Grass and agricultural byproducts for energy - an optimized anaerobic digestion technology

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Abstract

Anaerobic co-digestion of cattle manure with meadow grass was investigated in two-/one-step thermophilic continuous stirred tank reactors (CSTR) in lab-scale. Four lab-scale CSTRs (15L) were used at 20 days hydraulic retention time (HRT); the first two reactors which configured as two-steps (10 days HRT for each reactor) was fed with 95% cattle manure and 5% meadow grass; and the rest two reactors were fed with 95% cattle manure and 5% meadow grass (co-digestion & single step) or only with cattle manure (mono-digestion & single step), respectively. With a two-step configuration, the specific methane yield of 95%CM+5%MG and CM were 107.14 and 75.16 NL/kg VS, which corresponding to 23.96 and 10.48% increase compared with one-step co-digestion. 42.54 % and 27.05% increase of SMY can be achieved with 5% grass added at two-and one-step anaerobic digestion. However, the SMY obtained from lab-scale experiment were lower when compared with the BMP value.

Flexible biogas production with boost feeding regime was also investigated. Two boost with different substrates, maize silage and suger beet tops+Straw (4 g VS /L day), were compared in two different pilot scale reactors (R-Ms and R-CM) based on cattle manure feedings (4 g VS /L day) but with addition of new inoculum. Maximum increments of 200% and 60% of the methane yield were obtained in R-Ms and R-SBTSs respectively. However, transient inhibition was detected in R-Ms after the second boost. Reduction on CH_4 content (minimum of 40% CH_4), accumulation in VFA (maximum of 4000 mg L/L), pH decrease (minimum of 6.9) was observed.

Keywords: Co-digestion; Flexible biogas production; Meadow grass; Cattle manure; Multi-step.

1. Introduction

As an increasing energy demanding and critical environmental issues, more and more agricultural byproducts have been used as possible substitute resources for bio-energy/biogas production. In 2010, Denmark has decided to be the first country in the world to lead the transition and become a green and resource efficient economy entirely independently of fossil fuels by 2050. According to the ambitious target, the Danish government proposed to use 50% of livestock manure for green energy (The Danish Government, 2011) which means there will be a significantly increase for livestock manure utilization for energy purpose (only 5-7% of the available manure in Denmark were used for biogas production in 2013) (Birkmose et al., 2013).

Considering the lower carbon intensity and poor quality of available organic content in manure, more and more co-digested biomass were explored recent years, such as such as fruit/ vegetable wastes, energy crops and its residues, municipal solid waste (MSW), food waste, et al. (Callaghan et al., 2002; Cavinato et al., 2010; Lehtomäki et al., 2007; Macias-Corral et al., 2008; Zhang et al., 2013). The choices of co-digestion substrates is mainly determined by their availability and bio-degradability (Pokój et al., 2015) and also the environmental influence(Vega et al., 2014). Recently years, an increased attention about energetic utilization of agricultural by-products and meadow grass had risen around the world (Hohenstein & Wright, 1994; Klimiuk et al., 2010; McLaughlin et al., 2002; Murphy & Power, 2009; Prochnow et al., 2009; Vogel, 1996).This mainly due to the higher carbon intensity and sufficient supplement.

The objectives of this research were to 1) Flexibility biogas production from agricultural by-product based on demand-driven; 2) Optimization of biogas production by co-digestion of meadow grass as a multi-step configuration.

2. Materials and Methods

2.1. Substrates

Exp-1 Flexible biogas production

Sugar beet tops and straw were cultivated, mixed and chopped in sizes of 10 cm in Henry Kuhr’s farm. Afterwards, the mixture was preserved as silage in 5 liters vacuum bags during three weeks. The vacuum process was performed in AgroTech facilities.

The maize was collected from Danish Cattle Research Centre (8830 Tjele, Foulum). After harvesting was chopped in sizes of 3 cm approximately and storage as silage for 3 months.

Exp-2 Multi-stage

The cattle manure was collected from the animal facilities in Research Center Foulum (Aarhus University, Denmark) and kept at -18^{o}C during the whole period.

The meadow grass used in this experiment was grown in meadow area (Obbekærvej 59, 6760 Ribe, Denmark) and harvested at July 2015 which about 1 year from the last harvest. Predominant species in the mixed meadow grass were:

Phalaris arundinacea (80%), Holcus lanatus (10%) and Glyceria fluitans (5%).

All samples were sealed at airtight plastic bags and stored at -18 ^oC to prevent sample degradation.

Thermophilic inoculums were obtained from a thermophilic reactor located at Research Center Foulum (Aarhus University, Denmark) which had been running for more than 1 year under thermophilic condition. The inoculum was de-grassed with a screen before used. Before start-up, all reactors were completely filled with thermophilic inoculums. No feeding and discharge during the first two weeks.

2.2. Batch test

Ultimate methane yield from each feedstock was determined in a batch test at thermophilic temperature (52^{o}C) and converted as standard conditions (Angelidaki & Sanders, 2004). Prior to starting the batch test, the thermophilic inoculum used in semi- and BMP test were pre-incubated at 52^{o}C for 15 days to deplete the residual biodegradable organic material (degasification).

The batch test was carried out as mentioned by Moset et al. (2015). Biogas composition was analyzed by using gas chromatography (Agilent technologies 7890A, USA) once a week. Methane produced from each sample was corrected by subtracting the volume of methane produced from the inoculum control.

2.3. Lab-scale

The lab-scale experiment was carried out with a biogas test plant system (BTP2-control, UIT, Germany).The biogas test plant system consist 4 reactor units and a central control unit. Each reactor unit is equipped with a 15L CSTR reactor, gas drum, gas bag, heating control unit, agitator system and module SENSOconctol. Biogas composition (CH_4, CO_2, H_2S, O_2) was auto analyzed by a gas analyzing unit (SSM 6000 LT, PRONOVA, Germany). The reactors were manually fed and discharged daily with an amount based on the different hydraulic retention time. The stirring intensity was 100 r/min.

2.3.1 Flexible biogas production

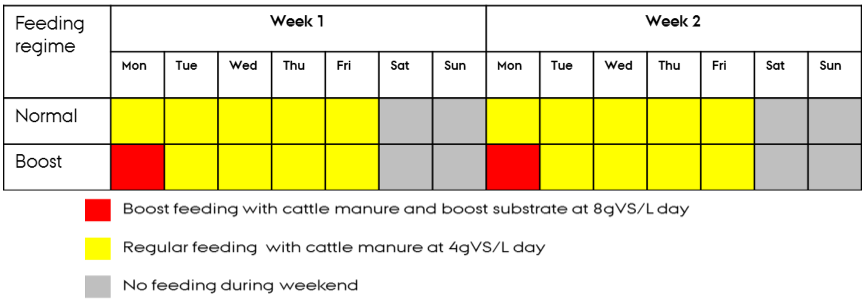


Figure 1. Schematic diagram of flexible biogas production

The flexible biogas test was divided in two periods of two weeks (Figure 1). 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.

2.3.2 Multistage

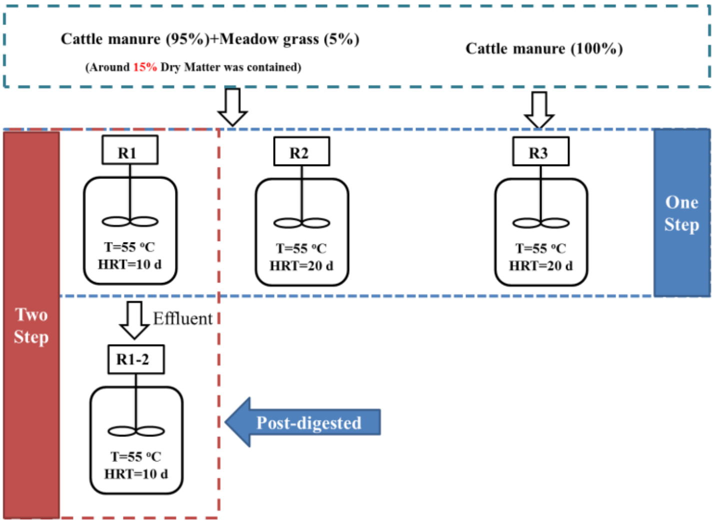


Figure 2.Schematic diagram of multi-stage anaerobic digestion experiments

As shown in Fig.2, four lab-scale CSTRs (15L) were used at 20 days hydraulic retention time (HRT); the first two reactors which configured as two-step (10days HRT for each reactor) was fed with 95% CM and 5% meadow grass; and the rest two reactors with 95% CM and 5% meadow grass (co-digestion & single step) or only with CM (mono-digestion & single step), respectively. The organic loading rate for R1, R2 and R3 was 12.45±0.01, 5.05±0.35 and 6.22±0.01 kg VS m-3 day-1 respectively. All co-digested reactors (R1, R1-2 and R3) were fed with cattle manure for 2 weeks before co-digestion.

During the experiment period, the records of pH value, temperature, biogas flow rate were measured and saved in a database in one minute interval. The biogas yield was measured with a gas drum and be recorded each day. Digestate samples was taken from each reactor 1-2 times a week and analyzed for total solids (TS) content, volatile solids (VS) content, dissolved VFA concentration, ammonia nitrogen concentration.

2.4 Analytical methods

Total solids (TS) content, volatile solids (VS) content were measured according to the standard methods(United States of America, 1985). Dissolved VFA were determined using a gas chromatograph (5560-D of APHA, 2005), equipped with a flame ionization detector (HP 68050 series, Hewlett Packard).Total ammonia nitrogen (TAN) was determined weekly from fresh digestate using photometric kits (Spectroquant kit, Merk, USA).

Samples for fibre analysis were dried (48 h at 60 °C) and milled to a particle size of 0.8 mm using CyclotecTM 1093 mill (FOSS North America). Fibre fractions (neutral detergent fibre (NDF), acid detergent fibre (ADF) and lignin (ADL)) were analyzed according to the Van Soest (1994) procedure.

3. Results and Discussion

3.1. Bio-methane potential

Table 1 given the ultimate bio-methane potential (BMP) from each fresh or dried substrate. The final BMP values from different substrates were ranked as follows: Maize silage > Sugar beet tops+Straw silage > Straw > Sugar beet tops > Meadow grass > Cattle manure. During all these biomass, the highest BMP was obtained from maize silage (338.05±30.86 NL/kg VS). The BMP value obtained from sugar beet tops was only 248.59±19.23NL/Kg VS, which much lower than the value (500 L/kg VS) has been reported (Fang et al., 2011). This may cause by the difference between sampling process, experimental condition and also the possibility to losing liquid part during batch preparation.

Table 1 Chemical composition and ultimate methane yield

Substrates	TS (%)	VS (%)	Ash (%)	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ultimate methane yield (NL/gVS)
Cattle manure [2,3]	12.42±1.2	10.09±0.71	2.33±0.50	23,99	16,57	15,51	216.39±30.48

Meadow grass-Mix [3]	75.36±0.21	68,39±5.35	3.17±0.35	33,17	34,47	6,07	236.64±16.73
Maize silage [2]	34.77±0.46	31.77±1.44	2.99±0.95	17,46	18,71	3,43	338.05 ± 30.86
Sugar beet tops [2]	12.80±0.27	9.95±0.17	2.85±0.10	ND	ND	ND	248.59±19.23
Straw [2]	80.24±1.07	74.28±1.82	6.02±0.82	ND	ND	ND	322.92±1.71
SBT+Straw Silagea [2]	30.11±0.48	26.79±0.47	3.32±0.02	39.89	20.98	7.35	331.24±3.28

1,2,3 The number means which part of experiment the biomass will be used .
a Silage for the mixture of sugar beet tops and straw

3.2. Flexible biogas production

3.2.1. General performance

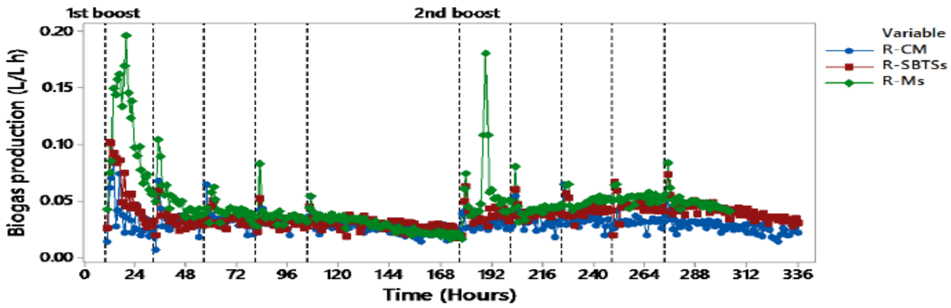


Figure 3.Hourly gas flow rate (Dashed line represent feed events)

During the first week (Figure 3), 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 h) after 4 hours of the first feeding. On the other hand, R-SBTSs and R-Ms presented their respective peaks (0.101 and 0.196 L/L h) after 2 and 10 hours respectively.

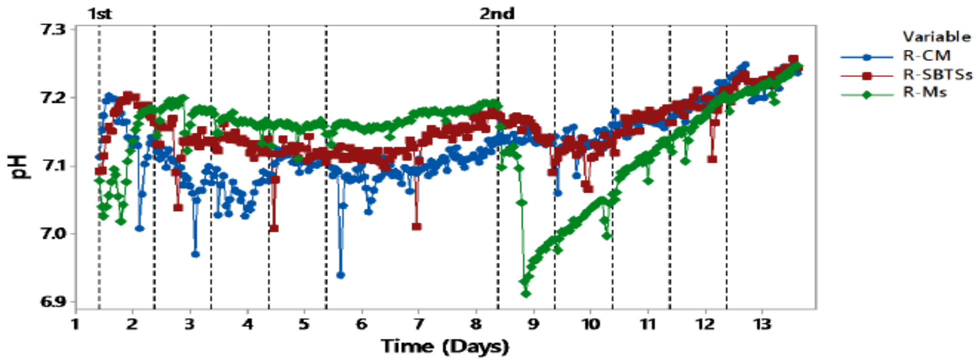


Figure 4.Changing of pH value

General decreasing tendencies were observed coupling with the decrease of CH4 content and the increase of VFA concentration along the first week. However, as cattle manure is a substrate with a high buffer capacity big pH changes could be neutralized. 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 CH4 drop. However the minimum pH (day 9) was reached previous to the maximum accumulation of VFA (day 10). It was caused by the fast conversion of maize silage (a readily degradable substrate) into VFAs thus lowering the pH.

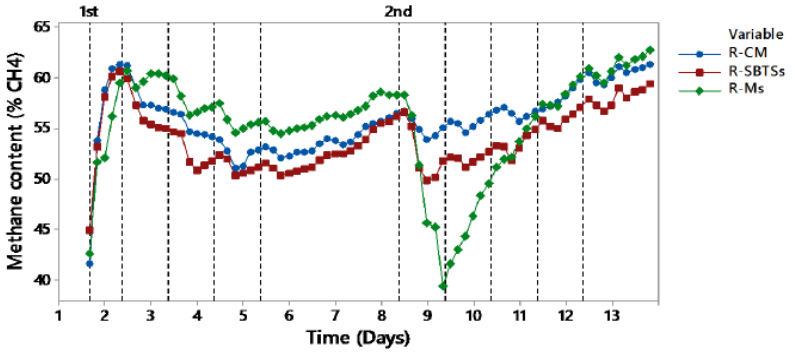


Figure. 5 Changing of methane contents

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 CH4 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.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 CH4 level with an increase on day 11 (due to changes in the feed strategy). On the other hand, high variability was observed in the experimental reactors. After the second boost, the CH4content in R-SBTSs was reduced an 11% in less than one day (from 56 to 50%). However, after reaching that minimum, the CH4 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.

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.

3.2.2. Energetic perspective

An extrapolation of the results obtained in this study, 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.

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 46 together with the two approximations. As can be observed with a boost of 4 kg m^{-3} d^{-1} of maize silage, a maximum of circa 10 Mwh could be produced.

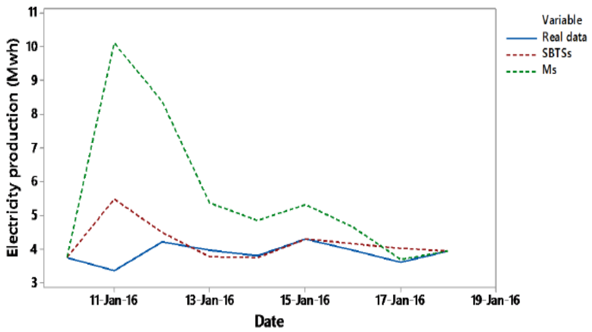
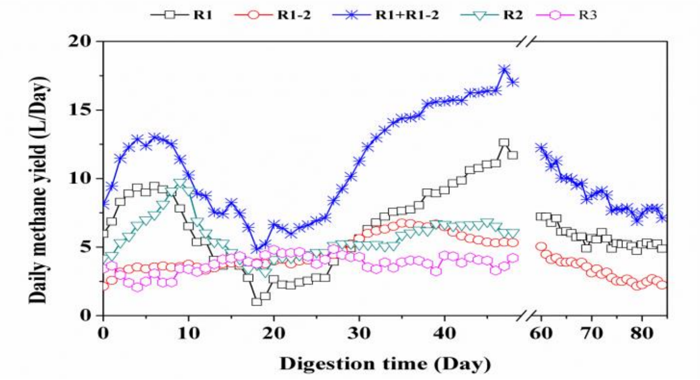
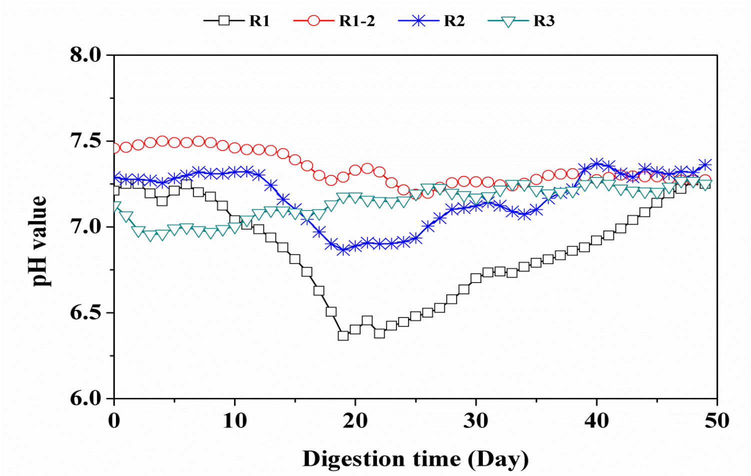


Figure 6. Extrapolation of the increments obtained during the first week. Blue line is the real electricity production from the main reactor in Foulum AU during one week of January 2016. Dashed lines represent the extrapolation, green for R-Ms and red for R-SBTSs.

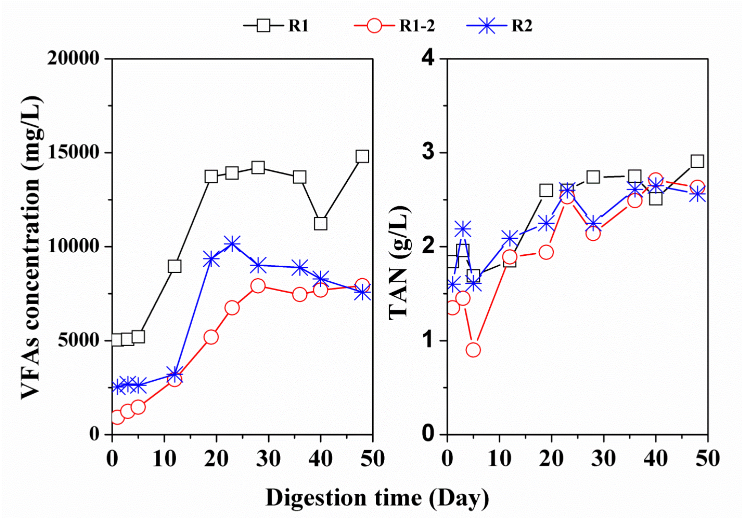
3.3. Multi-stage



(a) Daily methane yield



(b) pH value



(c) TVFAs and TAN concentration,

Figure.7 (a) Daily methane yield, (b) pH value, (c) TVFAs and TAN concentration during the two/single step co-digestion AD process.

The experimental results are shown in Fig.7 and Tab.2. As shown in Fig.2, daily methane yield (DMY) and specific methane yield(SMY) from both two-step and single-step (R1 and R2) increased at the beginning and decreased after 10 days' feeding with mixed substrates. The steady state period from two-/single-step were observed after 30 days' fluctuated process. All average value (DMY, SMY, VFAs etc.) were calculated based on the data acquired after 30 days' anaerobic digestion. The average SMY from two-step was 107.14±12.00 NL/kg VS (as shown in Tab.2), which was 23.96% higher than that of single step co-digestion (86.43±8.94 NL/kg VS). 17.83% increase on SMY can be achieved with 5% grass added when compared with mono-digestion. However, the SMY from all CSTR were lower than the BMP value acquired in batch assay. The highest SMY obtained in this study (95% cattle manure+5% meadow grass, two-step) was corresponding to only 45.3% of BMP value by batch assay. According to Lehtomäki et al. (2007), the SMY with a co-substrates of 70% cattle manure and 30% grass silage (based on weight) was corresponding to 85% of the BMP value determined by batch assay. The undesirable manipulate condition (higher OLR and TS content, shorter HRT,etc) and the lower C/N ratio in the substrates may lead to a lower SMY(Hilkiah Igoni et al., 2008; Søndergaard et al., 2015).Compared with co-digestion reactors, the performance of anaerobic digestion of cattle manure kept stable during the 48 days' experiments because of the reactor 3 was controlled as mono-digestion reactor for cattle manure at different HRTs for previous experiment.

As shown in Fig.7(b), pH values in R1-2 and R3 were stable during the whole period while R1-2 had the highest average pH value than the rest reactors. The same fluctuated trends were observed from R1 and R2. During the first 20 days, the pH value in R1 and R2 decreased from 7.2 to 6.3 (R1) and 6.8 and kept on increasing until the end. The PH value in all reactors was around 7.5 at the end, which corresponding to the steady VFAs level. According to Fig2 (a) and 2 (b), the decrease of biogas yield were accompany with a significantly pH decline.

The total volatile fatty acids (TVFAs) concentration was measured once a week (three times at the first week) in order to evaluate the process stability. As shown in Fig.2 c, the concentrations of TVFAs increased significantly from R1, R1-2 and R2 at the beginning 10-15 days and reached the peak concentration during the 20-30 days. The TVFAs concentration were in general 50% higher in R1 when compared to R1-2 (2nd step) and R2 (single step). The average TVFAs concentration from R1, R1-2 and R2 was 13482.66±1573.97, 7746.35±226.65 and 8443.18±658.61 mg/L, respectively. This higher VFAs level from R1 was mainly caused by the shorter hydraulic retention time (10 days). There is no obvious VFAs inhibition observed during the steady state. However, the possibility of overload in R1 was still higher than the rest three CSTRs.

The TAN concentration changed as well as the VFAs concentration. The average TAN after 30 days' digestion was 2.73 mg/L , 2.49 mg /L and 2.52 mg/L, respectively. According to Varel et al. (1977), the AD process could be inhibited by TAN concentration of above 1.7 g/L on the thermophilic anaerobic digestion of cattle waste at high OLR and pH value above 7.8. However, a severe inhibition was observed at TAN concerta

Table 2 Parameters from two/single step co/mono-digestion process after 30day' AD process

Parameters[a]	Two-step co-digestion			Single step co-digestion	Single step mono-digestion
	1st Step	2nd step	1st+2nd step		
Dailymethane yield (NL/day)	7.99±1. 73	5.35±0.46	13.34±1.49	5.38±0.56	3.43±0.32
Improvement [b] (%)	132.94	55.98	288.92	56.85	0
Improvement [c] (%)	48.51	-0.56	147.96	0	--
Specific methane yield (NL/Kg VS)	64.17±13.92	--	107.14±12.00	86.43±8.94	68.03±6.31
Improvement in SMY[b] (%)	-5.67	--	57.49	27.05	0
Improvement in SMY[c] (%)	-25.75	--	23.96	0	--
pH Value	6.96±0,19	7.28±0.02		7.25±0.1	7.23±0.03
Effluent TS (%)	10.58±0.83	8.12±1.33		9.06±0.68	--
Effluent VS (%)	8.86±0.75	6.56±1.15		7.05±0.61	--
Methane content (%)	47.19±2.01	57.38 ±1.56		50.29±1.57	50.32±4.55
H_2S concentration (ppm)	2238.84±500. 51	891.89±105.27		2423.08±402.99	1643.02±213.90
TVFAs (mg/L)	13482.66±1573.97	7746.35±226.65		8443.18±658.61	8932.45±1000.06
TAN(g/L)	2.73±0.16	2.49±0.25		2.52±0,18	2.69±0.17

[a] All data in this table were calculated as an average value based on the digestion after 30days.
[b] Compared with single step mono-digestion
[c] Compared with single step co-digestion

As shown in Tab.2, the most premium parameters both from the gas and liquid phase were observed from the 2nd step reactor. The methane contents, which represents the gas quality and heating value, from R1-2 was 57.38 ±1.5% in average. It is around 10% higher than 1st step and 7% higher than that from single step co-digestion. And the lowest concentration of H2S was also observed from R1-2, with 891.89 ppm in average, Furthermore, both the lowest effluent TS/VS and TVFAs, which indicates a higher bio-degradability, was acquired with a two-step configuration. The reason of this phenomenon was that the digested effluent from 1st step, which contained all important groups of microorganisms such as acidogens, acetogens and methanogens, and also VFA, nutrients and small amounts of residual soluble organics(Boe & Angelidaki, 2009), were more feasible to be utilized in the 2nd step.

From day 60 to day 85, as shown in Fig.7(a) and (c), the reactor R1 and R1-2 were changed to fed with cattle manure instead of mixed substrates. The data from day 75 to 85 were used as 'steady state' for average DMY and SMY calculation. The average DMY and SMY from two-step mono-digestion was 7.58±0.32 L/day and 75.16±3.17 NL/Kg VS, which corresponding to 120.99% and 10.48% improvement on DMY and SMY when compared with single step AD of cattle manure. The results obtained in this part was almost the same (11% higher of biogas yield) with Boe and Angelidaki (2009) when used two-stage AD of cattle manure and 13% lower than that of in the co-digestion experiment. It is obviously that relative percentage of SMY from two-step co-digestion and mono-digestion was different. The methane produced from the R1-2 (2nd step) alone constitute 40.1% and 33.59% of the total methane yield, in co-/mono-digestion, respectively. The 2nd reactors, or the last 10 days' retention time, provide more methane production yield with 5% grass added, which lead to a better performance during two-step process. In general, the performance of a two- step AD-process, such as specific methane yield, can be influenced by the initial substrates itself. A longer period of retention time may necessary when co-fed with higher lignocellulosic biomass which was difficult to be hydrolyzed completely fast.

4. Conclusion

With boost feeding regime and 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 experiment 1 and transient Inhibition was observed in experiment 2 after the second boost in R-Ms.

The application of co-digestion and two-step AD could improve methane yield when co-digested cattle manure with meadow grass. A two-step AD with total HRT of 20 days and 5% grass added could obtained specific methane yield 23.96% higher than a single step co-digestion and 57.49% higher than anaerobic digestion of cattle manure. The general performance of two-step AD can be affected by the initial substrate.

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