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Biogas production with horse dung in solid-phase digestion systems

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Abstract

Experiments on methanogenesis from horse dung were conducted in laboratory-scale batch reactors in order to determine the substrate performance in a solid-phase digestion process, more specifically in terms of potential energy recovery and suitable process technology. Dung from a horse stable with straw bedding was used. The temperature was kept in the mesophilic range. In the percolation process (with process water sprinkled over the stacked biomass) a proportion of 10-20% of solid inoculum (pre-digested horse dung) was found to be suitable. Comparative experiments with both percolation and flooding revealed a higher biogas production per volume for the flooded process, as no addition of solid inoculum was necessary. Methane yield from fresh material was similar in both processes: around 170 L_N CH_4 per kg VS added was obtained in six-week cycles with untreated material under optimized conditions. Methane production was increased after chopping the substrate. Pre-aeration resulted in decreased methane production.

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1. Introduction

Digestion with an elevated content of total solids (TS) is widely used for municipal solid waste (MSW). Since 1993, what are called dry digestion plants (>20% TS in the feed) have been constructed more often than wet digestion plants (<10% TS in the feed) (Bolzonella et al., 2003). Continuous single-phase processes are predominant. In Europe, only 11% of the total digestion capacity is offered by two-phase systems (De Baere, 2000), probably because single-phase systems are cheaper with regards to the investment and maintenance required (Mata-Alvarez et al., 2000). Several advantages of two-phase systems have been reported (Cho et al., 1995; Ghosh and Klass, 1978; Llabrés-Luengo and Mata-Alvarez, 1988; O'Keefe and Chynoweth, 2000; Raynal et al., 1998; Zoetemeyer et al., 1982). While during the first phase appropriate conditions accelerate liquefaction, the second phase converts soluble matter into biogas. This allows for a more rapid and more stable process compared to single-phase systems (O'Keefe and Chynoweth, 2000). However, distinct separation between hydrolysis-acidification and methanation is difficult to maintain (Cho et al., 1995; Christ, 1999; Mata-Alvarez et al., 2000; Raynal et al., 1998), which may eliminate the associated benefits of a two-phase system (O'Keefe and Chynoweth, 2000). Phase separation appears to be more difficult for slowly hydrolysable substrates (Chanakya et al., 1992). For solid materials with slow degradability, single-phase digestion is recommended (Christ, 1999; Wechs, 1985). For easily degradable materials, a two-phase system is considered more advisable (Mata-Alvarez et al., 2000; Pavan et al., 2000). With systems in which leachate trickles through a biomass bed, phase separation may be more difficult to achieve than with stirred digesters due to lack of mixing and low ion diffusion in a non-flooded matrix (O'Keefe and Chynoweth, 2000).

In agriculture, slurry-based liquid-phase digestion is widely applied today, but digestion with elevated TS contents promises further development. Research data has been published on the digestion of crop residues, herbs and leafy biomass (Anand et al., 1991; Chanakya et al., 1993; 1997; Jewell et al., 1982; Legrand and Jewell, 1987; Liu et al., 1987; Sun et al., 1987), dairy manure (Kalia and Singh, 2001; Linke, 2000; Schäfer et al., 2005; Weizhong et al., 1999),

pig dung (Zelter, 1978), yard waste (O'Keefe et al., 1993; Owens and Chynoweth, 1992), energy crops (Jewell et al., 1993) and various ensiled substrates (Linke and Schelle, 2001).

Batch digestion is a simpler method than continuous digestion (Ten Brummeler and Koster, 1990). In general, batch-operated solid-phase installations in agriculture have a volume of 100-150 m^3 per reactor (Weiland, 2004). In what is called the percolation process, liquid is recirculated and sprinkled over the stacked material in order to initiate biogas production and encourage bacteriological activity in the decomposing biomass throughout the process.

Biological activities are limited by an inadequate supply of moisture and associated organisms and nutrients (Chen and Chynoweth, 1995). A leachate flow through a biomass bed accelerates mass transfer by adding convective transport mechanisms to molecular diffusion (Martin, 1999). Leachate recycling has been found favourable for anaerobic decomposition of landfill material (Barlaz et al., 1992; Chan et al., 2002; Mata-Alvarez and Martinez-Viturtia, 1986; Mehta et al., 2002). However, it has also been reported that acidogenesis in particular may be enhanced, which may result in the inhibition of methanogenesis (Komilis et al., 1999a). Optimal conditions for methanogens are especially important in the initial stage; therefore, low leachate recirculation rates should be chosen when initiating the process (Vavilin et al., 2002; 2003). Recirculating leachate within an already acidified cell will not correct the problem (O'Keefe and Chynoweth, 2000). The exchange of leachate between a batch of previously stabilized waste and a batch of fresh waste enhances degradation (Chugh et al., 1999; Chynoweth et al., 1992; O'Keefe et al., 1993; Suna Erses and Onay, 2003). Compaction can hinder the proper distribution of leachate (Komilis et al., 1999a). For MSW, Chen and Chynoweth (1995) found a logarithmic relationship between hydraulic conductivity and packing density.

During start-up phases, the microbial community should contain sufficient levels of methanogens to prevent digester failure (Griffin et al., 1998). The conversion of hydrolyzed organics to volatile fatty acids (VFA) will result in VFA accumulation along with a drop in pH if acids are not metabolized by methanogens. Methane bacteria are sensitive to pH (Clark and Speece, 1970), values >6.5 (Christ, 1999) or >6.8 (Chen and Hashimoto, 1996) and in general <7.5 are recommended. The addition of an appropriate inoculum ratio is favourable during start-up (Chen and Hashimoto, 1996; Ten Brummeler and Koster, 1989), and inoculum quality is also important (Dirar and El Amin, 1988). The hypothesis has been proposed that in a bed of organic

substrate, anaerobic digestion is initiated by seed bodies around which reaction zones gradually develop (Martin et al., 2003). When densely seeded with inoculum, the expansion of methanogenic areas into the total volume of the digester occurs quite rapidly (Martin et al., 2003; Vavilin et al., 2002). Unnecessarily high levels of inoculum lead to increased digester sizes and would therefore be undesirable (Chanakya et al., 1997).

Greater particle-substrate surface areas increase contact between micro-organisms and organic mass (Barlaz et al., 1990). The positive effects on the biodegradability after size reduction of substrate particles have been discussed by Mata-Alvarez et al. (2000); biofibre degradation may be enhanced by shearing rather than by an actual change in size distribution. However, accelerated hydrolysis and acid generation might also inhibit methanogens. Contradictory results regarding the shredding of MSW have been discussed by Komilis et al. (1999b); increased compaction was also qualified as negative.

Pre-aeration is carried out in solid-phase digestion systems to reduce the anaerobic heat requirement by using the temperature increase resulting from the composting step. Pre-aeration may also reduce acidification during start-up phases (Ten Brummeler and Koster, 1990). It has also been hypothesized that pre-composted solid substrate is more easily degradable in an anaerobic system because the aerobic treatment allows a depolymerization of complex organic fractions (Komilis et al., 1999b; Mata-Alvarez et al., 1993). However, the pool of organic material for biogasification is lower due to the fact that easily degradable components are already metabolized (O'Keefe and Chynoweth, 2000; Ten Brummeler and Koster, 1990).

The anaerobic digestibility of horse dung is documented in literature (Mandal and Mandal, 1998). Kalia and Singh (1998) observed phase separation, with fibrous horse dung floating at the top of the liquid phase, which was found unsuitable for running a continuous slurry-based digester with higher ratios of horse dung. Gas production with mixtures of horse and cattle dung was slightly poorer than with cattle dung alone.

In this study, horse dung was digested in laboratory-scale solid-phase reactors both with percolation and in flooded mode. In an initial experiment the necessary proportion of solid inoculum (pre-fermented horse dung) was determined. In a second experiment percolation and flooding were directly compared, using process water containing methanogenic bacteria. Furthermore, the dung was flooded with potable water in order to investigate the possibility of biogas production without the addition of any inoculation material. One test cell was pre-aerated

before flooding in order to find out if an intensified aerobic phase leads to an improvement in the microbiological breakdown of the substrate structure and therefore to higher biogas production. Whether or not gasification is positively influenced by chopping the substrate was also investigated.

2. Materials and methods

2.1. Reactor design and operation

All nine reactors (Figure 1) were water-jacketed and thermostatted stainless steel cylinders (solid material content: around 50 L; inner diameter 30 cm). Leachate was collected in a liquid-phase reservoir at the base. Liquid pumps (average liquid flow: 4.7 L/min) were set with timers to sprinkle leachate over the biomass bed automatically. Substrate temperature in the middle of the digester and at a height of 13 cm above the substrate ground was measured (Pt1000) and recorded twice per hour with a data logger. Biogas was collected in aluminium coated PE/PTFE-gas bags. The digesters offered the possibility to aerate the substrate. Gas from the aerated phase was sent directly to a bellows-type gas flow meter.

2.2. Experimental configuration

Two experimental runs with 9 test cells each were performed; Table 1 summarizes the experimental set-up.

In experiment 1 fresh material (FM) was digested with percolation in mixtures with different proportions of solid inoculum (SI): 10, 20, 30, 40, 44 and 50% (w/w) on a TS-basis (further referred to as *SI_10, SI_20, SI_30, SI_40, SI_44, SI_50*). Pure solid inoculum was also digested with percolation (*SI_100*). Pure fresh material was digested both with percolation (*FM_percol*) and flooded with liquid inoculum (*FM_flood*).

In experiment 2, in response to the results of experiment 1, horse dung with 20% solid inoculum was digested with percolation in two replicates (*Percol1, Percol2*). In two reactors horse dung was flooded with liquid inoculum (*FloodLI1, FloodLI2*); in two others with potable water (*FloodPW1, FloodPW2*). Chopped dung (sent twice through a compost chopper; final straw particle size around 4 cm) was flooded with liquid inoculum in two replicates (*ChoppLI1, ChoppLI2*). In the 9th reactor (*Aeration*) digestion was started after pre-aeration (48 h, 160 L air/h).

For mixtures of fresh substrate and solid inoculum, fractions were first homogenized after a simplified quartering method (opposite quarters were not remixed): material was poured onto a flat surface and after intensive mixing, it was divided into quarters; then, paying special attention so as not to lose the fine material, each quarter was remixed and divided again into quarters until the desired volume was reached. The final mixture of fresh material with solid inoculum was thoroughly carried out by hand. The weights of the materials added were determined when preparing substrate mixtures and the volumes of the mixtures when filling the reactors under very low compaction by hand. At the beginning, the same amount of liquid inoculum (LI) was added to all percolated test cells until percolation (recirculation of liquid) for 15 minutes was possible in the first cell. Different amounts of potable water were added to the others until percolation was possible in all of them.

Experiment 1 was run over a period of 74 days and experiment 2 over a period of 46 days. The target temperature was 35°C. Leachate was recirculated twice daily for 15 minutes in all reactors (percolated and flooded cells). Additionally, leachate was recirculated (15 to 20 min) prior to sampling. It is assumed that this ensured an equalisation between free leachate and liquid retained in the substrate body. It is also assumed that analysing the liquid phase enabled the evaluation of the conditions in the substrate body, e.g. accumulation of inhibitory substances. Leachate was sampled periodically and analysed for pH, VFA, NH_4-N, Total Kjeldahl Nitrogen (TKN), chemical oxygen demand (COD), TS and volatile solids (VS), as was liquid at the beginning and the end of an experimental run. Solid material was sampled before and after digestion and analysed for TS and VS.

2.3. Differentiation between methane yield and methane production (release)

A certain amount of biogas is not released from the reactor but remains inside, filling pores of the substrate stack and empty spaces (e.g. as occurs after substrate compaction). The volume of void space above the solid residue was determined (by measuring the corresponding height), but biogas filling the substrate pores was neglected. Gas quality was assumed to be identical to the quality in the gas bag that was measured previously; thus, methane volumes remaining inside the reactors were calculated. These volumes are taken into account whenever methane yields and exploitation degrees are discussed. They are not taken into consideration when methane production is presented in cumulative form over the digestion time (methane release

from the reactor). At full-scale, only the methane released from the reactor would be usable and not the actual yield.

2.4. Calculation of CH_4-yield from fresh material (G_{FM}) in mixtures

In mixtures with solid inoculum and/or liquid inoculum the methane yield from the component fresh material G_{FM} can be derived from the total yield of the mixture if the individual yields from solid and liquid inoculum are known (assuming that the specific yields of inocula do not change in the mixture). For these calculations the total methane yield (methane released from digester plus methane remaining inside void digester spaces) was taken into account.

In experiment 1 the CH_4-yield from solid inoculum was determined using reactor SI_100. In experiment 2, trials to digest solid inoculum in 5 L bottles failed. Therefore, this value was calculated by assuming that the solid inoculum, which was the solid residue from experiment 1, consisted of two parts: *a*) material which in experiment 1 was previously solid inoculum and *b*) material which in experiment 1 was previously fresh material. The probable methane yield for part *a* in experiment 2 was determined by extrapolating the data from reactor SI_100 (according to the procedure for determining the total methane potential which will be described later, also see Figure 2). It was assumed that the CH_4-yield for part *b* during experiment 2 was 90% of the difference between the total methane potential (G_{pot}) and the yield already obtained during the 74 days of experiment 1.

CH_4-yield of the component liquid inoculum was determined using the equipment for the HBT method (Hohenheim biogas yield test) (Helffrich and Oechsner, 2003a; 2003b) and digesting 30 mL of liquid in three replicates.

2.5. Total methane potential (G_{pot}) and exploitation degree (q)

The total methane potential of horse dung (fresh material in experiment 1) was determined with the HBT (Helffrich and Oechsner, 2003a; 2003b). In a 100 mL glass syringe (flask sampler), 0.5 g of test substrate (dried at 60°C over 48 h, ground <1 mm) and 30 mL inoculum (pre-digested liquid manure) were digested at 37°C in three replicates. The volume and methane content of the biogas produced were recorded periodically. Inoculum without substrate was digested as zero variant with three replicates as well.

After 44 days the test was stopped. The total methane potential $G_{pot} = G(t \to \infty)$ was calculated by extrapolation, based on a non-linear curve fit for the experimental data of the decay phase (declining gas production rate) (Figure 2). According to the biochemical degradation of solid materials in landfills (Kruse, 1994), it was assumed that gas production in the decay phase could be best described by the sum of two decay functions in the form $G(t) = a + b_1 e^{-k_1 t} + b_2 e^{-k_2 t}$ (with G(t): methane yield at time t; a, b_i, k_i: const.).

The actual methane yield depends not only on the total methane potential but also on digestion time and degradation kinetics, which is influenced by substrate characteristics (including pre-treatment) and process conditions. The exploitation degree $q_{ti} = G_{FM,ti}/G_{pot}$ at a specific point in time t_i was determined after calculating the methane yield from the component fresh horse dung $G_{FM,ti}$ as described above (part 2.4.).

2.6. Substrates

The main substrate properties are summarized in Table 2. Horse dung was collected from a typical horse stable with straw bedding. The proportion of straw in the manure was high. The dung for the two experiments was collected separately; in experiment 2 the proportion of faeces appeared slightly higher upon visual inspection. Pre-digested substrate as solid inoculum for experiment 1 was taken directly from the large-scale digester at this stable, where flooded horse dung was digested as a mono substrate in a one-phase, batch-operated solid-phase process in six-week cycles (with no recirculation of process water within one reactor or between different reactors during this time, and no reactor heating), as described elsewhere (Kusch and Oechsner, 2004). Liquid inoculum for experiment 1 was taken from the same digester.

The solid inoculum used in experiment 2 was the mixed solid residue from all nine reactors of experiment 1. Liquid inoculum was also gained from experiment 1: at the end of experiment 1 the liquid of all nine reactors was mixed and diluted with potable water in a 1:1-ratio (v/v).

2.7. Analyses

CH_4- and CO_2-quality were determined by infrared spectroscopy (Siemens Ultramat, calibration before every reading). Gas quantity was determined with a bellows-type gas flow meter (GMT,

reading accuracy 0.1 L), calibrated to the flow of the vacuum pump (45 L/min). Gas volumes were corrected to norm litres (L_N), taking into account norm pressure and norm temperature (1.013 bar, 0°C).

Solid samples were analysed directly. Liquid samples were analysed either directly, within 24 hours after storage at 5°C or after being kept frozen at -22°C (but pH was always measured directly from the fresh sample). TS was determined by drying the samples at 105°C (solid samples 48 h, liquids 12 h) and VS by incineration of the samples at 550°C in a muffle kiln (ground solid samples >12 h, liquids approx. 8 h). The analytical methods used for COD, pH, NH_4-N and TKN conformed to DIN/EN-standards (DEV, 2004). VFA represent the sum of acetic, propionic, butyric, valeric and caproic acids and were measured by gas chromatography (GC) as follows: 1 µL supernatant (1 g sample, acidified with formic $acid_{conc}$, diluted 1:10, 15 min centrifuged at 16110 x g) was injected into a Varian CP-3800 GC (capillary column SGE BP 21, 25 m x 0.32 mm, 0.25 µm film; helium carrier gas at 40 mL/min in split 1:10; column temperature sequence: 40°C for 2 min, ramp of 15°C/min, 125°C for 1.5 min, ramp of 25°C/min, 180°C for 5 min; injector temperature 180°C), equipped with a flame ionization detector (280°C).

3. Results and discussion

3.1. Total methane potential

The total methane potential G_{pot} of the fresh horse dung used in experiment 1 was determined as 277.0 L_N CH_4/kg VS_{added} (Figure 3). This is lower than the methane potential reported for wheat straw by Tong et al. (1990) of 302 or 333 L_N CH_4/kg VS_{added} (two straw types, finely milled, 60 days in optimized batch test). Møller et al. (2004) found final methane yields of 195 L_N CH_4/kg VS_{added} for wheat straw (cut to 1 mm, 110 days), but quoted values of up to 241 L CH_4/kg VS_{added} from literature.

3.2. Ratio of solid inoculum

Among the percolated reactors in experiment 1, SI_20 had the highest methane production after 28 days (Figure 4), but differences were only marginal within group SI_10/20/30/40. This is due to the very high CH_4-yield of the solid inoculum. After 28, 42 and 74 days, the methane productions from SI_100 were 122.4, 159.2 and 188.9 L_N CH_4/kg $VS_{added,SI}$, respectively; these values are close to the amounts of methane produced by reactors containing fresh material and

indicate that digestion quality in the full-scale plant was poor. Indeed, severe problems, especially concerning temperature maintenance, were observed during the winter months when the materials were collected.

Figure 4 also shows the volumetric methane production. It should be kept in mind, however, that the contribution from solid inoculum was very high, which means that energy density in all test cells was quite similar. In 42 days, up to 10.4 and 10.5 L_N CH_4/L (SI_10 and SI_20) were produced with percolation.

FM_percol (no solid inoculum) had lower biogas production during the first 42 days. This option would be favourable only for longer digestion times. Inhibition during the start-up phase becomes less important with longer digestion times; consequently, the methane production of FM_percol was highest after 74 days (among percolated cells).

It was concluded that a proportion of 10-20% solid inoculum would be suitable for digestion of horse dung with percolation in six-week cycles. In the present study, despite the poor quality of the inoculum, 10% was sufficient. As the quality of horse dung may differ with regard to its contents, age, and storage prior to being digested, a higher proportion of 20% inoculum may be chosen in order to avoid process failure in full-scale applications.

Methane production in FM_flood was rapid, with no evident inhibition. It was, therefore, concluded that in the flooded mode no addition of solid inoculum would be necessary.

3.3. Comparison of flooding and percolation

Within 42 days Percol1/2 produced (released) 147.3 L_N CH_4/kg $VS_{added,mixture}$ (mean of both replicates) and FloodLI1/2 160.9 L_N CH_4/kg $VS_{added,mixture}$ (Figure 5). Although methane production from the flooded reactors was higher, the actual CH_4-yield from the component fresh horse dung ($G_{FM,42}$) was similar (Table 3): 173.8 and 171.5 L_N CH_4/kg $VS_{added,FM}$ for Percol1/2 and FloodLI1/2, respectively. This demonstrated that the digestibility of horse dung was comparable in the flooded and in the percolated mode.

Methane production per volume solid substrate, however, was higher with flooding, as no solid inoculum was necessary. Flooded reactors (FloodLI1/2) produced 11.4 L_N CH_4/L and percolated cells (Percol1/2) 8.4 L_N CH_4/L in 42 days. In experiment 1 the flooded cell (FM_flood) produced 11.7 L_N CH_4/L, while percolated ones generated up to 10.5 L_N CH_4/L (SI_20) in 42 days. Flooding therefore increased volumetric methane production by a factor of 1.11 in

experiment 1, but by a factor of 1.35 in experiment 2. This was due to the lower specific CH_4-yield from the component solid inoculum in experiment 2 compared to experiment 1 (a factor of 0.41). Methane yield $G_{FM,42}$ from the component fresh material itself was similar in both experiments (Table 3: 173.8 and 174.7 L_N CH_4/kg $VS_{added,FM}$ for Percol1/2 and SI_20, respectively). This showed that methane production in the percolated process was significantly influenced by the amount of solid inoculum added and also by the specific methane yield from the inoculum (very low in experiment 2 due to the long digestion in experiment 1; this would not be the case in a full-scale application).

The pH remained in a favourable range both in Percol1/2 and in FloodLI1/2 (Figure 6). Leachate COD was higher in Percol1/2 than in FloodLI1/2, with a more marked difference during the first weeks. In Percol1/2, up to 3810 ppm of VFA appeared on day 7, with up to 3210 ppm propionate (Figure 7). In FloodLI1/2, up to 2660 ppm of VFA with a maximum of 1560 ppm of propionate was measured. This indicated that percolated digesters may be more susceptible to VFA accumulation. In this experiment, however, no inhibition appeared; in both cell types VFA were completely degraded before day 18.

3.4. Flooding with potable water

Methane production from FloodPW1/2 was 151.2 L_N CH_4/kg $VS_{added,mixture=FM}$ after 42 days (Figure 5). This was 94% of the amount produced by FloodLI1/2, though production was considerably lower during the first 10 days. The inhibited performance during the first few weeks corresponded to a pH decrease to around 6.1 on day 7; afterwards, the pH gradually increased to 7.0 on day 34 (Figure 6). Although the optimum pH range for methanogens (6.8 to 7.5) was reached only on day 18, the overall digester stability was good. The microbiological removal of VFA was complete by day 25. This demonstrated that the microbiological population adapted well to this environment and that digestion of horse dung was possible without any addition of inoculum.

3.5. Chopping and pre-aeration

Cumulative methane production of ChoppLI1/2 was 180.1 L_N CH_4/kg $VS_{added,mixture}$ after 42 days. Methanisation was considerably enhanced during the first weeks: compared to FloodLI1/2, methane production increased by 22%, 18% and 12% after 15, 21 and 42 days respectively.

Anaerobic digestion of solid substrates is often rate-limited by the hydrolysis step (Christ, 1999), and mechanical pre-treatment can therefore improve digester performance (Mata-Alvarez et al. 2000). In the digestion of lignocellulosic material, enzymes must break the lignin barrier in order to gain access to the holocellulose; therefore, the reaction rate is directly related to the surface to which hydrolyzing bacteria can attach (Tong et al., 1990). Increased particle surface area enhances biodegradation but does not affect ultimate methane yield. Consequently, in the present experiment differences between chopped and unchopped substrate gradually became smaller with longer digestion times.

Pre-aeration did not enhance process kinetics. The test cell with pre-aerated horse dung produced 131.4 L_N CH_4/kg $VS_{added,mixture}$ in 42 days, which is 18% lower than FloodLI1/2.

3.6. Methane yield from component fresh horse dung (G_{FM}) and exploitation degree (q)

Table 3 summarizes methane yields G_{FM} from the component fresh horse dung in experiments 1 and 2. They were similar for all test cells in which no inhibition occurred and material without pre-treatment was used. Mean $G_{FM,42}$ (42 days) was 169.3 ± 3.4 L_N CH_4/kg $VS_{added,FM}$ (mean ± SD, n=5, SI_10/20/30/40/FM_flood) in experiment 1 and 172.6 ± 2.7 L_N CH_4/kg $VS_{added,FM}$ (n=4, Percol/FloodLI/1/2) in experiment 2, and was therefore not significantly different between the two runs (p=0.16 in unpaired, two-tailed Student's t-test). For both experimental runs mean $G_{FM,42}$ was 170.8 ± 3.4 L_N CH_4/kg $VS_{added,FM}$ (n=9, abovementioned cells). The corresponding exploitation degree q_{42} from the total methane potential G_{pot} was 0.62, which means that 62% of G_{pot} was converted in 42 days.

In 74 days, 74% of the total methane potential was converted (q_{74}=0.74; $G_{FM,74}$=205.5 ± 3.1 L_N CH_4/kg $VS_{added,FM}$; n=6, FM_percol/flood/SM_10/20/30/40). The proportion q_{42}/q_{74} was 0.83, which means that 83% of the yield in 74 days was converted during the first 42 days. This is more than in experiments with pig dung in solid-phase digestion with percolation conducted by Zelter (1978), in which 75% of the ten week yield was produced in the first six weeks.

Mean $G_{FM,28}$ was 144.6 ± 1.8 L_N CH_4/kg $VS_{added,FM}$ (n=4, SI_20/30/40/FM_flood). Only 52% of the total methane potential was obtained in 28 days (q_{28}=0.52).

Chopping enhanced biodegradability to q_{42}=0.69 and to mean $G_{FM,42}$=192.4 L_N CH_4/kg $VS_{added,FM}$ (ChoppLI1/2). Pre-aeration resulted in an exploitation degree of q_{42}=0.51.

Comparing this to q_{42}=0.62 of the substrate without pre-treatment, it was concluded that 11% of G_{pot} was lost through aeration.

3.7. VS removal

In Table 4, VS removal within 46 days is given for the solid phase (SP) and for the complete system (solid and liquid phase, SP + LP) because the two values differed (for calculation procedure see footnotes to Table 4). As considerable amounts of VS were found in the liquid phase, removal in SP was higher than the degradation in the system (SP + LP). The difference was more obvious with higher liquid volumes in the system. The liquid contained up to 19% of the final VS in the flooded system (ChoppLI1) and around 4% of the final VS in the percolated process.

In FloodLI1/2, the initial VS of the solid phase was reduced by 49%, and 44% of the total initial VS (SP + LP) was actually degraded. In the pre-aerated test cell, 46% of the total VS left the system during the experiment. This shows that the overall degradation of VS was comparable to the flooding without pre-aeration (FloodLI1/2), although energy recovery was lower. In Percol1/2, 40% of the VS in the solid phase was hydrolyzed and most of it (38% of the initial VS in the system) was metabolized by the methanogenic population. VS removal was lower with percolation than with flooding, which reflects poorer degradability of the organic material due to the addition of solid inoculum.

Mean methane production per kg VS removed was 397.0 ± 17.6 L_N CH_4/kg $VS_{removed}$ (n=8, Percol/FloodLI/PW/ChoppLI1/2), but the high standard deviation hinders further interpretation. The effect of lost VS when taking leachate samples was not considered in this study. Neither were various yet small amounts of material lost while emptying the reactors at the end of the experiment.

3.8. Further results

Biogas methane content of all reactors exceeded 50% after one week, although Percol1/2, Aeration and FloodPW1/2 demonstrated slightly poorer performance during the first few days (Figure 8). Methane contents of up to 60% were measured, with chopped substrate of up to 63%. All reactors reached a final gas quality of 52.5-53.5% CH_4. Mean methane content over the whole digestion time (46 days) ranged from 51.1-53.5% (Table 4).

Nitrogen accumulation did not reach inhibitory levels, final concentrations in the liquid phase were <520 mg/L NH_4-N and <1130 mg/L TKN (Table 4). Poggi-Varaldo et al. (1997) determined a critical ammonia concentration of 2800 mg/kg NH_4-N (MSW, mesophilic digestion at high TS content) and quoted from literature higher tolerable concentrations in liquid- or slurry-mode digestion. With better adapted bacteria, Weiland (1993) observed stable digestion of agro-industrial residues in the presence of 5000 mg/L NH_4-N.

Temperatures in the test cells of experiment 2 (Figure 9) were close to target temperature (35°C); differences of more than 1°C (median) occurred only in the middle of Percol1/2. In this study the replicates were positioned directly beside one another (no randomized distribution, which was not optimal). Percol1/2 were closest to the thermostat and so their position in terms of heat supply was optimal. The results indicate that problems with temperature maintenance may occur in percolated digesters. It may be assumed that, compared to flooded digesters, thermal energy transport is more difficult as pores inside the substrate stack are partly filled with gas and not with liquid.

4. Conclusions

Horse dung with straw was shown to be digestible as a mono substrate in batch-operated solid-phase digestion. Both in the percolation and in the flooded process, the specific methane yield of the fresh substrate was found to be around 170 L_N CH_4/kg $VS_{added,FM}$ in six weeks under optimal conditions and without pre-treatment (aside from the addition of inoculum). Compared to percolation, flooding itself did not enhance degradability of horse dung within six weeks. However, the volumetric methane production was higher in the flooded system as no solid inoculum had to be added. The results show that percolation and flooding can both achieve the same methane yield per kg VS of fresh horse dung added, but this requires larger reactor volumes in the percolated system.

In the percolation process a rapid start-up was ensured by the addition of 10-20% (w/w on TS-basis) pre-fermented horse dung as solid inoculum. Up to 10.5 L CH_4/$L_{solid\ substrate}$ were produced in six weeks with percolation and up to 11.7 L CH_4/L with flooding. Assuming a mean CH_4-content of 50%, 21 m³ biogas/m³ could be expected with percolation and 23 m³ biogas/m³ would be probable in the flooded process in six-week cycles with horse dung that did not

undergo any pre-treatment. Altered bulk density in full-scale applications may influence the result, a factor which could not be examined in this work.

The total methane potential of the horse dung was determined as 277 L_N CH_4/kg $VS_{added,FM}$. Of this, only 52% was converted in four weeks, 62% in six weeks and 74% in 74 days when using substrate without pre-treatment. Chopping the substrate with a compost chopper accelerated biogasification significantly. Aeration as a pre-digestion treatment failed to be successful. It resulted in a lower biogas yield with no enhancement of the process kinetics.

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Table 1
Experimental set-up (FM: fresh material, SI: solid inoculum, LI: liquid inoculum, PW: potable water)

	description	FM	SI	LI	PW
		kg	kg	L	L
experiment 1					
FM_percol	FM, percolated	7.32	-	6.0	4.5
SI_10	FM + 9.9% SI, percolated *)	7.18	1.79	6.0	3.5
SI_20	FM + 19.8% SI, percolated *)	6.30	3.50	6.0	3.5
SI_30	FM + 30.0% SI, percolated *)	5.39	5.15	6.0	3.0
SI_40	FM + 39.9% SI, percolated *)	4.55	6.76	6.0	3.0
SI_44	FM + 44.3% SI, percolated *)	5.25	9.32	6.0	2.5
SI_50	FM + 50.0% SI, percolated *)	3.72	8.29	6.0	1.0
SI_100	SI, percolated	-	16.24	6.0	-
FM_flood	FM, flooded with LI	8.71	-	37.0	-
experiment 2					
Percol1	FM + 20.7% SI, percolated *)	6.30	3.70	5.0	3.0
Percol2	FM + 20.7% SI, percolated *)	6.30	3.65	5.0	3.0
FloodLI1	FM, flooded with LI	11.73	-	36.0	-
FloodLI2	FM, flooded with LI	10.78	-	36.0	-
FloodPW1	FM, flooded with PW	9.41	-	-	37.0
FloodPW2	FM, flooded with PW	9.52	-	-	37.0
ChoppLI1	chopped FM, flooded with LI	11.20	-	37.0	-
ChoppLI2	chopped FM, flooded with LI	11.06	-	37.0	-
Aeration	FM + 2 L PW, aerated for 48 h, then flooded with LI	10.29	-	26.0	2.0

*) mixtures FM + SI are in % w/w on a TS-basis: (kg TS_{SI})/(kg TS_{FM} + kg TS_{SI})*100%

Table 2
Material characterization

	TS	VS	
	% wet w	% TS	% wet w
experiment 1			
fresh substrate	38.0	89.2	33.9
solid inoculum	17.0	81.4	13.8
liquid inoculum	2.2		0.8
experiment 2			
fresh substrate	32.2	85.8	27.6
solid inoculum	14.4	75.2	10.8
liquid inoculum	1.2		0.6

Table 3
Methane yield from component fresh material (G_{FM}) after different digestion times

	G_{FM} (L_N CH_4/kg $VS_{added,FM}$) *)		
	28 days	42 days	74 days
experiment 1			
FM_percol	106.3	149.5	207.0
SI_10	135.2	167.3	200.0
SI_20	145.9	174.7	208.0
SI_30	142.0	165.6	206.3
SI_40	145.9	169.7	207.7
FM_flood	144.4	169.3	204.0
experiment 2			
Percol1; Percol2		176.6; 170.9	
FloodLI1; FloodLI2		171.1; 171.9	
FloodPW1; FloodPW2		153.7; 158.8	
ChoppLI1; ChoppLI2		192.2; 192.7	
Aeration		140.2	

*) yield from component fresh material, determined according to section 2.4., methane remaining inside digesters was taken into account

Table 4
Final VS and nitrogen contents, VS removal from solid phase (SP) and from whole system (solid + liquid phase, SP + LP), methane yield per kg VS removed in experiment 2

	Percol1;2	FloodLI1;2	FloodPW1;2	ChoppLI1;2	Aeration
final VS in LP (% wet w)	1.01; 1.11	0.86; 0.82	0.42; 0.44	1.16; 0.98	0.83
final VS in SP (% wet w)	11.58; 12.19	10.84; 11.03	11.07; 11.44	8.85; 10.47	10.38
ratio VS in LP to total VS (LP + SP) (%) [a)]	4.05; 4.25	14.56; 14.35	8.82; 8.77	18.97; 16.80	15.13
final NH_4-N in LP (mg/L)	492; 512	320; 355	164; 131	415; 353	243
final TKN in LP (mg/L)	1051; 1049	798; 801	462; 463	1122; 902	790
VS removal from SP (%) [b)]	40.5; 39.8	50.7; 47.9	48.1; 46.3	54.7; 51.3	51.9
VS removal from SP + LP (%) [c)]	38.8; 37.9	45.6; 43.0	43.1; 41.1	47.5; 45.2	46.0
methane yield per kg VS removed (L_N CH_4/kg $VS_{removed,SP+LP}$)	412.3; 411.1	372.4; 396.2	369.8; 401.8	396.7; 416.0	(301.6)
mean biogas methane content (% v/v CH_4)	51.2; 51.1	53.5; 53.5	52.4; 52.8	53.0; 52.6	52.36

[a)] =(kg VS_{LP})/(kg VS_{LP} + kg VS_{SP})*100%; indicates which proportion of the final total VS was found in the liquid phase;

[b)] =(kg $VS_{removed,SP}$)/(kg $VS_{added,SP}$)*100%; with (kg $VS_{removed,SP}$)=(kg $VS_{added,SP}$ - kg $VS_{solid\ residue}$) and (kg $VS_{added,SP}$)=(kg $VS_{added,FM}$ + kg $VS_{added,SI}$); FM: fresh material, SI: solid inoculum;

[c)] =(kg $VS_{removed,SP+LP}$)/(kg $VS_{added,SP+LP}$)*100%; with (kg $VS_{removed,SP+LP}$)=(kg $VS_{added,SP+LP}$ - kg $VS_{solid\ residue}$ - kg $VS_{liquid\ residue}$) and (kg $VS_{added,SP+LP}$)=(kg $VS_{added,FM}$ + kg $VS_{added,SI}$ + kg $VS_{added,SI}$); LI: liquid inoculum;

Note: all data refer to experimental time of 46 days; the values for “final VS in SP”, “ratio VS in LP to total VS” and “VS removal from SP” are influenced by phase-separation between solid and liquid phase when emptying the reactors (not standardized in this study), therefore deviation between replicates might be high (due to different amounts of liquid retained in the solid phase); in general, phase-separation was carried out by repeatedly pumping out all liquid from the liquid-phase reservoir before removing the solid residue, but in two flooded reactors exterior filtering (PVC-cloth, mesh size 1.9 x 1.4 mm) was performed because liquid collected very slowly in the liquid-phase reservoir

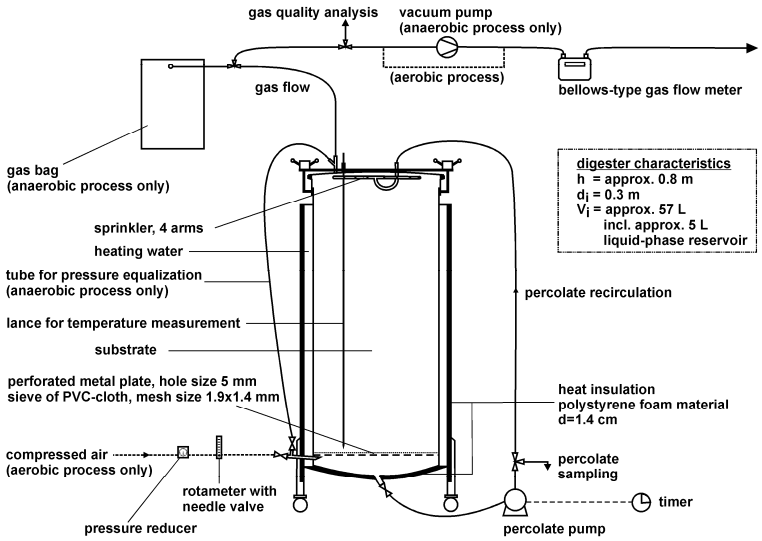


Fig. 1. Schematic diagram of laboratory-scale solid-phase digester

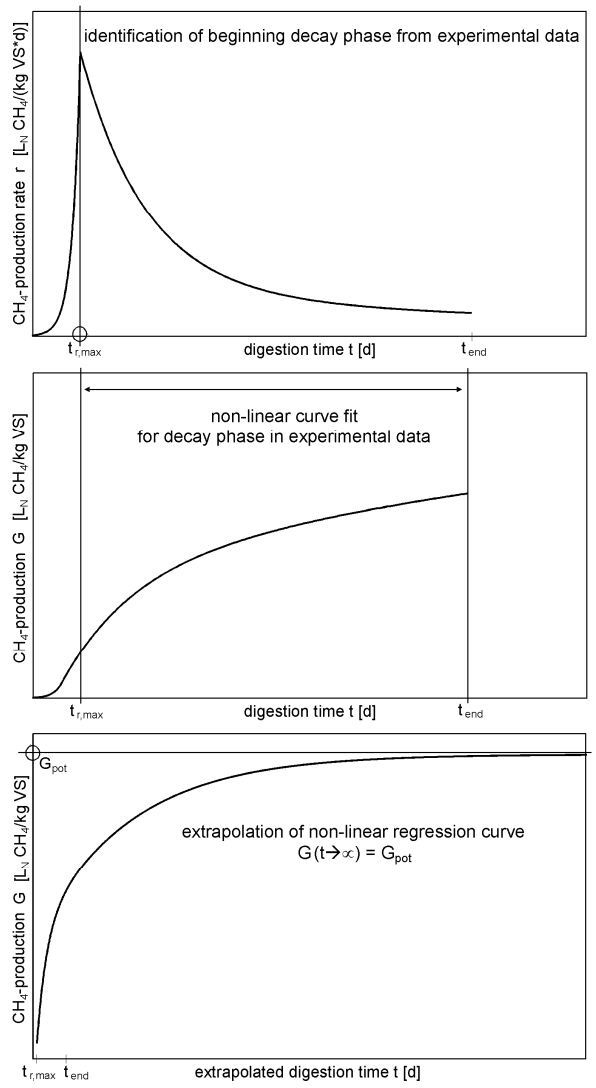


Fig. 2. Methodology for determination of total methane potential (G_{pot}) by extrapolation from experimental data

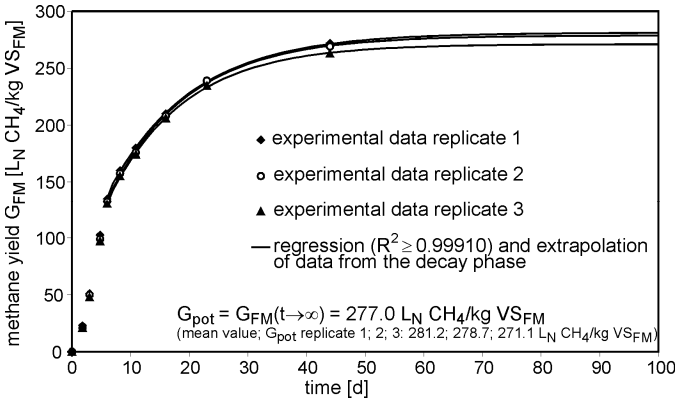


Fig. 3. Determination of total methane potential G_{pot} of horse dung by extrapolation from experimental data obtained with ground material in optimized batch-testing (Hohenheim biogas yield test HBT; contribution of inoculum is corrected)

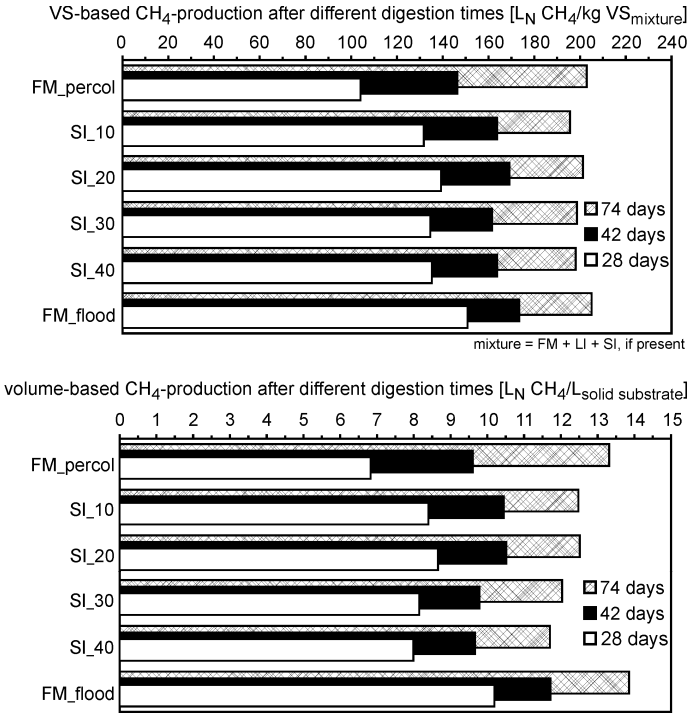


Fig. 4. Methane production in experiment 1

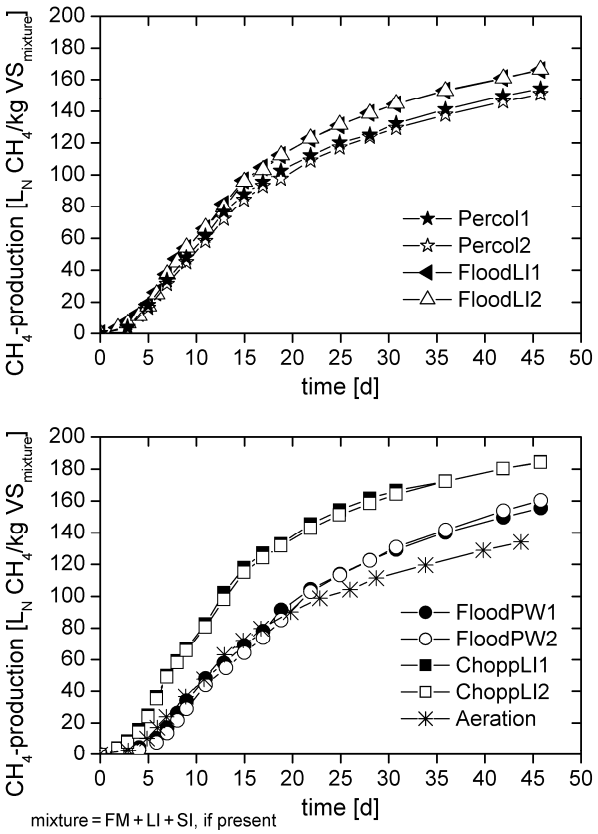


Fig. 5. Methane production in experiment 2

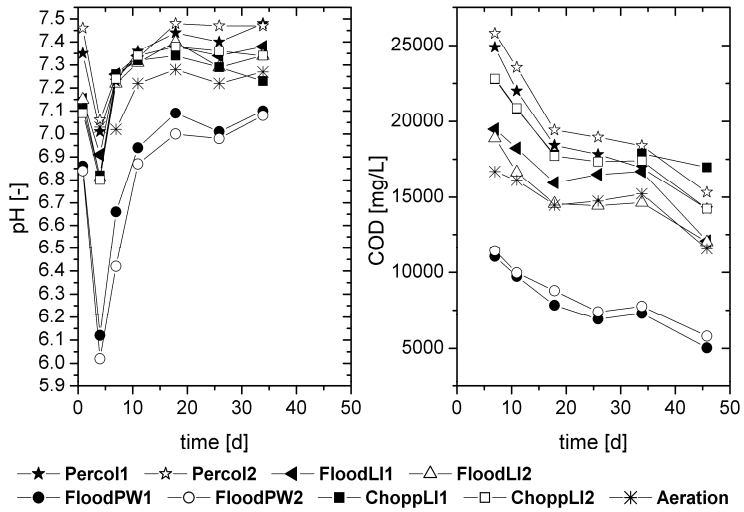


Fig. 6. pH and COD in leachate of experiment 2

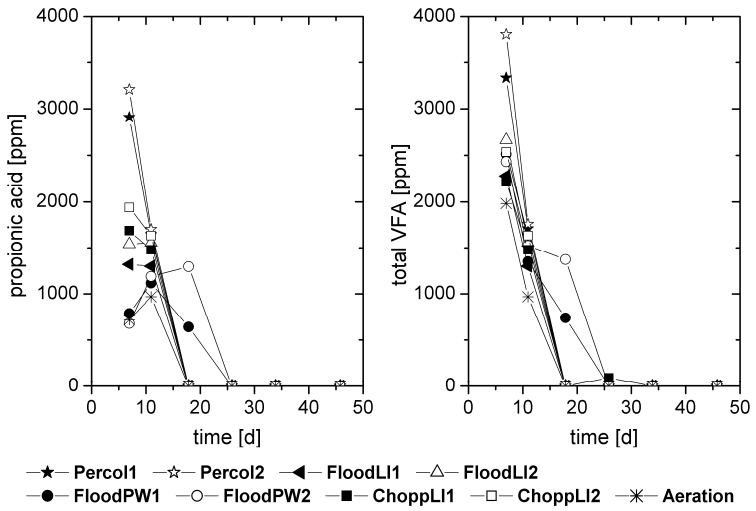


Fig. 7. Total VFA and propionic acid concentration in leachate of experiment 2

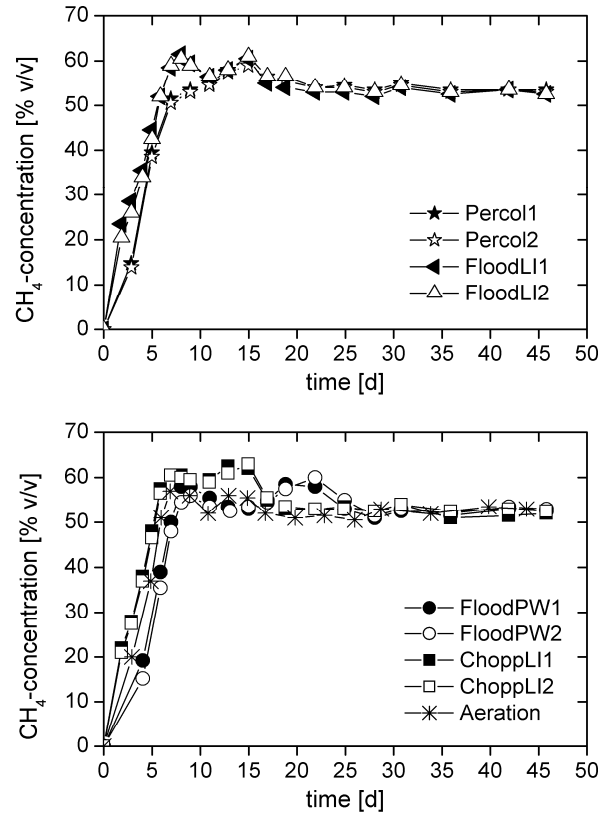
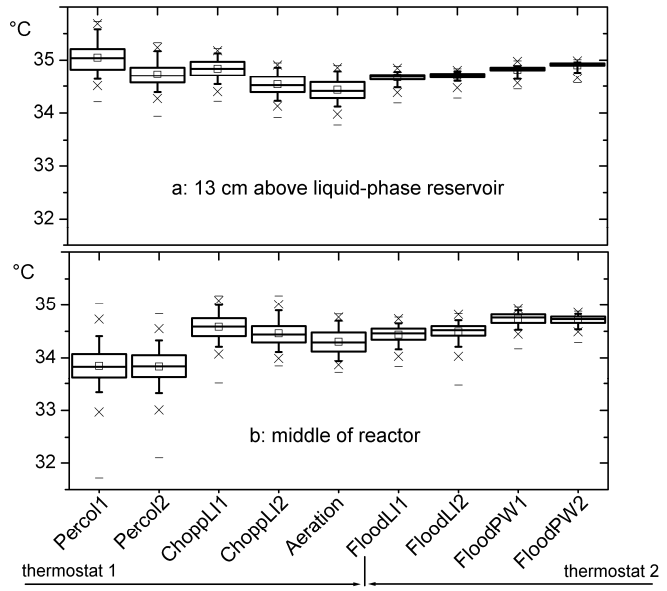


Fig. 8. Biogas methane content in experiment 2



box-whisker-plot: box contains 25th to 75th percentile, median (horizontal line) and mean (quadrat)
whiskers go to 5th and 95th percentile, X: 1st and 99th percentile, -: minimum and maximum
n=1877 for Percol/ChoppLI/FloodLI/FloodPW1/2; n=1679 for Aeration
all values during the first 24 hours of digestion are ignored

Fig. 9. Substrate temperatures during experiment 2