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The influence of biomass agitation on biogas and methane production using the high-solids thermophilic anaerobic digestion

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Abstract: In this study, we tested the high-solids thermophilic anaerobic digestion of separated cattle slurry solids inoculated by liquid digestate collected from the 1st stage of the wet process operated in an agricultural mesophilic biogas plant. The process without batch agitation (stirring) was carried out in a barrel bioreactor located in the incubator, whereas the process with batch agitation was carried out in a rotating drum bioreactor (rotation speed: 0.1 min⁻¹). In both cases, the biomass batch was non-liquid and the content of total solids was 18.8 wt%. The processes were conducted discontinuously without any addition for 21 days. The total solids content of both batches decreased to about 15 wt%, with only slight liquefaction. The bulk density increased from 500 to 750 kg m⁻³. The highest biogas production was achieved consistently on the 7th day. During the 21-day period, the unagitated batch produced 0.120 m₃ N³ of biogas, and the agitated batch produced 32.5% extra. The average CH₄ content in biogas from the unstirred batch amounted to 47.7 vol.%, and biogas from the stirred batch reached 46.2 vol.% The cumulative methane production rates from the unagitated and agitated batches were 0.095 and 0.121 m₃ N³ kg VS⁻¹, respectively. Thus, the agitation resulted in 28.5% higher methane yield.

Keywords: biogas; cattle slurry; high-solids (dry) anaerobic digestion; methane

1 Introduction

Agricultural fibrous biomass like maize silage is currently considered a promising renewable energy source [1]. Due to its multiple uses, this source delivers 12% of the worldwide energy demand. The proportion of this energy source has increased to 40%–50% in many developing countries [2]. In the anaerobic digestion (AD) process, biomass is utilized mainly for the production of biogas or methane [3].

The history of AD can be traced back 2000 years with the AD of animal manure in China and India [4]. In the modern age, after the discovery of methane emissions from natural anaerobic habitats by Volta in 1776, people began to collect natural biogas and use it as a fuel mainly for lighting. The first digestion plant was built in a leper colony in Bombay, India, in 1859. Since the 1980s, research have focused on dry digestion and several commercialized dry digestion systems, such as DRANCO [5], KOMPOGAS [6], and VALORGA [7], which have been developed to treat solid organic wastes.

AD [8–12] is a biochemical process that is not exposed to air, and its aim is to transform the organic substrate to material- and energy-profitable products. Combining this process with agriculture is very advantageous thanks to huge amounts of biological wastes generated in this industry [13–15]. The resulting biogas has resulted in better prospects regarding its future use in the energy market [16, 17].

On the basis of the total solids content of the feedstock, AD can be categorized as wet, also called low-solids AD (LSAD), semi-dry, and dry, both of which are categorized as high-solids AD (HSAD). The semi-dry process runs at approximately 15%–20% total solids in liquid biomass, whereas the dry process runs at more than 20% total solid in stackable biomass. LSAD is still mostly used, but HSAD technology is fast becoming more popular because it requires a smaller reactor volume as well as lower energy requirements for heating and material handling [18, 19]. However, there are still problems involved in the process of biomass agitation. Dry AD allows the processing of biomass with low moisture and high-fiber content [20, 21],

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such as corn silage, stow-manure, and even mixed municipal waste with liquid manure.

The total solids and consequently volatile solids content influence AD performance, especially biogas and methane production efficiency [22]. The HSAD process runs at a higher organic loading rate (2–3 times) but has a longer retention time (at least 2–3 times). When comparing the HSAD to LSAD running in liquid suspension, relatively smaller biogas and methane yields can be achieved, but the former generates less wastewater and larger amounts of digested solids that can be combusted [23, 24]. High-solids (dry) biogas stations often use garage-shaped (concrete bunker) fermenters. Such simple designs are effective and available even for small farmers. Nevertheless, there remains a need to develop alternative bioreactors that can efficiently process both solid and semi-liquid substrates on a small scale [25, 26]. The typical construction of the pilot-scale bioreactor for dry AD with batch agitation is mentioned in a previous work [27]. Stirring appeared to be necessary for the effective operation of biogas plants and the ideal consistency of feedstock fermentation inside the reactor, because it can better integrate micro-organisms and substrate as well as ensure the even distribution of pH levels and temperature.

Very rapid mixing disrupts the structure of flocs in the batch, which in turn, disturbs the syntrophic relationships between organisms, thereby adversely affecting biogas formation [28–30]. However, there is no clear information available in the literature regarding the threshold limits of digester stirring, other than a power input of 5.3–7.9 W m\(^{-3}\), which is recommended by the US EPA for the proper stirring of tank digesters used for the wet AD process [31].

Thus, the aim of the current paper is to determine the influence of biomass agitation on biogas and methane production using the high-solids thermophilic anaerobic digestion process.

### 2 Materials and methods

#### 2.1 Materials

Fresh cattle slurry, removed from the underground storage at a dairy farm in Zempsol Studénka Ltd. in Pustějov, Moravian-Silesian Region, Czech Republic, was delivered to the laboratory. The slurry (5 × 20 kg) was separated using the small industrial centrifuge BEHO CHC-61A (BeHo spol. s r.o., Krucemburk, Czech Republic) at the maximum obtainable rotational speed of 1200 min\(^{-1}\) for 10 min, thereby generating separated solids (solid phase) and liquid phase (see Table 1). The solid phase contained 23.1 wt% of total solids, and consisted mainly of fibrous particles with a length of less than 25 mm. Typically, the digested residues of corn silage and lucerne straw silage are easily identifiable. In the current study, the consistency was of wet bulk compost and the residues did not release any leachate; after the pummeling test, however, the solids changed to a rather muddy consistency. The separated solids were used as a model substrate. The liquid phase contained 4.6 wt% total solids and was not further exploited. The solid phase consisted of 17.6% of the initial sample.

Liquid slurry (digestate), obtained from the 1st stage of operation of a mesophilic agricultural biogas plant in Pustějov, contained active methanogenic microorganisms. This digestate was used as starting substrate (inoculum). This was cultivated for 21 days in a drum-type bioreactor INFORS HT TerraFors IS (Infor AG, Switzerland, Bottmingen) at thermophilic conditions without any addition, but with a single step change in temperature from 40°C to 55°C within the 5th to 10th hour of tempering. Photos of the cattle slurry separated solids (CSSS) and of the inoculum are shown in Figure 1.

#### 2.2 Laboratory model

Two parallel tests of high-solids thermophilic AD (55°C) were conducted. The control process without biomass batch agitation was performed in a steel barrel bioreactor (stationary bioreactor, total volume adjusted to 0.015 m\(^3\)) with a gas-tight removable lid, wherein the barrel was placed in the large volume incubator MEMMERT IF450 (Memmert GmbH + Co. KG, Germany, Schwabach) (see Figure 2). All inner surfaces of the barrel were coated with plastic film. The process with biomass batch agitation (stirring) was performed in a rotary drum-type bioreactor INFORS HT TerraFors IS with a total volume of 0.015 m\(^3\) (see Figure 3). The speed of rotation was set to the lowest

### Table 1: Biomass parameters.

<table>
<thead>
<tr>
<th>Biomass material</th>
<th>pH</th>
<th>Total solids (105°C)</th>
<th>Volatile solids (550°C)</th>
<th>Density</th>
<th>Carbon (105°C)</th>
<th>Nitrogen (105°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>TS (%)</td>
<td>VS (%)</td>
<td>VS (%TS)</td>
<td>P (kg m(^{-3}))</td>
<td>N (%TS)</td>
</tr>
<tr>
<td>Cattle slurry</td>
<td>7.4</td>
<td>8.0</td>
<td>5.9</td>
<td>73.2</td>
<td>1014</td>
<td>42.1</td>
</tr>
<tr>
<td>Cattle slurry separated solids (CSSS)</td>
<td>7.4</td>
<td>23.1</td>
<td>19.4</td>
<td>84.0</td>
<td>290</td>
<td>41.8</td>
</tr>
<tr>
<td>Cattle slurry liquid phase</td>
<td>7.4</td>
<td>4.6</td>
<td>3.1</td>
<td>68.3</td>
<td>1008</td>
<td>–</td>
</tr>
<tr>
<td>Inoculum-digestate from the Pustějov BGS (I)</td>
<td>8.2</td>
<td>6.2</td>
<td>4.5</td>
<td>72.0</td>
<td>1010</td>
<td>35.6</td>
</tr>
<tr>
<td>Experimental biomass batch-starting mixture of CSSS (74.4%) and I (25.6%)</td>
<td>7.7</td>
<td>18.8</td>
<td>15.6</td>
<td>83.0</td>
<td>500</td>
<td>40.21</td>
</tr>
<tr>
<td>Batch after 21 days (unagitated barrel reactor)</td>
<td>7.9</td>
<td>15.3</td>
<td>11.9</td>
<td>77.9</td>
<td>750</td>
<td>–</td>
</tr>
<tr>
<td>Batch after 21 days (agitated TerraFors reactor)</td>
<td>7.9</td>
<td>14.6</td>
<td>11.2</td>
<td>76.4</td>
<td>750</td>
<td>–</td>
</tr>
</tbody>
</table>


adjustable setting of 0.1 min⁻¹. This speed should be feasible and sufficient in full-scale practice. About 2.9 kg (74.4%) of CSSS and 1.0 kg (25.6%) of a liquid inoculum were weighed into the Terrafors bioreactor drum. An identical batch characterized by dry matter content of 18.8%, C : N ratio of 14 : 1, and volume of 0.0078 m³, was inserted into the control bioreactor. The initial volume weight of the CSSS was only 290 kg m⁻³, but this increased to about 500 kg m⁻³ after mixing with the liquid digestate. This resulted to an initial volume of 0.0078 m³ and corresponding initial organic load of approximately 65 kgVS m⁻³.

### 2.3 Measurements and analysis

The biomass batch temperature was measured using 1 jacketed thermocouple placed axially in the shaft of the rotary drum reactor, and an alcohol thermometer placed along the side wall of the barrel control reactor in the incubator.

The continuous measurements of raw biogas flow rate and the total volume of biogas were performed by water-calibrated drum-type flow meters Ritter TG05 (RITTER Apparatebau GmbH & Co. KG, Deutschland, Bochum) (transparent PVC, 0.001–0.060 m³ h⁻¹). The daily volume increase was written down manually. The gas volume was recalculated to 0°C, 1 atm under dry conditions. The composition of raw biogas was measured sequentially once a day (prior to dosing of the substrate at 9 am) by a portable IR/electrochemical analyzer Geotechnical Instruments (UK) Ltd. “Biogas5000” (Geotechnical Instruments (UK) Ltd, Queensway) (CH₄ 0%–70% ±0.5%, CO₂ 0%–60%±0.5%, O₂ 0%–25%±1.0%, H₂ 0–2000 ppm ±2.0% FS, H₂S 0–5000 ppm ±2.0% FS). The composition of biogas was measured once at hourly intervals for 24 h a day to correct the daily mean methane content. The gas sample for analysis (0.0002%–0.0004 m³) was evacuated via a gas valve on the lid of the bioreactor using a diaphragm pump. Once a week, the biogas composition was verified using a gas chromatograph Agilent Technologies 7890A (Agilent Technologies, Santa Clara, CA, USA) with a flame-ionisation detector. Upon completion of the test, the average value of the biogas/methane-specific production was calculated.

The pH was occasionally measured by a portable pH-meter with WTW SenTix® 940 electrode placed in the reactor after lifting the lid. The ratio of concentrations of volatile organic acids and total inorganic carbonate (VFA/TIC, also known as FOS/TAC) was measured offline by manual titration of 0.05 M H₂SO₄ to two end values (pH 5.0 and 4.4) at the end of the process.

The total solids (dry matter) content was measured by drying 10–20 g of the biomass sample at 105°C to constant weight with the use of halogen analyzer KERN DLB 160-3A (Kern & Sohn GmbH, Balingen, Germany) (repeatability of 0.2%) and verified by thermogravimetric analyzer LECO TGA 701 (LECO, Saint Joseph, MO, USA), through which the loss on ignition of the dry matter at 550°C to constant weight was also determined. The samples were stored in a refrigerator at 8°C.
3 Results and discussion

During the separation of cattle slurry by screw press, the amount of solids reached 17.6%. The same volume is reported by Rico [32].

In the first 7 days during the digestion, the biogas production rapidly increased in both reactors to different maximum values. In the case of a rotating drum, the increase of production was rather linear, whereas that in the case of a stationary reactor was rather exponential. The maximum daily production of raw biogas under laboratory conditions in the unagitated reactor reached 83.3% of the value given in the agitated drum reactor. From the 7th day onwards, biogas production in both bioreactors gradually decreased.

The agitated reactor produced 0.159 m₃ of biogas with an average volumetric CH₄ content of 46.2% in 21 days. The unagitated reactor produced only 0.120 m₃ of biogas but with an average CH₄ content of 47.7% during the same time (see Table 2). About 32.5% more biogas and 28.3% more CH₄ were produced due to agitation. Both values are considered very significant. The methane content in biogas rapidly reached 50% (within 4 days) and after that, it did not change much in both reactors. Due to the nature of the substrate (few quality organic solids), this result was expected. The cumulative methane productions from the unagitated and agitated batches were 0.095 and 0.121 m₃ CH₄/kg VS, respectively. These values are quite close to the findings of Massé [33] who reported that the agitated psychrophilic digestion of cow feces CH₄ production reached 0.154 m₃ CH₄/kg VS. However, both results are much lower than other data found for a similar substrate. For example, Rico [32] reported 0.265 m₃ CH₄/kg VS for the thermophilic process with percolation, whereas Jha [34] reported CH₄ yield of 0.358 m₃ CH₄/kg VS from the agitated mesophilic digestion of cow dung. The agitation in the case of Jha [35] was certainly imperfect in comparison to that in the rotating drum reactor, so it increased the CH₄ yield by only 7.5%. According to Karim [36], wet manure slurry digestion can possibly increase the production of biogas by mixing up to 30% of the slurry. Kaparaju [37] pointed out that agitation should be gentle and that it need not be continuous.

The total solids content of both batches fell to about 15 wt%, with only slight liquefaction. No loose leachate that could alternatively be recirculated to enhance
conversion efficiency was found underneath the unagitated batch. The layer thickness of the batch was too low. Given that 91.6% of batch total solids and 92.7% of organic solids came from the separated solid from cattle slurry, we can verify the methane production of this raw material. The final bulk density of both processed batches (digestates) was approximately 750 kg m$^{-3}$. If we calculate the intensity of biogas production to the corresponding batch volume (0.0052 m$^3$), we arrive at a value of 1.45 mN m$^{-3}$ d$^{-1}$. This is sufficient to ensure that the process could be conducted in practice after the transition to the continuous mode. The whole process is evident from the graph shown in Figure 4.

Rotating reactors with volumes of up to 100 m$^3$ could be used at small farms. These reactors could work discontinuously and at least three reactors should be used. However, the semi-continuous process is definitely advised (with daily dosing and digestate discharge).

The specific methane productions of the unstirred and stirred batches were 0.015 m$^3$ kg$^{-1}$ and 0.019 m$^3$ kg$^{-1}$, respectively. Both numbers correspond to the practice of garage biogas stations [27]. The result, however, did not exceed expectations. On the one hand, the DRANCO process is considered an effective approach in the treatment of solid wastes with 20%-50% TS. Its typical retention time is 15–30 days, and the biogas yield ranges between 0.100 and 0.200 m$^3$ kg$^{-1}$ of input waste. On the other hand, in the VALORGA system, the retention time is typically 18–25 days at mesophilic temperatures with biogas yields ranging from 0.080 to 0.160 m$^3$ kg$^{-1}$ of feedstock, depending on the type of solid waste [38]. A past study [39] mixed three types of waste (i.e. garbage and rejects from hotels, yard waste, and old paper) at various ratios to control the C/N ration before feeding to the KOMPÖGAS plant. The plant ran at stable operation for at least 2 years and generated biogas at a rate of about 0.820 m$^3$ kg$^{-1}$. The average heat input of biogas (according to net calorific value) amounted to 0.280 kW$_{in}$ m$^{-3}$ (related to the batch volume in the agitated reactor). This value is approximately in the middle of the range that has been verified, for example, by Besgen [40] who studies four small agricultural biogas stations with two-stage wet processes (0.128–0.415 kW$_{in}$ m$^{-3}$). This range, however, is significantly less than that indicated for dry digestion, especially residual municipal waste in the DRANCO system (up to 1.8 kW$_{in}$ m$^{-3}$) [41]. Meanwhile, Kayhanian reported that the values corresponded to 1.1–1.4 kW$_{in}$ m$^{-3}$ [42, 43].

In the case of laboratory drum bioreactor Terrafors IS, the specific power input of the electric motor used for the drum rotation (0.1 min$^{-1}$) was 2.0 W kg$^{-1}$ per batch. The difference between biogas net calorific values and batch densities of the agitated and unagitated reactors created the benefit of agitation, although it only increased energy production by 0.1 W kg$^{-1}$. The model was, therefore, very inefficient. We assume that the power input for rotation of approximately 0.1 W kg$^{-1}$ can be achieved in an industrial scale in a cylindrical bioreactor of 40 m$^3$ volume. At the same time, the increase of net calorific values of biogas...
should be higher than that at the lab-scale. This implies the probability of higher equipment efficiency than in the case of a garage fermenter with an external heated percolate tank and pumps.

### 3.1 High-solids digestate

The final digestate from the rotating drum had a pH value of 7.9 and 14.5 wt% of total solids containing 76.4 wt% of volatile solids. About 96 wt% of total carbon (40.2 wt% content) was formed by organic carbon. The total nitrogen content was 2.9 wt%. The C : N : P ratio was 28 : 2 : 1. This digestate meets the requirements for organic fertilizers with more than 13 wt% of total solids according to the CZ Regulation no. 131/2014 Coll. [44], Addendum no. 1, table no. 2b, as amended, except the Cu and Zn contents. The Cu content was three times higher than the 100 mg kg\textsuperscript{-1} limit, which may be due to the dairy cow hooves being rinsed in CuSO\textsubscript{4} disinfectant solution. The Zn content was 1.25 times higher than the 600 mg kg\textsuperscript{-1} limit, which may be due to the administration of cattle medication. The potassium, calcium and magnesium contents of 2.6, 1.7 and 0.7 wt%TS, respectively, were also sufficient for organic-mineral fertilizer.

The digestate also met the quality parameters for land or landfill reclamation set by the CZ Regulation no. 341/2008 Coll. [45], Addendum no. 5, table no. 5.1 and 5.3, as amended. Only the copper content limits its usage. The digestate would be usable as the reclamation material of the Group 2/Class II. This means that the digestate could be used outside the agricultural or forest lands, on sport and recreational grounds and green lands, in urban areas except those outside playing fields, in urban green fields, parks, forest parks, industrial zones, waste dumps, and sludge beds.

### 4 Conclusion

The experiment confirmed the possibility of increasing the anaerobic biogas and methane production by very slow continuous rotation (0.1 min\textsuperscript{-1}) of the reactor during discontinuous start-up of the process. With the experimental batch, 32.5% more biogas and 28.3% more CH\textsubscript{4} were produced as a result of this style of agitation. The results, however, must be complemented by energy consumption at the pilot scale and then used for the development of small biogas plants.

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### References

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