

CO FERMENTATION OF SUGAR BY-PRODUCTS WITH TYPICAL AGRICULTURAL SUBSTRATES

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Summary. Anaerobic digestion (AD) is a promising option for the environmentally friendly recycling of agricultural by-products. However, overloading of the digester with sugar, starch or protein might cause inhibition of the anaerobic processes. The aim of the present project was to investigate the influence of sugar beet by products on biogas yield from a typical mixture of energy crops and animal manure.

The investigated substrates have been: cattle slurry, maize, sorghum and grass silage, sugar beet pulp e (SBP) and sugar beet tail silage (SBT). The difference between untreated SBT to processed SBP. All substrates were digested in 1 l eudiometer-batch digesters at 37.5°C during 28 to 38 days. The specific methane yield of mixtures and various substrates examined. The experiments showed that edition of sugar beet by product to energy crop and slurry mixture results in high methane yield even the achieved methane yield of the mixture was lower the expected.

Key words: anaerobic digestion, biogas, methane yield, by-products, sugar beet pulp, sugar beet tail, potato peel pulp, potato fruit water.

INTRODUCTION

The worldwide high consumption of primary energy is not only causing the global climate change, but as well is the available of primary energy limited. Therefore, the primary energy sources need to be replaced by renewable ener-

gy sources. The energy recovery of animal manure, other organic wastes and energy crops in biogas plants gives a perspective to substitute natural gas by bio-methane. A new European study showed that about 500 billions m³ of bio-methane per year can be produced from organic wastes and energy crops [13]. This amount of bio-methane could completely cover the current demand of natural gas in Europe. Beside the independence of natural gas supplies, the usage of the biogas technology contributes to the reduction of greenhouse gas emissions, the development of rural areas and the creation of new jobs.

The limited knowledge of anaerobic digestibility and methane yield of by-products of sugar beet and starch potato processing is limiting their usage on the one hand and may cause biological-brake down of the biogas plants on the other hand.

Except of the present project, little work on AD and methane yield of by-products from the sugar and starch industry has been done [1, 6]. The low pH value and the high protein and sugar contents in these substrates may cause an acidification of the digester and therefore an inhibition of the methane production [9]. To avoid this danger in biogas plants, these by-products need to be investigated in laboratory experiments and the development of important process parameters has to be recorded. The most important parameters to indicate a possible inhibition of the AD process are: pH, volatile fatty acids and ammonia concentration. Beside these

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process parameters, it is also important to have knowledge about the development of the biogas composition (methane, hydrogen sulphide and carbon dioxide) during the AD.

The objectives of the present project was to determine the suitable volume and the co-fermentation effects of sugar beet by products within the mixture of other agricultural substrates and manure for biogas production.

MATERIAL AND METHODS

2.1. Substrates

Sugar beet pulp (SBP) and sugar beet tail (SBT) were collected as silages from the AGRANA Zucker Ges.m.b.H. in Tulln, Austria. The proofed mixture of agricultural substrates consists of cattle slurry, maize and sorghum was collected on the Farms in Lower Austria.

2.1.3. Inoculum

Active sludge from a commercial biogas plant in Lower Austria (table 1) was used as inoculum. The substrates of the biogas plant were vegetables, maize silage and sunflower silage. The inoculum was collected from the last part of the horizontal fermenter into a 50 l heatable container. Before sampling the transport

container was filled with argon to insure anaerobic conditions inside.

2.1.1. Determination of methane potential (Experiment A)

The present study included 14 experimental variants. There of six variants were explored in mono digestion. Sugar by-products were analyzed as silage and as dried material. To determine the co-fermentation effects of sugar by-products 6 mixtures with different content (30, 50 and 70% DM) of SBP and SBT were also digested. In the course of the experiment the fermentation process were detailed monitored to recognize any inhibitions or co-fermentation effects of different variants.

2.2. Anaerobic digestion experiments – Determination of the biochemical methane potential

The biochemical methane potential of the by-products was determined in 1 l eudiometer-batch digesters at 37.5°C.

Table 2 shows the nutrient content of the inoculum. In the course of the AD experiment in the laboratory, the specific methane potential of the inoculum was measured as well. The inoculum showed a low specific methane potential of only 15 l_N (kg VS)⁻¹.

Table 1. Parameters of the biogas plant from which the inoculum was taken

| Parameter | |
|-----------------------------|--|
| Digester type | Horizontal plug flow digester 1 mixing tank 193 m ³ |
| Digester | 4 horizontal plug flow digesters 160 m ³ each 1 vertical second stage digester 1885 m ³ 1 storage tank (uncovered) 4825 m ³ |
| Digested substrates | Energy crops, vegetables |
| Temperature in the digester | 37°C |
| Ø hydraulic retention time | 15 days h. digester + 55 days second stage |
| Electrical output | 330 kW |
| Energy production | 2 475.000 kWh a ⁻¹ |

Table 2. Nutrient content of inoculum

| Substrate | XP | XL | XF | XA | XX | N | C | GE | C/N | pH | DM | VS |
|-----------|------|-----|------|------|------|-----|------|------------------|-----|-----|-----|------|
| | % | % | % | % | % | % | % | MJ | | | % | % |
| | DM | DM | DM | DM | DM | DM | DM | kg ⁻¹ | | | FM | DM |
| | | | | | | | | DM | | | | |
| Inoculum | 14.5 | 0.8 | 10.0 | 47.2 | 27.5 | 6.3 | 27.7 | 18.0 | 4.4 | 7.4 | 2.4 | 52.8 |

XP = crude protein, XL = crude lipids, XF = crude fiber, XA = crude ash, XX = N-free extracts, N = nitrogen, C = carbon, GE = gross energy, DM = dry matter, FM = fresh matter, VS = volatile solids

The experiments were carried out in accordance with VDI 4630 [15] and DIN 38 414–8 [5].

Prior to AD, samples of all substrates were analysed for pH, DM, VS, crude protein, crude lipids, crude fibre, crude ash, N-free extracts, nitrogen and carbon using standard analysing procedures according to VDLUFA Band II.I [16] and VDLUFA Band III [16]. The gross energy content was measured with a calorimeter.

The substrates were digested together with 350 g inoculum. That means on average the DM ratio between substrate and inoculum was 1:3. The DM content in the digesters with SBP and SBT ranged from 3.8 to 4.0%, the DM content in the digesters with PP, PPP and PFW from 3.0 to 3.1% DM.

Each eudiometer consists of six digesters connected to equilibrium vessels, with a septum for gas extraction (Fig. 1). The digesters were placed on magnetic stirrers in a tempered water bath. Specific methane yield from each substrate was measured in three replicates. During AD, the digester content was mixed for 10 minutes every 30 minutes. Biogas was collected in gas-collection tubes connected to the digesters. The amount of biogas produced was monitored every day. Biogas quality (methane, hydrogen sulphide and ammonia) was analysed six times during the experiments. Methane (CH₄) concentration in the biogas was measured using a NDIR analyser (Dräger X-am 7000, Dräger Safety, Lübeck, Germany) with an accuracy of $\pm 1\text{--}3\%$ of the measurement reading. Before each measurement, the analyser was calibrated with CH₄ calibration gas containing 60% CH₄

and 40% CO₂. NDIR readings were validated at regular intervals with gas chromatographic analysis. Hydrogen sulphide (H₂S) and ammonia (NH₃) concentration in the biogas were analysed with the NDIR analyser in combination with Dräger tubes (accuracy $\pm 5\text{--}10\%$ and $10\text{--}15\%$ of the measurement reading, respectively). The biogas and methane production from the inoculum alone was also measured and subtracted from the biogas and methane production from the digesters containing the substrates and inoculum. The specific biogas and methane yields were calculated on the basis of norm conditions: 273 K and 1013 mbar and are given in norm litre per kg of volatile solids (l_N kg VS). In addition, the coefficient of energy efficiency of AD (η) was calculated for each substrate. This coefficient relates the produced methane energy to the gross energy of the substrate.

To control the quality and stability of the fermentation process, measurements of pH were done every second to third day and volatile fatty acids were measured twice during the experiment, at the beginning and at the end using gas chromatography. The fatty acid spectrum examined was C1-C6: acetic acid (HAC), propionic acid (PRO), iso butyric acid (i-BUT), butyric acid (n-BUT), iso valeric acid (i-VAL), valeric acid (n-VAL) and caproic acid (CAP).

2.3. Statistical data analysis

Statistical data analysis was carried out using the software package SPSS (version 12.0, SPSS Inc. 2006).

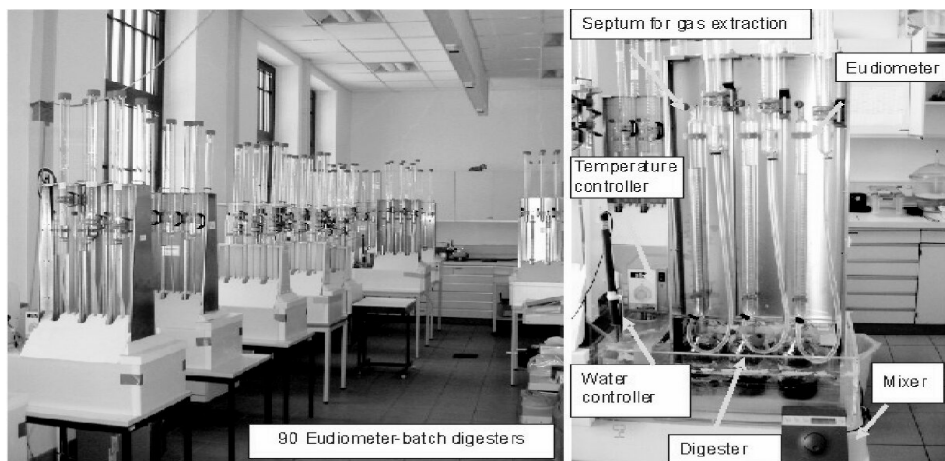


Fig. 1. Eudiometer-batch digester system

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In first step, the descriptive statistics were done, determining means, standard deviations and frequency distributions of the data. Differences in the specific biogas and methane yields were tested with a pair wise comparison of regression parameters by the Tukey-HSD-Test and T-Test. The level of significance was set to 0.05.

RESULTS AND DISCUSSION

3.1. Volatile fatty acid concentrations and pH during anaerobic digestion

The AD process of all substrates was carried out under optimal mesophilic conditions. The average temperature was 37.5°C and the pH values in the experiments ranged between 7.29 and 7.85. For SBP and SBT, at the beginning of the experiment the pH was 7.29 and 7.85, respectively. At the end of the experiment the pH for SBP and SBT was 7.34 and 7.79, respectively. That means during the whole experiment, the pH was lower in the digesters with SBP compared to digesters with SBT. From the beginning to the end of the experiment, the concentrations of acetic, propionic and butyric acid decreased in the digesters with SBP from 969 to 96.7, 113 to 4.2 and 8.8 to 0 mg l⁻¹, respectively. For SBT the values decreased from 791 to 58.0, 114 to 4.7 and 11.0 to 0 mg l⁻¹, respectively. The high concentrations of acetic and propionic acid at the beginning of AD are typical for the batch digester experiments. The low concentrations of acetic and propionic acid at the end of AD is a sign that the AD was not inhibited and the substrates were almost completely digested.

The pH was in all experimental variants in the range of 7.1 at the beginning of fermentation to 7.7 to 8.2 at the end of fermentation. Thus, there was optimum pH environment for the bacteria in the fermenters in experiment from the perspective of the. The optimal environment for the bacteria to a pH is between 6.4 and 8.0 (VDI 4630). If the pH is outside this range, there may be a worse gas yield and gas composition with a higher CO₂ content.

According to Wellinger [17], the AD runs optimal if the concentration of acetic, propionic

and butyric acid is less than 1000, 200 and 50 mg l⁻¹, respectively and the value for HAC/PRO lies between 5 and 10. When the total concentration of volatile fatty acids exceeds 3000 mg l⁻¹ or the propionic acid concentration becomes higher than 300 mg l⁻¹, an inhibition of the AD can take place. In the present experiments, except for PFW, the measured acetic acid concentrations were less than 1000 mg l⁻¹ (Fig. 2.). However, with SBT the total concentration of volatile fatty acids did not exceed 3000 mg l⁻¹ and with none of the substrates a propionic acid concentration higher than 300 mg l⁻¹ was measured. This demonstrates that in the present experiments the AD should not be inhibited.

3.2. Composition of the produced biogas

Table 3 displays the average composition of the biogas produced. Six times during the experiment the concentration of methane, hydrogen sulphide and ammonia were measured. The differences between the variants were not significant because the composition of the produced biogas varied during the experiments. In both experiments the concentrations of methane, hydrogen sulphide and ammonia increased during the first five days, then were more or less stable for the following 20 days and slightly decreased towards the end of the experiments. The present data are comparable with literature data [6, 8].

With regard to the by-products of sugar beet processing, SBP had higher concentrations of methane, hydrogen sulphide and ammonia compared to SBT (table 3).

As we can see the average methane concentration of grass and sorghum was higher than from the other substrates. The drying of sugar beet pulp silage reduced the methane content. It could be caused by the evaporation of fatty acids during drying process. The Mixtures with SBP shown a little higher methane content in biogas compared to the mixtures with SBT.

3.3. Specific biogas and methane yields as well as energetic efficiency of the investigated substrates

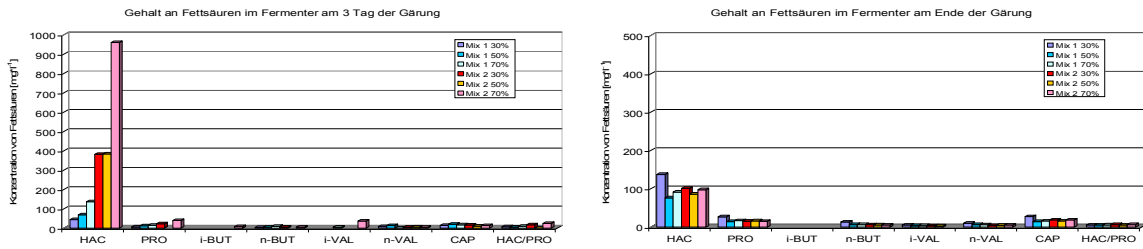


Fig. 2. Concentration of fatty acids in the fomenter to begin and to the end of digestion

Table 3. Concentration of methane (CH₄), hydrogen sulphide (H₂S) and ammonia (NH₃) in the biogas

| Variant | CH ₄ -Content | | | H ₂ S- Content | | | NH ₃ - Content | | |
|------------------------------------|--------------------------|---|------|---------------------------|---|-----|---------------------------|---|----|
| | % | n | ± | % | n | ± | % | n | ± |
| cattle slurry | 53,0 | 7 | 8,8 | 267 | 6 | 112 | 26 | 3 | 11 |
| maize | 55,1 | 7 | 3,7 | 214 | 6 | 58 | 29 | 3 | 16 |
| sorghum | 57,2 | 7 | 4 | 213 | 6 | 49 | 29 | 3 | 13 |
| grass | 57,6 | 7 | 4,2 | 281 | 6 | 149 | 32 | 3 | 30 |
| pressed beet pulp silage | 50,9 | 7 | 7,1 | 321 | 6 | 74 | 37 | 3 | 11 |
| beet-tail silage | 49,6 | 7 | 5 | 174 | 6 | 100 | 30 | 3 | 7 |
| Mix 1 30% | 56,7 | 7 | 3,7 | 209 | 6 | 23 | 33 | 3 | 1 |
| Mix 1 50% | 57,0 | 7 | 2,9 | 362 | 6 | 51 | 35 | 3 | 4 |
| Mix 1 70% | 57,3 | 7 | 3,3 | 176 | 6 | 97 | 32 | 3 | 10 |
| Mix 2 30% | 53,6 | 7 | 5,4 | 358 | 6 | 118 | 16 | 3 | 8 |
| Mix 2 50% | 54,7 | 7 | 7 | 387 | 6 | 45 | 16 | 3 | 13 |
| Mix 2 70% | 55,0 | 7 | 7,3 | 350 | 6 | 82 | 17 | 3 | 10 |
| pressed and dried beet pulp silage | 46,2 | 7 | 12,8 | 250 | 6 | 127 | 41 | 3 | 38 |
| dried beet-tail silage | 54,2 | 7 | 5,1 | 355 | 6 | 99 | 31 | 3 | 20 |

Table 4. Specific biogas and methane yield

| Variante | Biogas yield [Nl *(kg oTS)-1] | | | Methane yield [Nl *(kg oTS)-1] | | |
|------------------------------------|-------------------------------|---|--------|--------------------------------|---|--------|
| | Av | n | St.div | Av | n | St.div |
| cattle slurry | 249 | 3 | 2,6 | 132 | 3 | 0,5 |
| maize | 782 | 3 | 86,8 | 431 | 3 | 42,5 |
| sorghum | 608 | 3 | 26,8 | 348 | 3 | 14,9 |
| grass | 668 | 3 | 15,5 | 385 | 3 | 9 |
| pressed beet pulp silage | 845 | 3 | 33,3 | 430 | 3 | 18,1 |
| beet-tail silage | 970 | 3 | 68,7 | 481 | 3 | 32,4 |
| Mix 1 30% | 372 | 3 | 27,1 | 211 | 3 | 16,1 |
| Mix 1 50% | 405 | 3 | 15,5 | 231 | 3 | 8,1 |
| Mix 1 70% | 517 | 3 | 9,2 | 296 | 3 | 16,1 |
| Mix 2 30% | 668 | 3 | 24,0 | 358 | 3 | 10,2 |
| Mix 2 50% | 707 | 3 | 23,3 | 387 | 3 | 14,3 |
| Mix 2 70% | 812 | 3 | 50,0 | 447 | 3 | 24,9 |
| pressed and dried beet pulp silage | 641 | 3 | 21,4 | 296 | 3 | 13,2 |
| dried beet-tail silage | 506 | 3 | 27,9 | 274 | 3 | 14,9 |

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3.3.1. Specific biogas and methane yield of by-products of sugar beet processing (Experiment A)

The specific biogas and methane yield of the sugar by-products: sugar beet pulp silage (SBP) and sugar beet tail silage (SBT) were measured over 30 days. The measurements were carried out until the specific methane yield per day was less than 1% of the cumulative specific methane yield.

The specific biogas and methane yields of SBP and SBT were significantly different. With SBT the specific biogas and methane yields were higher. On average a specific methane yield of $481 \text{ l}_N (\text{kg VS})^{-1}$ was measured for SBT, whereas for SBP the specific methane yield was $430 \text{ l}_N (\text{kg VS})^{-1}$. In the literature similar values were reported [4, 6]. For sugar beet silage Hassan (2003) gave the methane yields between 400 and $468 \text{ l}_N \text{ kg}^{-1} \text{ VS}$.

SBT silage showed the highest methane yield of $480 \text{ Nl CH}_4 (\text{kg VS})^{-1}$. The lowest methane yield was achieved from cattle manure. The standard deviation of the average methane yield for the SBP-silage, meadow and Sudan grass silage was significantly lower than of SBT silage and corn silage. This indicates a different homogeneity of the samples.

Table 4 also gives results for η , the energetic efficiency. For SBP on average 87.4% of the gross energy was converted to methane energy. The average value for SBT was 88.5%.

In the literature we found, for SBP silage a specific methane production potential of 400 NL CH_4 per kg VS . SBT silage for a specific methane production potential of $96 \text{ m}^3 / \text{t FM}$ is, 52% CH_4 , 17% TS (Keymer 2002) and $75 \text{ m}^3 / \text{t FM}$ indicated (no indication TS) by Weiland. The specific methane yield from cattle manure, maize and grass silage were also in the folding back from the fields of literature [2, 9, 10, 13, 18, 19, 20, 21].

The efficiency of methane digestion was calculated in accordance with the methane yield and the gross energy content in the biomass. It was 24% for cattle manure, 84% for maize, 64% for sudan grass, 73% in meadow grass, 85% for SBP silage and 89% for SBT silage. The efficiency of methane fermentation shows the energy recovery and fermentability of constituents

of biomass in anaerobic fermentation process. The formula is described in chapter "Material and Methods".

To identify the optimal mixture ratio of SBP silage and SBT silage in the mixture of cow manure, corn silage, to see Sudan grass and grass silage, were digested separately and in the mixtures. The measured specific biogas and methane yields with the standard deviation of three replicates are shown in table 4. As shown in table 4, the biogas and methane yield of the mixtures increased with increasing amount of sugar by-products in the mixture.

Determination of co-fermentation effects

To clarify the co fermentations effects caused by the addition of SBP and SBT silage to the mixtures of cattle manure, maize silage, Sudan grass and meadow grass the substrates were digested in the mixture were digested in the mixture and separately. Based on the determined specific methane yields of the individual separately digested components and their content in the mixtures the expected specific methane yields were calculated.

Figure 3 shows the measured specific methane production potential of the mixtures 1 and 2 with different proportions of sugar beet by-products compared to the expected specific methane yield of these mixtures. As we can see in the fig 6 there was now co-fermentation effect achieved.

The lower achieved as calculated specific methane yield of the mixtures with SBP silage could be possibly caused by reduced activity of cellulolytic bacteria, and thus lower recovery of nutrients from corn, Sudan grass and meadow grass silage.

In animal nutrition we know that allowance of slightly soluble carbohydrates (sugars and starches) in ruminants may reduce the digestibility of other nutrients, particularly of protein and crude fiber. This decrease is referred to as "general digestive depression". According to primarily the cellulolytic bacteria (cellulolytische activity) could be inhibited. This could explain the reduced actual methane yield of the mixtures with SPB silage.

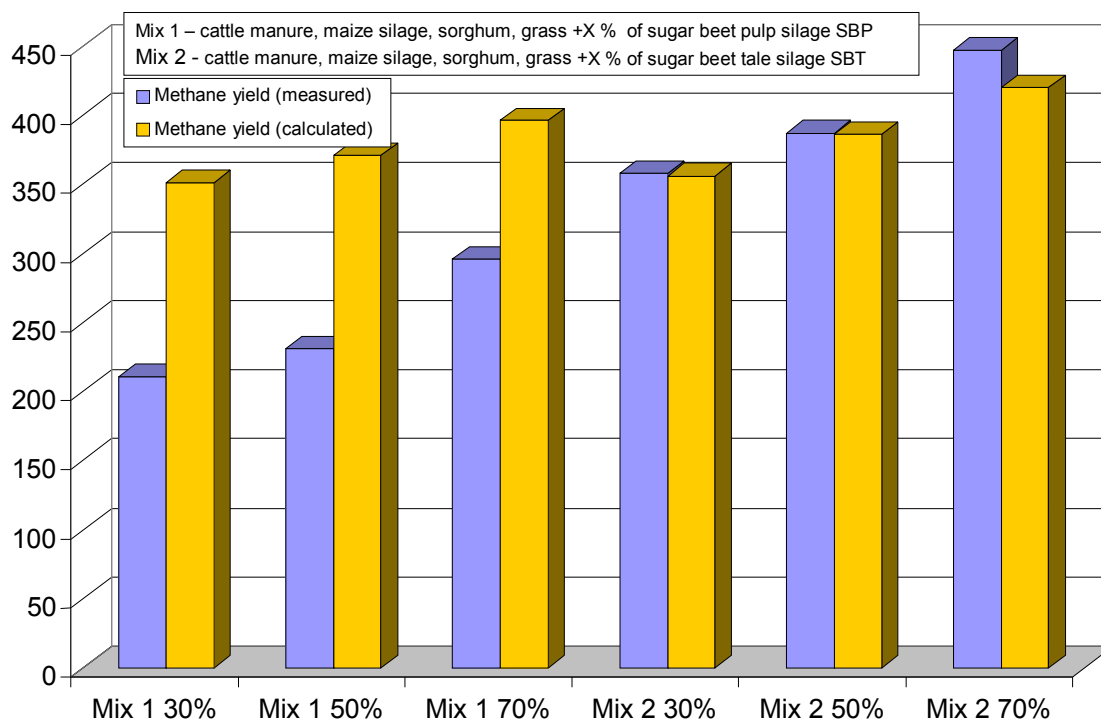


Fig. 3. Measured und calculated methane yield of agricultural substrates (determination of co-fermentation effects)

The mixtures of Group 2 with SBT silage showed only slight co-fermentations effects. The addition of 70% of the ZR-top silage, resulted maximal additional methane yield of 6%.

CONCLUSIONS

The fermentation of all variants was uniformly and stably without significant inhibition of methane fermentation. With increasing content of SBP silage in the mixture the specific methane production potential of the mixture increased. The addition of SBT silage (70% of DM fraction) to the mixture of energy crops and manure resulted in comparison to the monodigestion of the substrates – in a slightly higher methane yield as calculated. In other mixtures there was no co-fermentations effects achieved or they were even negative. For recommendations of the suitability of the ZR-pulp silage as performance-enhancing additive for biogas production, it is reasonable to test the transferability of the present test results in continuous experiments at laboratory scale.

Drying of sugar beet by-products:

The effect of drying of sugar beet-pulp silage and silage on top of their methane potential was tested in the present experiment compared to the non getrockneten ensiled biomass. The

results indicate that the drying of pulp silage-ZR and ZR-top silage to reduce the methane production potential of 30 and 43% resulted. The drying process causes the steaming out of free volatile fatty acids, which were formed during the ensiling process and can thus reduce the methane production potential of biomass.

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КО ФЕРМЕНТАЦИЯ САХАРА ПОБОЧНЫХ ПРОДУКТОВ С ТИПИЧ- НЫМИ СЕЛЬСКОХОЗЯЙСТВЕННЫХ СУБСТРАТОВ

Аннотация. Анаэробные пищеварения (AD) является перспективным вариантом для экологически чистого утилизации сельскохозяйственных побочных продуктов. Тем не менее, перегрузка в варочный котел с сахаром, крахмалом или белком может вызывать ингибирование анаэробных процессах. Целью настоящего проекта было изучение влияния сахарной свеклы по продукции на биогазовой выходом из типичной смеси энергетических культур и навоза.

Исследованные субстраты были: крупный рогатый скот суспензия, кукуруза, сорго и силос, жом с сахарной свеклы, силос (SBT). Разница между необработанной SBT к обрабатываемой СБП. Все основы были переведены в 1 л эвдиометр-пакетных варочных при 37,5 °C в течение 28 до 38 дней. Эксперименты показали, что выход по сахарной свеклы по продукции для энергетических культур и результатов суспензия смеси с высоким выходом метана может быть достигнута урожайность метана в смеси была ниже ожидаемого.

Ключевые слова: анаэробное сбраживания, биогаз, выход метана, побочный продукт, жом сахарной свеклы, картофельные корки, картофельно-фруктовая вода.