Use of laboratory anaerobic digesters to simulate the increase of treatment rate in full-scale high nitrogen content sewage sludge and co-digestion biogas plants

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Abstract

The aim of this study was to assess the effect of increasing feedstock treatment rate on the performance of full-scale anaerobic digestion using laboratory-scale reactors with digestate and feedstock from full-scale digesters. The studied nitrogen-containing feedstocks were i) a mixture of industrial by-products and pig slurry, and ii) municipal sewage sludge, which digestion was studied at 41 and 52 °C , respectively. This study showed the successful reduction of hydraulic retention times from 25 and 20 days to around 15 days, which increased organic loading rates from 2 to 3.5 kg volatile solids (VS) /m^{3}d and 4 to 6 kgVS/m^{3}d. As a result, the optimum retention time in terms of methane production and VS removal was 10–15% lower than the initial in the full-scale digesters. Accumulation of acids during start-up of the co-digestion reactor was

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suggested to be connected to the high ammonium nitrogen concentration and intermediate temperature of 41 °C.

Keywords

Anaerobic digestion; nitrogen; co-digestion; sewage sludge; hydraulic retention time

1 Introduction

Anaerobic digestion is an efficient technique for the treatment of organic wastes from different sectors, e.g. agriculture, industry and municipalities. Anaerobic digestion recovers renewable energy in the form of biogas, which can be used in combined heat and power plants, in vehicles and for grid injection; and it also allows recycling of nutrients through application of digestion residues in crop production. Both the Renewable Energy directive (2009/28/EC, European Parliament and the Council, 2009) and the Landfill directive (99/31/EC, European Council, 1999) have been strong drivers in promoting the use of anaerobic digestion for this application in recent years, and the EU Action Plant for the Circular Economy boosts the role of anaerobic digestion as a part of nutrient and material cycles (European Commission, 2015).

Full-scale anaerobic digestion plants aim to optimize energy production and solids removal in order to increase the waste treatment rate and the economy of the plant through increased gate fees, thus without compromising the digester process stability. The process stability of an anaerobic digester is dependent on the balance between micro-organisms, which are known to be vulnerable to inhibition or changes in the process variables e.g. hydraulic retention times (HRT) and organic loading rate (OLR) (McLeod et al., 2015). The optimization of the waste treatment rate can be achieved through a decrease in the HRT and increase of the OLR, which, to a certain point, can increase the methane production during digestion (Nges and Liu, 2010). However, the increasing OLR can lead to a process imbalance, accumulation of acids and a decrease in biogas production if the retention times are not sufficient for microbial growth (Regueiro et al., 2015). Additionally, the formation of increased amounts of inhibitory compounds, e.g. ammonium nitrogen (NH_4-N, Beale et al., 2016), can affect the process stability (Rajagopal et al., 2013). Nitrogen-containing feedstocks (e.g. slurries/manures, sewage sludges, food wastes and certain industrial by-products) naturally contain a large amount of proteins, which is degraded and mineralized into NH_4-N during digestion. Subsequently, NH_4-N has been reported to be the major toxic compound in full-scale digesters utilizing high nitrogen containing feedstocks (Fotidis et al., 2014). Additionally, the temporal and seasonal variation of the feedstock composition (e.g. sewage sludges, industrial organic wastes) can be challenging (Lee et al., 2016; McLeod et al., 2015; Regueiro et al., 2015). Thus, to improve the digestion process and increase the treatment rate also with challenging feedstocks such as industrial and food wastes, manures and sewage sludge, the stability of the digesters and the quality of the digestate are not to be compromised (Nges and Liu, 2010). With laboratory-scale experiments, the effect of the optimization of the digesters at full scale can be studied, without jeopardizing the actual full-scale process and plant economics. However, to simulate the full-scale operation with the natural changes and variation within the feedstock composition, the feedstock composition should not be as controlled and homogenized as it is with most of the laboratory-scale digestion studies.

The total nitrogen concentration in the protein-rich, high-nitrogen-containing feedstocks increasingly used in biogas plants varies, being around 3–9 gN/kg in animal manures (Moset et al., 2015b; Regueiro et al., 2015; Zhang et al., 2014), 7–8 gN/kg in food wastes (Banks et al., 2012; Haider et al., 2015; Tampio et al., 2014), 2–5 gN/kg (Leite et al., 2016; Lloret et al., 2013) and even 9 gN/kg (Zhang et al., 2014) in sewage/wastewater treatment sludges, while more specified biomasses, e.g. molasses residues, can contain nitrogen up to 15 gN/kg (Regueiro et al., 2015). Full-scale anaerobic digesters treating these nitrogen-containing feedstocks usually operate at

HRTs of around 18–30 days (OLRs 1–3 kgVS/m^{3}d, Hao et al., 2016; Leite et al., 2016; Lloret et al., 2013; McLeod et al., 2015; Menardo et al., 2011; Sundberg et al., 2013) in mesophilic conditions and at HRTs of 16–20 days (OLRs 2–3 kgVS/m^{3}d, Lee et al., 2016; Lloret et al., 2013; Sundberg et al., 2013) in thermophilic conditions. High nitrogen concentration within the feedstock affects the HRTs and OLRs applied as it leads to the formation of inhibitory NH_4-N during digestion. However, due to the relatively high HRTs currently applied in full-scale plants, there is a likely potential to improve the processes by increasing the treatment rate.

In this study, the aim was to assess the feasibility of increasing the feedstock treatment rate (HRT, OLR) in full-scale anaerobic digesters treating waste materials with high nitrogen (5–9 g/kg) concentrations. The mesophilic and thermophilic laboratory digesters were first operated with similar HRTs (25 and 20 days) to the representative full-scale reactors after which the HRTs were gradually reduced to 14–15 days. Digestates from the representative full-scale reactors were characterized in order to assess the conditions in the full-scale digester. The digestates were also used as inocula for the laboratory digesters, where the feedstocks originated from nitrogen-containing feedstocks: i) a mixture of industrial by-products and pig slurry, and ii) municipal sewage sludge. The process performance was assessed with batch and continuous digestion as well as chemical analyses so as to obtain the optimum reactor performance in terms of methane yield, volatile solids (VS) removal and process stability and in order to find the treatment rate when the process becomes unstable.

2 Materials and methods

2.1. Origin of materials

Two full-scale digesters were simulated, of which the first plant was co-digesting multiple feedstocks (referred to as co-digestion feedstock, CF) and the second digesting sewage sludge (SS) (Table 1). For this study, the feedstocks and digestate (used as an inoculum) were obtained from the two full-scale plants, which presented the actual operation of the full-scale digesters. Both digesters were showing a stable and steady performance before sampling. The digestates were collected from the reactor through a digestate outflow pipe. The feedstocks were collected after the hygienization/sterilization prior to digesters presenting the reactor feedstock used in the full-scale plants. During the 7 months of CSTR (continuously stirred tank reactor) studies, the CF feedstock was obtained in seven and SS feedstock in eight batches (Table 2). Sample batches were stored at 4 °C (up to 2-4 weeks) prior to analyses and feeding to reactors. The biochemical methane potential (BMP) assays were executed around 2 months prior to the CSTR experiments (Table 2).

2.2 BMP assays

BMP assays were performed at similar temperatures to those at which the representative full-scale digesters operated (40 ± 1 °C with C, 53 ± 1 °C with SS) using automated testing equipment (Bioprocess Control Ltd, Sweden). Assays were conducted in triplicate, each with an inoculum volume of 260 g. The substrate to inoculum ratio (S/I) was 1 in VS basis and distilled water was added to achieve a total liquid volume of 400 ml. $NaHCO_3$ (3 g/l) was used as a buffer. Carbon dioxide was absorbed by NaOH before the automated gas volume measurement, which was based on liquid displacement. The assays were mechanically mixed (84 rpm) for one minute per hour. Assays with inoculum alone presented the residual methane potential (RMP) of the digestate. The results are given as average values of the triplicate assays.

In assays with CF feedstock, methane production was low during the first 20 days of the assays. Subsequently, on day 24 two replicates were diluted to evaluate the potential inhibition/overloading of the batch assays. Dilution was done by mixing the content of two bottles (400 ml + 400 ml), after which 200 ml of this mixture was added in both bottles along with 200 ml of deionized water to achieve liquid volume of 400 ml in both bottles.

2.3 Reactor experiment

Two 11-litre stainless steel CSTRs (Metener Ltd, Finland) were operated at the temperature of the representative full-scale digesters, one at 41 °C (CF) and the second at 52 °C (SS). The reactors were fed manually five times a week (once or twice a day depending on daily feed volume) through an inlet tube which extended below the digestate surface, and which was also used for digestate sampling. Digestate overflowed from the reactors by gravity through a u-tube trap so as to prevent gas escape. Stirring (32 rpm) was semi-continuous with 5 seconds on and 60 seconds off (from day 33 onwards 5 seconds on and 30 seconds off in SS). Biogas volume was measured by water displacement in a volume-calibrated cylindrical gas collector (Ritter TG05/5), after which the gas was collected in aluminum gas bags.

The two laboratory CSTRs were inoculated with 11 liters of digestate from the full-scale digesters, CF and SS. Subsequently, the reactors were kept unfed for five days, after which feeding was started with feedstocks CF and SS (Table 3). The initial HRT with both reactors was higher than the original HRT in the full-scale reactors during days 5–14 for the acclimation of the processes. From day 15 onwards, the HRT corresponded with the full-scale digesters (Table 3). During days 92–211 the HRTs in the CF reactor were gradually reduced from 25 to 14 days, which increased the OLRs from 2 to 3.5 $kgVS/m^3d$. Simultaneously in the SS reactor, the HRTs were reduced from 20 to 15 d during days 54–189 (OLRs from 4 to 6 $kgVS/m^3d$). Reactors were initially fed once a day (five times a week) while with the increased feedstock amount (from days 160 and 61 onwards with CF and SS) the feeding was done twice a day (morning/evening). Due to the differences between the different feedstock batches in CF feedstock (Table 2), the HRT and OLR in CF and SS reactors do not correlate (Table 3).

Samples from the reactors were taken every week for analysis of NH_4-N, soluble chemical oxygen demand (sCOD) and volatile fatty acids (VFAs). Every two weeks, total and volatile solids (TS, VS) and total Kjeldahl nitrogen (TKN) were also analyzed in addition to NH_4-N, SCOD and VFA. Digestate pH was measured five times a week.

2.4 Chemical analyses

pH was determined using a VWR pH100 pH-analyzer (VWR International). TS and VS were analyzed according to SFS 3008 (Finnish Standard Association, 1990). TKN was analyzed by a standard method (AOAC, 1990) using a Foss Kjeltec 2400 Analyzer Unit (Foss Tecator AB, Sweden), with Cu as a catalyst and NH_4-N determined according to (McCullough, 1967). For analysis of sCOD, samples were pre-treated as described in Tampio et al. (2014), and analyzed according to SFS 5504 (Finnish Standard Association, 2002). VFAs (volatile fatty acids: acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric and caproic acids) were analyzed using a HP 6890 gas chromatograph, as described in Tampio et al. (2014). Biogas composition (methane CH_4, carbon dioxide CO_2) was analyzed using a portable Combimass GA-m gas analyzer (Binder Engineering GmbH, Germany).

2.5 Calculations

The reactors were fed for 5 days a week, but the OLR (kgVS/m^3day) is expressed as the average daily weight of substrate fed to the reactor over a one-week period. HRT was calculated based on feedstock densities. Methane yields in BMP assays were converted to STP conditions (0 °C, 100 kPa) according to the ideal gas law. Methane yields in the BMP and RMP assays were calculated by dividing the cumulative methane production by the VS of the added feedstock/inoculum. With BMP assays, methane production of the inoculum (RMP) was subtracted so as to achieve BMP of the feedstock. The standard deviations for BMP and RMP samples were calculated from the variances of the inoculum and feedstock bottles, where the feedstock variance was achieved by subtracting the variance of the inoculum.

3 Results and discussion

3.1 Feedstock and digestate characteristics

The characterization of the CF and SS feedstock showed variation in TS and other parameters, and in particular the composition of the CF feedstock varied due to the temporal and seasonal changes (Table 2, Lee et al., 2016; McLeod et al.; 2015; Regueiro et al., 2015). TS contents were 9–12% in both feedstocks, which is suitable for wet-type anaerobic digestion processes. The VS/TS ratio was low (50–56%) in the CF feedstock, which is most likely due to the characteristics of the industrial by-products in the feedstock mixture. In SS feedstock, the VS/TS ratio was higher, around 70%. Both feedstocks contained relatively high amounts of total nitrogen, 7–8 gN/kg in CF and 5–6 gN/kg in SS, while also the initial NH_4-N in feedstocks was high, around 4.3 gNH_4-N/kg, in CF feedstock (Table 2). The nitrogen content of the feedstock were due to the feedstock characteristics, where nitrogen concentrations of 3–9 gN/kg are generally reported for animal manures (Moset et al., 2015b; Regueiro et al., 2015; Zhang et al., 2014), 7–8 gN/kg for food wastes (Banks et al., 2012; Haider et al., 2015; Tampio et al., 2014), 2–5 gN/kg for sewage sludge (Leite et al., 2016; Lloret et al., 2013) and up to 14–16 gN/kg in enzyme industry by-products (unpublished result).

The digestates from the full-scale digesters, which were used as inocula in both batch and continuous experiments, had TS content of 7–9%, of which the VS content was 30%TS in CF and 45%TS in SS digestate (Table 2). Nitrogen concentrations were high (around 8 gN/kg in CF, 5.6 gN/kg in SS) as was expected based on the characteristics of the feedstocks, and 70% of the total nitrogen was in ammonium form in CF digestate and around 50% in SS digestate. The differences between ammonification in the full-scale digesters are mainly due to the amount and availability of nitrogen-containing molecules, e.g. proteins within the feedstock, where SS feedstock had already gone through one microbial process during the wastewater treatment. The relatively high NH_4-N concentrations, especially in CF digestate (5.5–6.5 gNH_4-N/kg), could potentially be inhibitive to the digester microbes (Rajagopal et al., 2013). Previously NH_4-N concentrations of 2–5 gNH_4-N/kg have been reported in full-scale plants mono- or co-digesting either manures, food wastes and different by-products (Fotidis et al., 2014; Lee et al., 2016; Menardo et al., 2011; Moset et al., 2015b; Sundberg et al., 2013). For SS digestates, lower NH_4-N concentrations have been reported (0.5–1.5 gNH4-N/kg, Hao et al., 2016; Sundberg et al., 2013), which are also lower than what was obtained in the present SS digestate (3.0 gNH_4-N /kg, Table 2) due to higher TS content of the feedstock (TS 12% in the present study, TS 4% in Hao et al., 2016).

With both digestates studied, the RMP values were similar, around 0.05 m^3/kgVS (Table 2), indicating efficient digestion within the full-scale plants. The values obtained were within the range of RMP values reported in the literature for full-scale digesters treating manure and different by-products (0.003–0.03 m^3/kgVS, Menardo et al., 2011; 0.13–0.17 m^3/kgVS, Moset et al., 2015b), where the RMP values are highly dependent on the reactor performance and operation, e.g. OLR (Menardo et al., 2011).

3.2 BMP assays

BMPs of around 0.20 m^3/kgVS were achieved with CF and SS feedstocks during the 100- and 64-day assays, respectively (Figure 1). Overall, the BMP potentials with SS were on the same level, as has been previously reported with sewage sludge (dewatered/thickened sewage sludge 0.17–0.20 m^3/kgVS in Abelleira-Pereira et al., 2015; Zhang et al., 2014). However, with the CF sample, the achieved BMP was 40% lower compared to batch studies with for example pig manure alone (0.32–0.36 m^3/kgVS in Kafle and Chen, 2016; Zhang et al., 2014) and over 50% lower than BMPs from digestion of food wastes (0.40–0.50 m^3/kgVS, Haider et al., 2015; Kawai et al., 2014).

With CF feedstock, the BMP assays showed low and delayed methane production during the first 20 days of the experiment and thus, the assay bottles were diluted in order to reduce the organic matter and nitrogen content within the assays. As a result, the diluted assays produced more than double the amount of methane (0.50 m^3/kgVS) than the undiluted assays (0.20 m^3/kgVS, Figure 1). The delayed methane production with the CF sample during the first 20 days of the experiment was observed with a long lag phase and low methane production, which were most likely due to the organic overloading of the assays, as the S/I ratio, in VS basis, was 1. Also, previous batch experiments with materials from the same full-scale digester had shown similar long lag-phase and delayed process start-up (unpublished results). However, the representative full-scale digester from which the CF digestate and feedstock were obtained showed stable process performance and gas production.

The overloading of the organic matter due to the too high S/I ratio induced the accumulation of VFAs, which caused acidification of the assays and reduced methane production, as the methane-converting micro-organisms were inhibited (Regueiro et al., 2015). However, the acidification phenomenon within the CF assays was observed to be reversible (Kawai et al., 2014), as the non-diluted assay was able to recover and produce methane after day 40, although with lower quantities compared to the diluted assay. It is apparent that within the diluted assay bottles the organic matter and VFA content decreased and pH stabilized, which enabled the recovery of the micro-organisms and improved methane production. In the literature, batch experiments are suggested to be executed with S/I ratios lower than 1 (Haider et al., 2015; Kawai et al., 2014; Moset et al., 2015a), where the higher amount of inoculum adds more active micro-organisms, e.g. methanogens, to the digestion process (Haider et al., 2015), which reduces the lag phase and improves degradation (Boulanger et al., 2012). However, too low ratios are not necessarily effective, as the activity of methanogens is increased to a certain point, after which other parameters, e.g. hydrolysis, becomes the rate limiting step (Boulanger et al., 2012). Additionally, too high inoculum amounts possibly affect the uncertainty of the results as the methane production of the inoculum increases (Angelidaki and Sanders, 2004). Overall, the effect of the S/I ratio is dependent on the substrate and inoculum type (Moset et al., 2015a), which explains the present differences between the BMP assays with CF and SS feedstocks, where e.g. no lag phase was observed with SS feedstock (Figure 1). For example, with sewage sludges an increase of S/I ratio to 2 has been reported to increase the cumulative methane production compared to S/I ratios of 1 and 0.5 as the amount of biodegradable substrate is increased (Braguglia et al., 2006).

3.3 Continuous experiments

Stable operation with increasing OLRs and decreasing HRTs was possible in both laboratory reactors during the around 200 days of experiments. At the beginning of the experiments, between days 15 to 92 in CF reactor and 15 to 54 in SS reactor, laboratory digesters were operated with similar HRTs to the representative full-scale digesters from which the inocula and feedstocks were obtained. Overall, methane yields were on the same level throughout the study as well as other parameters, which were slightly affected by the changes within the feedstocks (Figures 1 and 2), which is normal for full-scale digesters due to the temporal and seasonal variation of the feedstock composition (Lee et al., 2016; McLeod et al., 2015; Regueiro et al., 2015). This was seen with e.g. fluctuating TKN and NH_4-N concentrations in the CF reactor (Figure 2).

Methane yields of around 0.50–0.60 m^3/kgVS were achieved with the CF reactor, while SS reactor produced around 0.28–0.30 m^3/kgVS of methane (Table 4). Similar, though slightly lower, results were also obtained from the BMP assays, where diluted CF assays produced methane 0.50 m^3/kgVS and SS assays 0.20 m^3/kgVS (Figure 1). The differences between methane yields between BMP assays and continuous experiment may be due to the high S/I ratio applied in BMP assays. The lowest HRTs studied (14 d in CF, 15 d in SS reactor) showed higher methane yields compared to the initial HRT from the representative full-scale digesters (HRTs 25 d in CF, 20 d in SS). With both reactors, the increasing OLR and reducing HRT increased the methane yields; where the optimum HRT was around 10–15% lower than the initial HRT. In addition to the methane yields, the VS removal was also increased with the decreasing HRTs in both reactors; thus, the effect on VS removal was not linear (Table 4).

Although the CF reactor showed relatively stable methane yields during the experiment, the VFA and sCOD analyses showed that the digestion process was not stable during the first stage of the experiment (HRT 25 d, Figure 2), which was anticipated based on the long lag phase observed during the BMP assays. Shortly after the start of the feeding of the reactor, the VFA and sCOD concentrations started to increase and the VFAs reached a concentration of 12 g/kg, of which 63% was propionic, 22% acetic and around 10% iso-valeric acid. However, after the peak value, VFA concentrations started to decrease. Slight accumulations of VFAs (up to 5.8 g/kg) were detected after the HRT was reduced to 18 days in the SS reactor; thus, the acclimation of the microbial population to the increased loading was successful, and no further VFA peaks were discovered (Figure 3). During days 66–81 both reactors were fed with lower OLRs, which also affected the decrease and stabilization of the VFA concentrations to a level of <1.5 g/kg in CF and around 2.0 g/kg in SS reactor (Figures 2 and 3). The CF digestate obtained from the representative full-scale digester thus had a higher initial VFA concentration (2.9 g/kg) than the SS digestate (0.2 g/kg, Table 2), which indicated more stable process performance in the full-scale SS digester. Overall, also the laboratory SS reactor showed a more balanced VFA and sCOD performance throughout the experiment compared to CF (Figures 2 and 3).

With the CF digester, the process stability was affected at the beginning of the CSTR experiments, as the VFA concentrations increased (Figure 2). The increasing VFAs are usually due to overloading of organic material into the reactors, as the methanogens are not able to degrade the formed VFAs (Regueiro et al., 2015), or due to inhibition, which causes imbalances within microbial functions and increase of VFAs (Rajagopal et al., 2013). At the beginning of the experiments, the OLR was relatively low and HRT high, which reduces the risk for the VFA accumulation due to organic matter overloading. However, the NH_4-N concentration within the CF reactor was high (4–5 g/kg) throughout the study, which was due to the high TKN concentration of the feedstock (7 g/kg) consisting mainly of industrial by-products and pig slurry. The NH_4-N induces the inhibition of the methane-forming micro-organisms in high concentrations, of which concentrations around 1.5–2.5 g/kg are proposed to be inhibitive for un-acclimated and around 3–6 g/kg for acclimated inocula (reviewed in Rajagopal et al., 2013). According to these literature values, the NH_4-N concentration within CF reactor could potentially inhibit the digestion process. Thus, the inoculum used was already acclimated to high NH_4-N concentrations, as the full-scale reactor was already successfully fed with the same feedstock and the same OLR. Within the representative full-scale digester, no inhibition or accumulation of VFAs was observed, while the removal of the digestate from the reactor and inoculation and start-up of the laboratory reactors showed imbalanced digestion.

The reason for VFA accumulation in the CF digester could be related partly to the NH_4-N as well as the temperature range applied (41 °C), which possibly affected the function of the micro-organisms. Ammonium nitrogen has an effect on microbial consortia (Fotidis et al., 2014; Lee et al., 2016), and it is known that the hydrogenotrophic methane formation route is active when ammonia is high, while acetoclastic methanogens are inhibited by NH_4-N (Banks et al., 2012; Fotidis et al., 2014). Additionally, the digester temperature (41 °C) was near to the mesophilic upper range, which is the most vulnerable temperature zone (from 45 to 50 °C, Kim and Lee, 2016), where the microbial consortia are more susceptible against changes in environmental conditions, e.g. temperature shocks (Gao et al., 2011). It is thus suggested that the high NH_4-N concentration and temperature of 41 °C together reduced the microbial diversity already within the full-scale CF digester. As the inoculum was removed from the full-scale reactor and transported to the laboratory-scale reactors, the microbial consortia were affected due to a change in temperature and the microbial consortia were not able to quickly recover from the temperature shock, which caused the rapid VFA accumulation after the start of the CSTR experiments and possibly also affected the batch tests. The inoculum would probably have needed a longer acclimation/start-up period after introduced to the reactor and the mesophilic conditions, which was seen as the VFA concentrations stabilized around day 90 (Figure 2) indicating that the acidification was a reversible process (Kawai et al., 2014). A similar result was also obtained in a laboratory study with wastewater treatment sludge, where the temperature of 42 °C was observed to inhibit the start-up of the digestion process, and where the microbial community structure within the 42 °C reactor was also different compared to a control reactor operating at 37 °C (Beale et al., 2016). In the present study, the VFA accumulation did not correlate with the pH value (Figure 2), which was measured each day, supporting the reversible and temporary nature of the acidification phenomenon. Additionally, the high NH_4-N concentration within the reactor acted as a buffer and stabilized pH (Prochazka et al., 2012).

Another increase of VFAs in the CF reactor was observed during the lowest HRT (14 days) applied, where the increased organic matter and nitrogen loading most likely affected the methanogens and caused acidification. Unfortunately, the experiment was halted, and it is unclear what was the ultimate cause for the VFA imbalances. With the

SS reactor digesting sewage sludge, the NH_4-N concentration was lower (around 3 g/kg) compared to CF reactor (4–5 g/kg). SS feedstock had also more stable composition compared to CF (see Table 2), where the temporal variation within the feedstock composition was more evident, and was observed with e.g. varying TKN concentrations during the CSTR experiment (Figure 2). Overall, the composition of sewage sludge is more cohesive throughout the year, compared to the co-feedstock in CF (industrial waste), where the feedstock composition is affected by the current availability of the organic waste materials (Regueiro et al., 2015).

The present OLRs and HRTs achieved in CF and SS digesters were compared to literature values obtained from pilot and full-scale plants digesting similar feedstocks (mixtures of industrial/food wastes and manure, sewage sludge) (Table 5). The HRTs with industrial waste- and manure-based digestion plants are usually higher, around 25 to 30 days, thus with similar OLRs as in the studied CF reactor (around 3 kgVS/m^3d). However, the co-digestion with various feedstocks as well as varying mesophilic temperature ranges (from 35 to 41 °C) complicates the direct comparison of the reported literature values (Table 5). The thermophilic SS reactor with sewage sludge as feedstock, showed similar HRTs with full-scale applications (10–30 days) and VS removal (40–50%), thus higher OLR (4–6 kgVS/m^3d compared to 1.5–3 kgVS/m^3d within the full-scale digesters, Table 5). Overall, the HRT and OLR are dependent on feedstock characteristics, which affect the operational parameters in each digester. With lower HRTs and higher OLRs, the reactor volume can be reduced and the feedstock treatment rate increased, which increases the economy of the anaerobic digestion plants through increased gate fees without compromising the stability of the digestion process. In the present study, it was shown that the successful increase of the waste digestion rate by around 10–15% is possible also with feedstocks containing high nitrogen concentrations.

4 Conclusions

The reduction of HRT from 20–25 to 14–15 days was possible in laboratory-scale reactors simulating full-scale anaerobic digesters. In terms of methane yield and VS removal, the optimum HRTs were around 17 and 22 days, representing a 10–15% increase in digester efficiency and gate fee income in full-scale applications. The increase of the treatment rate of high nitrogen-containing feedstocks (5–9 g/kg) was possible, enabling more efficient utilization of these types of waste materials, thus necessary acclimation time is needed to prevent VFA accumulation during digester start-up.

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Figure captions

Figure 1. The biochemical methane potentials (BMPs) of the studied feedstocks and the residual methane potentials (RMPs) of the digestates, a) CF, b) SS. The standard deviations are plotted in 5-day intervals, where n=3 for inocula and SS feedstock, n=2 for diluted CF feedstock, and n=1 for the raw CF feedstock. Standard deviation for the diluted CF feedstock is not available from day 24 to 62 due to sample dilution. Note the different x- and y-axis between figures.

Figure 2. The process parameters within the CF reactor operating at 41 °C during the gradually reduced hydraulic retention times (HRTs). a) Methane content, methane yield and organic loading rate (OLR), b) soluble chemical oxygen demand (sCOD), volatile fatty acids (VFAs) and pH, c) total Kjeldahl nitrogen (TKN) and ammonium nitrogen (NH_4-N).

Figure 3. The process parameters within the SS reactor operating at 52 °C during the gradually reduced hydraulic retention times (HRTs). a) Methane content, methane yield and organic loading rate (OLR), b) soluble chemical oxygen demand (sCOD), volatile fatty acids (VFAs) and pH, c) total Kjeldahl nitrogen (TKN) and ammonium nitrogen (NH_4-N).

Table 1. The characteristics of the full-scale anaerobic digestion plants simulated in this study.

Digester	Co-digestion feedstock	Sewage sludge
Abbreviation	CF	SS
Feedstock	By-products from enzyme production (60%), pig slurry (25%), food industry by-products (15%)[a]	Dewatered municipal sewage sludge[b]
Treatment capacity (t/y)	100 000	75 000
Temperature (°C)	41	52
HRT (d)	25	20
OLR (kgVS/m^{3}d)	2.2–2.3	4

[a]Mixed and hygienized 60 min in 70 °C

[b]Diluted to TS 15% and sterilized (thermal hydrolysis <133 °C, <20 min, <3 bars)

Table 2. The characteristics of the digestate (used as an inoculum) and feedstocks used within the biochemical methane potential (BMP) assays and continuously stirred tank reactor (CSTR) experiments. All values presented on a fresh matter basis.

Reactor feedstock	Co-digestion feedstock (CF)				Sewage sludge (SS)			
Sample	*Digestate*		*Feedstock*		*Digestate*		*Feedstock*	
Experiment	BMP	CSTR	BMP	CSTR[a]	BMP	CSTR	BMP	CSTR[a]
pH	n.d.	8.3	n.d.	6.2 ± 0.2	n.d.	8.1	n.d.	6.1 ± 0.2
TS (g/kg)	96.2	76.9	121.5	85.9 ± 13.8	77.0	91.1	121.3	118.0 ± 4.1
VS (g/kg)	33.3	29.0	60.4	47.9 ± 7.8	43.4	46.9	88.8	83.3 ± 3.5
VS/TS (%)	34.6	37.7	49.7	55.8	56.4	51.5	73.2	70.6
TKN (g/kg)	8.8	7.6	8.4	7.0 ± 0.8	5.7	5.6	6.2	5.4 ± 0.2
NH_4-N (g/kg)	6.4	5.5	4.3	n.d.	3.0	3.0	1.1	n.d.
sCOD (g/kg)	n.d.	11.9	n.d.	n.d.	n.d.	10.9	n.d.	n.d.
VFAtot (g/kg)	n.d.	2.9	n.d.	n.d.	n.d.	0.2	n.d.	n.d.
BMP, RMP (m^3_{CH4}/kgVS)	0.048	n.d.	0.202[b]	n.d.	0.047	n.d.	0.191	n.d.

[a]n=6–7

[b]after dilution of assays 0.510 m^3_{CH4}/tVS

n.d., not determined

Table 3. The operation of the laboratory reactors during the experiment with gradually increasing organic loading rate (OLR) and decreasing hydraulic retention time (HRT).

Co-digestion feedstock (CF)			Sewage sludge (SS)		
Days	OLR ($kgVS/m^3d$)	HRT (d)	Days	OLR ($kgVS/m^3d$)	HRT (d)
15–92	1.8–2.1	25	15–54	3.9–4.3	20
93–111	1.5	22	55–125	4.4–4.7	18.3
112–125	1.9	17.1	126–137	4.8	16.6
126–158	2.5	22	138–158	5.0	15.9
159–189	3.0	16.7	159–189	5.5	15.9
190–211	3.5	14.4	190–212	6.0	14.7

Table 4. Methane yields, total and volatile solids (TS, VS) and VS removal during the study with decreasing hydraulic retention times (HRTs) and increasing organic loading rates (OLRs). The table presents average values and standard deviations from the representative HRTs. For days 0–4 the reactors were kept unfed, on days 5–14 the reactors were acclimated with HRT of 50 d (OLR 0.9 kgVS/m^3d) in CF and HRT of 50 d (OLR 0.9 kgVS/m^3d) in SS.

Co-digestion feedstock (CF)					
Days	HRT (d), OLR (kgVS/m^3d)	CH_4 yield (m^3/kgVS)	TS (g/kg)	VS (g/kg)	VS removal (%)
15–92	25 (OLR 1.8–2.1)	0.474 ± 0.109	74.0 ± 16.5	32.1 ± 7.6	40.1 ± 12.4
93–111	22 (OLR 1.5)	0.591 ± 0.027	53.6 ± 0	22.0 ± 0	33.7 ± 0
112–125	17 (OLR 1.9)	0.586 ± 0.015	50.2 ± 0	22.2 ± 0	33.3 ± 0
126–158	22 (OLR 2.5)	0.561 ± 0.008	60.5 ± 5.9	25.4 ± 1.5	53.8 ± 2.7
159–189	17 (OLR 3.0)	0.479 ± 0.007	71.3 ± 3.7	29.3 ± 1.4	41.6 ± 2.8
190–211	14 (OLR 3.5)	0.536 ± 0.010	72.5 ± 0	33.2 ± 0	34.3 ± 0
Sewage sludge (SS)					
Days	HRT (d), OLR (kgVS/m^3d)	CH_4 yield (m^3/kgVS)	TS (g/kg)	VS (g/kg)	VS removal (%)
15–54	20 (OLR 3.9–4.3)	0.280 ± 0.026	87.0 ± 2.6	47.0 ± 1.2	42.5 ± 4.7
55–125	18 (OLR 4.4–4.7)	0.279 ± 0.089	83.8 ± 3.9	45.7 ± 2.0	45.1 ± 2.5
126–137	17 (OLR 4.8)	0.297 ± 0.004	79.0 ± 0	44.3 ± 0	43.8 ± 0
138–158	16 (OLR 5.0)	0.288 ± 0.007	75.4 ± 0	42.5 ± 0	46.0 ± 0
159–189	16 (OLR 5.5)	0.279 ± 0.020	77.0 ± 3.7	44.4 ± 1.6	49.1 ± 1.8
190–212	15 (OLR 6.0)	0.286 ± 0.006	91.8 ± 0	49.8 ± 0	42.8 ± 0

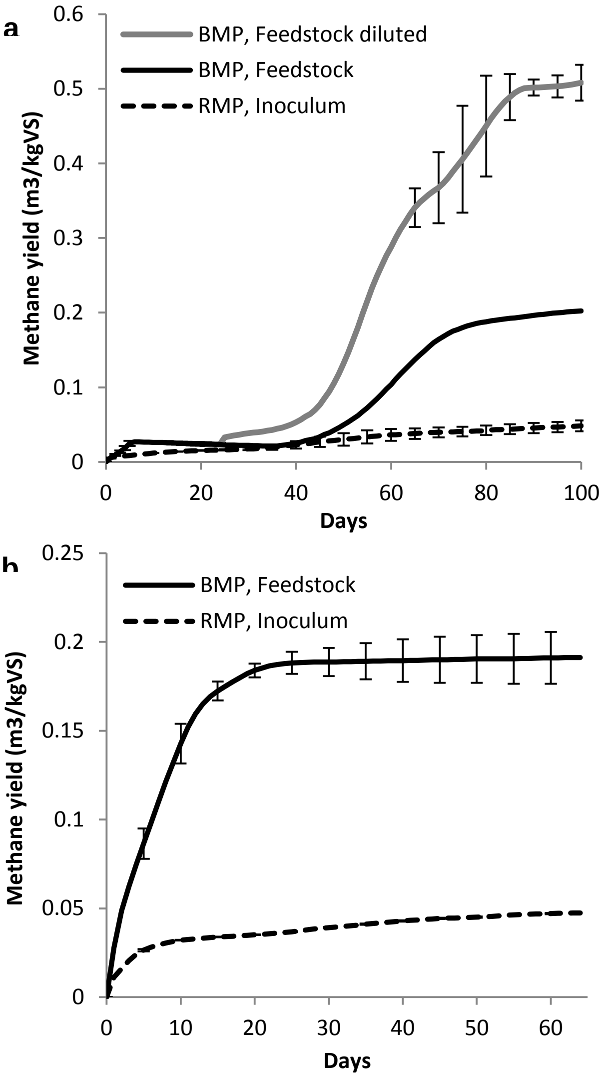
n=2–11 for CH_4 yield, n=1–5 for TS, VS and VS removal

Table 5. The operational parameters; temperature, hydraulic retention times (HRT), organic loading rate (OLR) and VS removal in full- and pilot-scale digesters digesting mixtures of industrial/food wastes and manure, and sewage sludge according to the literature.

	Temper-ature (°C)	OLR ($kgVS/m^3d$)	HRT (d)	VS removal (%)	Reference
Animal manure (70%), energy crops (20%), food industry by-products (10%)	41	2.25	105	-	Menardo et al., 2011
Pig slurry (87%), energy crops (17%)	41	0.85	51	-	Menardo et al., 2011
OFMSW (59%), food industry waste (21%), pig manure (9%)	37	3.2 –3.9	27–34		Sundberg et al., 2013
Pig and cow manure (69%), OFMSW (30%)	38	3.1	29	-	Sundberg et al., 2013
SHW (54%), pig and cow manure (33%), OFMSW (10%)	37	3.1	25	-	Sundberg et al., 2013
Cattle slurry, cattle manure, maize silage	38.5	-	25	-	Alburquerque et al., 2012
SHW (54%), pig and cow manure (33%), OFMSW (10%)	37	3.1	25		Sundberg et al., 2013
Pig manure, SHW sludge, biodiesel wastewater	37	-	21	-	Alburquerque et al., 2012
Cow manure	35	3.1	20	28.2	Moset et al., 2015b
Enzyme industry by-products (60%), pig slurry (25%), food industry by-products (15%)	41	1.5–3.5	14–25	33–54	Present study
Wastewater treatment sludge	52.3–53.9	1.5–2.5	16–28	39.4–46.1	Lloret et al., 2013
	55	2.2	20	34	Leite et al., 2016
	58.5	-	15.5–17.5	-	Lee et al., 2016
	51–53	2.9	11	-	Sundberg et al., 2013
	52	4–6	15–20	43–49	Present study

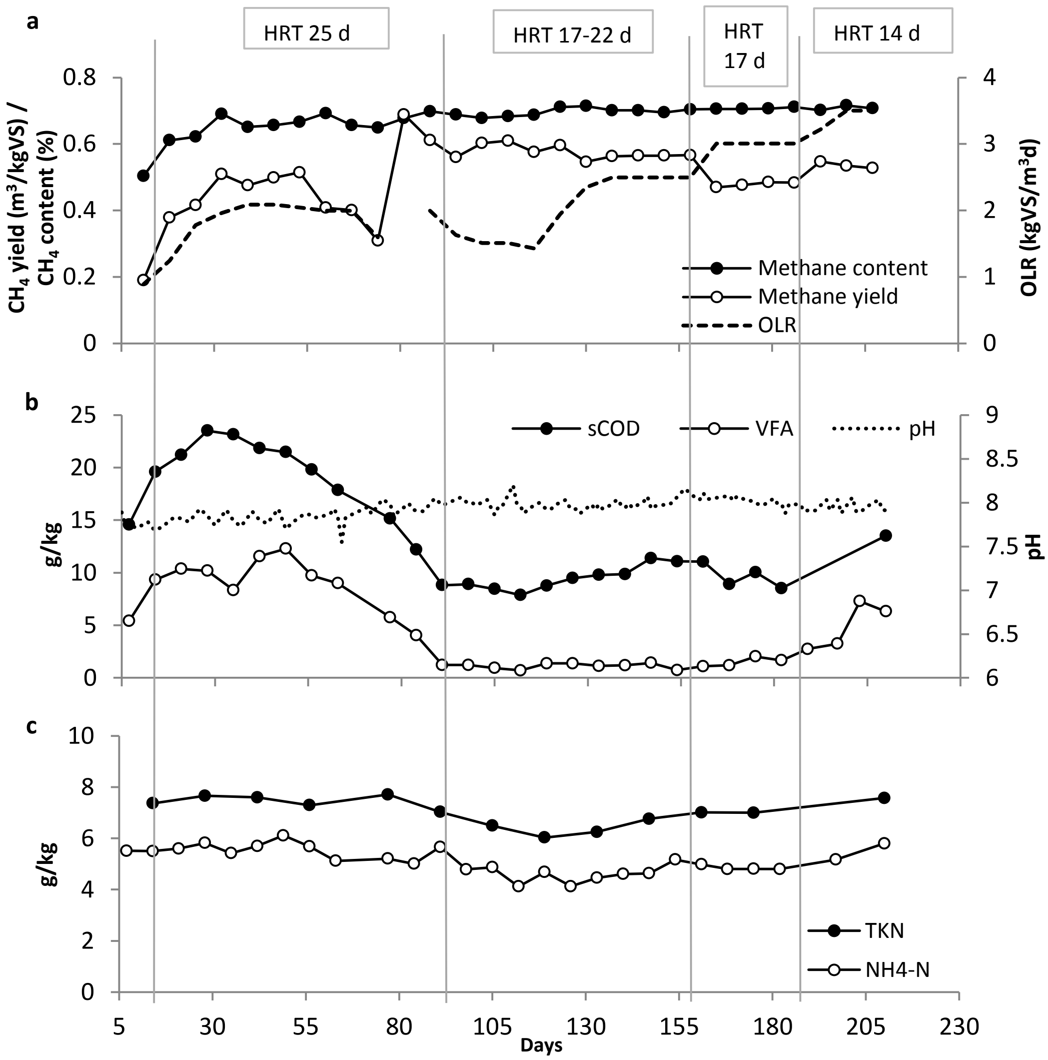
Organic fraction of municipal solid waste (OFMSW), slaughterhouse waste (SHW)
-, not available

Fig. 1.



a
BMP, Feedstock diluted
BMP, Feedstock
RMP, Inoculum
Methane yield (m3/kgVS)
0.6
0.5
0.4
0.3
0.2
0.1
0
0
20
40
60
80
100
Days
b
BMP, Feedstock
RMP, Inoculum
Methane yield (m3/kgVS)
0.25
0.2
0.15
0.1
0.05
0
0
10
20
30
40
50
60
Days

531 Fig. 2.



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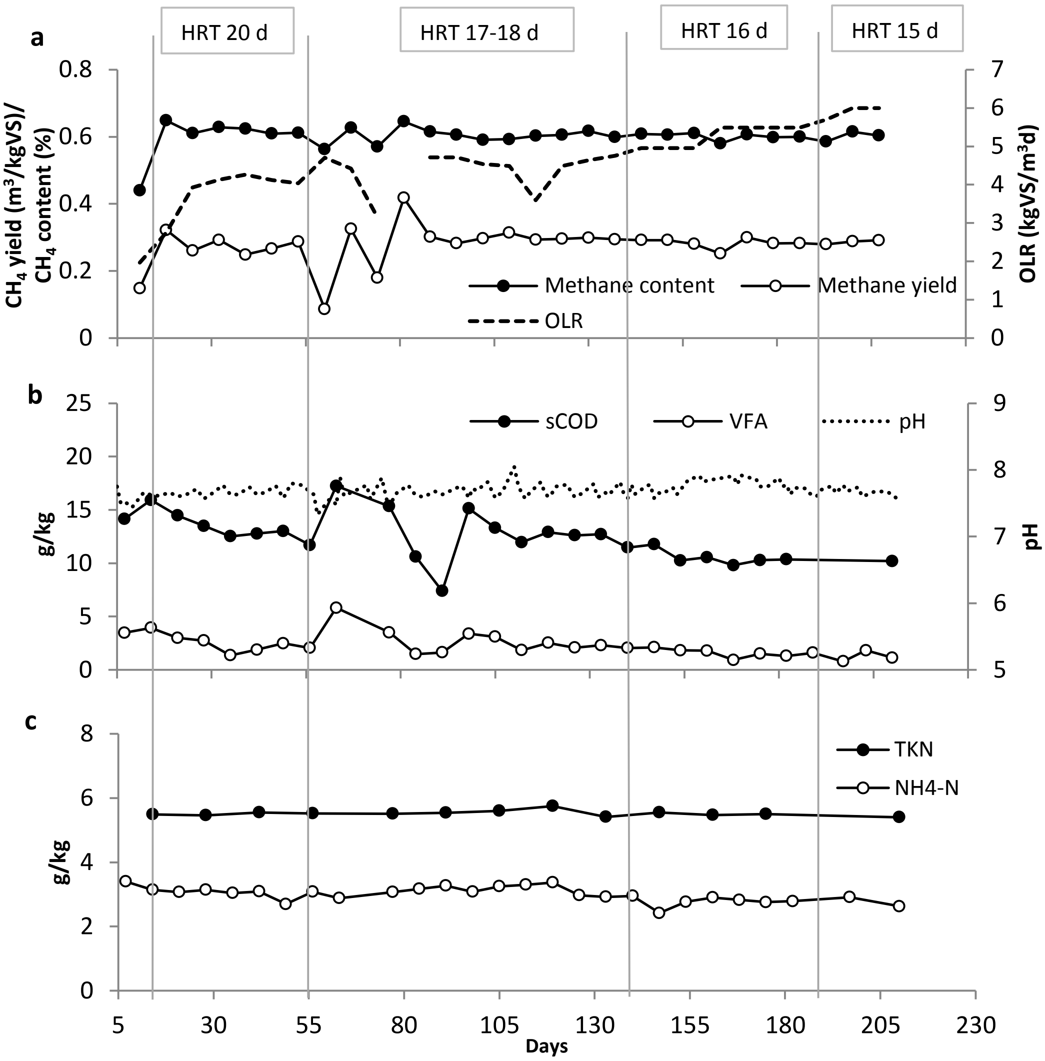
534 Fig. 3.



a
b
c
HRT 20 d
HRT 17-18 d
HRT 16 d
HRT 15 d
CH4 yield (m3/kgVS)/ CH4 content (%)
OLR (kgVS/m3d)
Methane content
Methane yield
OLR
sCOD
VFA
pH
g/kg
TKN
NH4-N
Days

535