

# Anaerobic co-digestion of sewage sludge and molasses

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**Abstract.** The efficiency of simultaneous digestion of sewage sludge and by-product of refining sugar beets (molasses) was investigated. The study was conducted for 28 days under mesophilic conditions. 0.5%, 1%, 1.5%, 2% and 3% (m/m) of molasses was added to the mixture of sludge. The result of the study showed that addition of molasses had positive effect the biogas production. The biggest biogas yield was achieved in sample with 0.5% of molasses (95.69 mL/g VS). In this sample biogas production increased by 21% in comparison with reference sample (without molasses). The biggest methane content (73%) was also observed in the sample with 0.5% of molasses. For comparison in reference sample was produced biogas with 70% content of methane. The dose over 0.5% of molasses caused inhibition of fermentation process. The minimal degree (38%) of degradation of organic matter was achieved in reference sample (38.53%) and in sample with 0.5% of molasses (39.71%) but in other samples was in the range of 35.61-36.76 % (from 3% to 1%, respectively). Digestion process have adverse effect on dewatering properties of sludge. Before co-digestion capillary suction time was from 31 s to 55 s, and after process increased from 36 s to 556 s (from 0% to 3% of molasses, respectively).

## 1 Introduction

Anaerobic co-digestion is process of simultaneous digestion two or more substrates come from different source, occur in four phases: hydrolysis, acidogenesis, acetategenesis and methanogenesis. The addition of co-substrate is the option to overcome the drawback of mono-digestion e.g. low organic load of sewage sludge, low organic load and high nitrogen concentration of animal manures, improper material and relatively high concentration of heavy metals in organic fraction of municipal solid waste, lack of nitrogen in crops and agro-industrial wastes and high concentration of nitrogen and long chain fatty acids in slaughterhouse wastes. Co-digestion is more economical process than mono-digestion because enhance the methane production. Moreover, co-digestion process lead in one fermentation tank could produce more methane than fermentation process conduct in two digesters. Another benefits of co-digestion are acceleration the digestion process and bringing higher degradation rates [1, 2].

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In the world in 2015/2016 year (from October to September) was produced 165.8 million t of sugar from sugar beet [3]. From each kilogram of sugar beet is obtained 75% water and 25% of dry substance. From dry substance is formed a product and the rest is turned into a by-product. The main by-product of the sugar factory are: pulp, carbonation-lime residue and molasses. First of all the sugar beets are washed, sliced and soaked in hot water to begin the process that separates sugar from the rest of the plant. As a result of extraction is made the hot sugary liquid and pulp. To hot sugary liquid is added lime and CO<sub>2</sub> to purification. The mixture is filtered so the runny syrup is made. As a by-product is obtained carbonation-lime residue. The runny syrup is evaporated and crystallization. At the end, hot water is sprayed over the light brown crystals to remove the remaining molasses, leaving pure naturally white sugar crystals. The sugar crystals are then dried [4]. Beet molasses contain 48% sugar (namely sucrose and invert sugar) and 79.5% dry substances. It is the most valuable by-product of the sugar factory because is used as a carbon source in the yeast fermentation and ethanol fermentation, as animal feed, as fertilizer and for biogas production [5, 6]. There are several reports of co-digestion molasses with other substrates [7–9] but is no report about co-digestion of molasses with sewage sludge.

The main objective of this paper was to examine the process of anaerobic co-digestion of sewage sludge and molasses. The attention was focused on biogas production, the composition of biogas, the VFA/A ratio and dewatering properties. The efficiency of the process in terms of molasses ratio was also examined.

## 2 Methods

### *Characteristics of the substrates*

Mixed thickened surplus activated sludge from municipal sewage treatment plant and by-product of refining sugar beets (molasses) was used in this investigation. Surplus activated sludge was obtained from sludge treatment system. Then, surplus activated sludge was inoculated by fermenting sludge from the digester. Molasses was obtain from yeast plant. The sample of molasses were dissolved by deionized water because of thick texture. The characteristics of supernatant liquor and molasses are shown in table 1. The sludge mixture (surplus activated sludge with 50 g of fermenting sludge) was mixed with 2, 4, 6, 8 and 12 g of molasses what corresponds to 0.5%, 1.0%, 1.5%, 2.0% and 3.0% ratio of molasses in the samples. The sludge without co-substrate was a reference. The weight of each sample was 400 g.

**Table 1.** Characteristics of supernatant liquor and molasses.

| Parameter                 | Unit                      | Supernatant liquor | Molasses |
|---------------------------|---------------------------|--------------------|----------|
| pH                        | -                         | 7.1                | 8.5      |
| COD                       | mgO <sub>2</sub> /L       | 603.2              | 13300.0  |
| Volatile fatty acid (VFA) | mg CH <sub>3</sub> COOH/L | < 28               | 545      |
| Alkalinity (A)            | mg CaCO <sub>3</sub> /L   | 475                | 935      |

### *Laboratory installation*

The sludge fermentation study was conducted for 28 days under static conditions, in 12 airtight glass flask (reactors) with a capacity of 500 mL each, at the mesophilic conditions (37°C). To keep temperature constant, the flasks were placed in a water bath. The installation was equipped with a device to collect biogas. The flasks were not mixed, therefore the mixture was stirred 2 times a day without disconnecting devices to receive biogas.

### Analysis

Before and after the experiment the following analyses were carried out:

- total solids (TS) and volatile solids (VS) according to PN-EN 12879:2004 [10] and PN-EN 12880:2004 [11];
- pH according to PN-EN 12176:2004 [12];
- total alkalinity according to PN-91/C-04540/05 [13];
- volatile fatty acid made based on cuvette test Hach Lange;
- capillary suction time according to PN-EN 14701-1 [14].

The volume of biogas was recorded every day and the composition of the produced biogas was measured by Multitec 540 analyser (Sewerin).

## 3 Results and discussion

### Efficiency of co-digestion

The results of the study showed that addition of molasses have positive influence on the cumulative biogas production. Fig. 1 shows the biogas yields in term of VS of the tested ratio of molasses. After 28 days of co-digestion the biggest biogas yield was reached in a sample with 0.5% of molasses (95.69 mL/g VS). In this sample biogas yield increased by 21% in comparison to the reference sample (79.17 mL/g VS). Additionally, in sample with 0.5% of molasses continued increase of biogas production was observed. However, enhancement the content of molasses over 0.5% resulted the inhibition of biogas production due to organic overloading but in sample with 1% of molasses the biogas production (75.55 mL/g VS) was similar to the reference sample. The lowest biogas yield was noticed in the sample when molasses fraction was 3% (27.20 mL/g VS).

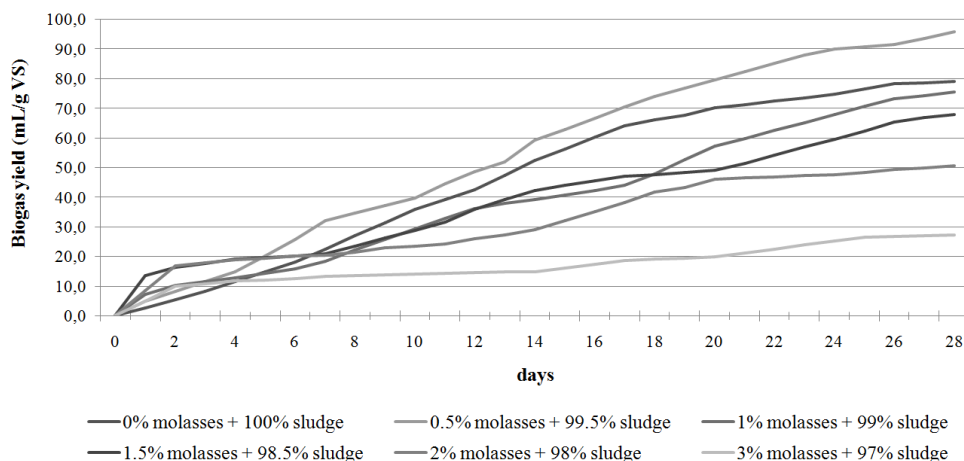
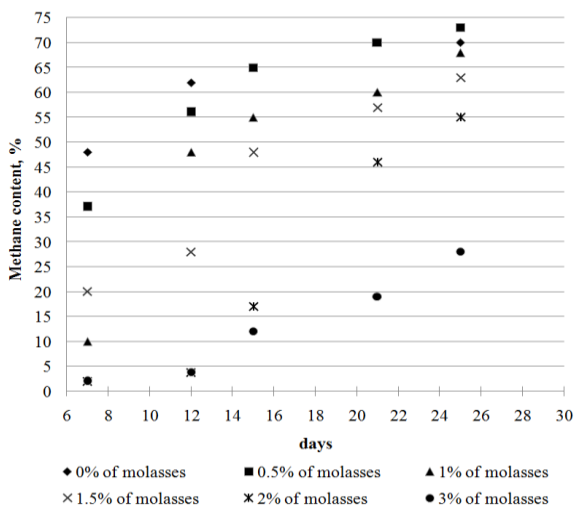


Fig. 1. Biogas yield during anaerobic digestion.

The addition of molasses had impact in the composition of biogas. Biogas produced in the anaerobic digestion of sewage sludge usually contains about 60% methane [15]. The results of this study confirm that addition of substrates had a positive influence on methane content in biogas (Fig. 2). In the reference sample biogas contained up to 70% of methane. The biggest content of methane in biogas (up to 73%) was detected in the sample with 0.5% of molasses. After 14 day of fermentation process in reference sample and in the sample with 0.5% of molasses the same methane content (65%) was reached. This situation continued up to 22 day of fermentation when methane content in both of sample reached 70%. Later methane content in reference sample remained on the constant level. However,

in sample with 0.5% of molasses the ratio of methane enhanced to 73%. It could be explained by a long time adaptation of microorganisms to molasses, probably due to the low susceptibility of molasses to biological degradation under anaerobic conditions. The lowest methane content (28%) was observed in the sample with 3% of molasses. As expected, this result was in agreement with the amount of biogas yield.



**Fig. 2.** The methane content in biogas during anaerobic digestion.

The degree of degradation of organic matter was calculated based on the changes in concentration of volatile solids before and after anaerobic stabilization (Tab. 2). The biggest degree of degradation (39.71%) was observed in sample with the biggest production of biogas (0.5% of molasses). According to Environmental Protection Agency the minimal reduction of volatile solids should be 38% [16]. In reference sample and in the sample with 0.5% of molasses the minimal degree of anaerobic stabilization was achieved. In the other sample sufficient degradation of organic matter was not reached but results (35.80%-36.76%) were very close to minimal degree of degradation of organic matter.

**Table 2.** The degree of degradation of organic matter.

| The amount of molasses, % | Volatile solids, g/L |                    | The degree of anaerobic stabilization, % |
|---------------------------|----------------------|--------------------|--|
|                           | Before co-digestion  | After co-digestion |  |
| 0                         | 31.66                | 19.46              | 38.53                                    |
| 0.5                       | 42.66                | 25.72              | 39.71                                    |
| 1.0                       | 43.88                | 27.75              | 36.76                                    |
| 1.5                       | 44.17                | 28.40              | 35.71                                    |
| 2.0                       | 47.71                | 30.72              | 35.61                                    |
| 3.0                       | 50.36                | 32.33              | 35.80                                    |

*The change of VFA/A ratio*

Concentration of VFA has a significant influence on the efficiency of anaerobic digestion. VFA is an important mid-product in the formation of methane. The result of study showed that the amount of the volatile fatty acids had effects the anaerobic digestion process. The higher ratio of molasses the higher concentration of VFA (Tab. 3). After co-digestion process in sample over 0.5% of molasses accumulation of VFA was observed. As a result, the methanogenesis was inhibited and pH was decline. Therefore, decrease of biogas production in sample over 0.5% was detected.

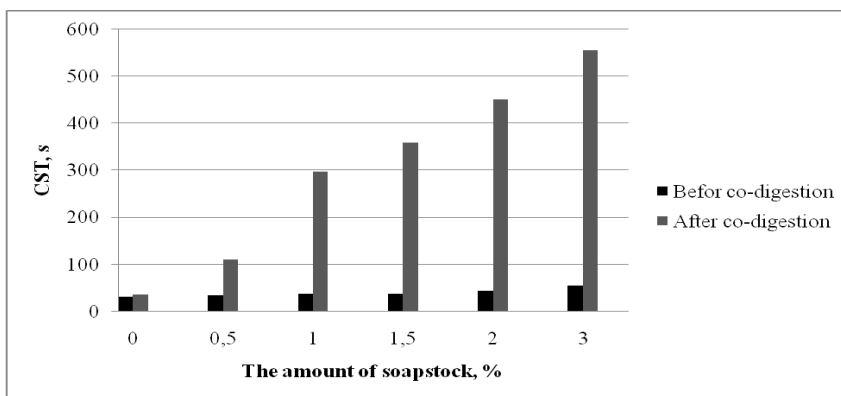
Examining concentration of VFA and A is recommended strategy to control co-digestion process. The VFA/A ratio up to 0.3 provides a balance between acidogenic and methane bacteria [17]. The results of the study confirmed this literature report. In the samples with 1%, 1.5%, 2% and 3% of molasses where ratio was over 0.3 the biogas yield, the methane content in biogas and the reduction of organic matter was lower than in reference sample.

**Table 3.** The physical-chemical analysis of supernatant liquid.

| The amount of molasses, %                     |                     | 0    | 0.5  | 1.0  | 1.5  | 2.0  | 3.0  |
|---|---------------------|------|------|------|------|------|------|
| pH  | Before co-digestion | 7.1  | 7.1  | 7.1  | 7.3  | 7.2  | 7.3  |
|   | After co-digestion  | 7.8  | 7.9  | 7.5  | 7.4  | 7.0  | 5.7  |
| A <sub>s</sub><br>mg CaCO <sub>3</sub> /L     | Before co-digestion | 475  | 400  | 375  | 450  | 375  | 475  |
|   | After co-digestion  | 2690 | 3019 | 2805 | 3063 | 2755 | 2465 |
| VFA <sub>s</sub><br>mg CH <sub>3</sub> COOH/L | Before co-digestion | 28   | 66   | 117  | 166  | 210  | 269  |
|   | After co-digestion  | 73   | 163  | 1106 | 1982 | 2743 | 3521 |
| VFA/A   | Before co-digestion | 0.06 | 0.17 | 0.31 | 0.37 | 0.56 | 0.57 |
|   | After co-digestion  | 0.03 | 0.05 | 0.39 | 0.65 | 1.00 | 1.43 |

*Dewatering properties*

The addition of molasses impacted dewatering properties of sludge. The capillary suction time (CST) test is widely used method to determine the filterability and the easiness of removing moisture from slurry and sludge [18]. Generally, digestion process have adverse effect on dewatering properties of sludge. The capillary suction time enhanced after co-digestion process in each sample (Fig. 3). In the reference sample CST increased from 31 s to 36 s but in other samples expansion was more significantly. After co-digestion, with increasing content of molasses, CST increased from 111 s to 556 s (from 0.5% to 3% of molasses, respectively).



**Fig. 3.** Changes of CST value for different contents of molasses.

*Comparison of biogas production for a different co-substrates at their optimum content*

The authors of this publication also done research process of co-digestion with other organic co-substrates. In this investigations were used: waste meat, soapstock, by-product after biodiesel production and waste from fat separator. These compounds have not yet been widely investigated as substrates for fermentation with sewage sludge. However, the results of such applications can be found in a few publications [19–22]. Table 4 shows the

results of the maximum biogas production and methane content in biogas under optimum conditions of the fermentation process for different organic co-substrates. Methane content in biogas was the biggest using waste meat (76%). The similar methane content (72–73%) was reached in soapstock, by-product after biodiesel production and molasses investigation. The lowest methane content in biogas was achieved in waste from fat separator investigation. The maximum biogas production using molasses was 95.69 mL/g VS and was only slightly higher than in case of application of waste from fat separator. For other substrates the biogas production was higher. However, compared to other substrates, the maximum biogas production and a relatively high methane content using molasses was obtained with the smallest content of this substrate (0.5%) in a mixture with sewage sludge. Therefore, molasses appears to be a substrate, which should be considered for use in wastewater treatment plants in the process of co-digestion, especially in the case when the digesters have a small supply of additional capacity.

**Table 4.** The maximum biogas production and methane content in biogas for different organic co-substrates.

| Organic co-substrates                  | Maximum biogas production (mL/g VS) | Methane content in biogas (%) | Content of substrate in the sample (%) |
|--|-------------------------------------|-------------------------------|--|
| Waste meat                             | 170.30                              | 76                            | 3                                      |
| Soapstock                              | 117.17                              | 73                            | 10                                     |
| By-products after biodiesel production | 99.91                               | 72                            | 10                                     |
| Molasses                               | 95.69                               | 73                            | 0.5                                    |
| Waste from fat separator               | 88.68                               | 70                            | 3                                      |

## 4 Conclusion

Addition of molasses had positive influence on biogas production. The biggest biogas yield was reached in sample with 0.5% of molasses and was 21% higher than in reference sample. Co-digestion process of sludge and molasses influenced on biogas composition. In sample with 0.5% of molasses methane content increased to 73%. The higher dose of molasses than 0.5% caused inhibition of the digestion process, thus 0.5% of molasses is optimum dose. According to the EPA regulations, the degrees of degradation of organic matter obtained in reference sample and in sample with 0.5% of molasses were significant, because it exceeded 38%. In the other samples sufficient degradation of organic matter was not reached, but obtained results (35.61%-36.76%) were very close to 38%. The result of VFA/A ratio indicated that 0.5% of molasses is the optimum dose because higher content caused inhibition of co-digestion. Generally, digestion process have adverse effect on dewatering properties. After co-digestion, with increasing content of molasses, CST increased from 36 s to 556 s (from 0% to 3.0% of molasses, respectively). This information should be taken into account in the design of sludge dewatering process.

Based on the obtained results it can be stated that molasses is a substrate which can be used in wastewater treatment plants in the process of co-digestion, especially in the case when the digesters have a small supply of additional capacity.

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