Boosting biogas production by pulse injection of waste biomass

Final report of the project:

Flexible CHP from Biogas based on Waste Biomass

Part 2.

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1. Background:

The purpose of this work package has been to carry out test in continuous biogas systems (CSTR) to demonstrate the possibilities and limitations of increasing loads by biomass with high biogas potentials both by pulse loading and increased loading over longer times. The tests have been done in different digester sizes and with different biomasses. The different tests are illustrated in Table 1.

Table. 1: Different biomass and feeding schemes

Biomass	Digester sizes	Pulse feeding	Long term increase in loading
Beet-straw silage (SBT)	15 liter, 10 m^3 and 30 m^3	15 liter	10 m^3 and 30 m^3
Pre-treated straw			10 m^3 and 30 m^3
Pre-treated grass	10 m^3 and 30 m^3	10 m^3 and 30 m^3	-
Maize silage	15 liter, 10 m^3 and 30 m^3	15 l, 10 m^3 and 30 m^3	-

2. Experiments in lab scale digesters

2.1 Experiment 1 with cattle manure and SBT

The main substrate was cattle manure from Foulum AU facilities. For the OLR increase, silage of SBT and straw was used (referred as silage). AgroTech supplied the silage from 2014 in a 500 g vacuum bag. The mixed ratio of SBT and straw was 3:1 and the straw was previously chopped as a pretreatment. Further cutting was necessary until straw length of approximately 5 cm (to avoid feeding problems). The duration of the experiment was one week. Two 10 liters (working volume) CSTR (referred as R1 and R2) were operated under thermophilic conditions (55°C) with cattle manure as the main substrate. The two reactors were operated under identical conditions including the same OLR (4 g VS $liter^{-1}$ day^{-1}, 500 g of cattle manure) and HRT (20 days)..

In order to assess the boosting capacity of a biogas system, a pulse step was applied to a reactor daily fed with cattle manure (R2). SBT and straw silage was used to increase the OLR and as fast degradable substrate. A first reactor (reactor 1, R1) was left as a control, thus no pulse step (silage) was applied. While the OLR of R2 was raised to 6.9 g VS $liter^{-1}$ day^{-1} (150 g of silage plus 350 g of cattle manure). In the remaining days the OLR was set as default (only cattle manure) at the first day of investigated week. Biogas production, biogas composition and pH were online measured and recorded. Furthermore, daily samples and special samples after 0.5, 1, 2, 3, 4 and 5 hours from pulse step in R2 were taken to characterize process parameters (such as VFA and TAN). Biogas production of both reactors increased after each feeding (Figure 1). However, due to the higher OLR, the maximum biogas produced after feeding was higher in R2 than in R1 (0.7 and 0.4 l/h respectively). Furthermore, it can be observed that the increase occurred faster in R2 (approx. 1 hour before). The reason of this behavior is the difference in degradation rates between SBT (found in silage) and cattle manure. On one hand, SBT have high amounts of non-fiber carbohydrates (NFC) and a low contents of neutral detergent fiber (NDF). On the other hand, cattle manure has low contents of NFC and higher amounts of NDF and crude proteins. The fast degradation of the high amount of easily degradable fraction in SBT found in the silage, also produced a faster decrease once the maximum is reached in R2 (approx. 2 hours after feeding). In addition, the biogas production during the following days was still higher. Straw, a slower degradable substrate than SBT, remained in the system thus increasing biogas production as it was degraded. An important fact was observed after the pulse feedings with only cattle manure. The difference between maximum productions after feeding seems to differ more than the hourly production during steady state after feeding. The difference between maximums biogas production (after feeding) was 0.33, 0.16 and 0.28 l/h for the second, third and fourth day respectively. However, the difference between averages of biogas produced by hour was 0.06, 0.08 and 0.09 l/h respectively. This behavior could be explained if the concentration of H_2 was higher in reactor 2, leading to an increase in biogas once new substrate was added (favoring hydrogenotrophic pathway).

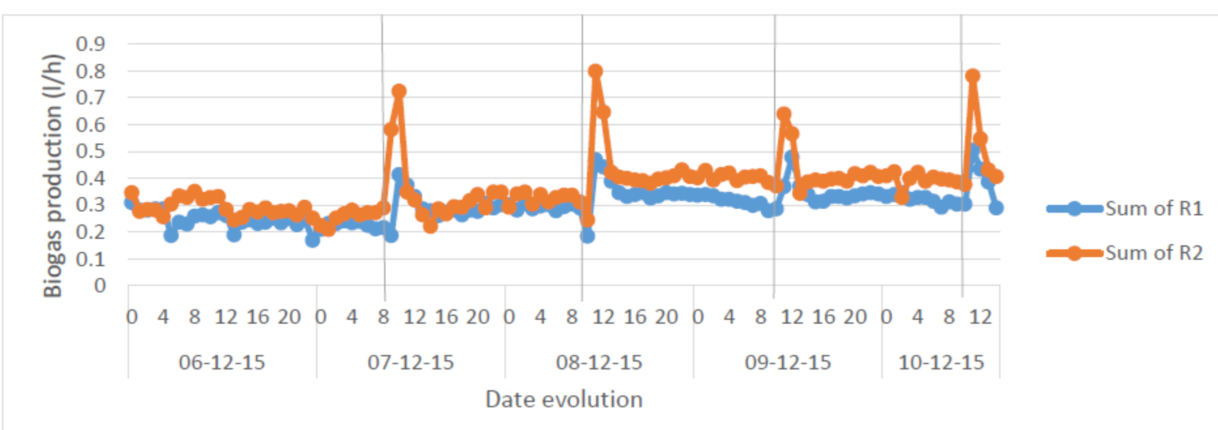


Figure 1. Biogas production. Lines show where the four feedings were applied.

Daily productions are shown in Figure 2. As can be observed, a difference already existed previous to the pulse step. This behavior was due to tests carried out before the experiment. However, the difference between R1 and R2 increased since the overload. The maximum daily production (10 l/d) was observed in the third day. As seen previously, the maximum peak on that day was the smallest one but the daily average was the highest. Finally, biogas production decreases on the fourth day. However, the difference between reactors remains. Further measurements were taken, daily biogas production stabilized and became similar after 10 days of the test.

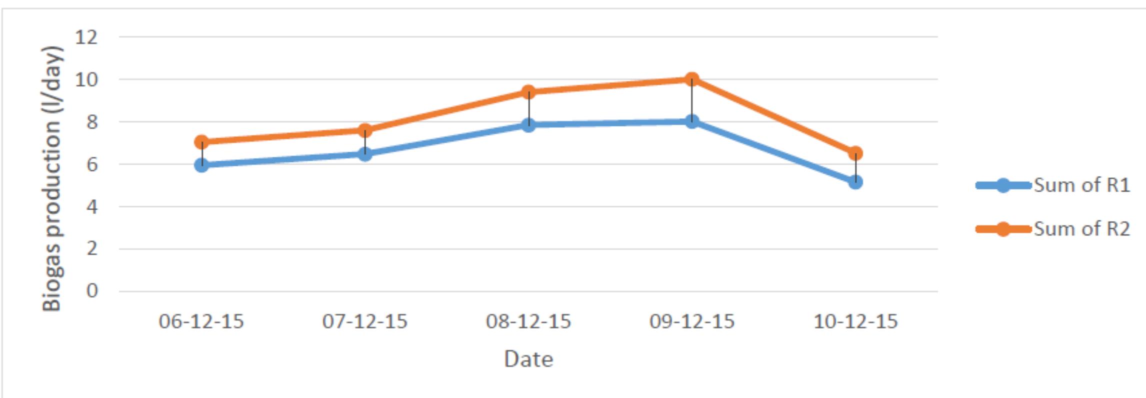


Figure 2. Biogas daily production.

Methane concentration is showed in Figure 3. Its concentration increased after the first feeding. In this case, the change of OLR did not make any difference between reactors. However, during the following days, R2 showed a faster decrease in CH_4 concentration than R1. Almost the same value (48.10% and 48.39% respectively) was hit for each reactor with a day of difference. The faster CH_4 decrease (thus higher CO_2 concentration) in R_2 could be due to a slightly accumulation of CO_2 as a product of fermentation. As a result of a higher OLR, a higher CO2 could be produced. However, in full scale plants, biogas is collected in the storage over several hours. Thus different qualities are mixed, leading to lower variations in the quality of the gas as it reaches the co- generation heat and power (CHP) (Mauky et al. 2015). Thus variations as observed, could be neutralized.

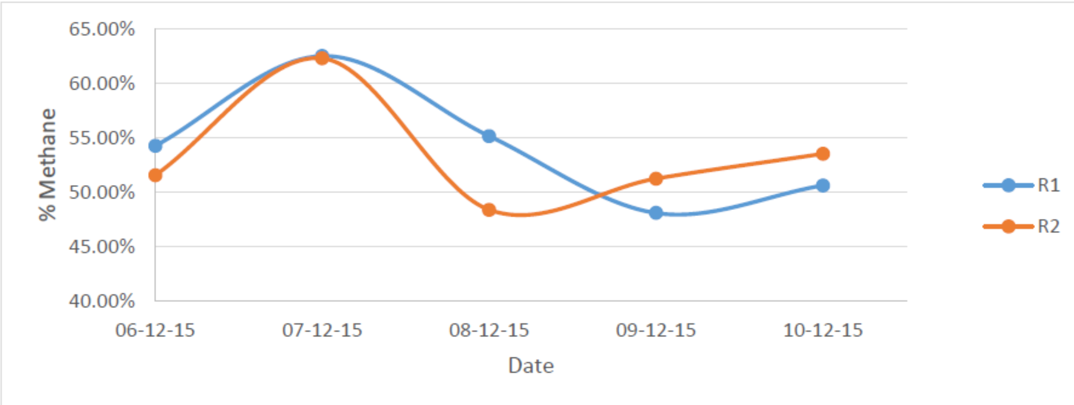


Figure 3. Methane concentration

Results shows that flexible biogas production is possible using a fast degradable substrate as a boost. Results showed that SBT and straw silage has a fast effect on biogas production. However, the higher biogas yield is observed during the following days rather than immediately after pulse step. Thus in this case, the flexibility is observed in a week scale rather than in a day scale. However, results of VFA, pH and TAN showed that there is still range of action. Higher OLR could be tested in order to stress the system and identify optimal process parameters. As stated previously, even though biogas composition showed variations, the impact could be neutralized by storage previous to CHP. To obtain more reliable results and stronger patterns, more frequent analysis should be taken (from both reactors) and for a longer period of time. Furthermore, special analysis such as substrate composition could make the picture clearer.

2.2 Experiment 2 with cattle manure, maize and SBT

15 liters CSTRs were used for the experimental work. Both reactors run on thermophilic conditions. The working volume was 10 liters and the hydraulic retention time (HRT) was 20 days. Prior to the experiment, the reactors were filled with screened inoculum from the main reactor at Foulum biogas plant controlled under stable configuration. The feeding was applied weekly thus no feeding was applied during weekends.

During phase 1, only sugar beet tops and straw silage (SBTSs) was tested as a boost substrate. The two reactors worked in parallel during two weeks (Figure 4). One reactor (R-CM), was used as a reference thus fed regularly with cattle manure. The other (R-SBTSs), was used as the boost feeding reactor. Boost feeding was given accompany with the normal feeding of cattle manure, which fed once at the start of the week.

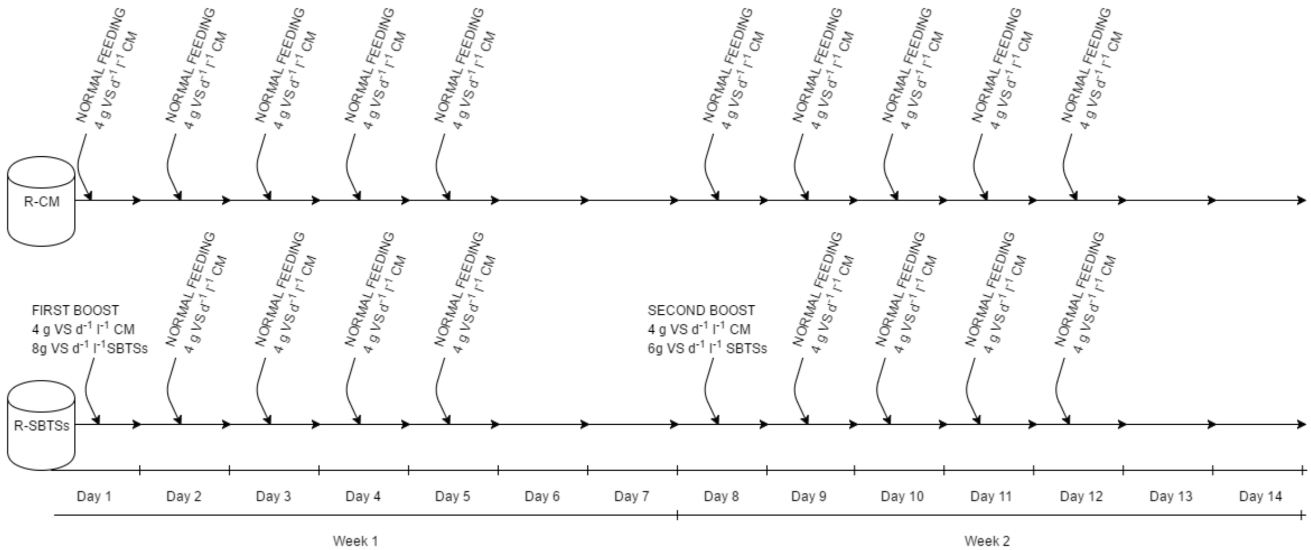


Figure 4. Experiment 1 setup.

Two different OLR were tested, 8 g VS l^{-1} d^{-1} and 6 g VS l^{-1} d^{-1} were applied during the first and second week, respectively. It has to be pointed that the boost was fed in addition of the regular cattle manure feeding, thus the total OLR was 12 and 10 g VS l^{-1} d^{-1}, respectively.

Due to inhibition in phase 1, both reactors were emptied and filled again with new inoculum from Foulum main reactor. The conditions were the same; 10 liters of working volume, thermophilic temperature and HRT of 20 days. Phase 2 was performed during one month. However, the experiment was divided in two periods of two weeks (Figure 4). During the first period, both reactors were used to test the boost effect in addition of normal cattle manure feedings. One reactor (R-Ms), was used to test maize silage (Ms) whereas the other (R-SBTSs) tested sugar beet tops and straw silage (SBTSs). On the other hand, during the second period, both reactors were used as a reference. One reactor (R-In), worked only with inoculum (no feeding was applied) whereas the other (R-CM) worked with cattle manure (CM) feedings. The inoculum data was used to subtract the effect of the inoculum during the whole experimental time.

The new inoculum was added in the first day of period one and replaced in the first day of period two. Thus new inoculum was used in each period. In order to maintain similar conditions with Foulum main reactor, no previous screening was applied. In this case, steady state conditions were not reached before the boost. As in experiment 1, the regular feeding were only applied during weekdays, no feeding was applied on weekends. The boost day was on Mondays.

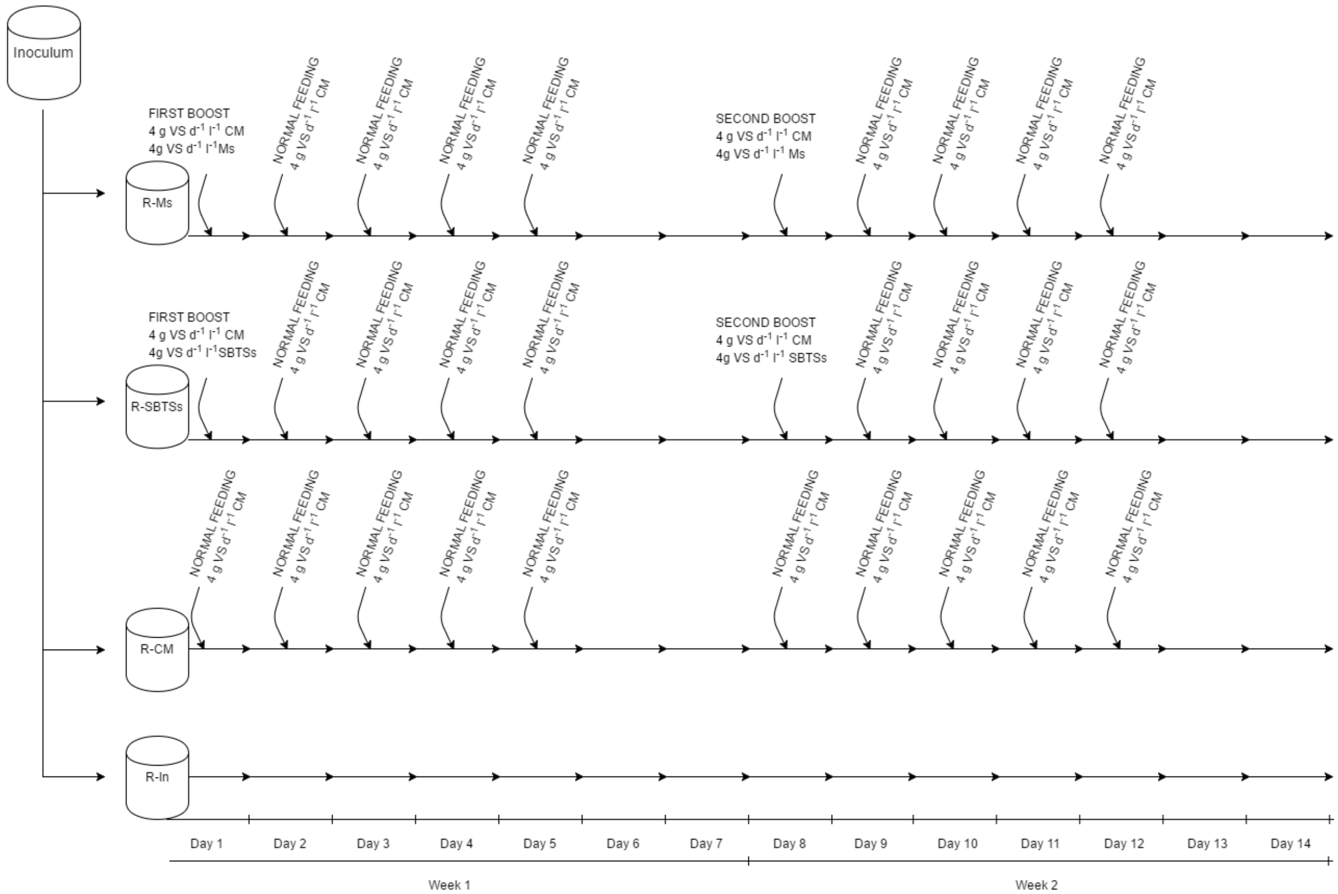


Figure 5. Experiment 2 setup. NOTE: R-Ms and R-SBTSs were run separate in time respect R-CM and R-In. In the scheme they are shown together to facilitate the understanding.

Phase 1

Experiment 1 (exp 1) was performed during two weeks. Two CSTRs were run in parallel in thermophilic condition and with a retention time (HRT) of 20 days. One reactor was daily fed with cattle manure and used as a reference (R-CM). The other, in addition of cattle manure was fed with sugar beet tops and straw silage (R-SBTSs) with higher OLR as boost substrate (Figure 5). Two different boosts were applied. During the first week, a boost of 8 g VS l^{-1} d^{-1} of SBTSs was tested. Due to a sever inhibition, a lower second boost (6 g VS l^{-1} d^{-1}) was applied during the second week.

Regular cattle manure feedings (4 g VS l^{-1} d^{-1}) were applied on week days, thus no feedings on weekends. The boost feed was applied on the first day of each week, to observe the whole week's performance. The hourly biogas production is shown in Figure 6. R-CM showed the expected behavior; biogas production increased during the week and decreased during the weekend. Furthermore, similar peaks were obtained after each feeding.

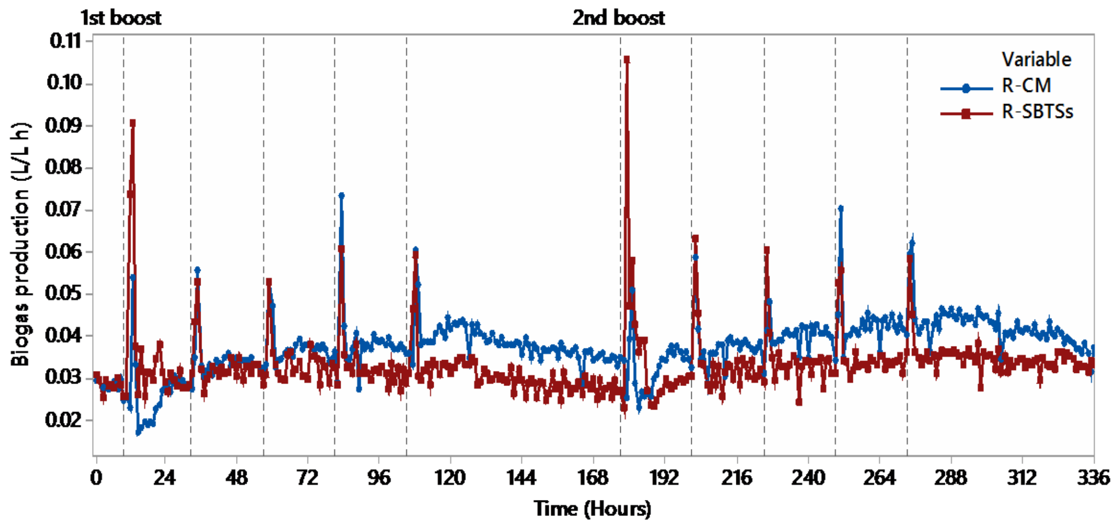


Figure 6. Phase 1. Online monitoring of the hourly biogas production. Dashed lines represent each feeding event.

On the other hand, R-SBTSs showed generally lower biogas yields than R-CM. However, higher peaks were observed after the first and second boost compared with regular feedings. Both boost effects were observed an hour after the feeding. The first, reached its maximum after 3 hours (0.09 L L^{-1} h^{-1}) whereas the second (0.11 L L^{-1} h^{-1}) after 2 hours. In addition, the second boost showed longer boost effect (4 hours and 8 hours respectively). It is interesting to state that even though the second boost had a lower OLR than the first (8 and 6 g VS L^{-1} d^{-1} respectively), the latter presented a higher effect. That could be caused by a pro-long accumulative effect of the first boost.

Daily biogas yields are shown in Figure 7. Only on boost days R-SBTSs presented higher yields than R-CM. As stated previously, R-CM showed the expected behavior but R-SBTSs did not. After both boosts, daily biogas yields decreased. Higher yields were observed during the second week. The total biogas production was 5.98 and 5.40 L L^{-1} during the first week and 6.65 and 5.72 during the second, for R-CM and R-SBTSs respectively.

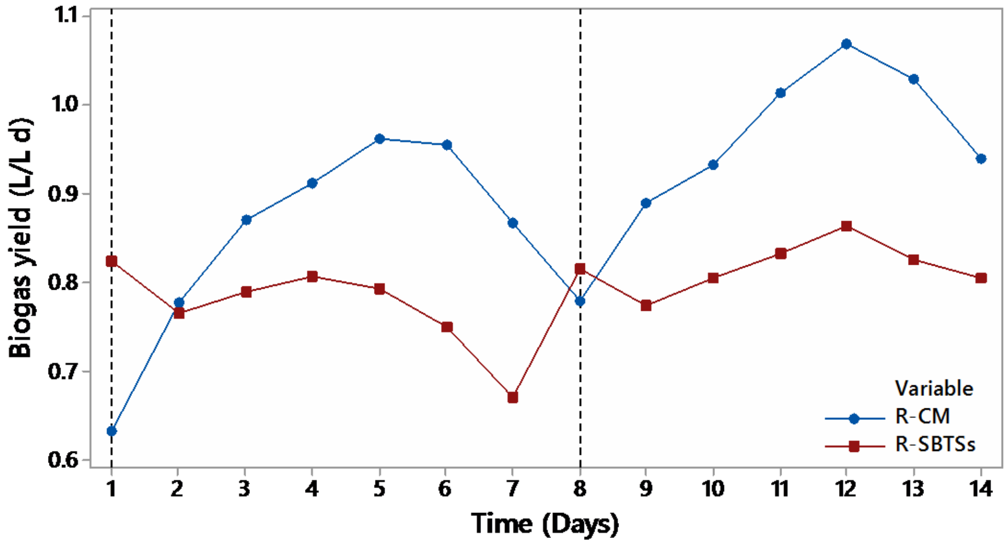


Figure 7. Phase 1. Daily biogas yields. Dashed lines represent boost feedings.

Differences between R-CM and R-SBTSs ranged between 0.2 and -0.2 l L^{-1} d^{-1} (Figure 8). During the whole experimental period, only on boost days a gas surplus was obtained. An overload could be the reason, thus the OLR was three times higher during the first boost and two times higher during the second.

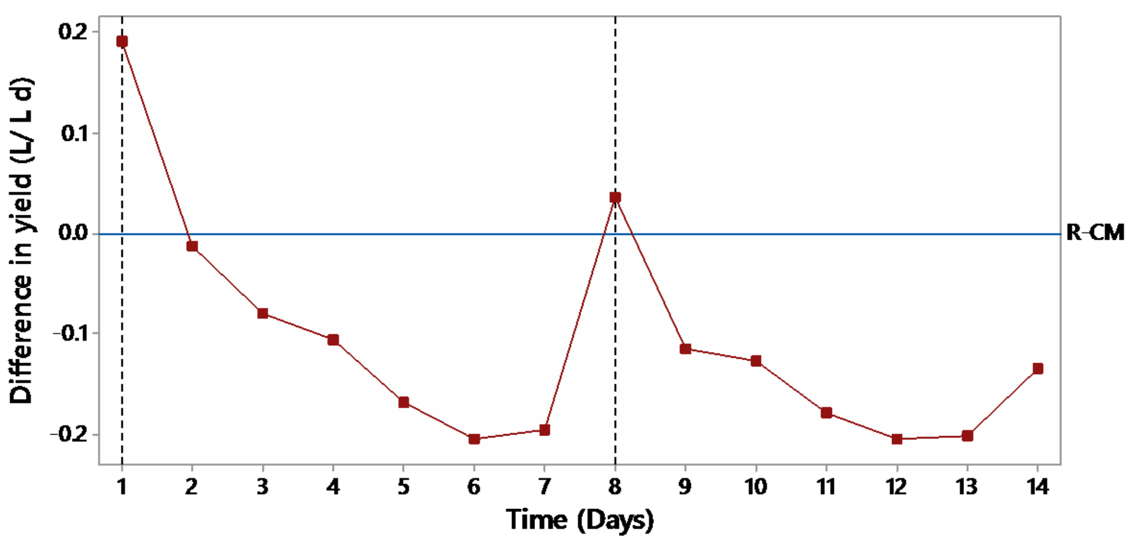


Figure 8. Phase 1. Difference in daily biogas yields between R-SBTSs and R-CM. Dashed lines represent boost days.

Methane content is shown in Figure 9. Different behaviors were identified. In R-CM similar patterns were observed in both weeks. Maximum CH_4 concentrations during the first days (59 and 62% respectively) and minimums during the last week days (52 and 53%). The decreasing tendency during the week was due to small reductions after each feeding. With the addition of new substrate, the concentration of CO_2 increases as a product of acetogenesis. Meanwhile this CO_2 generated is not used by methanogens to transform it to CH_4, remains accumulate in the reactors thus diluting the concentration of CH_4.

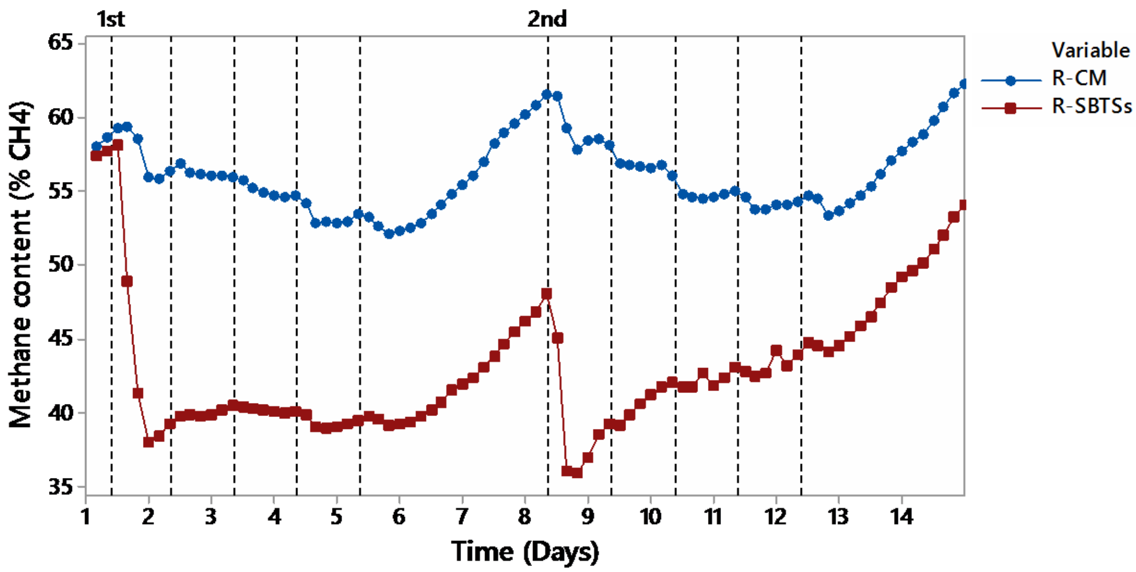


Figure 9. Phase 1. Online monitoring of the methane content during both weeks. Dashed lines represent each fed event. 1st and 2nd refer to the first and second boost.

On the other hand, biogas in R-SBTSs showed a big CH_4 drop after each boost. During the first day, a reduction of 20 points (equivalent to the 35%) was observed. After rising on weekend, another drop was observed with a reduction of 12 points (25%) after the second boost. The suitable pH range for anaerobic digestion is about 6.8 and 7.2. However, due to VFA accumulation a pH drop can occur. The pH change is shown in Figure 10. R-CM presented similar behaviors in both weeks; after a slightly increase on the first day, pH decreased during the whole week. However, during the weekend the pH lost was recovered. Even though, disturbances after feeding were observed, pH variations ranged about 0.10- 0.20 pH points.

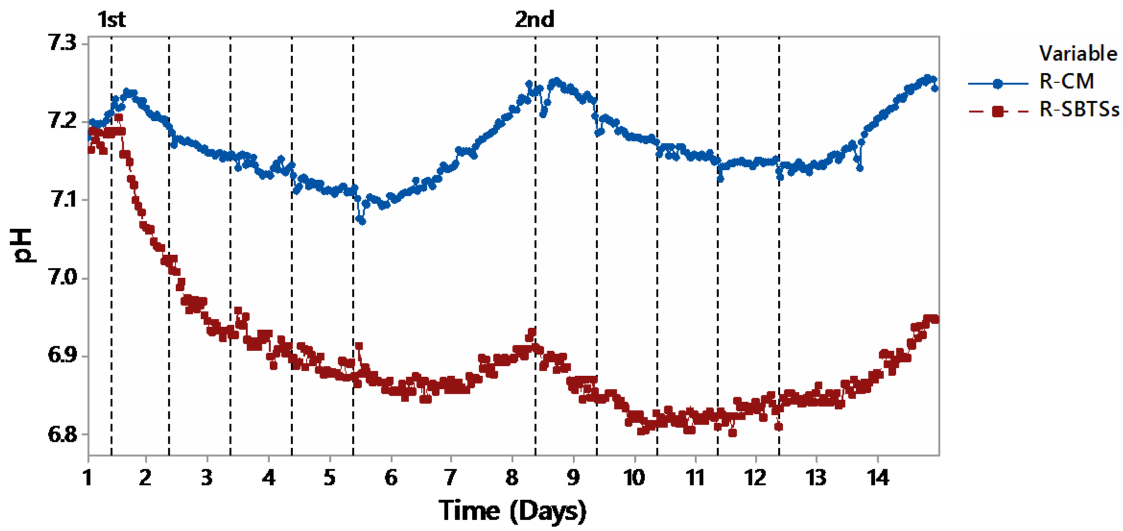


Figure 10. Phase 1. Online monitoring of pH value. Dashed lines represent feeding events. 1st and 2nd specifies the boost events.

On the other hand, higher variations were identified in R-SBTSs. A sudden pH drop was observed after the first boost; the pH dropped from 7.2 to 6.9 during the first two days. Then, the minimum pH was reached on the weekend (6.85). After the second boost, another pH drop was observed. In this case, the decreasing tendency only took two days until reaching its minimum (6.80) and seemed to recover along the weekend. As expected, the pH behavior complemented the VFA concentration. As the VFAs started to accumulate, the pH decreased. The maximum VFA concentrations reached to 8500 and 12000 mg L^{-1}, coincided with the minimum pH values (6.85 and 6.80). Although the optimum pH value is around 6.8 and 7.2 each process step has its own optimum values. Methanogenesis presents an optimum pH around 7.0 whereas hydrolysis and acidogenesis between 5.5 and 6.5. Thus in addition of the VFA accumulation due to overload, the fact of having a pH below 7 reduces the activity of methanogens while acetogens keep producing VFA and, as a consequence, contributing to a higher VFA accumulation. pH recovery could be possible by increasing the buffer capacity.

In phase 1, the only improvement on methane yield was observed during the first boost day. As a result of overload, poor performance of biogas production was acquired in R-SBTSs during the rest of the experimental period. Different signs of inhibition were observed. Methane content decreased 35% during the first boost and 25% during the second boost. VFA concentrations were generally high before starting the experiment, which indicated a low methanogenesis activity. In addition, VFA accumulation was observed after the first boost in R-SBTSs. Only changes in acetic acid and propionic acid were found to be relevant. Acetic acid was identified as the leading VFA to the total accumulation. The fact that the disturbance remained during the whole period, did not allow to identify an inflexion concentration. However, the inhibition started circa 7000 and 3200 mg L^{-1} of acetic and propionic acid respectively. As a consequence of the VFA accumulation, pH dropped during the first and second boost. Thus the pH range in R-SBTSs was permanently close to inhibition on methanogens (6.8). On the other hand, even though also high values of VFA were found in R-CM, the pH range was inside the optimum (6.8-7.2) during the whole period. Furthermore, the ratio IA:PA reflected the relation between the bicarbonate consumption and the VFA accumulation. A value around 0.8 was identified as impending system inhibition.

Phase 2

After the severe inhibition and high VFA concentration observed in Phase 1, it was decided to empty the reactors and add new inoculum. The new inoculum was obtained from Foulum AU main reactor (Table 9). Phase 2 was performed during one month, but the Phase was divided in periods of two weeks (Figure 5). During the experimental period (first two weeks), two different boost were tested in parallel (in addition of cattle manure feedings). One with sugar beet tops and straw silage (R-SBTSs) and the other with Maize silage (R-Ms). During the reference period (second period of two weeks), a reactor only fed with cattle manure (R-CM) and a reactor without feeding but running only with new inoculum were run in parallel.

In both periods, new inoculum was added only on the first day. By this manner, during the second week of each period an accumulative effect of the boost or regular feedings could be observed. The same boost OLR (4 g VS L^{1} d^{-1}) was applied in R-SBTSs and R-Ms in both weeks. Even though, the experimental and reference data were obtained in different periods, in this section the data will be shown as parallel on time. Some setbacks occurred during the second week of both periods. First, the heating mall of R-Ms broke during the last weekend, thus no biogas was produced. Second, due to public holidays, the feeding of regular cattle manure in R-CM was altered. Thus irregular behaviors were registered.

In order to facilitate the comparison between reactors, it has been assumed that the biogas production during the second week in R-CM was the same as the first week. Thus the data obtained from R-SBTSs and R-Ms of the second week will be compared with the data from R-CM of the first week. Besides, no biogas production will be shown from the last weekend, when R-Ms broke down.

However, the other parameters obtained will be compared with the samples taken on the correct time. Even though a different regime was applied, gas composition, pH, VFA, TAN and alkalinity behave as expected. In case of any irregularity, it will be specified.

The biogas production from R-In was subtracted from R-CM, R-SBTSs and R-Ms (Figure 11). Due to the time spent in the inoculum change, the collection of data started at 10am, when the first feeding/boost was applied. Different patterns were identified in both weeks. To facilitate the analysis and further comparisons, both weeks are divided and analyzed by separate.

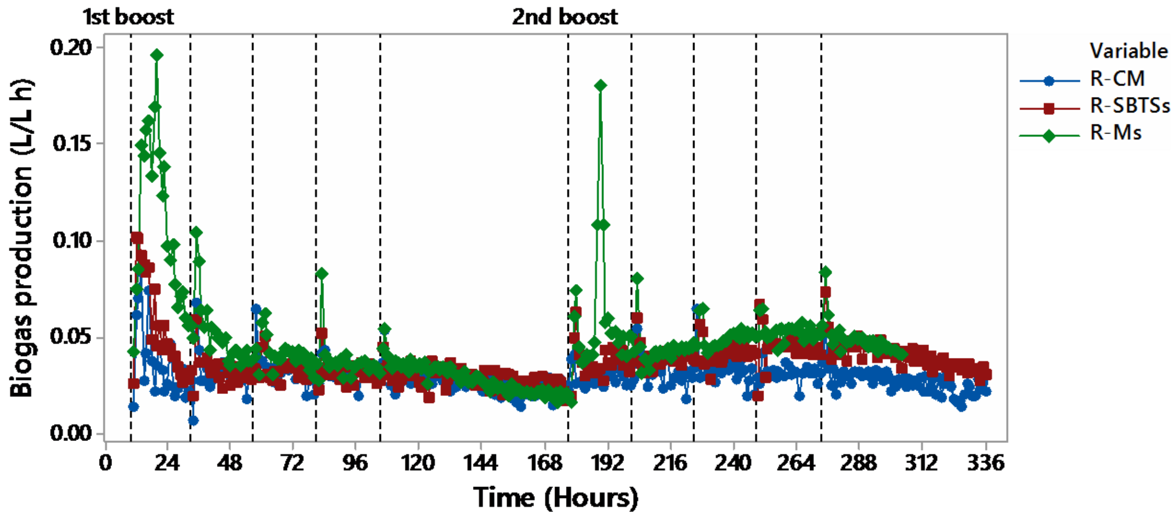


Figure 11. Phase 2. Online monitoring of the hourly biogas production. Dashed lines represent feed events.

During the first week (Figure 11), higher biogas productions were obtained in R-Ms, followed by R- SBTSs and R-CM. Furthermore, a higher boost effect was observed for R-Ms than for R-SBTs. However, after 72 hours the biogas production stabilized in all three reactors. R-CM reached its maximum production (0.083 L L L^{-1} h^{-1}) after 4 hours of the first feeding. On the other hand, R-SBTSs and R-Ms presented their respective peaks (0.10L and 0.196 L L^{-1} h^{-1}) after 2 and 10 hours respectively.

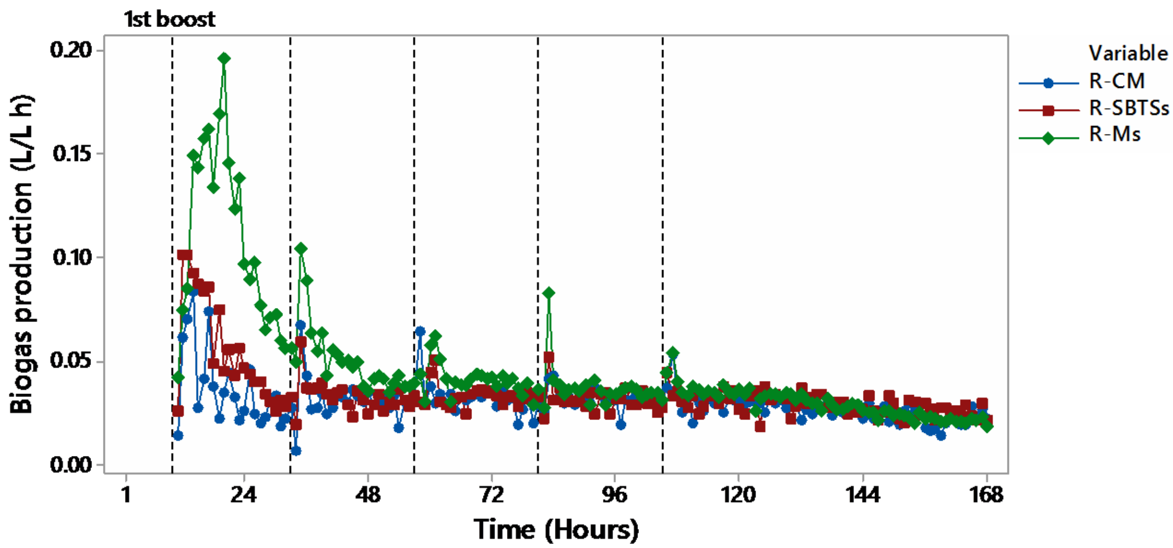


Figure 12. Phase 2. Detail of the online monitoring of the hourly biogas production during the first week. Dashed lines represent feed events.

Even though the response of R-SBTSs was faster (less time to reach its maximum), R-Ms presented a greater boost effect (approx. 24 hours). When the second feeding was applied (day 2), the boost effect of Ms still remained. Thus showing another boost peak (0.104 L $L^{-1}d^{-1}$) during the second day. During the second week (Figure 13), no boost effect was clearly observed. However, R-Ms presented a delayed boost peak after approx. 12 hours of the fed. R-CM reached its maximum peak (0.041 L L^{-1} h^{-1}) after 2 hours of feeding. On the other hand, R-SBTSs and R-Ms reached their respective maximums (0.063 and 0.074 L L^{-1} d^{-1}) after 3 hours. In addition, R- Ms showed a second maximum peak (0.18 l L L^{-1} h^{-1}) after 12 hours of the boost feeding.

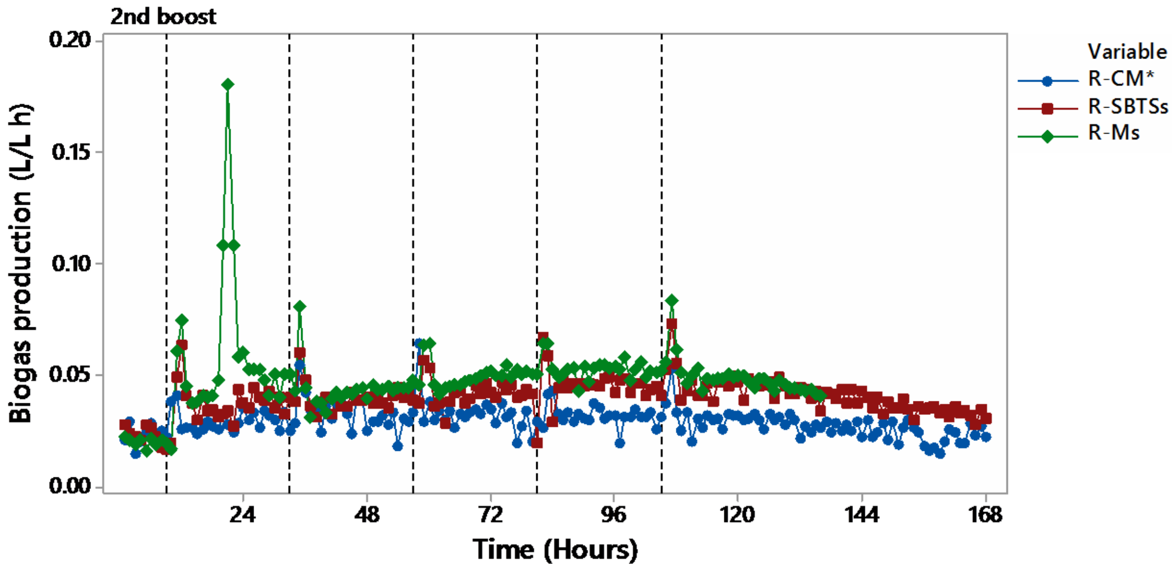


Figure 13. Phase 2. Detail of the online monitoring of the hourly biogas production during the second week. Dashed lines represent feed events. NOTE: R-CM is a remainder of the assumption for the biogas production in R-CM during the second week.*

Global differences became clear with daily biogas yields (Figure 14). During the first week, the expected behaviors were observed in all reactors; R-CM had an increasing tendency during the week and a decreasing one during the weekend; R-SBTSs and R-MS showed higher yields on the boost day and a decreasing production during the rest of the week. On the other hand, during the second week changes were observed. Even though R-SBTSs and R-Ms had higher yields than R-CM after the boost, a clear boost effect was not reflected. The total production of the first week was 4.8, 5.4 and 7.4 L L^{-1} $week^{-1}$ whereas for the second week was 6.4, 6.7 and 6.6 L L^{-1} $week^{-1}$ for R-CM, R-SBTSs and R-Ms, respectively.

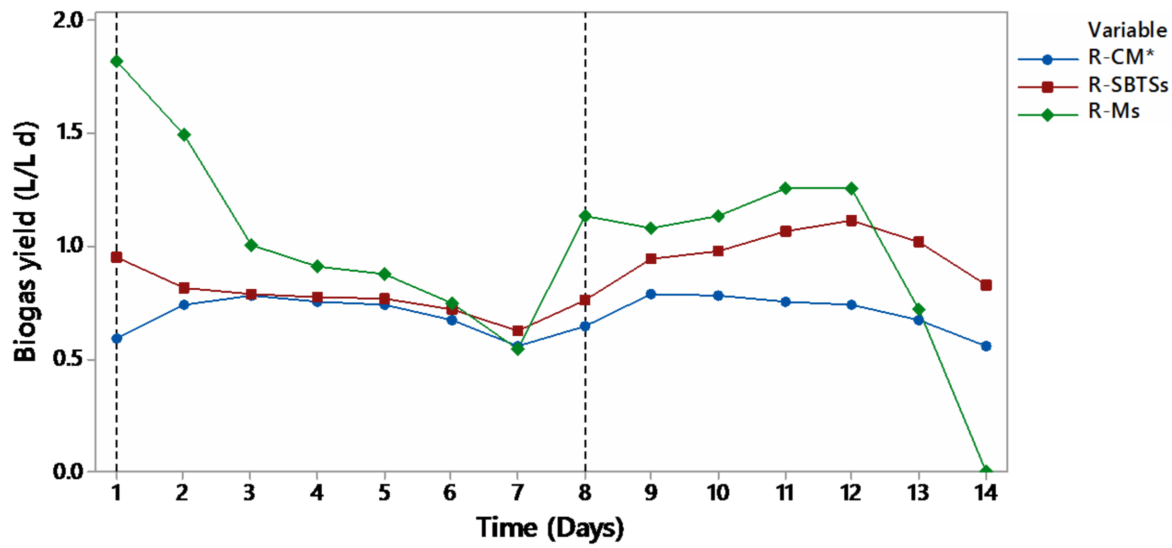


Figure 14. Phase 2. Daily biogas yield. Dashed lines represent boost days. NOTE 1: R-CM is a remainder of the assumption for the biogas production in R-CM during the second week. NOTE 2: The decrease after day 12 in R-Ms is due to the problem with the heating mall.*

Differences in yields between reference and experimental reactors are shown in Figure. For R- Ms, the maximum differences were found on day 1 and day 8 (boost days). On the other hand, for R-SBTSs were found on day 1 and 12. In general, R-Ms reached a higher biogas surplus than R-SBTSs.

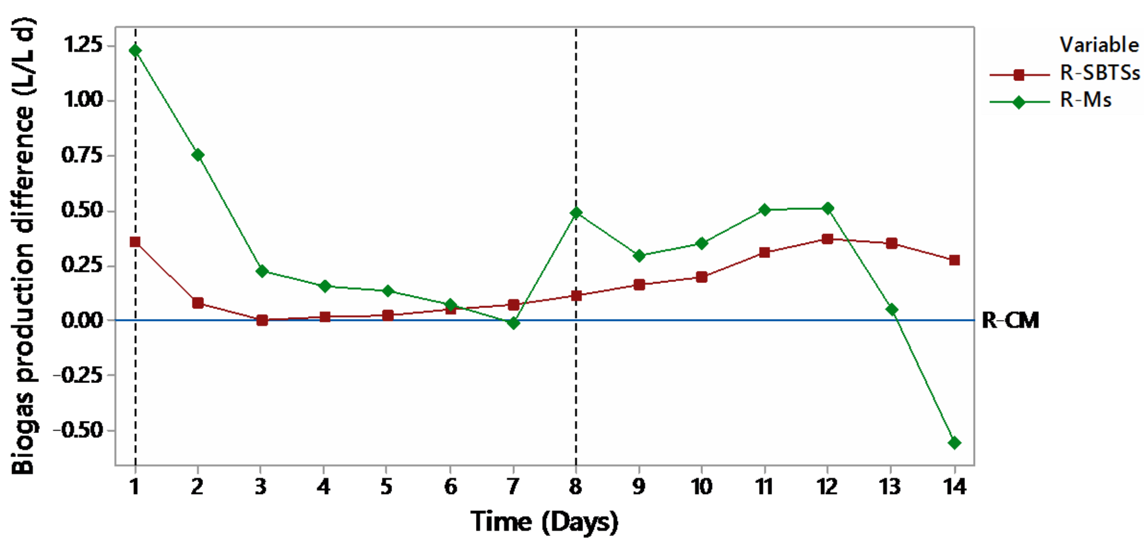


Figure 15. Phase 2. Difference in daily biogas yields between reference reactor (R-CM) and reactors (r- SBTSs and R-Ms). Dashed lines represent boost days. NOTE: The decrease after day 12 in R-Ms is due to the problem with the heating mall.

However, a strange behavior was observed during the second week (increasing productions rather than decreasing). This behavior could be explained by three factors. First, due to inhibition in R- SBTSs and R-Ms. Second, R-CM had different biogas yields during each week (thus wrong assumption). Third, alterations due to inoculum subtraction. The fact that R-SBTSs did not show a boost effect and R-Ms showed a delayed one, could be an indicator of inhibition or system imbalance. In order to clarify this point, special attention will be place during the second boost for the parameters studied. Assuming that R-CM had the same yields during both weeks is a delicate assumption. As the proportion in the reactor of inoculum decreases and manure increases, it would be expected a small increase in yields. Furthermore, small changes in feeding or in the operation actions during that period also may lead to variations.

As stated, one reactor was filled with inoculum without applying any feed. Then, its biogas production was subtracted from the others. While in R-CM, R-SBTSs and R-Ms a daily effluent was discharged (following the 20 days of HRT) in R-In no effluent was flushed out.

In that case, the proportion of biogas production lost from the effluent of R-CM, R-SBTSs and R-Ms (mainly inoculum during the first week) was not accounted. As a consequence, higher biogas productions were deducted, thus leading to lower biogas yields during the first week. During the second week, as the biogas production from the inoculum decreases exponentially and due to the lower proportion of inoculum, the effect was minor. Methane content is shown in figure 16. Due to the time spent in changing the inoculum and the consequent time until the gas bag had enough biogas to be analyzed, the first measure was taken at 4pm.

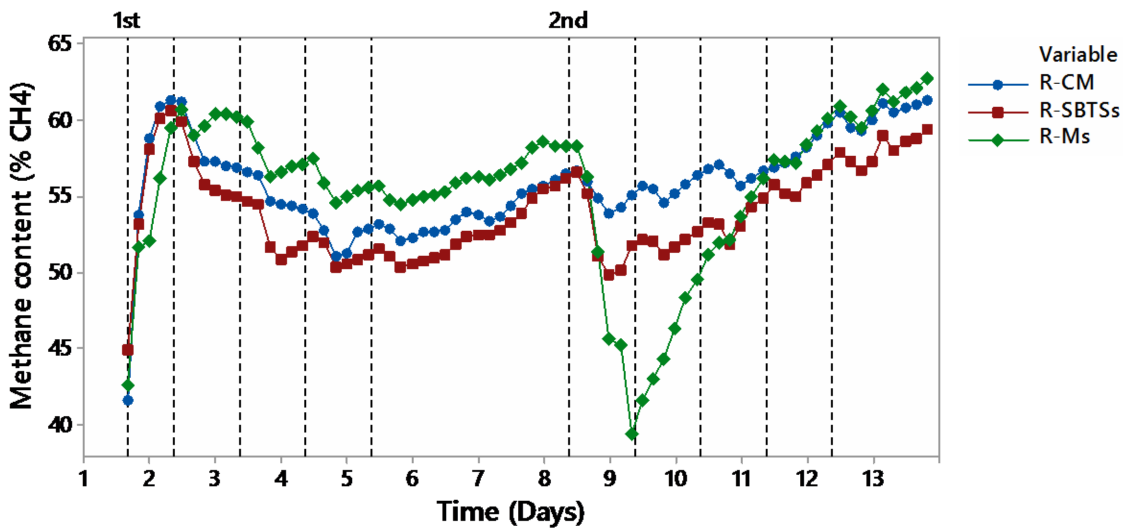


Figure 16. Phase 2. Online monitoring of the methane content during both weeks. Dashed lines represent each fed event. 1st and 2nd refer to the first and second boost.

During the first week, as a consequence of changing the inoculum, some atmospheric air was introduced into the reactor head space. As can be observed, during the first hours CH_4 concentration rose from circa 42% to 61%. Until the volume of the head space (5 liters) was not replaced with biogas, the composition was altered. As a consequence, the impact of the first boost respect the biogas composition could not be observed.

In general, higher CH_4 concentrations were observed in R-Ms, followed by R-CM and R-SBTSs. The maximum CH_4 content was observed on day 2, before the daily feeding (circa 61% in all reactors). Then, the concentration reached its minimum (55, 52 and 50% respectively) on day 6. As seen in Phase 1, the decreasing tendency during the week was due to small drops just after each feeding. The impossibility to recover before the next feed, caused the general decrease. The total CH_4 reduction was 11, 16 and 19% for R-Ms, R-CM and R-SBTSs, respectively.

During the second week, different patterns were observed. R-CM maintained a constant CH_4 level with an increase on day 11 (due to changes in the feed strategy). On the other hand, high variability was observed in the reactors. After the second boost, the CH_4 content in R-SBTSs was reduced an 11% in less than one day (from 56 to 50%). However, after reaching that minimum, the CH_4 rose until 59% on day 13. R-Ms instead, suffered a higher drop. In the same day, a reduction of 33% was observed (from 58 to 39%). Nevertheless, a faster recovery took place. During the following 3 days, the methane concentrations reached higher values than R-CM.

Average CH_4 content was used to calculate the daily CH_4 yield (Figure 17). In addition, differences respect CH_4 production in reference reactor were also calculated (Figure 17). In general trends, biogas yields and methane yields showed the same pattern. However, lower differences between reactors were observed when comparing methane yields. During the first week, R-SBTSs only showed higher yields on day 1 and 2. On the other hand, R-Ms showed higher yields during the whole week. During the second week, a different pattern was observed. R-SBTSs and R-Ms presented increasing tendencies and higher yields compared with the previous week. Besides, coinciding with the drop in CH4 content, R-Ms presented a drop in methane yield on day 9.

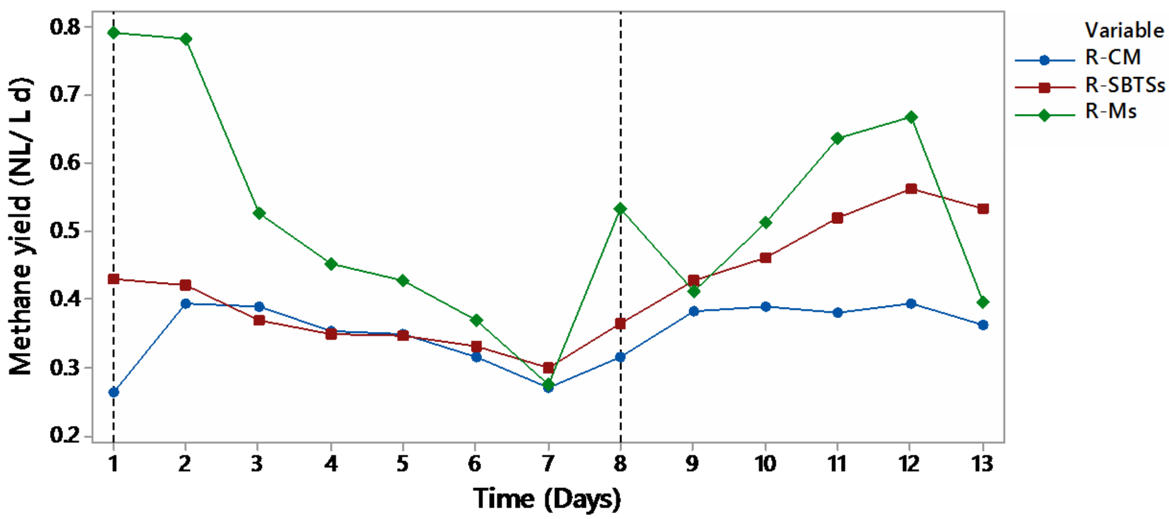


Figure 17. Phase 2. Daily methane yields. Dashed lines represent boost days.

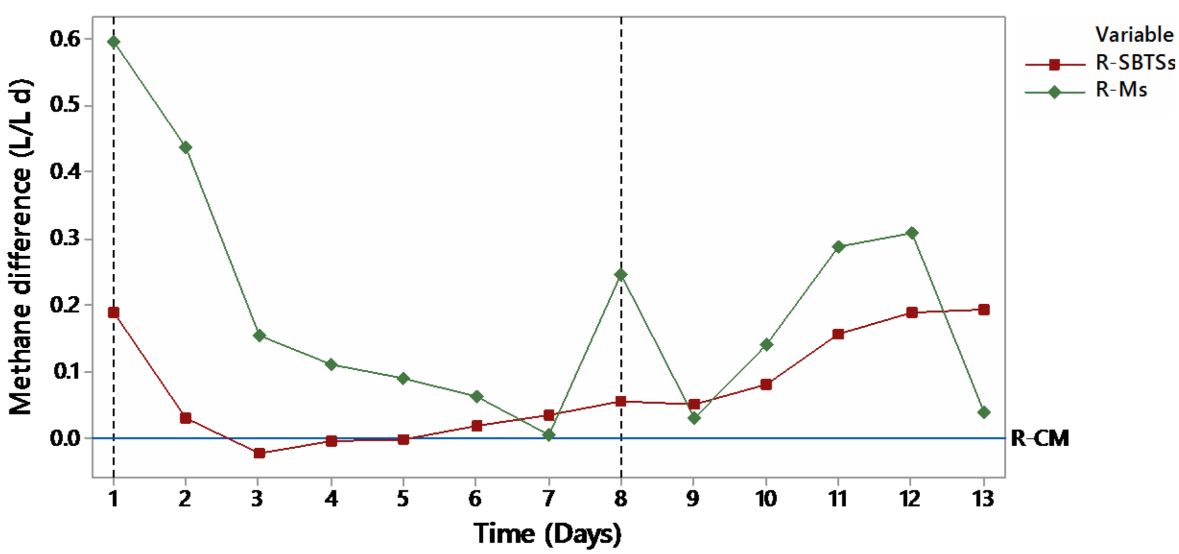


Figure 18. Phase 2. Differences in daily methane yields between reference reactor (R-CM) and Phaseal reactors (R-SBTSs and R-Ms). Dashed lines represent boost days.

The achieved methane surplus during Phase 2 is shown in Table. As can be observed, higher increments were obtained in R-Ms than in R-SBTSs. For instance, during the first boost day, an increment of 200% of the R-CM production was obtained in R-Ms.

Table 2. Increments of the daily methane yields with R-CM as a reference.

Day	**R-SBTSs**	**R-Ms**
1	63%	201%
2	7%	99%
3	-5%	35%
4	-1%	28%
5	-1%	23%
6	5%	18%
7	11%	2%
8	16%	70%
9	12%	7%
10	19%	32%
11	36%	67%
12	43%	70%
13	47%	9%

Even though the surplus of methane, the variation of the biogas quality relativized the improvement. As stated, after feeding events, higher biogas productions were observed but also yielded lower methane concentrations.

Small variations but in the optimum pH range (6.8-7.2) were observed during the first week (Figure 20). However, as a consequence of the inoculum addition, a clear behavior could not be identified after the first boost. General decreasing tendencies were observed coupling with the decrease of CH_4 content and the increase of VFA concentration along the first week. However, as cattle manure is a substrate with a high buffer capacity, the high pH changes could be neutralized.

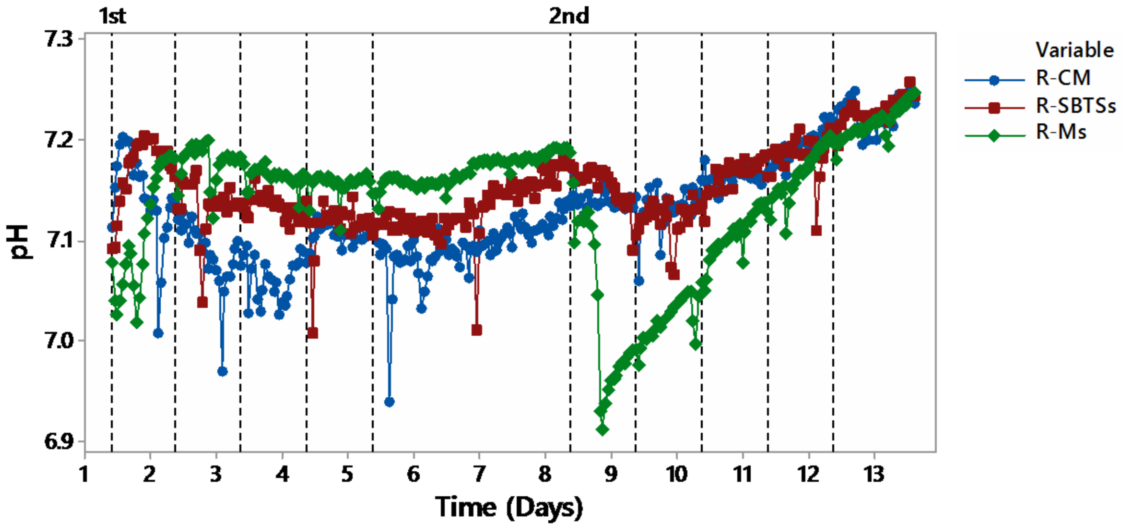


Figure 20. Phase 2. Online monitoring of pH. Dashed lines represent feed events. 1st and 2nd specifies the first and second boost.

During the second week, R-CM and R-SBTSs presented similar behaviors as the previous week. On the other hand, R-Ms showed a pH drop (from pH 7.2 to 6.9) after the second boost. However, once the minimum was reached on day 2, the pH rose up to a pH value of 7.2 on day 12. The sudden pH drop in R-Ms, matched with the CH_4 drop. However the minimum pH (day 9) was reached prior to the maximum accumulation of VFA (day 10). As stated previously, the pH drop is due to an overcome of VFA respect the buffer capacity of the system (Chen et al. 2008). It was caused by the fast conversion of maize silage (a readily degradable substrate) into VFAs thus lowering the pH. However, opposite to Phase 1, the lower VFA concentration of the inoculum helped to a rapid system recovery.

In this Phase, successful biogas improvements were achieved. A maximum increment of the 60% and 200% of the methane yield was obtained in R-SBTSs and R-Ms respectively (compared with R-CM). Due to the questionable results obtained during the second week, no emphasis will be placed on the increments obtained during that period.

However, some signals of process imbalance were observed in R-SBTSs and R-Ms. No boost effect was observed in R-SBTSs during the second week. On the other hand, R-Ms presented a delayed boost effect after 12 hours of fed (day 8). This probably due the inhibitory of accumulated VFA from the boost feeding of Ms. Under the boost feeding configuration, especially when feeding of easily degradable feedstock, the acetogenic becteria producing excess acids than the methanogen could used. The methane production will be inhibited due to the lower pH until returned to balance. This could also be reflected by the suddenly reduced pH and methane content at day 8 of R-Ms.

A reduction of CH_4 was observed in R-Ms and R-SBTSs during the second week. The methane content decreased 11% in R-SBTSs and 33% in R-Ms on day 9.Before the Phases, low concentrations of VFA were found in the inoculum. R-Ms and R-SBTSs showed an increasing tendency during the whole period. On the other hand, R-CM showed an increasing tendency during the first week but a constant concentration during the second.

Maximums concentrations of total VFA (circa 4000 mg L^{-1}) and acetic acid (circa 2700 mg L^{-1}) were found in R-Ms on day 10. Thus a day later than the CH4 drop. The different behavior of acetic acid and propionic acid, allowed the ratio propionic/acetic to be a good indicator of process imbalance. The maximum propionic/acetic ratio (0.65) was found on day 8, thus before the reduction of CH4 occurred. Furthermore, as a consequence of having a healthy inoculum, a clear increment of butyric acid (reaching a maximum of 10%) was detected. R-CM and R-SBTSs were always in the optimum pH range (7.2-6.8) (Ward et al. 2008). On the other hand, as a consequence of VFA accumulation, R-Ms showed a pH drop (circa 6.9) after the second boost (day 8). An extrapolation of the results obtained in Phase 2, can be made to assess the scope of a flexible electricity production from biogas. Only results from the first week are used in order to avoid using questionable data.

In Phase 2, the increments of the methane yield were calculated (reminder, Table 16). Maize silage provided a higher boost effect than silage of sugar beet top and straw. During the first day, R- Ms triplicated (200%) the methane production in R-CM. Besides, presented a daily increment during the whole week (week average of 55%). On the other hand, R-SBTSs increased the yield a 63% during the first day. However, some daily yields reductions were observed resulting in a total week average of 9%. These increments were applied to real data from Foulum AU main reactor. The real electricity production from the biogas plant is shown in Figure 21 together with the two approximations. As can be observed with a boost of 4 kg m^{-3} d^{-1} of maize silage and SBTs, a maximum of circa 10 and 5 Mwh could be produced, respectively.

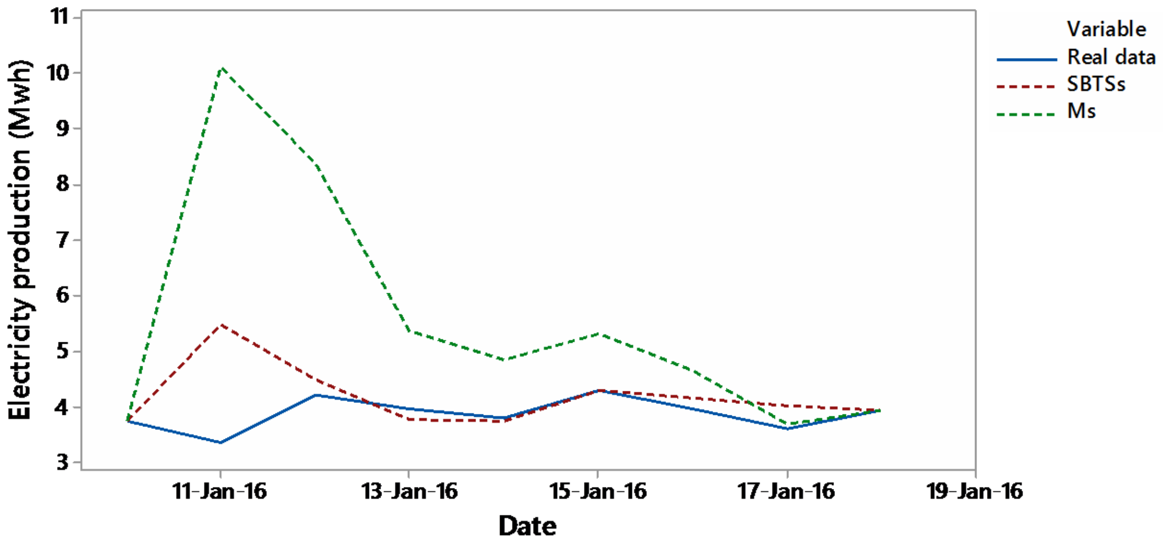


Figure 21. Extrapolation of the increments obtained during the first week of Phase 2. Blue line is the real electricity production from the main reactor in Foulum AU during one week of January 2016. Dashed lines represent the extrapolation, green for R-Ms and red for R-SBTSs.

However, special attention has to be placed to avoid system failure. It has been observed, that the application of two boost (with the same OLR) close in time, can lead to a temporary inhibition caused by sudden VFA accumulation.

To avoid this transient inhibition, some considerations could be made. First, the possibility of reducing the amount of OLR. Second, spaced boost application. Third, changing the feeding regime to smaller but more frequent feedings.

Conclusion:

It has been shown that boosting the biogas yield with SBTSs and Ms for a demand-driven biogas production is possible. Even though the specific methane yield (SMY), referring to the methane production per unit organic matter added, for Ms and SBTSs were similar (338 ± 31 and 326 ± 10 NL CH4 kg VS^{-1}), advantages of using SBTSs (as a second generation crop) instead of Ms (first generation crop) were not proved.

In Phase 2, using maize silage as the boost substrate, increments of 200%, 99%, 35%, 28%, 23%, 18% and 2 % were accomplished during each day of the first week. Representing a 55% surplus in a weekly perspective. On the other hand, using sugar beet tops and straw silage, an increment of 63%, 7%, -5%, -1%, 5% and 11% were accomplished. Representing a 9% in a weekly, perspective.

Special attention has to be placed when boost feedings are applied with a high OLR or in a short period of time. Prolonged inhibition was observed in Phase 1 and transient Inhibition was observed in Phase 2 after the second boost in R-Ms.

3. Boost experiments in pilot scale digesters

3.1 Substrates

Cattle manure was obtained from Aarhus University Foulum (Tjele, Denmark) in March 2017. Meadow grass was harvested from a meadow near Ribe (West Jutland, Denmark). The harvested grass was left in the field and dried naturally for three days before collection. The dominant species in the meadow grass were: Phalaris arundinacea (80%), Holcus lanatus (10%) and Glyceria fluitans (5%). The grass was hammer-milled with a 20-mm sieve (Cormall HDH770, Denmark) first and briquetted with BP 6500 briquetting unit (CF Nielsen, 9574 Bælum, Denmark) before being fed into pilot reactors. Maize silage was obtained in September 2016 and kept as a silage until the experiment. All pilot-scale reactors had been running mainly with cattle manure as main substrate and they had been monitored for around 1 year before pulse feeding.

Table 3. Composition and BMP of cattle manure, briquetted meadow grass, maize silage and digestate prior to pulse feeding.

Component	Unit	Cattle manure[a]	Briquetted grass	Maize silage	Thermophilic digestate[b]	Thermophilic digestate[c]	Mesophilic digestate[c]
TS	(%)	9.07±0.02	88.42±0.27	34.76±0.49	6.83±0.02	5.91±0.19	6.39±0.64
VS	($\%_{TS}$)	83.26±0.76	94.20±0.41	91.40±1.44	79.24±0.11	77.40±0.28	77.44±1.63
Ash	(%)	1.52±0.06	5.13±0.34	2.99±0.95	5.46±0.07	1.34±0.59	4.95±0.60
VFAs	(mg.L^{-1})	14744.43	ND	ND	413.41	689.69	462.72
pH		6.38	ND	ND	7.79	8.13	7.98
TAN	(g.L^{-1})	1.00	ND	ND	1.00	2.30	2.54

[a] Only the characteristics of cattle manure used in first experiment are presented since they were very similar.
[b] Experiment 1.
[c] Experiment 2.

3.2 Pilot-scale experiment

The pilot-scale experiment was conducted using three pilot-scale CSTRs with total working volumes of 10 (one digester) and 30 m^3 (two digesters). All these pilot-scale reactors were constructed of stainless steel and equipped with an external water jacket as the heating system. Biogas volumes were measured by differential pressure transmitter (EJX110A, Yokogawa Electric Corporation, Japan). Feeding and discharging of digesters were controlled automatically by weighing the substrates inside of digesters according to the experimental hydraulic retention time (HRT). All the reactors were fed with cattle manure two times per day, where the first feeding was normally given at 8:00 am followed the next feeding at 8:00 pm automatically. Prior to the boost (pulse feeding) test, all reactors were fed with 100% cattle manure and controlled separately for over 3 months. The organic loading rate of the pilot-scale reactors during manure-only feeding was *ca.* 2.60 kgVS m^{-3} d^{-1}, which was doubled (5.20 kgVS m^{-3} d^{-1}) by pulse feeding with maize silage or grass.

Test 1 Pulse feeding with briquetted grass
In the first experiment, two 30 m^3 thermophilic (51 ^oC) CSTRs (FR1 and FR2) were monitored during two consecutive weeks. The 1st reactor (FR1) was fed without any change and set as control, while the 2nd reactor (FR2) was pulse fed with briquetted grass once a week to double the daily OLR. The pulse feeding was given manually at 12:00 pm on the 1st day of each week.

Test 2 Pulse feeding with maize silage
In this experiment, flexibility of biogas production by pulse feeding of maize silage was investigated under both thermophilic and mesophilic conditions (52 and 41°C). Two pilot-scale reactors (FR1 and FR3) with different working volumes (30m^3 and 10 m^3, respectively) were monitored during two consecutive weeks. Both the reactors were fed with cattle manure and pulse fed with maize silage. Maize silage was manually fed only once at 12:00 pm during the 1st day. The week prior to pulse feeding was used as reference since no pilot-scale reactors were available as control in this experiment.

3.3 Biogas flow rate/yield

Pulse feeding with briquetted grass
Figure 22 and Table 4 shows the normalized volumetric biogas yield and biogas yield percent enhancement compared to the reference reactor. Weeks 1 and 2 showed similar results. The daily biogas yield was boosted by *ca.* 28-30 % at day 1 following each pulse with a maximum after two days of *ca.* 34-40% enhancement. After the second day following each pulse, the % enhancement decreased to *ca.* 17-20 % at day 7. There was around 28% more biogas produced in a week from a single pulse feeding of briquetted grass.

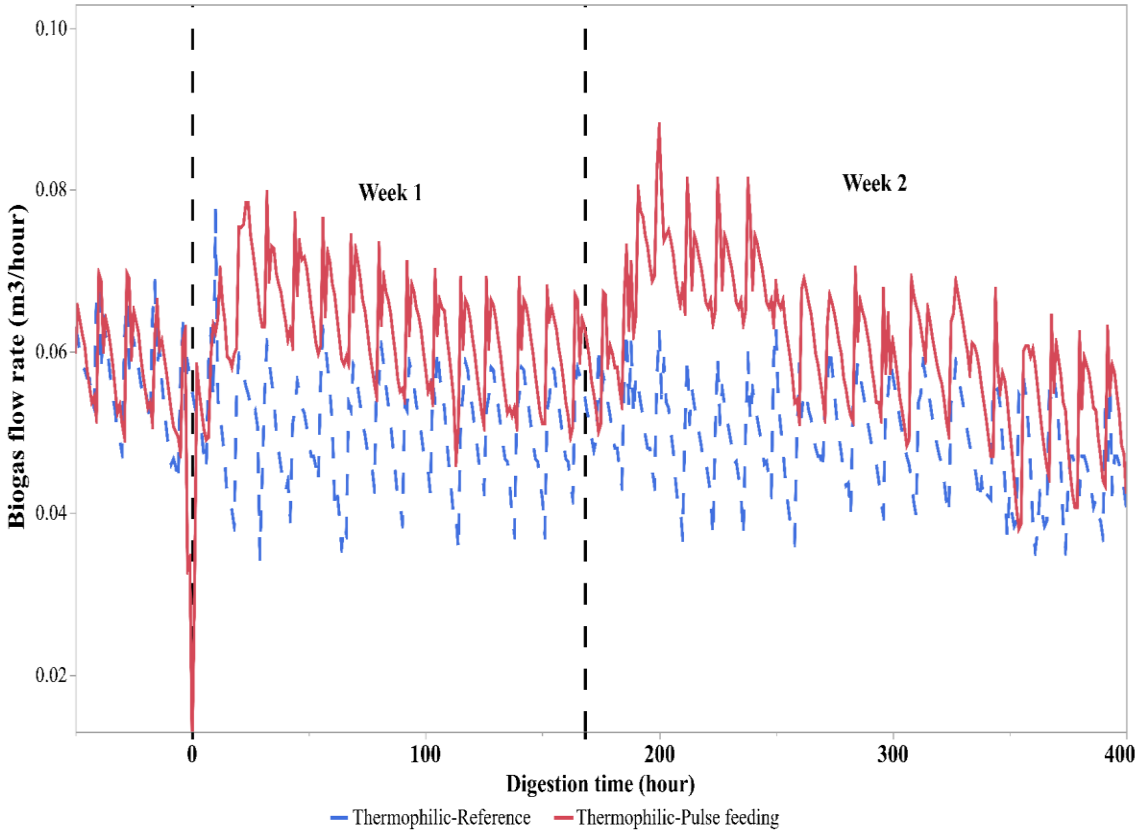


Figure 22. Volumetric flow rate after pulse feeding of briquetted meadow grass

Table 4. Volumetric biogas yield and the enhancement of pulse feeding of briquetted grass under thermophilic condition

Digestion time (d)	Volumetric biogas production ($m^3.m^{-3}_{reactor}$ d^{-1})			Pulse enhancement (%)[a]	
	Without pulse feeding	Pulse week 1	Pulse week 2	Week 1	Week 2
1	1.31	1.68	1.71	28.65	30.74
2	1.21	1.62	1.69	34.26	40.60
3	1.19	1.55	1.67	30.03	40.22
4	1.32	1.62	1.60	22.00	21.17
5	1.05	1.25	1.24	19.57	18.80
6	1.21	1.40	1.43	16.18	18.25
7	1.09	1.31	1.28	19.88	17.26
Total	8.38	10.43	10.62	--	--
Average	1.2	1.49	1.52	--	--

Pulse feeding with maize silage

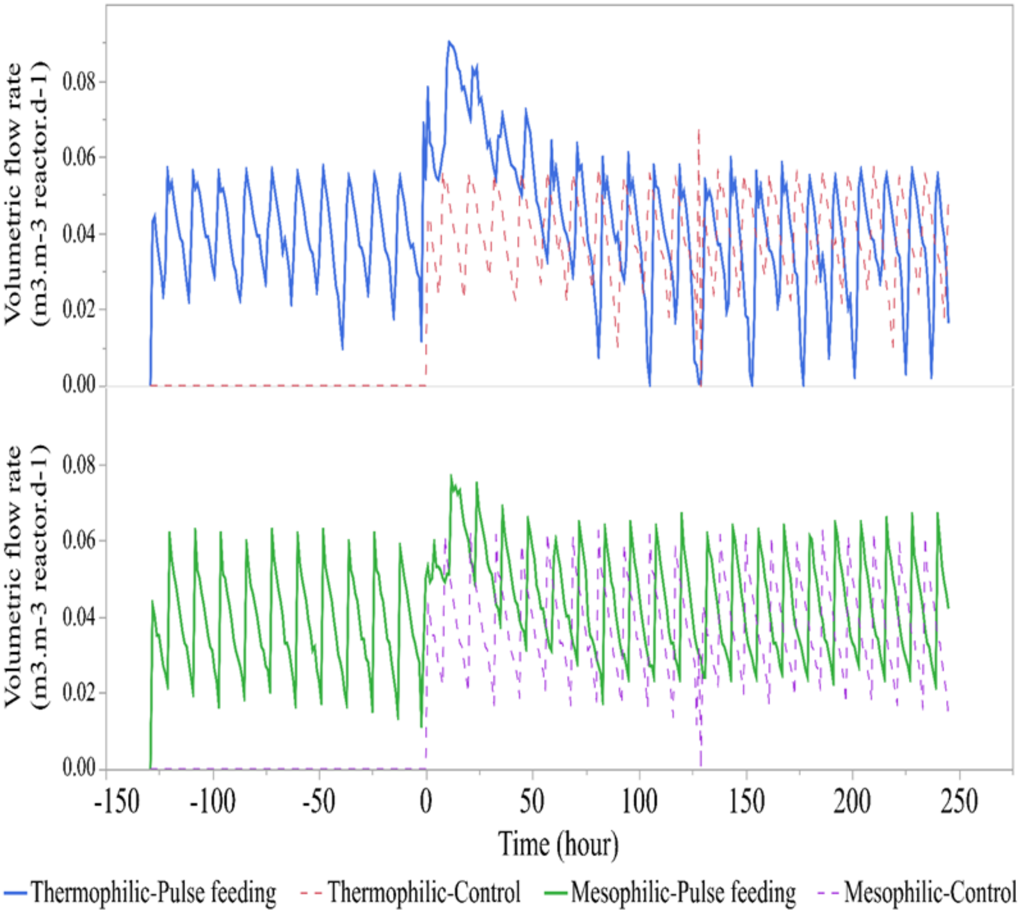


Figure 23. Volumetric flow rate after pulse feeding of maize silage

Both the Figure 22 and Table 5/6 show the volumetric biogas yield and enhancement compared to the yield without pulse feeding for thermophilic and mesophilic reactors, respectively. The volumetric biogas yield prior to the pulse feeding from both reactors was similar (0.9 m^3 per m^3 reactor) for both thermophilic and mesophilic digesters. However, different trends were observed after pulse feeding maize silage; the biogas yield from the thermophilic digester was found to be significantly enhanced during the 1st and 2nd day after pulse feeding. The biogas yields for days 1 and 2 was 84.75% and 55% (respectively) higher than the day without maize addition, and decreased to values less than the previous week before the pulse addition for days 5, 6 and 7). Compared to thermophilic AD, pulse feeding maize silage at mesophilic AD shows more moderated fluctuation. The daily biogas yield at day 1 was 57% higher than the day without maize silage, and 34% and 15% higher at days 2 and 3, respectively. The mesophilic digester was producing 6-9 % more biogas than the control during days 5-7, whilst the thermophilic digester was producing less than the control at this time after pulse addition. As reported by Forster-Carneiro et al. (2008), although thermophilic AD has advantages including a higher biogas yield and lower minimum retention time compared to mesophilic AD, mesophilic processes are normally more robust to temporary inhibitors and sudden environmental changes (Angelidaki & Ahring, 1994). In terms of the flexible biogas production, pulse feeding of maize silage showed a faster response regarding boosting of biogas production as well as a more rapid return to the prior level of gas production. The negative effect on biogas production from the thermophilic digester also shown that the system was more sensitive when fed with easily degradable substrate since no negative influence was observed when feeding briquetted grass at the same temperature. As reported by Labatut et al. (2014), substrate with higher fiber content can provide more surface area to absorb and decrease the inhibitor concentration.

Table 5. Volumetric biogas yield and the enhancement of pulse feeding of maize silage under thermophilic condition

Digestion time (d)	Volumetric biogas yield ($m^3.m^{-3}_{reactor}\ d^{-1}$)		Enhancement (%)[a]
	Without pulse feeding	Pulse feeding with maize	
1	0.99	1.83	84.75
2	0.97	1.51	54.71
3	0.99	1.13	14.15
4	0.91	0.94	4.08
5	0.94	0.86	-8.48
6	0.96	0.80	-16.52
7	0.96	0.83	-13.36
Total	6.72	7.91	17.60
Average	0.96	1.13	

[a] Compared with the same day of the week without pulse feeding.

[b] Average based on the monitored week.

Table 6. Volumetric biogas yield and the enhancement of pulse feeding of maize silage under mesophilic condition

Digestion time (d)	Volumetric biogas yield ($m^3\ m^{-3}_{reactor}\ d^{-1}$)		Enhancement (%)[a]
	Without pulse feeding	Pulse feeding with maize	
1	0.94	1.47	56.70
2	0.91	1.22	34.44
3	0.93	1.08	15.57
4	0.91	0.97	6.51
5	0.90	0.99	9.87
6	0.92	0.98	6.76
7	0.92	1.00	8.83
Sum	6.42	7.70	19.96
Average	0.92	1.10	

a Compared with the same day of the week without pulse feeding.

b Average based on the monitored week.

3.4 Process performance

The process data (methane content, VFAs concentration) prior to and after pulse feeding of briquetted grass are shown in Fig. 25. Biogas produced from the reference reactor consisted of 58 to 60% methane throughout the experimental period. Methane concentration fell slightly to *ca.*57% following the first pulse feeding, returning to a value similar to the reference digester after 5 days. After the 2nd pulse feeding, the changes of methane content followed the same pattern as the 1st week although the methane content dropped to 54% after 1.5 hours of the pulse feeding and returned to the reference level after 110 hours following the 2nd pulse feeding. The reduced methane content was attributed to the extra CO_2 released during acidogenesis process after pulse feeding. VFA concentration after pulse feeding of grass was found to be higher than the reference digester within 48 hours after pulse feeding. From hour 24 to 48, the VFA concentration from the pulse fed reactor was 650-660 $mg.L^{-1}$, which was 20% higher than the reference reactor in the same time period. However, it can be observed that the VFA concentration decreased to 440-480 $mg.L^{-1}$ at hour 120, which was similar to the reference digester.

Changes in process parameters after pulse feeding of maize silage were found to be more dynamic than those of grass. As shown in Fig. 24, after 30 mins of pulse feeding, the methane content from mesophilic reactor increased from 60% to over 65% and maintained a level over 60% during the 1st day. Conversely, the methane content from the thermophilic reactor dropped significantly from 58% to 44% after 30 mins of pulse feeding and gradually returned to 60% during the next 4 hours. VFAs concentrations from both thermophilic and mesophilic digesters enhanced from 500-700 and 460-570 $mg.L^{-1}$ during the 1st hour of pulse feeding and reduced to lower than 400 $mg.L^{-1}$ at hour 48.

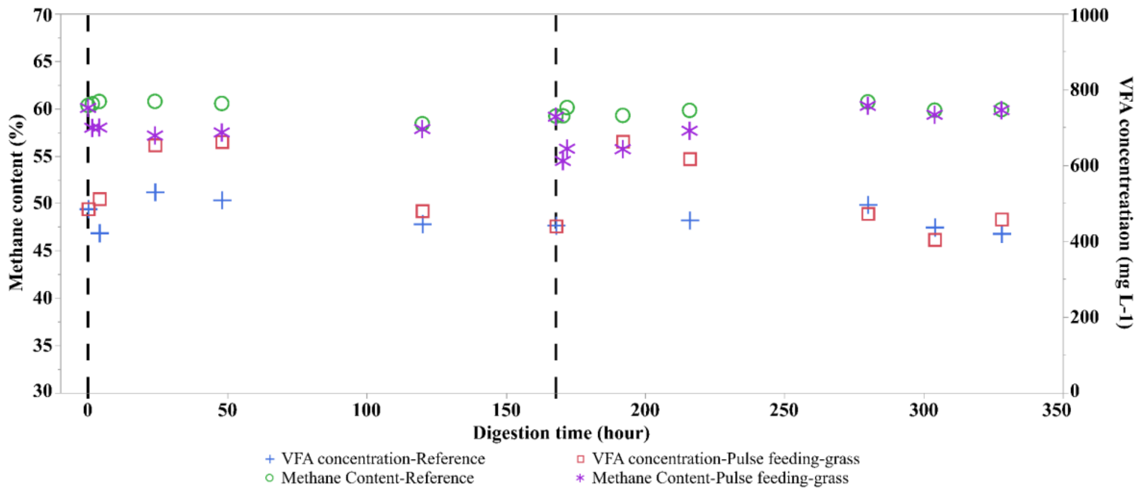


Figure 24. VFAs concentration and methane content -with/without pulse feeding of briquetted grass. (The dot blank line represent the time of pulse feeding)

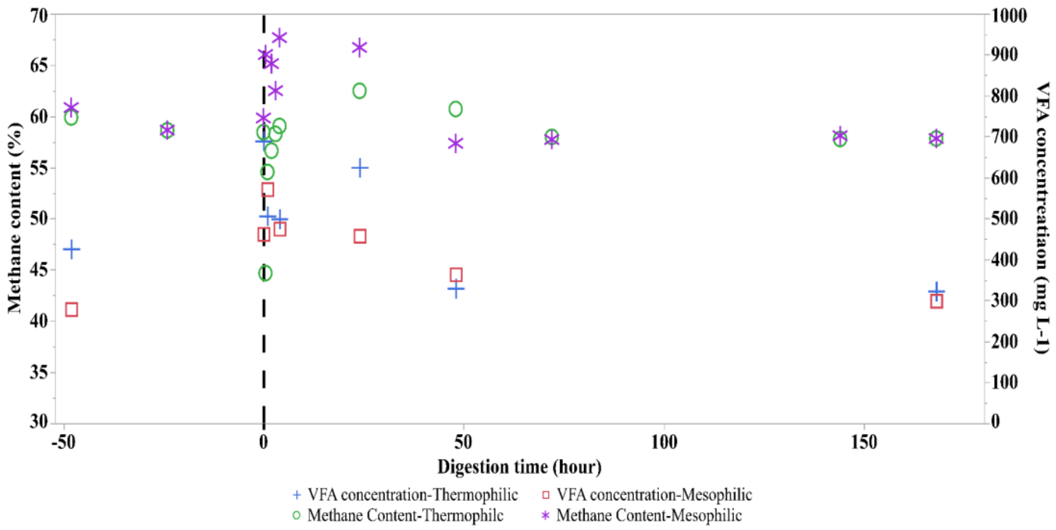


Figure 25. VFAs concentration and methane content-pulse feeding of maize silage. (The dot blank line represent the time of pulse feeding)

3.5 Conclusion

Pulse feeding of maize silage resulting from short time doubling the OLR show higher impact in providing flexibility on biogas production compared to using grass as sub-strate. The daily biogas production was 85% (thermophilic) and 57% (mesophilic) higher with pulse feeding of maize silage during the proceeding 24 hours, and returned to normal steady state production after 3 days.

4 Long term increase loading in pilot scale digesters

An experiment with two types of boosting material has been performed over a 5 months period. In the period 2 reactors with a volume of 10 m3 and 2 reactors with 30 m3 were used. The 30 m3 reactors was kept at thermophilic temperature (53 C) and 10 m3 reactors was kept at mesophilic temperature (35 C). 4 different starting inoculums coming from 4 different biogas plants was used in the 4 reactors to indicate if different biogas plants react different on straw rich substrates.
The basic substrate was cattle manure and in the first half of the period 3% straw pellets was added the digesters and the HRT was 25 days. In the second half of the experiments 3.75% silage made from beet leaves and macerated straw was used. In Figure 26 the results in terms of gas output from the digesters is illustrated. It can be seen that the use of straw pellets give a rapid increase in gas production in the 2 thermophilic digesters with one of them being slightly superior to the other indicating that different thermophilic plants react different. The mesophilic digesters react much slower on addition of straw and the biogas yield is in the period always considerably lower than the thermophilic. From the results with straw it can be concluded that thermophilic digesters react much faster on straw rich substrates. After shifting to the new silage substrate which is more degradable than straw the gas yield decline caused by the lower organic loading since the organic dry-matter in the substrate is less than half of that in straw. Towards the end of the experiment the mesophilc and thermophilic gas yields are quite similar indicating that mesophilic digesters works better with substrates that has a higher degradability.

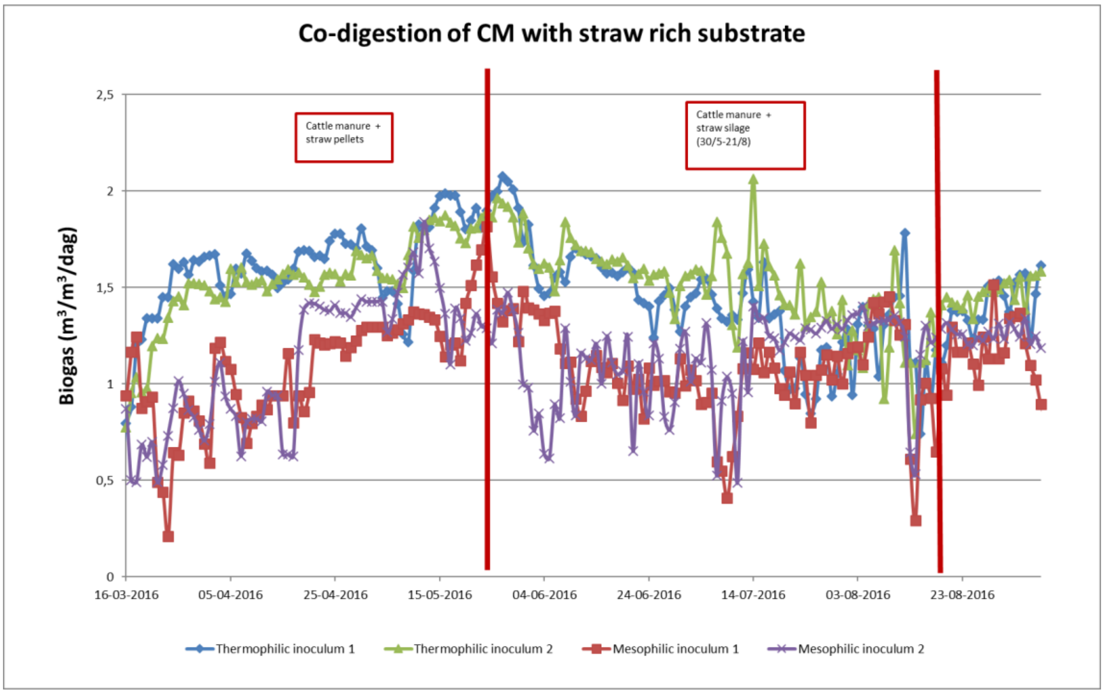
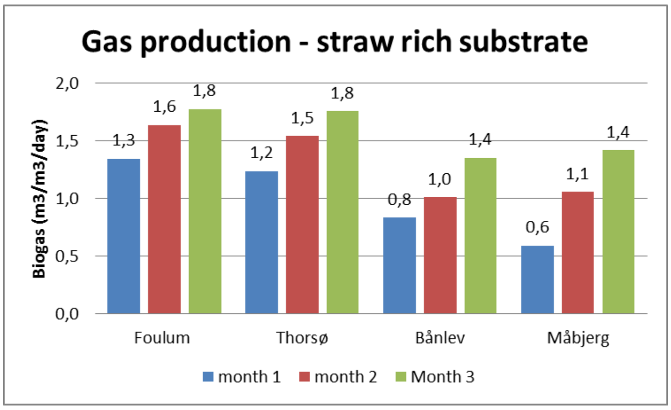


Figure 26. Gas production from co-digestion of cattle manure with straw pellets and silage from straw and sugar beet leaves.

In figure 27 the average gasproduction on a monthly basis is shown in the digesters co-digested with cattle manure and straw pellets/straw-beet leave silage. It can be seen that there is a long transition time before the straw rich substrates gives the maximum yield with the thermophilic digesters being superior. Thus if an increased production is planned the starting of adding the straw rich substrate should be started at least 1 month ahead. For the straw-beet leaves silage the yields are lower than for the straw pellets. The reason for this is that it is not possible to mix as much dry-matter in the cattle manure with the silage as with the pellets, since the size reduction is not as good and the silage process do not compensate sufficient for this. For the silage there is also a very positive effect by having a thermophilic temperature.



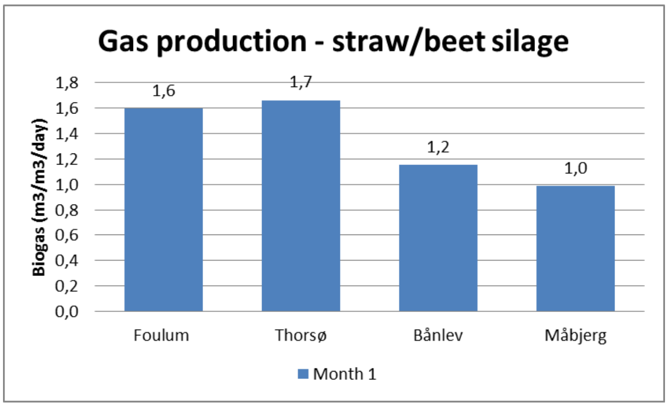
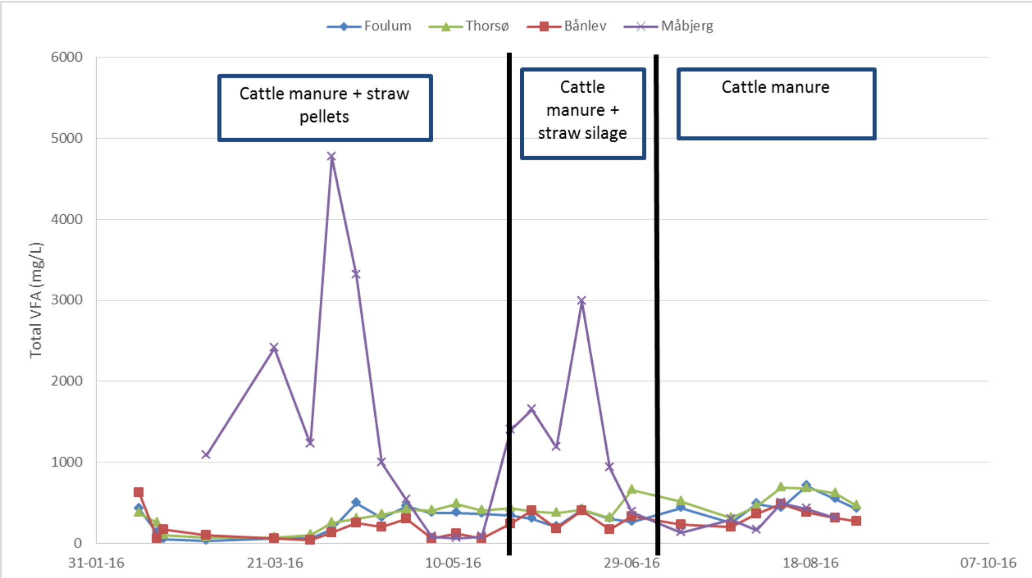


Figure 27. Gas production from co-digestion of cattle manure with straw pellets and silage from straw and sugar beet leaves

In figure 28 the VFA content and the methane concentration in gas is illustrated. It can be seen that during the start up with straw pellet one of the mesophilic digesters

(Måbjerg inoculum) is negatively affected with high VFA and low methane concentration. The rest of the digesters is not affected negative by the load of straw. The same picture is seen after the transition to straw-beet leave silage where the same digester is responding with increasing VFA level but in this case the methane concentration is not negatively affected. Overall it seems that responses to load of new substrate is inoculum depending.



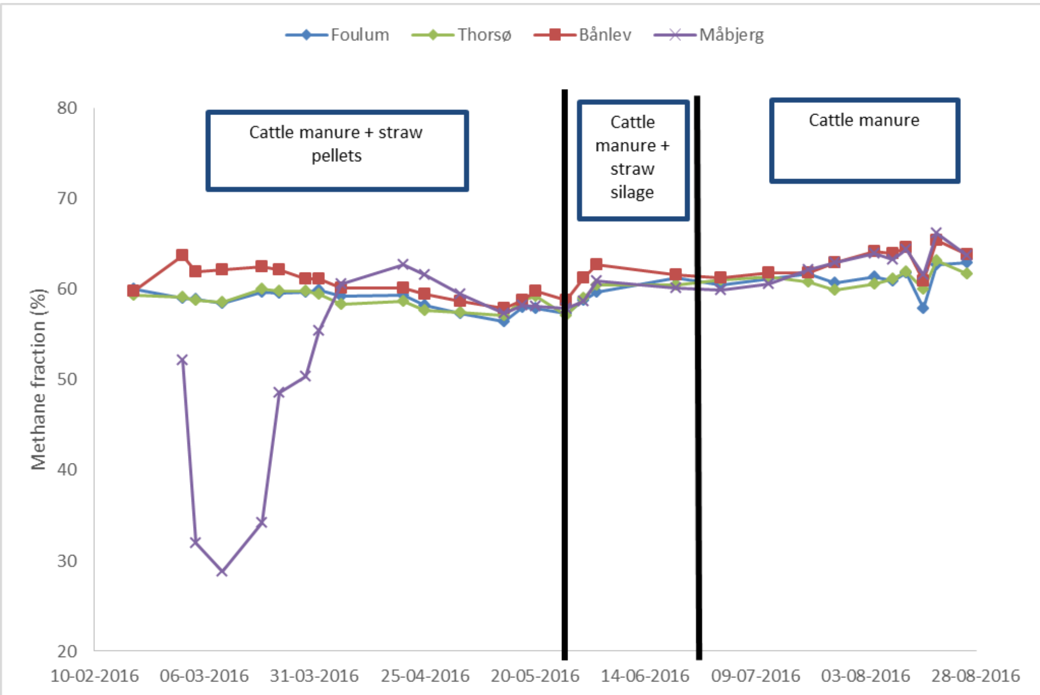


Figure 28. Total volatile fatty acids and methane concentration in gas

5. Final Conclusions

It has been proved from this study that boosting biogas yield for a demand-driven biogas production with pulse feeding is possible. This is true for all the tested substrates, sugar beet leaves-straw silage, meadow grass pellets and maize silage. Pulse feeding with maize silage shows higher flexibility than other substrates both in lab and pilot-scale. However, the risk of inhibition was also higher using maize silage compared to other substrates. The effect of pulse feeding varies depending on the feedstock, loading rate, intervals between pulsed feeding and the stability of AD system prior to pulse feeding. It is important to monitor the performance more frequently after pulse feeding to evaluate the stability and prepare for next pulse feeding. In our study, it was possible to give pulse feeding once a week without process failure. The stability of mesophilic AD was higher than thermophilic AD. Reactors pulse fed once a day (in short term) lead to a transient accumulation of fermentation products (VFA and CO_2) and a temporal variation of the pH. A moderate pulse feeding strategy could be considered, such as feeding several times per day or to keep on constant feeding instead of many pulses, to raise the stability of AD system. Further research in order to identify optimal substrate loads, boost frequencies and optimal substrates for a successful boost performance is necessary. Though maize seems to be very suitable boosting substrate in future it will be less acceptable due to demand for a more environmental sound biogas production. In this study straw pellets, straw-beet silage have proven to be good substrates for increasing gas production and especially for a gradually increase in biogas production e.g. going from summer to winter period. Hence the full effect of these substrates takes more than a month to reach. Straw pellets is easier to use than the straw silage used in our experiment since the ability to mix with manure is better for the pellets enabling higher organic load. The experiments shows that different reactors react different on the same load and in general the gasyield of the straw rich digesters are significant higher at thermophilic conditions.

6. Dissemination AU:

Pau Grima Guixe. 2016. Regulating biogas yield to meet fluctuations in energy demand. Thesis supervisors: Henrik B. Møller, Lu Feng. Department of Engineering, Aarhus University.

Lu Feng*, Henrik Bjarne Møller, Pau Grima Guixé and Erik Fløjgaard Kristensen. Grass and agricultural byproducts for energy - an optimized anaerobic digestion technology. International Conference on Agricultural Engineering 2016, Aarhus, Denmark.

Feng, L., Møller, H.B., Moset, V., & Ward, A. J. 2017. Flexible biogas production by pulse feeding of maize silage briquetted meadow grass/ or briquetted meadow grass

maize silage using pilot-scale continuous stirreding tank reactors. Prepared for submission to Bioresource Technology.