Kusch, S, Oechsner, H. and Jungbluth, T. (2012) 'Effect of various leachate recirculation strategies on batch anaerobic digestion of solid substrates', Int. J. Environment and Waste Management, Vol. 9, Nos. 1/2, pp.69-88.

DOI: 10.1504/IJEWM.2012.044161

http://www.inderscience.com/info/inarticle.php?artid=44161

http://www.inderscience.com/info/inarticletoc.php?jcode=ijewm&year=2012&vol=9&issue=1/2

Effect of various leachate recirculation strategies on batch anaerobic digestion of solid substrates

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Abstract: Various leachate recirculation strategies were applied to a batch-wise operated one-stage solid-phase digestion system in laboratory-scale tests. Comparative experiments with both continuous and intermittent leachate recirculation revealed no advantages of a continuous flow. Results suggest that leachate recirculation should not be carried out continuously during process initialisation when methanogenesis is the rate-limiting step. Continuous watering resulted in the accumulation of Volatile Fatty Acids (VFA) during process start-up. In addition, no need for continuous water circulation was found for the following digestion process when hydrolysis was rate-limiting. Even in the absence of liquid recirculation, degradation was only slightly retarded when the biomass moisture content was adjusted.

Keywords: biogas, dry digestion, leachate recirculation, one-stage system, batch process

Reference to this paper should be made as follows: Kusch, S., Oechsner, H. and Jungbluth, T. (2012) 'Effect of various leachate recirculation strategies on batch anaerobic digestion of solid substrates', *Int. J. Environment and Waste Management*, Vol. 9, Nos. 1/2, pp.69-88.

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Hans Oechsner, Head of the State Institute for Machinery and Construction in Agriculture (an institution affiliated with the University of Hohenheim), received his PhD after researching aerobic treatment of agricultural residues (slurry). His current research areas cover the evaluation of technologies and substrates for biogas production, the operation of sterling engines with low-calorific gases, and the combustion of hey and corn for energy generation. He teaches undergraduate and graduate courses in Bioenergy and has vast experience as consultant to Public Institutions, Farmers and Engineering Firms.

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1 Introduction

In order to treat municipal solid waste so-called 'dry digestion' processes with >20% total solids content (TS) are implemented to at least a similar extent than 'wet digestion' processes (wastes are generally slurred to <12% TS) (Bolzonella et al., 2003; Forster-Carneiro at al., 2008). Dry digestion is also referred to as 'solid-state' digestion (Martin et al. 2001; 2003; Veeken and Hamelers, 2000) or 'solid-phase' digestion (Anand et al., 1991; Chanakya et al., 1997; 1999). Continuous one-stage processes are most common, though some discontinuous (batch-operated) plants do exist (e.g. systems Bekon, Biocel). In agriculture, slurry-based liquid-phase digestion is widely applied today, but the use of yard manure and especially of energy crops is becoming more attractive (Amon et al., 2007; Weiland, 2006). Dry digestion has been studied with different agricultural substrates, e.g. solid manure (Kalia and Singh, 2001; Kusch et al., 2008; Schäfer et al., 2006; Weizhong et al., 1999; Zelter, 1978) or energy crops or other plants including crop residues (Anand et al., 1991; Andersson and Björnsson, 2002; Chanakya et al., 1997; 1999; Jewell et al., 1993; Linke et al., 2006; Liu, 1987; Parawira et al., 2008; Sun et al., 1987; Svensson et al., 2006). In full scale, digestion of solid biomass is limited in conventional slurry-plants, due to technical restrictions (e.g. related to mixing and feeding devices), and technologies appropriate for operation with elevated TS content are imperative. Due to the fact that smaller throughputs are sought in agriculture in general, direct transfer of expensive municipal systems is difficult. As batch digestion is simpler than continuous fermentation (Ten Brummeler and Koster, 1990), it will likely be the object of further development. In batch digestion, anaerobic degradation begins anew with each filling of the reactor.

The addition of an appropriate inoculum ratio prevents digester failure during start-up (Chen and Hashimoto, 1996; El-Mashad et al., 2006; Ten Brummeler and Koster, 1989). Inoculum quality is also important (Dirar and El Amin, 1988; Forster-Carneiro et al., 2007). If acetic acids are not metabolized by methanogens, the conversion of hydrolysed organics to volatile fatty acids (VFA) will result in VFA accumulation along with a drop in pH (Griffin et al., 1998). The inhibition of methanogenesis by acidity is well documented (Hori et al., 2006; Liu et al., 2002). Prevailing acidity suppresses methanogenesis but does not completely eliminate the methanogens; archea are still present (Jjemba et al., 2008). Even after several weeks of acidity, methane generation was rapid when pH was adjusted to a favourable level (Kusch et al., 2005). To assure process stability, pH values of >6.5 (Christ, 1999; Chugh et al., 1998) or >6.8 (Chen and Hashimoto, 1996; Gallert and Winter, 2008) and in general <7.5 are recommended.

Hydrolysis may also be inhibited by acidity (Vavilin et al., 2006). Especially with slowly degrading solid biomass (low hydrolysis rate) prevailing acidic conditions may significantly reduce initial hydrolysis and therefore retard the overall degradation.

According to Vavilin et al. (2006), inhibition of hydrolysis of slowly degrading biomass is among the most important factors affecting the anaerobic digestion process.

In batch-wise digested stacked biomass, liquid is sprinkled over the substrate in order to initiate biogas production and encourage bacteriological activity in the decomposing biomass throughout the process. Leachate recirculation influences degradation of solid material through two factors: moisture movement through the biomass bed and moisture content change (Chugh et al., 1998). Supplemental water also dilutes inhibitory substances (Sanphoti et al., 2006). Transport of acids to methanogens, in particular acetic acid as pricipal precursor for methanogenesis, may be a more important mechanism than the reverse process, since methanogens transported to the fresh biomass would become inactive due to acid inhibition (Martin, 1999). Leachate recirculation has been found favourable for anaerobic decomposition of landfill material (Barlaz et al., 1992; Bilgili et al., 2006; Chan et al., 2002; Demir et al., 2004; Francois et al., 2007; Ledakowicz and Kaczorek, 2004; Mata-Alvarez and Martinez-Viturtia, 1986; Mehta et al., 2002; Pohland and Al-Yousfi, 1994; Reinhart et al., 2002, Sanphoti et al., 2006), where lack of moisture is generally responsible for retarding mineralization (Chugh et al., 1998).

But it has also been reported that leachate recirculation may enhance acidogenesis in particular, which may result in inhibition of methanogenesis (Komilis et al., 1999). The risk of acidification can be limited by pre-aeration (Ten Brummeler and Koster, 1990; Vavilin et al., 2006), by leachate exchange between batches of stabilized and fresh waste (Chugh et al., 1998; 1999; Chynoweth et al., 1992; O'Keefe and Chynoweth, 2000; O'Keefe et al., 1993; Suna Erses and Onay, 2003; Poggi-Varaldo et al., 2005), by neutralising the acidifying leachate (Vavilin et al., 2003) and through buffer addition (Sanphoti et al., 2006). To prevent imbalance between acidogenesis and methanogenesis, the two key phases can also be isolated in separate reactors. Two-stage concepts are highly efficient and more stable but also more complex and more expensive (Lissens et al., 2001), and therefore, less suitable for smaller throughputs.

In this study, various leachate recirculation strategies were applied to a batch-wise operated one-stage solid-phase digestion system. Optimization strategies for biogas systems operated in batch-mode are limited since no direct process control is possible and leachate management may be one key factor. In order to study a simple process type, leachate was recirculated within the reactor and no chemicals were added. This concept may be interesting when small throughputs are desired and mainly solid substrates are available, e.g. in rural areas, developing countries and generally when decentralized biogas production is favoured. Two experimental runs were performed. In experiment 1, a substrate with a high proportion of readily degradable organic material was used, assuming a risk of acidification during process start-up if process conditions were not favourable. In experiment 2, biomass with a high initial proportion of methanogens and no assumed risk of acidification was digested, in order to study a system in which not methanogenesis but hydrolysis was rate-limiting. Process stability and methane yields were studied using the various leachate recirculation methods. Continuous and discontinuous process water recirculation was performed. Whether degradation was retarded when no water recirculation took place after that the initial water content of the biomass had been adjusted to at least water holding capacity was also determined.

2 Material and Methods

2.1 Reactor Design and Experimental Configuration

Leachate of the one-stage solid-phase digestion reactor (Figure 1: cylindrical reactor, thermostatted at 35°C, around 50 L stacked biomass with no mixing during digestion) was collected in a liquid-phase reservoir and sprinkled over the biomass (average liquid flow 4.7 L/min, four-arm sprinkler with 24 holes of 2.5 mm). Biogas was collected in aluminium coated PE/PTFE-gas bags.

'Insert Figure 1'

All tests were initiated with duplicates (two parallel test cells). Two experimental runs with eight test cells each were performed. Ensiled maize was mixed with solid inoculum (pre-digested biomass consisting mainly of municipal green cut with woody components). Mixtures of structure material (e.g. green cut or straw) and readily degradable biomass (e.g. grass or maize) are assumed to be favourable in this reactor type, since they limit compaction while still enabling satisfactory biogas yields. According to Kusch et al. (2005) batch-wise digestion of maize with intermittent leachate recirculation requires the addition of around 70% inoculum. In this study, in experiment 1 a mixture of 25% (w/w on a TS-basis) maize and 75% solid inoculum was digested. It was assumed that this substrate had a risk of acidification during process start-up. In experiment 2 the proportion of maize was 15% (w/w on TS-basis), representing a biomass with a high initial proportion of methanogens and no assumed acidification risk. This permitted the study of the effect of leachate circulation in a system with stable methanisation. Table 1 summarizes the experimental set-up. Liquid inoculum was added to the solids in all test cells to ensure the presence of free leachate for recirculation.

'Insert Table 1'

In experiment 1 (25% maize) leachate was recirculated discontinuously (twice daily for 15 minutes) in two replicates (hereafter referred to as M25_disc1 and M25_disc2). Continuous leachate recirculation was applied in two other test cells (M25_cont1, M25_cont2). For two cells (M25_stat1, M25_stat2) the biomass was completely soaked with liquid before beginning digestion (thus reaching at least water holding capacity of the substrate by allowing the presence of free water) and no process water recirculation took place throughout the digestion process. In order to derive methane yields from the component maize from the total yields of mixtures, solid inoculum was also digested separately with discontinuous leachate recirculation (SI1_disc1, SI1_disc2).

In experiment 2 (15% maize) leachate recirculation was 2 x 15 min/d in two reactors (M15_disc1, M15_disc2) and continuous in two others (M15_cont1, M15_cont2), as in experiment 1. In two test cells (M15_vari1, M15_vari2) recirculation was changed during the process: after continuous liquid sprinkling during the first six days, watering was discontinuous afterwards (2 x 15 min/d). Pure solid inoculum was also digested (SI2_disc1, SI2_disc2).

Experiment 1 was run over a period of 42 days and experiment 2 over a period of 56 days, although all test cells still released some methane. Experiments were terminated when methane productivity of mixtures reached the level of methane productivity of pure

solid inoculum. It was assumed that all significant further methane production would originate from the solid inoculum, which contained slowly hydrolysable green cut.

2.2 Substrate Characterization and Handling

In experiment 1, ensiled maize with a TS-content of 29.7% and a VS/TS-content of 95.6% was used. In experiment 2 the processed maize had a TS-content of 25.3% and a VS/TS-content of 95.7%. Maize was taken out of a full-scale silo at one of the university's experimental farm facilities.

Solid inoculum for both experimental runs was collected at a full-scale solid-phase digestion plant. In experiment 1, the inoculum (TS = 36.8%, VS/TS = 58.7%) was a pre-digested mixture of green cut (high proportion of woody components from bush cut, chopped to approx. 10 cm before digestion) with 15% ensiled grass. In experiment 2, the solid inoculum (TS = 34.2%, VS/TS = 51.2%) was a pre-digested mixture of green cut (abovementioned type) with 30% ensiled maize. Liquid inoculum (experiment 1: TS = 0.7%, VS/TS = 45.5%; experiment 2: TS = 0.4%, VS/TS = 72.5%) for each experiment was gained from a previous solid-phase digestion laboratory experiment (with maize; collected liquid was diluted 1:1 v/v with potable water).

No intermediate storage of maize took place prior to starting the experiments. Solid inoculum was stored not longer than 12 hours at 5°C under anaerobic conditions. Due to operational requirements between different experimental runs, liquid inoculum had to be stored several (5-7) days. The stored liquid was thermostatted (35°C, anaerobic), stirred twice daily and fed with small proportions of ensiled maize on day 1 and 3.

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.

2.3 Sampling and Analyses

In reactors with discontinuous process water recirculation, leachate was additionally 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 of all reactors (aside from M25_stat1, M25_stat2) was sampled periodically and analysed for pH, VFA, 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.

Solid samples were analysed directly. Liquid samples were analysed either directly, either within 24 hours after storage at 5°C or 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 (solids 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 and pH conformed to DIN/EN-standards. VFA represent the sum of acetic, propionic, butyric, valeric and caproic acids and were measured by gas chromatography. Further details of analytical methods were provided by Kusch et al. (2008).

Biogas was analysed for CH_4 and CO_2 content (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, 1.013 bar, 0°C).

2.4 Consideration of Methane Remaining in Reactors and Calculation of Methane Yield from Fresh Material in Mixtures with Solid Inoculum

A certain amount of biogas is not released from the reactor but remains inside, filling pores of the substrate stack and void spaces (e.g. as occurs after substrate compaction). Methane volumes remaining inside fermenters at the end of an experiment were determined as follows: fresh air was blown into the reactor at the reactor bottom (approx. 120 L air at a flow rate of 45 L/min) and the released gas was collected in gas bags and analysed for its methane content and its exact volume. Multiplying those two values determines the methane volume previously present inside the fermenter, under the assumption that all biogas was released from the reactor and replaced by fresh air.

These volumes are not taken into consideration when methane release from a reactor is presented in cumulative form over digestion time. But they are taken into account when deriving the methane yield of maize from the total mixture yield.

In mixtures with solid inoculum and/or liquid inoculum the methane yield from the component fresh material (ensiled maize) 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). In both experiments the methane yield from solid inoculum was determined by digesting the inoculum separately (SI1_disc1/2, SI2_disc1/2). Methane yield from liquid inoculum was determined aside. For these calculations the total methane yields of all fractions (methane released from digester plus methane remaining inside void digester spaces) were taken into account.

3 Results and Discussion

3.1 Experimental Results

In this study, VFA throughout the digestion process were monitored as indicator of the development of a stable methanogenic population. Total VFA concentrations are given for both experimental runs in order to facilitate direct comparison (Figure 2 and 5) between the experiments and with literature data. In addition, in experiment 1 with a risk of accumulation of long-chain fatty acids, the most relevant acids are given with Figure 3. In the degradation of complex substrates, breakdown of long-chain fatty acids is usually regarded as a rate-limiting step (Salminen et al., 2000).

‘Insert Figure 2’

‘Insert Figure 3’

In experiment 1, with a high proportion of readily hydrolysable biomass (25% maize), the pH dropped to 5.3 on day 4 before gradually rising to values between 7.6 and 8.0 with both continuous and discontinuous leachate recirculation (Figure 2). The initial drop in pH indicates that the system was not adequately buffered with alkalinity. Chanakya et al. (1993) observed no pH change with VFA peaks up to 4 g L^{-1}. It has also to be considered that maize silage itself is sour. The VFA pattern is in agreement with literature data, rapid hydrolysis of herbaceous biomass and conversion to VFA within few days has been reported for different feedstock (Chanakya et al., 1997). But continuous recirculation (M25_cont1/2) resulted in longer acidification with more persistent VFA accumulation. With discontinuous leachate recirculation (M25_disc1/2), long-chain VFA (e.g. butyric, caproic acids) occurred in a comparable or slightly lower intermediate concentration than with continuous water circulation, but degradation in M25_disc1/2 was more rapid (Figure 3). According to Chanakya et al. (1993) predominance of acetic and propionic acids suggests that fermentation is normal. Acetic acid, the principal direct substrate for methanogens, represented around half of the total VFA concentration in acidified test cells, whereas the other dominant component was n-butyric acid (up to 13,000 ppm). In particular branched chain iso-butyric or valeric acids were attributed to digester failure (Hill and Bolte, 1989); their levels were low in this study (data not shown). As mentioned by Gallert and Winter (2008), propionate oxidizers seem to be the most critical organisms in the anaerobic food chain and should be paid particular attention during restart periods. Pullammanapallil et al. (2001) observed no effect on methane generation in the presence of 2.8 g L^{-1} propionic acid at a pH below 6.5 and cite tolerable concentrations up to 8 g L^{-1}, though in some cases lower concentrations were reported inhibitory; they concluded that accumulation of propionic acid may be the result and not the cause of imbalanced methanogenesis. After digester overload in the initial presence of high levels of VFA, persistence of propionate was observed even after consumption of other VFA (Griffin et al., 1998). In experiment 1, propionic acid was found in concentrations of up to 4,000 ppm; in general, maximum levels appeared later in time than maximum levels of other acids. All leachate VFA were degraded below detectable levels when experiment 1 was terminated after 56 days, aside from M25_cont1 where some propionic acid was still measured. Depletion of accumulated VFA indicates that methanogenesis is no longer the rate-limiting step and that degradation is controlled by the hydrolysis rate. Measurement data suggest that continuous water circulation significantly retarded the establishment of the methanogenic population.

Despite intermediate VFA concentrations of >25,000 ppm methane generation was not suppressed. A level about 30 g VFA L^{-1} is often referred to as inhibiting anaerobic digestion (Vavilin et al., 2004). Vavilin et al. (2006) report inhibition of both hydrolysis and methanogenesis in the presence of 5 g VFA L^{-1}; also Cho et al. (1995) observed significant methanisation at VFA levels below 5 g L^{-1}. VFA toxicity to the microbial community is higher at low pH (Dearman et al., 2006; Pullammanappallil et al., 2001); non-ionized VFA and/or proton concentrations are important factors (Vavilin et al., 2006). With a pH value around 3.7, Ghanem et al. (2001) observed methane production at 9.4 g VFA L^{-1} while it was stopped at 11.4 g VFA L^{-1}. In experiment 1, methane production from continuously watered test cells (M25_cont1/2) was lower throughout the eight weeks of operation (Figure 4) compared to intermittent water recirculation (M25_disc1/2). Although the duplicates M25_cont1 and M25_cont2 did not show complete parallelism, when compared to other test cells, trends can be identified. After an inhibited start-up with low initial methane production rates, maximum production rates

were reached later and final methane production rates were still high, indicating that degradation had not yet been completed after eight weeks. All test cells still released some methane after eight weeks, most probably due to degradation of the solid inoculum.

'Insert Figure 4'

M25_stat1/2 with no process water recirculation showed only slightly poorer performance than M25_disc1/2 with discontinuous watering. After a comparable start-up phase, methane production was 5% lower after 6 weeks and 4% lower after 8 weeks. Methane concentrations were quite similar in all four test cells, with intermediate maxima of around 70% (v/v) after three weeks, final concentrations of 55% after eight weeks and mean concentrations of around 50% over the whole digestion time (data not shown).

'Insert Figure 5'

In experiment 2 with mixtures containing a low proportion of readily degradable substrate (15% maize), differing process water recirculation strategies had no significant influence on pH value or VFA concentration in the leachate (Figure 5). In addition, no influence on the VFA spectrum (data not shown) or the methane production (Figure 6) was observed. Methane production began similarly and there was little difference in the methane productivity throughout the process, regardless of whether process water was recirculated continuously (M15_cont1/2) or discontinuously (M15_disc1/2). Switching from continuous to intermittent recirculation (M15_vari1/2) was of no impact.

'Insert Figure 6'

Table 2 lists methane yields from the component maize for all test cells. Differences in experiment 2 were marginal. In experiment 1, deviation of the duplicates M25_cont1 and M25_cont2 was high, but neither yielded the same amount of methane as M25_disc1/2 after 42 days. After 56 days, yield from M25_cont2 was in the range of M25_disc1/2, while it was still 21% lower from M25_cont1. With no water circulation (M25_stat1/2), a 5% lower methane yield than M25_disc1/2 was achieved after 42 days.

In experiment 2, the methane yield of maize was 398.2 ± 3.5 L_N CH_4 kg^{-1} VS_{added} (mean ± SD; n = 6; M15_disc/cont/vari/1/2), indicating an efficient digestion process. In experiment 1, the methane yield of maize in M25_disc1/2 was only slightly lower. According to Oechsner et al. (2003) the maximum methane potential of maize is 400 L_N CH_4 kg^{-1} VS_{added} (determined in optimized batch-test with ground material and different types of maize). Amon et al. (2007) determined an average specific methane yield of 398 L_N CH_4 kg^{-1} VS_{added} and no significant differences between maize varieties.

'Insert Table 2'

Using experimental data (methane yields of mixtures and inocula including CH_4 remaining inside reactors, total mass and VS content of solid and liquid digestates), the methane yield of the component ensiled maize per kg VS removed was calculated. A value of 424.2 ± 8.6 L_N CH_4 kg^{-1} $VS_{removed}$ (n = 5; M25_disc/stat/1/2, M25_cont2; 56 days) was determined in experiment 1, and 419.8 ± 8.2 L_N CH_4 kg^{-1} $VS_{removed}$ (n = 6; M15_disc/cont/vari/1/2, 42 days) in experiment 2. Methane yield per kg VS removed is more than the theoretical value for carbohydrates alone (395 L_N CH_4 kg^{-1} VS based on

Buswell's equation). Contents of carbohydrates, proteins and lipids of maize were not determined in this study (proteins: 497 L_N CH_4 kg^{-1} VS, lipids: 850 L_N CH_4 kg^{-1} VS based on Buswell's equation). Lemmer (2005) reports an average crude protein content of 7% expressed as % of TS for different maize silages; Oleskowicz-Popiel et al. (2008) use a standard value of 8%. With 421.8 ± 8.3 L_N CH_4 kg^{-1} $VS_{removed}$ (n = 11; experiment 1 and 2, abovementioned cells), the average methane yield per kg VS removed is only around 6% higher than per kg VS added, indicating a high degradation efficiency (94% VS removal). According to Resch et al. (2008), in an efficient digestion process 11% of the initial VS of maize will not be degraded, of which 6% represent residual methane potential and hence 5% non convertible VS.

However, it should be kept in mind that in this study all methane yields for maize were derived from the total methane yield of the mixture, assuming that yields of inocula do not change in mixtures. In this study, it was not possible to verify this assumption. If the methane yield of inoculum was increased when digesting the mixtures, calculation procedure results in an overestimation of the methane yield of maize since any additional methane generation is assumed to originate from maize.

3.2. Evaluation of Experimental Findings

Results demonstrate that the solid-phase digestion process was highly efficient and high methane yields were achieved when no inhibition occurred.

Further evaluation of the experimental data reveals that biogas production with solid biomass in batch-wise operated one-stage reactors is sensitive to the leachate recirculation strategy. No positive effect was found for continuous recirculation, neither while methanogenesis was the rate-limiting step and development of a methanogenic population was essential for the further degradation, nor while methanisation was in a stable phase and hydrolysis was the rate-limiting step.

Optimal conditions for methanogens are especially important in the initial stage. Continuous water recirculation resulted in a higher risk of acidification. This is in agreement with literature data. When initiating an anaerobic digestion process, low leachate recirculation rates were also recommended by Veeken and Hamelers (2000) and Vavilin et al. (2002; 2003). In small full-scale applications with very simple technical equipment the liquid flow of pumps can often not be regulated (direct variation of leachate sprinkling rate is not possible). Results of this study provide evidence that the intermittent and short operation of pumps is an appropriate strategy.

In a bed of organic substrate, anaerobic digestion is initiated by seed bodies around which reaction zones gradually develop (Martin et al., 2003). Kalyuzhnyi et al. (2000) assumed seed particles with high methanogenic activity and highly degradable particles of fresh waste with low (if any) methanogenic activity. Chanakya et al. (1997) mentioned acidogenic and methanogenic pockets. Veeken and Hamelers (2000) postulated that increasing the transport of VFA from acidogenic to methanogenic pockets would be positive unless the methanogenic activity of the seed was too low to consume the acids. A low liquid flow in the initial stage is beneficial for the expansion of methanogenic areas (Vavilin et al. 2002; 2003). Otherwise, when incoming acids cannot be sufficiently degraded in methanogenic areas, acidogenic areas expand. High rates of mass transfer may spread inhibition after overwhelming the assimilative capacity of the methanogens, while low rates may simply retard the process (Martin, 1999). During start-up, a vigorous mixing was also found unsuitable since it might dissipate methanogenic centres (Vavilin

and Angelidaki, 2005; Vavilin et al., 2004). The existence of separate methanogenic and acidogenic areas might also explain why digestion was found more stable when fresh biomass and solid inoculum had not been mixed, but were placed into the reactor in layers (Forster-Carneiro et al., 2004). When digesting the substrate from experiment 2 in two layers (instead of carefully mixing fresh material and solid inoculum) and with discontinuous process water recirculation, a reduced risk of acidification was found, as was a significantly slower biogas generation (data not shown).

Veeken and Hamelers (2000) and Vavilin et al. (2002) recommend increasing the leachate recirculation rate when it is hydrolysis that is rate-limiting and no longer methanogenesis. A higher rate of mass transfer between methanogenic and acidogenic areas (Vavilin et al., 2002) and improved solubilisation (Chugh et al., 1998) were associated with higher recirculation rates. Results of the present study do not indicate any beneficial effects from a continuous leachate recirculation in a system with stable methanogenic population (experiment 2). Continuous (compared to intermittent) leachate recirculation did not have any significant impact. Degradation kinetics and methane generation followed the same pattern throughout the process. When switching from continuous to intermittent watering, no effect on microbiological activity could be observed. When process water recirculation was applied discontinuously, leachate COD and VS content were slightly higher during the first 15 days of operation (Figure 7) than with continuous watering, while methane generation showed no difference. This indicates that a continuous water flow through the biomass bed did not improve biomass solubilisation in this study (no higher proportion of organic material found in the liquid while methanisation was similar). The average leachate COD/VS-ratio gradually fell from initially 2.21 ± 0.19 (n = 6, M15_disc/cont/vari/1/2) to a final value of 1.32 ± 0.10. The relationship between COD and VS is known to vary considerably with changing nature of the liquid's content. For example, the COD/VS-ratio for carbohydrates is about 1.1, whereas for a lipid it is about 2.9 and for a protein 1.5 (Burton and Turner, 2003).

'Insert Figure 7'

Besides a moisture flow through a biomass bed, the biomass moisture content itself is an important factor in anaerobic degradation (Pommier et al. 2007; Hao et al., 2008). According to results from experiment 1, digestion of biomass with its water content adjusted to water holding capacity may proceed quite similarly both with and without leachate recirculation. Methane yield was only 5% higher after six weeks when leachate recirculation was applied. This observation is only partly in agreement with results reported by Klink and Ham (1982) for landfill material. They found that moisture flow significantly increased methane production (at least +25%) relative to waste at the same moisture content with no moisture flow.

Leachate recirculation has been found to be the most practical approach to moisture content control for landfills, but waste characteristics, most notably large pore volumes and heterogeneity, lead to rapid vertical flow of leachate along preferential flow paths and consequently, incomplete use of available moisture storage (Reinhart et al., 2002). The adjustment of biomass water content prior to digestion seems advisable and is easily feasible in biogas generation, while its use is limited in landfill operations.

Hydrogen was not measured in this study. H_2 plays a key role in the anaerobic process and may be a good indicator for imbalanced methanogenesis (Mormille et al., 1996). Methanogens occupy the terminal position in the anaerobic food chain and are

classified in two groups based on their substrate conversion capabilities. Around 70% of the overall methane originates from acetate, converted by acetoclastic methanogens. Hydrogenotrophic species convert H_2 and CO_2 to CH_4 and maintain the H_2 partial pressure low (< 10 Pa), which is necessary for the functioning of intermediate groups which convert acids and alcohols to direct methane precursors (Montero et al., 2008).

4 Conclusions

Discontinuous digestion with elevated TS content promises further application in agriculture and MSW treatment, especially when smaller throughputs are sought. Robustness and flexibility (Forster-Carneiro at al., 2008), and low water consumption (Köttner et al., 2003) are some of the advantages associated with this concept. In addition, no problems will occur with fibrous materials floating on top of the liquid, a phenomenon often observed in wet digestion of lignocellulosic substrates such as straw or straw-containing dung, e.g. from horses (Kalia and Singh, 1998) or sheep (Shan, 1992). Both simple and appropriate for smaller throughputs, batch-operated one-stage solid-phase digestion has potential for application not only in developing countries, but in general when decentralized biogas production is favoured. The main disadvantages of this concept are the high ratio of seeding material needed to prevent irreversible acidification at the start-up of the process and long retention times in the reactor (Veeken and Hamelers, 2000). Optimization strategies are limited since no direct process control is possible during batch-wise digestion. Leachate management is one key factor.

In this study various leachate recirculation strategies were researched. Continuous recirculation of leachate was not found favourable. Neither during process start-up nor during the further digestion process could any positive effect be observed. During process start-up a higher risk of acidification and consequently an inhibited methane production was found. In a system with stable methanisation no significant difference between discontinuous and continuous leachate recirculation was observed. Switching from continuous to intermittent recirculation had no effect. Energy consumption is higher when pumps are operated continuously. Since no positive impact on biogas production can be concluded from the results of this study, intermittent operation of recirculation pumps is advisable both during start-up and during the further digestion process.

Adjusting the moisture content of solid biomass prior to process initiation is advisable, as biological activities are limited by an inadequate supply of water. Initial watering of the whole substrate stack seems more appropriate than gradual watering through leachate recirculation, which will efficiently adjust water content only when operated continuously. Provided the water content of the biomass is favourable and the proportion of solid inoculum added is adequate, long process water recirculation intervals can be chosen and even a complete lack of leachate recirculation will still result in stable methane production. With very little leachate recirculation, methanisation may be retarded but the digestion process may fail if recirculation is too intensive.

Acknowledgements

This work was funded by the Ministry for Nutrition and Agriculture of Baden-Württemberg, Germany, as part of a research project. The authors also thank graduate

student Roman Poltoratskyy for excellent assistance with the experiments and biologist Annette Buschmann for help with the laboratory analysis.

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Table 1 Experimental set-up (M: ensiled maize; SI: solid inoculum; LI: liquid inoculum)

		M	SI	LI
		kg	kg	L
experiment 1	25.1% (w/w TS) M + 74.9% SI*			
M25_disc1, M25_disc2	discontinuous leachate recirculation	5.3	12.8	10
M25_cont1, M25_cont2	continuous leachate recirculation	5.3	12.8	10
M25_stat1, M25_stat2	no leachate recirc. after initial soaking	5.3	12.8	10**
SI1_disc1, SI1_disc2	100% SI, discontinuous leachate recirc.	-	19.0	10
experiment 2	15.4% (w/w TS) M + 84.6% SI*			
M15_disc1, M15_disc2	discontinuous leachate recirculation	4.0	16.3	10
M15_cont1, M15_cont2	continuous leachate recirculation	4.0	16.3	10
M15_vari1, M15_vari2	variation of leachate recirculation	4.0	16.3	10
SI2_disc1, SI2_disc2	100% SI, discontinuous leachate recirc.	-	20.3	10

*mixture proportions are given in % w/w on a TS-basis, the proportion of maize (M) is determined as follows: (kg TS_M)/(kg TS_M + kg TS_{SI}) x 100%

**Although no process water recirculation was performed, the same amount of liquid inoculum was added in order to ensure that all test cells contained the same amount of total organic material; some of the liquid was retained by the biomass during the initial soaking and surplus liquid remained in the liquid reservoir at the reactor bottom.

Table 2 Methane yield of ensiled maize (derived from total methane yields of mixtures by calculation, assuming that methane yields of inocula do not change in mixtures; contributions of inocula are corrected, all methane volumes remaining inside reactors by the end of the experiment are taken into account as described in section 2.4)

	methane yield ensiled maize [$L_N\ CH_4\ kg^{-1}\ VS_{added}$]	
	42 days	56 days
experiment 1		
M25_disc1; M25_disc2	369.1; 357.8 (363.5)	390.0; 387.6 (388.8)
M25_cont1; M25_cont2	81.2; 311.1	308.2; 386.3
M25_stat1; M25_stat2	344.6; 343.7 (344.1)	371.3; 373.8 (372.6)
experiment 2		
M15_disc1; M15_disc2	403.7; 398.7 (401.2)	
M15_cont1; M15_cont2	400.7; 394.7 (397.7)	
M25_vari1; M25_vari2	396.5; 394.9 (395.7)	

Data in brackets represent mean value of duplicates; no mean value given for M25_cont1/2 since deviation of the replicates is high.

Figure 1 Schematic diagram of laboratory-scale solid-phase digester

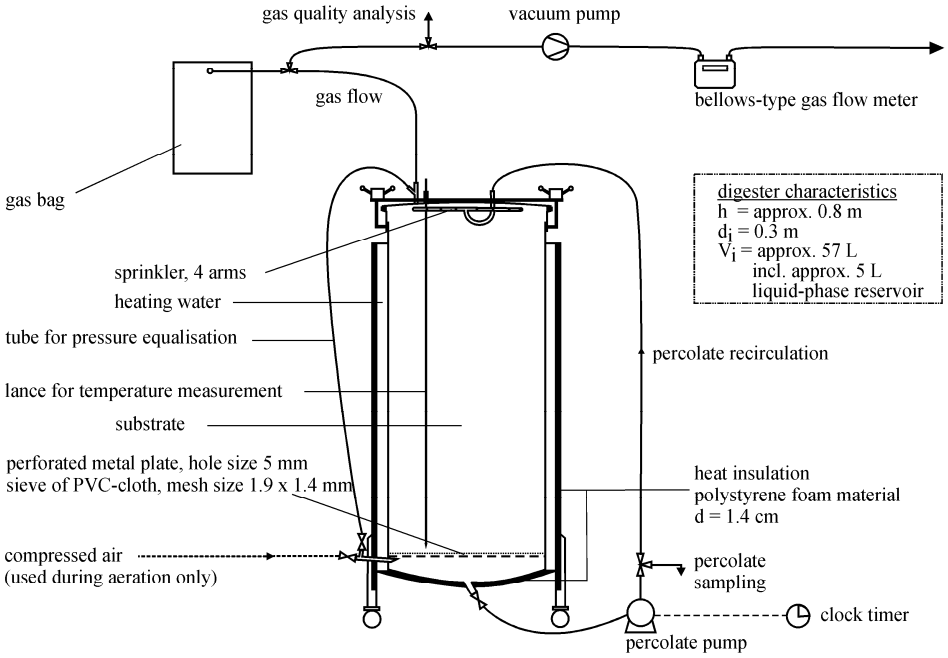

Figure 2 pH values and VFA concentrations in experiment 1 with a high proportion of readily degradable organic material (25% ensiled maize)

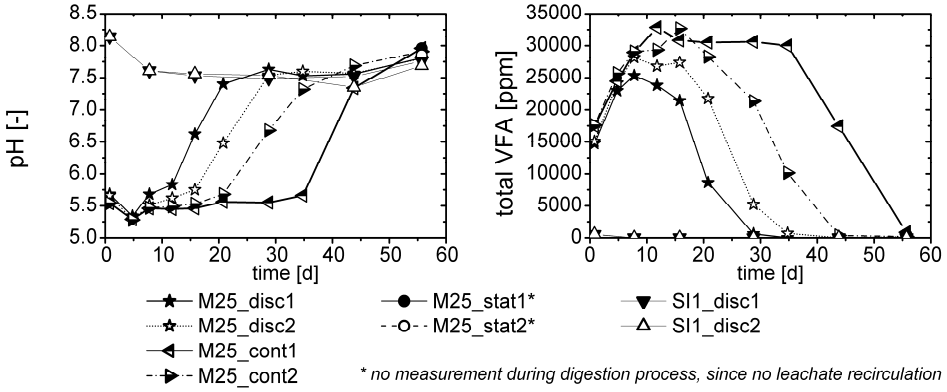

Figure 3 VFA spectrum in experiment 1 (high proportion of readily degradable organic material) over digestion time

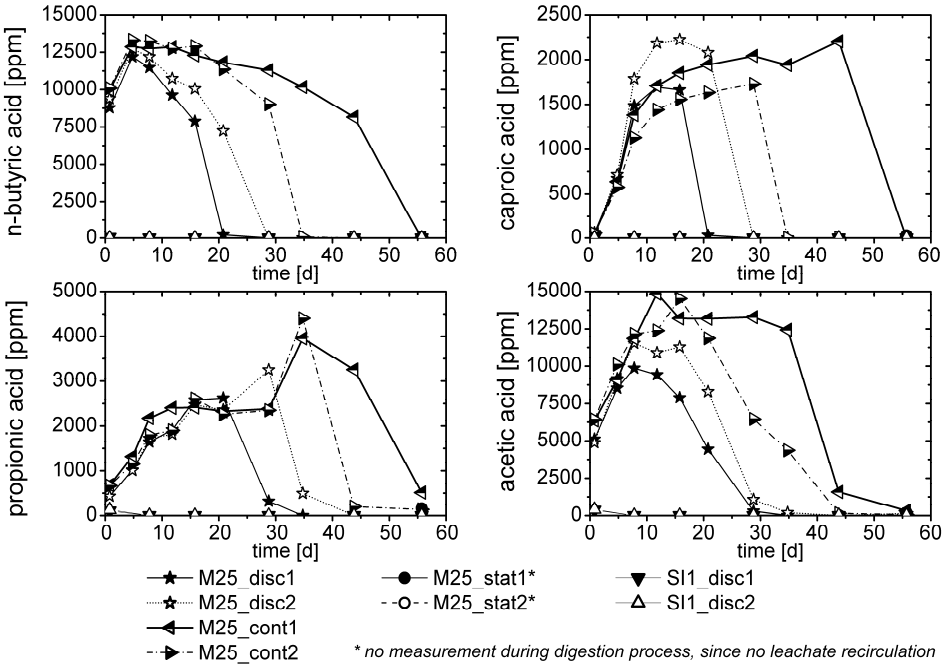

Figure 4 Methane production and methane production rate over digestion time in experiment 1 with a high proportion of readily degradable organic material (all values represent methane released from test cells, gas remaining inside reactors is not considered; production rate: mean value in reading interval displayed in middle of interval)

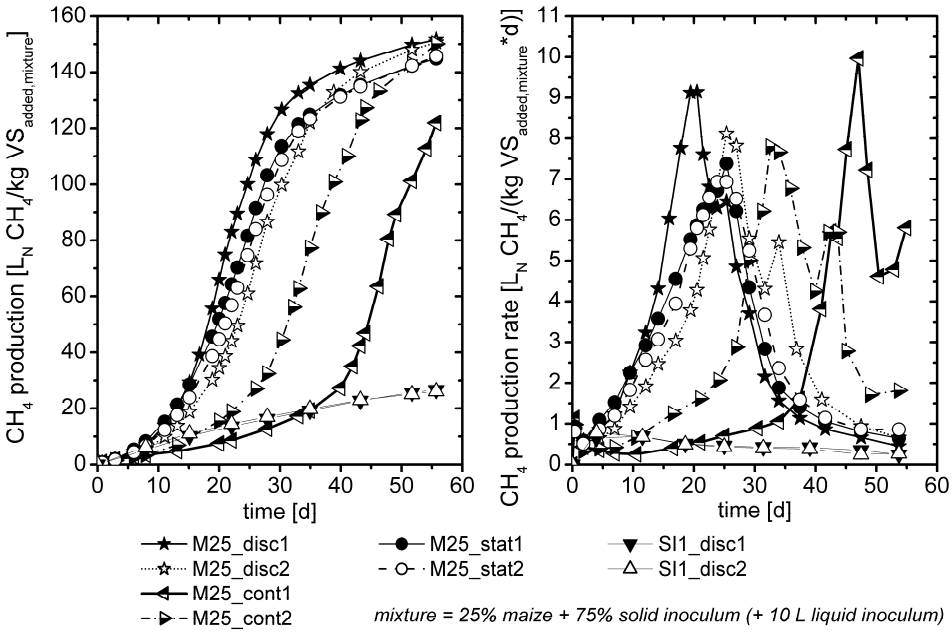

Figure 5 pH values and VFA concentrations in experiment 2 with a small proportion of readily degradable organic material (15% ensiled maize)

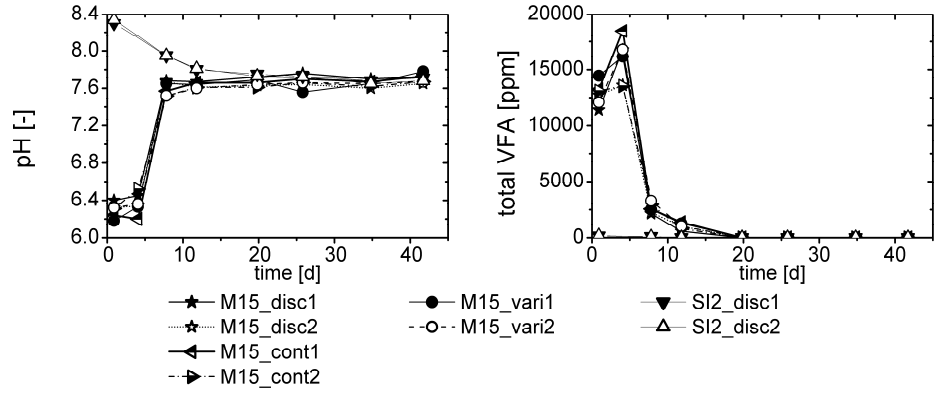

Figure 6 Methane production and methane production rate over digestion time in experiment 2 with a small proportion of readily degradable organic material (all values represent methane released from test cells, gas remaining inside reactors is not considered; production rate: mean value in reading interval displayed in middle of interval)

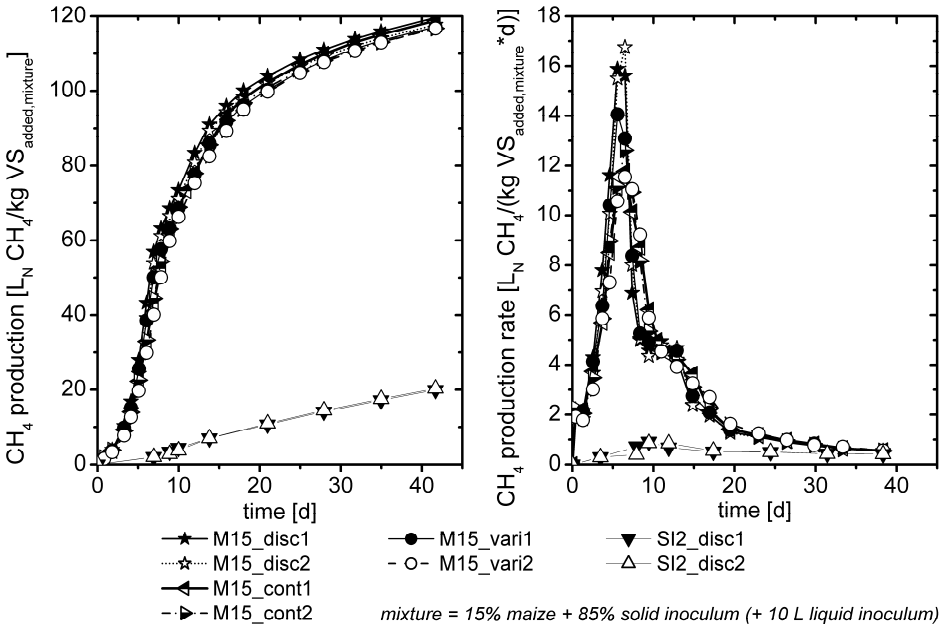

Figure 7 COD and VS content in leachate of experiment 2