Pre-treatment of Fiber-rich Biomasses for Biogas Production

Forbehandling af fiberrige biomasser til biogasproduktion

Project No.:	EUDP j.nr. 64011-0335 /Internal project 10842
Made by:	Edited by APJE + SBG + FR
Issued:	December 3 2013

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1 Summary and Conclusions

During the period from January 2012 to May 2013 Xergi A/S has carried out a EUDP-supported research and development project with the aim of improving the applicability of fibre-rich biomasses for biogas production.

Special focus of the project was documentation of Xergi's patented pre-treatment technology, NiX®. The technology is based on pressure cooking with base addition in order to achieve improved degradability and/or removal of nitrogen, which can be inhibitory to the biogas process.

The specific objectives of the project were to (with reference to EUDP work packages at the end):

1. Investigate the effect of Xergi's patented NiX® pre-treatment method on the biological methane potential of various agricultural residues including fibre-rich and nitrogen-rich biomasses (WP 2).

2. Compare this effect of the NiX® method with various mechanical pre-treatments of fibre-rich biomasses (WP 3 & 4).

3. Improve the efficiency of nitrogen removal from nitrogen-rich biomasses by the NiX® pre-treatment (WP 5).

4. Screen various types of cattle deep litter for key parameters (dry matter, nitrogen, biological methane potential) (WP 6).

5. Conduct long-term, continuous "proof of concept" pilot-scale testing of pre-treated biomasses (WP 7).

6. Design a full-scale biogas system with NiX® pre-treatment for a full-scale demonstration of the technology based on both fibre- and nitrogen-rich biomasses and plan the commercialisation of the NiX® technology (WP 8).

The results obtained and conclusions made during the project are summarised in this report with more detailed reporting in the following annexes to the report:

Annex 1: Pre-treatments of Agricultural Residues (WP 2+3+4)
Annex 2: Mineralisation of Organically Bound Nitrogen (WP 5)
Annex 3: Screening and BMP Analysis of Cattle Deep Litter (WP 6)
Annex 4: CSTR Experiments on Chicken Litter (WP 7.1+7.3+7.4)
Annex 5: CSTR Experiments on Straw (WP 7.2)
Annex 6: Design of Demonstration Plant (WP 8)

Overall conclusions of the project:

1. A very high nitrogen removal has been demonstrated through a new pre-mineralisation step for the NIX process on poultry manure. This allows for a higher relative amount of poultry manure as substrate in AD plants.

2. A patent PCT-application for the pre-mineralisation process has been filed.

3. A new and simple pH-adjustment process has been introduced in the NiX process and a full documentation has been produced.

4. A patent application for the pH-adjustment process has been filed.

5. The NIX concept including the 2 new inventions has been demonstrated in a stable CSTR trial run for now up to 8 months with chicken litter as a mono-substrate for the AD process. The trial run will continue after the EUDP project to secure a full proof of concept.

6. The engineering part of the project in combination with the process test has given Xergi the basis for selling and implementing the first3 MW full scale NIX project based on 45 % poultry manure in UK.

7. A new pressure cooker construction has been designed through the project. This design will reduce the price/capacity ratio with at least 25 % and reduce the high electricity consumption with 60 %. The design has formed the basis for a patent application.

8. From the market response from investors, Xergi has concluded that a full-scale demonstration project for both the new batch cooker and the concept for operation with 100 % poultry manure is a pre-requisite for selling full-scale plants. Xergi and a project group has applied EUDP to support this phase.

9. The project focus on treatment of deep litter from cattle shows now extra gas yield with mechanical pre-treatment where a nix pre-treatment shows an extra gas yield of 23 to 38 %. But mechanical treatment can solve the problem with floating layer in the digesters.

10. The project focus on pre-treatment of straw has showed that different pre-treatment systems can solve the problem with floating layer and extra gas yield can be obtained in the range from 15 to 35 %. But the treatment cost seems to be too high for most technologies.

11. Xergi has chosen to develop their own pre-treatment system for handling cattle deep litter, straw, grass etc. in a simple and cheap way to handle the problem with floating layers and the high content of stones in the deep litter. The technology, named X-chopper, has been produced in a full-scale prototype and successfully tested.

12. The general conclusion for handling deep litter, straw, grass etc. is to do a cheap pre-treatment with the X-chopper to deal with the floating layer problems and then get the extra gas yield by a NiX treatment on separated and recirculated fibres from the backend of the biogas plant.

2 Pre-treatments of agricultural residues (WP 2+3+4)

The effect of the NiX treatment, grinding, hammer-milling and extrusion on the methane yield of various biomasses was investigated using a standard batch test method for methane potential (see figure 1, below).

Figure 1. Batch test bottles in climate chamber for determination of biological methane potential.

In table 1, below, the results of these batch tests are shown as well as a calculation of the predicted yields in a 2-step continuous CSTR system[1] based on the kinetic parameters determined from the batch test.

[1] Primary digester at 52 °C with 15 days HRT followed by secondary digester at 37 °C with 15 days HRT.

Biomass & pre-treatment	Final methane yield obtained in batch test (Nml CH4/gVS)	Increase of final yield in batch BMP test compared to untreated	Increase of yield in two stage CSTR compared to un-treated
Wheat straw			
Straw 1			
Untreated	269		
Grinded – Fine	324	20%	36%
Grinded – Medium	291	8%	20%
Grinded – Coarse	287	7%	18%
NiX	299	11%	26%
Straw 2			
Untreated	263		
Hammermilled	273	4%	14%
Hammermilled and pelletized	274	4%	15%
Degassed fibres from AD plants			
Fibre 1 (maize silage + cattle slurry)			
Untreated	150		
NiX (low TS)	238	59%	83%
NiX (high TS)	200	33%	45%
Fibre 2 (maize silage only)			
Untreated	142		
NiX (low TS)	236	66%	98%
NiX (high TS)	174	23%	47%
Cattle deep litter			
Bull calves (3 samples)			
Untreated (reference to extrusion)	258-288		
Extrusion	260-281	0%	0%
Untreated (reference to NiX)	232-274		
NiX	272-341	17-25%	30%-38%
Dairy cows (3 samples)			
Untreated	225-313		
NiX	**291-340**	**5-30%**	**23%-37%**
Poultry litter			
Egg layers			
Untreated	293		
NiX	292	0%	0%
Broilers (meat chicken)			
Untreated	300		
NiX	~300[2]	0%	0%

Table 1. Summary of obtained methane yields from untreated and treated biomasses.

2.1 NiX (WP 2)

The NiX pre-treatment resulted in a significant increase in final gas yield for all fibre-rich biomasses tested while no effect was found on poultry litter. The positive effect was smallest with wheat straw (11%) and highest with degassed fibres (59-66% on low TS treatments) while the effect on cattle deep litter was intermediate but more variable (5-30 %).

These results indicate that a low initial degradability (in this case expressed as low gas yield in the untreated biomass) means a high improvement potential. Degassed fibres from AD plants have already undergone a long degradation process where most of the readily accessible components have been converted to biogas. Cattle deep litter is a mix of straw bedding, cattle slurry and feed residues, and will therefore contain straw at different levels of decomposition, while straw represents plant fibres in a raw form, and therefore contains the full (however limited) amount of easily degradable components. The lacking effect on poultry litter can be explained by the fact that bed-

[2] Samples from various steps in the NiX-process showed some variation around 300.

ding material made up less than 10 % of the dry matter and mainly consisted of wood shavings, which normally are considered non-degradable under anaerobic conditions.

The calculated yield in a CSTR process shows even greater effects of the NiX treatment due to increases in the rate of degradation making the pre-treatment effect more profound during the first part of the anaerobic digestion. Still, the pattern with higher improvements the lower the initial yield is maintained. A remarkably high improvement was obtained from degassed fibres where the yield from Fibre 2 was nearly doubled and increased by more than 80 % from Fibre 1 (both in low TS NiX pre-treatments).

2.2 Mechanical pre-treatments (WP 3 & 4)

The mechanical pre-treatments were applied to wheat straw (grinding and hammermilling) and bull cattle deep litter (extrusion). Only grinding of wheat straw showed any significant effect on the final gas yield and only in the most energy consuming operational mode where a 20 % improvement was obtained. In comparison NiX treatment of the same substrate yielded 11 % improvement in methane production.

However, our test results show that the energy balance of grinding is not favourable, and even at the coarse grinds the energy consumption for this treatment outweighs the energy content of the additional methane yield.

Mechanical pre-treatment had a positive effect on the rate of gas production as can be seen from the higher improvement levels calculated for the CSTR process. Still, the rate improvements were smaller than the ones obtained from the NiX pre-treatment on the same biomasses with the exception of the finely grinded straw.

2.3 Conclusions on pre-treatments

NiX pre-treatment (see figure 3, below) resulted in significant increases in the final gas yield from all eight fibre-rich biomasses tested except one out of three samples of dairy cow deep litter. The relative improvements in gas yield are positively correlated to the content of heavily degradable components in the biomass. In the most fibre-rich biomass samples (degassed fibres from AD plants) the final gas yield was improved on average by 63 %. The gas yield from poultry litter was not improved by the NiX pre-treatment.

The pattern of relative improvements of the calculated CSTR-yield was the same as for the final yields and due to rate increases the improvement levels were higher and reaching nearly 100 % for the most fibre-rich sample (degassed fibre from AD plant).

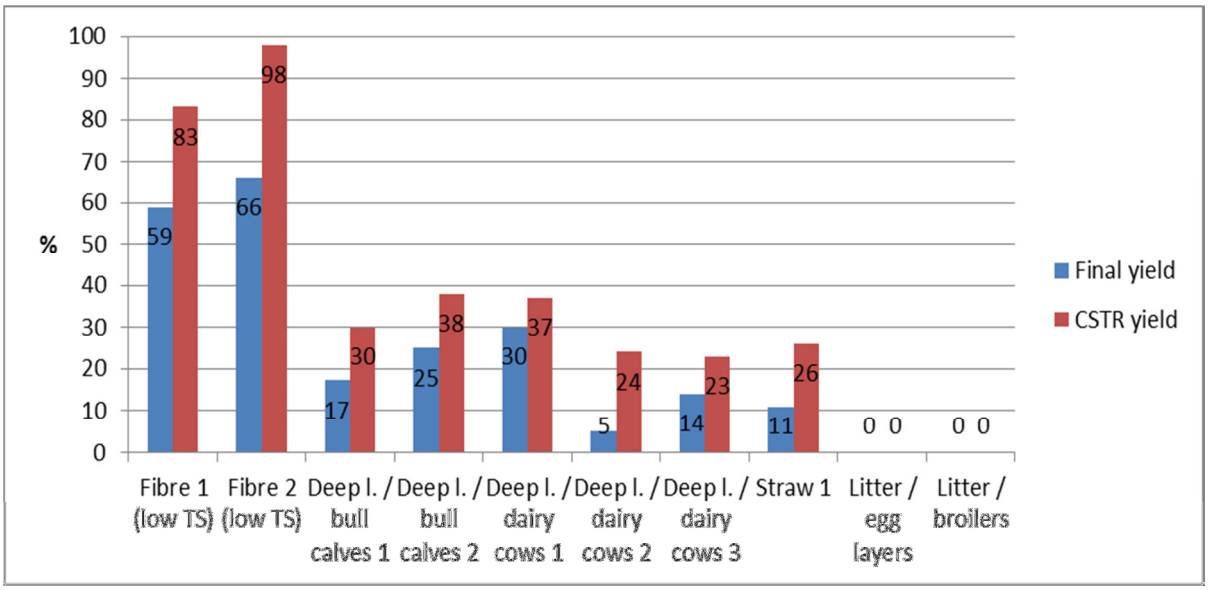

Figure 3. Summary of gas yield improvements obtained by the NiX pre-treatment.

The energy balance of the NiX treatment is discussed in Annex 6.

Significant improvements of the final gas yield could not be achieved using mechanical pre-treatment (see figure 4, below) of straw and cattle deep litter, except when using the finest grind setting in a special grinding technology, which consumes more electrical power than the energy content of the yield increase.

Rate increases were obtained by the mechanical pre-treatments but at levels below the increases obtained by the NiX pre-treatment.

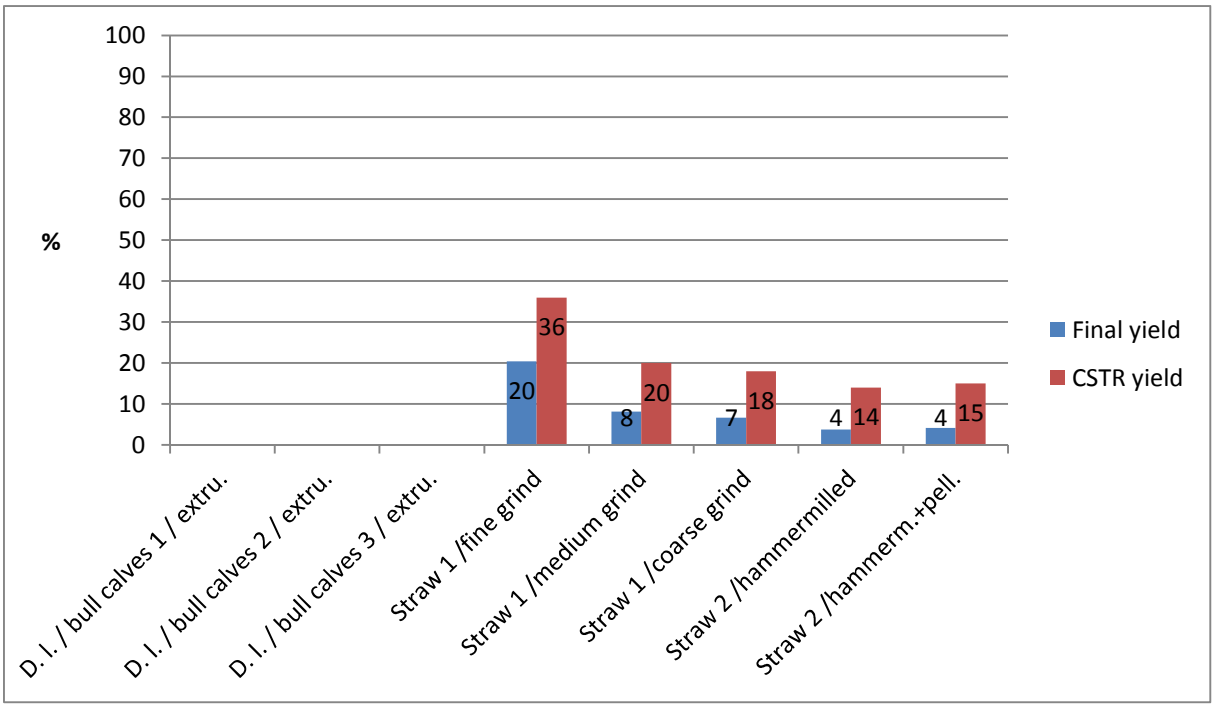

Figure 4. Summary of gas yield improvements obtained by the mechanical pre-treatments.

3 Improvement of method for nitrogen removal during NiX pre-treatment (WP 5)

As reported in section 1.4.1 of this report, the pilot scale experiment on NiX pre-treated chicken litter underwent some method improvements related to the nitrogen removal and subsequent pH effect in the digester.

The first method improvement focused on degrading the organic nitrogen pool of the chicken litter to ammonium-N as removal during the NiX treatment requires nitrogen to be on this form. The effect of NiX treatment of chicken litter is therefore limited, since typically only around 20 % of the nitrogen pool is present as ammonium with the remaining nitrogen bound in proteins and uric acid.

This was achieved by a biological method causing 70 – 80 % of the organic nitrogen to be mineralised to ammonium. Further method optimisations have indicated that the efficiency of the treatment could reach almost 100 %. The method works equally well on hen and chicken litter.

Loss of organic dry matter during the mineralisation process has after final optimisation reached a negligible level after correction for the intended loss of organic nitrogen compounds.

Patent applications have been filed for the improved NiX treatment comprising the mineralisation of organic nitrogen.

4 Screening of cattle deep litter (WP 6)

Cattle deep litter is not commonly used as a major biomass input for biogas production. This is due to difficult handling and low degradability. The definition “cattle deep litter” covers a wide array of straw based solid manure from cows.

In order to get an overview of the variability within this biomass category, a total of 6 cattle deep litter samples were analyzed for dry matter and nitrogen content. In addition, as summarized in section 1.1 above, the effect of pre-treatments on the methane yield was investigated.

The analysis of dry matter and nitrogen content showed a lot of variation that can be attributed to differences in bedding material, feeding regime, storage conditions and age of the animals. The screening served as a first assessment of the variability of cattle deep litter, which will be more thoroughly and extensively investigated in the coming EUDP-supported project (j.nr. 64013-0167).

Figure 5. Cattle deep litter in stack.

5 Pilot-scale testing of pre-treated biomasses (WP 7)

Testing of the feasibility of using pre-treated chicken litter and straw, respectively, as the only or dominating biomass for biogas production was carried out in pilot-scale plants in continuous operation (see figure 6, below).

Figure 6. Pilot-scale CSTR anaerobic digestion plant for continuous testing.

5.1 NiX pre-treated chicken litter

Chicken litter is a high-solid and high-nitrogen biomass which cannot be used as mono-substrate for biogas production without pre-treatment unless extensive dilution of the dry matter and nitrogen levels is carried out.

A pilot-scale biogas plant (200 L mesophilic single step) was operated for more than 18 months on daily feedings of NiX pre-treated chicken litter mixed with recirculated liquid from separation of the effluent stream from the biogas plant.

Figure 7. Pilot-scale NiX cooker for biomass pre-treatment.

The objective was to determine the optimal process conditions for a high and stable gas yield with focus on minimizing the need for external water addition. NiX pre-treatment was therefore applied

in order to remove the major part of the inhibitory ammonium nitrogen, while recirculation of separated digester liquid ensured that external water input for a obtaining a pumpable mixture could be kept at a minimum.

The first test phase resulted in levels of free ammonia nitrogen above critical inhibitory levels, due to release of ammonium from organic nitrogen compounds during the biogas process. In chicken litter organic nitrogen typically makes up more than 70 % of the total nitrogen, and organic nitrogen is not available for removal during the NiX treatment where only volatile nitrogen (free ammonia, NH3) can be removed. This led to strongly fluctuating methane yields and accumulation of process intermediates.

The second test phase comprised an improved nitrogen removal process, where 70 - 80 % of the organic nitrogen is broken down to ammonium by a biological process before the NiX treatment. However, process stability was still not achieved as pH in the digester increased rapidly and soon caused the pH dependent free ammonia to cross the threshold level for potential inhibition.

It was therefore decided to abort the test and look for ways of controlling pH in the digester.

A solution to this problem was found by an innovative application of CO2 in the produced biogas in a step between NiX-treatment and anaerobic digestion.

The third and last test phase demonstrated stable process conditions with free ammonia concentrations below the inhibitory level, low VFA's and a specific methane yield (250 L CH4 per kg VS added) above the expected based on the obtainable yield determined in the labs tests performed as part of WP 2 (see figure 8, below).

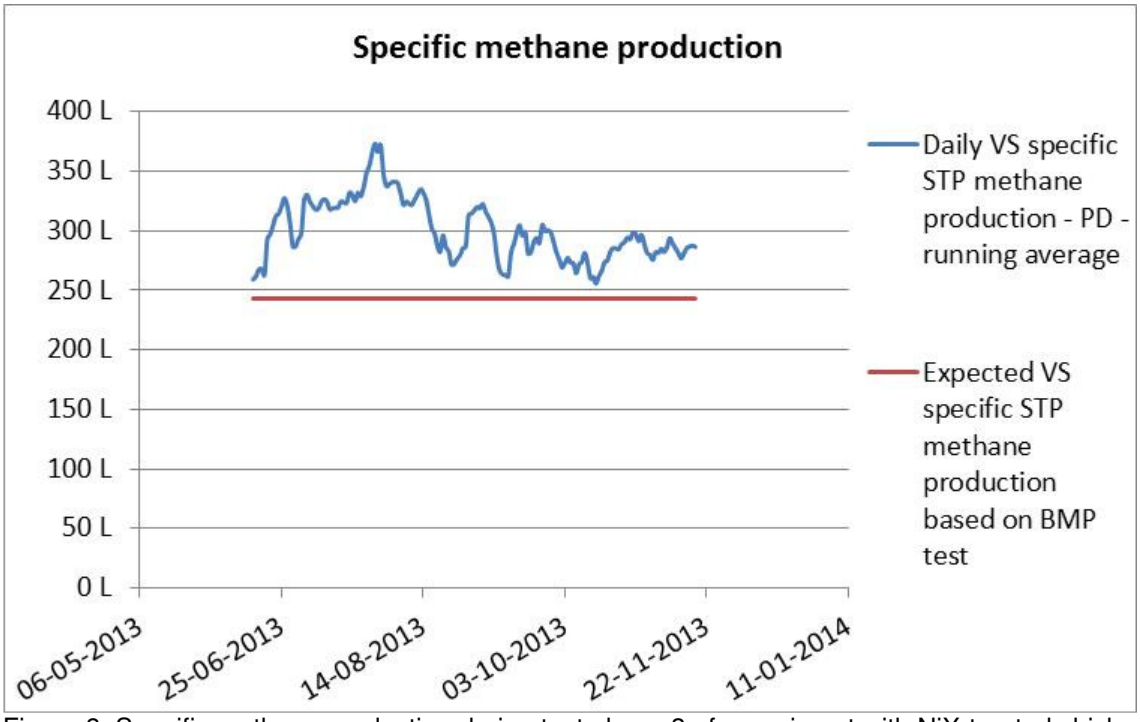

Figure 8. Specific methane production during test phase 3 of experiment with NiX-treated chicken litter.

For both the abovementioned improvements to the NiX method patent applications have been filed.

The water addition required for stable operation was determined to be at a ratio of 0.6 – 0.9 relative to the chicken litter input. The indicated range in water consumption is a function of the pH in the digester and since the newly developed method for pH control seems very effective, the low water consumption ratio of 0.6 is expected to be feasible.

This limited addition of external water during the biogas process is of great importance to the competitiveness of the concept as the cost of back-end treatment and disposal are substantial.

5.2 Grinded wheat straw

The suitability of grinded wheat straw as a dominating substrate for biogas production was investigated in a pilot scale set-up similar to the one described in the previous section on chicken litter although without NiX pre-treatment. The purpose of the experiment was to investigate the stability of the biological process and identify possible technical issues in relation to pumping and mixing.

The level of grinding of the wheat straw was the same as in the fine grind quality of the wheat straw tested in the previous section 1.1. This means a grinding level requiring a power input higher than the extra energy potential generated by the increased gas production. Still, the fine grinding was chosen as it was expected to minimize the risk of blocking and clogging.

A mixture of pig slurry with an increasing additional input of grinded wheat straw was used as biomass input to the pilot-scale biogas plant.

During stage 1, a baseline biogas production from pig manure was successfully established. However, shortly after introduction of the diluted mixture of wheat straw and pig manure, the pump associated with feeding into the anaerobic digester experienced problems due to the viscosity of the mixture, and had to be replaced with a pump with a higher capacity. Although this solution alleviated the problem during stage 2, the final mixture of wheat straw powder and pig manure was so viscous, that is was not possible to go through with stage 3 without substantial modifications to the pilot plant.

It was therefore not possible to determine the biological stability of a process in which the main biogas production comes from wheat straw.

6 Design of full-scale biogas system with NiX pre-treatment (WP 8)

During the engineering part of the project the following results has been reached and documented:

1. Detailed mass balance, energy balance and nitrogen balance has been calculated for different combination of feedstocks and plant size as well for a demonstration plant in Ribe.

2. Optimization of heat/steam consumption has been performed in the project.

3. In connection with item 1 a general NIX calculation tool has been developed, and is today in use for calculation of new NiX projects in Xergi.

4. General plant layout (1,5 MW, 3 MW, 6 MW) as well as specific Ribe biogas plant concept and site layout has been made as well as building layouts and optimizing activities has been made.

5. PI diagrams, corresponding component list and functional description have been made.

6. For the key components lime dosing, nitrogen flash and ammonium absorption intensive design activity and evaluation of different potential sub suppliers has been carried out.

7. Different straw handling technologies has been evaluated including capacity test and efficiency test done at a supplier in Germany. The feasibility of the evaluated technologies was too bad to continue with these technologies. Instead Xergi decided to develop its own technology, named X-chopper.

8. The new X-chopper has been made as a full scale prototype and installed at Vester Hjermitslev Biogas plant. It has shown a god operational performance and the first commercial version has been sold to a Danish biogas plant.

9. An intensive development work has been made to design a new batch cooker, dedicated to biogas plant. The new cooker will reduce the heavy power consumption to only 40 % of the original and at the same time the price per capacity (DKK/tons pr. day treated) will be reduced by 25 %. Xergi has filed a patent application for this new design.

10. The activities done in the project have formed the basis for Xergi to obtain a supply contract for a 3 MW plant in UK using the Nix technology as well as a design contract for a 3 MW plant to Northern Ireland operating purely on chicken litter.

11. Xergi´s market evaluation of tariffs for biogas production and biomass situation has led to the conclusion our focus for the poultry litter concept (Poultry Power®) will be UK where Xergi already is present and Italy, which is a new Country for Xergi to handle. For the Manu Power® concept focused on cattle deep litter and fibers from the back end of biogas plant, we need a commercializing of the new batch cooker, after which Denmark will be the main market in the first round and probably followed by France.

12. Xergi has identified the need for a demonstration plant for testing the new batch cooker in a full scale prototype version before it can be implemented in bigger commercial plants. Xergi and its project partners have applied EUDP for a grant to do this testing with special focus on deep litter.

Annex 1 Pre-treatments of Agricultural Residues (WP 2+3+4)

Project No.:	EUDP j.nr. 64011-0335 /Internal project 10842
Made by:	SBG + APJE
Issued:	December 3 2013

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1 Abbreviations

AD	-	Anaerobic digestion
CSTR	-	Continuously stirred tank reactor
HRT	-	Hydraulic retention time
OLR	-	Organic loading rate
OrgN	-	Organic Nitrogen
RSD	-	Relative standard deviation
SD	-	Standard deviation
SOP	-	Standard Operating Procedure
TAN	-	Total Ammonium Nitrogen
TS	-	Total solids (dry matter)
VFA	-	Volatile fatty acids
w/w%	-	Weight percentage

2 Introduction and summary

The effect of NiX treatment, hammer-milling and extrusion on the methane potential of various biomasses was investigated using a standard batch test method for methane potential (as described in Appendix 5.1). The batch method was validated by an independent GTS institute[1] (see Appendix 5.2), and the obtained batch results are converted to predicted yields in a 2-step continuous CSTR system.

The tested biomasses were:

1) Wheat straw (hammermilling and/or NiX)
2) Degassed fibre fraction from biogas plants based on energy crops (NiX)
3) Cattle deep litter (NiX or extrusion)
4) Hen litter from egg layers (NiX)
5) Chicken litter from broilers (NiX)

For biomass 2) the effect of different TS levels during NiX treatment was investigated. For biomass 3) the specific effect of the element of lime addition to the NiX treatment was investigated. The results of the tests are briefly reviewed in this report. Details can be found in internal reports.

In summary the following effects on methane yield was obtained by the pre-treatments:

Biomass	**Treatment**	**Effect on two stage CSTR digestion yield when compared to untreated**
Wheat straw	Grinded – Fine	36%
	Grinded – Medium	20%
	Grinded – Coarse	18%
	NiX	26%
	Hammermilling	14%
	Hammermilling and pelleting	15%
Degassed fibre fraction from mono-substrate energy crop plant	NiX (low TS content)	83%
	NiX (high TS content)	45%
Degassed fibre fraction from energy crop plant	NiX (low TS content)	98%
	NiX (high TS content)	47%
Cattle deep litter from bull calves	Extrusion	0%
	NiX	30%-38%
Cattle deep litter from dairy cows	NiX	23%-37%
Hen litter from egg layers	NiX	0%
Chicken litter from broilers	NiX	0%

[1] Advanced technology providers (GTS institutes) are officially certified by the Danish government to maintain and develop the technological infrastructure in Denmark (http://www.teknologiportalen.dk/en)

3 Overview of results

3.1 Wheat straw

The following section summarizes the results of the experiments and analyses performed on wheat straw.

The purpose of the investigations was to

- Determine the biomethane potential (BMP) value of untreated wheat straw
- Determine the effect of the Jäckering grinding technology on the BMP value of wheat straw
- Determine the effect of NiX pretreatment on the BMP value of wheat straw
- Determine the effect of hammermilling and pelleting on the BMP value of wheat straw.

Key results from the experiments are shown in Table 1 and Table 2, and the main conclusions are listed below each table. The consequences of the treatments on methane production in a CSTR setup are shown in Figure 1 and Figure 2.

Grindings and hammermilling experiments were performed at remote test facilities. NiX treatments were performed at Xergi's own test facility. The BMP analysis of the treated wheat straw was carried out in two separate setups. However, due to technical problems the first BMP analysis could only be used to determine relative improvements from the NiX treatment..

	Units	Wheat straw	Nix treated wheat straw	Grinded wheat straw – Fine	Grinded wheat straw – Medium	Grinded wheat straw – Coarse
BMP	NmL CH_4/g VS *NmL CH_4/g wet weight*	269 (±3) *235 (±3)*	+11%[2]	324 (±6) *298 (±5)*	291 (±4) *268 (±4)*	287 (±4) *264 (±4)*
TS	w/w %	89% (± 0,5)		95% (± 0,1)	95% (± 0,1)	95% (± 0,1)
VS	w/w %	87% (± 0,7)		92% (± 0,1)	92% (± 0,1)	92% (± 0,1)

Table 1 – Overview of key results on NiX treated and grinded wheat straw.

- NiX treatment of wheat straw resulted in an 11% increase in final batch BMP, and a 60% increase in digestion speed[3].
- Grinding caused an increase of 20% in batch BMP and 56% in digestion speed in the fine grind version. The effects on the medium and coarse grinds were 8% on batch BMP, and 44% on digestion speed.

[2] From the first BMP analysis (see **Fejl! Henvisningskilde ikke fundet.**, **Fejl! Henvisningskilde ikke fundet.**)
[3] Digestion speed is a term used to describe how fast a reaction reaches completion.

- Grinding causes a small rise in TS/VS, likely due to heating of the product during processing.

	Units	Wheat straw	Wheat straw Hammermill	Wheat straw – Hammermill - Pelleted
BMP	NmL CH_4/g VS *NmL CH_4/g wet weight*	263 (±4) *224 (±3)*	273 (±5) *238 (±4)*	274 (±3) *236 (±3)*
TS	w/w %	88% (± 0,5)	89% (± 0,1)	89% (± 0,1)
VS	w/w %	85% (± 0,7)	87% (± 0,1)	86% (± 0,1)

Table 2 – Overview of key results on hammermilled wheat straw.

- Hammermilling and pelleting of wheat straw resulted in a 4% increase in final batch BMP, and a 30% increase in digestion speed.

The expected methane yield, when digesting the treated and untreated samples in a two-stage CSTR setup, may be seen below. The calculation combines the digestion speeds and BMP obtained in batch with the setup of the CSTR system to predict the methane production.

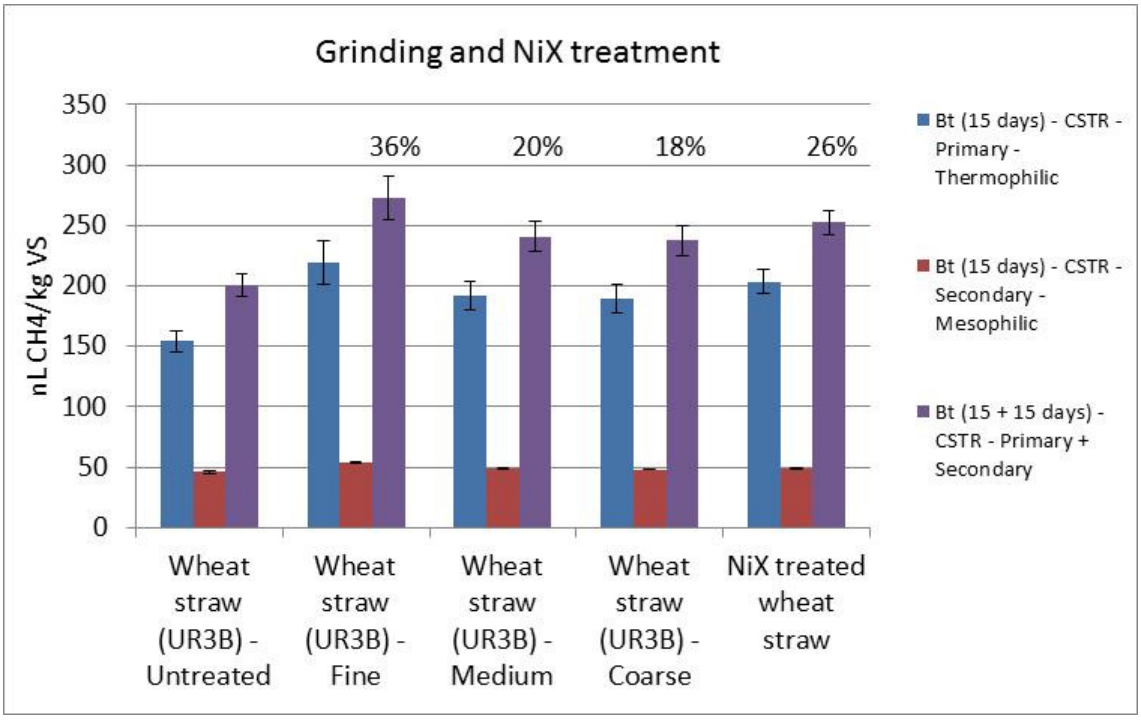


Figure 1 – Expected methane yields from NiX treatment and grinding in a CSTR setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

Comparison of untreated and NiX treated wheat straw in a CSTR setup shows NiX treatment results in a 26% improvement in methane yield. In comparison, grinding yields improvements ranging from 18%-36% depending on the particle size of the grind.

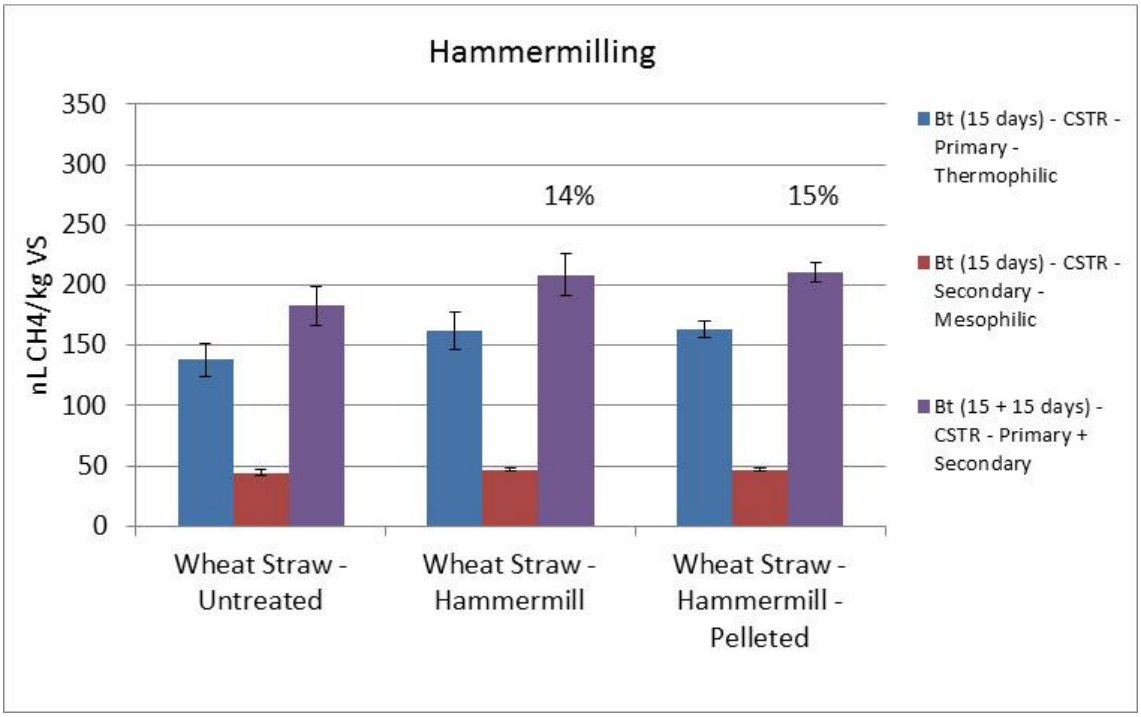


Figure 2 – Expected methane yields from hammermilling and pelleting in a CSTR setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

Comparison of untreated and hammermilled wheat straw in a CSTR setup shows hammermilling results in a 14% improvement in methane yield. There is no additive effect of pelleting subsequent to hammermilling.

3.2 Degassed fibre fraction from biogas plants based on energy crops

The following section summarizes the results of the experiments and analyses performed on degassed fibre.

The experiments were carried out on two types of degassed fibre fraction. The first fibre (Fibre 1) was from an AD plant processing maize silage and cattle slurry, while the second fibre (Fibre 2) was from an AD plant processing maize silage only.

The purpose of the investigations was to

- Determine the biomethane potential (BMP) value of both fibre types
- Determine the effect of NiX pre-treatment on the BMP value of both fibre types
- Determine the influence of the TS level in the NiX treated material on the effect of NiX treatment.

Key results from the experiments are shown in Table 3 and the main conclusions are listed below the table. The consequence of the treatments on methane production in a CSTR setup is shown in Figure 3.

Parameter	Units	Fibre 1	Fibre 1 – NiX (low TS)	Fibre 1 – NiX (high TS)	Fibre 2	_Fibre 2 - NiX (low TS)	Fibre 2 - NiX (high TS)
BMP	Nml CH_4/g VS *Nml CH_4/g wet weight*	150 (± 6) *24*	238 (± 5) *21*	200 (± 4) *32*	142 (± 7) *29*	236 (± 5) *21*	174 (± 5) *35*
TS	w/w %	18.4	10.1	18.4	23.0	10.2	23.0
VS	w/w %	15.8	8.6	15.8	20.2	8.8	20.2

Table 3 – Overview of key results on degassed fibres from energy crop plants.

The conclusions from the experiments are:

- The two fibres show similar BMP profiles, with fibre 1 (maize and other materials) having a slightly higher BMP value than fibre 2 (maize only).
- NiX treatment improves the BMP of both types of fibres.
- The TS level of the treated samples is a significant factor. Improvements range from 23-33% for the high TS treatments, and 59-66% for the low TS treatments.

The high TS NiX treatments were performed with only the addition of burnt lime (i.e. no added water), and were thus performed at the inherent TS level of the substrate. The TS of the 'high TS' treatments thus vary between the two fibres with fibre 2 having a higher inherent TS level than fibre 1. In accordance with the observation that low TS NiX treatments lead to a higher BMP improvement, 'Fibre 1 – NiX (high TS)' shows a higher improvement by NiX treatment than 'Fibre 2 NiX (high TS)'.

The expected methane yield, when digesting the treated and untreated samples in a two-stage CSTR setup, may be seen below. The calculation combines the digestion speeds and BMP obtained in batch with the setup of the CSTR system to predict the methane production.

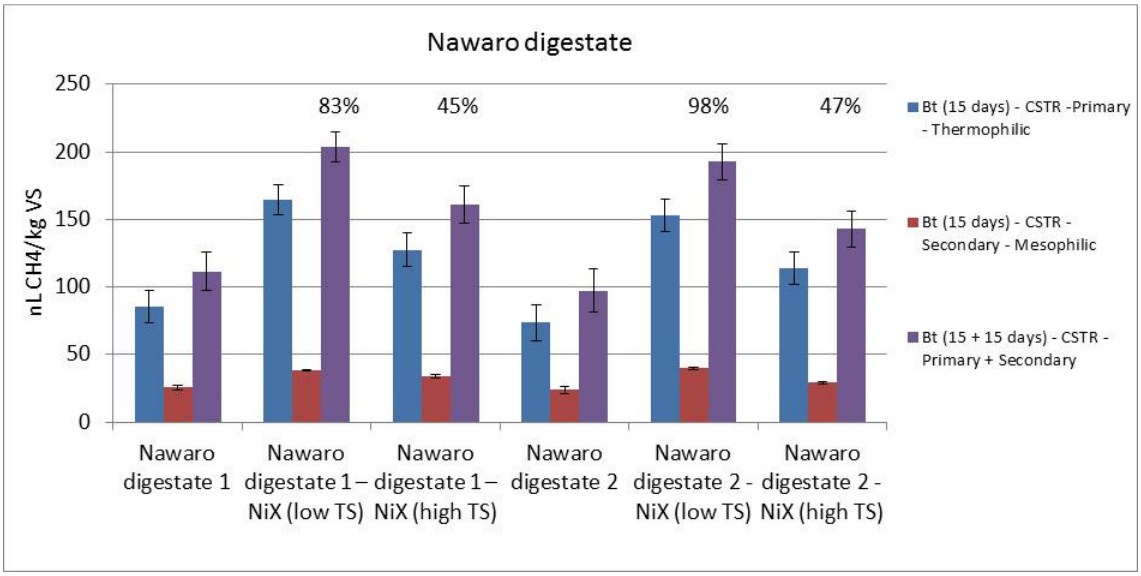


Figure 3 – Expected methane yields of deep litter from degassed fibres in a CSTR setup based on results from the BMP setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

Comparison of untreated and NiX treated degassed fibres straw in a CSTR setup shows NiX treatment results in a 83% - 98% improvement in methane yield in the samples with low TS content. In comparison high TS levels yields improvements of 45%-47%.

3.3 Cow deep litter

The following section summarizes the results of the experiments and analyses performed on deep litter from cows.

The purpose of the investigations was to

- Determine the biomethane potential (BMP) value of
    a. Deep litter from bull calves[4]
    b. A mixture of deep litter from dairy farms**Fejl! Bogmærke er ikke defineret.**
- Determine the effect of extrusion on the BMP value of litter from bull calves

[4] Bull calves are from 0-12 months of age and bred for slaughter. The dairy farms produce deep litter from calves (0-5 months of age), dry cows (dairy cows that have stopped lactating) and/or heifers.

- Determine the effect of NiX pretreatment on the BMP value of bull calves and dry cows/calves.
- Evaluate the variation in dry matter and nitrogen content within and between different types of cattle deep litter

Deep litter was obtained from three different suppliers of bull calves, and three different dairy farmers. The inherent heterogeneity of deep litter required that all deep litter samples were homogenised using a Biomixer[5] prior to sampling smaller portions used for each experiment. The results below are a result of three different BMP assays. Due to natural variance between assays the effects of the tested treatments should always be compared to the accompanying untreated sample. The main results from the experiments are summarized in Table 4 and Table 5.

	BMP setup	Units	Laursen	Hindbo	Korsholm
Deep litter type		NmL CH_4/g VS	Bull calves	Bull calves	Bull calves
BMP (untreated)	1	NmL CH_4/g VS	274 (±7) *88 (±3)*	258 (±6) *76 (±3)*	288 (±6) *117 (±3)*
BMP (with extrusion)	1	NmL CH_4/g VS	264 (±5)	260 (±4)	281 (±4)
BMP (untreated)	2	NmL CH_4/g VS	*Not included*	232 (±6) *68 (±2)*	273 (±6) *110 (±3)*
BMP (with NiX treatment)	2	NmL CH_4/g VS	*Not included*[6]	272 (±5)	341 (±5)
TS			32% (±0.5)	29% (±0.3)	40% (±1.5)
VS			28% (±0.3)	26% (±0.3)	37% (±1.5)
Total Ammonium Nitrogen		g N/kg sample	2.8 (±0.1)	3.5 (±0.1)	1.9 (±0.1)
Total Nitrogen		g N/kg sample	8.3 (± 0.4)	7.7 (± 0.1)	7.7 (± 0.6)
Organic Nitrogen		g N/kg sample	5.5 (± 0.4)	4.2 (± 0.1)	5.8 (± 0.6)

Table 4 – Overview of key results from BMP and TS/VS analysis of untreated and NiX treated deep litter from bull calves suppliers.

- The batch BMP for untreated deep litter from bull calves suppliers in the first BMP setup ranged from 258 – 288 NmL CH_4/g VS.
- Extrusion had no significant effect on the batch BMP or the digestion speed

[5] From Konrad Pumpe Gmbh

[6] Laursen was not NiX treated. See section **Fejl! Henvisningskilde ikke fundet.** for details.

- The batch BMP for untreated deep litter from bull calves suppliers in the second BMP analysis ranged from 232 – 273 NmL CH_4/g VS. These potentials are a little lower than the value obtained in the first BMP analysis (compare BMP setup 1 and 2 in the above table). The reason for this is likely natural variance between samples and BMP setups.
- The age of the deep litter may be a determining factor in BMP quality of the deep litter.
- NiX treatment resulted in an increase in final batch BMP of 17%-25%, and a 50% increase in digestion rate.
- NiX treatment without addition of burnt lime had no effect on the batch BMP (data from the first BMP analysis – data not shown)
- Dry matter contents ranged from 29% - 40%.

	BMP setup	Units	KFC	Jacobsen	Olesen
Deep litter type			Dry cows/calves/ heifers	Dry cows/calves	Dry cows/calves
BMP (untreated)	3	NmL CH_4/g VS	225 (±7) *58 (±3)*	313 (±7) *69 (±6)*	298 (±6) *70 (±5)*
BMP (with NiX treatment)	3	NmL CH_4/g VS	291 (±5)	329 (±4)	340 (±5)
TS			26% (± 0.5)	22% (± 0.6)	23% (± 0.3)
VS			23% (± 0.5)	19% (± 0.5)	19% (± <0.3)
Total Ammonium Nitrogen		g N/kg sample	0.6 (± <0.1)	1.3 (± <0.1)	2.1 (± <0.1)
Total Nitrogen		g N/kg sample	5.2 (± 0.4)	4.6 (± 0.2)	5.1 (± <0.1)
Organic Nitrogen		g N/kg sample	4.6 (± 0.4)	3.3 (± 0.2)	3.0 (± 0.2)

Table 5 – Overview of key results from BMP and TS/VS analysis of untreated and NiX treated deep litter from dairy farmers.

- The batch BMP for untreated deep litter from dairy farmers ranged from 225 – 313 NmL CH_4/g VS.
- NiX treatment resulted in an increase in final batch BMP of 5%-30%, and a 30% - 70% increase in digestion rate.
- Dry matter contents ranged from 22% - 26%.

The TS and VS levels for both categories of deep litter (bull calves and dairy cattle) show that the deep litter from bull calves productions is significantly dryer than deep litter from dairy cattle. The reason for this is possibly the dry cow section, which in a number of smaller Danish farms is not mucked out on a regular basis and hence tends to be very wet.
The deep litter from bull calves is higher in both total nitrogen and total ammonium nitrogen. This is a bit surprising since as one would expect a dryer substrate to contain less urine and hence less

TAN. The reason may be found in the dry cow diet, which is usually nutrient poor. This would lead to less protein being absorbed and hence less nitrogen excreted.

The expected methane yield, when digesting the treated and untreated samples in a two-stage CSTR setup, may be seen below. The calculation combines the digestion speeds and BMP obtained in batch with the setup of the CSTR system to predict the methane production.

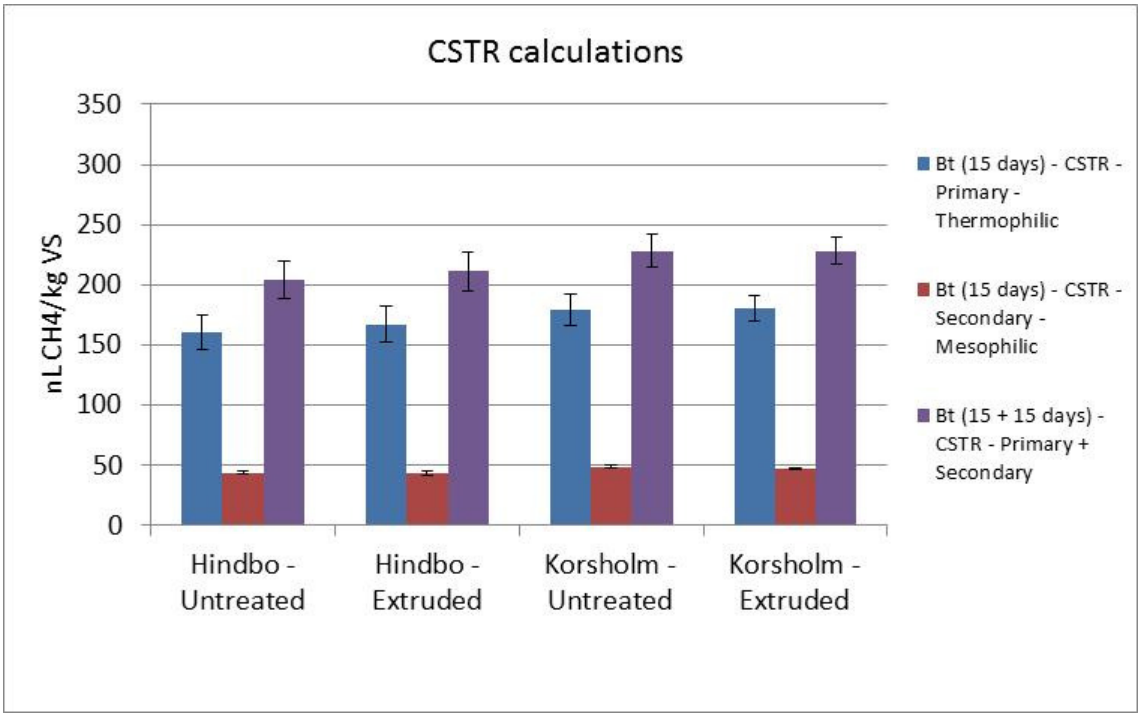


Figure 4 – Expected methane yields of deep litter from bull calves in a CSTR setup based on results from the first BMP setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

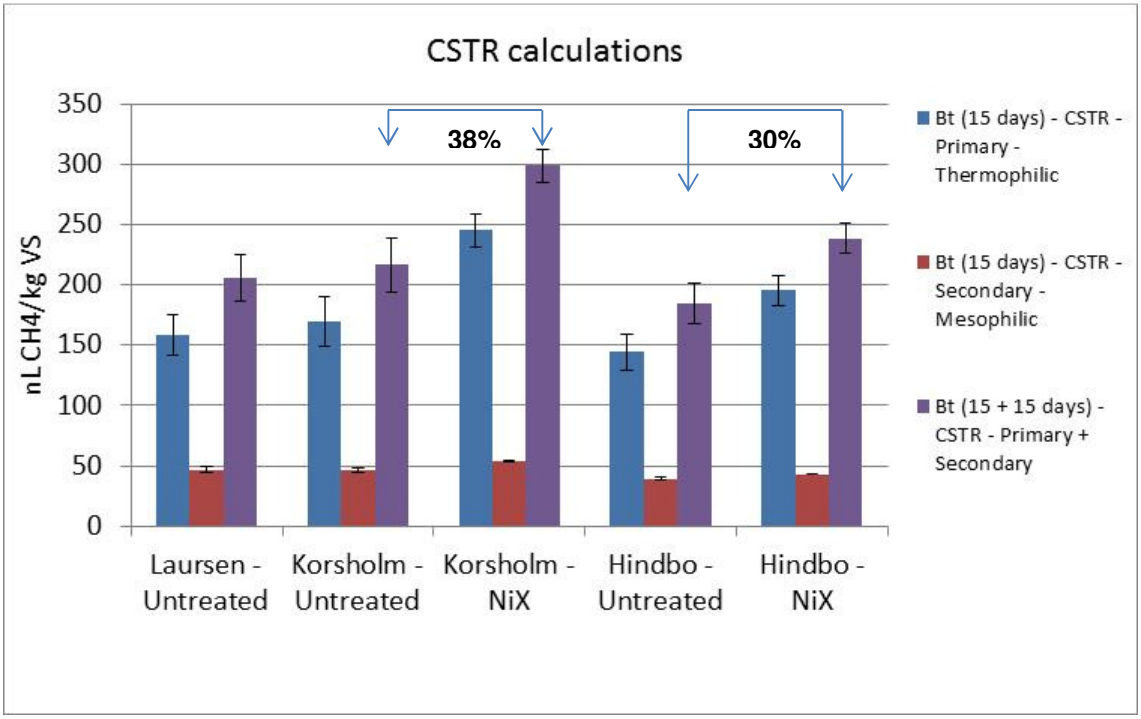


Figure 5 – Expected methane yields of deep litter from bull calves in a CSTR setup based on results from the second BMP setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

When comparing the methane yields in Figure 4, it can be seen that extrusion has no significant effect on the expected methane yield in a CSTR. On the other hand NiX treatment causes an increase in methane yield by 30%-38% in a two-stage thermophilic/mesophilic system with a 15 days retention time in each digestion step (see Figure 5).

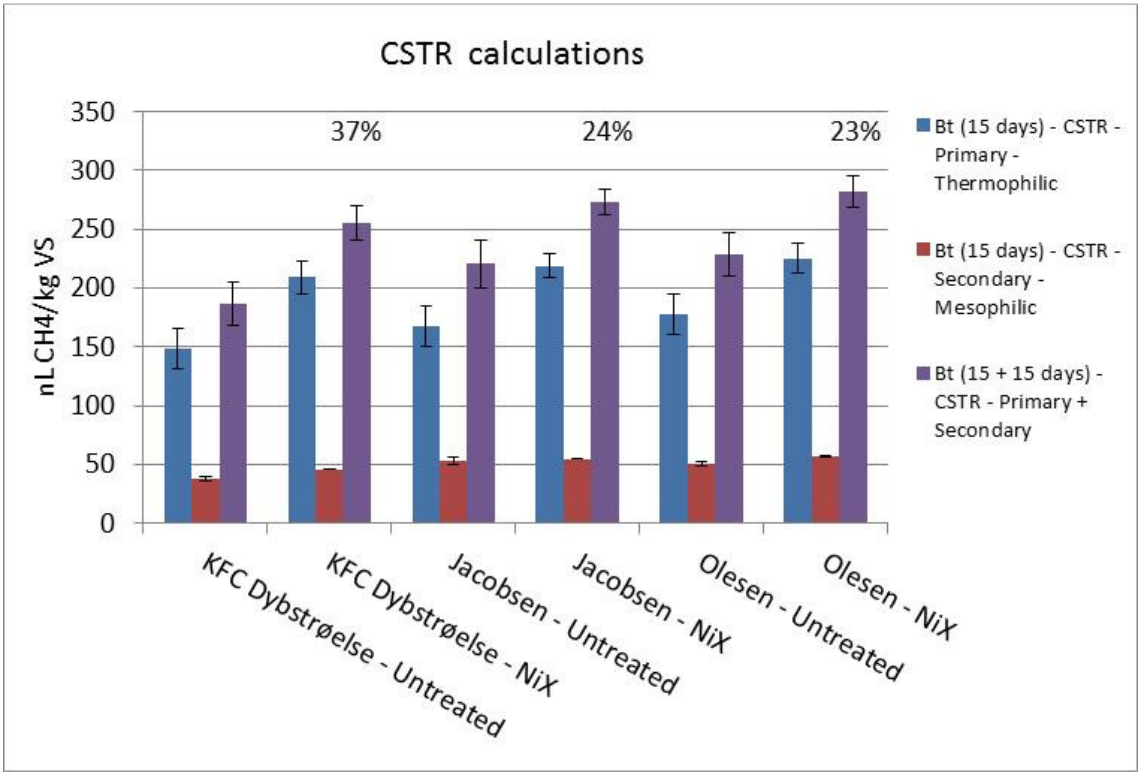


Figure 6 – Expected methane yields of deep litter from dairy farmers in a CSTR setup based on results from the third BMP setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

The expected CSTR methane yield from NiX treated deep litter from dairy farmers ranges from 23%-37% as seen from Figure 6. As mentioned previously deep litter from KFC is a mixture between litter from heifers, dairy cows and calves, whereas the litter from Jacobsen and Olesen is a mixture of litter from dairy cows and calves. It cannot be concluded from this study whether the higher improvement on the KFC litter is due to the heifer fraction or some other factor such as e.g. the lower batch BMP value of the untreated sample or some other factor. More experiments will be needed to conclude this.

3.4 Hen litter from egg layers

The following section summarizes the analyses performed on hen litter from egg layer.

The purpose of the investigations was to

- Determine the TS/VS and nitrogen content of hen litter
- Determine the biomethane potential (BMP) value of untreated hen litter
- Determine the effect of NiX pre-treatment on the BMP value of hen litter

Broiler litter was obtained from an egg producer in the United Kingdom, and transported to the Xergi research centre, where it was homogenized and stored. The conclusions from the analysis may be seen from Table 6.

Parameter	Unit	Hen Litter
BMP (Untreated)	Nml CH_4/g VS	293 (± 9)
	Nml CH_4/g wet weight	*137*(± 5)
BMP (with NiX treatment)	Nml CH_4/g VS	292 (± 6)
	Nml CH_4/g wet weight	*136* (± 4)
Total Solids content	w/w %	46.6 (± 0.9)
Volatile solids content	w/w %	33.9 (± 0.5)
Total Ammonium Nitrogen	g N/kg sample	3.7 (± 0.3)
Total Nitrogen	g N/kg sample	25.7 (± 0.4)
Organic Nitrogen	g N/kg sample	22.0 (± 0.5)

Table 6 – Overview of key results associated with the analysis of hen litter. Organic Nitrogen is calculated by subtraction of total ammonium nitrogen from total nitrogen.

- The final batch BMP for untreated hen litter reached 293 NmL CH_4/g VS. 90% of this value was obtained after 11 days anaerobic digestion.
- NiX treatment did not result in an increase in neither BMP nor in digestion speed

The expected methane yield, when digesting the treated and untreated samples in a two-stage CSTR setup, may be seen below. The calculation combines the digestion speeds and BMP obtained in batch with the setup of the CSTR system to predict the methane production.

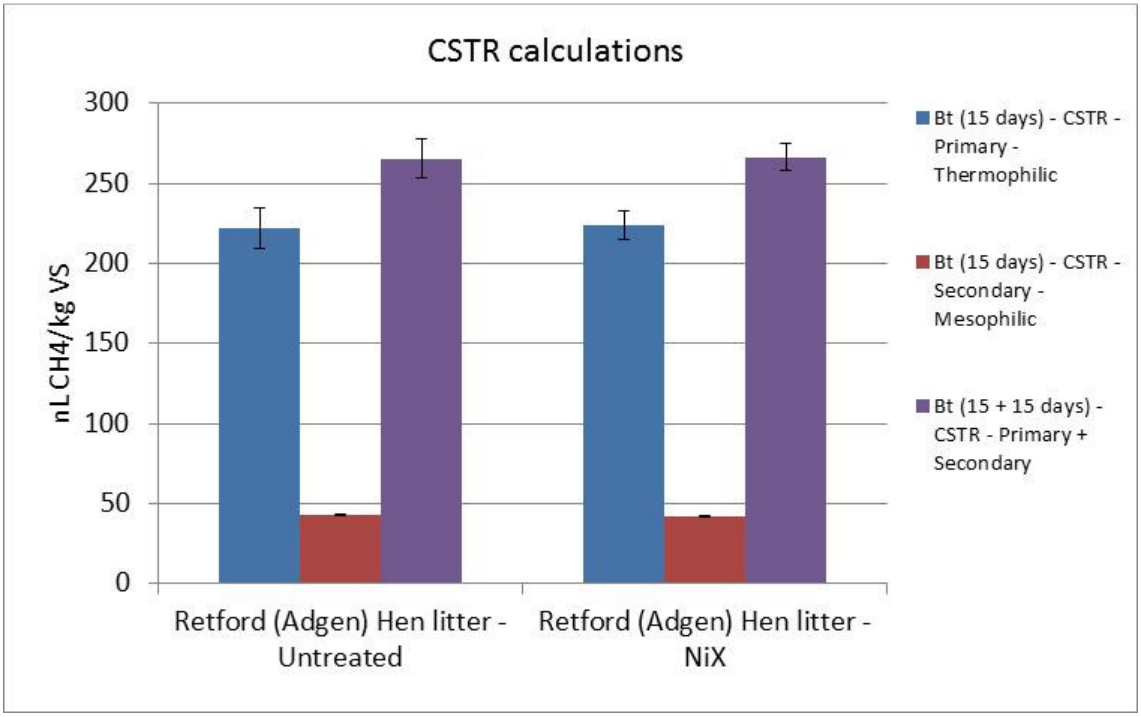


Figure 7 – Expected methane production in a two-stage CSTR with a thermophilic primary digester and a mesophilic secondary digester, both with 15 days retention time. Bt = methane yield after time t. Error bars are produced from 95% confidence intervals.

From Figure 7 it can be seen that NiX treatment cannot be expected to have a significant effect on the expected methane yield in a CSTR.

3.5 Chicken litter from broilers

The following section summarizes the results of the experiments and analyses performed on broiler litter. The broiler litter is used in a continuous pilot plant biogas trial during which it undergoes a number of treatments including nitrogen mineralisation, NiX treatment and pH adjustment prior to anaerobic digestion[7]. The analyses reported here investigates the effect on biomethane potential (BMP) from each of the different treatments.

The purpose of the investigations was to determine the BMP value of:

- Broiler litter from slaughter chickens.
- Broiler litter after nitrogen mineralisation.
- Broiler litter after nitrogen mineralisation and NiX treatment.
- Broiler litter after nitrogen mineralisation, NiX treatment, and pH-adjustment.

The BMP analyses were carried out in two separate setups. In the first setup the effect of mineralisation and NiX treatment at 4 barg was investigated. In the second setup the investigation included

[7] See Annex 4 to the main report.

mineralisation, NiX treatment at 0 barg and pH adjustment. In the following the samples which are repeated in both setups are averaged.

Broiler litter was obtained from a chicken farmer in Northern Ireland, and transported to the Xergi research centre, where it was homogenised and stored. Details regarding the different treatments can be found in Annex 4 to the main report. After each treatment samples were collected and analysed. Key results are presented in Table 7.

	Units	Untreated	Mineralised	Mineralised, NiX treated (4 barg)	Mineralised, NiX treated (0 barg)	Mineralised, NiX treated (0 barg, pH adjusted)
BMP	NmL CH_4/g VS *NmL CH_4/g wet weight*	300 (±10) *194 (±5)*	300 (±10)	279 (±4)	297 (±6)	307 (±7)
TS	w/w %	64% (±<0.1)				
VS	w/w %	53% (±<0.5)				
Total Ammonium Nitrogen	g N/kg sample	7.3 (± <0.7)				
Total Nitrogen	g N/kg sample	27.7 (± 1.8)				
Organic Nitrogen	g N/kg sample	20.4 (± 1.9)				

Table 7 – Overview of key results from BMP and TS/VS analysis of broiler litter.

- Untreated litter from broiler chickens reached a final batch BMP of 300 NmL CH_4/g VS, and is comparable to previous analysis of hen litter (293 NmL CH_4/g VS[8])
- Mineralisation seems to cause a slight reduction in both the BMP of the VS (-5%) and the digestion speed (-25%) compared to untreated broiler litter in the first investigation (data not shown). However, in the second BMP setup there is no significant change in either batch BMP or digestion speed (data not shown).
- NiX treatment at 4 barg shows a small drop in BMP and an increase in digestion speed. However, the net effect is practically zero and the apparent effects are likely due to small measuring deviations in the beginning of the analysis period.
- NiX treatment at 0 barg shows no deviations compared to the untreated sample

[8] Reference: Xergi Internal report "Hen Litter (Retford) - Analysis Report"

- pH adjusted material shows a slight increase in both digestion speed and batch BMP. The net effect is not significant though, and since this sample has only been analysed once, there is no conclusive effect from the treatment.

The expected methane yield, when digesting the treated and untreated samples in a two-stage CSTR setup, may be seen below. The calculation combines the digestion speeds and BMP obtained in batch with the setup of the CSTR system to predict the methane production.

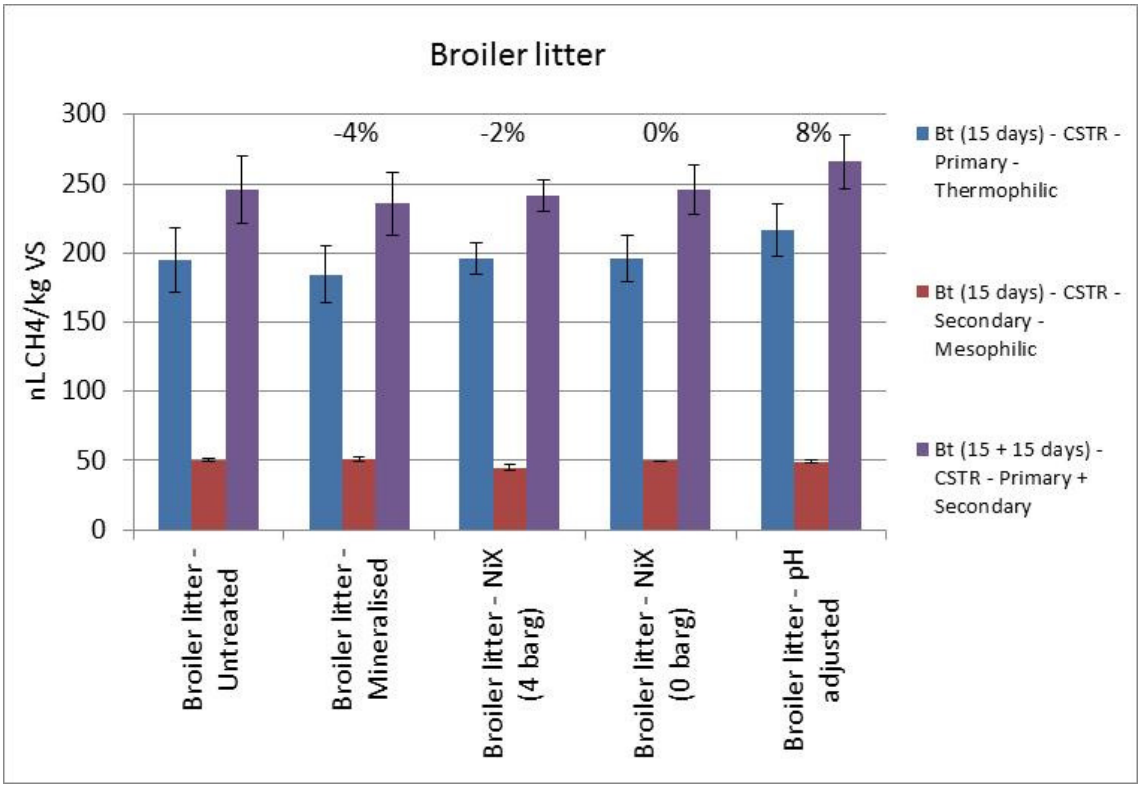


Figure 8 – Expected methane yields of broiler litter in a CSTR setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

Comparison of the methane yield of untreated broiler litter with litter that has been mineralised, NiX treated, and pH adjusted, show that none of the treatments yield any significant effect on the quality of the VS.

4 Conclusion

The biomethane potential of a number of substrates was investigated. The effect on biomethane potential by several mechanical treatments was also elucidated and compared to NiX treatment.

The mechanical treatments tested were grinding and hammermilling with wheat straw as the substrate. Grinding showed the highest improvements with up to 18 % - 36% more methane expected in a two-stage CSTR biogas plant. All three levels of grinding showed significant improvements in batch BMP and digestion speed. Hammermilling can be expected to result in up to 15% more methane produced in a CSTR setup. This increase is mainly due to an effect on digestion rate.
In comparison NiX treatment of the same substrate causes a 26% improvement in methane yield in a CSTR setup. This effect is dependent on lime addition, since NiX treatments in the absence hereof showed no significant effects.

The effect of NiX treatment on a number of biomasses was investigated. NiX treatment seems to be more effective the lower the original batch BMP of the untreated substrate and there is a positive correlation between the amount of fibrous material in the substrate and the improvement obtained by NiX treatment. For example, poultry manure contains little or no fibrous material and is not improved by NiX treatment, whereas fibre fractions from already digested energy crops are improved in batch BMP by up to 98%.

The significance of the dry matter content of the NiX treated substrate was also investigated. There is a clear positive correlation between water content and efficacy of the NiX treatment. In the NiX treated degassed fibres the samples with the lowest TS contents improved in batch BMP by twice the amount of the high TS content samples.

5 Appendix

5.1 Standard Operating Procedure – BMP batch assay

Biogas potential tests are made to evaluate whether a substrate is biologically degradable and to measure the amount of methane that it can produce (batch BMP).

1 Equipment

- 500 mL infusion bottles
- rubber stoppers
- aluminium screw lids
- distilled water
- nitrogen gas
- incubator
- scale (precision 0,01 g)
- 200 uL syringe with pressure lock
- gas-chromatograph equipped with FID detector
- 100% methane (standard gas)
- crystalline cellulose
- equipment to measure TS and VS

2 Anaerobic inoculum

The anaerobic inoculum is collected from a well-functioning thermophilic reactor, sieved through a 2mm sieve and stored at 52 ℃. The inoculum has to be in the incubator for 7 – 15 days before use to minimize the production of gas. The amount of inoculum to be collected is 2 – 3 times more than required.

3 Preparation of the batches

A biogas potential test consists of a series of bottles containing the substrate to be tested and inoculum (substrate bottles), a series of bottles with cellulose and inoculum (cellulose controls), and a series of bottles with inoculum only (blanks).

For each substrate to be tested it is necessary to include two substrate concentrations (high and low). This is not necessary for the cellulose controls. The amount of substrate to be added is calculated on the basis of the VS content of the inoculum. With a few exceptions, the VS added should always be less than 50% of the total amount of VS from the inoculum. A minimum of three replicates should be included for each substrate level. Blanks and Cellulose controls should minimum be in quadruplicates.

4 Procedure to prepare the batches:

1. Measure the total volume of the bottles (an average based on 10 bottles can be enough). Store this result as V_{bottle}.

2. Weigh off an exact amount of substrate in each substrate bottle. Take great care to do so homogenously. Record the amount of added substrate and label the bottles.
3. Weigh off an exact amount of cellulose in each cellulose control bottle. Record the amount of added substrate and label the bottles
4. Transfer 200 mL inoculum to each of the bottles (error 0,5% is acceptable). While adding inoculum to the bottles the inoculum stock should be thoroughly stirred to avoid precipitation of VS. However, great care should be exhibited, not to add air into the inoculum during stirring.
5. At evenly distributed intervals during inoculum addition, add inoculum to the blanks (no substrate is added to these bottles).
6. Flush the headspace of the bottles with nitrogen gas as they are filled with inoculum, to expel any air and seal them with rubber stoppers and screw lids.
7. Measure TS and VS of the inoculum.
8. Calculate the headspace ($V_{headspace}=V_{bottle}-V_{inoculum}-V_{substrate}$) of each bottle (the density of the substrate can be assumed to be 1g/ml)
9. The bottles are incubated for 6 – 12 weeks and methane production is measured;

5 Measurements

The production of methane is measured at least twice/week the first two weeks of incubation, once/week the third and fourth week of incubation and once/two weeks during the remaining incubation period.

Procedure to measure methane:

1. Inject a standard row of 100% methane into the GC (150-100-75-50-25 uL). This injection is made by adjusting the syringe to the desired volume at the temperature and atmospheric pressure of the laboratory.
2. Make a pressure test of the syringe: Fill the syringe with 150 uL methane as in step 1. Reduce the volume of gas with the pressure lock closed until 75 uL (equal to 2 bar) and maintain this volume/pressure for 10 seconds. Withdraw the plunger to 150 uL again and inject the content into the GC. The methane loss should be <5% compared to the standard curve.
3. Record the temperature and atmospheric pressure of the laboratory
4. Insert the needle of the syringe into the infusion bottle through the rubber stopper and sample 100 uL (V_{inj}). Close the pressure lock of the syringe while the needle is still inside the bottle. In this way the biogas is sampled at the temperature and pressure inside the bottle. Inject this sample into the GC.
5. After 3-5 days, 10-13 days and 24-26 days, the pressure in the bottles should be released: Measure the gas inside the bottles, release the gas with a needle, measure the gas again.

Make a standard curve at the end of the measurement to ensure no drifting has occurred.
Do not let the bottles become cold to avoid disturbing the microorganisms. It is not required to measure the temperature and pressure inside the bottles.

6 Calculations

The gas-chromatograph gives an area for each injection.

The calculations are made on mass-basis as the area given by the GC depends on the number of molecules of methane that have been injected. The results are then converted into volume and the yield is calculated as Nml/gVS.

Procedure to calculate the yield:

1. Calculate the slope of standard curve (the correlation coefficient should be >0,99). Convert the x-axis into moles using the ideal gas law and the recorded ambient temperature and pressure during injection.
2. Divide the area of each substrate peak with the slope to obtain the mole injected for each substrat bottle.
3. Multiply the result with the $V_{headspace}$:V_{inj} ratio to obtain the total amount of methane in the headspace.
4. Convert the result to normal volume using the ideal gas law and STP conditions (1 atm, 273 K).
5. When ventilations have been performed, calculate the volume released by substracting the methane volume after ventilation from the methane volume before ventilation. Add this value to the subsequent measurements.
6. Calculate the average of the methane produced by the blanks
7. Subtract the volume of gas produced by the blanks from the volume of methane produced by the substrate/cellulose bottles.
8. Calculate the specific methane yield by dividing the volume of methane produced with the VS added during the preparation of the batches.
9. Normalize values using the cellulose control. The cellulose control is expected to yield 390 mL methane/g VS.

5.2 Validation of the Xergi Standard Operating Procedure by Teknologisk Institut.

TEKNOLOGISK INSTITUT

Report

Validation of Xergis method for methane potential analysis in 500 ml biogas flasks

used in the EUDP project, jnr. 64011-0335: Forbehandling af fiberrige biomasser til biogasproduktion

Conclusions

Danish Technological Institute (DTI) has validated the method for methane potential measurements undertaken by XERGI in the EUDP project jnr. 64011-0335. The verification included both the test setup and the methane analysis.

The results at DTI varied slightly (deviation max. 16.8 %) from the biogas potential, XERGI obtained. Most results corresponded within one standard deviation, all samples within two standard deviations. The deviations were in the region of what can be expected as variations by handling of the samples by different technicians and the non-homogeneity of the substrates, as the used straw is far from homogeneous by nature in regard to small sample volumes to be used.

Introduction

Danish Technological Institutes (DTI) task was a validation of the methane potential analysis, XERGI applied in the small scale digestion tests for different fiber containing substrates (biomasses) with and without preliminary treatment.
XERGI delivered the substrates and slurries to be tested and decided the levels for substrate addition.

Method

Three batch tests were performed parallel at DTI and Xergi with 66 flasks each, containing triplicate flasks for each biomass and pretreatment and 6 flasks each for seeding sludge (inoculum background measurements) and cellulose (reference).

The used method for measuring methane potential is based on the measurement protocol which is used by Xergi (described in the appendix). The only deviation from the method was usage of a 60 % methane standard, which has been taken in account in the calculations. The basic measurement principle is based on the following steps:

- Performing batch experiments with incubation in flasks with rubber stoppers according to description in VDI 4630 including the demand (organic dry matter of sub-

strate)/(organic dry matter of seeding sludge) < 0,5 to avoid/minimize inhibition

- Incubation at thermophile conditions at 52 °C
- Measurement of methane production is based on a direct measurement of moles methane present in head space of the flasks by taking a pressurized sample with known volume (etc. 100-200 µl) and injecting the sample into a gas chomatograph for measurement of moles methane in the sample. From the volume of head space in the flasks the amount of moles methane in the head space can then be calculated.
- Ventilating flasks when pressure is high with measurement of methane content after ventilation

The flasks had a volume of 500 ml containing 200 ml inoculum and substrate of two different levels volatile solids of substrate, for to take possible inhibition in account.

The tests at DTI were carried out synchronous with the tests at XERGI using the same seeding sludge (inoculum) as XERGI.
The setting up of the flasks at DTI was done by a technician briefed by XERGI of the procedure of careful homogenization of the substrates without destroying fibers, weighing with a precision of 0.01 g and replacing the air in the flasks by nitrogen. Special care was taken of stirring the seeding sludge before filling of each flask to avoid precipitation of VS. The flasks were closed with thick rubber stoppers and an aluminum screw cap. The flasks were incubated at 52 °C ± 0.5 °C during the whole test period.

Gas chromatographic measurements of methane were carried out equally distributed over the following test periods: 4 times in the first 2 weeks, 2 times in the following 2 weeks and 2 times in the remaining time of the test period, which lasted up to 3 months.

The gas chromatograph used for analysis was equipped with two packed columns (Pora Pak Q and Molsieve 5, length: 1 m each) and a Thermal Conductivity Detector.

Before and after measurement of all flasks a 4 point standard curve was measured with 60 % methane (injection volumes 50 µl, 150 µl, 250 µl and 500 µl), as well as the pressure tight syringe (200 µl) was tested for tightness by filling the syringe with 150 µl of standard, reducing the volume to 75 µl with the pressure lock closed (equal to 2 bar) and maintaining this pressure for 10 seconds. The maximum loss for the measurement compared to the standard curve was < 5 %.

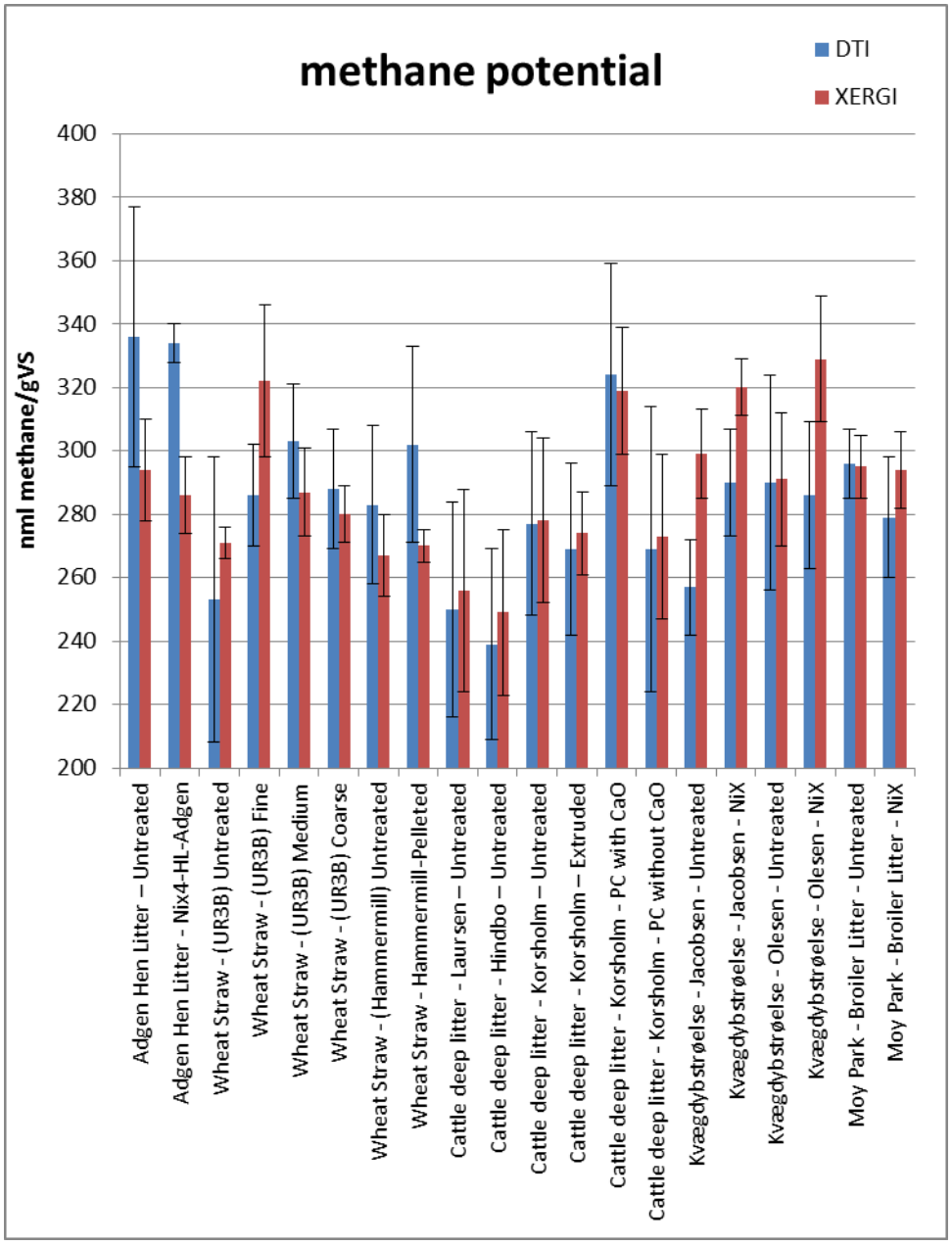
Results and discussion

Distributed to 3 tests a total of 26 substrates were tested in triplicates for high and low VS loading. The substrates were wheat straw, cattle deep litter, hen litter and broiler litter both untreated and treated with different methods chosen by XERGI.

The following table shows the maximum value of methane potential for the different substrates as the average of 6 flasks for each substrate. The average value for the low and high VS addition of substrate is shown. The values are background corrected and normalized for cellulose control (3 points at maximum potential). The maximum cellulose methane potential is set to 390 nml methane/g VS. This value is based on results from typical measurements in laboratories using measurements of produced gas volume and measurement of methane concentration according to VDI 4630. The theoretical maximum methane potential is 415 nml methane/g VS. Due to different decomposition velocities for the different substrates, the reading was done at the day of maximum methane potential. For the cases of replicate treatments, the mean value is given in the table. Data for both treatments are to be seen in the figures of the appendix. The samples named "Kvægdybstøelse" are dairy cattle deep litter.

Substrate name	Methane potential B(0) [Nml_{CH4}/g_{VS}]		Standard deviation at day of reading [ml_{CH4}/g_{VS}]		Day of reading	
Test 1	DTI	XERGI	DTI	XERGI	DTI	XERGI
Adgen Hen Litter – Untreated	336	294	41	16	50	51
Adgen Hen Litter - Nix4-HL-Adgen	334	286	6	12	28	31
Wheat Straw - (UR3B) Untreated	253	271	45	5	98	94
Wheat Straw - (UR3B) Fine	286	322	16	24	98	94
Wheat Straw - (UR3B) Medium	303	287	18	14	98	94
Wheat Straw - (UR3B) Coarse	288	280	19	9	98	94
Wheat Straw - (Hammermill) Untreated	283	267	25	13	98	94
Wheat Straw - Hammermill-Pelleted	302	270	31	5	98	94
Test 2						
Cattle deep litter - Laursen – Untreated	250	256	34	32	27	43

Cattle deep litter - Hindbo – Untreated	239	249	30	26	27	43
Cattle deep litter - Korsholm – Untreated	277	278	29	26	27	43
Cattle deep litter - Korsholm – Extruded	269	274	27	13	27	43
Cattle deep litter - Korsholm - PC with CaO	324	319	35	20	27	17
Cattle deep litter - Korsholm - PC without CaO	269	273	45	26	27	21
Test 3						
Kvægdybstrøelse - Jacobsen - Untreated	257	299	15	14	28	32-85
Kvægdybstrøelse - Jacobsen - NiX	290	320	17	9	28	32-85
Kvægdybstrøelse - Olesen - Untreated	290	291	34	21	28	32-85
Kvægdybstrøelse - Olesen - NiX	286	329	23	20	28	32-85
Moy Park - Broiler Litter - Untreated	296	295	11	10	28	32-85
Moy Park - Broiler Litter - NiX	279	294	19	12	28	32-85



The methane potential of the different substrates measured by DTI and Xergi with the error bars, indicating one standard deviation.

In nearly all cases, the results derived from Xergi corresponded with the data derived by DTI within one standard deviation. Two samples though showed more than one standard deviation (Adgen Hen Litter - Nix4-HL-Adgen and Kvægdybstrøelse- Jacobsen-Nix). All data corresponded within two standard deviations. The results at DTI varied slightly (deviation max. 16.8 %) from the biogas potential XERGI obtained, which can be explained by the very inhomogeneous samples.

Aarhus June 13th, 2013

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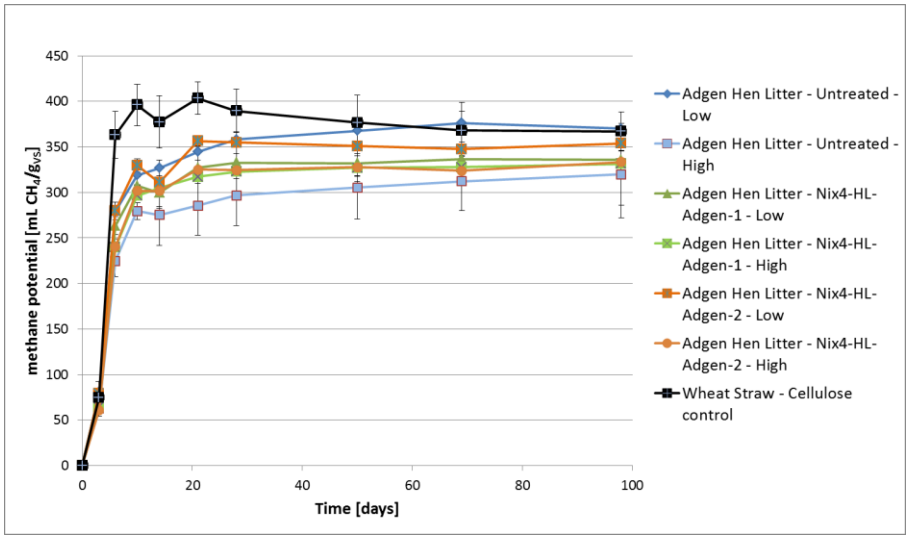
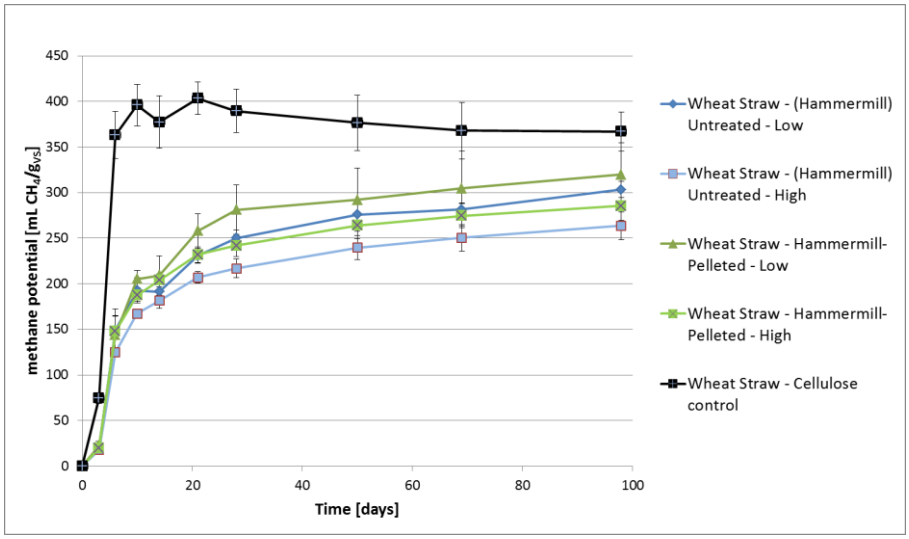
Appendix

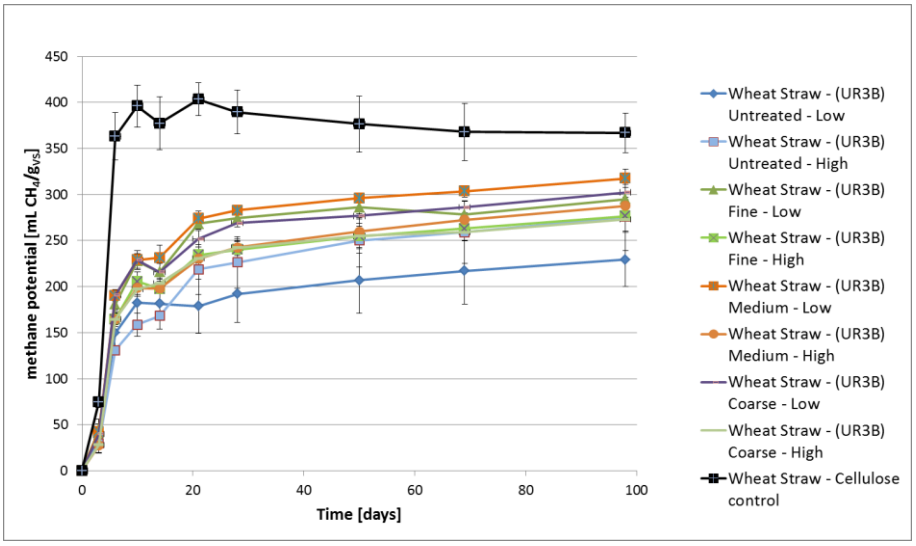
The following figures show the data background subtracted, specific to VS and normalized to the average value of cellulose (3 points at maximum potential). The maximum cellulose methane potential is set to 390 nml methane/g VS.

DTI data

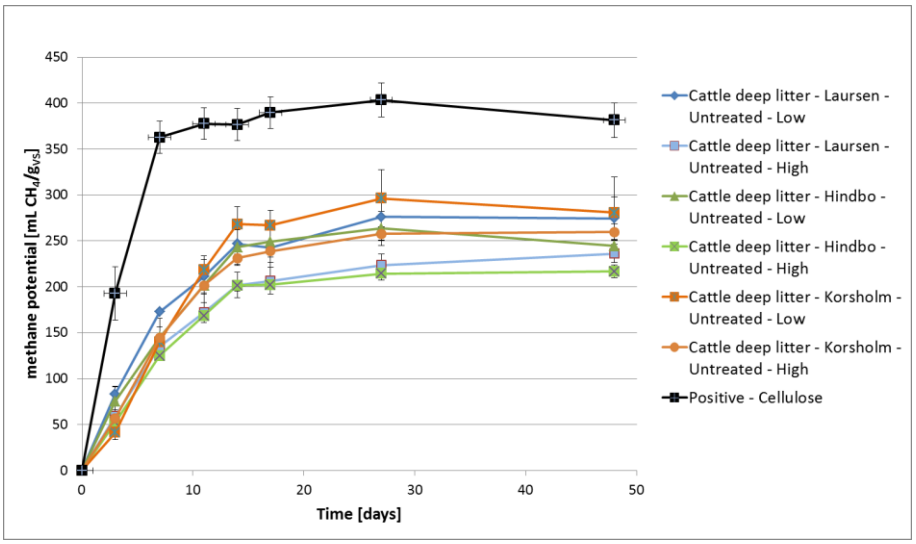
Test 1

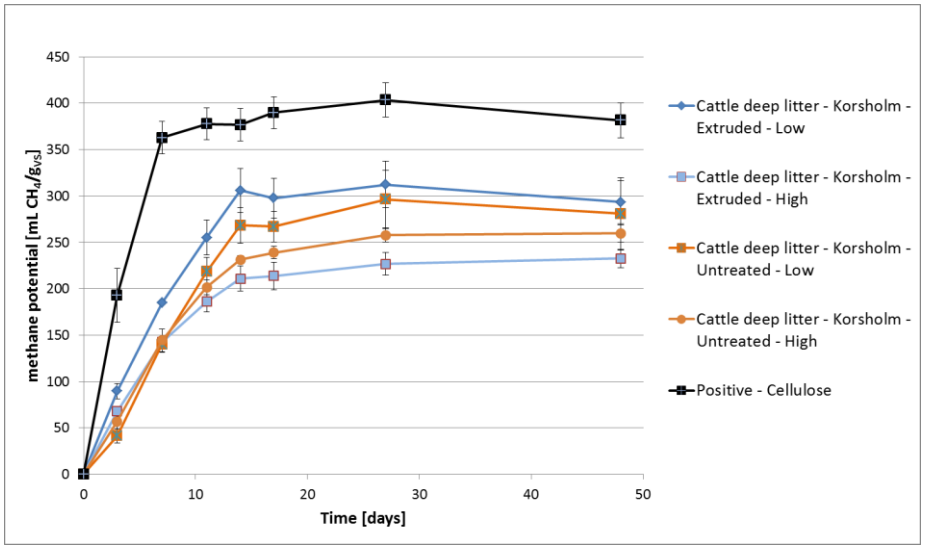


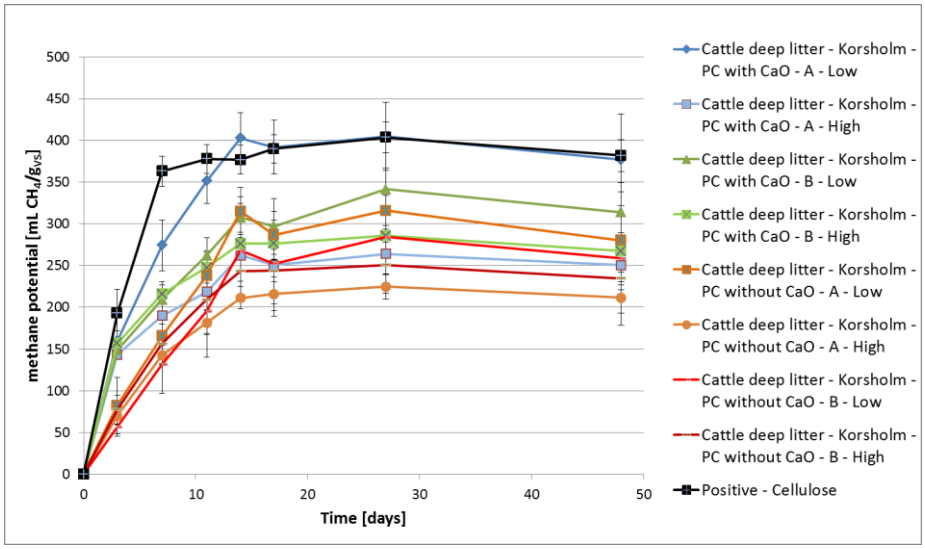



methane potential [mL CH4/gVS]
Time [days]
Wheat Straw - (UR3B) Untreated - Low
Wheat Straw - (UR3B) Untreated - High
Wheat Straw - (UR3B) Fine - Low
Wheat Straw - (UR3B) Fine - High
Wheat Straw - (UR3B) Medium - Low
Wheat Straw - (UR3B) Medium - High
Wheat Straw - (UR3B) Coarse - Low
Wheat Straw - (UR3B) Coarse - High
Wheat Straw - Cellulose control

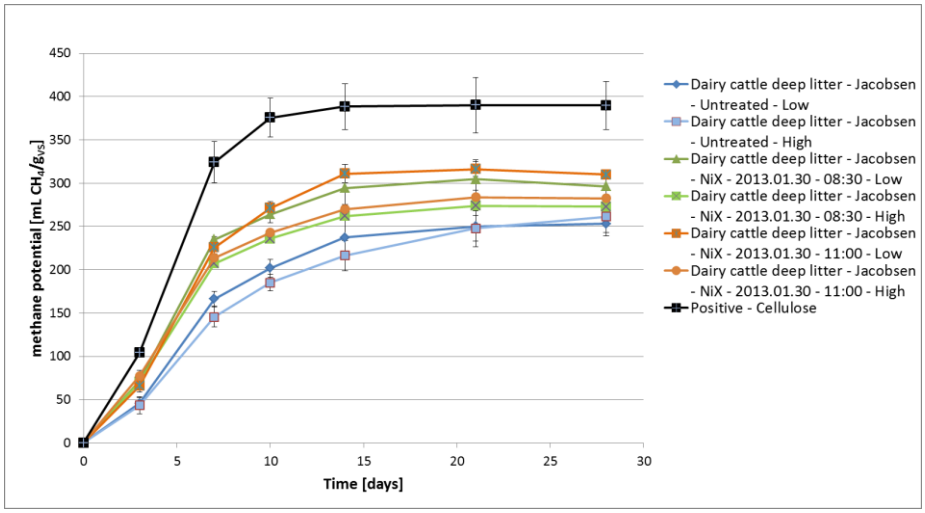
Test 2

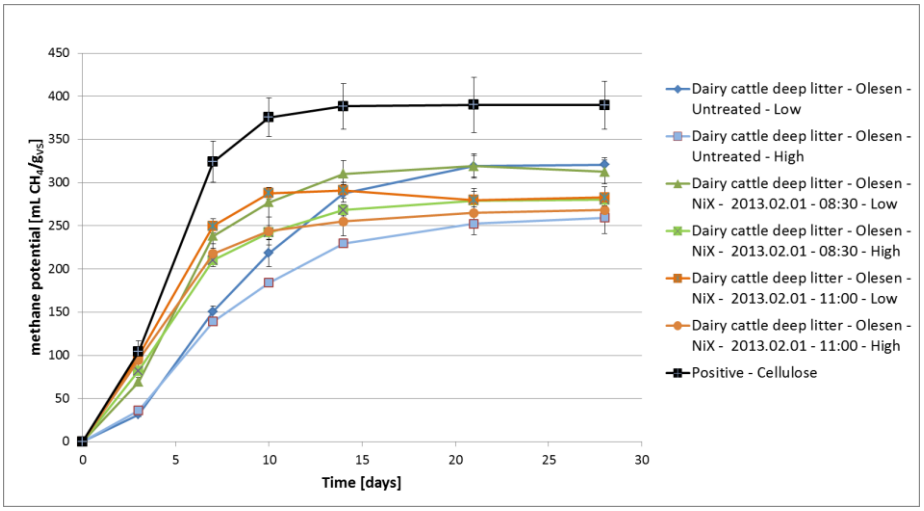

methane potential [mL CH4/gVS]
Time [days]
Cattle deep litter - Laursen - Untreated - Low
Cattle deep litter - Laursen - Untreated - High
Cattle deep litter - Hindbo - Untreated - Low
Cattle deep litter - Hindbo - Untreated - High
Cattle deep litter - Korsholm - Untreated - Low
Cattle deep litter - Korsholm - Untreated - High
Positive - Cellulose

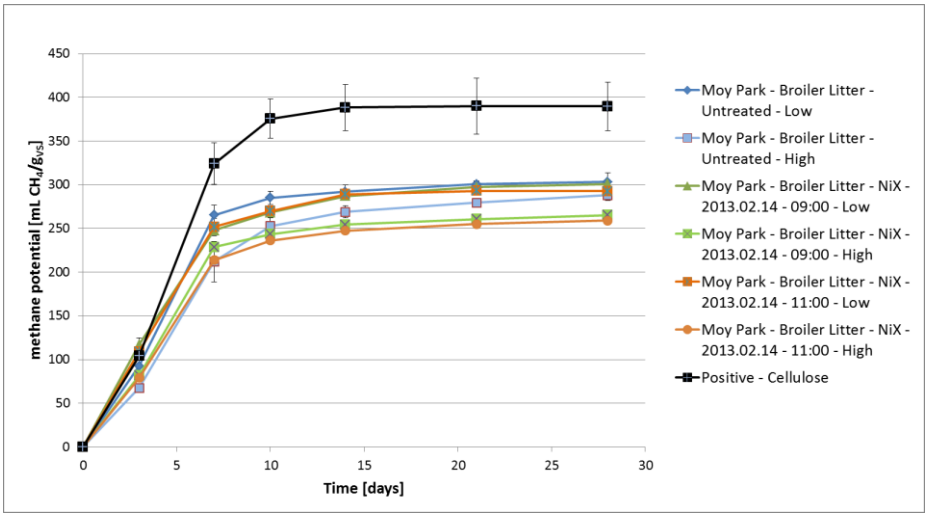

methane potential [mL CH4/gVS]
Time [days]
Cattle deep litter - Korsholm - Extruded - Low
Cattle deep litter - Korsholm - Extruded - High
Cattle deep litter - Korsholm - Untreated - Low
Cattle deep litter - Korsholm - Untreated - High
Positive - Cellulose


methane potential [mL CH4/gVS]
Time [days]
Cattle deep litter - Korsholm - PC with CaO - A - Low
Cattle deep litter - Korsholm - PC with CaO - A - High
Cattle deep litter - Korsholm - PC with CaO - B - Low
Cattle deep litter - Korsholm - PC with CaO - B - High
Cattle deep litter - Korsholm - PC without CaO - A - Low
Cattle deep litter - Korsholm - PC without CaO - A - High
Cattle deep litter - Korsholm - PC without CaO - B - Low
Cattle deep litter - Korsholm - PC without CaO - B - High
Positive - Cellulose

Test 3


methane potential [mL CH4/gVS]
Time [days]
Dairy cattle deep litter - Jacobsen - Untreated - Low
Dairy cattle deep litter - Jacobsen - Untreated - High
Dairy cattle deep litter - Jacobsen - NiX - 2013.01.30 - 08:30 - Low
Dairy cattle deep litter - Jacobsen - NiX - 2013.01.30 - 08:30 - High
Dairy cattle deep litter - Jacobsen - NiX - 2013.01.30 - 11:00 - Low
Dairy cattle deep litter - Jacobsen - NiX - 2013.01.30 - 11:00 - High
Positive - Cellulose

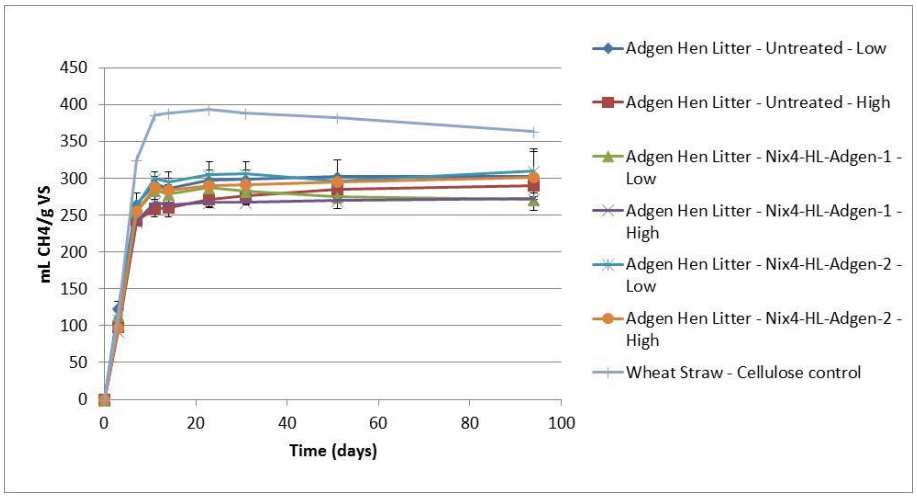


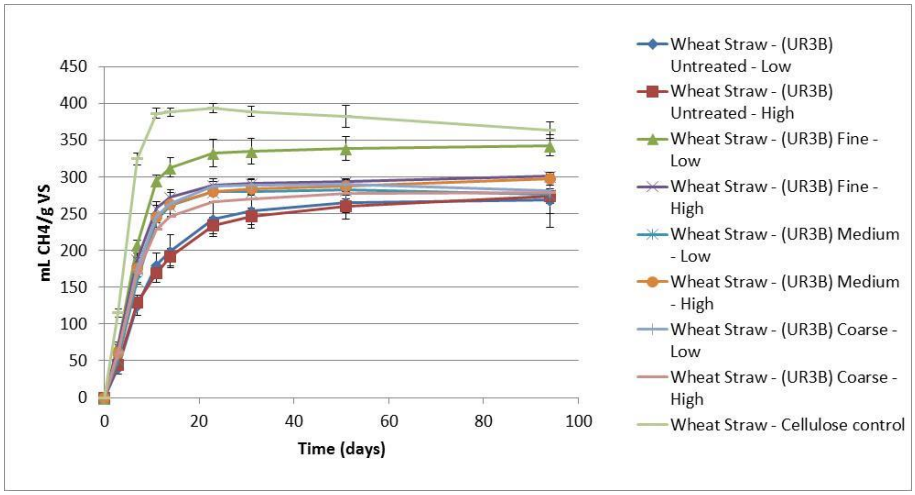


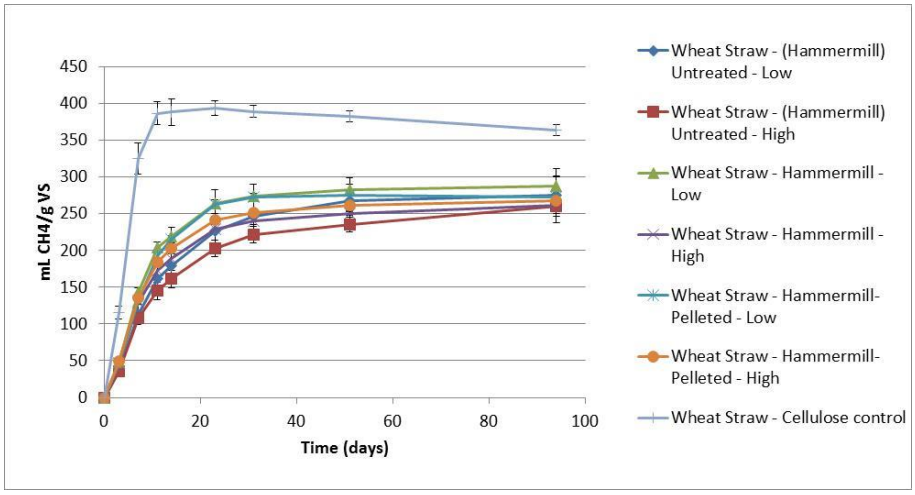
As seen in the figures above all data at 10 days production for cattle Deep litter Korsholm, Dairy Cattle deep litter Jacobsen and Dairy Cattle deep litter Olsen show a significant increase in accumulated methane when comparing NIX treated deep litter with non-treated deep litter.

Xergi data

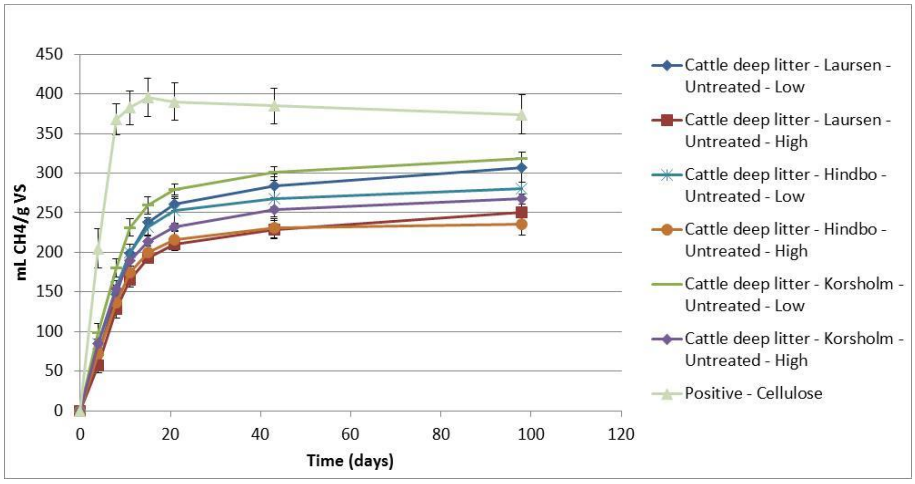
Test 1

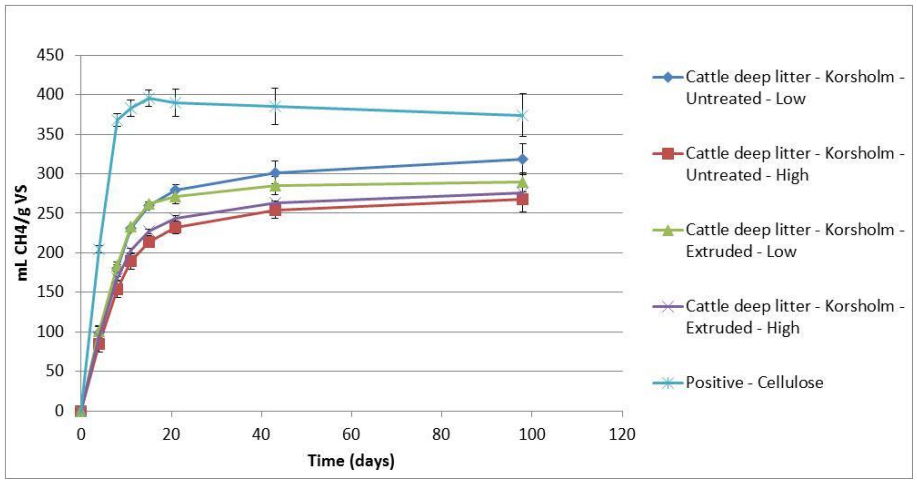


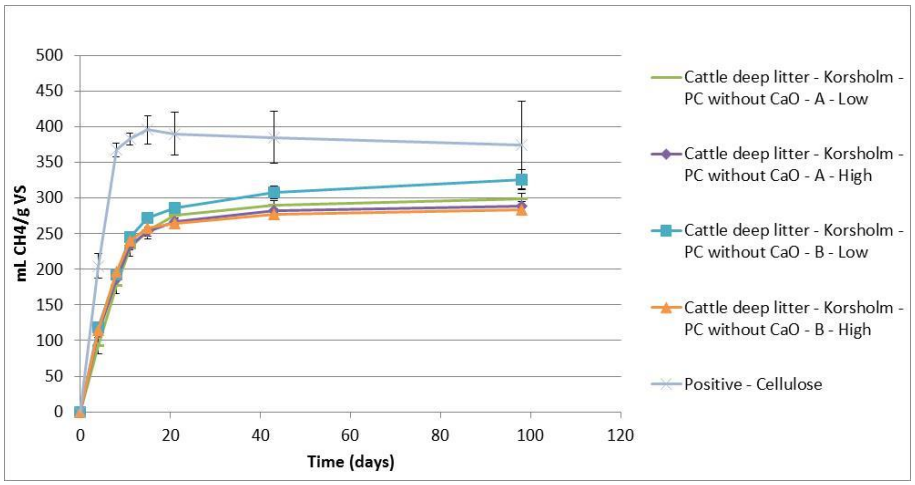




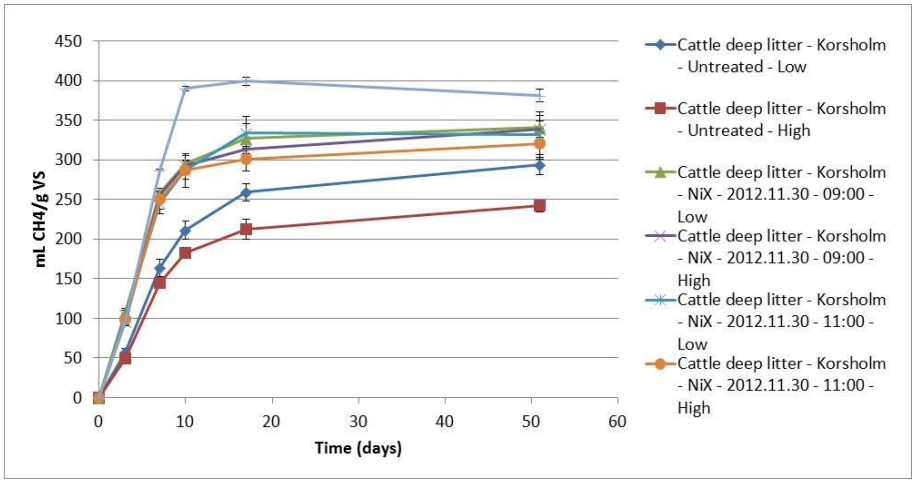
Test 2



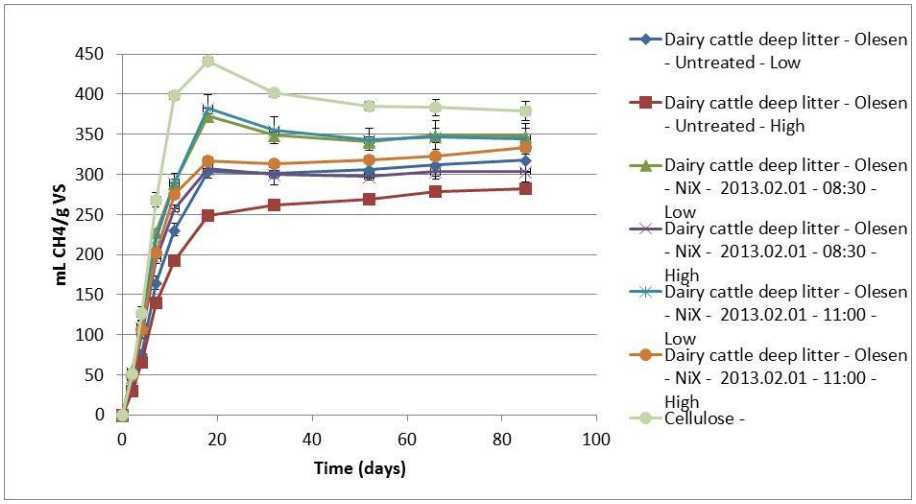


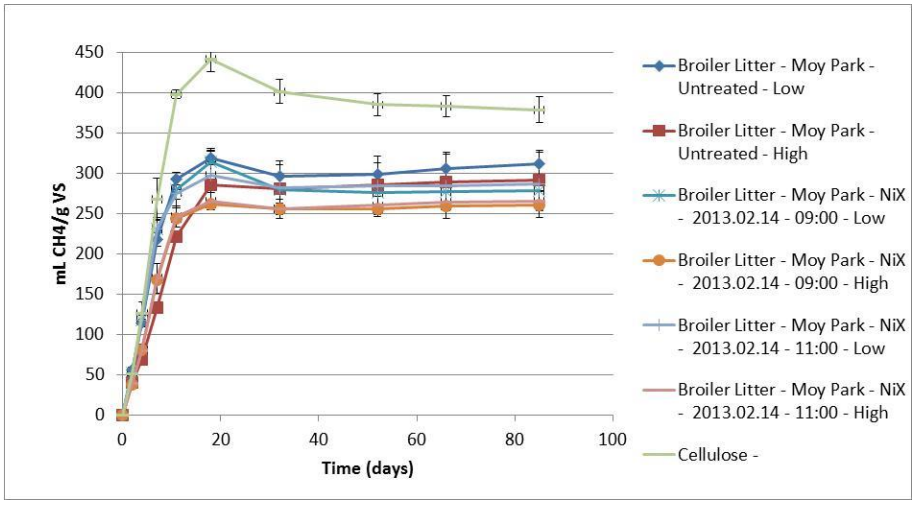


The first test of cattle deep litter Korsholm with Nix treatment didn't show valid data for which reason it was repeated (se data below).


mL CH4/g VS
Time (days)
Cattle deep litter - Korsholm - Untreated - Low
Cattle deep litter - Korsholm - Untreated - High
Cattle deep litter - Korsholm - NiX - 2012.11.30 - 09:00 - Low
Cattle deep litter - Korsholm - NiX - 2012.11.30 - 09:00 - High
Cattle deep litter - Korsholm - NiX - 2012.11.30 - 11:00 - Low
Cattle deep litter - Korsholm - NiX - 2012.11.30 - 11:00 - High

Test 3


mL CH4/g VS
Time (days)
Dairy cattle deep litter - Olesen - Untreated - Low
Dairy cattle deep litter - Olesen - Untreated - High
Dairy cattle deep litter - Olesen - NiX - 2013.02.01 - 08:30 - Low
Dairy cattle deep litter - Olesen - NiX - 2013.02.01 - 08:30 - High
Dairy cattle deep litter - Olesen - NiX - 2013.02.01 - 11:00 - Low
Dairy cattle deep litter - Olesen - NiX - 2013.02.01 - 11:00 - High
Cellulose -


mL CH4/g VS
Time (days)
Broiler Litter - Moy Park - Untreated - Low
Broiler Litter - Moy Park - Untreated - High
Broiler Litter - Moy Park - NiX - 2013.02.14 - 09:00 - Low
Broiler Litter - Moy Park - NiX - 2013.02.14 - 09:00 - High
Broiler Litter - Moy Park - NiX - 2013.02.14 - 11:00 - Low
Broiler Litter - Moy Park - NiX - 2013.02.14 - 11:00 - High
Cellulose -

Biogas potential tests (protocol from Xergi)

The test

Biogas potential tests are made to evaluate whether a substrate is biologically degradable and to measure the amount of methane that it can produce (yield).

Equipment
- 500 mL infusion bottles
- rubber stoppers
- aluminum screw lids
- distilled water
- nitrogen gas
- incubator
- scale (precision 0.01 g)
- 200 µL syringe with pressure lock
- gas-chromatograph equipped with FID detector
- 100 % methane (standard gas)
- crystalline cellulose
- equipment to measure TS and VS

Anaerobic inoculum

The anaerobic inoculum is collected from a well-functioning thermophillic reactor, sieved through a 2mm sieve and stored at 52 °C. The inoculum has to be in the incubator for 7 – 15 days before use to minimize the production of gas. The amount of inoculum to be collected is 2 – 3 times more than required.

Preparation of the batches

A biogas potential test consists of a series of bottles containing the substrate to be tested and inoculum (substrate bottles), a series of bottles with cellulose and inoculum (cellulose controls), and a series of bottles with inoculum only (blanks).
For each substrate to be tested it is necessary to include two substrate concentrations (high and low). This is not necessary for the cellulose controls. The amount of substrate to be added is calculated on the basis of the VS content of the inoculum. With a few exceptions, the VS added should always be less than 50 % of the total amount of VS from the inoculum. A minimum of three repli-

cates should be included for each substrate level. Blanks and Cellulose controls should minimum be in quadruplicates.

Procedure to prepare the batches:

1. Measure the total volume of the bottles (an average based on 10 bottles can be enough). Store this result as V_{bottle}.
2. Weigh off an exact amount of substrate in each substrate bottle. Take great care to do so homogenously. Record the amount of added substrate and label the bottles.
3. Weigh off an exact amount of cellulose in each cellulose control bottle. Record the amount of added substrate and label the bottles.
4. Transfer 200 mL inoculum to each of the bottles (error 0.5% is acceptable). While adding inoculum to the bottles the inoculum stock should be thoroughly stirred to avoid precipitation of VS. However, great care should be exhibited, not to add air into the inoculum during stirring.
5. At evenly distributed intervals during inoculum addition, add inoculum to the blanks (no substrate is added to these bottles).
6. Flush the headspace of the bottles with nitrogen gas as they are filled with inoculum, to expel any air and seal them with rubber stoppers and screw lids.
7. Measure TS and VS of the inoculum.
8. Calculate the headspace ($V_{headspace}=V_{bottle}-V_{inoculum}-V_{substrate}$) of each bottle (the density of the substrate can be assumed to be 1g/ml)
9. The bottles are incubated for 6 – 12 weeks and methane production is measured.

Measurements

The production of methane is measured at least twice/week the first two weeks of incubation, once/week the third and fourth week of incubation and once/two weeks during the remaining incubation period.

Procedure to measure methane:

1. Inject a standard row of 100 % methane into the GC (150-100-75-50-25 µL). This injection is made by adjusting the syringe to the desired volume at the temperature and atmospheric pressure of the laboratory.
2. Make a pressure test of the syringe: Fill the syringe with 150 µL methane as in step 1. Reduce the volume of gas with the pressure

lock closed until 75 µL (equal to 2 bar) and maintain this volume/pressure for 10 seconds. Withdraw the plunger to 150 µL again and inject the content into the GC. The methane loss should be < 5 % compared to the standard curve.
3. Record the temperature and atmospheric pressure of the laboratory
4. Insert the needle of the syringe into the infusion bottle through the rubber stopper and sample 100 µL (V_{inj}). Close the pressure lock of the syringe while the needle is still inside the bottle. In this way the biogas is sampled at the temperature and pressure inside the bottle. Inject this sample into the GC.
5. After 3-5 days, 10-13 days and 24-26 days, the pressure in the bottles should be released: Measure the gas inside the bottles, release the gas with a needle, measure the gas again.

Make a standard curve at the end of the measurement to ensure no drifting has occurred.
Do not let the bottles become cold to avoid disturbing the microorganisms. It is not required to measure the temperature and pressure inside the bottles.

Calculations

The gas-chromatograph gives an area for each injection.
The calculations are made on mass-basis as the area given by the GC depends on the number of molecules of methane that have been injected. The results are then converted into volume and the yield is calculated as Nml/gVS.

Procedure to calculate the yield:

1. Calculate the slope of standard curve (the correlation coefficient should be > 0.99). Convert the x-axis into moles using the ideal gas law and the recorded ambient temperature and pressure during injection.
2. Divide the area of each substrate peak with the slope to obtain the mole injected for each substrate bottle.
3. Multiply the result with the $V_{headspace}$:V_{inj} ratio to obtain the total amount of methane in the headspace.
4. Convert the result to normal volume using the ideal gas law and STP conditions (1 atm, 273 K).
5. When ventilations have been performed, calculate the volume released by subtracting the methane volume after ventilation

from the methane volume before ventilation. Add this value to the subsequent measurements.
6. Calculate the average of the methane produced by the blanks
7. Subtract the volume of gas produced by the blanks from the volume of methane produced by the substrate/cellulose bottles.
8. Calculate the specific methane yield by dividing the volume of methane produced with the VS added during the preparation of the batches.
9. Normalize values using the cellulose control. The cellulose control is expected to yield 390 mL methane/g VS.

Annex 2 Mineralisation of Organically Bound Nitrogen (WP 5)

Project No.:	EUDP j.nr. 64011-0335 /Internal project 10842
Made by:	SBG
Issued:	December 3 2013

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4.2 Method optimisation 8

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1 Abbreviations

AD	-	Anaerobic digestion
AD liquid	-	Liquid from an anaerobic digester
BMP	-	Biological methane potential
cTAN	-	Centrifuged TAN
N	-	Nitrogen
NmL	-	Normal mL (volume at 1 bar and 273 K)
OrgN	-	Organic Nitrogen
PC	-	Pressure cooking
RSD	-	Relative standard deviation
RT	-	Room temperature of the research facility (between 17 °C and 20 °C)
SD	-	Standard deviation
TAN	-	Total Ammonium Nitrogen
TKN	-	Total Kjeldahl Nitrogen
TS	-	Total Solids
VS	-	Volatile Solids
w/w%	-	Weight percentage

2 Summary and Conclusion

This report summarizes experiments on mineralisation of organically bound nitrogen in poultry litter.

Poultry litter contains high amounts of nitrogen. The majority of the nitrogen pool (approx. 80%) is stored in organic compounds such as uric acid or proteins, while the rest is inorganically bound in the form of ammonium or ammonia (collectively referred to as TAN).

The high nitrogen content of poultry litter makes it unattractive as a substrate in biogas plants. During anaerobic degradation, organically bound nitrogen (OrgN) is converted to ammonium and ammonia, the latter of which is toxic to the biogas producing microbial community. It is possible to remove a large portion of the TAN pool before it enters the biogas plant using NiX technology. However, the effect of this approach is limited by the fact that less than 20% of the total nitrogen pool in poultry litter exists as TAN.

The purpose of the experiments summarized here has been to develop and characterise a mineralisation reaction in which OrgN is efficiently converted to TAN, hence increasing the amount of strippable nitrogen before the NiX step.

The primary objectives for the investigations have been to determine

1. Is it possible to shift nitrogen from the OrgN pool in hen litter into TAN and how much of the OrgN pool in hen litter may be mineralised into TAN under optimal conditions?
2. What is the effect of oxygen, temperature, seeding and water content on the reaction?
3. Is the mineralisation process substrate specific or can it be repeated in other poultry litters?
4. What is the extent of mineralisation in protein bound nitrogen and in uric acid bound nitrogen, respectively?
5. Does mineralisation affect the VFA and TS/VS content?

The primary conclusions from the experiments are:

Ad 1. It is possible to mineralise OrgN into TAN. Under optimal conditions it is possible to convert 70-80% of the OrgN fraction into TAN. Combined with the TAN pool already present, the resulting strippable TAN fraction becomes 75-80% of the total nitrogen pool.

Ad 2. Nitrogen mineralisation is somewhat sensitive to oxygen contents, as high oxygen amounts are inhibitory to the reaction. The reaction seems indifferent to variations below atmospheric oxygen levels. The temperature optimum for the process is in the range 33 – 37 °C. Reaction rates are approximately doubled when the mineralisation mixture is seeded with material from a previous incubation. Decreasing amounts of water cause a reduction in mineralisation rate.

Ad 3. Mineralisation of OrgN to TAN is equally effective in both hen litter and chicken litter.

Ad 4. Uric acid nitrogen constitutes 50% of the OrgN fraction, and was completely degraded in all analysed samples. Degradation of the remaining OrgN pool (primarily protein-bound nitrogen) ranged from 48-60%.

Ad 5. Both VFA's and TS/VS levels change during mineralisation. There is a considerable production of acetate and butyrate. TS and VS levels are reduced by about 20%. The mass balance analysis is incomplete, but some of the lost VS are converted to CO_2. More experiments are needed to determine the nature of the remaining VS loss.

3 Materials and Methods

3.1 Substrate characterisation

Figure 1 – Hen litter.

Figure 2 – Chicken litter.

3.1.1 Dry Matter

Total solids (TS) and volatile solids (VS = organic dry matter) were determined in triplicate according to Danish Standard[1], TS were determined by heating the samples to 105°C for a minimum of 24 hours to constant weight. VS were determined by burning the samples at 550°C for 3-4 hours. The methods are modified to take into account the evaporation of volatile fatty acids during drying.

[1] TS: DS/EN 12880. VS: DS/EN 12879.

3.1.2 Substrate information

Substrate name	Hen Litter	Chicken litter
Substrate description	Litter from egg layers	Litter from broilers. 0.8 tons of straw/1.7 tons of wood chips/27.5 tons of chicken faeces
Production type	Cages	Stables (25400 birds)
Sample date	May 2012	November 2012
Substrate age (at time of sampling)	< 1 day	42 days
Sampled by	APJE	SBG
Supplier information	Stud Farm, Rufford, Newark-on-Trent, Nottinghamshire, NG22 9HB United Kingdom	Mr Peter McWilliams 56 Tullyaran Road Donaghmore Dungannon Co. Tyrone BT70 3HL Northern Ireland

Table 1 – Substrate, sampling and supplier information

3.1.3 Nitrogen content analysis

Samples were analysed for Total Ammonium Nitrogen (TAN) and Total Kjeldahl Nitrogen (TKN) according to the Kjeldahl method.

Destruction of samples was performed on a Tecator™ Digestion Unit Auto Lift 20 and distillations were performed on a Büchi K355 distillation unit.

3.1.4 Volatile fatty acid analysis

Volatile fatty acids (VFA) were analysed on an GC (Shimadzu 2010) equipped with a capillary column (wax 0.53 mm ID, 30 m) and a FID detector.

3.1.5 Uric acid analysis

Uric acid was analysed using the method outlined in Pekic Chromatographica v.27 no. 9/10 p.467, 1989.

3.1.6 Measurements and analysis

The CH_4 content in the headspace of the bottles was measured by GC (Shimadzu 2010) equipped with a capillary column (wax 0.53 mm ID, 30 m) and a FID detector.

By a standard curve created by injection of various volumes of 100 % pure CH_4, the number of CH_4 molecules in the headspace were determined at regular intervals. Based on this the volumetric CH_4 production could be calculated during the test period. The biogas produced by the batches was released several times during the experiment in order to maintain low pressure in the bottles.

The specific methane yield (Nml methane per gram substrate VS added) is calculated by subtraction of background, normalizing to standard pressure and temperature (STP) and relating the yield to the quantity of VS added.

3.2 Experimental setup

An experiment typically consisted of a mixture of poultry litter, water or centrifugal liquid from an anaerobic digester and/or seeding material[2]. All substrate materials were homogenized and mixed in appropriate amounts in a suitable container. The final weight was registered and the mineralisation mix covered with parafilm. The mixture was then incubated at the appropriate temperature for varying time intervals and sampled either during or after incubation. Before sampling from the experiments, masses were registered and adjusted with water to compensate for evaporation.

4 Results

4.1 Substrate characterisation

The substrates used for the following experiments have all been characterised with respect to TS/VS, TAN/TKN, VFA and Uric acid content. The results may be seen in Table 2.

	Unit	Hen litter	Chicken litter	AD liquid
Total Solids	w/w%	46.5% (±0.7%)	63.6% (±1.2%)	7.5% (±<0.1%)
Volatile Solids	w/w%	33.9% (±0.1%)	54.2% (±1.4%)	4.3% (±<0.1%)
TAN	g N/kg sample	3.7 (± 0.3)	7.1 (± 0.9)	5.0 (± <0.1)
TKN	g N/kg sample	25.7 (± 0.4)	27.8 (± 0.9)	7.4 (± 0.2)
OrgN	g N/kg sample	22.0 (± 0.5)	20.7 (± 1.3)	2.4 (± 0.2)
VFA	ppm	3400 (± 580)	4000 (± 500)	49 (± 13)
Uric acid	g N/kg sample	12.7 (± 0.6)	7.1 (± 0.2)	ND[3]

Table 2 – Characterisation of the individual substrates used in the mineralisation setups.

[2] Seeding is a term that describes the addition of material from a previous mineralisation reaction.
[3] ND – Not detectable

4.2 Method optimisation

Preliminary experiments showed at an early stage that it was possible to shift the nitrogen pool in hen litter from the organic fraction to the inorganic fraction. However, the process required incubation times of up to 17 days. Hence, the method was optimised with respect to oxygen level, incubation temperature, seeding and water content. Meanwhile the reactions were monitored for development of various parameters, such as TAN, VFA and TS/VS.

4.2.1 Oxygen, temperature and seeding

The effect of oxygen on the reaction speed and final mineralisation yield was investigated by either flushing the mineralisation mixtures with N_2 (low oxygen level), O_2 (high oxygen level) or without flushing (normal oxygen level).There was no observable differences between low and normal oxygen levels with respect to mineralisation speed and ultimate yield. However, high oxygen levels significantly impaired mineralisation speeds (data not shown). The remaining experiments were thus performed with normal oxygen levels.

The effect of temperature on the reaction speed and final mineralisation yield was initially investigated at four different temperatures (see Figure 3). The choice of temperature intervals was based on the assumption that the bottle neck in the reaction might be the urease enzyme, which catalyses the conversion of urea to ammonia, a prerequisite of converting uric acid to TAN. Urease has an activity profile which increases steadily until it reaches an optimum around 68 °C after which it drops off sharply.

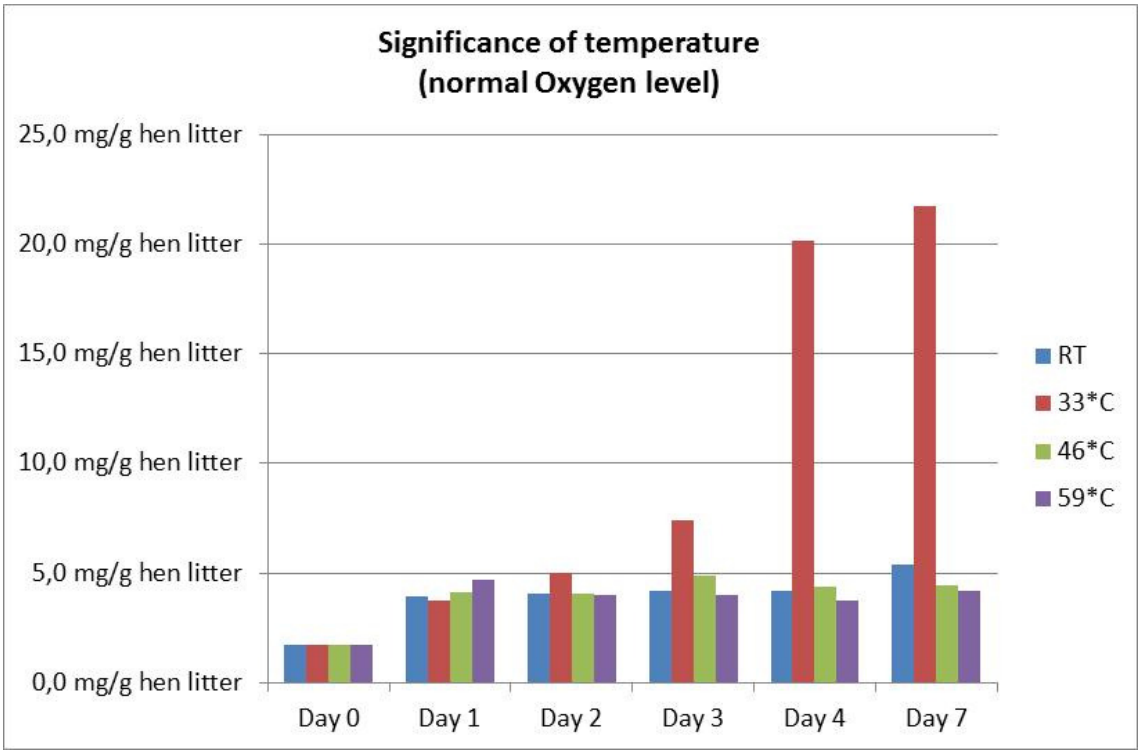

Figure 3 – Development of TAN. TS = 20%. No seeding. RT = Room temperature.

Figure 3 shows TAN development at normal oxygen levels. It is evident that 33 °C results in faster mineralisation speeds compared to the other temperatures tested. Within the time interval analysed, temperatures below or above 33 °C did not show any TAN development. Incubation at room temperature had previously been observed to produce TAN after 17 days of incubation, and might be expected to have reacted after a longer process time than 7 days in this experiment. The fact that no TAN development could be observed at temperatures above 33 °C indicates that the reaction bottle neck is not the urease enzyme.

The fact that the process is inhibited at high oxygen levels and that it has a temperature optimum around 33 °C, is a strong indication that the mineralisation process is biological. Chemical reactions are not likely to be inhibited by increasing oxygen levels, and would be expected to increase its speed with increasing temperatures.

To further narrow down the optimal temperature range, and considering that the microbial community in the hen litter stems from the intestines of hens, the physiological temperatures 30 °C and 37 °C were investigated.

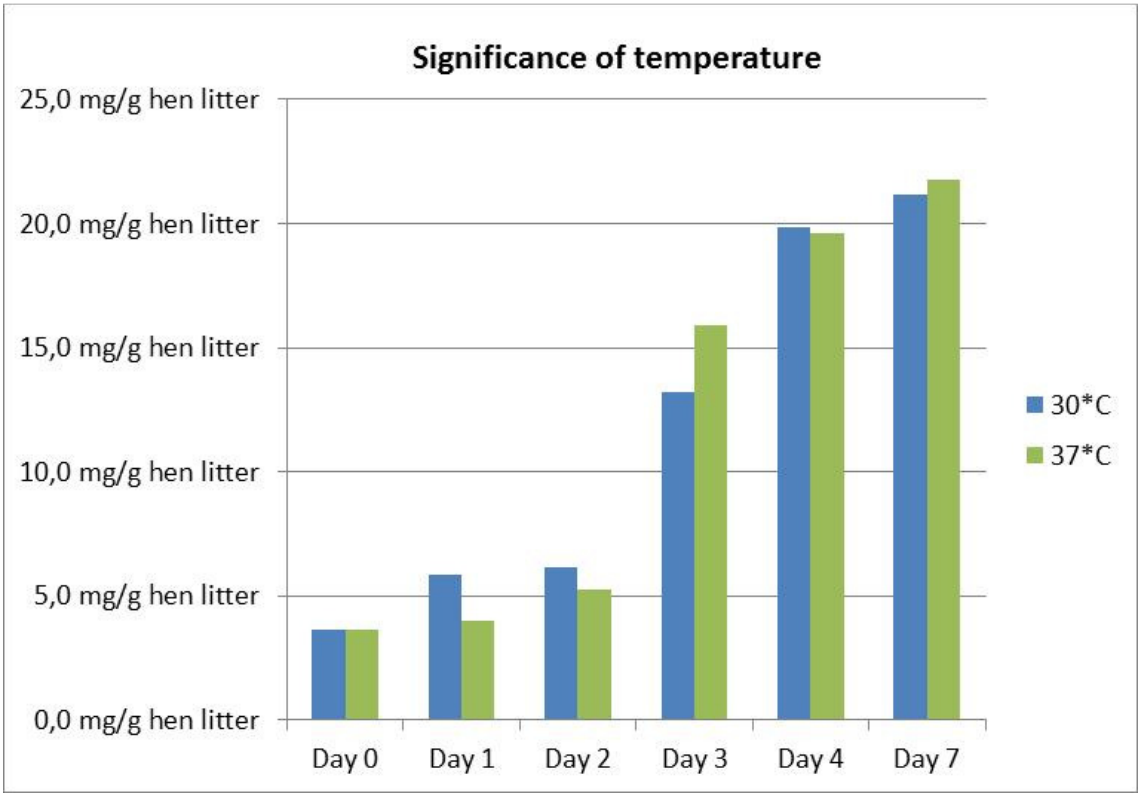

Figure 4 – Development of TAN at physiological temperatures. TS = 20%, normal oxygen levels.

The TAN development showed that the mineralisation process at both temperatures tested was comparable to 33 °C (compare Figure 3 and Figure 4). It is not possible to conclude if the process is faster at temperatures between 37 °C and 46 °C. However, since the process is likely microbiologically catalysed and most intestinal microbes have temperature optima around 37 °C it is likely that the process will not increase significantly beyond this temperature.

If the mineralisation process, as assumed, is catalysed by bacteria, these are likely to be dormant due to the high TS content of the hen litter. A certain time interval will thus be expected after hydration to activate their metabolism, causing a lag-phase before TAN development may be detected. Seeding of the sample with material from a previous mineralisation reaction should be expected to decrease the lag-phase, since the bacterial cultures in this material are actively growing and metabolising.

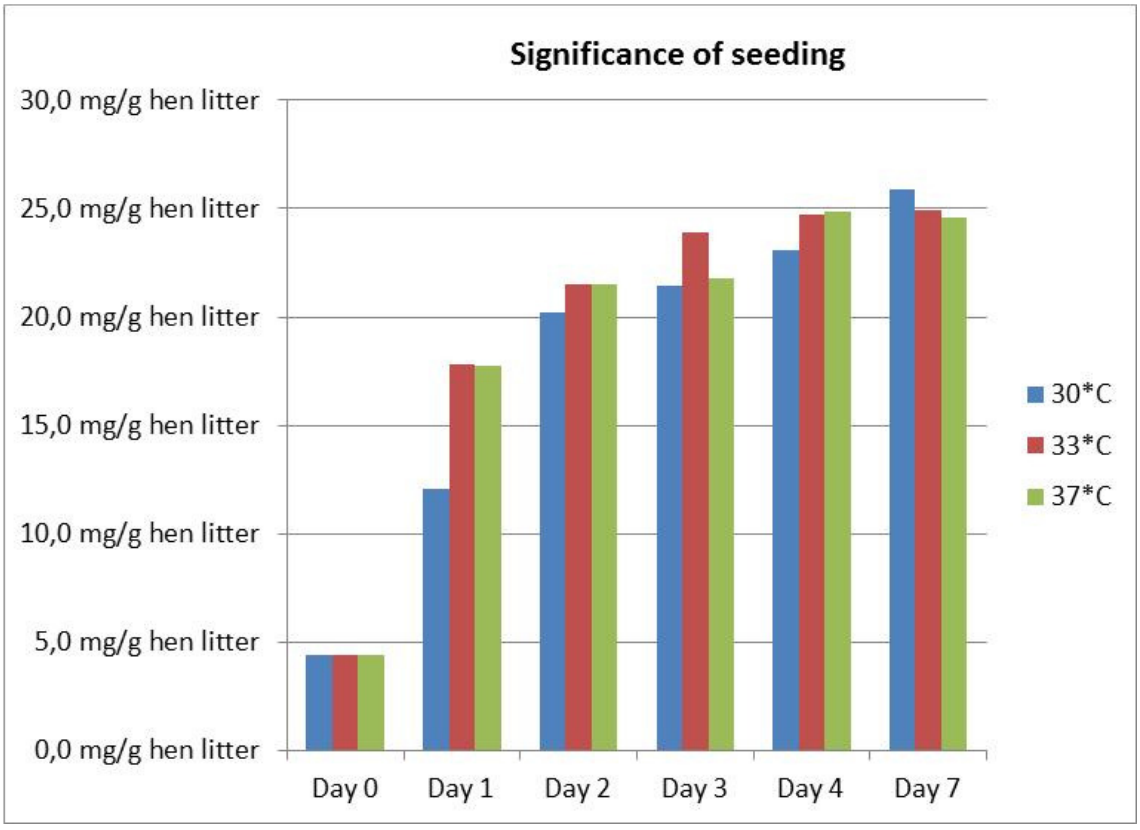

Figure 5 – Effect of seeding on the mineralisation process. TS = 20%. 15% of the final mineralisation mixture is seeding material. TAN from the seeding material has been subtracted.

Seeding of the mineralisation experiments with 20% material from previous incubations resulted in significant increases in reaction speed at all temperatures (compare Figure 4 and Figure 5). After 48 hours more than 90% of the final yield is obtained. The TAN development in this experiment is a little slower at 30 °C than at 33 °C and 37 °C.

4.2.2 Water content

To determine the influence of water content on mineralisation speed and final TAN yield, hen litter was mixed with water and mineralised at varying TS levels. Initial experiments showed no or very little activity above 30% TS in the initial mixture. The experiments here show the difference between mineralising at 20%, 25% and 30% TS.

Figure 6 shows that the variations in water content tested here only have a small influence on the mineralisation reaction. There is a small tendency that the TAN development is slower with higher TS contents, but the effect is not pronounced. The initial experiments that showed very little mineralisation taking place at TS levels higher than 30% were performed without addition of seeding material, and it is thus uncertain at which TS level mineralisation is significantly inhibited.

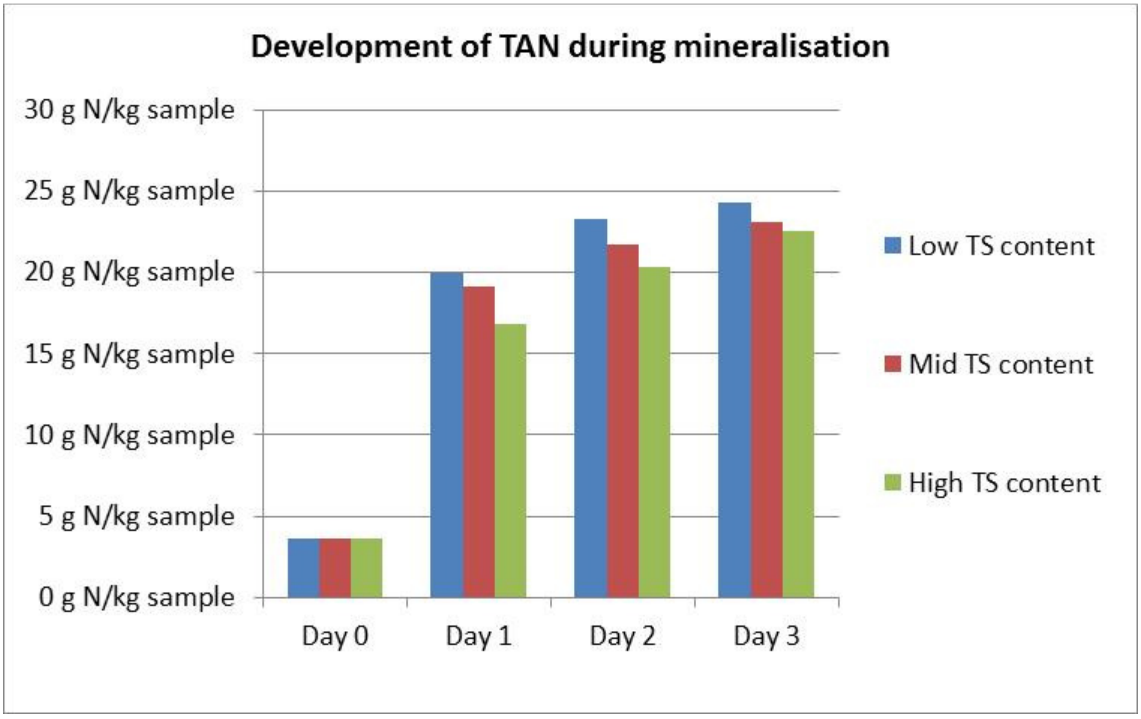

Figure 6 – Development of TAN during mineralisation. Blue bars: 20% TS initially in mineralisation setup – Red bars: 25% TS initially in mineralisation setup. Green bars: 30%TS initially in mineralisation setup.

4.2.3 Substrate specificity

All of the above experiments have been carried out using hen litter as substrate. However, as other poultry litters also contain large amounts of nitrogen the specificity of the mineralisation process was tested using chicken litter as substrate.

Figure 7 shows the development of TAN during mineralisation of chicken litter. The efficiency of the process is comparable to that in hen litter (compare Figure 6 and Figure 7), although the final yield is a little less in chicken litter. This is not surprising since the chicken litter used here contains significantly less nitrogen per gram TS than hen litter (see Table 2). Moreover, the chicken litter used here contains up to 8% bedding material, which is not expected to be mineralised to the same extent as the poultry faeces.

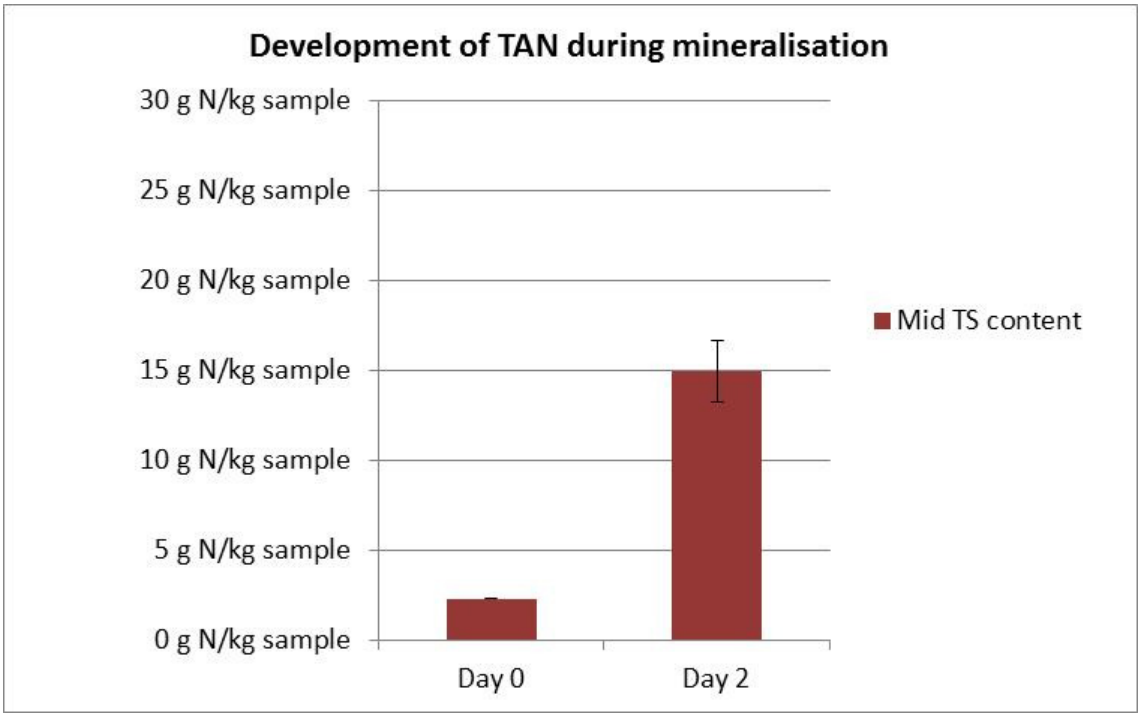

Figure 7 – Development of TAN in chicken litter during mineralisation. Red bars – intitial TS in mineralisation setup – 25%.

4.3 Method characterisation

4.3.1 Organic nitrogen

The organic nitrogen fraction in poultry litter is primarily made up of uric acid and proteins. Uric acid is a much smaller molecule than proteins, and could be expected to be easier to degrade. Analysis of the uric acid and protein content were made on untreated and mineralised hen litter to determine any differences in degradation patterns for the two fractions.

From Figure 8 it can be seen that uric acid is 100% degraded while protein bound nitrogen is degraded by approximately 40%. On average total mineralisation levels range from 71% - 80%.

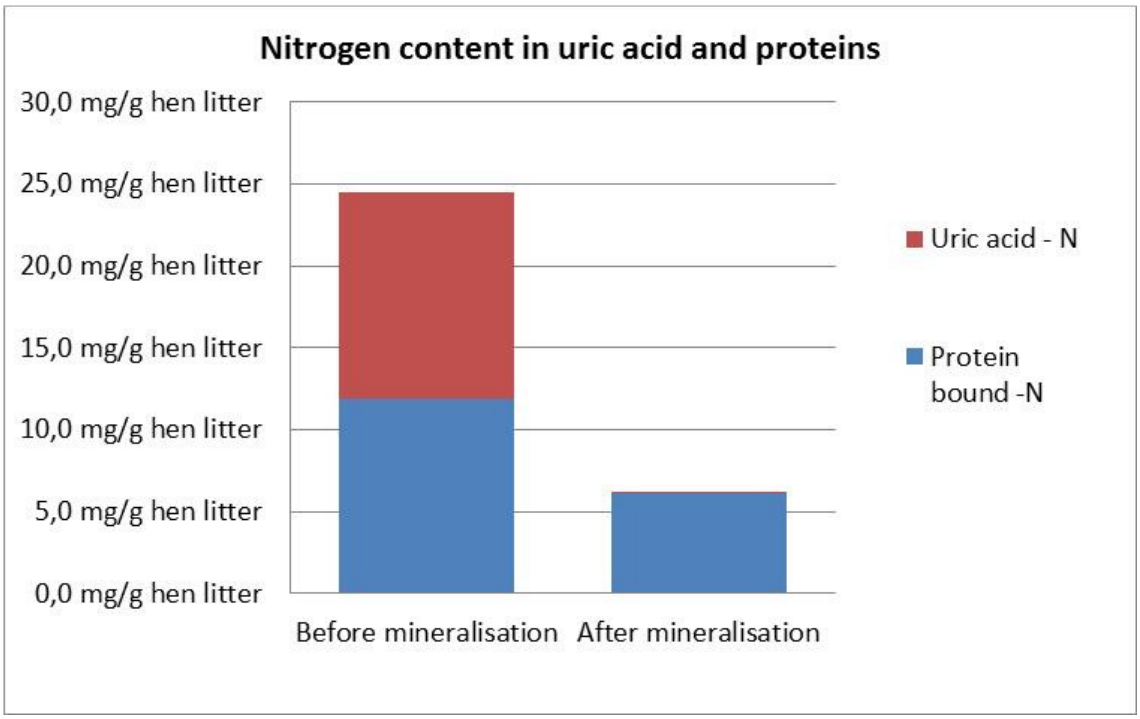

Figure 8 – Content of uric acid bound nitrogen and protein bound nitrogen before and after mineralisation.

4.3.2 Volatile Fatty Acids

VFA's are breakdown products from hydrolytic action on carbohydrates, protein and fat. Acetate is also produced during anaerobic breakdown of uric acid. Since the mineralisation reaction is facilitated by bacterial degradation of OrgN, the TS and VS content is monitored to see how much is lost due to microbial metabolism (see figure 9).

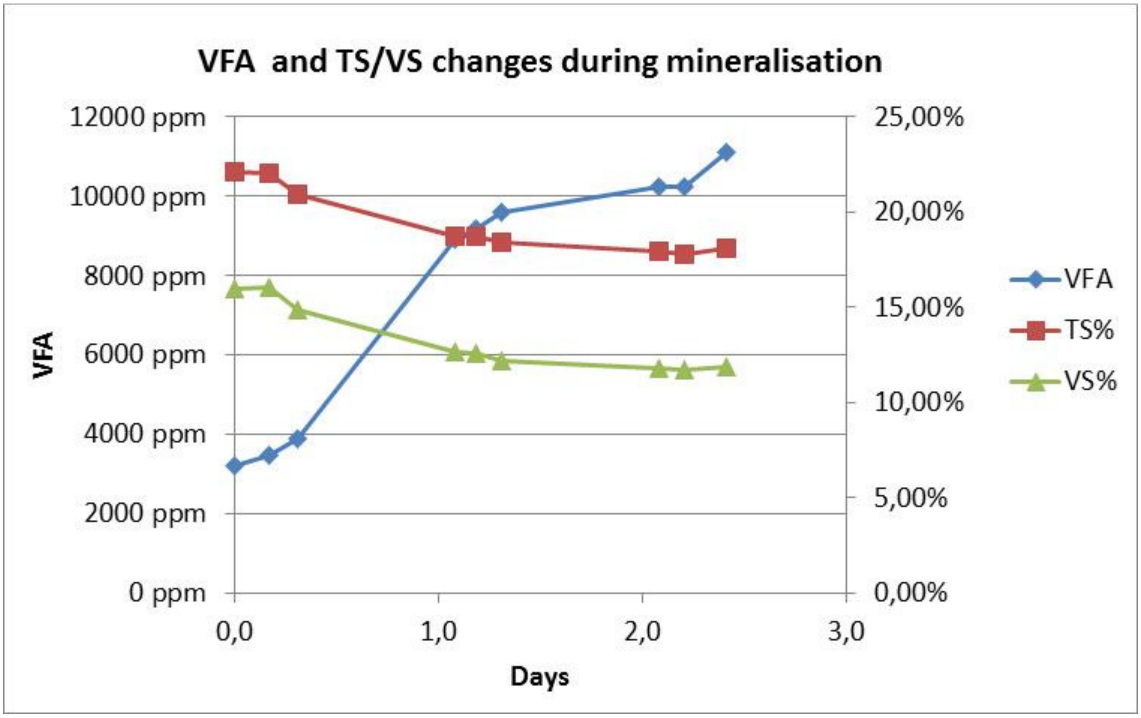

Figure 9 – Total VFA and TS/VS changes during incubation.

There is a significant production of total VFA's during the mineralisation process. In particular acetate is increased during incubation but also propionate and butyrate show significant increases (data not shown). There is a significant TS and VS loss during incubation.

4.3.3 Methane and CO_2 production

The observed VS loss during incubation (see Figure 9), made it interesting to investigate the composition of the gas being produced during mineralisation.

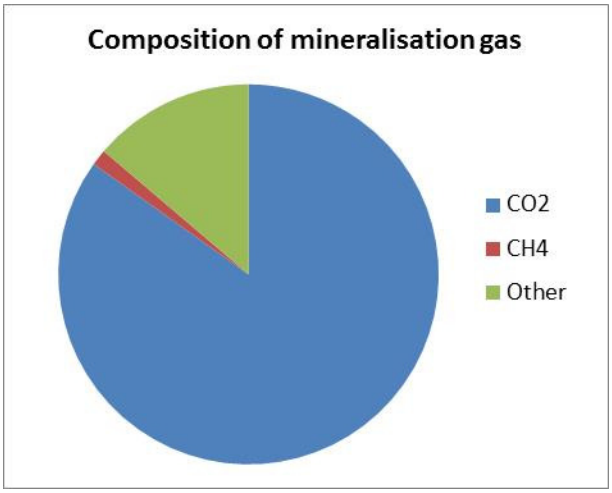

Figure 10 – Relative amounts of CO_2 and CH_4 in the gas produced during mineralisation.

From Figure 10 it can be seen that the main constituent of the produced gas is CO_2. Methane only accounts for a small percentage (~1%) of the entire gas production. Methane production occurs solely during anaerobic digestion, whereas CO_2 production is a typical product of aerobic digestion. This is a strong indication that the microbial metabolism leading to gas production is aerobic. It should be noted that ~14% of the mineralisation gas cannot be accounted for by either methane or CO_2 (green segment "Other" in Figure 10). The unknown gas is likely residual nitrogen or hydrogen, which is known to be produced during similar hydrolysis reactions. Neither compound can be detected with the methods available to our lab.

5 Conclusion and discussion

It has been shown that OrgN in poultry litter may be converted to TAN using a biologically facilitated mechanism of mineralisation. The optimal parameter settings determined in these experiments may be seen from Table 3.

Parameter	Value
Oxygen levels	Atmospheric or below
Temperature	33 ℃ - 37 ℃
TS content	Below 30%
Seeding	10-15% (w/w) of the total mass

Table 3 – Optimal parameters for mineralisation of poultry litter

When using these settings we have shown that OrgN from hen litter can be converted into TAN with an efficiency of 70%-80%. We have also shown that mineralisation is also possible on chicken litter with comparable efficiencies.

Parallel to the degradation of OrgN (as seen by the production of VFA's), there is a concurrent TS and VS loss of 20%-25% of the VS pool. The fact that the loss is observed in both the TS and VS measurements indicate that VS is converted into a volatile compound.

An explanation for this could be that the metabolised VS is converted into CO_2 or another gaseous compound and emitted from the mixture, which would result in a drop in both the VS and TS fractions. However, one discrepancy in this explanation is a difference between the amount of collected gas during mineralisation and the expected gas production due to the VS loss. In an experiment like the one referred to in Figure 9, the measured VS loss can be calculated. In this case, conversion of VS to CO_2 is expected to yield ~4 liters of gas. However, the measured gas production is only about 0.8 – 1 liters.

If the VS is not converted into gas, but is lost during VS analysis, it can only be caused by production of volatile compounds, which evaporate during drying. The procedure for TS/VS measurements already takes into account the production of VFA's, which evaporate during drying, but there may be other compounds which we do not routinely measure (e.g. alcohols and aromatic compounds). Clearly more investigations are needed to clarify if the VS loss during mineralisation is real or an artefact due to evaporation of volatile organic compounds during TS/VS analysis.

Annex 3 Screening and BMP Analysis of Cattle Deep Litter (WP 6)

Project No.:	EUDP j.nr. 64011-0335 /Internal project 10842
Made by:	SBG
Issued:	December 3 2013

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1 Abbreviations

AD	-	Anaerobically digested
BMP	-	Biomethane potential
CSTR	-	Continously stirred tank reactor
HRT	-	Hydraulic retention time
KFC	-	Kvægbrugets Forsøgscenter (The Cattle Associations Research Center)
N	-	Nitrogen
NmL	-	Normal mL (volume at 1 bar and 273 K)
OrgN	-	Organic Nitrogen
PC	-	Pressure cooking
RSD	-	Relative standard deviation
SD	-	Standard deviation
TAN	-	Total Ammonium Nitrogen
TKN	-	Total Kjeldahl Nitrogen
TS	-	Total Solids
VS	-	Volatile Solids
w/w%	-	Weight percentage

2 Summary and Conclusion

This analysis report summarizes experiments performed on deep litter from cows.

The purpose of the investigations was to determine the

1. Biomethane potential (BMP) value of
    a. Deep litter from bull calves[1]
    b. A mixture of deep litter from dairy farms[1]
2. Effect of extrusion on the BMP value of litter from bull calves
3. Effect of NiX pre-treatment on the BMP value of bull calves and dry cows/calves.

Deep litter was obtained from three different suppliers of bull calves, and three different dairy farmers. Due to the inherent heterogeneity of deep litter, all deep litter samples were homogenised using a Biomixer (Pumpe), prior to sampling smaller portions used for each experiment. The results below are a result of three different BMP assays. Due to natural variance between assays the effects of the tested treatments should always be compared to the accompanying untreated sample. The main results from the experiments are summarised in Table 1 to Table 3.

	BMP setup	Units	Laursen	Hindbo	Korsholm
Deep litter type		NmL CH_4/g VS	Bull calves	Bull calves	Bull calves
BMP (untreated)	1	NmL CH_4/g VS	274 (±7) *88 (±3)*	258 (±6) *76 (±3)*	288 (±6) *117 (±3)*
BMP (extruded)	1	NmL CH_4/g VS	264 (±5)	260 (±4)	281 (±4)
BMP (untreated)	2	NmL CH_4/g VS	*Not included*	232 (±6) *68 (±2)*	273 (±6) *110 (±3)*
BMP (NiX treated)	2	NmL CH_4/g VS	*Not treated*[2]	272 (±5)	341 (±5)
TS			32% (±0.5)	29% (±0.3)	40% (±1.5)
VS			28% (±0.3)	26% (±0.3)	37% (±1.5)
Total Ammonium N		g N/kg sample	2.8 (±0.1)	3.5 (±0.1)	1.9 (±0.1)
Total Nitrogen		g N/kg sample	8.3 (± 0.4)	7.7 (± 0.1)	7.7 (± 0.6)
Organic Nitrogen		g N/kg sample	5.5 (± 0.4)	4.2 (± 0.1)	5.8 (± 0.6)

Table 1 – Overview of key results from BMP and TS/VS analysis of untreated and pre-treated deep litter from bull calf suppliers.

[1] Bull calves are from 0-12 months of age and bred for slaughter. The dairy farms produce deep litter from calves (0-5 months of age), dry cows (dairy cows that have stopped lactating) and/or heifers.

[2] Laursen was not NiX treated. See section 4.3.1 for details.

- The methane yields for untreated deep litter from bull calves suppliers in the first BMP set-up ranged from 258 – 288 NmL CH_4/g VS.
- Extrusion had no significant effect on the BMP yield or the digestion speed (see Table 11)
- The methane yields for untreated deep litter from bull calves suppliers in the second BMP analysis ranged from 232 – 273 NmL CH_4/g VS. The age of the deep litter may be a determining factor in BMP quality of the deep litter. This final yields reached in this setup are a little lower than the value obtained in the previous BMP analysis (see Table 1). The reason for this is likely natural variance between samples and BMP setups.
- NiX treatment resulted in an increase in final BMP yield of 17%-25%, and a 50% increase in digestion rate (see Table 12)
- NiX treatment without addition of burnt lime had no effect on the BMP yield (data from the first BMP analysis – data not shown)

	BMP setup	Units	KFC	Jacobsen	Olesen
Deep litter type			Dry cows/calves/ heifers	Dry cows/calves	Dry cows/calves
BMP (untreated)	3	NmL CH_4/g VS	225 (±7) *58 (±3)*	313 (±7) *69 (±6)*	298 (±6) *70 (±5)*
BMP (NiX treated)	3	NmL CH_4/g VS	291 (±5)	329 (±4)	340 (±5)
TS			26% (± 0.5)	22% (± 0.6)	23% (± 0.3)
VS			23% (± 0.5)	19% (± 0.5)	19% (± <0.3)
Total Ammonium Nitrogen		g N/kg sample	0.6 (± <0.1)	1.3 (± <0.1)	2.1 (± <0.1)
Total Nitrogen		g N/kg sample	5.2 (± 0.4)	4.6 (± 0.2)	5.1 (± <0.1)
Organic Nitrogen		g N/kg sample	4.6 (± 0.4)	3.3 (± 0.2)	3.0 (± 0.2)

Table 2 – Overview of key results from BMP and TS/VS analysis of untreated and NiX treated deep litter from dairy farmers.

- The methane yields for untreated deep litter from dairy farmers ranged from 225 – 313 NmL CH_4/g VS.
- NiX treatment resulted in an increase in final BMP yield of 5%-30%, and a 30% - 70% increase in digestion rate (Table 14).

The expected methane yield, when digesting the treated and untreated samples in a two-stage CSTR setup, may be seen in Figure 2 and Figure 3. The calculation combines the digestion speeds and final methane yields obtained in batch with the setup of the CSTR system to predict the methane production.

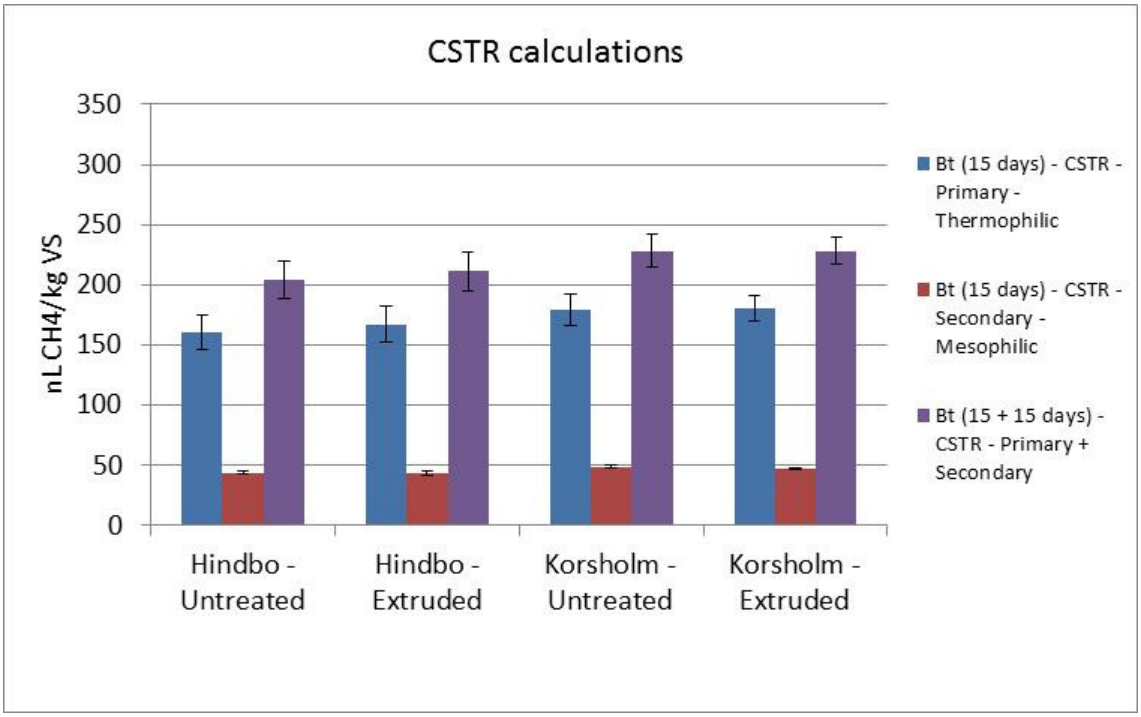

Figure 1 – Expected methane yields of deep litter from bull calves in a CSTR setup based on results from the first BMP setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

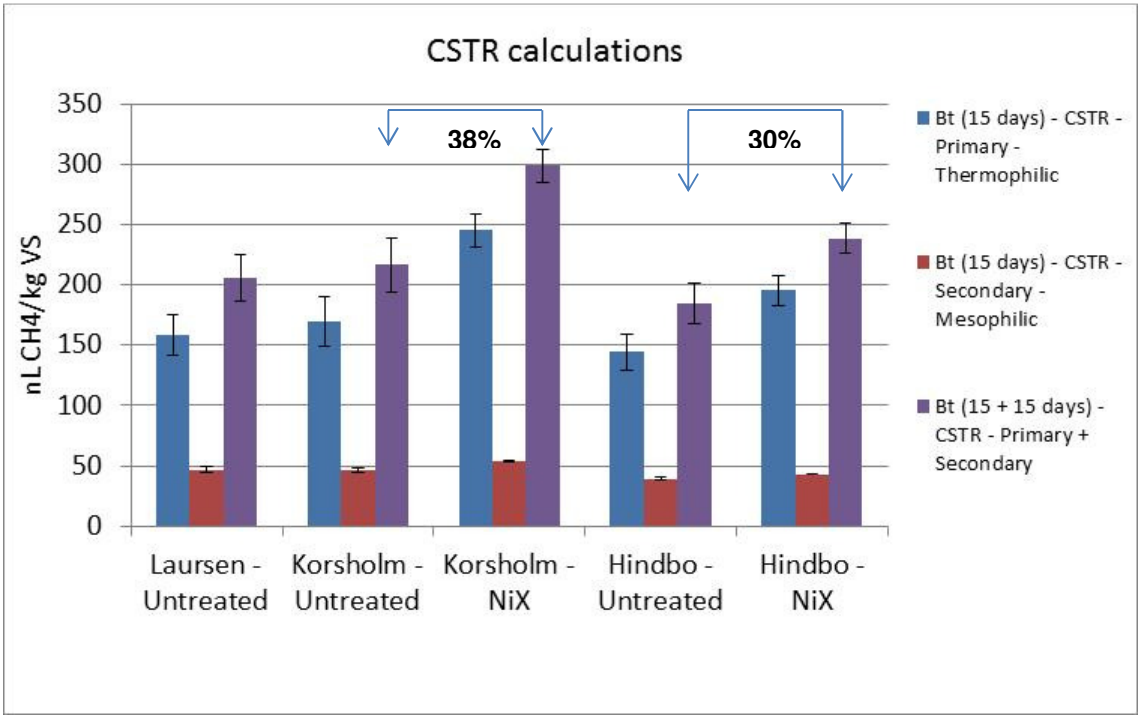

Figure 2 – Expected methane yields of deep litter from bull calves in a CSTR setup based on results from the second BMP setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

When comparing the deep litter from bull calves, it can be seen that extrusion has no significant effect on the expected methane yield in a CSTR type anaerobic digester (see Figure 2). NiX treatment however, is expected to increase the methane yield by 30%-38% in a two-stage thermophilic/mesophilic system with a 15 days retention time in each digestion step (see Figure 3).

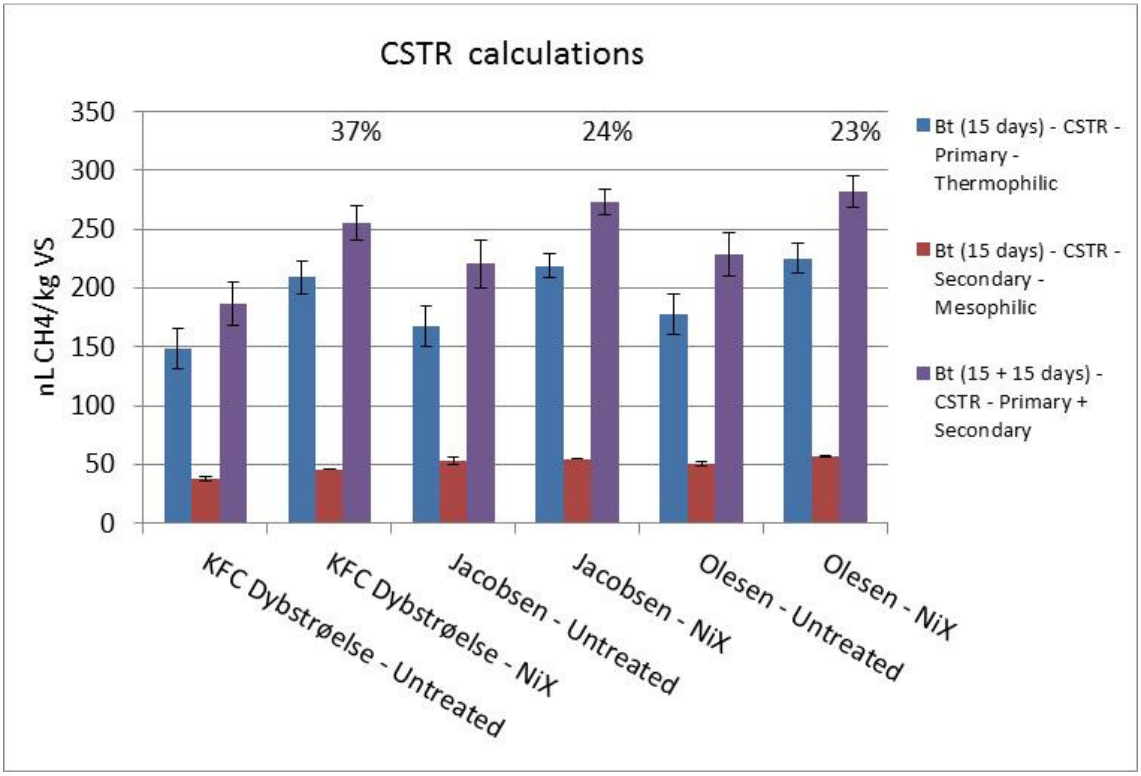

Figure 3 – Expected methane yields of deep litter from dairy farmers in a CSTR setup based on results from the third BMP setup. B_t=Methane yield at time t. Blue bar: Methane yield in a primary thermophilic reactor with 15 days retention time. Dark-red bar: Methane yield in a secondary mesophilic reactor also with a 15 day retention time. Purple bar: Total methane yield in both reactors.

The methane yield from NiX treated deep litter from dairy farmers ranges from 23%-37% as seen from Figure 3. Deep litter from KFC is a mixture between litter from heifers, dairy cows and calves, whereas the litter from Jacobsen and Olesen is a mixture of litter from dairy cows and calves. It can not be concluded from this study whether the higher improvement on the KFC litter is due to the heifer fraction or some other factor such as e.g. the lower batch BMP value or some other factor. More experiments will be needed to conclude this.

The effect of NiX treatment on methane yield is amplified in the continuous setup because of the effect on digestion speeds. This effect is more pronounced at shorter retention times and becomes less evident with increasing retention times. Raising the HRT of the system will thus decrease the difference in methane yield between untreated and treated deep litter.

3 Materials and Methods

3.1 Substrate Characterization

3.1.1 Dry Matter

Total solids (TS) and volatile solids (VS = organic dry matter) contents of the deep litter were determined in triplicate prior to BMP analysis. TS were determined by heating the samples to 105 °C for a minimum of 24 hours. VS were determined by burning the samples at 550 °C for 3 - 4 hours.

3.1.1 Substrate information – deep litter from suppliers of bull calves

Substrate name	**Laursen**	**Hindbo**	**Korsholm**
Substrate description	Deep litter. Bedding used is app. 75% barley, 25% wheat.	Deep litter. Bedding used is app. 1/3 barley, 2/3 wheat.	Deep litter. Bedding used is app.50% barley, 50% wheat.
Production type	Bull calves. Deep litter height at time of sampling app. 1-1.5 m.	Bull calves. Deep litter height at time of sampling app. 1-1.5 m.	Bull calves. Deep litter height at time of sampling app. 1-1.5 m.
Sample date	August 2011	October 2011	September 19th 2012
Substrate age (at time of sampling)	Deep litter age at mucking ~6 months. The sample came from two week old pile outside the stable.	Deep litter age at mucking 7-8 months. Sampled from a pile outside the stall. The pile was continuously supplied with deep litter from stables.	Deep litter age at mucking ~4 months. Sampled straight from the stable.
Sampled by	Laursen	Hindbo	Korsholm/SBG
Supplier information	Henry Laursen Lundgårdvej 2 6900 Skjern Phone: +4520784327 Email: lundgaard2@hotmail.com	Henrik Hindbo Holkvej 7, Bølling 6900 Skjern Phone:+45 97368444/ +4540286070 E-mail: h.hindbo@post.tele.dk	Sven Erik Korsholm Herningvej 118 Ringkøbing Phone: +4520143151 henriettesv@hotmail.com

Table 3 – Substrate, sampling and supplier information

3.1.2 Substrate information – deep litter from dairy farmers

Substrate name	KFC	Jacobsen	Olesen
Substrate description	Litter from dry cows, heifers and young calves (0-6 months) in quantities proportional to the yearly production of each type. Ratios (based on weight) were 2.5:5:2.5 respectively.	Litter from dry cows and young calves (0-6 months) in quantities proportional to the yearly production of each type. Ratios (based on weight) were 1:2 respectively	Litter from dry cows and young calves (0-6 months) in quantities proportional to the yearly production of each type. Ratios (based on weight) were 2:1 respectively.
Production type	Dairy cows on slits. Dry cows on deep litter. Calves on litter. Heifers on deep litter.	Dairy cows on slits. Dry cows on deep litter. Calves on litter.	Dairy cows on slits. Dry cows on deep litter. Calves on litter.
Sample date	December 2012	January 2013	January 2013
Substrate age (at time of sampling)	Sampled directly from stable	Sampled directly from stable	Sampled directly from stable
Sampled by	KFC/SBG	Jacobsen	Olesen
Supplier information	Kvægbrugets Forsøgscenter Forskningscenter Foulum Burrehøjvej 49 Boks 50 8830 Tjele Phone: 87991500	Henning Jacobsen Højbjerg Møllevej 21 Rødkærsbro Phone: +4551740007	Niels Olesen Overgårdsvej 9, Mollerup, 8830 Tjele Phone: +4522821065

Table 4 – Substrate, sampling and supplier information

3.2 Extrusion

A twin-screw extruder with two counter-rotating screws (Bio-extruder MSZ-B74e) was used for this experiment. The extruder is driven by two 37 kW motors. The extruder was cleaned by addition of app. 3-4 m^3 deep litter. Samples were collected at the outlet opening and stored at -12 °C until analysis.

3.3 NiX treatment

The Nix technology consists of a thermochemical treatment of the substrate. Substrate was mixed with burnt lime (CaO) and water to a final concentration of 1.5 wt.-% burnt lime and 30 % TS, prior to subjecting the mixture to elevated temperatures and pressure. The treatment was performed in a pilot scale pressure cooker in which saturated water steam was used to raise the pressure to 4

barg and the temperature to 146 ℃. After treatment pressure was released over a period of 20-30 minutes. Samples were collected and processed according to the flowchart below.

Pres-sure	Tempera-ture	Pressure hold time	Pressure release time	Sample TS	Sample VS	pH after treatment
4 barg	146 ℃	20 min	20-30 min.	20%	7,3%	>11

Table 5 – Pressure cooking parameters.

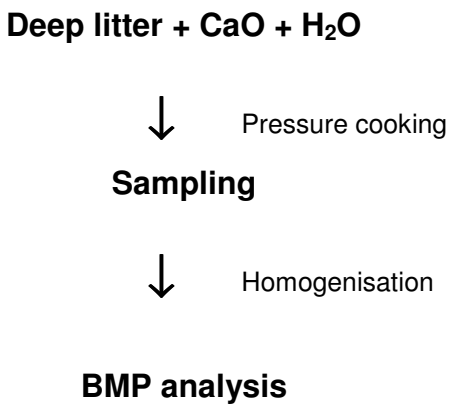

Figure 4 – Overview of treatment and analysis flow

Figure 5 – Pilot scale pressure cooker

Two independent NiX treatments were performed on each substrate. Samples were collected after treatment and stored at -12 ℃ until analysis.

3.4 Biological Methane Potential (BMP) Test

3.4.1 BMP assay

The BMP assay was carried out according to the German VDI4630 protocol for analysis of methane potentials in agricultural biomasses with minor modifications. The batches were prepared in 500 ml infusion glass bottles. The inoculum was taken from the thermophilic main digester of Foulum biogas plant and incubated at 52 ± 1 ℃ for 10-14 days before substrate addition in order to

minimize the relative contribution from the inoculum to the total gas production. 200 ml of inoculum were used per bottle.

To avoid inhibition due to organic overloading, the BMP assay was carried out at two different concentrations of deep litter. Each batch bottle was prepared by addition of either 1 or 2 g VS followed by addition of 200 ml inoculum. Resulting substrate VS concentration in each of the substrate batch bottles was 5 and 10 g substrate VS/L inoculum respectively. For each of two different substrate concentrations 3 replicates were incubated.

Figure 6 - Batch bottles in heat cabinet

For examination of inoculum quality, 6 bottles of 1.0 g cellulose per 200 ml inoculum were incubated (positive controls). For determination of the contribution from the inoculum to the CH_4 production 6 control bottles of 200 ml inoculum were also incubated (blanks).

After addition of inoculum and substrate all bottles were flushed with N_2 and closed with gas tight rubber stoppers and aluminium screw lids before incubation at 52 ± 1 ℃ in a heat cabinet for the duration of the batch test.

3.4.2 Measurements and analysis

The CH_4 content in the headspace of the batch bottles was measured by GC (Shimadzu 2010) equipped with a capillary column (wax 0.53 mm ID, 30 m) and a FID detector.

By means of a standard curve created by injection of various volumes of 100 % pure CH_4, the number of CH_4 molecules in the headspace was determined at regular intervals. Based on this the volumetric CH_4 production could be calculated during the test period. The biogas produced by the batches was released several times during the experiment in order to maintain low pressure in the bottles.

The specific methane yield (Nml methane per gram substrate VS added) is calculated by subtraction of background, normalizing to standard pressure and temperature (STP) and relating the yield to the quantity of VS added.

4 Results

4.1 TS and VS analysis

TS and VS contents of the deep litter were determined prior to NiX treatment and BMP analysis.

	Units	Laursen	Hindbo	Korsholm
TS	w/w %	32% (± 0.5)	29% (± <0.3)	40% (± 1.5)
VS	w/w %	28% (±<0.3)	26% (±<0.3)	37% (± 1.5)

Table 6 – TS and VS levels of deep litter from suppliers of bull calves.

	Units	Laursen	Hindbo	Korsholm
TS	w/w %	26% (± 0.5)	22% (± 0.6)	23% (± 0.3)
VS	w/w %	23% (± 0.5)	19% (± 0.5)	19% (± <0.3)

Table 7 – TS and VS levels of deep litter from dairy farmers.

4.2 Nitrogen analysis

The deep litter was analysed for total ammonium nitrogen (TAN) and total nitrogen (TKN). Table 8 and Table 9 below, shows the content of TAN and TKN in the substrate. Organic nitrogen (OrgN) is calculated by subtraction of TAN from TKN.

	Units	Laursen	Hindbo	Korsholm
Total Ammonium N	g N/kg sample	2.8 (± <0.1)	3.5 (± <0.1)	1.9 (± 0.1)
Total Nitrogen	g N/kg sample	8.3 (± 0.4)	7.7 (± 0.1)	7.7 (± 0.6)
Organic Nitrogen	g N/kg sample	5.5 (± 0.4)	4.2 (± 0.1)	5.8 (± 0.6)

Table 8 – Overview of key results associated with the analysis of deep litter from bull calves. Organic nitrogen is calculated by subtraction of total ammonium nitrogen from total nitrogen.

	Units	KFC	Jacobsen	Olesen
Total Ammonium N	g N/kg sample	0.6 (± <0.1)	1.3 (± <0.1)	2.1 (± <0.1)
Total Nitrogen	g N/kg sample	5.2 (± 0.4)	4.6 (± 0.2)	5.1 (± <0.1)
Organic Nitrogen	g N/kg sample	4.6 (± 0.4)	3.3 (± 0.2)	3.0 (± 0.2)

Table 9 – Overview of key results associated with the analysis of deep litter from dairy farmers. Organic nitrogen is calculated by subtraction of total ammonium nitrogen from total nitrogen.

4.3 BMP analysis

Specific methane yields obtained during the BMP analysis of untreated and treated deep litter are shown in Figure 7 – Figure 10. The specific methane yield (mL methane per gram substrate VS added) is calculated by subtraction of background and normalizing according to VS concentration.

4.3.1 Deep litter from suppliers of bull calves

Deep litter from bull calves was analysed for BMP in two separate setups. The first setup included Laursen, Hindbo and Korsholm deep litter. All deep litters were extruded and one (Korsholm) was also NiX treated. Due to technical problems the NiX treated samples were discarded, and another BMP assay was carried out, this time with NiX treatment of two deep litter samples (Hindbo and Korsholm).

	BMP setup	Units	Laursen	Hindbo	Korsholm
BMP (untreated)	1	NmL CH_4/g VS	274 (±7)	258 (±6)	288 (±6)
BMP (with extrusion)	1	NmL CH_4/g VS	264 (±5)	260 (±4)	281 (±4)
BMP (with NiX treatment)	1	NmL CH_4/g VS	Not included	Discarded	Discarded
BMP (untreated)	2	NmL CH_4/g VS	Not included	232 (±6)	273 (±6)
BMP (with NiX treatment)	2	NmL CH_4/g VS	Not included	272 (±5)	341 (±5)

Table 10 – Summary of BMP values obtained in the two different BMP setups.

As seen from Table 10 the second BMP setup has slightly lower BMP values compared to the first setup (13-25 NmL CH4/g VS). This variation is within what is generally accepted for BMP assays. Results from the first BMP setup may be seen in Figure 7 – Figure 9, and in Figure 10 for the second BMP setup. In all graphs data points represent empirical data, and solid lines represent the best-fit curve.

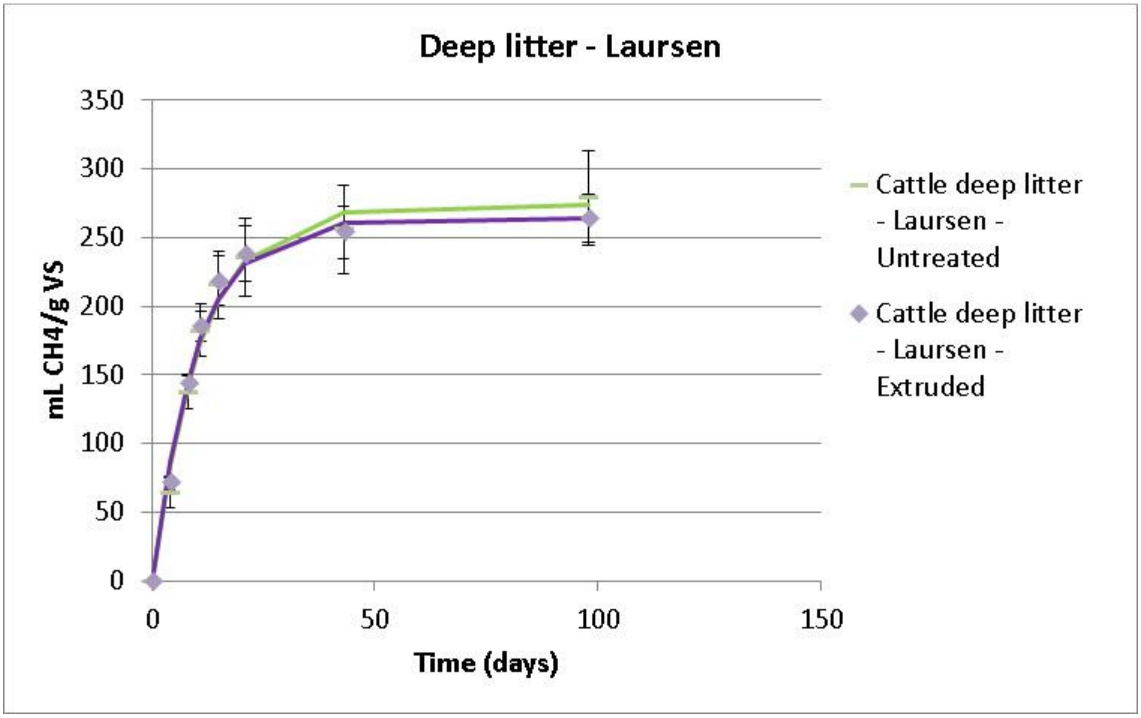

Figure 7 – Accumulated methane yield from BMP setup 1. Untreated and extruded deep litter from Laursen. Errorbars = +/- 1xSD.

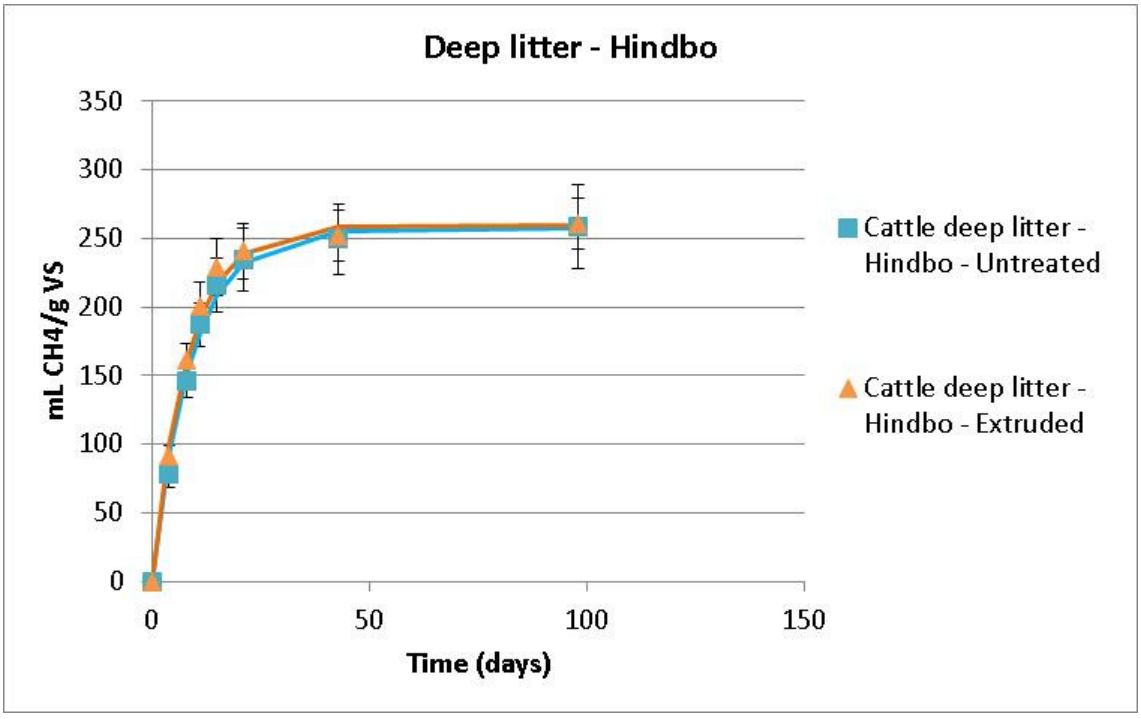

Figure 8 – Accumulated methane yield from BMP setup 1. Untreated and extruded deep litter from Hindbo. Errorbars = +/- 1xSD.

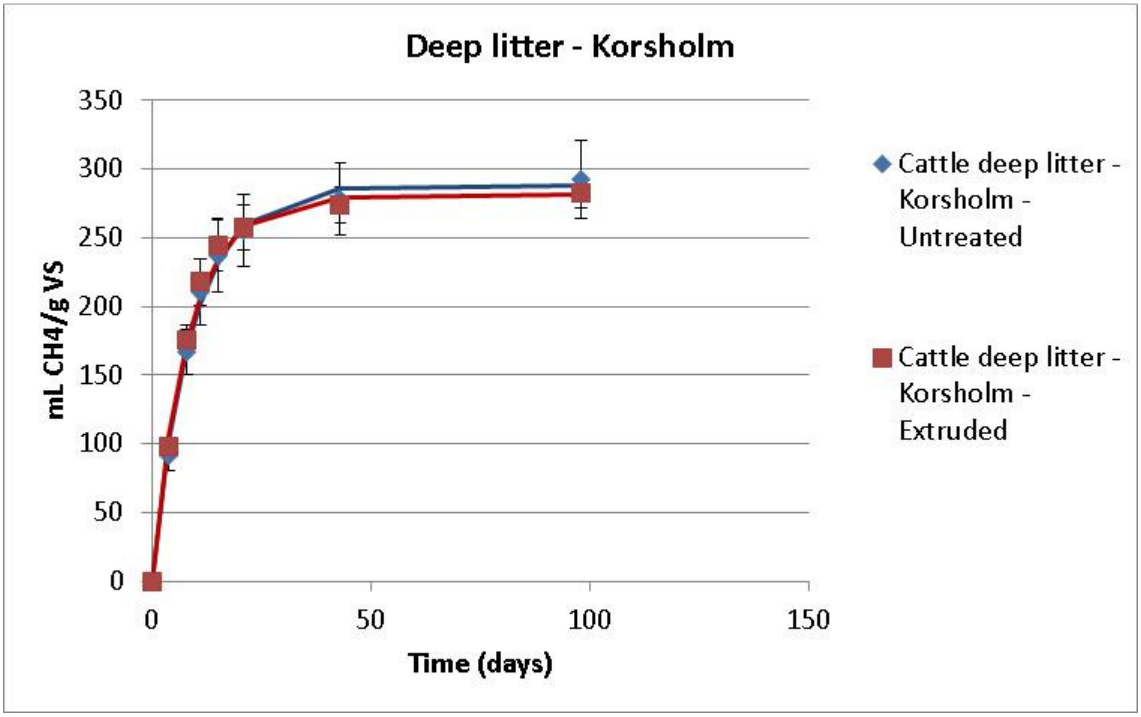

Figure 9 – Accumulated methane yield from BMP setup 1. Untreated and extruded deep litter from Korsholm. Errorbars = +/- 1xSD.

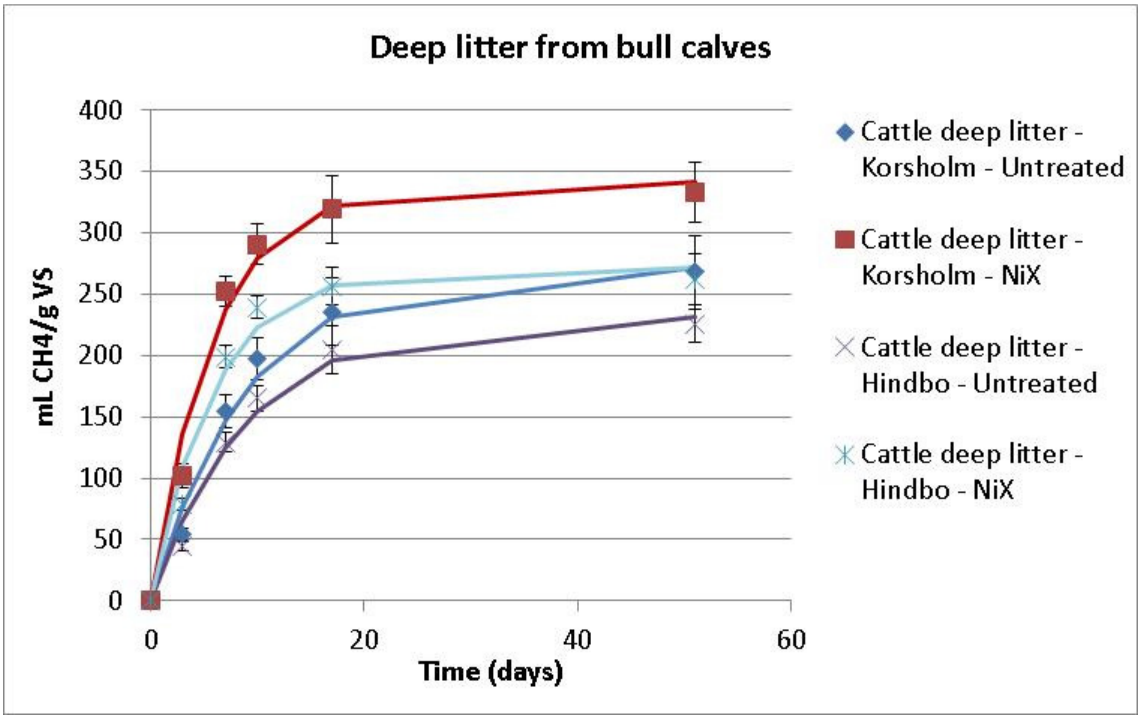

Figure 10 – Accumulated methane yield from BMP setup 2. Untreated and NiX treated deep litter from Korsholm and Hindbo. Errorbars = +/- 1xSD.

All untreated deep litter samples have the same digestion rate (compare k-values in Table 12). However, the final BMP yield varies between 232 for Hindbo and 273/274 NmL CH_4/g VS for Korsholm and Laursen, respectively. The reason for the lower BMP value for Hindbo may partly be explained by experimental variations. However, the most likely explanation may be that the Hindbo deep litter was the oldest litter of the three tested, and has likely been more composted than the two other deep litter types.

	Laursen - Untreated	**Laursen - Extruded**	**Hindbo - Untreated**	**Hindbo - Extruded**	**Korsholm - Untreated**	**Korsholm - Extruded**
BMP - First order equation						
Best-fit values						
B	274	264	258	260	288	281
K	0,091	0,10	0,11	0,12	0,11	0,12
Std, Error						
B	7,2	4,5	6	4,4	6	3,7
K	0,0066	0,0051	0,0069	0,0067	0,0069	0,0052
95% Confidence Intervals						
B	259 to 288	255 to 274	247 to 269	251 to 269	276 to 300	273 to 289
K	0,078 to 0,10	0,092 to 0,11	0,095 to 0,12	0,11 to 0,14	0,096 to 0,12	0,11 to 0,13
Goodness of Fit						
Degrees of Freedom	46	46	46	46	46	46
R^2	0,94	0,97	0,95	0,97	0,95	0,98
Absolute Sum of Squares	26413	11438	17573	12212	21180	8847
Sy,x	24	16	20	16	21	14
Constraints						
B	B > 0,0	B > 0,0	B > 0,0	B > 0,0	B > 0,0	B > 0,0
K	K > 0,0	K > 0,0	K > 0,0	K > 0,0	K > 0,0	K > 0,0
Number of points						
Analyzed	48	48	48	48	48	48
Outliers (excluded, Q=1,0%)	0	0	0	0	0	0

Table 11 – Non-linear best-fit analysis of untreated and extruded deep litter from suppliers of bull calves

There is a nice correlation between deep litter age and BMP value (see Table 3 and Table 10). If the amount of TAN present in the litter may be taken as an indication of decomposition, due to conversion of OrgN to TAN, this also corroborates the picture of a generally more decomposed deep litter in Hindbo.

	Laursen – Untreated	**Korsholm – Untreated**	**Korsholm - NiX**	**Hindbo - Untreated**	**Hindbo - NiX**
BMP - First order equation					
Best-fit values					
B	274	273	341	232	272
k	0,091	0,11	0,17	0,11	0,17
Std, Error					
B	7,2	8,40	5,00	6,00	4,10
k	0,0066	0,01	0,01	0,01	0,01
95% Confidence Intervals					
B	259 to 288	256 to 290	331 to 351	220 to 244	263 to 280
k	0,078 to 0,10	0,096 to 0,13	0,15 to 0,18	0,098 to 0,13	0,15 to 0,19
Goodness of Fit					
Degrees of Freedom	46	34,00	70,00	34,00	70,00
R^2	0,94	0,95	0,97	0,97	0,97
Absolute Sum of Squares	26413	15728,00	29632,00	7882,00	20716,00
Sy,x	24	22,00	21,00	15,00	17,00
Constraints					
B	B > 0,0	B > 0,0	B > 0,0	B > 0,0	B > 0,0
k	K > 0,0	K > 0,0	K > 0,0	K > 0,0	K > 0,0
Number of points					
Analyzed	48	36	72	36	72
Outliers (excluded, Q=1,0%)	0	0	0	0	0

Table 12 – Non-linear best-fit analysis of untreated and NiX treated deep litter from suppliers of bull calves

4.3.2 Deep litter from dairy farms

Deep litter from three different dairy farms was analysed for BMP. All deep litters were a mix of at least two different deep litter types.

On a typical dairy farm there are three main categories of deep litter. The first type of litter stems from young calves from the age of 1 – 6 months who live in an enclosure with straw bedding, where they are slowly accustomed to solid food. The litter produced from this type is referred to as deep litter from young calves. This type of litter is not very compressed and consists mostly of straw and smaller amounts of manure. The second type of litter comes from cows that are separated from the rest of the dairy cows when they are dried out. During the drying period they will usually be in a separate enclosure with straw bedding. This litter type is very diverse, and can be compact or loose in structure, and contain varying amounts of manure. These two types of deep litter will be present in more or less all dairy farm productions. The third litter type stems from production units where the calves are heifers are kept from 6 months until about a month before calving. This type is usually very compact and is seemingly similar to the deep litter of bull calves.

The litter obtained from KFC was a mixture of heifers, young calves and dry cows. The litter from Jacobsen and Olesen was a mixture of litter from young calves and dry cows.

	BMP setup	Units	KFC	Jacobsen	Olesen
BMP (untreated)	3	NmL CH_4/g VS	225 (±7) *58 (±3)*	313 (±7) *69 (±6)*	298 (±6) *70 (±5)*
BMP (with NiX treatment)	3	NmL CH_4/g VS	291 (±5)	329 (±4)	340 (±5)

Table 13 – Summary of BMP values obtained from anaerobic digestion of deep litter from dairy farms.

Graphical results from the first BMP setup may be seen in Figure 11 – Figure 13. In all graphs data points represent empirical data, and solid lines represent the best-fit curve.

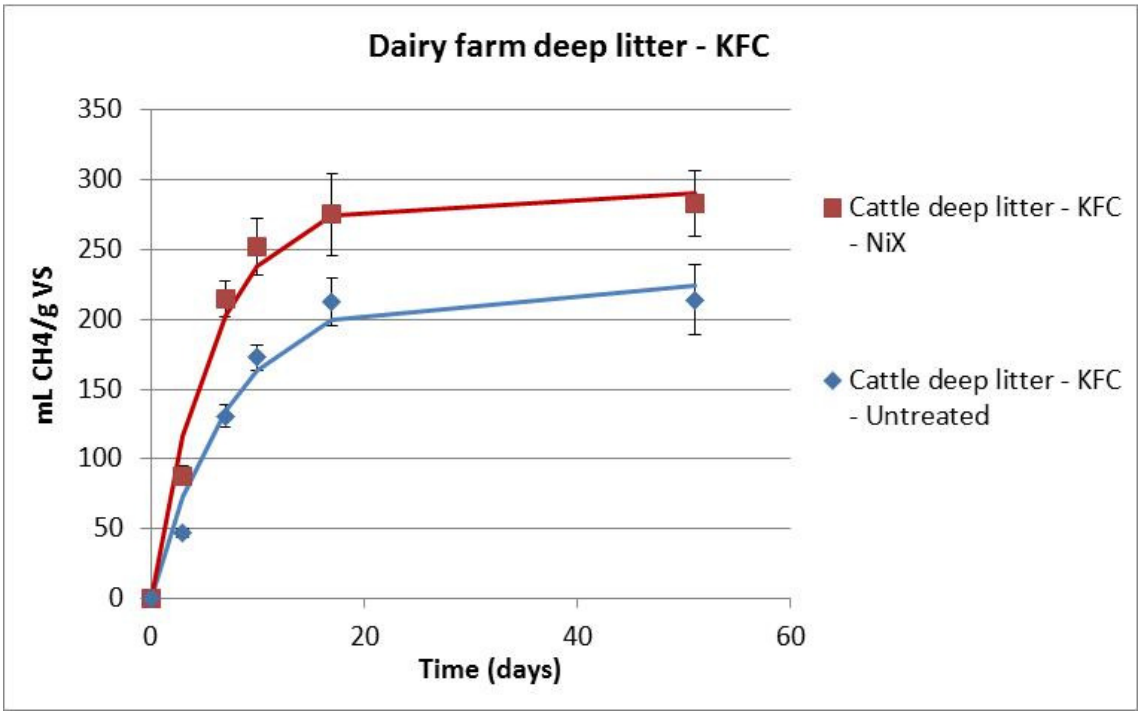

Figure 11 – Accumulated methane yield from BMP batch setup. Untreated and NiX treated deep litter from KFC. Errorbars = +/- 1xSD.

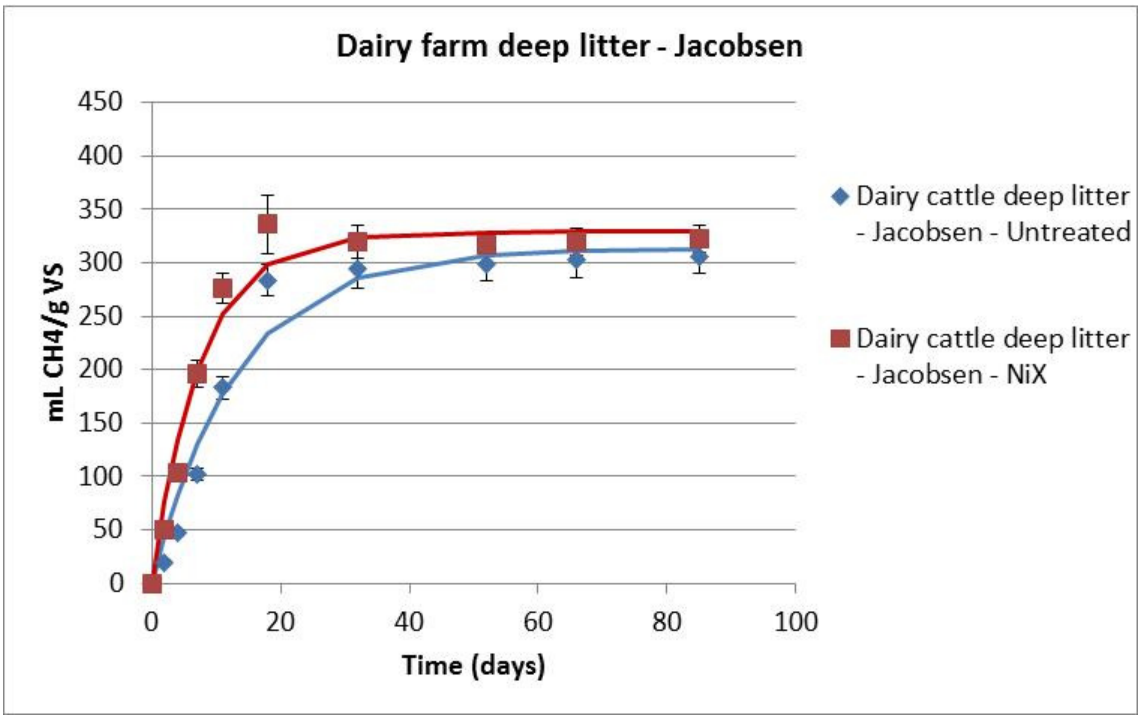

Figure 12 – Accumulated methane yield from BMP batch setup. Untreated deep litter and NiX treated deep litter from Jacobsen. Errorbars = +/- 1xSD.

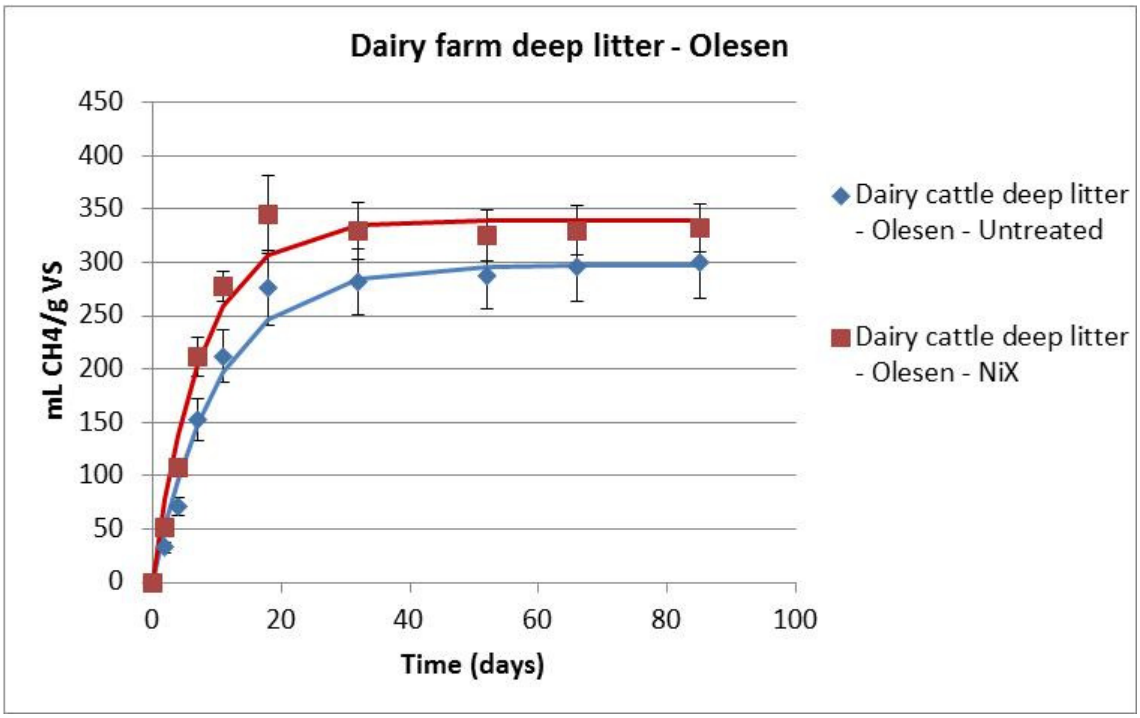

Figure 13 – Accumulated methane yield from BMP batch setup. NiX treated deep litter from and NiX treated deep litter Olesen. Errorbars = +/- 1xSD.

Final BMP values for the two dry cows/young calves litter mixtures from Jacobsen and Olesen have similar BMP values (313 vs. 298), whereas the heifer/dry cows/young calves mixture from KFC has a significantly lower BMP value (225). This could indicate that the addition of heifer deep litter results in a decrease in BMP values. In support of this, the BMP values for bull calves deep litter range from 232-273 NmL CH_4/g VS. However, it should be noted that the KFC BMP value was obtained in the same setup as the low values for bull calve deep litter (see Table 10), meaning that the BMP span between KFC and Jacobsen/Olesen deep litter may be smaller than indicated by Table 13.

The digestion rates of the untreated deep litter samples range from 0.07 to 0.13 (see Table 13). In comparison the digestion rates of the bull calves range from 0.09 to 0.11 (see Table 10). After NiX treatment all deep litter samples digested faster, as seen by *k*-values of 0.13 for Jacobsen/Olesen and 0.17 for KFC.

Best-fit values	Jacobsen - Untreated	Jacobsen - NiX	Olesen - Untreated	Olesen - NiX	KFC - Untreated	KFC - NiX
B	313	329	298	340	225	291
K	0,072	0,13	0,099	0,13	0,13	0,17
Std. Error						
B	6,36	3,47	5,88	4,37	7,00	5,20
K	0,005	0,005	0,007	0,006	0,01	0,01
Lower 95% conf. limit						
B	300	322	286	331	211	281
K	0,07	0,12	0,085	0,12	0,11	0,15
Upper 95% conf. limit						
B	326	336	310	349	239	302
K	0,0867	0,142	0,112	0,143	0,15	0,19
Goodness of Fit						
Degrees of Freedom	58	118	57	106	34,00	70,00
R^2	0,956	0,962	0,947	0,955	0,95	0,96
Absolute Sum of Squares	40468	68506	40647	81360	11550,00	32299,00
Sy.x	26,4	24,1	26,7	27,7	18,00	21,00
Constraints						
B	B > 0.0	B > 0.0	B > 0.0	B > 0.0	B > 0,0	B > 0,0
K	K > 0.0	K > 0.0	K > 0.0	K > 0.0	K > 0,0	K > 0,0
Number of points						
Analysed	60	120	59	108	36	72
Outliers (excluded, Q=1.0%)	0	0	1	0	0	0

Table 14 – Non-linear best-fit analysis of deep litter from dairy farms.

Annex 4 CSTR Experiments on Chicken Litter (WP 7.1+7.3+7.4)

Project No.:	EUDP j.nr. 64011-0335 /Internal project 10842
Made by:	APJE + SBG
Issued:	December 3 2013

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Energy balance? ..... **Fejl! Bogmærke er ikke defineret.**

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1 Abbreviations

AD	-	Anaerobic digestion
HRT	-	Hydraulic retention time
OLR	-	Organic loading rate
OrgN	-	Organic Nitrogen
RSD	-	Relative standard deviation
SD	-	Standard deviation
STP	-	Standard temperature and pressure (273 K and 1 bar)
TAN	-	Total Ammonium Nitrogen
TS	-	Total solids (dry matter)
VFA	-	Volatile fatty acids
w/w%	-	Weight percentage

2 Summary

Using chicken litter as the only substrate input the performance of a 200 L mesophilic single step anaerobic digester was evaluated for more than 18 months of continuous operation. The chicken litter was mixed with recirculated liquid fraction of the digester output and pre-treated by a patented method called NiX, which comprises pressure cooking and lime addition in order to remove the major part of the inhibitory ammonium pool.

The primary performance indicators was in prioritised order methane production, concentration in the digester of free ammonia, concentration of intermediate volatile fatty acids (VFA) and dry matter.

The first test phase resulted in levels of free ammonia above critical inhibitory levels reported in the scientific literature. This led to strongly fluctuating specific methane yields from less than half to more than twice of expected yield. Parallel to this the VFA pool grew to levels not normally seen in anaerobic digester unless process breakdown is imminent.

The results of the first test phase led to an optimisation of the method for NiX-treatment with respect to efficiency of the ammonium stripping. This was achieved by introducing a pre-incubation before NIiX-treatment thereby converting the major part of the organic nitrogen to ammonium. This allows for stripping of a much bigger part of the total nitrogen pool before anaerobic digestion.

The second test phase comprised this improved nitrogen conversion step, but process stability was not achieved as pH in the digester increased rapidly and soon caused the pH dependent free ammonia to cross the threshold level for potential inhibition. It was therefore decided to abort the test and look for ways of controlling pH in the digester.

A solution to this problem was found by an innovative application of CO2 in the produced biogas in a step between NiX-treatment and anaerobic digestion.

The third and last test phase demonstrated stable process conditions with free ammonia concentrations below the inhibitory level, low VFA's and a high specific methane yield of more than 250 L CH4 per kg VS added.

For both the abovementioned improvements to the NiX method patent applications have been filed.

3 Introduction

In the reported investigation the performance of a continuously operated CSTR using only chicken litter as substrate was evaluated. The process was based on recirculation of separated digester liquid and application of the NiX-method on both this liquid and the chicken litter. The purpose of the investigation was to demonstrate under which conditions stable plant operation with uninhibited gas yields could be achieved.

Using chicken litter for anaerobic digestion (AD) is attractive due to the high energy density of the material. Organic dry matter levels (VS) are typically in the range from 35 to 55 % of total mass and the obtainable methane yield is relatively high for animal manure with reported yields around 300 Nm3 CH4 per ton VS. Realistic yields per ton of wet weight is therefore at the same level or higher for chicken litter (100-150 Nm3 CH4) than for most energy crops (often less than 100 Nm3 CH4).

However, chicken litter also has a high nitrogen (N) content, which can be inhibitory to the AD process, by release in the form of ammonium-N (TAN). TAN is the sum of ammonium (NH_4^+) and free ammonia (NH_3), the latter of which has been identified as the inhibiting agent. The NH_3-fraction of TAN is positively correlated with process temperature and pH. Thus, at a certain TAN-level the potential inhibitory effect increases with increasing temperature and (particularly) pH. The actual threshold value for ammonia inhibition cannot be universally defined as microbial adaptation and potential neutralizing effect of other ions can be in play. Most studies indicate however, that the concentration of free ammonia should be kept below 1,000 mg/L in the digester. Some studies even suggest that the level for significant inhibition is to be found as low as 600 mg/L. In this investigation an inhibitory limit for free ammonia (NH_3) at 700 mg/L was assumed.

Traditionally chicken litter is mixed with other biomasses low in N to a manageable average N-level. This limits the use of chicken litter to AD-plants with access to sufficient N-poor biomasses and to using typically less than 10 % chicken litter in the biomass input. Alternatively, chicken litter can be diluted with water to reduce both dry matter and N to acceptable levels for the AD process. This approach results in extra costs for water consumption and process heat and more importantly in excess production of effluent, which can be fatal to the economic feasibility of a project.

Recirculation of digester effluent possibly after removal of suspended solids can potentially be a solution to reducing water and heat consumption as well as effluent production, but nitrogen content in the effluent stream will be at the same level or higher than in the digester. Controlling the N-balance in the digester is thus the key to a stable AD process based on chicken litter as mono-substrate with recirculation of separated digester liquid. Water addition can be necessary for maintaining the water balance.

4 Procedure

The investigation was carried out in a pilot scale plant during the period from November 2011 to July 2013. Before the start of the investigation, the plant had already been running on poultry litter for several months in connection to previous investigations (EUDP 64010-0083).

The test was based on a single-step digestion at 37 °C with hydraulic retention times and organic loading rates as specified below for each of the phases. Average dry matter content in the applied chicken litter was 62 % TS and 52 % VS.

Theoretical calculation before and during the experimental plant operation had established that the process could be maintained below the assumed inhibitory limit for free ammonia (NH3) at 700 mg/L when the following parameter values were applied:

1. 65 % removal of ammonium-N (TAN) during NiX treatment
2. pH in digester ≤ 8.1
3. 70 % conversion of organic N to TAN in digester
4. Water to chicken litter ratio 0.33 (weight basis)
5. Recycled liquid to chicken litter ratio 3.1 (weight basis)
6. Lime (CaO) to chicken litter ratio 0.07 (weight basis)
7. TS 4 % in liquid fraction after separation of effluent for recirculation.

The feasibility of the required TAN removal was demonstrated during previous experiments, but the use of lime to increase pH during NiX-treatment can potentially disturb the pH-level in the digester above the maximum acceptable limit. As the concentration of free ammonia is highly pH-dependent, the achieved effect of TAN-removal can be partially or completed counteracted by increases in pH in the digester.

Another critical assumption to be verified by the experiment was the ability to maintain the water balance of the system with the determined water addition. This basically would depend on the efficiency of separation of the effluent stream.

During the test period the obtained results gave rise to the addition of two novel features of the NiX-concept. The test period is therefore divided into three phases as follows:

Phase 1	NiX-treatment of chicken litter and recycled separated effluent liquid
Phase 2	Pre-incubation of chicken litter and recycled separated effluent liquid for increase of ammonium pool before NiX treatment
Phase 3	As phase 2 but with addition of post-treatment of NiX treated mixture in order to control pH

The key performance indicators (KPI) of the test run were the following:

1) Methane yield
2) NH3 concentration in digester (TAN/pH)
3) TS in digester and recycled liquid / water balance

KPI 1) and 2) were primary as the NH3 concentration is expected to be the main potential inhibitor of the methane yield. When stable NH3 concentration and a stable and high methane yield is obtained the focus changes to also include the remaining KPI's.

In addition to the KPI's the Volatile Fatty Acids (VFA) in the digester were followed as a generally accepted indicator of process stability.

The specific methane production is calculated as the average methane production in the last 7 days relative to the average amount of VS added over the same period. All volumes are reported at STP conditions. Expected methane production is calculated from a batch BMP test taking into account the retention time in the digester.

5 Results

5.1 Phase 1. Nov 2011 – Jun 2012

Before initiation of phase 1 on the 1st of January 2012 the plant had been operated for more than 3 months with gradual increases in the organic loading (OLR). The increase was obtained by reducing the HRT in two steps from 85 days to initially 34 days and later 29 days. This final HRT corresponded to an OLR of 3.49 g VS/(L digester x 24h), which can be considered acceptable for testing the plant performance under realistic loading conditions. At the onset of Phase 1 the plant had been in stable operation at this OLR for more than 6 weeks (since 15th of Nov 2011).

During Phase 1 the performance of the digester was highly dependent on the organic loading rate, which led to a number of changes in the loading in attempts to reach stable and high yields. Phase 1 has therefore been divided into four periods the results of which will be presented individually.

5.1.1 Period 1A. Nov 15 2011 – Feb 9 2012. High OLR (3.49)

Methane yield (Figure 1) was stable around 300 L CH4/kg VS for more than the first 4 weeks of the period but a decrease began in mid-December and continued until early January where a new stable yield level slightly above 200 L CH4/kg VS was reached. This was followed by unstable peaks of higher yields leading to a sudden drop to less than 200 L CH4/kg VS at the end of the period.

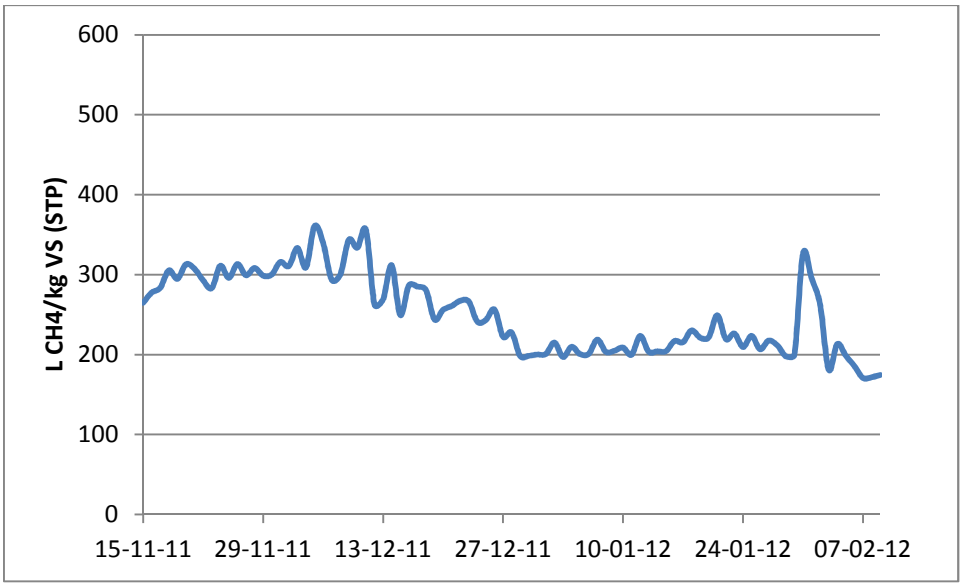

Figure 1 Specific methane yield per kg chicken litter VS added (5 days rolling average).

The concentration of TAN (Figure 2) remained quite constant during the first 4 weeks at 6,100-6,200 mg/L followed by an increase within two weeks around mid-December to a new plateau around 6,700 mg/L with a peak value of nearly 7,000 mg/L on the last day of measurement.

NH3 showed little variation around a mean value of 900 mg/L during the first 4 weeks but peaked at 1,100 mg/L during the following TAN increase which coincided with a pH increase from 8.1 to 8.2 (Figure 3). NH3 dropped consecutively to a level of 700-800 mg/L due to a pH decrease from 8.2 to 8.0. This was followed by an increase in pH from around 8.0 to more than 8.1 causing the NH3 to return to the previous peak value at nearly 1,100 mg/L.

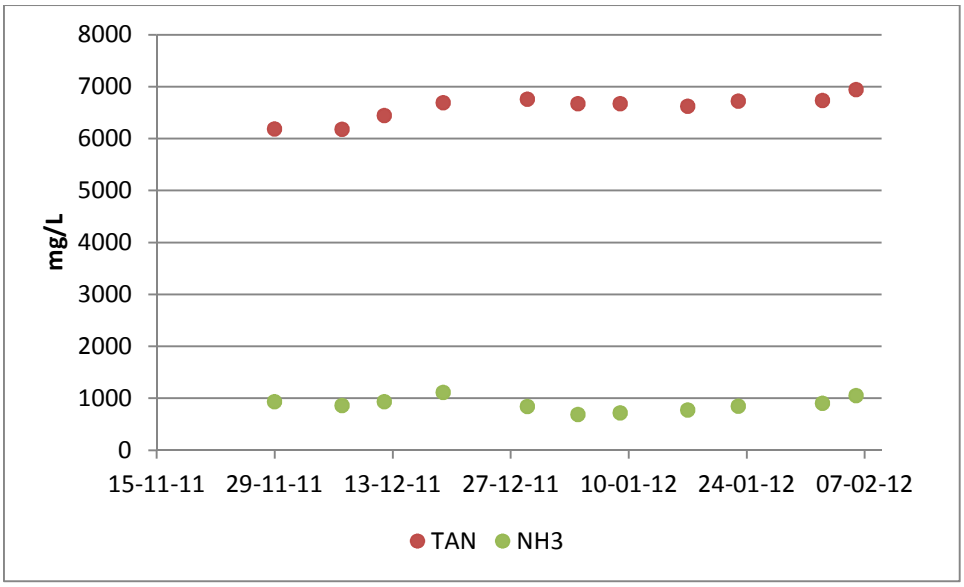

Figure 2 TAN and NH3 concentrations in the digester.

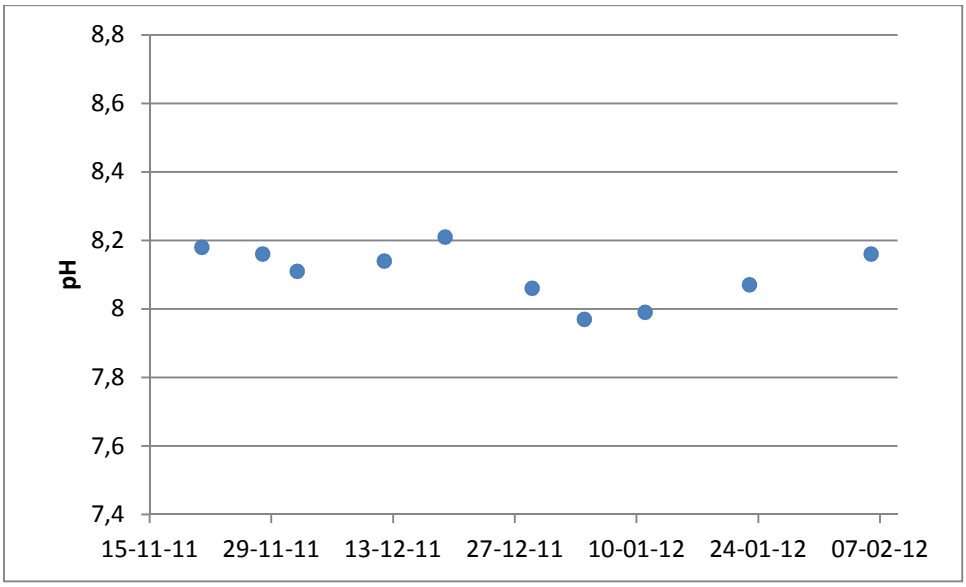

Figure 3 pH in the digester.

VFA's were not measured during the first 4 weeks of the period. The first analysis in the second half of December showed a total level of 2,300 mg/L and a healthy profile with acetic acid as the dominating species and with propionic acid as the only other VFA present at a significant concentration (Figure 4 and Figure 5). The following 3 measurements showed drastic increases in acetic as well as propionic acid to levels of 12,000 and 6,000 mg/L, respectively. The longer-chained VFA also increased from levels close to zero to several hundreds of mg/L.

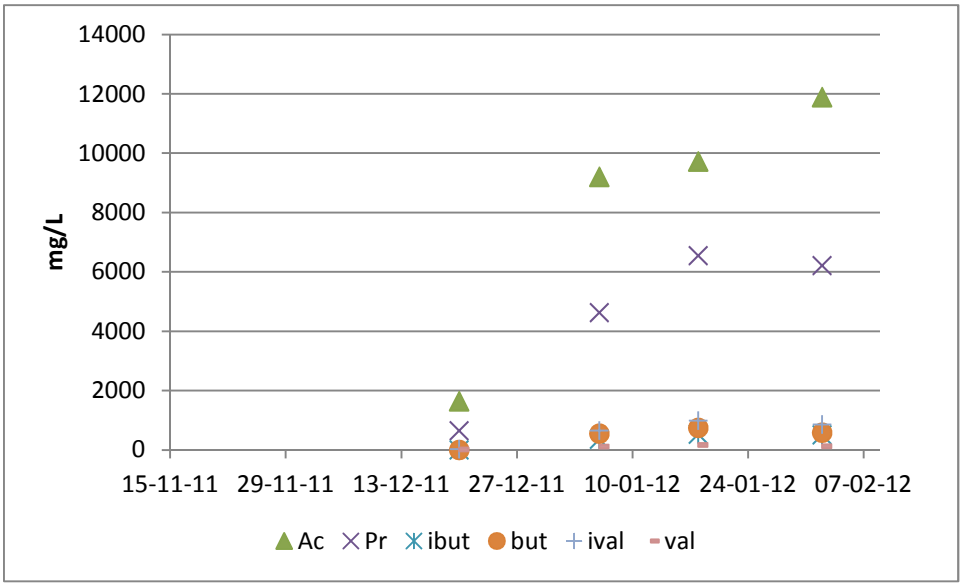

Figure 4 VFA in digester.

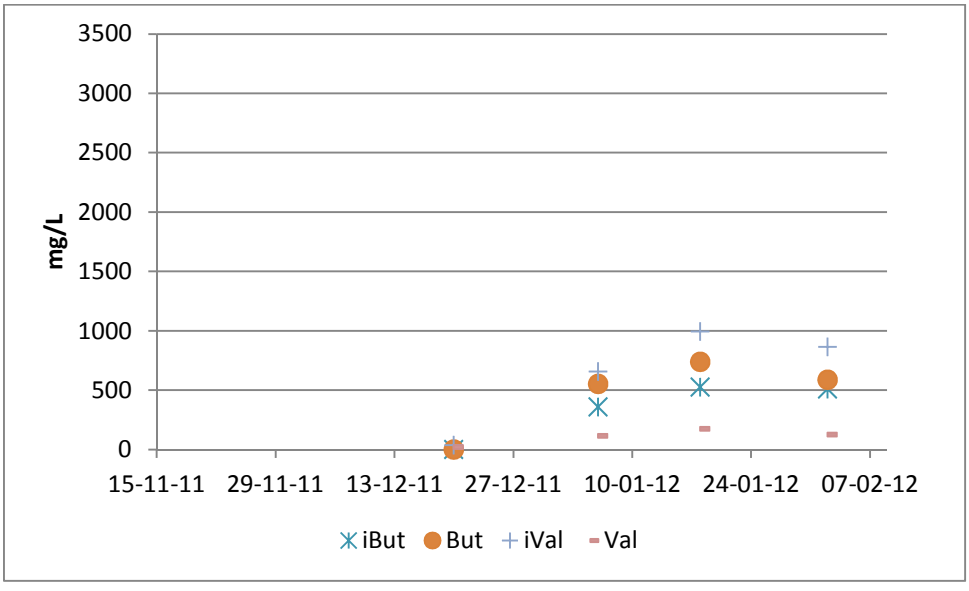

Figure 5 Blow-up of C4-C5 VFA in digester.

The TS content in the digester (Figure 6) increased gradually from 10 % during 8 weeks to a plateau around 12.5 % in the first half of January and stayed at this level during the rest the period.

The TS content in the recycled liquid showed the same development from an initial level at 5 % to more than 8 %.

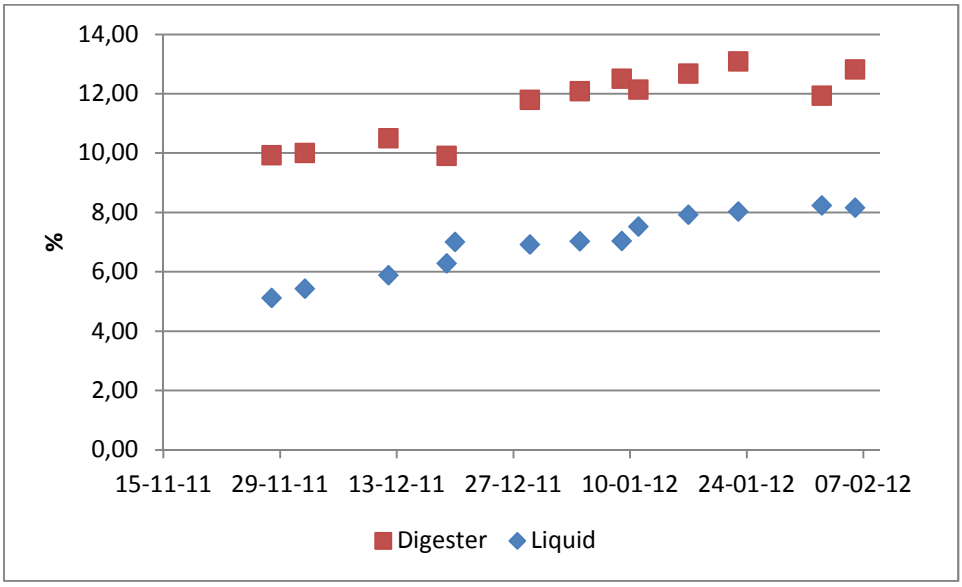

Figure 6 TS in digester and recycled liquid.

5.1.2 Period 1B. Feb 10 2012 – Apr 1 2012. Low OLR (1.26)

As a consequence of the falling gas yield and the increasing VFA levels during Period 1A the loading was reduced by 64 % by a reduction of the daily input without any changes in the composition of the input. In addition to this, 25 % of the digester content was replaced by water on February 27 in an attempt to save the process.

Methane yield (Figure 7) immediately started to go up from less than 200 L CH4/kg VS and peaked within a few days at +260 L CH4/kg VS followed by a very rapid drop during a week to less than 100 L CH4/kg VS. This level continued for three weeks after which the gas yield increased dramatically to +560 L CH4/kg VS during the last 3 weeks of the period.

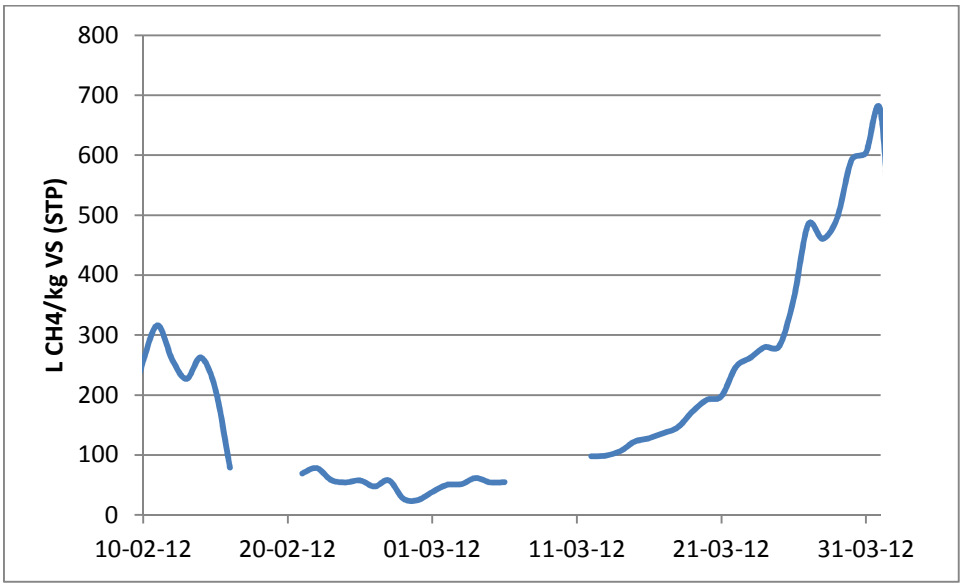

Figure 7 Specific methane yield per kg chicken litter VS added (5 days rolling average).

The concentration of TAN (Figure 8) increased gradually from just below 6,900 mg/L to 7,300 mg/L within the first 3 weeks followed by a drop to 5,600 mg/L at the end of February due to dilution of the digester content with water. This level was maintained for another 2 weeks followed by a slight increase to approx. 5,800 mg/L for the rest of the period.

NH3 decreased from 900 to 800 mg/L during the initial increase in TAN due to falling pH from 8.1 to an estimated 8.0 (Figure 9). Water dilution reduced NH3 to 600 mg/L followed by a further drop to 400 mg/L caused by a continued drop in pH to less than 7.8. NH3 then rose gradually to 700 mg/L during the rest of the period caused by the pH returning to a level above 8.0.

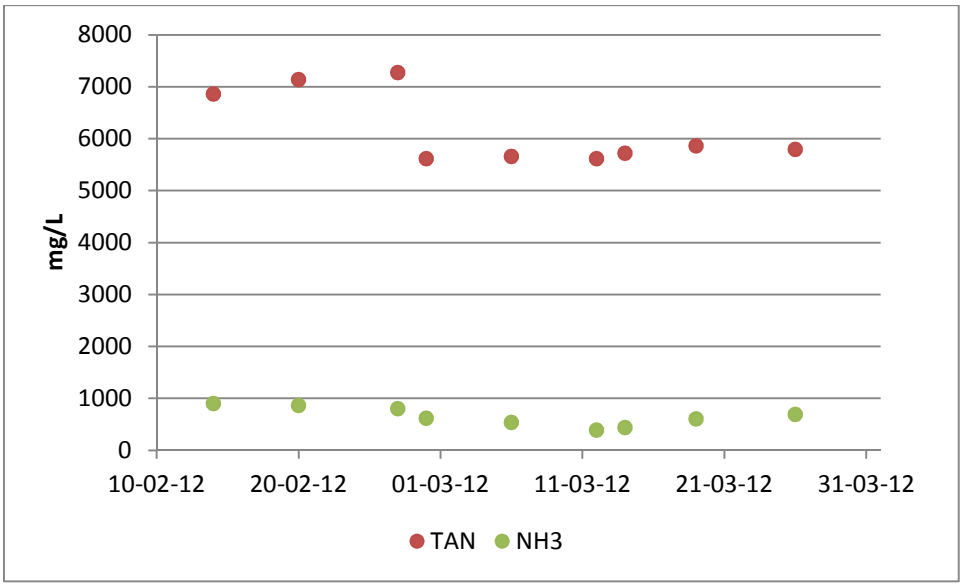

Figure 8 TAN and NH3 concentrations in the digester.

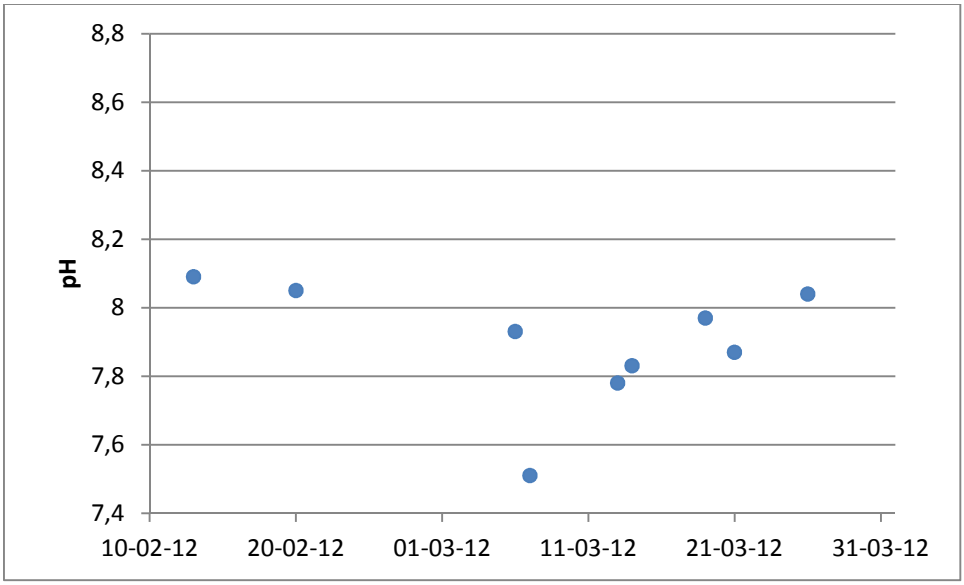

Figure 9 pH in the digester.

The VFA levels (Figure 10) were quite constant during the first 2 weeks of the period, but for propionic acid at a much higher level than at the end of the previous period (10,000 vs. 6,000 mg/L).

Propionic acid stayed at a level around 10,000 mg/L during the entire period with fluctuations (+/- 2,000 mg/L) but no clear tendency. Acetic acid on the other hand started increasing at the end of February and peaked at a level of more than 25,000 mg/L in mid-March followed by a rapid decrease during the next 2 weeks to 10,000 mg/L.

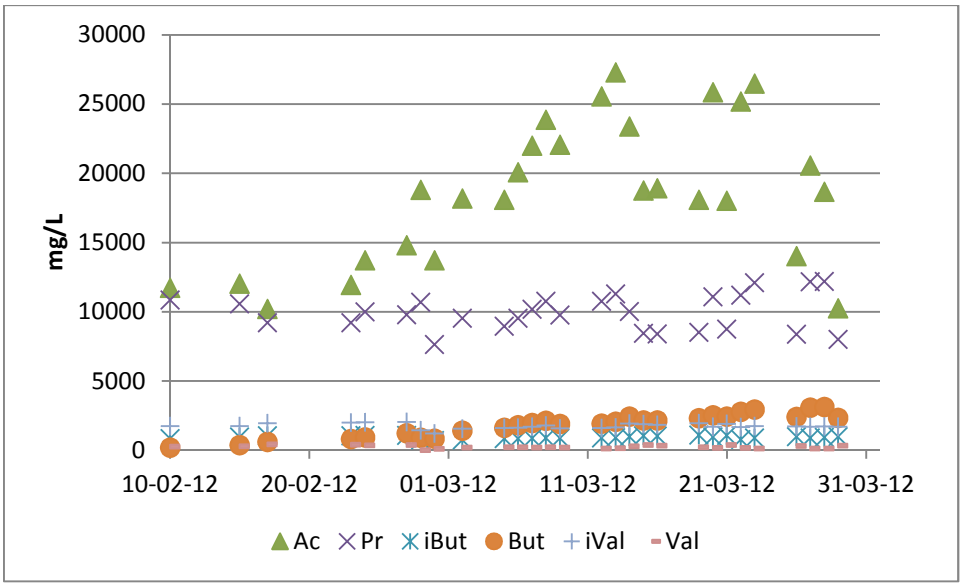

Figure 10 VFA in the digester.

The longer-chained VFA's showed different trends during the period (Figure 11).

Butyric acid increased constantly during the entire period from less than 200 mg/L reaching a final level of more than 3,000 mg/L (although with fluctuating values from 2,300 to 3,100 mg/L during the last week).

Iso-butyric acid remained relatively constant around 900 +/- 200 mg/L.

Valeric acid dropped after water dilution from an initial level around 350 mg/L to below the detection limit and then stabilized at a level around 200 mg/L.

Iso-valeric acid gradually increased from 1,700 to 2,000 mg/L during the first week and stayed there until the water dilution 10 days later. The concentration then dropped first to a level corresponding to the degree of dilution (1:3) and then further to 1,200 mg/L before increasing gradually to 1,900 mg/L followed by a drop to a final level of 1,700 mg/L.

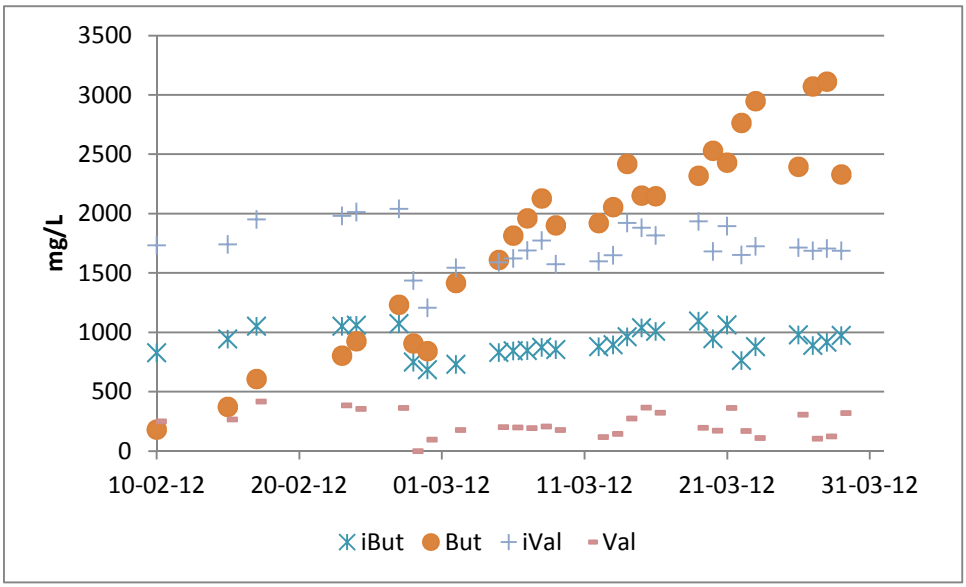

Figure 11 Blow-up of C4-C5 VFA in digester.

The TS content in the digester (Figure 12) had increased since the end of Period 1a to a level of more than 13 %. Water dilution reduced this level 10.7 % but TS continued to increase slowly during the rest of the period ultimately reaching 11.4 %.

The water addition had no immediate effect on TS in the recycled liquid, which remained at around 8.5 % but consecutively decreased and reached 6.9 % before the end of the period.

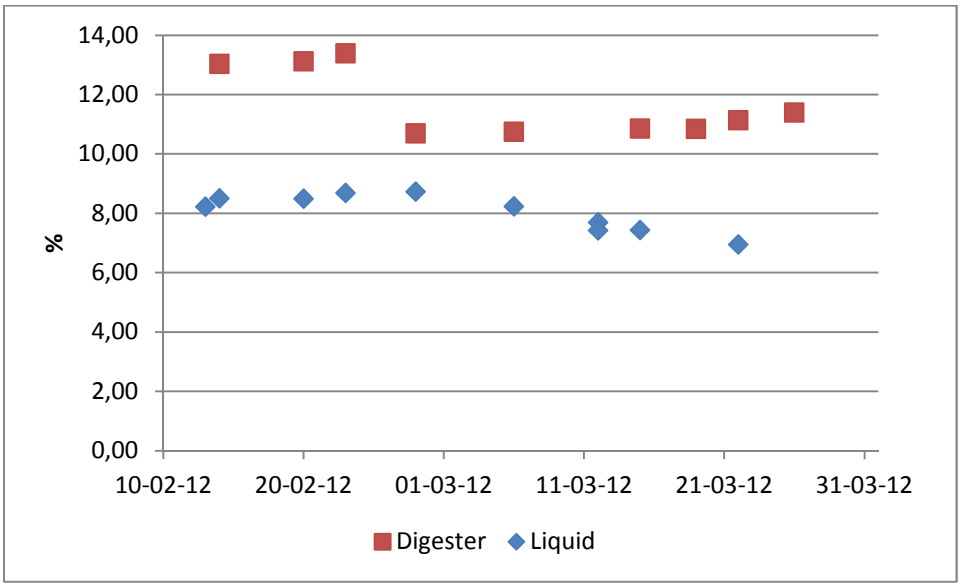

Figure 12 TS in digester and recycled liquid.

5.1.3 Period 1C. Apr 2 2012 – May 21 2012. Medium (2.26) → high OLR (3.24)

The very high specific yields during the second half of Period 1C led to the decision to start increasing the loading of the digester. This was done in two steps from 1.26 to 2.26 g VS/(L digester x 24h) at the start of the period and from 2.26 to 3.24 g VS/(L digester x 24h) after two weeks. The second increase in loading meant a return to a loading level similar to Period 1a (3.49 g VS/(L digester x 24h)).

Methane yield (Figure 13) immediately began to drop from the peak level of more than 560 L CH4/kg VS at the end of Period 1B to around 300 L CH4/kg VS three weeks later and finally stabilizing at 250 L CH4/kg VS after 6 weeks.

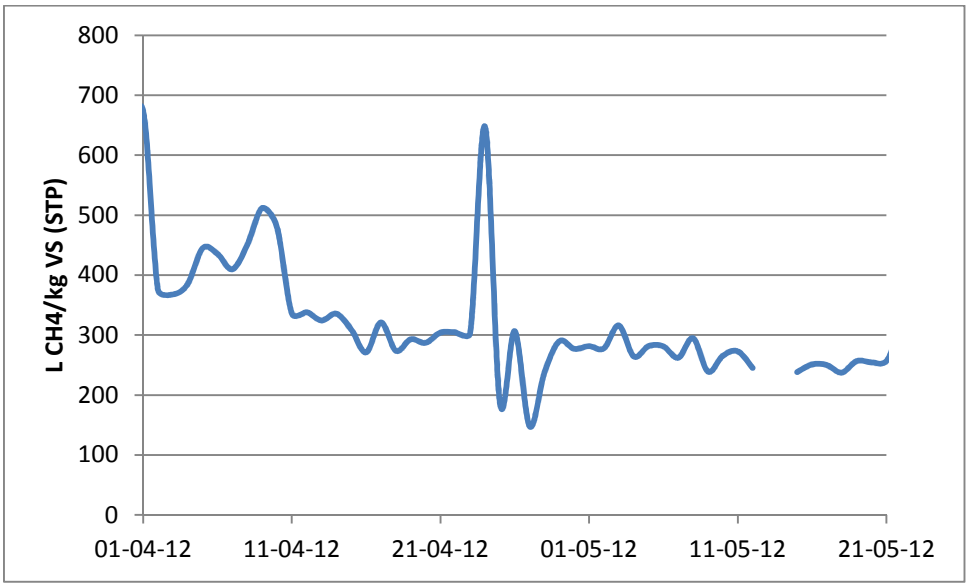

Figure 13 Specific methane yield per kg chicken litter VS added (5 days rolling average).

The concentration of TAN (Figure 14) was stable at 5,500 mg/L during the first 3 weeks followed by a drop to 5,000 mg/L, which was maintained during the rest of the period. NH3 increased from 900 to 2,100 mg/L during the first 3 weeks due to increasing pH from an estimated 8.2 to an 8.7 (Figure 15). Consecutively, NH3 dropped to 1,000 mg/L during the second part of the period as a result of pH dropping to 8.3 while also TAN decreased.

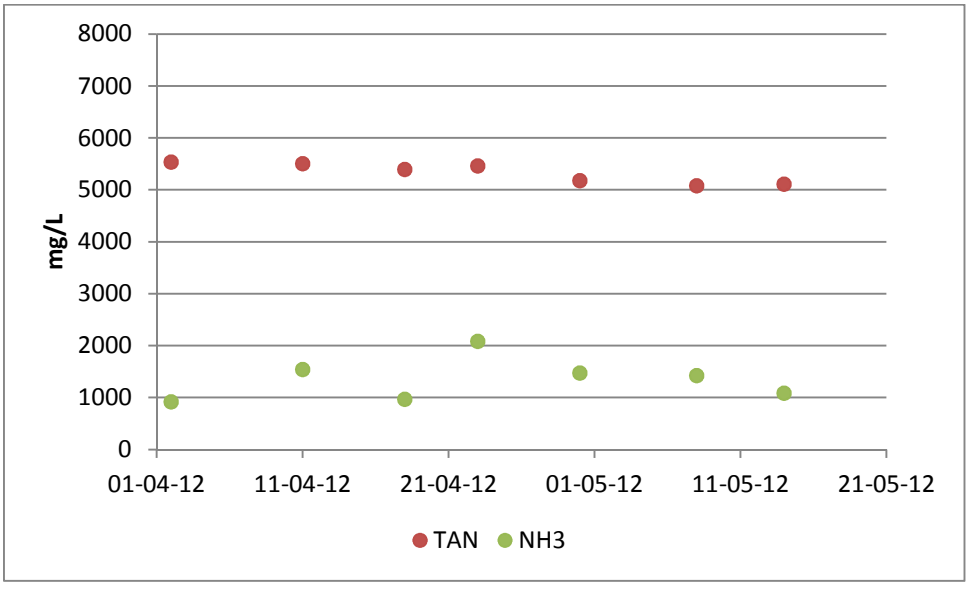

Figure 14 TAN and NH3 concentrations in the digester.

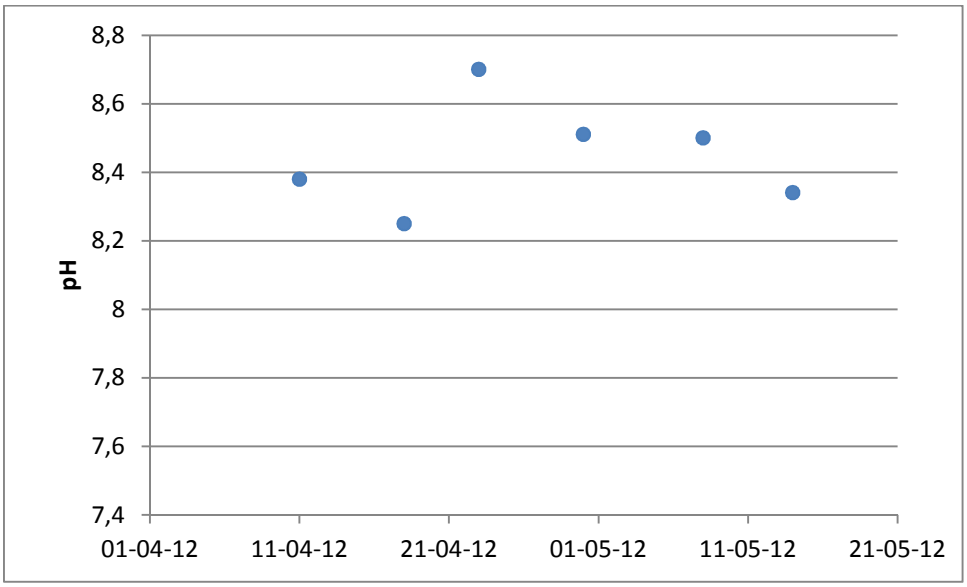

Figure 15 pH in the digester.

The composition of the VFA pool (Figure 16) changed markedly during the period with acetic acid continuing the drop that had started in the last part of Period 1B.

Acetic acid levels had thus dropped to 4,000 mg/L at the start of the period and continued to drop to a final 1,500 mg/L.

Propionic acid, on the other hand, increased in concentration from 9,000 mg/L to peak values above 20,000 mg/L before dropping again to 11,000 mg/L at the end of the period.

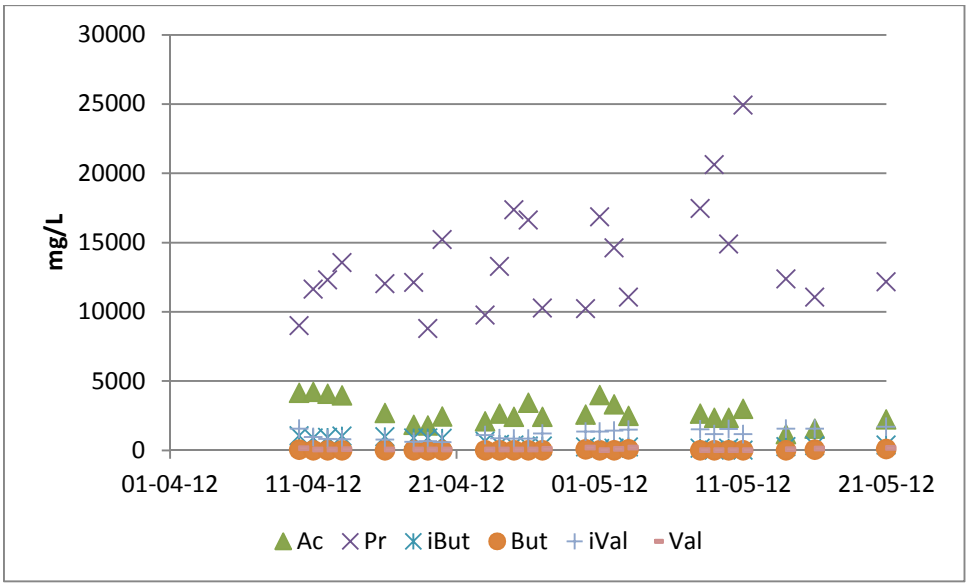

Figure 16 VFA in digester.

The longer-chained VFA's showed different developments during the period.

Butyric acid had dropped from a level of more than 3,000 mg/L at the end of the previous to less than 100 mg/L at the first analysis after little more than a week and stayed at this low level during the entire period.

Iso-butyric acid maintained the level from the previous period around 900 - 1,000 mg/L for the first 3 weeks after which the level dropped to less than 300 mg/L within a week. After a further drop to around 150 mg/L the level increased to around 350 mg/L at the end of the period.

Valeric acid fluctuated between 0 and 200 mg/L during the entire period with a tendency towards values in the high end during the last week of the period.

Iso-valeric acid dropped from an initial 1,600 mg/L to around 600 mg/L during the first three weeks followed by a gradual increase to 1,700 mg/L at the end of the period.

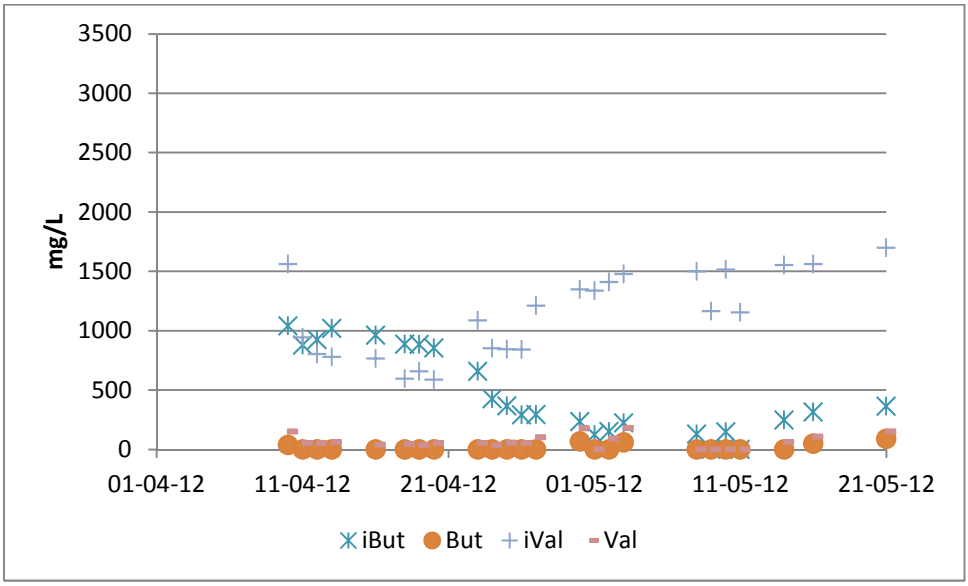

Figure 17 Blow-up of C4-C5 VFA in digester.

The TS content in the digester (Figure 18) had decreased since the end of Period 1b to 10.3 %. TS increased gradually during the entire period reaching a final level of 11.7 %.

TS in the recycled liquid remained at around 7.0 % during the first 4 weeks where after a gradual increase to 7.5 % within 2 weeks was observed.

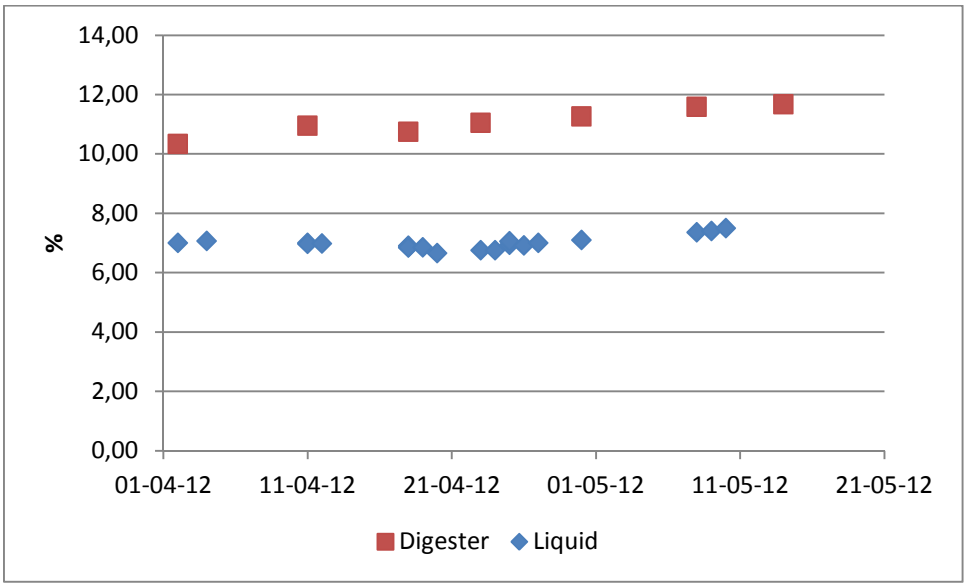

Figure 18 TS in digester and recycled liquid.

5.1.4 Period 1D. May 22 2012 – June 24 2012. Medium OLR (2.03)

The relatively low gas yield at the end of Period 1C with a preceding long period of declining yield in combination with a persistently very high concentration of propionic and iso-valeric acid led to the decision to decrease the digester loading by 37 % in order to avoid the risk of process break-down.

Methane yield (Figure 19) was quite constant during the entire period around a mean of 270 L CH4/kg VS but with drops to 230-240 L CH4/kg VS several times. However, at the end of the period the yield showed an increasing tendency towards a yield level around 300 L CH4/kg VS.

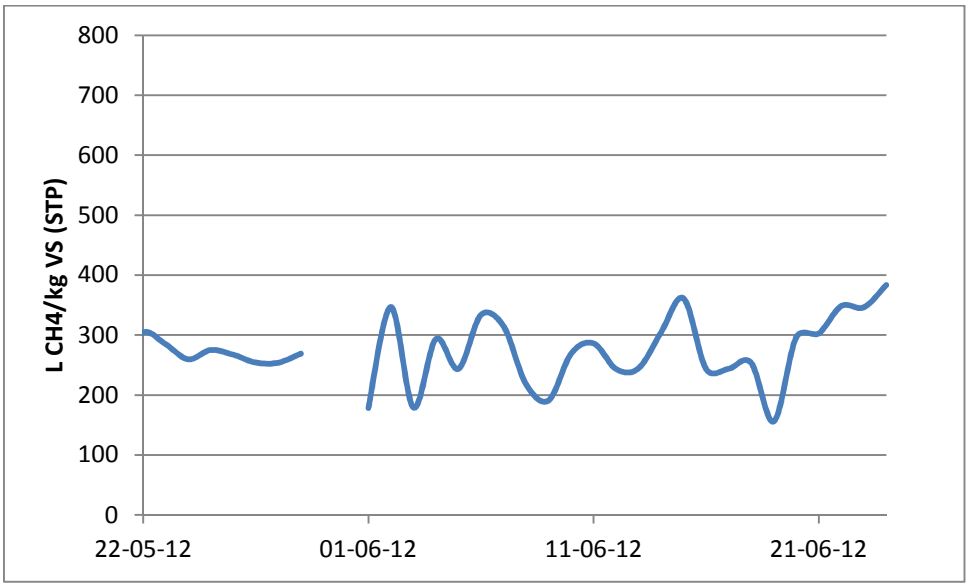

Figure 19 Specific methane yield per kg chicken litter VS added (5 days rolling average).

The initial concentration of TAN (Figure 20) at 5,200 mg/L increased during the first 2 weeks to a final level of 5,600 mg/L.

NH3 increased from 700 to 1,200 mg/L during the first week due to increasing pH from 8.1 to 8.3 (Figure 21). A week later a stable NH3-level at 1,100 mg/L had been reached while the pH had stabilized at around 8.2.

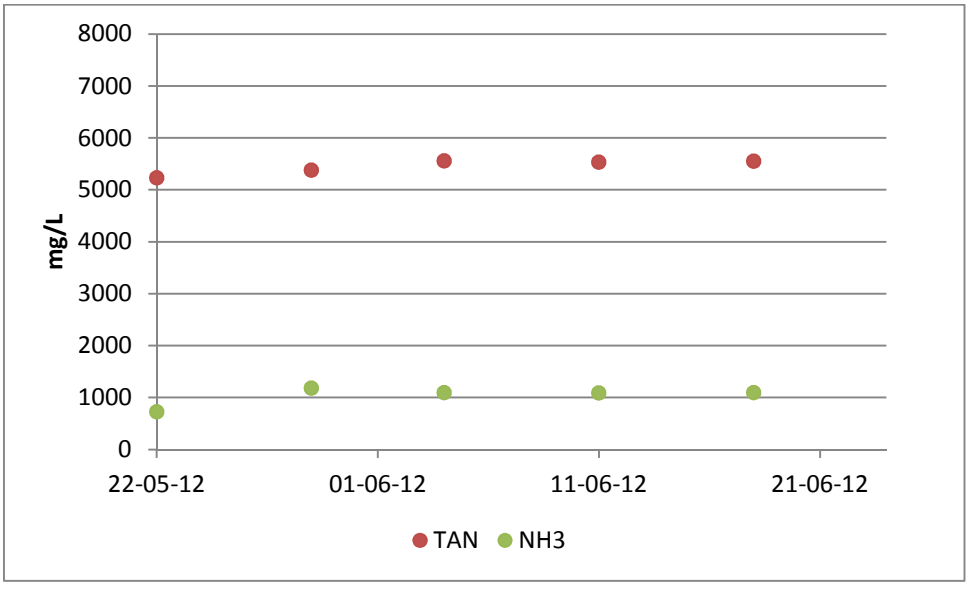

Figure 20 TAN and NH3 concentrations in the digester.

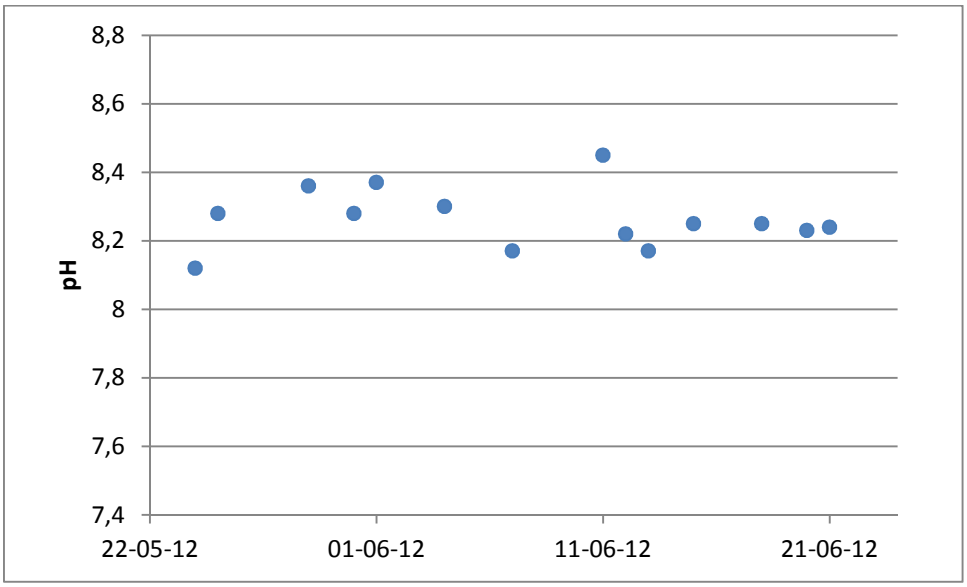

Figure 21 pH in the digester.

The VFA pool (Figure 22) stabilised during the period with acetic acid levels around 1,500 +/- 200 mg/L.

Propionic acid varied between 11,000 and 19,000 mg/L with no clear trend and with most values at 15,000 +/- 2,500 mg/L.

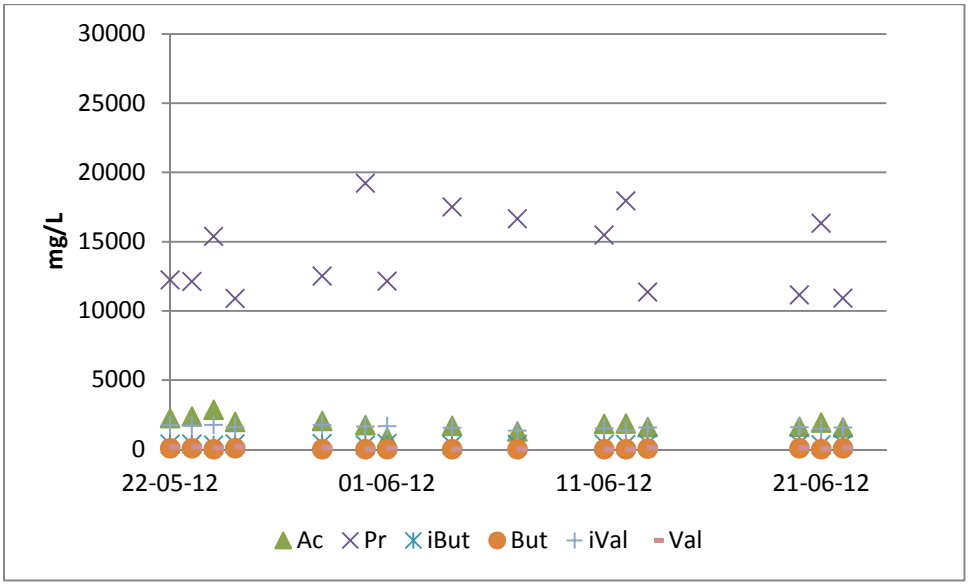

Figure 22 VFA in digester.

Butyric and valeric acid levels were constant at ≤ 100 and ≤ 200 mg/L, respectively, while iso-butyric and iso-valeric acid were constant at 300-400 mg/L and 1,300-1,800 mg/L, respectively.

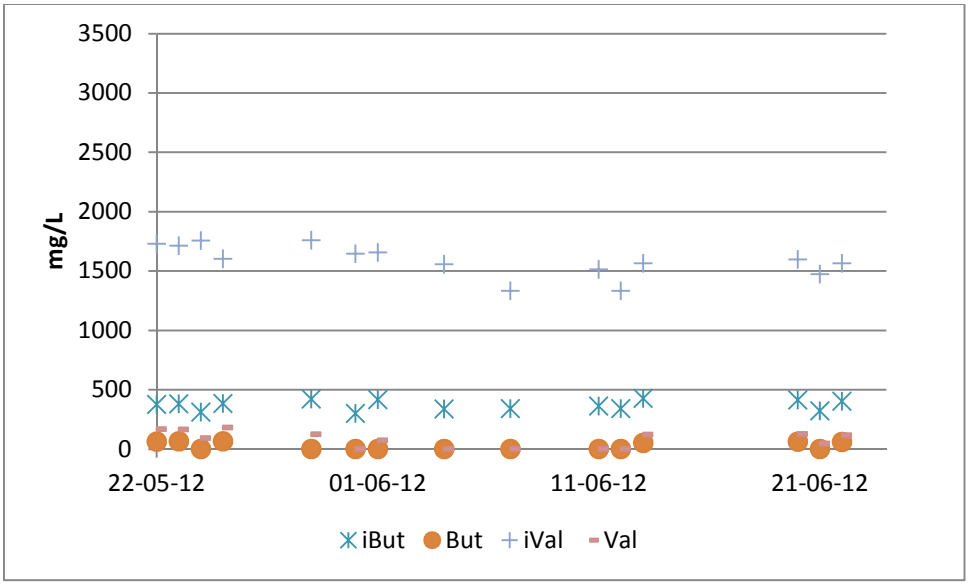

Figure 23 Blow-up of C4-C5 VFA in digester.

The TS content in the digester and the recycled liquid (Figure 24) had increased slightly since the end of Period 1c from 11.7 to 12.0 % and 7.5 to 8.0 %, respectively.

TS in the digester remained in a range from 12.0 to 12.4 % during the entire period. TS in the recycled liquid remained in a range from 7.7 to 8.0 % during the entire period.

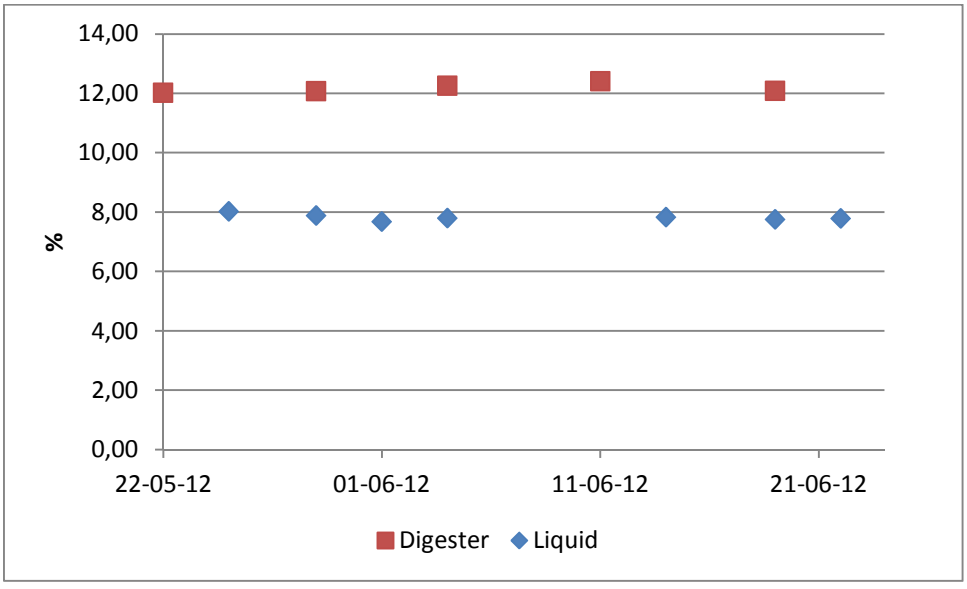

Figure 24 TS in digester and recycled liquid.

5.2 Phase 2. November 2012 – January 2013. High OLR (5.0)

At the end of Phase 1, it was clear that with TAN levels at 5-6,000 mg/L and pH levels at 8.1-8.2 for extensive periods, it would be difficult to maintain stable anaerobic digestion at non-inhibitory ammonia levels (below 700 mg/L).

Since ammonia is a product of TAN and pH, it is possible to decrease the ammonia levels by decreasing either TAN or pH levels. In chicken litter the TAN pool constitutes ~25% of the total nitrogen pool. The rest is organically bound as uric acid and proteins and therefore not available for stripping during the NiX treatment. The the majority of the organic nitrogen is mineralised during digestion and released as TAN in the digester. To lower the amount of TAN in the digester a novel concept was thus developed to degrade the organic nitrogen to inorganic nitrogen (TAN) prior to the digester, thus allowing for it to be removed in the NiX treatment.

The nitrogen mineralisation (or simply mineralisation) method developed made it possible to increase the strippable nitrogen fraction more than 5 fold by mineralisation of a large part of the organic nitrogen into TAN. Details regarding the method may be seen in section 7 or in Annex 2 to the main report. In summary, nitrogen mineralisation comprises incubation of the chicken litter at 36 ℃ with an appropriate mixture of liquid from separated digestate and a microbially active seeding material at 36 ℃. The seeding material stems from a previous mineralisation, and contains a viable and active microbial culture, which facilitates conversion of up to 75 % of the nitrogen contained in organic compounds into TAN within 24 hours.

Nitrogen mineralisation allows for a more efficient stripping process since the amount of nitrogen available per unit chicken litter is much higher. However, the increased TAN removal potential also necessitates addition of more lime during NiX treatment, which may potentially counteract the lowered TAN levels by increasing digester pH and hence the free ammonia. The assumption in the following phase was that the buffer capacity of the digester is strong enough to maintain pH levels at 8.1-8.2.

The development of TAN, NH3 and pH during Phase 2 may be seen in Figure 25 and Figure 26.

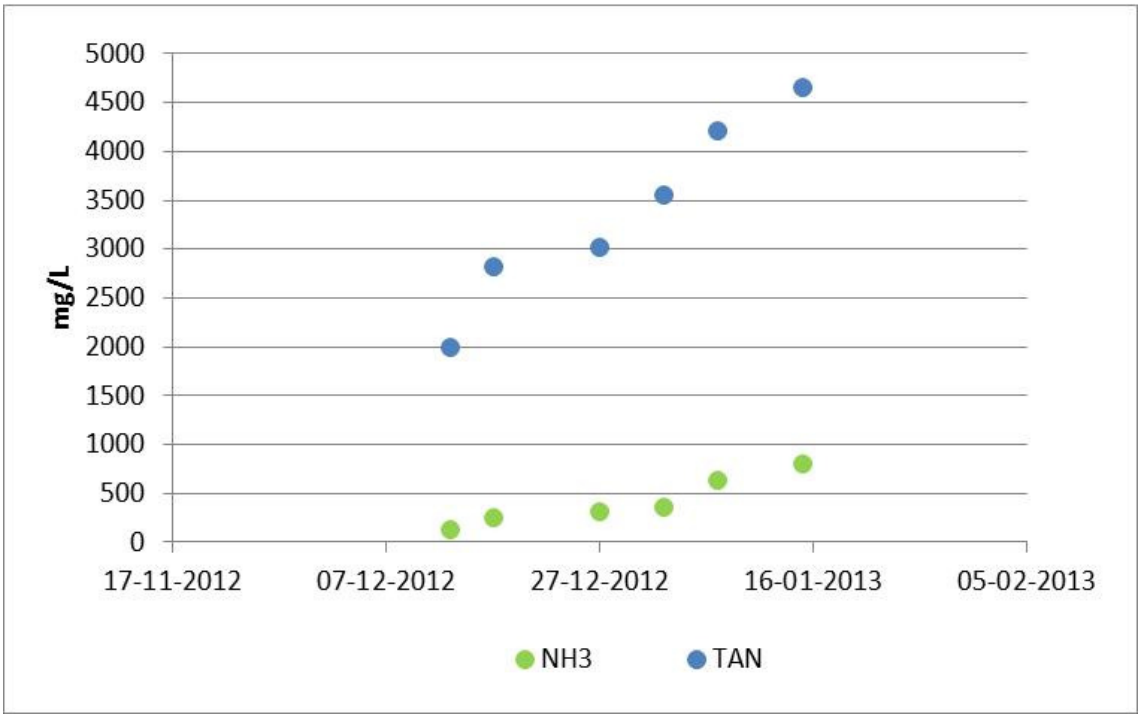

Figure 25 TAN and NH3 concentrations in the digester

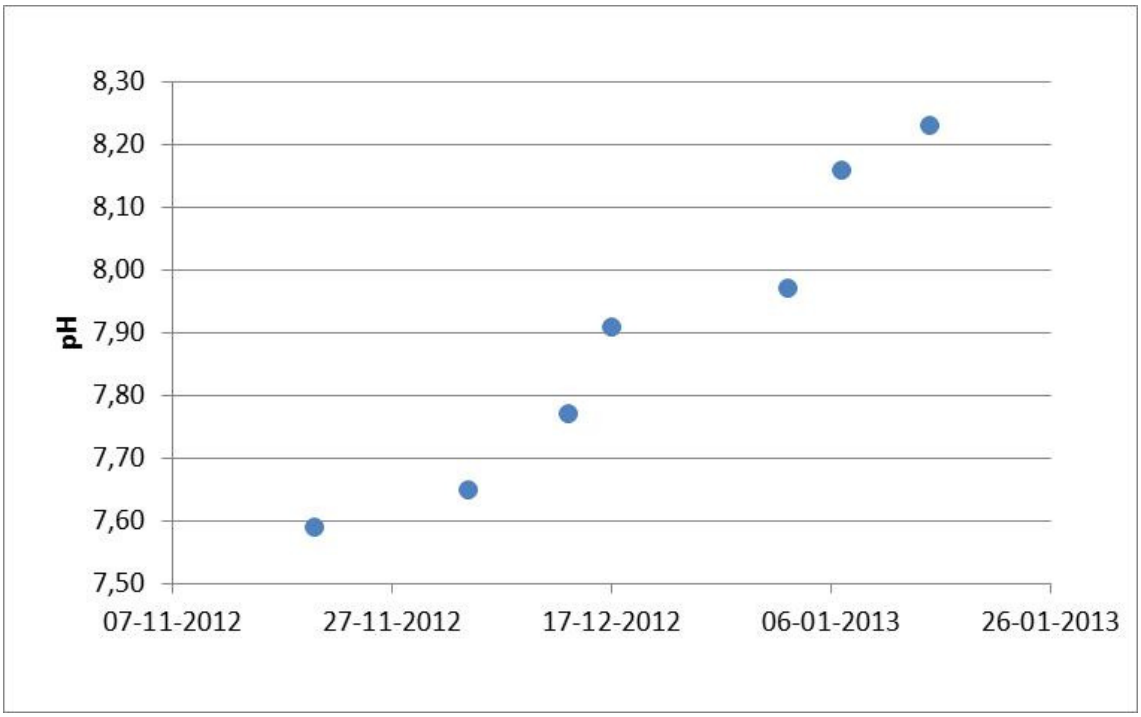

Figure 26 pH in the digester

As may be seen from Figure 26 and Figure 25, pH and TAN levels were steadily increasing all throughout the experiment. Although mineralisation allowed stripping of significantly higher amounts of nitrogen (data not shown), the buffer capacity of the digester was not strong enough to maintain pH at the desired level. At the end of Phase 2, pH had reached 8.3 and TAN concentration was above 4500 mg/L. At this point the reactor contained 800 mg/L NH3, and the trial was therefore aborted before the microbial community could be severely affected by the rising ammonia concentrations.

TAN levels in the reactor had not reached equilibrium when Phase 2 was terminated. The effect of mineralisation on the TAN level in the digester at steady-state could therefore not be determined.

5.3 Phase 3. March 2013 – July 2013. High OLR (5.0)

At the end of Phase 2 it was clear that the introduction of a mineralisation reaction prior to NiX treatment was not sufficient to ensure non-inhibitory ammonia levels. Two additional measures were thus undertaken to reduce the TAN content and the pH level in the digester.

Experiments showed that the nitrogen stripping efficiency in the NiX treatment could be improved with 10-15% by slaking the lime 15 minutes prior to usage. Henceforth all NiX treatments were performed with slaked lime.

Moreover, a method was developed to decrease the pH and reduce the residual lime after NiX treatment. The method developed, referred to as “pH neutralisation”, makes it possible to adjust the pH of the influent material to <7 (compared to pH 8.5 – 9 without pH neutralisation). Details regarding the pH neutralisation method are confidential as the method is patent pending. In summary the effect of the method is to neutralize the base effect of the added lime using the biogas produced by the AD process.

The development of key performance indicators 1-4 during Phase 3 may be seen in Figure 27 to Figure 30.

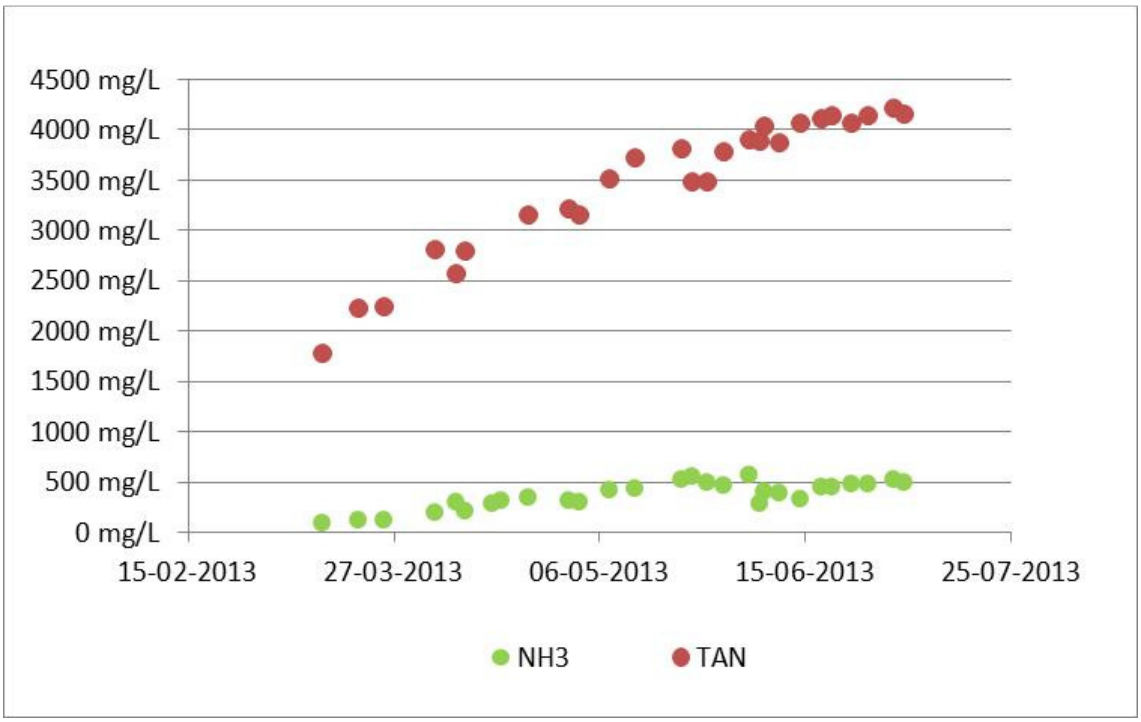

Figure 27 TAN and NH3 concentrations in the digester

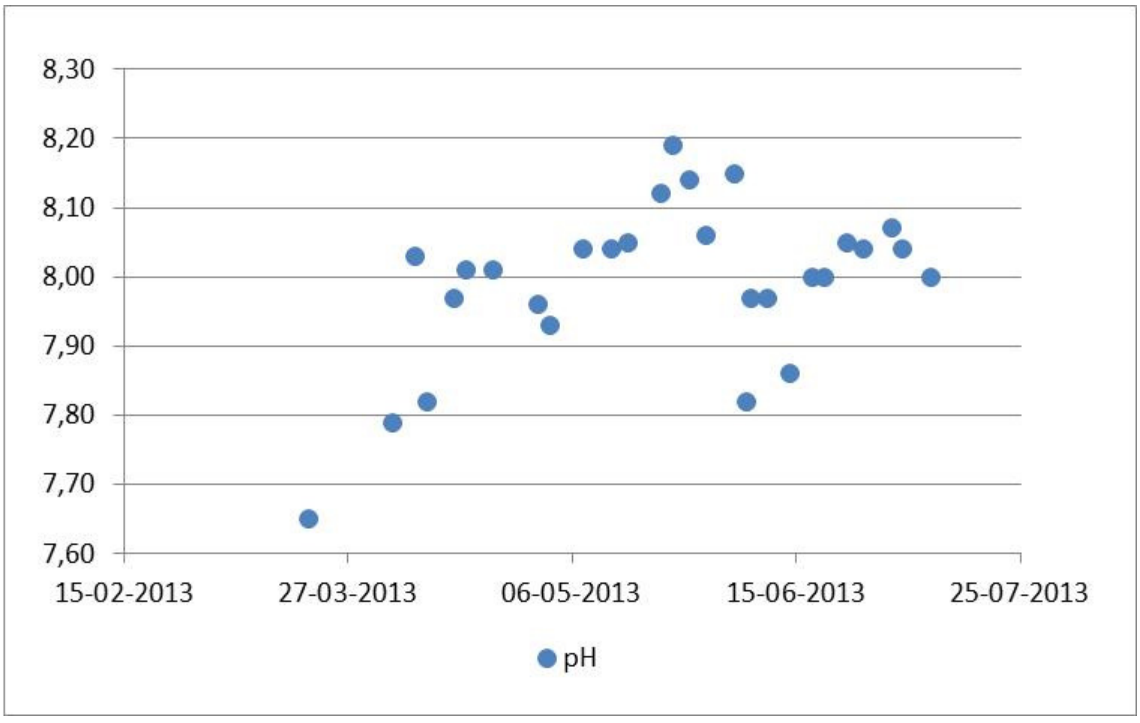

Figure 28 pH in the digester

As can be seen from Figure 27, TAN levels increase at a slower rate than in Phase 2 (see Figure 25). Steady state has not yet been reached but it appears that the equilibrium will be <4500 mg/L. Ammonia levels are also increasing at a significantly slower rate and are presently relatively stable at ~500 mg/L. Since TAN levels seem close to reaching equilibrium, the major contributor to the ammonia level is pH. As can be seen from Figure 28, pH is not yet stable although there is weak indication that it may stabilise between 8.0 and 8.1. If TAN levels settle at 4500 mg/L and pH at 8.1, ammonia levels will stay just below 600 mg/L which is generally considered to be the limit below which no ammonia inhibition can be observed.

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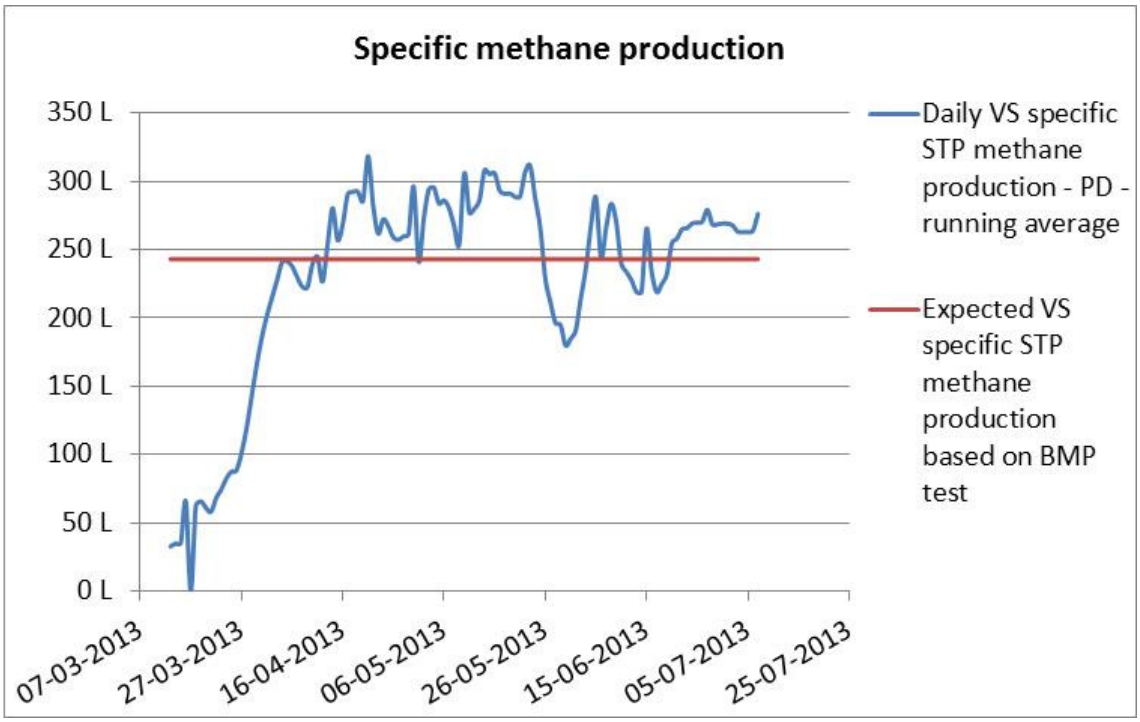

Figure 29 Specific methane yield per kg chicken litter VS added (7 day rolling average)

As seen from Figure 29 the methane production is relatively stable with an average of 267 (±3) NL/kg VS over the last 14 days. In comparison the predicted methane production is 243 (±18) NL/kg VS. The observed methane production is thus 10% higher than expected. More data are needed to determine the reason for this, but it may be explained by adaptation of the bacterial community to the specific substrate. In batch BMP assays the inoculum is taken from a digester which is not necessarily accustomed to the substrate being tested.

In the first half of the measured period the methane production seemed to stabilise close to 300 NL/kg VS. However, during this period the substrate used contained large amounts of wood shavings which had a tendency to clog the pipes in the digester. It is likely that the shavings in the outlet pipe functioned as a sieve filtering the VS material and artificially increasing the retention time and hence the degradation in the digester.

The large drop in methane production in the beginning of June is a result of technical difficulties due to the build-up of wood shavings in the pipes. At this point the digester material was cleaned for excessive shavings and the clots in the pipes removed. In the material used now the shavings are much smaller and make up a significantly less proportion.

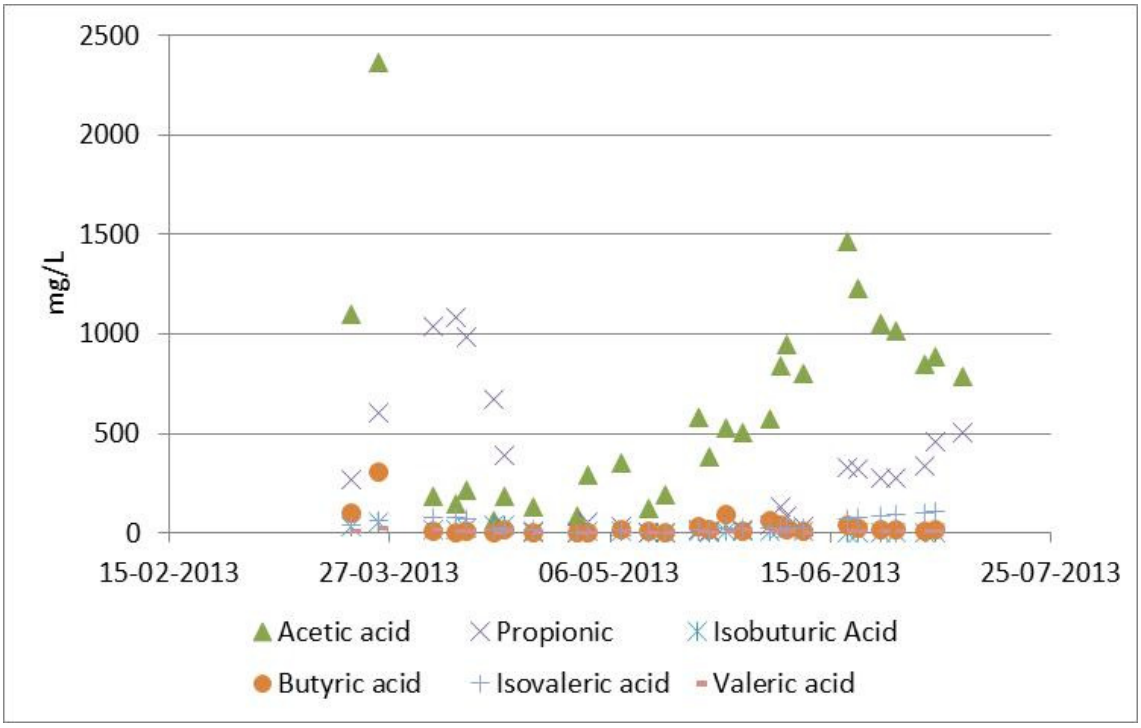

Figure 30 VFA in digester

Although the VFA development has not stabilised yet, the profile and distribution of VFA's seems healthy. The dramatic increase in the beginning coincides with the first substrate feedings to the pilot plant and a VFA increase as a consequence of this would be expected. Since then there has been a gradual increase of primarily acetic acid with propionic acid tailing the acetate increase. The high peak coincides with the technical problems mentioned in p. 27. During the last two weeks acetate decreased while propionate increased. Consecutive measurements have shown a decrease in both acetic and propionic acid concentrations (data not shown).

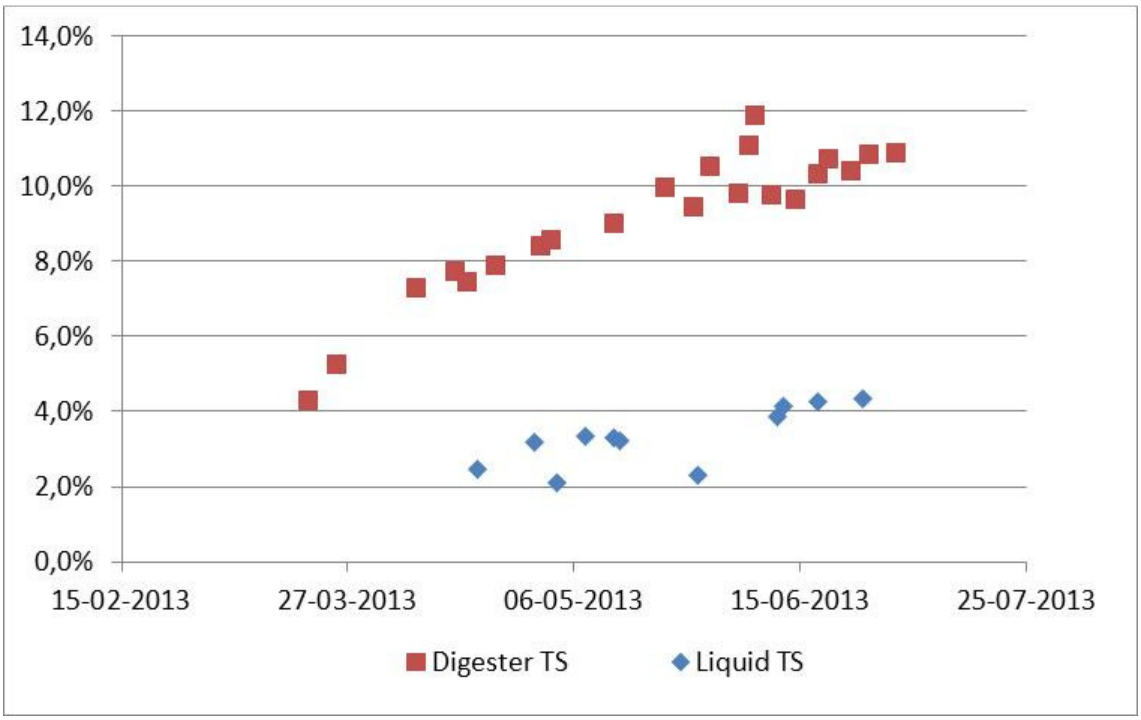

Figure 31 TS in digester and recycled liquid

The amount of total solids in the digester is still increasing but shows a tendency to stabilise around 11% TS. The peak at the beginning of June is due to the previously mentioned technical problems with the wood shavings in the biomass. The predicted TS level is between 10.5% and 11% depending on the extent of degradation in the digester.

The ability to maintain an effective separation is indicated by the TS content of the separated liquid. As seen by Figure 31 the total solids content is still rising. It is difficult to predict at what TS level the liquid will stabilise. However, since the TS content of the liquid in Phase 1 settled at approx. 8%, a similar value may be expected here.

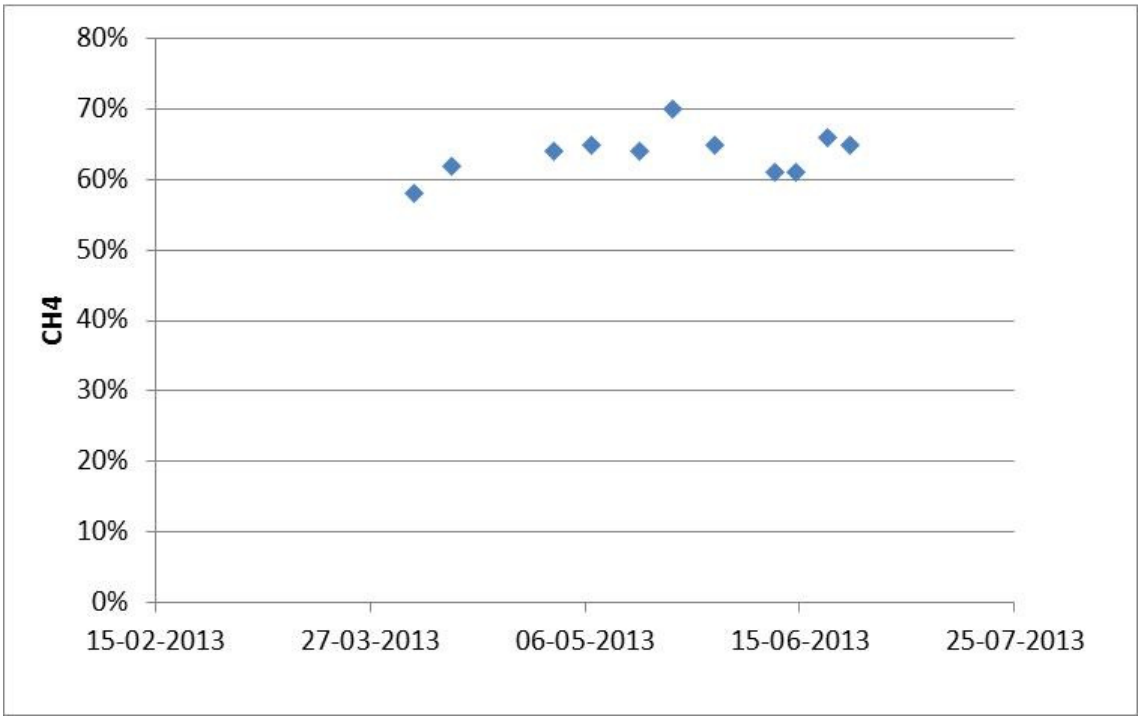

Figure 32 CH_4% in the biogas

The methane percentage in the reactor seems to have stabilised around 60-65%. The large peak at the beginning of June coincides with the technical difficulties mentioned on p. 27.

6 Discussion

6.1 Phase 1

The initiation of the constant drop in methane yield in Period 1A during the second half of December from 300 to 200 L CH4/kg VS coincides with [NH3] increasing to more than 1,000 mg/L. This could be seen as a confirmation of the expectation that a threshold value for NH3 inhibition exists although at a higher concentration level than assumed. Predicted methane yield in a continuous AD process under the given conditions is 250 – 260 L/kg VS indicating that the yield was negatively affected by the process conditions. The decrease in methane yield is followed by a very significant increase in all VFA species thus confirming the signs of inhibition of the methanogenic process.

The decrease in OLR in Period 1B initially led to an increase in the methane yield and a stabilisation of the VFA levels. However, within the first week methane yield began dropping steeply reaching a level of 70-80 L/kg VS a week later and continuing the decline during the next two weeks to a record low 40 L CH4/kg VS. Again the drop in methane yield occurred after a peak in [NH3] which went above 1,000 mg/L just before the start of the period. The gas yield stayed below 100 L CH4/kg VS for more than three weeks while the VFA's skyrocketed to more than 35,000 mg/L with unusually high contributions from the longer-chained VFA's. With [NH3] decreasing to around 600

mg/L by water dilution and later decreasing to less than 400 mg/L due to a drop in pH the methane began to increase to levels above maximum obtainable yields, which can explained by degradation of accumulated VS and specifically the very significant decrease in the acetic acid pool.

The step-wise increase in OLR from a low level in Period 1B to 3.24 g VS/(L digester x 24h) in Period 1C took place while the methane yield dropped to around 250 L CH4/kg VS thereby stabilising at a yield level close to the calculated prediction. Interestingly this yield level was maintained during a period with very high NH3 concentrations ranging from just below 1,000 to more than 2,000 mg/L. The high NH3 was due to high pH values as the TAN level was lower during the entire period than during the previous periods. The high pH values could be explained by the decrease in the total VFA pool, which dropped from an average 33,000 mg/L in the previous period to 18,000 mg/L. Adaptation of the AD process to high NH3 concentrations could be an explanation for the observed stable methane yield.

During period 1D a medium-level OLR was maintained and methane yields were quite stable during the entire period at a level around 270 L CH4/kg VS with an increasing trend. The tendency from the previous period with stable yields at a level corresponding to the calculated prediction was thereby continued and still with [NH3] above 1,000 mg/L. However, the continued high levels of particularly propionic acid (around 15,000 mg/L) but also other VFA's (e.g. iso-valeric acid at around 1,500 mg/L) indicated a strongly un-balanced AD process. Lack of pH-control in the digester was seen as the main reason for this by triggering a constantly high level of NH3 in the digester. It was therefore decided to terminate the experiment until a method for ensuring an acceptable NH3 level in the digester had been identified.

Adding to the NH3-level was the fact that TAN removal was not maintained at an average 65 % during the entire phase. This target level was achieved during longer periods but for reasons of optimisation of the NiX method where also energy and water consumption is critical sub-optimal TAN-removal was obtained during parts of the phase.

Dry matter content in the recycled liquid could not be kept at the target level of 3 % but increased to at or slightly above a constant level of 8 %. The issue of dry matter content in the recycled liquid is of importance not only for the water balance but also for the not well-understood potential negative effects on the AD process of a high suspended solids level in the digester. However, in spite of the elevated TS content in the recycled liquid and the consequential increased TS in the digester, methane yields at the end of the period were stable and high.

6.2 Phase 2

Prior to the initiation of Phase 2 effort was put into developing a method for ensuring an acceptable NH3 level in the digester. The method developed converts organically bound nitrogen into TAN and increases the strippable TAN pool fivefold, thus allowing for the removal of a significantly larger amount of nitrogen from the substrate. However, in order to remove more TAN, higher concentrations of burnt lime was needed, which in turn could lead to higher residual amounts of base being introduced into the reactor.

The pH development during Phase 2 was thus closely monitored to determine if the buffer capacity of the system was strong enough to counteract the increase in residual base. Unfortunately the ammonia concentration reached inhibitory levels after two months. At the end of the trial period pH had reached 8.3 and TAN concentrations were above 4500 mg/L. At this point the reactor contained 800 mg/L NH3, and since neither pH nor TAN showed any signs of stabilising, the experiment was terminated. It was decided to investigate methods for decreasing the residual amount of base after NiX treatment.

6.3 Phase 3

Between the termination of Phase 2 and the onset of Phase 3 a method was developed to adjust the pH and lower the residual amount of base in the substrate entering the digester. Specifics regarding this pH neutralisation method are confidential as it is patent pending. In summary the method makes it possible to decrease the amount of residual base using biogas and adjust the pH of the substrate to below 7. Without pH adjustment the pH of the substrate entering the digester would be 8.5 – 9.

With the inclusion of the pH neutralisation method the experiment has been running for 4 months and there is no sign of ammonia inhibition. The specific methane production is high (>250 L CH_4/kg VS) and the VFA levels are low (<1500 mg/L) indicating that the process is uninhibited. TAN and TS levels in the reactor have not yet stabilised completely but they show a solid trend and can be expected to stabilise at approximately 4500 mg/L and 12% respectively.

The rise in pH is significantly slower compared to Phase 2. However, pH levels are not yet stable and it is uncertain where they will settle. So far the combination of pH and TAN has not resulted in ammonia levels exceeding 600 mg/L at any time.

The TS level in the centrifuged liquid has not yet reached equilibrium. Presently the dry matter content of the liquid is approx. 5% and is still rising. During Phase 1 the TS of the centrifuged liquid reached 8%, and it is possible that this will be the steady state value of Phase 3 as well.

7 Appendix – Standard Operating Procedures

7.1 Mineralisering

Mineralisering foretages ved at blande substrat, væske samt seeding i en 120 L tønde. Seeding består af substrat og væske inkuberet ved 37 °C (står på øverste hylde i incubator 4).
Der ønskes 25 % TS i mineraliseringsmassen, samt 20 % seeding (ved 24 timers inkubering). Der opsættes ugentligt en mineralisering til 2 * 16 kg kogninger, samt til at opretholde seeding-puljen (ca. 2 kg ekstra), dvs. en samlet mineralisering på 34 kg.
Beregning af **Moypark** mængde (TS i Moypark er 59 %):

$$m_{Moypark} = \frac{m_{mineralisering} * TS\%_{mineralisering}}{TS\%_{Moypark}}$$

$$m_{Moypark} = 14{,}41\ kg$$

Beregning af **recirkulat** (der tages ikke højde for TS- % i recirkulat):

$$m_{recirkulat} = m_{mineralisering} - m_{Moypark}$$

$$m_{recirkulat} = 19{,}59\ kg$$

Beregning af **seeding** (20 %):

$$m_{seeding} = m_{mineralisering} * 0{,}20$$

$$m_{seeding} = 6{,}8\ kg$$

Efter blanding sættes tønden i incubator 4 (37° C) i 24 timer.
Der udtages prøve til analyse af den mineraliserede blanding. Prøveglasset mærkes ved hjælp af de forud printede labels (ligger i skuffen i bordet i forsøgshallen), hvorpå datoen for udtaget noteres. Prøveglasset sættes på køl til senere analyse.
HUSK: at notere ALLE afvejninger på data ark 1 "mineralization for pilot plant 1"

7.2 Læskning af kalk (1:3-blanding)

1. Der koges en portion vand i el-kedlen.
2. Der afvejes 1 kg H_2O, som et mix af det kogte vand og koldt vand. Start temperatur i denne portion H_2O skal være på 55° C
3. Afvej 1 kg CaO (står i en hvid spand øverst på stålreolen, ved den tekniske vægt) i en spand for sig
4. Afvej yderligere 2 kg H_2O i en spand for sig
5. Spanden med det 55° C varme vand placeres nu i et koldt vandbad, da der under de næste trin i processen vil ske en kraftig varmeudvikling
6. Tillsæt skiftevis under omrøring CaO og H_2O lidt ad gangen. Der fortsættes til alt det afvejede H_2O og CaO er brugt
7. Efter endt blanding efterlades denne fortsat i vandbad med løst låg i mindst 15 min. før end den læskede kalk kan anvendes
   HUSK: at notere ALLE afvejninger på data ark 5 "Slaked lime"

7.3 NIX-treatment/kogning

Inden behandling af det mineraliserede Moypark substrat, skal der udtages en prøve i et 180 ml prøveglas (se ark om mineralisering).

Der måles pH på det mineraliserede materiale, som noteres i data ark 2 ”NIX treatment for pilot plant 1”. Værdien noteres også på prøveglassets label.

NIX- kogeren opvarmes mens nedenstående procedure gennemføres. Opstart og klargøring af NIX-kogeren foretages i henhold til ”SOP for the NiX pressure cooking unit”.

Data ark 2 anvendes til at notere alle afvejningerne under hele nedenstående procedure:

- Afvej gryden (på gulvvægt)
- Tarér vægten med gryden på
- Afvej 16 kg mineraliseret Moypark i gryden (på gulvvægt)
- Afvej 1,792 kg* læsket kalk (svarende til 2,8 % kalk i en prøve på 16 kg) til kogningen (på teknisk vægt) – se beregningen længere nede.
- Afvej vand (på teknisk vægt)
- Tilsæt det afvejede kalk til gryden, skyl kalk-beholderen af med det afvejede vand før det også tilsættes gryden. Grydens indhold mixes grundigt – anvend evt. den elektriske om-rører.
- Tarér vægten uden gryden på
- Afvej gryden med indhold
- Gryden kan nu monteres i NIX-kogeren. **HUSK** at måle pH på grydens indhold inden låget monteres og noter denne på data ark 2. Målingen skal foretages 15 min. efter tilsætning af kalken

Følgende kogeparametre anvendes:

- Forvarmning ved 2 bar i 10 min
- Reducér tryk mellem forvarmning og kogning hurtigt (ca. 5-7 min med hanen åben til gryden og til slut også i kappen)
- Kogning ved 1,5 bar i 15 min. Hanen åbnes, når holdetiden startes og der laves en kraftig afblæsning over de 15 minutter. Det tilstræbes at holde et tryk i gryden på omkring 0,8 bar.

Straks efter endt kogning afmonteres gryden og afvejes igen med indhold. Vægten noteres på data ark 2. Der udtages en repræsentativ prøve i et 180 ml. prøveglas, som mærkes med de forud prin-tede labels, hvorpå datoen og klokkeslettet for behandlingen noteres. Der måles pH på den kogte

masse i prøveglasset efter afkøling til stuetemperature, som ligeledes noteres på data ark 2 og på prøveglasset. Prøveglasset sættes på køl til senere analyse.

Grydens indhold flyttes til en 60 L tønde. Skal det kogte materiale anvendes til indfødning på anlægget med det samme, foretages der straks efter endt kogning en pH-neutralisering af massen. Skal det IKKE anvendes med det samme fryses det straks.

Ved frysning mærkes tønden med en af de forudprintede labels, hvorpå der noteres dato og klokkeslet for kogningerne, samt hvor mange kilo kogt materiale, der opbevares i tønden. Dagens to kogninger fryses/opbevares i en og samme beholder (så længe den samlede kogeportion ikke overstiger 32 kg af gangen).

HUSK: at notere ALLE afvejninger på Data ark 2 - NIX treatment for pilot plant 1

*Beregning af kalktilsætning i form af læsket kalk gælder som følgende:

$$m_{læsket\ kalk} = \frac{m_{prøve} * 0{,}028}{0{,}25}$$

7.4 PH-neutralisering

Confidential.

7.5 Separation

Der foretages separation af det udpumpede materiale fra reaktoren. Væskefraktionen kaldet recirkulat genanvendes til både mineralisering og til pH-neutraliseringen før det igen tilføres reaktoren.

1. Afvej udpumpningstønde før start af separation.
2. Afvej recirkulatsbeholder og solid beholder (beholderen til den faste fraktion) før start af separation.
3. Der udtages prøve til TS/VS hver gang der startes på en ny udpumpningstønde. Der skal udtages prøve af de tre forskellige fraktioner (udpumpet materiale, væske og den faste fraktion). Vær opmærksom på at udpumpningstønden homogeniseres grundigt før udtaget.
4. Separation foretages ved hjælp af centrifugen. Tarér på centrifugeflasken med låg.
5. Afvej 600 gram udpumpet materiale (inkl. låg)
6. Centrifuger ved 3500 RPM i 20 min (indtil ny centrifuge modtages, da øges omdrejninger til 4000 RPM)
7. Når flaskerne er blevet centrifugeret hældes væskefraktionen i recirkulatsbeholderen (hvid 21,8 L spand) og den faste fraktion skrabes over i solid-beholderen. Herefter kan startes en ny centrifugering.

8. Når dagens separation er færdig afvejes udpumpningstønde, recirkulats beholder og solid beholder. Afvejningerne noteres på data-ark 4 (”Separation”).
9. Efter registrering af slut vægt på recirkulats beholder og solid beholder kan recirkulatet fryses i 16 kg portioner og mærkes med forud printede labels. Solidfraktionen skal gemmes i de blå 120 L tønder ved frysecontaineren med henblik på senere hygiejnisering. Hver tønde mærkes tydeligt med leverandørnavn (e.g. ”Moy Park”) samt at indholdet efterfølgende skal hygiejniseres før bortskaffelse.
10. Udpumpningstønde, recirkulats beholder samt solid beholder opbevares ALTID med låg på under og mellem de enkelte separationer for at undgå fordampning.

NB! Flaskerne og kopperne fra centrifugen bør rengøres/vaskes en gang om ugen.

Annex 5 CSTR Experiments on Straw (WP 7.2)

Project No.:	EUDP j.nr. 64011-0335 /Internal project 10842
Made by:	SBG
Issued:	December 3 2013

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1 Summary

This short analysis report summarizes the development of an experiment testing the suitability of grinded wheat straw as a substrate in a pilot scale CSTR. The purpose of the experiment has been to investigate the stability of the biological process and identify possible technical issues.

The wheat straw used was finely ground using a milling technology supplied by Jäckering Group[1] to a particle size below 0.5 mm to facilitate suspension and increase the biogas potential. In order to suspend the wheat straw powder, pig manure was used as a carrier substrate.

The CSTR trial was divided into three stages. The purpose of the first stage was to establish a baseline for the biogas production from pig manure only. In the second stage a diluted mixture of wheat straw powder and pig manure was introduced to reveal any technical or biological problems early on and allow the biological process time to acclimatise to a higher organic load. In the third stage the final concentrated mixture of wheat straw and pig manure was introduced.
The duration of the first and third stage was three hydraulic retention times (~75 days), since the purpose of these stages was to determine the biogas production and stability of the process. The second stage needed only to be one HRT since it was introduced as an intermediate step towards the high concentration substrate.

The parameters of the CSTR setup may be seen in Table 1.

Parameter	Value
Substrate (first stage)	Pig manure
Substrate (second stage)	Wheat straw (6%) and pig manure (94%)
Substrate (third stage)	Wheat straw (18%) and pig manure (82%)
Reactor temperature	37 ℃
HRT	25 days

Table 1 – CSTR trial conditions. Substrate percentages are weight percentages in terms of wet weight.

2 Conclusion

During stage 1, the baseline biogas production of pig manure was successfully established. However, shortly after introduction of the diluted mixture of wheat straw and pig manure, the pump associated with feeding into the anaerobic digester experienced problems due to the viscosity of the mixture, and had to be replaced with a pump with a higher capacity.

Although this solution alleviated the problem during stage 2, the final mixture of wheat straw powder and pig manure was so viscous, that is was not possible to go through with stage 3 without substantial modifications to the pilot plant. The experiment has shown that mixtures up to 6%

[1] Jäckering Gruppe, Vorsterhauser Weg 46, 59067 Hamm

wheat straw powder are pumpable and may be used as a substrate without modifications, whereas higher mixture ratios need a feeding system designed for high solid products. It was not possible to determine the biological stability of a process in which the main biogas production comes from wheat straw.

Annex 6 Design of Demonstration Plant (WP 8)

Project no.:	EUDP j.nr. 64011-0335 /Internal project 10842
Made by:	FR
Issued:	December 3 2013

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0 Resumé

During the engineering part of the project the following results have been reached and documented:

1. **Detailed mass balance, energy balance and nitrogen balance** have been calculated for different combination of feedstocks and plant size as well for a demonstration plant in Ribe.
2. Optimizing **heat/steam consumption** has been performed in the project.
3. In connection with item 1 a **general NIX calculation tool** has been developed, and is today in use for calculation of new Nix projects in Xergi.
4. **General plant layout** (1,5 MW, 3 MW, 6 MW) as well as specific **Ribe biogas plant concept** and site layout has been made as well as building layouts and optimizing activities has been made.
5. **PI diagrams**, corresponding **component list and functional description** have been made.
6. For special component **lime dosing, nitrogen flash, ammonium absorption t**here have been an intensive design activity an evaluation of different potential sub suppliers.
7. Different **straw handling technologies** has been evaluated included capacity test and efficiency test done at a supplier in Germany. The feasibility of the evaluated technologies was too bad to continue with these technologies. Instead Xergi decided to develop its own technology, named X –chopper.
8. The new **X chopper** have been made in a **full scale proto type** which is installed in V. Hjermitslev Biogas plant. It has shown a god operational performance and the first commercial version has been sold to a Danish biogas plant.
9. An intensive development work has been made to design **a new batch cooker**, dedicated to biogas plant. The new cooker will reduce the heavy power consumption to only 40 % of the original and at the same time the price per capacity (DKK/tons pr. day treated) will be reduced by 25 %. Xergi has filed a **patent application** for this new design.
10. The activities done in the project had formed the basis for Xergi to obtain a **supply contract for a 3 MW plant in UK using the Nix technology** as well as a design contract for a 3 MW plant to NI operating purely on chicken litter.
11. Xergi´s **market evaluation** of tariffs for biogas production and biomass situation has led to the conclusion our focus for the **Poultry Power®** concept will be **UK** where Xergi already are present and **Italy** which is a new Country for Xergi to handle. For the **Manu Power®** concept focused on cattle deep litter and fibers from the back end of biogas plant, we need a commercializing of the new batch cooker, which will **Denmark** in the first round and properly **France** afterwards.
12. Xergi has identified the need for **demonstration plant** for testing the new batch cooker in a **full scale proto type** version before it can be implemented in bigger commercial plants. Xergi and its project partners had therefore **applied EUDP** for a grant to do this testing with special focus on deep litter.

1 Massebalancer og kapacitetsoptimering

Tidsoptimering

For at kunne opnå størst mulig gevinst ved et NiX forbehandlingsanlæg på et biogasanlæg, er det nødvendigt designe anlægget, således komponenterne har den rigtige størrelse og kapacitet i forhold til at opnå optimal udnyttelse af hele anlægget.

Overordnet set optimeres reaktorer og gasmotoranlæg således at netop driften af gasmotoranlægget vil være i omegnen af 8000 timer per år. Dvs. mængden af biomasse til rådighed afstemmes med en given motorstørrelse. Derudover afstemmes reaktorkapaciteten sammen med kapacitet på modtagefaciliteter. Til sidst kan størrelsen af trykkogeren bestemmes.

Der findes i dag nogle forskellige størrelser af trykkogere på markedet som der i projektet er arbejdet på i forbindelse med at vurdere/beregne kapaciteter. De størrelser der har været arbejdet med er på 5000 liter, 8500 liter, 10.000 liter, 16.000 liter og 28.000 liter. Derudover er der regnet på 2 styk mindre trykkogere som alternativ én større trykkogere.

Generelt er dimensioneringen af selve biogasanlægget ikke problemstillingen. Problemstillingen har været at vurdere hvilke kapaciteter der kan opnås med de forskellige trykkogere. Kapaciteten afhænger af følgende parametre: fyldningstid, opvarmningstid, holdetid, trykaflastningstid og tømmetid. Det er særligt opvarmningstid, trykaflastningstid og tømmetid som er de vanskelige at bestemme. Opvarmningstiden bestemmes af, hvor hurtigt dampen kondenseres i trykkogeren og af biomassen. Trykaflastningstiden bestemmes af hvor vanskeligt det vil være at trykaflaste uden risiko for medrivninger/opkogninger af biomassen til trykaflastningssystemet. Tømmetiden bestemmes af hvor hurtigt biomassen kan ledes ud af udgangsstudsen ved hjælp af omrøreren i trykkogeren. Fyldningstiden beregnes forholdsvist simpelt da pumpe-/sneglekapaciteter kan oplyses af producenter.

Til bestemmelse af kapaciteten på givne trykkogere er der udarbejdet et Excel regneark, hvori de enkelte trykkogere er opstillet/sammenlignet. Det er således muligt at ændre input biomassen, og deraf få beregnet kapacitet på baggrund af specifik biomasse. Som eksempel på en beregning er anvendt nedenstående mængde biomasse:

Biomasse i eksempel

Biomasse	**Ton/år**	**Vægtfylde, kg/m³**	**Tørstof, %**	**Specifik varmekapacitet**
Gødning, æglægger (etagesystem)	34.000	600	35	3,22
Væskefraktion fra separation	24.000	1000	3,5	4,10
Kalk	1624	900	100	1,40
Total	**59.624**	**723**	**24,1**	**3,53**

Som eksempel tilføres der således ca. 163 ton/døgn. I beregningen af hvad de enkelte trykkogere kan præstere er forudsætningen af alt biomassen blandes i en fortank det det forinkuberes ved 30-37 °C (anden teknologi som ikke beskrives her). Det forudsættes at blandingen i tanken kan omrøres og er pumpbart. I scenarie 1 forudsættes en fyldningskapacitet på 40 m³/time. Opvarmnings, trykaflastnings- og tømmetiderne er målt på et eksisterende biogasanlæg hvor NiX forbehandling er implementeret. Via op- og nedskalering er tiderne for opvarmning, trykaflastning og tømning estimeret.

Scenarie 1

	5000 liter	**8500 liter**	**10000 liter**	**16000 liter**	**28000 liter**
Fyldningstid, min.	4,7	7,9	9,3	14,9	26,1
Opvarmningstid, min.	1,3	2,2	2,6	4,2	7,3
Holdetid, min.	0	0	0	0	0
Trykaflastningstid, min.	18	20	20	23	30
Tømmetid, min.	4	8	10	15	20
Total tid, min	**54**	**66**	**70**	**87**	**120**
Kapacitet, ton/døgn	**58**	**81**	**90**	**114**	**146**

Af scenarie 1 fremgår det således til at behandle 163 ton biomasse/døgn ikke kan opnås ved brug af kun én trykkoger. Aktuelt vil 2 stk. 10.000 liter kunne løse opgaven.

Massebalance

Udover at der anvendes energi til opvarmning/trykkogning af biomassen, går der også energi til opvarmning af stålet i trykkogeren, som skal tages med i beregningen af energiforbrug.
Med udgangspunkt i beregningen i scenarie 1 hvor 2 stk. 10.000 liter trykkogere kan håndtere de 163 ton biomasse/døgn er det beregnet, hvor meget energi der skal anvendes for at opvarme biomassen fra ca. 30 °C til 140 °C og stålet fra 95 °C til 140 °C, hvilket ses i nedenstående tabel.

Energiforbrug 10.000 liter trykkoger

Energiforbrug	**10.000 liter**
Biomasse, kWh/batch	469
Stål, kWh/batch	100
Total varmeforbrug	569
Antal batch/år	13.745
Total energiforbrug, kWh/år	7.821 MWh

Anlægsdesign

Med udgangspunkt i scenariet, hvor 34.000 ton æglæggergødning behandles, er der designet følgende anlæg:

Modtagetank	2 x 250 m³
Buffertanke	2 x 250 m³
Trykkogere	2x10000 liter (+1 x 10000 liter)
Kalksilo + læskningstank	50 m³
Xergi doseringsmoduler	6 x 20 m³
Primær reaktortanke	2 x 8000 m³
Sekundære reaktortanke	1 x 4500 m³
Dekanter	1 x 30 m³/h
Buffertank	1 x 50 m³
Efterlagertanke	1 x 3500 m³
Gasrensning + køling	1 x 1500 m³/h
Gasmotoranlæg	2 x 1487 kW
Majslinje	
Faststofmodtagning + neddeling	2 x 80 m³

2 Energibalancer og energioptimering.

Energioptimering

I forsøget på at gøre NiX forbehandling så økonomisk som muligt er det nødvendigt at have en så høj starttemperatur på biomassen. Udover at det kræver meget energi at producere damp, kræver det også en særlig forbehandling af vandet, der laves til damp, hvilket er omkostningstungt. Derfor har én af problemstillingerne været at lave et design, hvor biomassen forvarmes mest muligt med lavtemperaturvarme fra enten varmeveksling med færdig trykkogt biomasse (NiX behandlet biomasse) eller ved anvendelse af overskudsvarme fra kølevandkredsen på gasmotoren.

Biomassen, der typisk forbehandles ved NiX metoden, er husdyrgødninger som dybstrøelse fra kvæg/mælkeproduktion, fjerkrægødning fra æglæggere i etagesystemer eller dybstrøelse fra skrabeægsproduktion og slagtekyllingegødning.

Det trykkogersystem, der er arbejdet med, består af en vandretliggende tank i størrelserne 5-28 m³. I selve trykbeholderen er der monteret en vandretliggende omrører som skal holde biomassen omrøreret og fordele dampen i biomassen. Erfaringer fra producent af trykkogersystemet samt erfaringer fra håndtering af fiberholdige biomasser i biogasanlæg viser, at trykkogningsudstyret formentlig vil kunne håndtere biomassen, såfremt tørstofindholdet i trykkogeren ikke overstiger 22-24 % før trykkogning. Efter trykkogning vil tørstofindholdet falde til omegnen af 20-22 %. Da gødningerne kan have et tørstofindhold på op mod 60-65 %, kan det være relevant at iblande væske for at nå et tørstofindhold på 22-24 %. Der vil kunne anvendes flere væsker til formålet heriblandt, gylle, vand og væskefraktion fra bagende separeret afgasset biomasse. Da mængden af væske/afgasset biomasse fra bagenden af biogasanlægget har betydning for driftsomkostningerne for biogasanlægget, er det sjældent en økonomisk fordelagtig at bringe biomasse ind i biogasanlægget, der ingen gaspotentiale har og som senere skal bringes ud på marken. Derfor vil det optimalt set være bedst at anvende gylle eller alternativt væskefraktionen fra en separation.

I forsøget på at nedbringe energiforbruget på forbehandling ved NiX er det som tidligere nævnt relevant at øge temperaturen af biomassen før dampindblæsningen startes. Da dybstrøelse og fast gødning har et højt tørstofindhold, er det en udfordring at ville hæve temperaturen af den del. Derimod findes der udstyr i form af varmevekslere som kan håndtere gylle og væskefraktion fra en separator med et begrænset tørstofindhold. Det er således realistisk at gyllen/væsken, der anvendes til at opnå det rette tørstofindhold i trykkogeren, kan hæves fra 5-10 °C til 60-80 °C.

Alternativt kan væsken/gyllen opvarmes ved at anvende den som kølemedie i "flash-tanken", hvori trykaflastning fra trykkogning sker. Da formålet med trykkogning er afstripning af ammoniak fra biomassen, vil ammoniakken blive absorberet i kølemediet med mindre kølemediet er pH reguleret med kalk. Det betyder, at gyllen/væsken efterfølgende kan anvendes i trykkogeren sammen med dybstrøelsen/fast gødning.

De to metoder vil give samme resultat, at gyllen/væsken forvarmes med et reduceret energiforbrug i trykkogningen til følge. For at illustrere ovenstående er der udarbejdet beregninger, der viser besparelsen ved at forvarme biomassen.

I scenarie 1 er der opstillet et eksempel for et anlæg for væsken, der anvendes til fortynding til ca. 22 % tørstof ikke forvarmes, men i stedet pumpes direkte i trykkogeren med en temperatur på 32 °C svarende til temperaturen i den sekundære reaktortank inkl. et mindre varmetab. I skemaet nedenfor ses mængden der kunne anvendes i et NiX-anlæg, tørstof for biomassen, indgangstemperaturen samt den estimerede specifikke varmekapacitet for hver enkelt biomasse.

Scenarie 1:

Biomasse	Mængde	Tørstof	Temperatur	Specifik varmekapacitet (Estimeret)
Slagtekyllingegødning (NiX)	23.500 ton/år	60 %	15 °C	2,52 kJ/kg°C
Væskefraktion fra separation (NiX)	49.000 ton/år	2,5 %	32 °C	4,13 kJ/kg°C
Kalk	1.088 ton/år	100 %	60 °C	1,40 kJ/kg°C
Sum/Gennemsnit	**73.588 ton/år**	**22,3 %**	**27,0 °C**	**3,58 kJ/kg°C**
Energiforbrug	**112 kWh/ton**			

Af scenarie 1 ses det, at der skal anvendes 124 kWh/ton biomasse for at opvarme biomassen fra 27 °C til 140 °C. Tilsvarende er energiforbruget beregnet hvis væsken opvarmes til 80 °, se scenarie 2.

Scenarie 2:

Biomasse	Mængde	Tørstof	Temperatur	Specifik varmekapacitet (Estimeret)
Slagtekyllingegødning (NiX)	23.500 ton/år	60 %	15 °C	2,52 kJ/kg°C
Væskefraktion fra separation (NiX)	49.000 ton/år	2,5 %	80 °C	4,13 kJ/kg°C
Kalk	1.088 ton/år	100 %	60 °C	1,40 kJ/kg°C
Sum/Gennemsnit	**73.588 ton/år**	**22,3 %**	**58,9 °C**	**3,58 kJ/kg°C**
Energiforbrug	**81 kWh/ton**			

Af scenarie 2 ses det at der kan spares i omegnen af 38 % energi hvis væskefraktionen, her væskefraktion efter separation opvarmes til 80 °C. Udover at kunne spare energi i form af damp for man samtidig nyttiggjort varme fra processen enten energi fra trykaflastning eller varmen fra kølekredsløb på gasmotoren.

Varmevekslingssystemer

Ved anvendelse af et varmevekslingssystem vil der være nogle problemstillinger, som der skal tages højde for ved dimensionering. Når biomasse som gylle eller væskefraktion fra en separation opvarmes i en veksler som typisk kan være en spiralvarmeveksler eller en rørvarmeveksler, vil der være risiko for, at biomassen brænder fast på veksleren, hvorved varmeledningen reduceres.

Ifølge flere producenter af varmevekslere opstår på brændingen fordi temperaturdifferencen på biomassen og fjernvarmevandet er for stor. På brænding kan dels afhjælpes ved at dimensionere vekslerne til et højt nok flow af biomasse igennem eller via rengøring med kemikalier. Hvis flowet er tilstrækkeligt højt (over 20 m^3/time) vil vekslerne være selvrensende. I alle tilfælde vil det være nødvendigt at dimensionere ud fra enten at have en høj nok kapacitet på varmevekslerne eller at have ekstra kapacitet til rådighed i perioder hvor vekslerne er ude til rensning.

3 P&I diagrammer

For at forberede designet af NiX forbehandlingsanlæg for nem tilpasning til forskellige typer af biogasanlæg (biomasser) har vi valgt at lave Proces og Instrumenterings diagrammer (PI-diagrammer) for de enkelte delmoduler, som et NiX anlæg kan sammensættes af.

For en nærmere forklaring af hele NiX processen henvises til funktionsbeskrivelsen, der understøttes af PI-diagrammerne.

4 Komponentlister

Der er lavet en samlet komponentliste, der dækker over de komponenter, der indgår i et NiX forbehandlingsanlæg. Komponentlisten er ikke færdigstruktureret fuldstændigt, og der skal arbejdes videre med at opsplitte listen i en række under komponentlister, således det bliver nemt at tilpasse et NiX forbehandlingsanlæg til forskellige typer af biogasanlæg (biomasser).

5 Trykkoger

Udvikling og design af trykkoger

NiX konceptet er nøje forbundet til trykkogning af biomassen og et effektivt udstyr til kogning, og håndtering af biomasser med et meget højt tørstofindhold er derfor afgørende for processen.

De typer af trykkogere/tørsmeltere, der findes på markedet i dag, er som udgangspunkt udviklet til fremstilling af benmelsprodukter. Produktion af benmel stiller imidlertid andre krav til omrøring under kogeprocessen, og trykkogeren er derfor forsynet med meget store gearmotorer med et meget højt energiforbrug. Ligeledes er kogeprocessen baseret på nedtørring af benmelet i kogeren. De eksisterende trykkogere på markedet giver anledning til lange tømme- og fyldetider, og ikke baseret på kogning af biomasser med et højt fiberindhold.

Ud fra ovenstående problemstillinger er der iværksat et udviklingsprojekt, hvor der ønskes designet en ny trykkoger, hvor designet er optimeret i forhold til de biomasser der ønskes forbehandlet til biogasproduktion.

Det forventes at kunne producere en højeffektiv trykkoger, der har et energiforbrug, der er ca. 60 % lavere end ved den kendte teknologi. Ligeledes forventes det at tømme- og fyldetider vil kunne reduceres væsentligt, hvorved kapaciteten øges betydeligt.

5.1 Udvikling af ny type batchkoger

Det er besluttet at udvikle trykkogeren sammen med maskinværdstedet Lildal der har mange års erfaring indenfor produktion og levering af trykkogeranlæg. Da det samtidig er besluttet at søge patent på de basale metoder og udformninger der ligger til grund for den nye trygkoger er det ikke muligt at beskrive disse i denne rapport.
Den nye trykkoger forventes at ville :

- kunne komprimere luftholdige biomasser som dybstrøelse bedre.
- kunne tømme batchkogeren betydeligt hurtigere.
- Reducere prisen med ca. 30 %
- Reducere elforbrug og installeret effekt med op til 60 %. (traditionel 16m3 koger er der monteret ca. 110 kW)

Prøve model (trykløs)er sendt fra Lildal til test plads i Jammerbugt kommune for opbygning af test opstilling. Test af forskellige dybstrøelses typer er gennemført.

Film fra test kan ses på Xergi arkiv S:\Product Developement\Nix udvikling\1.0 Generelt\Foto og film.

Testen har vist at de ønskede effekter kan opnås. Næste step er at designe og fremstille en prototype / demo model.

Lildal har udarbejdet grunddesign af den nye type trykkoger og den grundlæggende form og konjektur er fastlagt.

Parterne er klar til at lave demomodel til Ribe projektet.

6 Indfødning af biomasser

6.1 Generel problemstilling Fjerkræmøg.

Indfødningssystemet til både æglægger og slagtekyllinge møg er relativt ukompliceret sammenlignet med øvrige biomasser der normalt håndteres, hvorfor der ikke er fokuseres disse teknologier i dette projekt.

6.2 Generel problemstilling kvægdybstrøelse

De generelle problem med kvæg dybstrøelse til biogasanlæg er :

1. Svær at håndtere i indfødning og giver flydelag i reaktor, samt stenbelastning.
2. Meget lavt energi udbytte (40 – 45 % nedbrydning)
3. Stor mængde fiberfraktion til bortskaffelse p.a. lav nedbrydning

Ovenstående problemstillinger er så væsentlige, at kvæg dybstrøelse stort set ikke anvendes til biogasproduktion, på trods af at der er meget store tilgængelige ressourcer, samt at mange biogasanlæg mangler biomasser.
NIX forbehandling har ved batch udrådning, vist et forbedret udbytte på ca. 45 % og samtidig forventes flydelags opbygningen reduceret markant. NIX forbehandling ser således ud til, at kunne eliminere de væsentligste problemstillinger omkring brug af dybstrøelse i biogasprocessen.

Nye problemstillinger der skal håndteres er håndteringsproblemer i tilknytning til selve NIX forbehandlingen. Hvilket udviklingsarbejdet er målrettet mod.

Kvæg dybstrøelse findes i mange varianter, men kan i vores sammenhæng inddeles i 4 hovedtyper.

A. Dybstrøelse baseret på snittet eller andre former for neddelt rent halm
B. Dybstrøelse fra mælkebesætninger(traditionel dybstrøelse)
C. Dybstrøelse fra tyrekalveopdræt(komprimeret hårdt, 6mnd)
D. Dybstrøelse fra drægtige køer og kvieopdræt. (meget halm)

Type A er relativt uproblematisk, men kan fortsat give flydelagsproblemer.

Type B og C (som også indeholder en andel af D) er den traditionelle dybstrøelse med høj vægtfylde, men som kræver oprivning og neddeling.

Type D der typisk kommer som et sideprodukt i forbindelse med malkekvægs besætninger på spalte gulve. Dybstrøelsen stammer fra opdræt kalve og ungdyr og kan have en stor variation fra næsten rent halm med foderrester, til en mere bastant og gennemvædet type. Denne store variation, vil være den mest problematiske at håndtere. Der findes imidlertid meget store mængder med denne sammensætning og det vil være attraktivt for besætninger af denne type at få dybstrøelsen vekslet til gylle. Denne omveksling giver ligeledes en positiv fordel for opgørelse af dyreenheder. Ydermere vil der være en bedre udnyttelse af gødningsværdien i den konverterede dybstrøelse. Der vil derfor kunne komme meget af denne type biomasse til biogasanlægene
Der gennemført udrådnings forsøg med forskellige typer af dybstrøelse fra kvægbesætninger. Forsøgene er afsluttet og dokumenterede (se WP3 rapport)

Det kan umiddelbart konkluderes at mængden af halm og halmtypen har indflydelse på gaspotentialet i dybstrøelsen.

Dybstrøelse indeholder ofte lange halmstrå, der gør det svært at transportere og samtidig kan vægtfylden være meget lav, så en fylde proces kan være meget tidskrævende, hvorved kapaciteten bliver lav.

6.3 Basis løsning

Biomixer af typen Echart (leveres til Tiper)eller Konrad pumpe (leveret til Foulum) der kan neddele halm blandet med dybstrøelse.

Dobbelt snegl til batch koger.

Udviklingstest gennemført viser at alm dybstrøelse (Typer abc) kører med forventet kapacitet gennem biomixer i Foulum og er ligeledes testet fuldskala på Echart i Østrig.

Udviklingstest med type D dybstrøelse for udmadningskapacitet (tons pr time) er testet i Foulum. Resultatet af testen viste at kapaciteten på udfødningen fra biomixeren er afhængig af i hvilken grad at dybstrøelsen er neddelt, varierende fra 1200 kg/time ved maks. nedelling til 4932 kg/time ved en middel neddeling. Den gennemsnitlige vægtfylde for den mixede dybstrøelse vil være ca. 430 kg/m3. Den endelige vægtfylde vil blive beregnet ved testen på OL.

Test af trykkoger på Over Løjstrup er gennemført. Formålet med testen var at belyse hvilken fyldningsgrad der kan opnås med neddelt dybstrøelse. På trods af en relativ høj vægtfylde i dybstrøelsen (520 kg/m3) kunne der ikke opnås den ønskede fyldningsgrad på 360 kg/m3 volumen i trykkoger. Den opnåede fyldningsgrad på 237 kg/m3 volumen i trykkogeren. Der er udarbejdet en samlet rapport over test forløbet med biomixeren i Foulum og trykkogeren i Over Løjstrup.
Som en nøglekomponent til forbehandling i NIX konceptet, er kæde-neddeleren identificeret som en solid og mulig løsning. Det er besluttet at udvikle Xergi`s egen model af dette udstyr. Dette vil sikre at der vil kunne opnås en meget høj fyldningsgrad i trykkogeren. Endvidere vil kædeneddeleren formentlig gøre det muligt at føde direkte med kalvedybstrøelse.

Der er gennemført et udviklingsprojekt med udvikling af en kæde-neddeler specifikt designet til neddeling af biomasser som dybstrøelse m.v.. Produktionsgrundlaget er udarbejdet og ejes af Xergi.

Kæde-neddeleren færdig produceret. Vester Hjermitslev Biogas er klar til at indgå i et testforløb. Vester Hjermitslev Biogas stiller faststofmodtager til rådighed for testen.

De første test er gennemført og det tegner meget positivt.

Kæde-neddeleren indgår nu i den daglige drift hos Vester Hjermitslev Energiselskab. Der er pt. neddelt ca. 100 ton dybstrøelse fra kvæg.
Testen har trods udfordringer med kæden vist en meget positiv tendens hvad angår kapacitet og effektforbrug.

Der ligger stadig en udfordring i at finde den optimale udformning af kæden til neddelingen. Kæden skal ses som er en sliddel, som bør have en acceptabel levetid. Det står i imidlertid klart, at dybstrøelse generelt udøver et voldsomt slid på kæden. Dette kan skyldes at der er en relativ stor mængde at sand i dybstrøelsen.

7 Kalkdosering og base

I NiX konceptet indgår kalk eller anden base som en vigtig komponent i processen med at afdrive kvælstof fra biomassen der ønskes behandlet. For at kunne regulere pH i processen nøjagtigt er det vigtigt at kunne dosere basen i automatisk anlæg og med en pasende nøjagtighed.

Der ønskes derfor udviklet et system der er i stand til at udføre denne proces, uden at der skal anvendes manuelle resurser i processen.

De kontinuerte test i Foulum (se WP 4 rapport) har vist at der kan blive opløsningsproblemer med brændt kalk i pulverform. Det er derfor fundet nødvendigt at ændre systemet fra en direkte tørstofdosering til en våddosering, hvor den brændte kalk læskes op i et forbehandlingssystem.

Hermed mistet varmebidraget fra læskningsprocessen, men det giver større sikkerhed for at der ikke opstår ph problemer i den efterfølgende biogasproces.

8 Kontinuert kogning

Det nuværende anlæg bygger på en batchproces, hvor biomassen pumpes ind i en tryktank. Her opvarmes den til ved tilførsel af damp direkte ind i biomassen - eller energien overføres via trykkogerens kappe.

I projektet er der gennemført tekniske overvejelser om konceptet kan ændres til et kontinuert kørende system. Men der er ikke fundet en løsniing der på en gang kan håndtere kravene om tryksætning af frisk biomasse, flash af ammonium og efterfølgende udmadning i trykløs tilstand.

9 Flashsystem

Mulige flashsystemer

Helt principielt kan flashsystem udføres enten i separat flashtank – eller eventuelt i kombination med absorbersystem.

I forsøg på at klarlægge mulighederne er der ført dialog med svensk/tysk firmakonstellation om kombinationsløsning samt tidligere samarbejdspart om separat løsning for flash tank med efterstående luftabsorber.

Efter gennemgang af modtagne tilbud – på kombinationsløsning og ved sammenligning med tidligere indhentede priser på luftabsorber i kombination med tilbudt flashtank, så indikeres det overordentlig kraftigt, at anlægsprisen for en kombinationsløsning er mindst dobbelt – måske nærmere 3-dobbelt i forhold til separat flashtank med efterstående absorber.

Valgt løsning

I medfør af den åbenlyst væsentlige prisforskel er det vurderet at fortsætte med "kendt" løsning med separat flashtank og efterstående absorber.
Dette uanset at driftsomkostningen til især dampfremstilling hermed ikke reduceres – som alternativ løsning måske muliggør.

Produktudviklingstiltag for flash tank

Fremstilling af flashtank er aftalt med vor faste leverandør af koger. I samarbejde er der udviklet et xls beregningsark, som kan håndtere den energimæssige side – idet at nødvendigt kølemiddel kan være både vand – og alternativt dekanteret biomasse, typisk fra biogasanlæggets bagside.
Dette beregningsark er allerede taget i praktisk anvendelse som grundlag for kontraheret flashtank på engelsk projekt.

10 Ammoniakabsorber

Mulige absorbersystemer

Valget af absorbersystem hænger nøje sammen med valget af flashsystem. Idet det er valgt at fortsætte med separat flashtank løsning – så er absorbersystem implicit også valgt som en luftabsorber.

Valgt løsning

Der er ført intensiv – og meget langvarig dialog med både p.t foretrukken mulig dansk underleverandør.

I medfør af den åbenlyst væsentlige prisforskel er det vurderet at fortsætte med at frembringe en løsning, som er baseret på fremstilling i PE materiale, af prismæssige årsager.

Der har supplerende været ført indledende dialog med alternativ dansk leverandør – som primært fremstiller absorbere i rustfri stål materialer.

Produktudviklingstiltag for absorber

Dialogen med fortrukken leverandør har været midlertidig underdrejet grundet sygdom hos leverandøren. Dialogen er genoptaget pr. maj 2013, og leverandøren er fremkommet med ønske om inddragelse af Teknologisk Institut. Der vil snarest blive berammet mødeafholdelse som grundlag for kontrahering til engelsk projekt.

11 Dampproduktion

Scenarier for dampproduktion med forskellige størrelser af biogasmotorer

I forbindelse med udarbejdelsen af et optimalt design for et NiX anlæg er der arbejdet med dimensionering af gasmotorinstallationen. NiX forbehandling vil kræve en større mængde biomasse for at opnå økonomisk gevinst at en ekstra investering. Derfor er der lavet beregninger med gasmotorstørrelser på 1.487, 1.817, 2.433, 1.560 og 2.000 kWel (gasmotorfabrikater: GE Jenbacher og MWM).

For alle tilfælde er der regnet med følgende mængde biomasse:

Biomasse	Mængde	Tørstofindhold
Slagtekyllingegødning	23.500 ton/år	60 %
Kalk	1.088 ton/år	100 %
Vand	10.000 ton/år	0 %

Ovenstående biomasse giver et gasudbytte på ca. 5.677.000 Nm³ metan/år. Med udgangspunkt i den mængde metan, er det således beregnet, hvor meget elektricitet, varmt vand (kølekreds gasmotor), varmt vand (vha. Economizer) og damp, der kan produceres. Derudover er der beregnet hvor meget energi i form af naturgas, der skal anvendes for at dække dampforbruget ved NiX forbehandling, som ikke dækkes af dampproduktionen på gasmotoren.

	Driftstid, gasmotor	Elproduktion	Varmtvands-produktion, 90°C	Varmtvands-produktion Economizer, total	Varmtvands-produktion, Economizer, fødevand, 8°C-90°C	Varmtvand Economizer , other purpose, ~100°C
1 pcs. Jenbacher 420 - 1487 kWel	8.000 h/år	11.896.000 kWh/år	6.648 MWh/år	1.672 MWh/år	520 MWh/år	1.152 MWh/år
1 pcs. Jenbacher 612 - 1817 kWel	7.611 h/år	13.829.880 kWh/år	6.858 MWh/år	2.093 MWh/år	583 MWh/år	1.510 MWh/år
1 pcs. Jenbacher 616 - 2433 kWel	5.709 h/år	13.890.358 kWh/år	6.869 MWh/år	2.089 MWh/år	645 MWh/år	1.444 MWh/år
1 pcs. MWM TCG 2020 V16 - 1560 kWel	8.000 h/år	12.480.000 kWh/år	6.713 MWh/år	2.248 MWh/år	624 MWh/år	1.624 MWh/år
1 pcs. MWM TCG 2020 V20 - 2000 kWel	6.792 h/år	13.583.120 kWh/år	7.131 MWh/år	2.391 MWh/år	666 MWh/år	1.725 MWh/år

	Dampproduk-tion i gasmo-tor	Gasoverskud	Dampproduk-tion fra natur-gas	Naturgas, metan	Dampfor-brug, NiX	Energi, flash, 140°C-100°C, ~65°C
1 pcs. Jenbacher 420 - 1487 kWel	3.652 MWh/år	435.246 Nm³/år	3.495 MWh/år	398.365 Nm³/år	7.147 MWh/år	4.022 MWh/år
1 pcs. Jenbacher 612 - 1817 kWel	4.519 MWh/år	0 Nm³/år	2.628 MWh/år	299.535 Nm³/år	7.147 MWh/år	4.022 MWh/år
1 pcs. Jenbacher 616 - 2433 kWel	4.522 MWh/år	0 Nm³/år	2.626 MWh/år	299.307 Nm³/år	7.147 MWh/år	4.022 MWh/år
1 pcs. MWM TCG 2020 V16 - 1560 kWel	4.346 MWh/år	254.096 Nm³/år	2.801 MWh/år	319.253 Nm³/år	7.147 MWh/år	4.022 MWh/år
1 pcs. MWM TCG 2020 V20 - 2000 kWel	4.664 MWh/år	0 Nm³/år	2.484 MWh/år	283.122 Nm³/år	7.147 MWh/år	4.022 MWh/år

Ud fra ovenstående tabel fremstår Jenbacher 616 og MWM TCG 2020 V20 som for store gasmotorer at anvende med mindre biomassemængden øges. Til gengæld er Jenbacher 420 for lille en motor. Et valg af MWM TCG2020V20 og Jenbacher 420 kan omvendt set godt være et fornuftigt valg, hvis biomassemængden tilpasses, fordi disse gasmotorer er de største i hver deres serie og er derved en relativt billig motor i forhold til kapacitet.

I nedenstående opstilling er der lavet en detaljeret oversigt over design med en Jenbacher 420 (1.487 kW) gasmotor.

Gas balance		
Methane production from biogas plant	3.408.298 Nm³/year	100%
Consumption gas engine	2.993.441 Nm³/year	88%
Consumption steam boiler	414.857 Nm³/year	12%
Total gas consumption	3.408.298 Nm³/year	100%

Heat balance	
Steam production, gas engine	3.656 MWh/year
Steam production boiler	3.651 MWh/year
Total steam production	7.307 MWh/year
Steam demand for NiX® process	7.307 MWh/year
Excess heat production for sale (95°C)	6.656 MWh/year

Electrical production	11.896 MWh/year

Jenbacher 420	Data	Efficiency
Energy input in gas engine	3.702 kW	100%
Electrical output	1.487 kW	40%
Heat exhaust gas (for steam)	457 kW	12%
Heat (jacket, oil and turbo) (95°C)	832 kW	22%
Operation time	8.000 hours/year	

12 Damplager

Princip for damplager

I forsøget på at gøre NiX forbehandling så økonomisk som muligt, er det nødvendigt at have et "damplager", som potentielt kan udjævne forbrugsforskelle mellem produktionen (røggas fra gasmotorer) – og forbruget, batchvis, i NiX Cooker udstyr.
Rent praktisk er der tale om at etablere et tryksat lager af overhedet vand - som ved efterspørgsel har mulighed for at genfordampe - og således forsyne NiX Cooker udstyr, selvom dampproduktion er midlertidig stoppet.

Design grundlag og forudsætninger

Lagerstørrelsen er principiel afhængig af "dampproduktionens størrelse" under motordrift samt dampforbrugets størrelse under både motordrift og ved motor stilstand.
Med baggrund i at damproduktion altid forløber – fuldlast og med døgngennemsnitlige værdier svarende til årsproduktionsydelse, og at dampforbruget tilsvarende er jævnt fordelt over døgnet – med årsgennemsnitsbelastning – så er der lavet et beregningsark (xls), der klarlægger lagerets nødvendige størrelse.

Nødvendig inddata for beregningsark.

For anvendelse af beregningsark skal følgende oplysninger foreligge:

Årlige antal trykkogte batch (fra Xergi Gascalculation tool)
Ønsket opvarmningstid/batch (fra Xergi Gascalculation tool)
Beregnet årligt dampforbrug til trykkogning (fra Xergi Gascalculation tool)
Ligeledes skal dampproduktionen og trykket hidrørende fra motorer indtastes.
På baggrund af ovenstående beregnes den nødvendige størrelse af hedtvandslager.

13 Biofilter

Motivering for udeladelse

Biofilter var nævnt i oprindelig opdeling – og derfor blot en bemærkning om, at efterfølgende vurdering konkluderede, at det anses irrelevant for biofilterets samlede performance at fokusere særskilt på til ledning fra NiX Cooker flash.

Idet mængden anses som procentuelt lille i forhold til den samlede luftmængde på biofilter på biogasanlæg, er emnet ikke yderligere behandlet i undersøgelsen.

14 Instrumentering

For at forberede designet af NiX forbehandlingsanlæg for nem tilpasning til forskellige typer af biogasanlæg (biomasser) har vi valgt at lave Proces- og Instrumenteringsdiagrammer (PI-diagrammer) for de enkelte delmoduler, som et NiX anlæg kan sammensættes af.
For en nærmere forklaring af hele NiX processen henvises til funktionsbeskrivelsen, der understøttes af PI-diagrammerne.

De fleste af de monterede instrumenter findes som standard i industriel udførelse til de givne tryk og temperaturer.

Hvis, der skal monteres en pH måling i Cookeren, skal der søges mere specielt på pH elektroder og deres montering, da den typiske maksimale driftstemperatur er 135 °C.

Som flow- og densitetsmåler på scrubber systemet kan der sandsynligvis anvendes en masse-flowmåler efter Coriolis pricippet.

I det følgende ses forslag til instrumentering:

1. NiX Koger:
   Temperatur i koger
   Tryk i koger (dobbelt måling)
   Vejeceller
   Effektoptag på omrører
   Damptryk

2. Cyklon og Flashtank:
   Afgangsluften/dampen: temperatur
   Tryk i tank
   Niveau i sump
   Flowmåling på væskecirkulation
   Cirkulerende vand: pH (enten måling direkte i tank eller gennemløb/by-pas til tank)

3. Scrubber
   pH (til styring af svovlsyredosering)
   temperatur
   Ammoniumsulfat koncentration (densitet)
   Væske flow
   Niveau i sump
   Overvågning af differenstryk absorberdel
   Overvågning af differenstryk dråbefang

4. Ud af scrubber
   temperatur

15 Funktionsbeskrivelse

15.1 NiX Pre-treatment system

15.1.1 In general

The Background for this project is to design, test and document a pre-treatment system for using alternative biomass in AD-plants.

Today there is a different reasons why litter from chickens, hens and deep litter from cows hasn´t been used in bigger amounts in AD-plants. Often it is chemical and biological reasons but there are also some mechanical issues to be solved using biomass containing a lot of straw.

By using the NIX pretreatment system it is possible to increase the Biogas potential on some biomass and on other it can be used to strip of ammonia which is inhibitory for the digestion process.

The purpose of the project is to investigate:

The possibility to increase the gas potential for biomass which is difficult to biodegrade by:

- Mechanical processing
- Heat treatment (Cooking)
- Chemical treatment (Alkaline treatment)

The possibility to reduce the risk of floating layers and mechanical problems in the AD plant by:

- Compressing biomass with high-volume, low-density
- Increase absorbency.
- Pressing with liquid addition.
- Grinding / shredding.

Remove inhibitory substances (ammonia):

- NH3 flash on the NIX Cooker.
- Stripping of recirculate.

This following describes the overall automatic operation of the NiX Pre-treatment System.

15.2 Intake system

15.3 Solid Receiving System

The solid receiving system could consist of tip pits and a crane to move biomass from tip pits to storage areas and to feed an intake system above the cooker.
The crane has its own local control system. The local control system is interfaced to the overall control system.

Filling:

The tip pits are filled from truck. When the truck has finished emptying and the gate/port is closed, a crane will move the biomass from the receiving area to the storage area or directly to the intake buffer above the cooker.
The buffer/hopper can be of different type according to the exact type of biomass.
Some hoppers will also mix or even shred the solids.

Emptying:

The crane will dump the biomass into a solids receiving hopper.
Feeding to the NIX Cooker has priority over moving solids to the storage area.
The solids receiving hopper can empty to some pre-treatment such as a Chain Chopper, a Hammer Mill or directly to the NIX cooker.

Emptying to the cooker module:
Emptying to the cooker is activated when called from a feeding sequence. Emptying to the cooker involves the following components:

- Crane started filling solids into the hopper [TAG-No]
- Receiving Hopper unit running [TAG-No]
- Weighing transmitter hopper unit [TAG-No]
- Outlet screws from hopper to hammer mills running [TAG-No]
- Hammer mills running [TAG-No]
- Outlet screws from hammer mills to conveyer running [TAG-No]
- Chain conveyor to cooker running [TAG-No]
- Overfill switch on chain conveyer [TAG-No]
- Speed monitor on chain conveyer [TAG-No]
- Inlet damper on the actual cooker open [TAG-No]
- Weighing transmitter on the actual cooker [F1G1G1TF1]

15.4 NiX Cooker System

15.4.1 Cooker System in General

The Cooker is used for weighing, mixing and heating/cooking of biomass from the pre-storages, recirculate as the liquid fraction from the separation system and lime. After filling the cooker with the biomass and the lime, the mix of biomasses is mixed via a slow running agitator while heated up to the set point. In some occasions the biomass is held at the set point temperature for some minutes in order to obtain the wanted effect or if the biomass is going to be sterilized. After this the flash valve is opened in order to release the pressure slowly through the cyclone, the flash tank and the ammonium scrubber system.
When the pressure is suitable low the discharge valve will open and the cooker will empty all biomass to a mixer tank under the Cooker. In this mixer tank it is possible cool down the biomass or ad some other biomass in order to bring down the temperature. It can be necessary to install digester cooling to secure the right temperature inside the digester.

A number of batches are treated every day. A batch consists of biomass from one or more of the pre storages. The amount of batches, intervals between batches and content in batches are set up via recipes for each primary digester on the SCADA-system. Data for each batch are logged and listed in reports and detailed reports can be printed out. Quantities are registered for the day, the day before and in total.
The Batch Cooker can be filled with several products.

Filling can include:

- Raw material from Solids intake silo line 1(Not pump able) [P1V1V1QM8]
- Liquidized lime line 2 [TAG-No P1V1V1QM9]
- Liquid Biomass pipe line 3 [TAG-No P1V1V1QM10]
- Liquid Biomass pipe line 4 [TAG-No P1V1V1QM11]
- Sludge from cyclone places above Batch Cooker line 5 [TAG-No P1V1V1CM1]

In filling mode must the agitator in the Batch Cooker run forward [P1V1V1HW1] and the pressure must be below 0.3 Bar [P1V1V1BP3]

Filling from Solids intake silo line 1 can involve the following components:

- Crane started filling solids into the hopper [TAG-No]
- Receiving Hopper unit running [TAG-No]
- Weighing transmitter hopper unit [TAG-No]
- Outlet screws from hopper to hammer mills running [TAG-No]
- Hammer mills running [TAG-No]
- Outlet screws from hammer mills to conveyer running [TAG-No]
- Conveyor to cooker running [TAG-No]
- Overfill switch on chain conveyer [TAG-No]
- Speed monitor on chain conveyer [TAG-No]
- Inlet damper on the actual cooker open [TAG-No]
- Weighing transmitter on the actual cooker [P1V1V1TF1]
- Biomass inlet valve [P1V1V1QM8]

Filling from Lime from pipe line 2 involves the following components:

- Weighing cell [P1V1V1TF1]
- Pump [TAG-No]
- Fluid valve [P1V1V1QM9]

Filling from Liquid Biomass pipe line 3 involves the following components:

- Weighing cell [P1V1V1TF1]
- Pump [TAG-No]
- Biomass inlet valve [P1V1V2QM10]

Filling from Liquid Biomass pipe line 4 involves the following components:

- Weighing cell [P1V1V1TF1]

- Pump [TAG-No]
- Fluid valve [P1V1V1QM11]

15.4.2 Cooker Module No. 1

The below description is for Cooker module No. 1

15.4.2.1 Cooker Module, Batch Sequence

Every batch is treated according to a predefined sequence.

The feeding sequence consist of the following steps:

1. Waiting for next batch
2. Starting new batch
3. Pre.-mixing
4. 1. intake of biomass
5. 2. intake of biomass
6. 3. intake of biomass
7. 4. intake of biomass
8. Steam injection
9. Waiting for temperature set point
10. Heating in time (possibility for sterilization)
11. Flash
12. Emptying
13. Finishing batch

Waiting for next batch

In this step the module is ready to start a batch and is waiting to be called from the digester.

Starting new batch

In this step data for the recipe to be executed is read from the digester recipe.

Pre-mixing

In this step pre.-mixing in relevant pre.-tanks are made to secure that the biomass is mixed before pumping in. The lime will also have been blended with water before pumping in. This will normally always have been done before the batch is started.

1. - 4. Intake of biomass

In these steps up to eight different biomasses can be taken in. Each intake consists of a biomass type and amount of biomass. Components involved in pumping in to the Cooker module are described under emptying of the individually pre.-tanks.

Steam Injection

In this step the system are opening the steam control valve so the biomass can be heated up to the temperature set point defined in the recipe.

The heating system involves the following components:

- Heating Cooker module 1 with direct steam injection. Open valve [P1V1V1TQN1]
- Biomass temperature in Cooker module [P1V1V1BT1]

Waiting for temperature set point
In this step the system are waiting for the biomass to be heated up to the temperature set point defined in the recipe. Components involved in heating are described in chapter “Cooker modules, heating system”.

Heating in time
In this step the temperature is kept at the specified temperature set point in the time specified in the recipe.
In sterilization condition the biomass must be treated at 2 Bar and 133°C in 20 minutes. Before emptying the Batch Cooker the vapour must be released.

Sterilization condition includes the following components:

- Agitator in Batch Cooker [P1V1V1HW1]
- Direct steam valve [P1V1V1QN1]
- Batch Cooker pressure control transmitter [P1V1V1BP3/4]
- Batch Cooker temperature control transmitter [P1V1V1BT1]

Flash
The Steam pressure will be released by a control valve keeping a certain flow through the Cyclone and the Flash tank and further on into the Ammonia Scrubber. The set point for the flow will depend on the capacity in the flash tank and the Scrubber but also secure that only a minimum of material from the cooker is carried out via the flash. In this step the steam is cooled down in the flash tank to the temperature requested in the Ammonia Scrubber. The process in the ammonia scrubber will bring up the temperature in the air supposed to be cleaned in a bio filter so it will be necessary to install an air cooler after the ammonia scrubber.

Emptying
The Batch Cooker can only empty to the Mixing tank under the Cooker
Emptying is activated when Batch Cooker has completed heating or sterilization sequence and the pressure in the chamber is below a set point (0.5 Bar).
Components involved in emptying are described in the following.
The outlet valve is opened when batch is ready.
The agitator is reversing under emptying.

Emptying feeding module 1 to mixer tank involves the following components:

- Pressure under set point [P1V1V1BP3]

- Outlet valve on module open [P1V1V1QM1]
- Agitator running in Outlet mode. [P1V1V1HW1]
- Outlet valves on other modules closed
- Weighing transmitter on Cooker module 1 [P1V1V1TF1]
- Level transmitter in the mixer tank [TAG-No]
- Max level switch in in the mixer tank [TAG-No]

Finishing batch
In this step data for the batch are logged. The logged data includes the below listed:

- Batch-ID [Unique number]
- Set point and actual value for pre mixing time
- Media type [Pre tank no], set point [kg] and actual value [kg] of amount for 1. intake
- Media type [Pre tank no], set point [kg] and actual value [kg] of amount for 2. intake
- Media type [Pre tank no], set point [kg] and actual value [kg] of amount for 3. intake
- Media type [Pre tank no], set point [kg] and actual value [kg] of amount for 4. intake
- Heating set point [℃]
- Heating time [minutes]
- Sterilisation done [Yes/No]
- Digester [No]
- Batch time [Minutes]
- Batch start time [date and time]
- Batch stop time [date and time]

Cooker Module Mixing
The end to end mounted agitator is released for operation when the weight in the module is higher than a specified set point. The agitator is frequency converter controlled. The agitator speed is specified via the recipe.

Mixing in module No. 1 involves the following components:

- Agitator started [P1V1V1HW1]
- Weighing transmitter on feeding module [P1V1V1TF1]

15.4.3 Cyclone and Flash Tank

The system involves a cyclone to take out bigger particles in the flash and a flash tank to cool the steam before it is brought to the scrubber.

The cooling system must provide a cooling spray fog inside the tank so all flash steam is condensed. The cooling system involves the following components:

- Cooling pump [P1V1V1GP2]
- Temperature transmitter [P1V1V1BT2]

- Mixing valve [P1V1V1QMx]

The level in the Flash Tank is controlled and excess liquid will either be discharged to end storage or in some systems the separated digestate, which is going to be used for dilution of the solids input to the cooker, will be used as cooling media for the flash tank. The energy obtained by this will save some steam production for the next batches.

15.4.4 Ammonia Scrubber

The ammonia scrubber will remove the ammonia by adding sulphuric acid and water. The process will run weak acidic (app. pH 5) and make ammonium sulphate, which will be removed from the process. The ammonium sulphate will be removed on basis of the density of the liquid inside the scrubber. The air from the scrubber will probably have to be cooled before entering the bio filter.

15.5 Heat System

15.5.1 In General

The steam supply to the NiX cooker is normally produced on the exhaust gas from biogas engines or on a steam boiler with a gas fired burner. Steam supply can also be produced on a wood chip boiler or a straw boiler. The gas engines also normally produce the low temperature (<96°C) energy for process and building heating for the AD-plant

15.5.2 Gas Engine

The gas engine has its own local control system which handles the operation and internal auxiliaries. Hardwired alarms and states from the gas engine control panel are connected to the overall control system. Also a profibus connection with detailed data is connected to the overall control system.

Start/Stop of the gas engine is described in section “Gas System”

Operation of gas engine No. 1 involves the following components:

- Start gas engine No. 1 [U1G1G1GA1]
- Inlet temperature to the engine [U1G1G1BT3]
- 3-way valve controlling the inlet temperature to the engine [U1G1G1QN1]
- Circulation pump started and controlling the forward temperature [U1G1G1GP1]
- Forward temperature from the exhaust boiler [U1G1G1BT10]
- Intercooler started [U1G1G1EP3]
- Dump cooler ready / started [U1G1G1EP2]
- Room ventilation ready / started [A1W6W1GQ1], [A1W6W1GQ2]
- Forward temperature from heat production unit [U1G1G1BT1]
- High pressure switch accumulation tank [U1C1C1BP1]
- Low pressure switch accumulation tank [U1C1C1BP2]

Gas Engine Intercooler

The gas engine intercooler is started when the gas engine calls for auxiliary equipment.
The intercooler is keeping the intercooler water temperature at a specified set point by pumping the water through a dry air cooler with 3 frequency regulated fans.

Operation of the intercooler involves the following components:

- Start gas engine [U1G1G1GA1]
- Intercooler water temperature [U1G1G1BT11]
- Intercooler circulation pump [U1G1G1GP3]
- Pressure switch low pressure intercooler [U1G1G1BP2]
- Dry air cooler fans [U1G1G1GQ1], [U1G1G1GQ22], [U1G1G1GQ23]
- 3-way valve controlling the inlet temperature to the engine [U1G1G1QN2]

Gas Engine Dump cooler

The gas engine dump cooler is started when the return water temperature to the gas engine is higher than a specified set point.
When the water temperature has been lower than the set point for a specified time, the dump cooler is stopped.
The dump cooler is keeping the return water temperature at a specified set point by pumping the water through a dry air cooler with 20 frequency regulated fans.
Operation of the dump cooler involves the following components:

- Start gas engine [U1G1G1GA1]
- Gas engine return water temperature [U1G1G1BT8]
- Dump cooler circulation pump [U1G1G1GP2]
- Dump cooler forward temperature [U1G1G1BT6]
- Dump cooler return temperature [U1G1G1BT5]
- Pressure switch low pressure intercooler [U1G1G1BP1]
- Dry air cooler fans [U1G1G1GQ2-21]

Gas Engine Room Ventilation

Ventilation is started on a specified speed set point when requested from GEJ.
Later GEJ will send a signal, releasing fans for regulating.

Room ventilation involves the following components:

- Engine room ventilation fan started [A1W6W1GQ1, A1W6W1GQ2]
- Engine room temperature [A1W6W1BT1]

15.6 Steam system

15.6.1 In General

The steam to the NIX Cooker is produced on the steam boiler, which is fired by the exhaust from the gas engines, via tubes installed at the top of the boiler and/or via the gas fired burner.

15.6.2 Steam boiler

The steam boiler/burner has its own local control system which handles the operation and internal auxiliaries. Hardwired alarms and states from the burner control panel are connected to the overall control system. Also a profibus connection with detailed data is connected to the overall control system.
The burner can operate on biogas or natural gas. This will be selected by the operator on the control system.
If biogas is selected the burner will be released for biogas operation when the biogas level is above a specified set point. Likewise will be burner be blocked for operation when the biogas level goes below a specified set point.

If the steam pressure gets below a certain value, the burner will be started on biogas unless it's blocked for operation.

Operation of gas burner/boiler involves the following components:

- Boiler ready signal
- Feed water pumps
- De-aerator
- Condensate tank
- RO Plant
- Boiler pressure
- Steam heat exchanger system for hot water
- Heat exchanger for feed water
- Room ventilation ready / started (burner operation)
- Blow down system

15.6.3 Heat Accumulation Tank

When the heat consumption is less than the heat production the heat surplus is stored in the accumulation tank.

Operation of the heat accumulation tank involves the following components:

- Indication of heat content via temperatures in tank [U1C1C1BT1 - 6]
- High pressure switch accumulation tank [U1C1C1BT1]
- Low pressure switch accumulation tank [U1C1C1BT2]

15.6.4 Main Heat Distribution Pump

The heat is distributed to the heat consumers on the biogas plant via the distribution pump [U1G2G1GP1. The pump is controlled by a frequency converter. The pump is running according to the differential pressure between forward and return pressure.
The three pumps are running in master/slave operation. One pump is chosen as the master pump and the others are then slave pumps. The master pump is always running. The slave pumps will start if the pressure goes below specified set points.

Operation of the main heat distribution pumps involves the following components:

- Heat distribution pump started [U1G2G1GP1].
- Pressure transmitter after the pumps [U1G2G1BP2]
- Return pressure transmitter [U1G2G1BP3]

The following measurements are made:

- Flow temperature transmitter [U1G2G1BT1]
- Return temperature transmitter [U1G2G1BT2]
- Flow pressure transmitter before pump [U1G2G1BP1]
- Flow pressure transmitter after pump [U1G2G1BP2]
- Return pressure transmitter [U1G2G1BP3]

15.6.5 Heat Metering

The consumed heat for feeding module 3 is measured with the heat meter F1G1G3KF1.
The consumed heat for feeding module 4 and 5 is measured with the heat meter F1G1G0KF1.

15.6.6 Expansion System

The heat system has included an expansions system [U1C2C1CM1]. The expansion system has a local control system. Alarms from the expansion system are connected to the control system.

15.6.7 Water Softening Plant

The heat system is filled and topped up via a water softening plant [A1H2H1HQ1], which is also generating softened water for the RO plant, which is producing feeding water for the steam plant. The plant has a local control system. Alarm from the plant is connected to the control system.

15.6.8 Hydro-X Unit

The heat system has included a hydro-x unit [A1H2H2HQ1] which controls the pH in the heat water. The system has a local control system. Signal for pH in the water is connected to the overall control system.

15.6.9 Feed water system

The boiler control system calls for water from the RO plant, according to the level in condensate tank [U1E1E0CM2]. The water is pre-heated by heat exchanger [U1E1E0EP1] and controlled by control valve [U1E1E0QN2] according to the water level in the boiler.
Water from the condensate tank is pumped to the de-aerator by pump [U1E1E0GP3], controlled by level measurement in the de-aerator.

Pumping from the condensate tank to the de-aerator involves the following components:

- Condensate tank level measurements [U1E1E0BL1, U1E1E0BL2, U1E1E0BL3, U1E1E0BL4]
- Transfer pump [U1E1E0GP3]
- De-aerator level measurements [U1E1E0BL5, U1E1E0BL6]

15.6.10 Chemical dosing system

The boiler has a chemical dosing system, which adjust the level in the condensate tank to ensure the feed water quality.

15.6.11 De-aerator

In order to ensure no oxygen in the feed water to the boiler a de-aerator system is also installed.

16 Bygningslayout

Eksempler på Bygningslayout og Sitelayout

17 Halm

17.1 Generel problembeskrivelse

Der er store tilgængelige halmressourcer verden over der kan leveres til rimelige priser. Halm er en udmærket ressource til lavtemperatur (under 100 grad.)varmeforsyning, men har vist sig yderst vanskelig til el fremstilling pga. slagge og tæringsproblemer. Der satses således meget på halm som brændstof til 2. generations bioethanol.

Halm er energimæssigt et god biomasse til biogasfremstilling, men er yderst svær at håndtere som følge af et stort iltindhold der giver lav massefylde og en stor opdrift og dermed store flydelagspro-blemer. Lignin beskyttelsen i halmstrå gør endvidere at vandoptageligheden er meget langsom, hvorfor flydelags problematikken er langtidsholdbarheden.

Det er denne langtidsholdbarhed der nedbrydes gradvist, når halm ligger som dybstrøelse i urin og afføring, samtidig med at denne mases ind i halmen.

Det er velkendt i landbruget, at fordøjeligheden af halm kan øges markant ved tilsætning af ammoniak (halmludning).

Brug af halm til biogas kræver derfor en for bearbejdning, der neddeler eller opløser halmen, så sugeevnen øges markant.
Dette kan ske mekanisk (formaling, bankning, tryksætning), kemisk
(baser, syrer osv.) eller termisk (kogning) eller i kombination med førnævnte med biologisk (enzymer, mikroorganismer).

Halm indeholder endvidere en relativ stor del sand/sten og metalstumper, som behandlingsteknologien skal kunne håndtere, uden at det medfører omfattende drift- og vedligeholdelsesomkostninger.

17.2 Proces

Halm er tidligere blevet beskrevet som en meget tungt nedbrydelig biomasse med udbytter på under 200 Nm3 CH4/ton VS. De seneste års øgede fokus på halm til biogas har medført en del forsøgsafprøvninger parallelt med Xergis egne, og dette har dannet grundlag for en revideret opfattelse. Rapporterede udbytter ligger nu typisk på over 250, og Xergis egne data viser et niveau på 300 – 320 og med en relativ hurtig nedbrydningsprofil. Forbehandling ved formaling hhv. NiX-behandling og kombination af disse to metoder har vist udbyttestigninger på hver ca. 10 % og med næsten fuld additiv effekt. Forbehandling har dog især effekt på nedbrydningshastigheden, som øges meget markant. Beregnede CSTR-udbytter med 15+15 dage termofil/mesofil øges fra et niveau omkring 250 til omkring 300 ved af forbehandlingerne og kombinationen giver ca. 340. (Se afrapportering for WP 3)

Halm indeholder ikke ret meget kvælstof i forhold til energiindholdet og kan derfor anvendes til at balancere biomasser med lavt TS og højt kvælstof. Et oplagt koncept er derfor kombinationen af svinegylle og halm.

17.3 Bearbejdning og neddeling

Formaling til pulver med tysk formalings maskine er udvalgt som den mest lovende teknologi, at undersøge nærmere efter succesfyldt indledende formalingstest med efterfølgen feasibitity beregning.

17.4 Formaling af halm ved Jäckering

Under besøget hos Jäckering 30/5-2012 blev der formalet hvedehalm i tre forskellige grovheder – Fin, Mellem og Grov.

Partikelstørrelser for de tre formalinger er vist nedenunder:

	<1000 µm	<500 µm	<200 µm
Fin	100%	95%	75%
Medium	99,9%	89%	56%
Grov	97%	78%	36%

Kapacitetstest

Der blev foretaget el-forbrugsmålinger for hver af de 3 formalingsgrader. Under forsøgene forsøgte teknikerne at fastholde maximalt ampereforbrug for ultrarotoren i et afmålt tidsinterval ved at håndføde i en hastighed så el-forbruget gennemsnitligt lå på 150 A for rotoren. Den formalede mængde halm blev derefter vejet for at bestemme maximal kapacitet ved den givne formalingsgrad.
Det var kun Ultra-rotorens forbrug der kunne observeres direkte. De to andre komponenters forbrug beror på Jäckerings udtalelser. Der var et display i hallen der viste forbruget for hele hallen, under testen. Den viste et forbrug der svingede fra ca. 90 kW – 140 kW under testen (se evt. video fra Henrik Hansen).

Den installerede kapacitet i systemet:

	Installeret kapacitet	Forbrug under test
Ultra-Rotor	75 kW	Ca. 60 kW (målt)
Ventilator	45 kW	12 kW (opgivet af Jäckering)
Filter med rotationsventil	1,1 kW	1,1 kW (opgivet af Jäckering)

Formalingsgrad	**Kapacitet**
Fin	250 kg/time
Medium	270 kg/time
Grov	310 kg/time

Beregning af specifikt effektforbrug:

	Registreret elforbrug				Oplyst elforbrug		
	Effektforbrug	Kapacitet			Effekt-forbrug	Kapacitet	
	kW	kg/h	kWh/t		kW	kg/h	kWh/t
Fin	140	250	560		72	250	288
Medium	140	270	519		72	270	267
Grov	140	310	452		72	310	232

Testen viste et markant højere elforbrug end oprindeligt oplyst (3-5 gange og tilsvarende viste kapaciteten sig at være tilsvarende lavere.

Den umiddelbare konklusion er, at anlægsomkostninger samt vedligeholdelsesomkostninger nok bliver det dobbelte af det forudsatte i feasibility studiet, lige som elforbruget stiger med en faktor 3 eller mere. Det vurderes at der derfor ikke er økonomi i dette koncept.

17.5 Alternative løsninger

Extrudering har været nævnt som en anden forbehandlingsmetode til Halm. Men test gennemført at Xergi ved testcenteret i Folum viser at ekstruderens kapacitet og elforbrug er alt for højt til at dette kan gøre rentabelt.
Xergi har idriftsætter i 2013 et biogasanlæg med Hammermøller som forbehandling af dybstrøelse og halm, og vil i den forbindelse evaluere hammermølle teknologiens muligheder for forbehandling af halm.

For en NIX behandling af halm skal der findes en mekanisk løsning på at få født tilstrækkeligt halm ind i batchkogeren. Xergi ser her muligheden for at anvende en kædeneddeler som er nærmere omtalt under afsnit 6 Indfødning af biomasser.

18 Demonstrationsanlæg til Ribe Bioenergi

18.1 Generelt

På basis af Xergi´s udviklingsarbejde omkring de enkelte komponenter i NIX konceptet og den principielle systemopbygning har Xergi i samarbejde med de ansatte på Ribe biogasanlæg og rådgivningsfirmaet Gascon projekteret et NIX demonstrationsanlæg der kan indpasses i faciliteterne på Ribe biogasanlæg.

Processen har været kompliceret pga.at de ideelle testfaciliteter først er tilstede efter at Ribe har udbygget deres biogasanlæg med ny reaktorkapacitet samt faststofdoseringsanlæg. Ribe biogasanlæg har søgt om anlægstilskud til disse arbejder.

Der er udarbejdet et færdigt design der passer ind i et udbygget Ribe biogasanlæg, med layouttegninger energi og masseballancer, flow diagrammer og komponentlister, ligesom projektet er prissat.

Der søges om EUDP midler til at støtte demonstrationsprogrammet herunder den efterfølgende målefase. Demonstrationsanlægget omfatter bl.a. den nyudviklede batchkoger omtalt nærmere i denne rapport.

Nedenstående vises leveringsomfanget for demonstrationsprojektet fordelt mellem parterne.

18.1.1 Anlægsbeskrivelse

Anlægsdelene er beregnet til indfødning af faste biomasser såsom fintsnittet majsensilage (Maksimalt 20.000 ton/år) samt hygiejninsering af maksimalt 22.000 ton af flydende industri biomasser pr. år. Det forventes at de flydende biomasser ikke indeholder materialer, der er større end kravene til hygiejnisering ved 70°C (12mm).

Det forventes, at den mængde gylle der er nødvendig, for at lave batches med en acceptabel tørstofprocent samt den biomasse der skal hygiejniseres, forvarmes til mindst 25°C i eksisterende gylle/gyllevarmevekslere samt at indpumpnings flowet til doseringsmodulerne er mindst 80 m^3/h.

18.2 Leveringsomfang for parterne.

		Xergi leveringsomfang	**Ribe Biogas leveringsomfang/Leveringsgrænse**
1.	Modtagelses faciliteter	1 stk. Faststofmodtager, type SF-S-XS-60, Volumen 60 m^3 med 2 stk. snegleudtag.	
		2 stk. Snegle for indfødning i doseringsmoduler.	
2.	Forbehandling af biomasser	2 stk. Xergi FlexFeed® doseringsmoduler, type FM-S-HD-20-3, Volumen 20 m^3	Kunden leverer varme til de 2 moduler (1 MW, 95 °C, 20 m^3/time)
		1 stk. Skumtank, type FT-F-XS-30, volumen 30 m^3. Tanken er forbundet til FlexFeed® modulerne.	
3.	Rørinstallationer	1 stk. Rørinstallationspakke, se bilag 1.1, dokument: *Xergi Pipe Installation Package*	• Alle rør afsluttes i flanger på moduler. Der er således ikke inkluderet rørforbindelser til eksisterende installationer hverken for biomasse, varme, trykluft eller andet.
4.	Elektriske installationer	1 stk. Xergi El installationspakke, se bilag 1.1, dokument: *Xergi Electrical Installation*	• Der er kun interne installationer på moduler og udstyr leveret af Xergi inkluderet. Forbindelser til eksisterende anlæg samt forsyningskabler er ikke inkluderede • Telefon/internet: Skal bestilles og leveres af kunden inkl. tilslutningsboks monteret på væggen i tavlerummet.
5.	Elektriske tavler	1 stk. Xergi El tavle pakke, se bilag 1.1, dokument: *Xergi Electrical Panels*	• Det forudsættes, at der er tilladelse til nulling.

		• Der er kun inkluderet tavler for anlægsdele leveret af Xergi.
6. Kontrolsystem	1 stk. Xergi Kontrolsystem, se bilag 1.1, dokument: *Xergi SCADA system*	• Kontrolsystemet omfatter kun udstyr leveret af Xergi. Kommunikation med eksisterende SRO er forudsat at kunne foretages via Siemens Profibus DP Protokol. Omfang af signaludveksling aftales med bygherres SRO-leverandør. • El tavler og kontrolsystem leveres til installation i separat rum i umiddelbar nærhed af doseringsmodulerne. SRO systemet leveres desuden med mulighed for en fjernbetjent operatørstation.
7. Anlægsarbejder og bygninger	Tilbuddet omfatter ikke anlægsarbejde, bygningsarbejde, udgravning og fundering.	
8. Materialer og installationer under opførelse	Kran til aflæsning og installation af Xergi moduler.	Forberedelse af byggeplads, indhegning omkring byggepladsen og forberedelse af midlertidige veje til byggeperioden anlægges af bygherre. Adgangskontrol og vagtservice udenfor arbejdstiden varetages af bygherre. Bygherre leverer vand og elektricitet gennem hele byggeperioden ligesom bygherren sørger for bad og toiletfaciliteter. Affaldshåndtering håndteres af bygherren.
15. Test og idriftsættelse	Detaljeret design, opsyn, test og idriftsættelse er inkluderet. • Koldtest • Varmtest • Idriftsættelse • Ydelsestest • Brugermanual	Bygherre skal levere og betale for: Elektricitet Varme Vand Biomasser

	• Dokumentation	Kemikalier
	Relevante tegninger og teknisk information til myndigheder.	Myndighedsgodkendelse inklusiv dokumentation til myndigheder og forhandlinger med myndigheder.
	Træning af bygherres personel på biogasanlægget gennem idriftsættelsesperioden.	Maksimalt 2 dages træning er inkluderet.

19 Markedsevaluering og -planer for NiX® teknologien.

Markedssegmenter:

- Poultry Power®:
  - Fjerkræproducenter.
  - Æg producenter.
- Manu Power®
  - Blandede N holdige biomasser (fjerkræ+ afvandet fiber+restfraktion biogasanlæg etc.).
  - Efterforgasning af fiberfraktion på større biogasanlæg.

Forretningsmodeller:

Modeller for håndtering af forskellige markeder.

1. Turnkey biogas projekter med NIX linje(R)
2. Forbehandlingssystem + licens + design+support
3. Licens-, design og supportpakke
4. Licenspakke pr. anlæg eller pr behandlet tons løbende.

Geografiske markeder:

- **Nordirland** drevet af 3 ROC betaling i alt ca. 1,6 kr./kwh op til 5 MW. Afsætningsproblemer for kyllingemøg
- **UK** drevet af 2 Roc betaling i alt ca. 1,2 kr/ kWh.
- **Italien** speciel høj tarif ved mere end 30 % N fjernelse tarif på 1,85 kr/kwh. <1 MW og 1,46 kr/kwh <5 MW. (N fjernelse 22 øre/kWh eller DKK 5,3 mio/ år for 3 MW).
- Identifikations program for øvrige lande.
  -Holland, Belgien

Markedførings aktiviteter:

- Udarbejde markedsføringsmateriale (næste slide)
- Deltage i udvalgte brancheseminarer.
- Brug af ”LinkedIn” præsentation.
- Partner/kunde identifikation via:
  - internet

- teknologi net (agro business park)
- brancheorganisationer

Markedsførings-/salgs materiale:

- Brochure rettet mod fjerkræsegment.
- Standard "notat" (til udlevering til interesserede kunder der ønsker at gå dybere) med gennemgang af koncept via principskitser, billeder og tabeller med massebalance.
- Feasibility beregningsopsætning i gaskalkulation 3 MW i UK 1 MW og 3 MW Italien. Udleveringsnotat om feasibility beregning.
- Hjemmeside præsentation
- PP præsentation

Kommercielle milestones Poultry Power® 2014:

• Samarbejds aftale investor for UK underskrevet	Q1
• Turnkey contract for NI	Q1
• Identifikation af øvrige markeder og handleplaner	Q1
• Identifikation af kommercielle partnere Italien	Q1
• Kontrakt med partner i Italien	Q2
• Identifikation af partnere i 3. land	Q2
• Første projektudviklingsaftaler i Italien	Q3

Kommercielle milestones Manu Power® 2014:

• Demoanlæg (EUDP) med ny batch koger i drift	Q2
• Videbæk Biogasanlæg anlægsstart	Q3
• Identifikation af øvrige markeder og handleplaner	Q3
• Identifikation af kommercielle partnere Frankrig	Q4