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Methane production during laboratory-scale co-digestion of cattle slurry with 10 wt. % of various biowastes

Jiří Rusín, Kateřina Chamrádová, Karel Obroučka, Roman Kuča

VŠB, Technical University Ostrava, Centre of environmental technologies, 9350, 17. listopadu 15, Ostrava – Poruba 708 33, e-mail: jiri.rusin@vsb.cz, katerina.chamradova@vsb.cz, karel.obroucka@vsb.cz, roman.kuca@vsb.cz

The paper summarizes the results from twenty model tests of continuous one-stage mesophilic anaerobic co-digestion of cattle slurry (90 wt. %) and various biowastes (10 wt. %). Digestion was conducted in 0.06 m^3 reactors with hydraulic retention times ranging from 60 to 98 days during the research period 2007–2010. Methane production intensity and specific methane production are discussed. The highest methane production intensity (0.85 $m_N^3.m^{-3}.d^{-1}$) was from a mixture of 63 wt. % of total solids from biscuit meal EKPO – EB and from 37 wt. % of total solids from cattle slurry. The highest specific methane production from 1 kg of added organic compounds (0.67 $m_N^3.kg_{VSp}^{-1}$) was given by a mixture containing 61 wt. % of total solids using spring barley Aksamit (milled grain) and 39 wt. % of total solids from cattle slurry. The highest substrate-specific methane production (0.92 $m_N^3.kg_{VSp}^{-1}$) was from milled grains of winter rye Aventino.

Keywords: biowaste, cattle slurry, anaerobic co-digestion, specific methane production.

INTRODUCTION

The Centre for Environmental Technology of VŠB – TU Ostrava in cooperation with VÍTKOVICE POWER ENGINEERING a.s. dealt with a research project designed to increase anaerobic biogas production from cattle slurry by co-digestion with various biowastes. The aim of this project was to verify specific biogas and methane production from various biowastes under continuous mesophilic conditions and to compare the results with the data from literature. The comparison of specific biogas and methane production data from literature is difficult due to the fact that the data are most often related to co-digestion with sewage sludge[1] or pig slurry[2] often at discontinuous tests[3] or to very different hydraulic retention time and fermenter load. For some an-aerobically degradable wastes such as wastes from confectionery production, G-phase from rape oil methyl ester production and so on, specific methane production data was not found at all. The purpose of this paper is to compare the results of a series of model anaerobic digestion tests performed with input mixtures containing 90 wt. % of cattle slurry and 10 wt. % of biowaste.

MATERIALS AND METHODS

The cattle slurry (reference substrate and inoculum) was from the dairy-farm Zemspol Studénka, a.s. This cattle slurry was actually digested in reference agricultural biogas station of the company VÍTKOVICE POWER ENGINEERING, a.s. in Pustějov. Biowastes gained from co-operating companies in Moravian-Silesian region were mainly agricultural commodities (feedstuff residues) and wastes from food industry, for example from sugar refinery, distillery, brewery, confectionery production, etc. (Table 1).

No mechanical or physical treatment of biowaste was used. The input mixtures of biowaste with cattle slurry were always prepared in spare amount from 3 to 5 days while hydrolysis and acidification partially occurred (at laboratory temperature 24±3°C).

Six model fermenters of same construction with the loading volume of 0.06 m^3 and continuous stirring (Figure 1) were used for the realization of the long-term tests of continuous mesophilic anaerobic digestion or co-digestion. Semi-continuous feeding equal to 0.001 m^3 of input mixture containing 90 wt. % of cattle slurry and 10 wt. % of biowaste (1.67 % of volume reactor per day) proceeded only during the working days. Average digestion temperature was kept on 40±3°C with a continuous run of the low-speed stirrer. The measurements of biogas production were carried out with the laboratory drum-type gas flow meters and the composition of biogas was measured daily by mobile analyser with IR and electrochemical sensors and occasionally controlled by gas chromatography. The total solids (TS), volatile solids (VS), chemical oxygen demand (COD), pH and volumetric mass (bulk density) were measured for input mixtures and digestates twice a week. After each digestion test, average values of parameters characterizing the input mixture, digestate, biogas and process itself were calculated. The anaerobic process was characterized by average volume loading of reaction area and by organic compounds (OL), theoretical hydraulic retention time (HRT) and removal efficiency of added organic compounds. The average values of biogas and methane production were calculated according to one-day-process; according to volume unit of input mixture; according to mass unit of added total solids; according to the mass unit of added / decomposed organic compounds and according to volume unit of reaction area (related to normal conditions 0°C, 101.325 kPa).

Reference test

The reference test was mesophilic anaerobic digestion of cattle slurry containing 7.5 wt. % of total solids, 80.0 wt. % of volatile solids and 80000 $mg.dm^{-3}$ COD_{Cr}. Average organic load of the fermenter was 1.032 $kg_{VS}.m^{-3}.d^{-1}$. Average values of specific biogas and methane production were calculated after 500 days of process running at average HRT 69 d.

Co-digestion tests

Each model co-digestion test started with several-days

Table 1. Analytically measured parameters of biowaste (average values)

BIOWASTE	Biowaste Producer	pH	Total solids	Volatile solids (Ignition loss)	Total carbon	Total nitrogen	Carbon : Nitrogen	Ammonia	Phosphorus	Lipids	Amyl	Total fiber
			TS	VS	TC	TN	TC:TN	N-NH_4^+	TP	L	A	TF
		–	wt. %	$\%_{TS}$	$\%_{TS}$	$\%_{TS}$	–	$\%_{TS}$	$\%_{TS}$	$\%_{TS}$	$\%_{TS}$	$\%_{TS}$
CATTLE SLURRY (long-term averages)	ZEMSPOL Studénka. a.s. Pustějov	6.9	7.50	80.00	35.70	5.83	6.1	2.14	0.81	3.20	7.00	14.67
Corn silage - hybrid LG32.66		4.3	32.30	95.80	42.50	1.90	22.4	0.65	2.30	9.00	0.34	7.61
Corn grain - hybrid LG32.66		5.8	60.10	98.00	43.70	1.56	28.0	0.27	0.25	10.00	7.02	2.00
Winter rye Aventino (milled grain)		–	86.00	97.50	39.10	2.15	18.2	0.00	0.86	1.50	0.61	3.80
Spring barley Aksamit (milled grain)		–	87.40	97.80	40.80	2.19	18.6	0.00	0.27	1.80	0.66	4.00
Processed sugar beet cuttings	Moravskoslezské cukrovary. a.s. Opava	3.9	19.50	91.70	35.70	2.63	11.0	0.16	0.14	0.64	23.50	25.19
Stillage (distillery residues) from the fruit mixture	Lihovar a likérka Velká Polom. s.r.o.	3.6	8.00	90.60	35.70	1.30	27.3	0.18	0.17	0.65	3.08	10.61
Stillage from the waste of confectionery production		3.7	9.00	95.40	30.10	3.15	9.6	0.98	0.26	16.10	1.54	2.18
Malt dust from the brewery	Plzeňský Prazdroj. a. s.. Radegast Nošovice	–	94.70	95.10	41.10	2.84	14.5	0.30	0.42	0.87	15.90	12.90
Wastes from purging of cabbage	Zemědělského družstva vlastníků Nošovice	4.1	7.20	83.20	34.50	6.17	5.6	1.09	0.67	2.24	1.00	22.95
Fresh green grass	VŠB - TU Ostrava - Poruba	6.4	16.60	86.30	39.70	3.33	11.9	0.05	0.56	0.30	3.60	3.64
G-phase – secondary product from rape oil methyl ester production	GLYCONA. s.r.o. Otrokovice	8.3	99.90	91.20	31.30	0.10	313	0.01	0.00	6.10	0.02	0.00
Biscuit feeding meal EKPO	CERVUS. s.r.o. Olomouc	–	95.30	94.00	45.70	1.62	28.2	0.04	0.21	7.37	19.00	7.10
Biscuit meal EKPO-EB for biogas production		–	94.20	98.70	52.30	1.14	45.9	0.04	0.12	16.40	38.80	0.40
Mixed waste wafer material	Opavia - LU. a.s. Opava	–	90.70	99.00	43.30	1.70	25.5	0.02	0.09	0.76	70.00	0.60
Dough mixed waste		7.9	83.00	98.00	41.30	1.48	27.9	0.01	0.27	12.40	48.80	0.90
Waste of confectionery filling DELI		6.5	98.50	98.40	53.70	1.37	39.2	0.04	0.18	32.30	9.57	0.58
Mixed waste chocolate parts		–	96.20	98.80	48.00	1.48	32.4	0.03	0.16	12.00	10.00	0.66
Waste starch from the confectionery gel production		–	89.80	99.90	38.10	0.12	317.5	0.00	0.03	0.17	92.80	0.16
Yew needles extracted by methanol	Ivax Pharmaceuticals s.r.o.. Opava (TEVA)	–	46.60	89.20	45.90	2.58	17.8	0.06	0.49	0.96	0.87	16.27

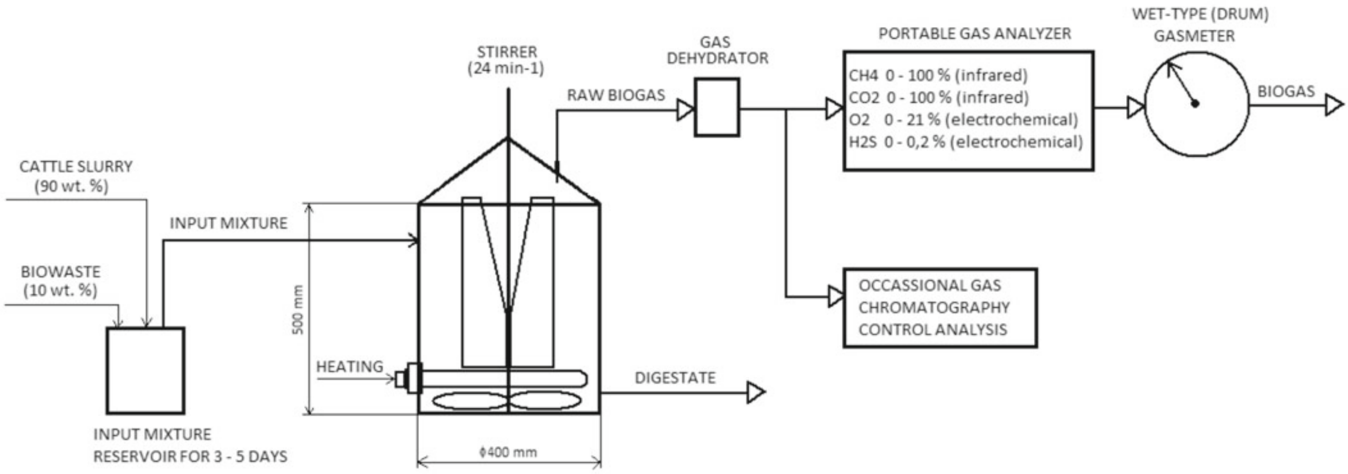


Figure 1. Layout of laboratory apparatus

implementation of model fermenter for cattle slurry (from 10 till 15 days of cultivation without dosage), further dosage of 1.0 $dm^3.d^{-1}$ of cattle slurry till stable biogas production and biogas composition. Then the daily dosage of input mixture containing 90 wt. % of cattle slurry and 10 wt. % of biowaste started. The measured parameters were compared with the average results of the reference test.

RESULTS AND DISCUSSION

In table 1, the measured parameters of nineteen tested biowastes (co-substrates) and cattle slurry are introduced. The calculated parameters of model input mixtures prepared by mixing of cattle slurry (90 wt. %) with biowaste (10 wt. %) are introduced in table 2. These parameters were calculated as weighted averages of appropriate values of used biowastes with significance 1 – mass part of biowaste, additionally with significance 2 – mass part of total solids of biowaste. The input mixtures comprised 10 wt. % of liquid biowaste – for example stillages (distillery residues) contained only 6 wt. % of total solids, whereas in the case of solid biowaste (e.g. malt dust), the input mixtures contained even 17 wt. % of total solids. The COD of the input mixtures was in the range of 80000 to 270000 $mg.dm^{-3}$. The crude lipids content was in the range of 1.8 to 21 wt. % of total solids.

In table 3, there are the averages of measured parameters of input mixtures (pH, TS, VS), process parameters (OL – fermenter loading by VS, HRT – theoretic hydraulic retention time), further methane production intensity M_r (daily methane production expressed according to volume unit of active reaction area), specific methane production from added organic compounds of input mixtures (M_{VSp}) and co-substrate specific methane production (CM_{VSp}). It is clear that the average values of the measured total solids (TS) of input mixtures are in most cases lower than the total solids calculated (Table 2) on the basis of biowaste analyses from table 1. The differences are mainly due to a limited number of the analysed samples of biowastes and also due to the fact that the prepared input mixtures went under partial hydrolysis and acidification before analysis. The measured loading of the model fermenter by volatile solids varied in the range of 0.7 to 1.7 $kg_{VS}.m^{-3}.d^{-1}$.

During the series of model co-digestion tests, high differences in biogas or methane production rates were measured. This fact is not only due to the different content of volatile solids (VS), their composition (e.g. content of lipids, carbohydrates, proteins) and real anaerobic dissolubility but also due to not the same retention time of substrate in all model tests (which were carried out during 4 years of the research). The average theoretical hydraulic retention time was in the range of 60 to 98 days (due to the omitted samples during the days off work). However, according to the opinion of the authors, the obtained results have sufficient testify ability. The biowaste-specific methane production (CM_{VSp}; $m_N^3.kg^{-1}$) is depicted in the ascending order (Figure 2).

Methane production intensity

The highest methane production intensity Mr as 0.85 $m_N^3.m^{-3}.d^{-1}$ (Table 3) was gained during co-digestion of biscuit meal for biogas production (EKPO-EB from the firm CERVUS, s.r.o. Olomouc). The second highest methane production intensity (0.75 $m_N^3.m^{-3}.d^{-1}$) was measured during co-digestion of G-phase (secondary product from rape oil methyl ester production). These co-substrates contained significant values of lipids or fatty acids. Mean methane production intensity was reached within co-digestion of biowastes composed mainly from carbohydrates and starch. For example during co-digestion of melted rye grain, methane production intensity was 0.68 $m_N^3.m^{-3}.d^{-1}$. In case of co-digestion of 10 wt. % of waste starch from confectionery gel production, methane production intensity was 0.59 $m_N^3.m^{-3}.d^{-1}$. Only 4 from totally 19 model mixtures performed lower methane production intensity than 0.28 $m_N^3.m^{-3}.d^{-1}$, which is the value equivalent to digestion of cattle slurry itself.

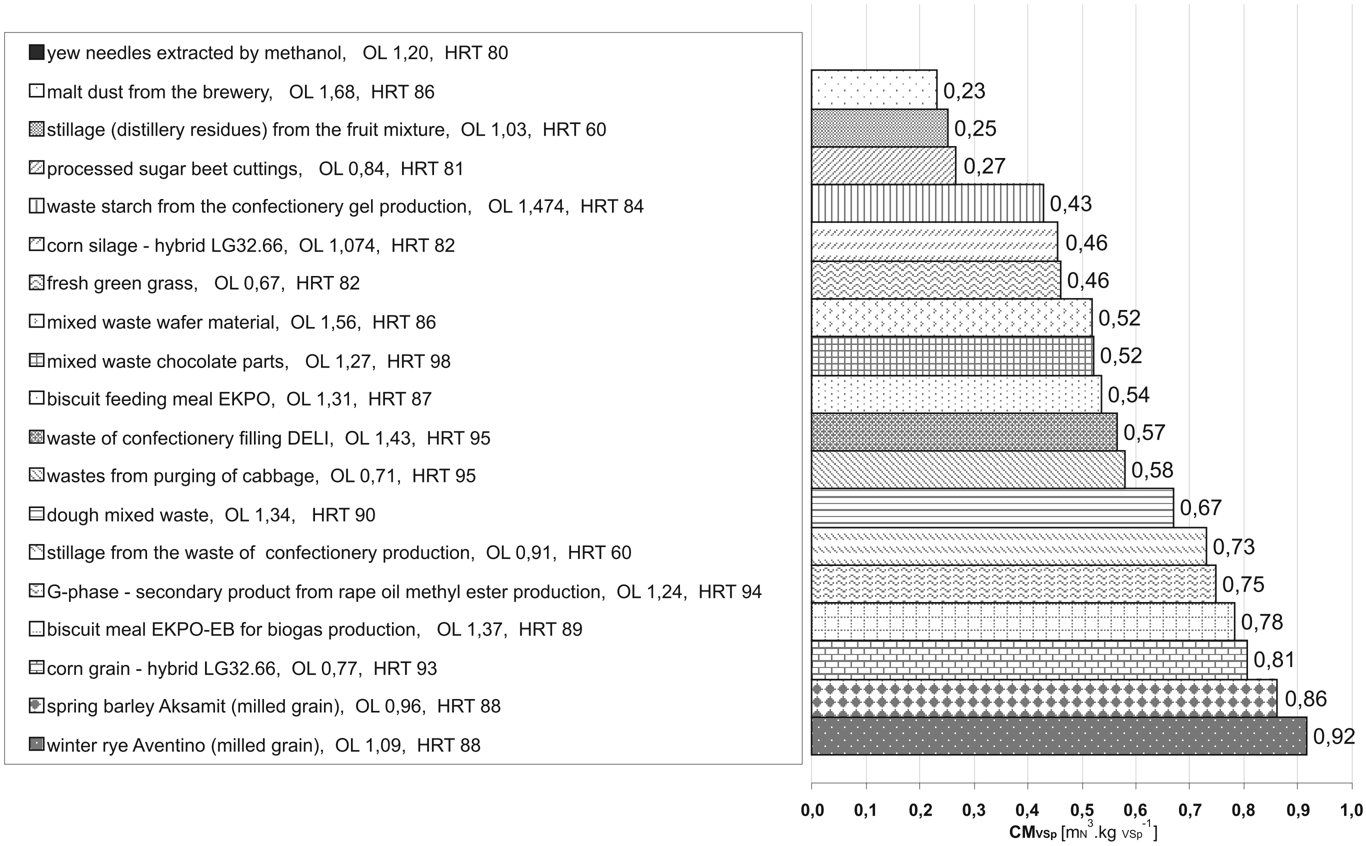
Specific methane production of the input mixture

Considering the mass unit of the added volatile solids (VS) to the input mixture, the highest specific methane production (0.67 $m_N^3.kg_{VSp}^{-1}$) was reached by co-digestion of spring barley Aksamit (Table 3).

The second highest specific methane production (0.65 $m_N^3.kg_{VSp}^{-1}$) conformed to 10 wt. % of corn grain. The third highest specific methane production (0.63 $m_N^3.kg_{VSp}^{-1}$) conformed to 10 wt. % of biscuit meal EKPO-EB. On the contrary, the lowest specific methane

Table 2. Calculated parameters of model mixtures of cattle slurry (90 wt. %) and biowaste (10 wt. %)

BIOWASTE (10 % co-substrate in the mixture)	Biowaste Producer	Total solids	Total solids derived from co-substrate	Volatile solids (Ignition loss)	Volatile solids derived from co-substrate	Carbon : Nitrogen	Ammonia	Phosphorus	Lipids	Amyl	Total fiber
		TS	K_{TS}	VS	K_{VS}	TC:TN	N-NH4^{+}	TP	L	A	TF
		wt. %	%	$\%_{TS}$	%	–	$\%_{TS}$	$\%_{TS}$	$\%_{TS}$	$\%_{TS}$	$\%_{TS}$
Corn silage - hybrid LG32.66	ZEMSPOL Studénka. a.s. Pustějov	9.98	32.36	85.11	36.43	8.3	1.47	1.29	5.08	4.85	12.39
Corn grain - hybrid LG32.66		12.76	47.10	88.48	52.17	10.3	1.26	0.43	6.40	7.01	8.70
Winter rye Aventino (milled grain)		15.35	56.03	89.80	60.83	10.0	0.94	0.84	2.25	3.42	8.58
Spring barley Aksamit (milled grain)		15.49	56.42	90.04	61.28	10.2	0.93	0.51	2.41	3.42	8.65
Processed sugar beet cuttings	Moravskoslezské cukrovary. a.s. Opava	8.70	22.41	82.62	24.88	6.7	1.70	0.66	2.63	10.70	17.03
Stillage (distillery residues) from the fruit mixture	Lihovar a likérka Velká Polom. s.r.o.	7.55	10.60	81.12	11.83	6.7	1.93	0.74	2.93	6.58	14.24
Stillage from the waste of confectionary production		7.65	11.76	81.81	13.72	6.4	2.01	0.75	4.72	6.36	13.20
Malt dust from the brewery	Plzeňský Prazdroj. a. s.. Radegast Nošovice	16.22	58.38	88.82	62.52	9.5	1.06	0.58	1.84	12.20	13.64
Wastes from purging of cabbage	Zemědělského družstva vlastníků Nošovice	7.47	9.67	80.31	10.02	6.1	2.04	0.80	3.11	6.42	15.47
Fresh green grass	VŠB - TU Ostrava - Poruba	8.41	19.74	81.24	20.97	6.8	1.73	0.76	2.63	6.33	12.49
G-phase – secondary product from rape oil methyl ester production	GLYCONA. s.r.o. Otrokovice	16.74	59.68	86.68	62.79	13.7	0.87	0.33	4.93	2.83	5.92
Biscuit feeding meal EKPO	CERVUS. s.r.o. Olomouc	16.28	58.54	88.20	62.39	12.3	0.91	0.46	5.64	14.02	10.24
Biscuit meal EKPO-EB for biogas production		16.17	58.26	90.89	63.26	14.6	0.91	0.41	10.89	25.53	6.36
Mixed waste wafer material	Opavia - LU. a.s. Opava	15.82	57.33	90.89	62.45	11.6	0.93	0.40	1.80	43.12	6.60
Dough mixed waste		15.05	55.15	89.93	60.10	11.3	0.97	0.51	8.27	30.05	7.08
Waste of confectionery filling DELI		16.60	59.34	90.92	64.22	14.6	0.90	0.43	20.47	8.52	6.31
Mixed waste chocolate parts		16.37	58.77	91.05	63.77	13.1	0.90	0.43	8.37	8.76	6.44
Waste starch from the confectionery gel production		15.73	57.09	91.36	62.42	14.4	0.92	0.37	1.47	55.98	6.39
Yew needles extracted by methanol	Ivax Pharmaceuticals s.r.o.. Opava (TEVA)	11.41	40.84	83.76	43.50	8.9	1.29	0.68	2.29	4.50	15.32



Note: OL – volatile solids loading of the model fermenter, HRT – theoretical hydraulic retention time.

Figure 2. Specific methane production from biowaste (maximum obtained values at stated conditions)

production was measured for 10 wt. % of yew needles extracted by methanol (0.11 $m_N^3.kg_{VSp}^{-1}$). The extracted yew needles contained a minimal part of anaerobic decomposable organic compounds, moreover presumably induced some inhibition.

Specific methane production of the biowaste (co-substrate)

In the reference test the specific methane production equal to 0.28 $m_N^3.kg_{VSp}^{-1}$ for cattle slurry was measured. Demirer[4] measured the average specific methane production 0.19 $m_N^3.kg_{VSp}^{-1}$ for cattle slurry (in a laboratory fermenter mixed by biogas). Ahring et al.[5] presented the highest measured value 0.20 $m_N^3.kg_{VSp}^{-1}$. Chen[6] and Habig[7] presented specific methane production for cattle slurry from 0.13 to 0.32 $m_N^3.kg_{IL}^{-1}$ (related to a kilogram of organic compounds calculated as total solids loss by annealing at 550°C). The value 0.28 $m_N^3.kg_{VSp}^{-1}$ of specific methane production obtained from our tests seems to be real, also with consideration that it is related to a kilogram of organic compounds reached from burning of total solids at 800°C, so that with consideration of decomposable effect over 550°C. Considering mainly the positive effect on anaerobic decomposition of organic compounds from cattle slurry during the co-digestion with biowastes, it may be presumed that specific methane production higher than 0.30 $m_N^3.kg_{IL}^{-1}$ is reached during the co-digestion of cattle slurry with biowastes. Straka[8,9] presented the value 0.36 $m_N^3.kg_{VSp}^{-1}$ as the most common methane production from cattle slurry.

For the calculation of specific methane production of the tested biowastes, the ratio of biowaste volatile solids (VS) in the input mixture (Table 3) and specific methane production from cattle slurry (0.36 $m_N^3.kg_{VSp}^{-1}$) were used. The highest specific methane production (0.92 $m_N^3.kg_{VSp}^{-1}$) from the tested biowastes had winter rye Aventino milled grains. The second highest specific methane production (0.86 $m_N^3.kg_{VSp}^{-1}$) fell on spring barley Aksamit milled grains. Specific methane production of EKPO-EB biscuit meal or G-phase is around 0.75–0.78 $m_N^3.kg_{VSp}^{-1}$. Selly[10] presented the maximal specific methane production from stillages (distillery residues) as 0.60 $m_N^3.kg_{VSp}^{-1}$. Our results of methane production from stillages (distillery residues) of confectionery production were about 0.73 $m_N^3.kg_{VSp}^{-1}$. Das[11] and Sales[12] presents that energetic biogas content from mesophilic digestion of stillages is higher than energy needed for distillation when stillages are formed. It can be presumed that stillages from fruit will be always rather at lower limit of biogas production (opposite to, for example, stillages from ray, corn or potatoes). Different wastes from confectionery production as, for example, chocolate waste part or wafer matter gave the specific methane production from about 0.50 to 0.60 $m_N^3.kg_{VSp}^{-1}$. Low values of specific methane production were measured for processed sugar beet cuttings (0.27 $m_N^3.kg_{VSp}^{-1}$), while the literature[13] indicates the value 0.37 $m_N^3.kg_{VSp}^{-1}$. Methane production from malt dust from brewery is about 0.23 $m_N^3.kg_{VSp}^{-1}$.

CONCLUSION

The results from nineteen model tests realized by the same way of a continuous mesophilic anaerobic digestion of cattle slurry with 10 wt. % of biowaste with retention time of about 85–95 days were compared. The ratio of carbon to nitrogen (from 6.0 to 15.0) was for all input

Table 3. Measured parameters of input mixtures. loading of the fermenters and specific methane production

BIOWASTE (10 % co-substrate in the mixture)	Measured pH of input mixture	Measured total solids	Total solids derived from co-substrate	Volatile solids derived from co-substrate	Organic load of the fermenter	Theoretical hydraulic retention time	Methane production intensity	Specific methane production *(added organics)*	Co-substrate spec. methane production *(added organics)*
	pH	TS	K_{TS}	K_{VS}	OL	HRT	M_r	M_{VSp}	CM_{VSp}
	–	% hm.	%	%	$kg_{VS}.m^{-3}.d^{-1}$	d	$m_N^3.m^{-3}.d^{-1}$	$m_N^3.kg_{VSp}^{-1}$	$m_N^3.kg_{VSp}^{-1}$
Biscuit meal EKPO-EB for biogas production	4.7	13.4	58.3	63.3	1.369	89	0.85	0.63	0.78
G-phase – secondary product from rape oil methyl ester production	6.5	12.7	59.7	62.8	1.240	94	0.75	0.60	0.75
Dough mixed waste	4.8	13.0	55.2	60.1	1.337	90	0.73	0.55	0.67
Mixed waste wafer material	4.2	14.4	57.3	62.5	1.558	86	0.72	0.46	0.52
Waste of confectionery filling DELI	4.5	15.0	59.3	64.2	1.425	95	0.70	0.49	0.57
Winter rye Aventino (milled grain)	4.3	10.6	56.0	60.8	1.092	88	0.68	0.62	0.92
Spring barley Aksamit (milled grain)	4.4	9.6	56.4	61.3	0.964	88	0.64	0.67	0.86
Corn grain - hybrid LG32.66	6.4	8.4	47.1	52.2	0.765	93	0.62	0.65	0.81
Waste starch from the confectionery gel production	4.7	13.2	57.1	62.4	1.474	84	0.59	0.40	0.43
Mixed waste chocolate parts	4.9	13.7	58.8	63.8	1.273	98	0.59	0.46	0.52
Biscuit feeding meal EKPO	4.9	12.6	58.5	62.4	1.309	87	0.52	0.40	0.54
Malt dust from the brewery	5.0	16.7	58.4	62.5	1.680	86	0.47	0.28	0.23
Corn silage - hybrid LG32.66	6.1	10.2	32.4	36.4	1.074	82	0.42	0.40	0.46
Stillage (distillery residues) from the fruit mixture	7.3	6.8	10.6	11.8	1.032	60	0.36	0.35	0.25
Wastes from purging of cabbage	5.9	8.6	9.7	10.0	0.708	95	0.30	0.38	0.58
CATTLE SLURRY	7.1	7.9	–	–	1.032	69	0.29	0.28	0.28
Processed sugar beet cuttings	6.8	8.3	22.4	24.9	0.839	81	0.28	0.34	0.27
Stillage from the waste of confectionary production	6.9	6.6	11.8	13.7	0.905	60	0.25	0.28	0.73
Fresh green grass	6.5	6.9	19.7	21.0	0.666	82	0.25	0.37	0.46
Yew needles extracted by methanol	6.5	11.0	40.8	43.5	1.204	80	0.13	0.11	0.00

mixtures at the low level of optimum for anaerobic digestion. The optimal relation C : N (approximately from 20 : 1 to 40 : 1) can be reached for 30 wt. % of biowastes in mixtures with cattle slurry. The highest specific methane production from 1 kg of added organic compounds (0.67 $m_N^3.kg_{VSp}^{-1}$) gave the mixture whose total solids was composed from 61 wt. % of total solids from spring barley Aksamit and 39 wt. % of total solids from cattle slurry.

The totally highest specific methane production from biowaste itself was found during the model co-digestion test of cattle slurry with 10 wt. % of winter rye Aventino (0.92 $m_N^3.kg_{VSp}^{-1}$).

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