The C/N ratio for manure was equal to 16.5 ± 0.3, for *Amaranthus retroflexus* L.: 7.8 ± 0.35 and *Amaranthus cruentus* L.: 11.1 ± 0.2.

Table 3 presents the results, where AR is biomass of *Amaranthus retroflexus* L., and AC is biomass of the *Amaranthus cruentus* L.

Very high content of calcium and potassium was observed in the dry biomass of plants leaves.

A statistical analysis was performed. The experimental biogas productions were always referred to average values. Analysis of variance (ANOVA) was used to assess the influence of the analyzed factor. The significance threshold was set at 0.05. Table 4 presents the results of the analysis of variance.

There is a significant difference in the average biogas yield for all substrates with a probability of 95 %. Since the value of F is higher than the value of F_{crit} for a given number of groups, the dispersion between groups makes a greater contribution to any sum of dispersions than that within the groups. In other words, the

Table 2

Elemental composition of substrates

	C, %	H, %	O, %	N, %
Dairy manure	27.13	4.72	41.54	1.64
Amaranthus retroflexus L.	32.61	5.24	40.45	4.22
Amaranthus cruentus L.	32.33	5.49	40.62	2.91

Table 3

Content of macro- and microelements in the dry biomass, %

Name	Ca	K	P	S	Fe	Mn	Sr	Br	Si	Cl	Mg	Zn
AR	54.58	32.32	2.81	3.47	0.44	0.17	0.10	0.08	0.65	0.84	4.48	0.07
AC	58.74	20.82	3.95	3.15	1.22	0.19	0.10	0.05	2.51	3.35	5.83	0.07

Table 4

One way ANOVA for biogas yield

SUMMARY

Groups	Number of observations	Sum	Average	Variance
Sample No. 1	37	3 943.29	106.58	3 932.01
Sample No. 2	55	6 137.14	111.58	6 069.09
Sample No. 3	55	7 712.55	140.23	7 769.96
Sample No. 4	55	3 666.73	66.67	6 141.98

ANOVA

Source of variation	Sums of squares	Degrees of freedom (df)	Mean square (MS)	Fisher criterion F	p-value	Critical value F_{crit}
Between groups	151 238	3	50 412.69	8.18	$3.69 \cdot 10^{-5}$	2.65
Within groups	1 220 528	198	6 164.28			
Total	1 371 766	201				

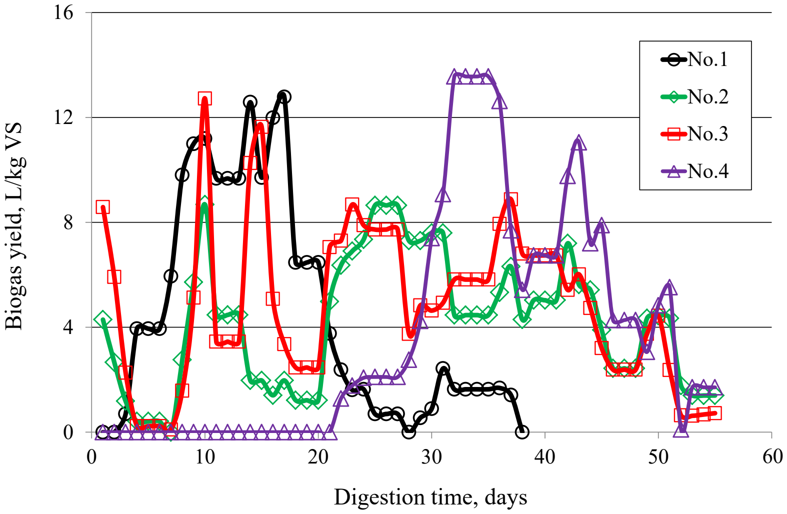
co-digestion of dairy manure and co-substrates in the anaerobic digesters has a significant effect on the biogas yield. This effect is accounted for an improvement in the nutrient balance in the mixture of manure and biomass of *Amaranthus retroflexus* L. and *Amaranthus cruentus* L. for co-digestion, which increases the solubisation, decomposition and production of biomethane.

The amount of daily produced biogas is an important indicator for comparing the various substrates used to produce biogas. The Figure 3 presents the daily specific biogas yield for the four considered substrates.

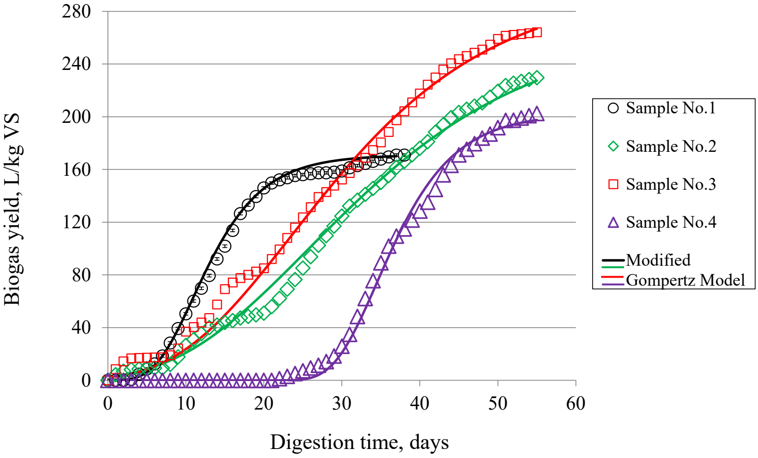
In their study Z. Wang and co-authors reported that, “when carrying out experiments on the co-digestion of acorn slag and dairy manure with bio-based carbon, two peaks of biogas production were also observed” [32]. The authors explain this by “the degradation of carbohydrates and crude protein in the anaerobic digesters” [32]. The two peaks observed in biogas production occur because the carbohydrates decomposition can be very fast within a few days, and sometimes it can take several weeks to transform crude proteins.

Figure 4 shows the cumulative specific methane yield for the four considered substrates. Numerous literature data on the production of biogas from cattle manure indicate a specific biogas yield in the range between 140 and 266 L/kg VS [33]. The obtained experimental data (sample No. 1, control) 171 L/kg VS are consistent with the literature data. Specific biogas yield for sample No. 2 was 228 L/kg VS. The use of ultrasonic pre-treatment allowed increasing the specific biogas yield to 264 L/kg VS (sample No. 3). Specific biogas yield for sample No. 4 was 202 L/kg VS. Thus, *Amaranthus retroflexus* L. is as good as the herbal supplement *Amaranthus cruentus* L.

The content of lignin in the diet of animals has a key influence on biogas production at the digestion of dairy manure. In spring and summer, when fresh grass is in the diet of animals, an increase in the



F i g. 3. Kinetics of gas formation



F i g. 4. Cumulative biogas yield

specific biogas yield is observed at anaerobic digestion of dairy manure [33]. Because experimental studies were carried out in the autumn when animals fed on hay, the specific production of biogas was lower than it could be in summer.

The computer program developed at the Kazan Scientific Center of the Russian Academy of Sciences (certificate No. 2018662045) was used to obtain modified Gompertz models describing the kinetics of biogas formation[2]. Table 5 presents

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[2] [Kinetic Analysis of Biogas Production]: Certificate of State Registration of the Program for ECM 2 018 662 045 Russian Federation. Appl. 25.09.2018; publ. 25.09.2018. (In Russ.)

the obtained kinetic constants. The maximum biogas production rate is higher in the experiment with biomass of *Amaranthus cruentus L*. According to the biogas production potential, the dry biomass of *Amaranthus retroflexus* L. treated with ultrasound would be highly competitive with the biomass of *Amaranthus cruentus* L. The longest lag-phase was observed in the experiment *Amaranthus cruentus L.*

Figure 5 shows the total biogas production end the methane content in biogas [34]. The maximum methane production was achieved on the second or third week of digestion. The maximum methane yield in biogas was observed for sample No. 3. It was 65.9 %.

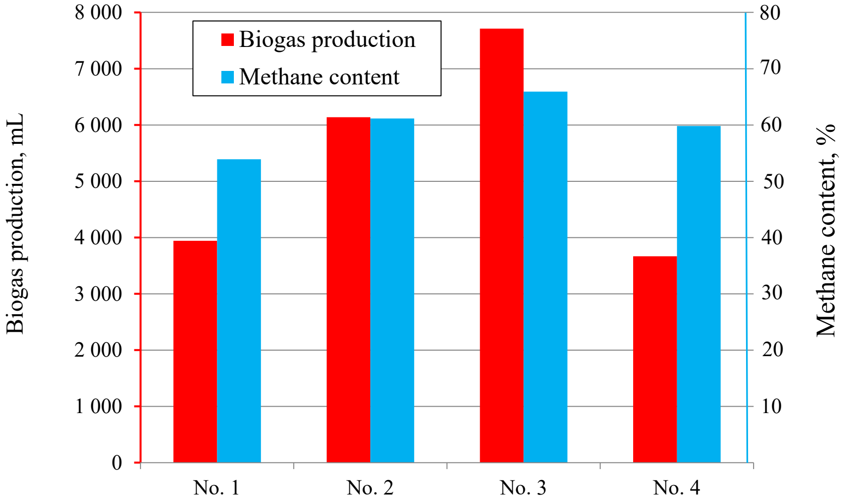
Thus, the specific methane yield for sample No. 1 was 92 L/kg VS, for No. 2 – 140 L/kg VS, for No. 3 – 174 L/kg VS, for No. 4 – 139 L/kg VS. When using *Amaranthus retroflexus* L. biomass, the specific methane yield is 25 % higher than when using the *Amaranthus cruentus* L. biomass.

The key control parameters for a conditionally continuous-action methane tank (HRT, OLR) were determined using the computer program developed at the Kazan Scientific Center of the Russian Academy of Sciences[3]. The initial data were the results of experimental studies for the batch methane tank, namely, the function of the cumulative biogas yield and the biogas yield rate as a function of time (see Fig. 6

Table 5

Kinetic constants of the modified Gompertz model

No.	W, L/kg VS	R_{max}, L/kg VS·day	α, day	R^2	Pearson's correlation coefficient R^2	Standard error, %
1	170.91	12.78	6	1.0000	0.9978	5.09
2	270.00	6.00	9	1.0000	0.9941	2.64
3	300.00	7.60	9	1.0000	0.9958	3.59
4	202.49	13.57	29	0.9999	0.9964	9.30



F i g. 5. Total biogas production and methane concentration

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[3] [Optimization of Operating Modes of the Apparatus]: Certificate of State Registration of the Program for ECM 2 015 662 618 Russian Federation. Appl. 27.11.2015; publ. 20.12.2015. (In Russ.)

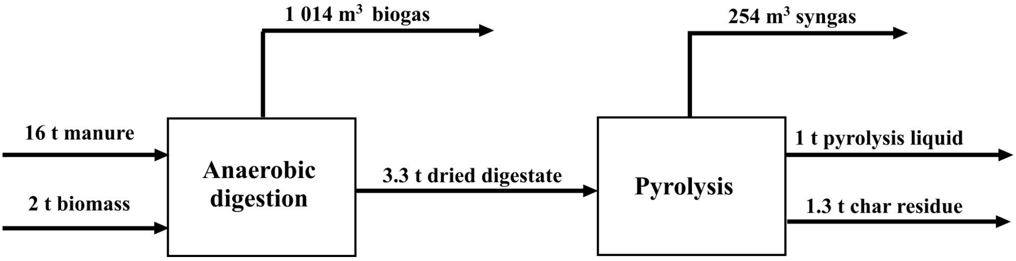

Fig. 6. Material balance of the AD-Py technology

for an example). The optimal HRT value was 10 days, OLR 4.1 kg VS/m^3 of digester per day. Let us consider a biogas plant, processing daily 18 tons of organic waste. When changing the composition of the processed substrate, 2 tons of *Amaranthus retroflexus* L. and 16 tons of manure should be added every day to the biogas plant. The material balance of anaerobic digestion was compiled based on the experimental data presented in this work. The material balance of the pyrolysis of the dried digestate was compiled based on the experimental data presented by the scientists [10].

After applying the preliminary ultrasonic treatment (an oscillation frequency of 22 kHz and an exposure intensity of at least 10 W/cm^2) and introducing a new herbal additive into the methane tank, the daily biogas yield will increase to 1 014 m^3. Daily use of biogas will make it possible to obtain 5 576.7 kW·h, synthesis gas – 846.7 kW·h, and pyrolysis liquid 5 611 kW·h.

Discussion and Conclusion

In this study, a higher biogas yield was due to a synergistic effect caused by technological (ultrasonic pre-treatment) and microbiological (co-digestion) effects. The paper presents the use of a new phytogenous co-substrate. A weed plant cosmopolite amaranth (*Amaranthus retroflexus* L.) was used for this purpose. The biogas production potential at mono-digestion of dairy manure was 170.91 L/kg VS when using dry amaranth (*Amaranthus retroflexus* L.) – 270 L/kg VS when using the preliminary ultrasonic treatment and dry amaranth (*Amaranthus retroflexus* L.) – 300 L/kg VS.

The use of new co-substrate *Amaranthus retroflexus* L. allowed increasing the cumulative biogas yield from dairy manure by 52.2 % and the ultrasonic pre-treatment in combination with the herbal supplement by 89.1 %.

The biomass of *Amaranthus retroflexus* L. was found to be comparable with the biomass of *Amaranthus cruentus* L. Therefore, further studies on the use of the co-substrate *Amaranthus retroflexus* L. in biogas plants are needed. It is especially important in the production of biogas from difficultly fermented substrates.

The optimal HRT value was 10 days, OLR 4.1 kg VS/m^3 of digester per day.

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