

Use of ethanol production and stillage processing residues for biogas production

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Abstract. In Latvia, ethanol is produced mainly from wheat grains. The production process involves the formation of the by-products of wheat bran, grains residues and stillage. By-products from production of alcohol distilling dregs (stillage) contain much organic matter therefore could be useful for the production of the biogas. The product with high protein content usable for feed can be produced from the stillage too. A liquid residue is formed during the production process. Purpose of study is the assessment of the methane volume obtainable from the stillage processing residue mixed with wheat brans and grains residues in anaerobic fermentation process and from wheat brans and grains residues mixed only with inoculum. Investigation was provided in 16 bioreactors operated in batch mode at 38 °C. Stillage processing residues mixed with the wheat brans and inoculum were filled into 4 bioreactors, mixed with grains residues were filled into 4 bioreactors and only inoculum was filled into two bioreactors for control. Wheat brans with inoculum were filled into 3 bioreactors. Into others 3 bioreactors were filled grains residues with inoculum. The yield of biogas from wheat brans was 1.151 L g⁻¹_{DOM} and methane 0.593 L g⁻¹_{DOM} after 30 days of anaerobic digestion. The yield of biogas from wheat brans with stillage processing residue was 1.098 L g⁻¹_{DOM} and methane 0.600 L g⁻¹_{DOM}. The yield of biogas from grains residues was 0.915 L g⁻¹_{DOM} and methane 0.451 L g⁻¹_{DOM}. The yield of biogas from grains residues with stillage processing residue was 1.01 L g⁻¹_{DOM} and methane 0.523 L g⁻¹_{DOM}. The study demonstrates that the investigated products are very good raw material for the production of methane. Stillage processing residue acted as a catalyst for the process.

Key words: methane; stillage, anaerobic digestion, wheat brans, grains residues, stillage processing residue.

INTRODUCTION

Most biogas plants built in Latvia are large and therefore require a lot of raw materials. Many of them do not have enough land to grow own raw materials and therefore raw materials are transported even from a long distance. The prices on raw materials increased significantly (Atanasiu, 2010). There has been fierce competition for arable land, and farmers who have been able to rent cheap land so far are particularly dissatisfied. Now, due to the development of biogas production, land prices have risen. Although there is a lot of unused or underutilized land in Latvia (Dubrovskis & Adamovics, 2012), competition is getting worse and owners of dairy farms, who do not have biogas plants, are putting pressure on the Ministry of Agriculture and the Ministry

of Economy to limit the use of arable land for biogas producers. At the same time, some food production facilities produce waste and it is difficult to dispose of such waste (Al Seadi et al., 2008). For example, a bioethanol plant stillage processing product still contains a lot of chemical oxygen demand (COD) and cannot be easily cleaned in biological treatment plants, because its pH is also low. One of solutions of this problem is its use for the biogas production, but effectiveness of anaerobic digestion process can be lowered due to too low dry matter content (Wilkie et al., 2000; Westerholm et al., 2012).

However, with rising energy prices bioethanol plants will need to optimize energy consumption in order to avoid a negative impact on the costs of ethanol production (Drosg et al., 2008).

Many researchers have been investigated the potential of biogas from the stillage (Stover et al., 1984; Wilkie et al., 2000; Schaefer, 2006; Kim et al., 2008; Schaefer & Sung 2008; Kaparaju et al., 2010; Ghorbani, 2011; Dubrovskis & Plume, 2017a). The study of the biochemical potential of methane (BMP) from the stillage gave methane yield $0.409.8 \text{ Nm}^3 \text{ CH}_4 \text{ kg}^{-1}_{\text{DOM}}$ from the raw grain and $0.467.6 \text{ Nm}^3 \text{ CH}_4 \text{ kg}^{-1}_{\text{DOM}}$ from greenery (Errata, 2015). Investigation conducted by the Swedish Boras University (Awosolu, 2008) identified and compared the theoretical methane potential for stillage produced from wheat $0.473 \text{ m}^3 \text{ kg}^{-1}_{\text{DOM}}$. For cellulose fibre it was $0.407 \text{ m}^3 \text{ kg}^{-1}_{\text{DOM}}$. Practically got methane $0.288 \text{ m}^3 \text{ kg}^{-1}_{\text{DOM}}$ from wheat stillage and $0.218 \text{ m}^3 \text{ kg}^{-1}_{\text{DOM}}$ from cellulose stillage. University of Vienna have been investigated (Drosg et al., 2013) found BMP for each stillage fraction. There are a lot of data on anaerobic fermentation of thin stillage. Methane BMP of thin stillage (TS = 7.5%) it was $500 \text{ Nm}^3 \text{ t}^{-1}_{\text{VS}}$ added. However, the amount of methane extracted from the stillage processing is a highly liquid product (1 to 1.5% dry matter), which is produced in the Iecava's bioethanol plant, was not found in the literature. In Latvia such the research has been carried out for the first time.

Biogas potential from the wheat bran has been investigated by several (Becker et al., 2007; Drosg, 2008; Wellinger et al., 2013) researchers. The substrate with and without pre-treatment gave daily methane yields of $0.430 \text{ m}^3 \text{ kg}^{-1}_{\text{DOM}}$ and $0.389 \text{ m}^3 \text{ kg}^{-1}_{\text{DOM}}$ respectively.

Researchers was investigated the potential of biogas and methane from different grains residues in the LULST Bioenergy Laboratory. Biomass were taken from a dryer where various grains were processed. In the first 2017 year study (Dubrovskis & Plume, 2017b) an average of $0.694 \pm 0.098 \text{ L g}^{-1}_{\text{DOM}}$ biogas and $0.383 \pm 0.08 \text{ L g}^{-1}_{\text{DOM}}$ methane were obtained. In another investigation (Dubrovskis et al., 2018) was yield of biogas from grains residues average $0.721 \pm 0.06 \text{ L g}^{-1}_{\text{DOM}}$ and methane $0.376 \pm 0.02 \text{ L g}^{-1}_{\text{DOM}}$. But preliminary investigation results (Dubrovskis & Plume, 2017b) were following: yield of biogas - $0.517 \pm 0.06 \text{ L g}^{-1}_{\text{DOM}}$ and methane $0.268 \pm 0.03 \text{ L g}^{-1}_{\text{DOM}}$. The great difference in results can be explained by the composition of the different grains residues and how many in there are whole grains.

The aim of this work is to find out the suitability of three different bioethanol waste products - stillage processing residue, wheat bran and grain residues for biogas production.

MATERIALS AND METHODS

The stillage contains not less than 17 different amino acids, the total content of which is 35.6% of the absolute dry matter. Carbohydrates account for an average of 13.5%, fat for 7–8% and mineral salts for 2.4%. One of the most valuable properties of the stillage is that the stillage contains the full spectrum of the B group vitamins, as well as vitamin B (folic acid), tocopherol, ergosterol, which are the regulators of animal metabolism. The dry matter of the stillage is also characterized by the presence of trace elements such as iron, zinc, manganese, copper, etc. rich content. After nutritional value, the stillage dry matter exceeds the standard compound feed and bran. Protein is produced from the stillage. Feed protein contains a large amount of raw protein that reaches and exceeds 37%, and is equivalent to sunflower cake protein after use efficiency and nutritional value. This amount of protein is determined by the course of yeast life processes during the fermentation of the alcoholic raw materials. Protein production from the stillage process produces a liquid residue. The stillage processing residue is shown in Fig. 1. This is the product that results from the residue in the protein product manufacturing process at the bioethanol plant.

Wheat bran is a product of grain milling residue. Grain casings consist mainly of fibre (cellulose, hemicellulose and lignin), minerals (potassium, calcium, magnesium, iron, etc.), group B vitamins, carotenoids and proteins. Grain germ contains fats of high-quality fatty acids (linoleic acid, linolenic acid, monounsaturated oleic acids). Grinding the grains, the casings and germ of high-quality nutrients are mechanically separated, bran still contains many valuable substances facilitating also anaerobic digestion process. Bran is an excellent product that mechanically cleans the digestive tract while providing the body with many high-quality substances. There are few simple sugars in bran, but they are very rich in protein and contains also soluble fibre. Fibres have a high absorption capacity, which absorbs 25 times more water than their volume.

Grain residues are very different depending on the type of grain have been treated to dryer. Also, the content of biogas and methane varies depending on the grain composition and content of the husks. The wheat and triticale grains residues of JP Iecava plant are shown in Fig. 3.

In the investigation, digestate, which was taken from the bioreactor of the Bioenergy Laboratory, operating with the cows manure in a continuous mode, was used. Wheat bran, grain residues and stillage processing residue (Figs 1, 2, 3) from JP Iecava's bioethanol plant were used as raw materials for anaerobic fermentation research.



Figure 1. Stillage processing residue.



Figure 2. Wheat bran.



Figure 3. Grain residues.

The methodology described below and similar with German VDI 4630 (VDI 4630, 2006) guideline and the German Methodenhandbuch Energetische Biomassenutzung (Thran, 2010) were used for the present study. The widely applied methods (Angelidaki et al., 2009) were used for the AD process investigation in 16 experimental bioreactors with volume of 0.75 litres. 2 bioreactors for control were filled with 400.0 ± 0.2 g inoculums and rest bioreactors were filled with mixtures of inoculums (400 g) and added biomass, according to experimental plan, see Table 1. Dry organic matter (DOM) content was determined by weighting of the initial biomass samples, drying in dry matter weights Shimazu at $105\text{ }^{\circ}\text{C}$ and then placed for ashing in oven ('Nabertherm' type) at $550\text{ }^{\circ}\text{C}$. All the components were carefully mixed together and filled in bioreactors. All bioreactors were placed into heated thermostat SNOL in the same time before starting of anaerobic digestion. Gas released from each bioreactor was collected in storage bag positioned outside of the thermostat container. Gas volumes were measured using flow meter (Ritter drum-type gas meter). The composition of gases, including oxygen, carbon dioxide, methane, and hydrogen sulphide was measured help by gas analyser (model GA 2000). The substrate pH value was measured before and after finishing off the AD process, using a pH meter (model PP-50) with accessories. Scales (Kern, model KFB 16KO2) was used for weighting of the total weight of substrates before and after the AD process. Fermented cattle manure (from 120 L bioreactor working in continuous mode) was used as the inoculum. Batch mode AD process was ongoing at temperature $38 \pm 0.5\text{ }^{\circ}\text{C}$. Biogas released was collected in gas bags for further measurements of gas volume and elemental composition. Biogas and methane volumes and gases composition were measured during AD process at regular time intervals. The AD process was provided until biogas emission ceases. Obtained experimental data were processed using appropriate statistical methods.

Three bioreactors (R2–R4) were filled with 400 g of inoculum (digestate) (weighing up to 0.2 g accuracy) and 10 g of wheat bran WB. The other two bioreactors (R5–R6) were filled with 400 g of inoculum (weighing up to 0.2 g) and 10 g of wheat bran and 100 g of stillage processing residues. The other two bioreactors (R7–R8) were filled with 400 g of inoculum and 5 g of wheat bran and 100 g of stillage processing residues. The R9–R11 bioreactor was filled with every 400 g of inoculum and 10 g of grain residues. Other two bioreactors (R12–R13) were filled with 400 g of inoculum and 10 g of grain residues and 100 g of stillage processing residues. Other two bioreactors (R14–R15) were filled with 400 g of inoculum and 5 g of grain residues and 100 g of stillage processing residues. In the bioreactor R1, R16 was filled with 400 g of inoculum - digestate (control sample) each. All data was recorded in the experiment log and on the computer. All bioreactors were connected to calibrated gas storage bags and taps, placed in an oven and set at a working temperature of $38 \pm 0.5\text{ }^{\circ}\text{C}$. The amount and composition of the released gas was measured daily. Bioreactors were also shaken daily by mixing the substrate to wet and reduce the floating layer. The fermentation took place in a single filling (batch) mode and lasted until the biogas was released (25 days).

RESULTS AND DISCUSSION

The data on sample analysis and on amount of biogas and methane produced was estimated for all 16 bioreactors, and average results were calculated. The LUA

laboratory identified the main organic matter composition of the wheat bran sample: Protein 15.18%; Lipids 4.58%; Carbohydrates 16.83%.

The results of raw material analyses before anaerobic digestion are shown in Table 1.

Table 1. Results of analysis of raw materials

Raw material	pH	TS %	TS g	ASH %	DOM %	DOM g	Weight g
R1, R16 inoculum 400 g In	7.5	4.99	19.96	15.69	84.31	16.828	400
R2–R4 10 g WB		85.66	8.566	9.76	90.24	7.730	10
R2–R4 400 g In+10 g WB	7.5	6.96	28.526	13.91	86.09	24.558	410
R5–R6 10 g WB+400 g In+100 g SPR		5.89	30.026	13.47	86.53	25.982	510
R7–R8 5 g WB +100 g SPR+400 g In		5.10	25.743	14.09	85.91	22.117	505
R9–R11 10 g GR		88.35	8.835	13.39	86.61	7.652	10
R9–R11 10 g GR+400 g In		7.02	28.795	14.99	85.01	24.480	410
R12–R13 10 g GR+400 g In+100 g SPR		5.94	30.295	14.49	85.51	25.904	510
100 g SPR		1.50	1.50	5.10	94.90	1.424	100
R14–R15 5 g GR +400 g In+100 g SPR		5.12	25.878	14.68	85.32	22.078	505

Abbreviations: TS – total solids; ASH – ashes; DOM – dry organic matter; In – inoculums, 400 g In – 400 g inoculum; WB – wheat brans; GR – grains residues; SPR – stillage processing residues; 100 g SPR – 100 g stillage processing residues.

The results of biogas and methane from all raw materials are shown in Table 2 and in the figures. The table shows the results from the R2–R15 bioreactors, where the amount of gas obtained from the inoculum is already calculated.

Table 2. Biogas and methane yields

Raw material	Biogas, L	Biogas, L g ⁻¹ _{DOM}	Methane, aver. %	Methane L	Methane, L g ⁻¹ _{DOM}
R1 400 g In	0.3			0.09	
R16 400 g In	0.6			0.089	
R2WB10 g+ In 400 g	9.2	1.19	53.69	4.939	0.639
R3 WB 10g+ In 400 g	9.0	1.164	52.06	4.684	0.606
R4 WB10 g+ In 400 g	8.5	1.100	48.64	4.134	0.535
R2–R4	8.9	1.151	51.46	4.586	0.593
± st.dev.	± 0.36	± 0.046	± 2.58	± 0.411	± 0.053
R5 WB 10 g+ In 400 g+100 SPR	10.5	1.147	55.97	5.924	0.647
R6 WB 10 g+ In 400 g+100 SPR	9.6	1.049	52.72	5.065	0.553
R5–R6 WB	10.05	1.098	54.35	5.495	0.600
± st.dev.	± 0.64	± 0.069	± 2.30	± 0.607	± 0.066
R7 WB 5 g+ In 400 g+100 SPR	7.2	1.361	51.95	3.740	0.707
R8WB 5 g+ In 400 g+100 SPR	6.4	1.210	55.28	3.539	0.669
R7–R8	6.8	1.286	53.62	3.640	0.688
± st.dev.	± 0.57	± 0.107	± 2.35	± 0.142	± 0.027
R9 GR10 g+ In 400 g	7.7	1.006	47.22	3.633	0.475
R10 GR 10 g+ In 400 g	6.6	0.863	50.98	3.364	0.440
R11 GR 10 g+ In 400 g	6.7	0.876	50.11	3.363	0.439
R9–R11GR	7.0	0.915	49.44	3.453	0.451
± st.dev.	± 0.61	± 0.079	± 1.97	± 0.156	± 0.021

Table 2 (continued)

R12 10GR+ 100 SPR + In 400 g	6.8	0.749	55.27	3.757	0.414
R13 10GR +100 SPR + In 400 g	7.6	0.837	51.59	3.909	0.431
R12–R13	7.2	0.793	53.43	3.833	0.423
± st.dev.	± 0.57	± 0.062	± 2.60	± 0.107	± 0.012
R14 5GR +100 SPR + In 400 g	5.3	1.01	50.30	2.666	0.508
R15 5GR +100 SPR + In 400 g	5.3	1.01	53.28	2.824	0.538
R14–R15	5.3	1.01	51.79	2.745	0.523
± st.dev.	± 0.0	± 0.00	± 2.11	± 0.112	± 0.021

Abbreviation: L g⁻¹DOM – litres per 1 g dry organic matter added (added fresh organic matter into inoculum).

From the table data it was estimated that the inoculum (digestate) was still slightly digested. The digest of DOM cannot completely decompose because it contains many microorganism cells.

As shown in the table, methane was extracted more from bio-reactors, where 10 g of wheat brans and 100 g of stillage processing residue were filled. Also in bio-reactors, where 10 g of grain residues and 100 g of stillage processing residue, methane was formed more than in those bioreactors containing only 10 g of wheat bran or 10 g of grain residues. This shows that adding 100 g of the stillage processing residue is useful. Methane is derived from wheat bran more than from grain residues. Specific biogas and methane yields from bioreactors filled with wheat brans, grain residues and stillage processing residues shown in Fig. 4.

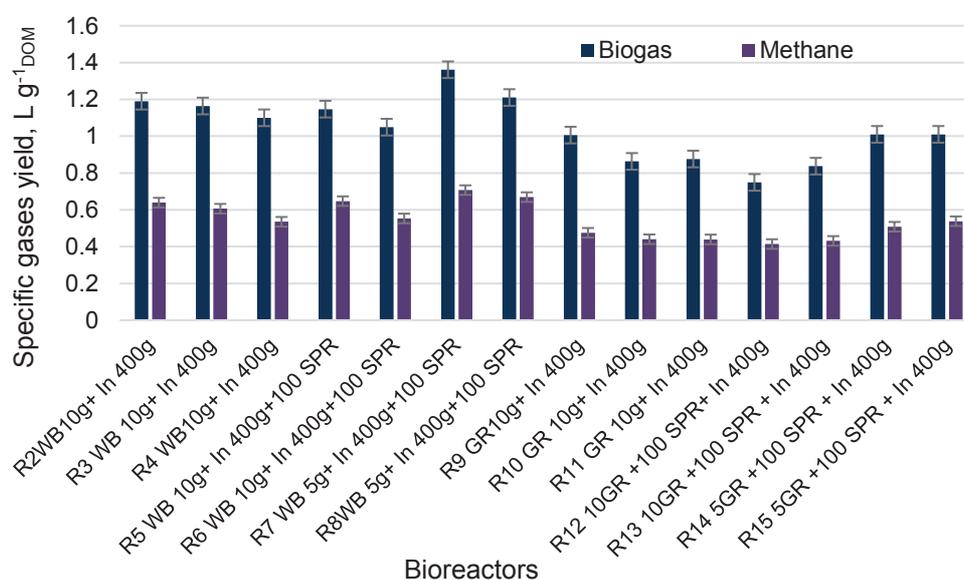


Figure 4. Specific biogas and methane yields from bioreactors filled with wheat brans, grain residues and stillage processing residues.

As seen from the figure, most methane is obtained from bioreactors, where 100 g of stillage processing residue was added to wheat bran and grain residues 5 g. From the bioreactors, where 10 g of wheat bran and the grain residues were filled, were obtained

less methane, because the optimum organic load was obviously exceeded and the AF process slightly inhibited.

Fig. 5 shows the average methane content of each bioreactor with wheat bran, grain residues and stillage processing residue.

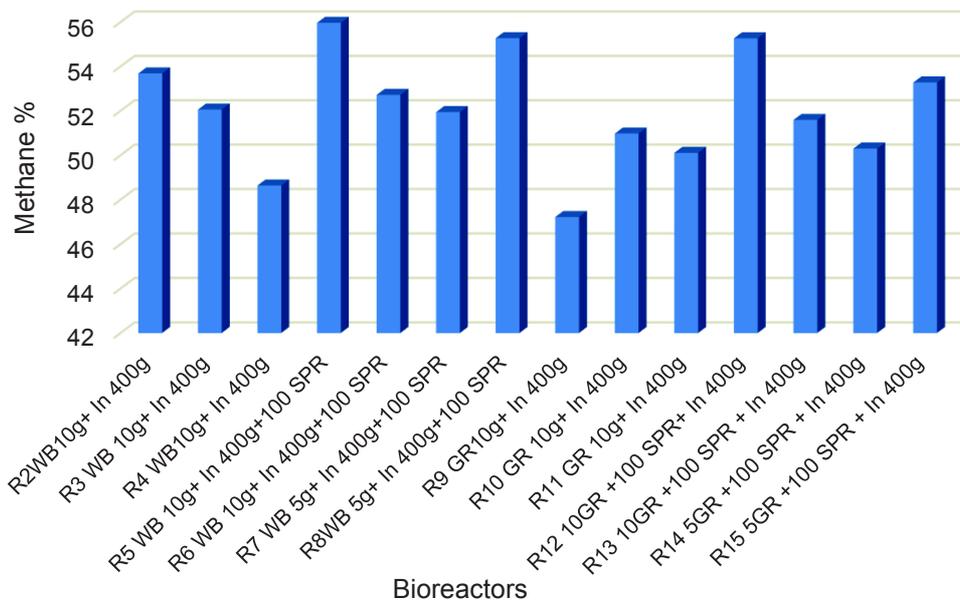


Figure 5. Average methane content of each bioreactor with wheat bran, grain residues and stillage processing residue.

To find out how fast methane production took place in the bioreactors of each raw materials group, the results were analyzed on average after 7, 14 and 25 days (Table 3).

Table 3. Average biogas and methane contents and yields after 7, 14 and 25 days

Bioreactor	Biogas, L			Methane, %			Methane, L			Methane, L g _{DOM} ⁻¹		
	7d	14d	25d	7d	14d	25d	7d	14d	25d	7d	14d	25d
R2–R4	6.033	8.33	8.9	74.1	47.37	51.46	2.944	4.374	4.586	0.381	0.566	0.593
R5–R6	4.95	8.4	10.1	74.95	60.65	54.34	2.369	4.709	5.495	0.259	0.514	0.600
R7–R8	5.6	6.2	6.8	72.25	46.55	53.62	3.205	3.57	3.64	0.606	0.675	0.688
R9–R11	4.2	6.17	7.0	67.23	54.87	49.44	1.876	3.071	4.455	0.245	0.402	0.451
R12–R13	4.45	6.15	7.2	73.55	47.3	53.38	2.607	3.646	3.833	0.287	0.402	0.423
R14–R15	4.35	4.95	5.3	66.1	49.55	51.78	2.322	2.642	2.745	0.442	0.504	0.523

The production of methane L g⁻¹_{DOM} from wheat bran and wheat bran with stillage processing residue after 7, 14 and 25 days is shown in Fig. 6. Methane produced very fast in bioreactors, with 5 g of WB and 100 g of stillage processing residues. Already in the first week, 88.05% of the total 25-day production of methane was produced. This proves that this proportion of wheat bran and stillage processing residues is very good and provides the correct AF process. The results obtained compared to the results of

other biomass are very good. Also, wheat bran and grain residues produce higher yields of methane as shown by other researchers and obtained from our previous research. This could be explained by the good quality of these biomass.

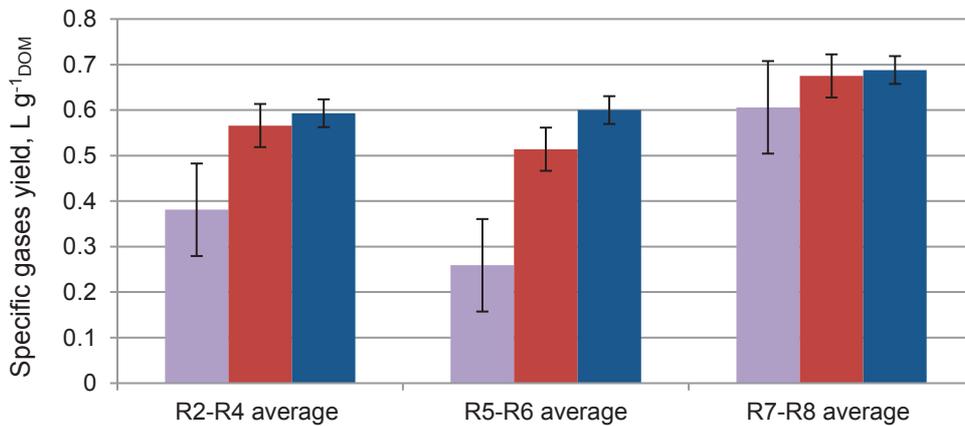


Figure 6. The production of methane L g⁻¹DOM from wheat bran and wheat bran with stillage processing residue after 7, 14 and 25 days.

Methane L g⁻¹DOM from grain residues and stillage processing residues is shown in Fig. 7. Extracting it to organic dry matter content exceeded not only the obtained from 10 g grain residues, but also from the 10 g grain residues and 100 g stillage processing residues. This can be explained by the inhibition of the AF process due to organic overload. Here, inhibition was greater than that of bioreactors with wheat bran.

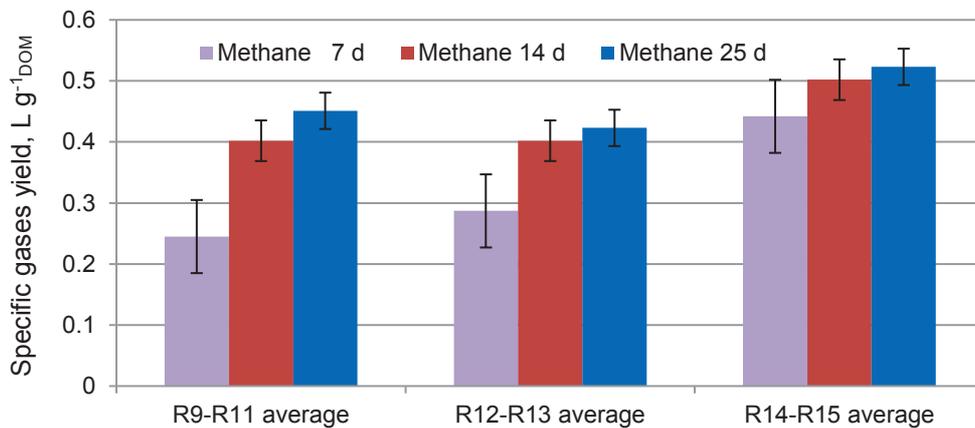


Figure 7. Methane L g⁻¹DOM from grain residues and stillage processing residues.

The results obtained from wheat bran, stillage processing residue and grain residues from natural mass are summarized in Table 4.

Table 4. The results obtained from wheat bran, stillage processing residue and grain residues from natural mass

Raw material	TS%	DOM%	Biogas L g ⁻¹ _{DOM}	Methane L g ⁻¹ _{DOM}	Vnm biogas m ³ t ⁻¹	Vnm methane m ³ t ⁻¹
WB	85.66	90.24	1.191	0.593	920.63	458.39
WB10+100SPR	9.15	90.94	1.098	0.600	91.36	49.93
WB5+100SPR	5.50	91.46	1.286	0.688	64.69	34.61
GR	88.35	86.61	0.915	0.451	700.15	345.10
GR10+100SPR	9.40	87.82	0.793	0.423	65.46	34.92
GR5+100SPR	5.64	88.71	1.010	0.523	50.53	26.17

Abbreviation: Vnm – volume obtained from natural mass.

CONCLUSIONS

1. Both wheat bran and grain residues produced a high yield of methane. Such raw materials can be well used in Latvian conditions.

2. When adding 100 g of stillage processing residue to wheat bran, methane yields increased, but only slightly when the optimum organic load was exceeded (10 g WB + 100 g SPR).

3. Adding 100 g of stillage processing residue to grain residues increased the yield of methane, but only did not when the optimum organic load was exceeded (10 g GR + 100 g SPR).

4. When 100 g of stillage processing residues were added to 5 g wheat bran, the methane yield increased by 16.02% compared to bioreactors with only wheat bran, although twice more. This proves the good effect of stillage processing residue.

5. When 5 g of grain residues was added to 100 g of stillage processing residues, the methane yield increased by 15.96% compared to bioreactors, containing 10 g of grain residues. It also proves the good effect of stillage processing residue.

6. In bioreactors, where the stillage processing residues were filled, the anaerobic fermentation process starts more rapidly and the organic matter decomposed more rapidly.

7. Comparison of raw materials by natural methane yields shows that wheat bran, which can yield 458.39 m³ t⁻¹, is the most valuable raw material. This is very good compared to other biomasses. A good raw material is also a grain residue that can produce 345 m³ t⁻¹ methane.

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